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The effect of heat treatments on the microstructure of Inconel 718 produced by additive manufacturing: a comparative study on LPBF, LDED and casting

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The effect of heat treatments on the microstructure of Inconel 718 produced by additive manufacturing: a comparative study on LPBF, LDED and casting

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TURIN 2021 This work is dedicated to my dog, Toquinho, who was a great company during the writing of this work, and did not live to see its completion. It is also dedicated to my parents, who always seek to support and encourage me to go further and beyond.

ABSTRACT

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Additive manufacturing (AM), popularly known as 3D printing, has been increasingly popular in recent times, drawing attention from both academia and industry. It has been developing at a fast pace, and in recent years it has left its original purpose of being a prototyping technology to become a true manufacturing technique, suitable for the production of end parts, especially through metal AM. The industry that currently uses AM to the greatest extent is the aerospace sector, in which cost is not a strong constraint, and the benefits of additive manufacturing are profoundly valuable (optimized design, weight reduction, internal channels for cooling, improved mechanical properties). In this industry, a key material is Inconel 718, a precipitation hardened nickel superalloy used on turbine blades and disks, due to its extraordinary mechanical properties and high temperature resistance. Inconel 718 is difficult to process through traditional operations, due to its high liquidus temperature, high hardness and yield strength and low thermal conductivity, making it a great candidate for AM routes. For metal AM, some of the most employed processes are laser powder bed fusion (LPBF) and laser powder directed energy deposition (LDED), each with different benefits and challenges. Regardless of the manufacturing technique, the condition of the parts in the asproduced state is not ideal. Thermal treatments are necessary in order to optimize the microstructure, dissolve brittle precipitates and promote the formation of strengthening precipitates, relieve residual stresses and obtain better mechanical and metallurgical properties. The most commonly used heat treatment for AM Inconel is the standard heat treatment AMS 5662 for wrought Inconel 718. In this work, Inconel 718 parts produced by LPBF, LDED and casting (the traditional process for this alloy, for comparison purposes) are submitted to different heat treatments (no heat treatment, solubilization, solubilization plus single aging and solubilization plus double aging) based on AMS 5662 and characterized in terms of microstructure and microhardness, in order to evaluate whether the AMS 5662 heat treatment is suitable for parts fabricated through AM. The effect of the heat treatments on the microstructure and microhardness is assessed and compared with the as-produced state, through OM, SEM, EDS and microhardness tests. Both AM techniques yield superior hardness in comparison to cast samples, and LPBF leads to higher numbers than LDED, due to the finer microstructure. Despite the high cooling rates, microsegregation of Nb and Mo was detected even in LDED and LPBF samples. Aging treatment successfully promoted increased hardness when compared to the non-treated samples, but the solubilization treatment failed to dissolve Nb-rich phases such as Laves and delta, and failed to eliminate microsegregation. As a consequence, the second aging proved ineffective, with inconsistent effect on hardness, due to the low Nb availability. Samples produced with different methods were affected differently, and the LDED sample reached 95% of the hardness of the LPBF sample after the full heat treatment. These results highlight the importance of the starting microstructure and evidences the need of developing an adequate heat treatment specifically tailored to additive manufacturing processes, since the standard heat treatment for wrought parts did not produce the desired results on AM parts.

Keywords: Additive manufacturing; 3D printing; LPBF; SLM; LDED; DED; PBF; IN718; Inconel 718; Nickel alloys; Heat treatments; Thermal treatments.

RIASSUNTO

Riassunto della Tesi di Laurea Magistrale

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"The effect of heat treatments on the microstructure of Inconel 718 produced by additive manufacturing: a comparative study on LPBF, LDED and casting"

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1. Introduzione

Inconel 718 è una superlega di nichel con un'eccezionale resistenza alle alte temperature ed elevata resistenza alla corrosione. Mantenendo un'alta resistenza meccanica (carico di snervamento e carico di rottura) anche ad altissime temperature, e avendo un'elevata resistenza al creep, questa lega è utilizzata soprattutto nell'industria aerospaziale, come pale e dischi di turbine jet in aerei ed anche in turbine a gas per la generazione di energia. Il materiale ha una temperatura solidus pari a 1255 °C e può essere utilizzata fino ai 700 °C [33].

Tuttavia, lo stesso insieme di proprietà che rendono la lega idonea per applicazioni in ambienti sottoposti ad alte temperature o altamente corrosivi, fanno sì che la lega sia difficile da processare e formare con i metodi convenzionali. Ad esempio, l'alto carico di snervamento anche ad elevate temperature rende le operazioni di forgiatura difficili da eseguire, essendo necessari carichi e temperature molto alti per deformare il materiali come si vuole. La fresatura o la tornitura sono anche metodi non-ottimali per il processamento di Inconel 718, visto che l'elevata durezza e la bassa conducibilità termica della lega fanno sì che i tool taglienti della fresatrice o tornitrice subiscano un riscaldamento esagerato durante la lavorazione, il che diminuisce la vita dell'utensile e aumenta il costo dell'operazione. Tra i processi tradizionali, la fonderia è quello più adatto, nonostante l'alta temperatura richiesta (in paragone all'aluminio, per esempio). Comunque, i processi di fusione e colata risultano pezzi con bassa resistenza meccanica, poiché non si sfrutta l'incrudimento. Inoltre, oggetti fatti in Inconel 718 frequentemente hanno geometrie complesse e dettagli intricati, che sono difficili da ottenere tramite i metodi tradizionali, particolarmente senza l'uso di fresatura [67].

Le pale di turbine, per esempio, hanno canali interni per possibilitare il raffreddamento del pezzo durante il suo uso. In questo scenario, la manifattura additiva si presenta come un'alternativa interessante per la produzione di parti in Inconel 718. La manifattura additiva (AM, per *Additive Manufacturing*) si basa sulla costruzione di pezzi in modalità bottom-up, tramite l'addizione di strato dopo strato, da una piattaforma o un substrato fino ad arrivare all'oggetto finale con la forma voluta, definita da un file CAD. L'AM consente la produzione di parti con geometrie estremamente complesse (inclusi canali interni) praticamente senza incrementi nei costi (rispetto alla produzione di parti con geometrie semplici tramite lo stesso metodo). Inoltre, siccome è un metodo additivo, si riesce ad addizionare materiale solo là dove sia necessario e, di conseguenza, si riesce a ridurre la massa dei componenti, un vantaggio particolarmente gradito in applicazione nelle quali i componenti si muovono, con la spesa di energia, normalmente da un carburante. In questi casi, la riduzione massica porta a un risparmio energetico e quindi economico [69].

Le più importanti limitazioni dell'AM sono l'alto costo delle attrezzature e polveri, la ridotta gamma di leghe commercialmente disponibili, e la bassa velocità di produzione. Nonostante ciò, per componenti in Inconel 718, un materiale comunque costoso, rivolti ad applicazioni avanzate come quelle nell'industria aerospaziale o energetica, in che il costo non è un fattore limitante dei progetti, e che richiedono pezzi il più leggeri possibile, con geometrie complesse, l'utilizzo di tecniche AM viene giustificato dai suoi vantaggi. Infatti, l'Inconel 718 è uno dei tre materiali più utilizzati nella manifattura additiva [22].

L'AM è una famiglia di processi basati sulla produzione di pezzi in metodo additivo, ma ci sono differenti tecnologie dentro questa categoria di processi. Per i metalli, le due tecnologie più spesso utilizzate sono la fusione a letto di polveri (PBF, per *Powder Bed Fusion*), responsabile per l'85% delle vendite di macchine AM in 2020 (in fatturato), e la deposizione diretta (DED, per *Directed Energy Deposition*), responsabile per l'8% dei ricavi dalle vendite di macchine per l'AM in 2020 in tutto il mondo [22].

La tecnologia LPBF prevede l'addizione di strati di polvere e la successiva fusione selettiva (cioè, solo nelle regioni che dovranno consolidarsi per formare il componente desiderato) di ogni strato. Un utensile ricopre la piattaforma dal primo strato di polvere, poi il laser fonde lo strato in modo a consolidare una sessione trasversale dell'oggetto in costruzione. Lo strato successivo viene spalmato sullo strato precedente, e poi fuso nello stesso modo, seguendo la forma del pezzo che si desidera realizzare. Il processo avviene dentro una camera sotto vuoto oppure con atmosfera controllata di gas inerti (solitamente Ar). Con questa tecnologia, si producono pezzi con alta precisione dimensionale, però in un modo lento. Così, questo processo è più adatto alla produzione di componenti piccoli con geometria complessa e dettagli fini. La sorgente di calore può essere un laser o un fascio di elettroni. La sorgente a laser è quella più utilizzata, e in questo caso il processo viene chiamato LPBF (che sta per *Laser Powder Bed Fusion*) [21].

La tecnologia DED, invece, prevede la deposizione di metallo direttamente su un substrato, attraverso un ugello che soffia la polvere (con l'utilizzo di un gas carrier, di solito l'Ar) verso la zona fusa del substrato, che viene fusa dall'ugello stesso contenente un laser. Questa modalità di DED con la sorgente laser ed il metallo sotto forma di polvere viene chiamata LDED, ed è la forma più comune della tecnologia. La LDED lavora con un tasso di deposizione più elevato rispetto alla LPBF, in modo che sia possibile l'ottenimento di componenti più grandi in modo più veloce. Questo è possibile grazie a un maggiore diametro del laser e a polveri più grosse (in paragone a quelle utilizzate in LPBF). D'altra parte, queste caratteristiche portano a una precisione dimensionale meno accurata. Di conseguenza, la tecnologia LDED è più adatta alla produzione di componenti più grandi e meno complessi di quelli idonei per il processo LPBF [21].

L'Inconel 718 è una lega trattabile termicamente che può subire indurimento per precipitazione, attraverso la formazione dei precipitati nanometrici e coerenti γ " (Ni₃Nb, di struttura tetragonale a corpo centrato) e γ ' (Ni₃(Al,Ti), di struttura cubica ordinata). La fase γ " è la principale fase indurente, poiché il contenuto di Nb è superiore a quelli di Al e Ti, e soprattutto perché causa una tensione di coerenza con la matrice superiore a quella causata dalla fase γ '. Il trattamento termico di questa lega è complesso, visto que la fase γ " è metastabile e può facilmente trasformarsi nella fase δ (sempre Ni₃Nb, però con una struttura ortorombica invece che cubica) se sottoposta a temperature al di sopra dei 700 °C [24]. La trasformazione nella fase δ , che è la fase stabile, viene accompagnata da una riduzione della resistenza meccanica (durezza, carico di snervamento), perché questa fase è incoerente con la matrice, oltre che più grossolana. Inoltre, durante la solidificazione della lega, spesso si forma la fase di Laves ((Ni, Cr, Fe)₂(Nb, Ti), con struttura esagonale), che è una fase dura e fragile, e che porta alla consumazione di Nb e Ti che altrimenti sarebbero utilizzati per formare le fasi desiderate γ " e γ ' [24].

Le tecniche AM hanno un tasso di raffreddamento estremamente elevato, grazie alla sorgente termica altamente localizzata, portando a una microstruttura molto fine. Il tasso di raffreddamento, naturalmente, è diverso per le diverse tecnologie AM. Il processo LPBF, usando un laser di diametro più ridotto, una potenza più elevata e una maggiore velocità di scansione, ha un tasso di raffreddamento più alto, portando a una microstruttura più fine (e quindi migliori proprietà meccaniche). Il processo LDED, invece, con un laser di diametro maggiore, una potenza più alta e una velocità di scansione più bassa, presenta un minore tasso di raffreddamento, risultante una microstruttura meno fine (ma comunque fine). I valori tipici di tasso di raffreddamento per processi LPBF sono all'ordine di 10⁶ K/s, mentre i processi LDED presentano valori all'ordine di 10³-10⁴ K/s. Il processo di fusione a cera persa, per esempio, che è uno dei processi di fonderia più comunemente utilizzati per l'Inconel 718, comporta un tasso di raffreddamento dell'ordine di 1 K/s [58, 75].

Normalmente, la microstruttura di pezzi in Inconel 718 prodotti per AM comporta grani colonnari spesso orientati lungo la direzione di costruzione o deposizione. I grani sono fini, e spesso comportano sottostrutture allungate (comunemente verso la direzione di crescita dei grani), che tornano la microstrutture complessivamente ancora più fine. La direzione di crescita dei grani e delle sottostrutture è definita dalla direzione di massima estrazione di calore e dalle direzione cristallografiche preferenziali di accrescimento della lega, che per l'Inconel 718 è la direzione <100> [61]. In modo macroscopico, l'estrazione del calore avviene dallo strato più recentemente formato (quello più in alto) verso il substrato (in basso), e quindi nella direzione di massima estrazione di calore e verticale). Comunque, localmente, nella pozza di fusione, la direzione di massima estrazione di calore è perpendicolare al bordo della pozza di fusione, e quindi è comune che i grani non siano perfettamente verticali, ma sì leggermente inclinati. La direzione di moto della sorgente di calore può anche influenzare i flussi di calore locali, rafforzando questo effetto [60].

Il raffreddamento rapido caratteristico dei processi AM non permette la formazione dei precipitati indurenti γ " e γ ', i quali hanno una cinetica lenta. Tipicamente, questi precipitati non sono osservati in componenti in Inconel 718 nello stato "come prodotto" (senza trattamenti termici), mentre l'indesiderata fase di Laves ci è frequentemente presente. Per questo motivo, trattamenti termici si fanno necessari anche per componenti prodotti tramite AM [49].

Per i metodi tradizionali di manifattura, i quali forgiatura e fonderia, ci sono ormai trattamenti termici efficaci, definiti da norme e tradizionalmente utilizzati nell'industria. Questi trattamenti prevedono degli step volti a solubilizzare la fase di Laves, per aumentare la concentrazione di Nb nella matrice, per poi promuovere la precipitazione di γ " (e anche γ ') negli step successivi. Per componenti forgiati, la norma AMS 5662 stabilisce l'utilizzo del trattamento termico definito dalla norma AMS 2774, che include un trattamento termico di solubilizzazione a 980 °C per 1 ora, tempra in acqua, e poi un invecchiamento doppio, prima a 720 °C per 8 ore, poi raffreddamento in forno fino a 620 °C, e mantenimento a questa temperatura per 8 ore aggiuntive, finendo con raffreddamento in aria. Per componenti fusi, la norma AMS 5383 raccomanda il trattamento termico definito dalla norma AMS 2774, con l'aggiunta di un trattamento di omogeneizzazione a 1080 °C per 1 ora come primo step, prima del trattamento di solubilizzazione [50-53].

Per componenti prodotti per AM invece, non esiste un consenso su quali trattamenti utilizzare, visto che l'AM è una tecnica nuova e che non è ancora completamente compresa nella totalità dei fenomeni e effetti che comporta. Comunque, la strategia più comunemente impiegata è quella di utilizzare lo stesso trattamento previsto per le parti forgiate. Recentemente, sono state sviluppate norme per la produzione di metalli tramite LPBF, e queste norme (ASTM F3055 e ASTM F3301) difendono l'uso del trattamento termico AMS 2774, lo stesso usato per componenti forgiati [55, 56]. Non esiste ancora una norma per il trattamento termico di Inconel 718 prodotto per LDED.

La scelta del trattamento sviluppato per parti forgiate si basa sul fatto che la microstruttura di componenti prodotti tramite AM è molto fine, e si assomiglia più alla microstruttura di pezzi forgiati che a quella di pezzi fusi, visto che pezzi fusi hanno una microstruttura piuttosto grossolana, grazie ai bassi tassi di raffreddamento del processo, mentre i pezzi forgiati presentano una microstruttura più fine dovuta alla rottura dei precipitati e alla ricristallizzazione dei grani, che avvengono durante la lavorazione meccanica ad alte temperature.

2. Materiali e metodi

In questa tesi, si ha studiato l'effetto di trattamenti termici basati sull'AMS 5662/2774 sulla microstruttura di componenti in Inconel 718 prodotti per LPBF, LDED e anche per fusione a cera persa. L'obbiettivo è stato quello di verificare se il trattamento termico più comunemente utilizzato per parti prodotte tramite AM è di fatto in grado di portare avanti le modificazioni microstrutturali desiderate (solubilizzazione della fase di Laves e precipitazione delle fasi γ " e γ ') sulle parti prodotte per LPBF e LDED. Inoltre, si vuole scoprire in che modo campioni prodotti da tecniche AM diverse (in questo caso, LPBF e LDED) rispondono allo stesso trattamento termico. Si desidera anche analizzare l'influenza della microstruttura iniziale nel risultato del trattamento termico. Per questo motivo, oltre ai campioni prodotti con due differenti tecniche di AM, si ha considerato anche dei campioni prodotti per fusione a cera persa, uno dei metodi di fonderia più utilizzati per la produzione di pale di turbina in Inconel 718. Così, si riesce a valutare l'effetto del trattamento termico su campioni con una microstruttura solidificata in uno stato prossimo all'equilibrio, senza interferenze di lavorazione meccanica. In più, si vuole individuare l'effetto dei singoli step facenti parte del trattamento AMS 5662/2774 e verificarne l'efficacia.

Per fare ciò, sono stati prodotti 12 campioni: 4 campioni per ogni metodo. I 4 campioni realizzati per LDED sono stati fabbricati da un partner, la Scuola Universitaria Professionale della Svizzera Italiana (SUPSI), a Manno, Svizzera. Si ha utilizzato una macchina Prima Additive Laserdyne 430, e il processo è avvenuto con una velocità di scansione pari a 900 mm/s, una potenza del laser di 600 W, uno hatch spacing (distanza tra due track adiacenti) del 0.3 mm, uno strato di spessore 0.2 mm, un diametro del laser di 1064 nm, e una strategia di scansione a rotazione (0°/90°/45°/135°/60°/120°). I 4 campioni prodotti per fusione a cera persa e i campioni prodotti per LPBF sono stati forniti da un partner per effetto di confronto. I parametri di processo utilizzati sono confidenziali.

Per ogni metodo di fabbricazione, un campione non ha subito alcun trattamento termico, un campione ha subito soltanto il trattamento di solubilizzazione a 980 °C per 1 ora e poi tempra in acqua, un campione ha subito il trattamento di solubilizzazione (980 °C per 1 ora e poi tempra in acqua) più un trattamento di invecchiamento a 720 °C per 8 ore e poi tempra in acqua, e un ulteriore campione ha subito il completo trattamento AMS 5662/2774, cioè solubilizzazione (980 °C per 1 ora e poi tempra in acqua) e doppio invecchiamento, prima a 720 °C per 8 ore, poi raffreddamento in forno fino a 620 °C, poi mantenimento a 620 °C per 8 ore aggiuntive, e finalmente raffreddamento in aria. Il trattamento completo è riportato in Figura I. I campioni LDED sono stati trattati mentre ancora collegati al substrato (un disco in Inconel 718 prodotto per fusione e colata). I trattamenti

termici sono stati eseguiti alla sede di Alessandria del Politecnico di Torino, in un forno tubolare Nabertherm RHTH 120-600/16.



Figura I: Trattamento termico AMS 5662/2774 [50, 51] (schematico). RT=room temperature.

Dopo i trattamenti termici, presso la sede di Torino, i campioni LDED sono stati tagliati con una lama di SiC (Remet TR 100s) in campioni con dimensioni non oltre i 25 mm, per permetterli di entrare nella inglobatrice. Anche gli altri campioni hanno osservato lo stesso limite dimensionale. Dopo la tagliatura, i campioni sono stati inglobati in resina acrilica tramite un'inglobatrice (Remet IPA 30) per facilitarne la manipolazione. In seguito, i campioni sono stati spianati e lucidati in una lucidatrice (Presi Minitec 233), con rotazione di 240 rpm e carte abrasive di SiC con #FEPA 240, 400, 800, 1200, 2400 e 4000, successivamente. Finita la lucidatura con la carta da #FEPA 4000, si è proceduto alla lappatura in due step, con pasta diamantata di granulometria 3 µm e 1 µm, rispettivamente.

Come fase finale della preparazione dei campioni, essi sono stati sottoposti a un attacco chimico (etching) con il reagente di Kalling #2 (preparato con 10 g di CuCl₂, 200 ml di $HCl_{(aq)}$ (37% vol.) e 200 ml di etanolo (100% vol.)). Ogni provino è stato sommerso nel reagente per 20 s, e poi immediatamente sommerso in acqua distillata.

Tutti i provini sono stati caratterizzati tramite il microscopio ottico – OM, per "optical microscope" – (Leica DMI 5000), con ingrandimenti di 100x, 200x e 500x. Inoltre, i provini non trattati, quelli solo solubilizzati, e quelli che hanno subito il trattamento completo (solubilizzazione e doppio invecchiamento) sono stati osservati al microscopio elettronico a scansione – SEM, per "scanning electron microscope" – (Thermo Fischer Scientific Phenom XL), con ingrandimenti pari a 2000x, 3000x, 5000x, 10000x e 20000x. In aggiunta, sono stati eseguite analisi chimiche per EDS in tutti i campioni osservati al microscopio elettronico. Finalmente, si è proceduto a valutare la microdurezza Vickers, con un tester (Leica VMHT) con carico di 500 gf, e un periodo di indentazione di 15 s (in accordo con la norma ASTM E384). Per ogni campione, sono stati fatte 5 misure e si ha calcolato la microdurezza media e la deviazione standard.

Le polveri utilizzate per le tecniche LPBF e LDED sono anche stati caratterizzate (in accordo con la norma ASTM F3049), con osservazioni delle particelle delle polveri al microscopio elettronico a scansione (ingrandimento 500x) e osservazioni della sezione trasversale delle polveri al microscopio ottico (ingrandimento 500x per la polvere da LPBF e 250x per la polvere da LDED) dopo inglobatura in resina acrilica e lucidatura. Dalle immagini ottenute al microscopio elettronico si ricava la distribuzione granulometrica delle polveri. In aggiunta, la fluidità delle polveri è stata misurata con un imbuto Hall (tempo necessario per che 50 g di polvere attraversino l'imbuto). L'angolo di riposo del cono di polvere formato dopo lo scorrimento è anche stato misurato. Sono state eseguite 3 misure. La densità apparente e la tap density sono state misurate con un volumetro Scott (misrando la massa del volume di polvere contenuto nel volumetro di 25 cm³). La caratterizzazione delle polveri è stata eseguita dai partner del gruppo Hybrid 4D.

3. Risultati e discussione

3.1 Polveri

Entrambe le polveri sono state prodotte per atomizzazione a gas, e quindi hanno una morfologia prevalentemente sferica. Le distribuzione di dimensioni delle particelle delle polveri sono notevolmente diverse per LPBF e LDED. Il diametro mediano (d_{50}) per la polvere LPBF è pari a 17.5 µm, mentre il diametro mediano della polvere LDED è pari a 57.5 µm.

3.2 Fusione a cera persa

3.2.1 Campione non trattato

Le osservazioni al microscopio ottico per il provino prodotto per fusione a cera persa (Figura II) mostrano una microstruttura dendritica equiassica con fasi secondarie presenti nelle zone interdendritiche. Vi è una fase grossolana, con morfologia irregolare, e una fase più fine, con morfologia ad ago. Queste morfologie sono tipiche delle fasi di Laves e della fase δ , rispettivamente. La fase irregolare è vista sia in una tipologia eutettica (con interiore forato da isole di γ) sia in una tipologia primaria (interiore chiaro e omogeneo). In aggiunta, sono anche visibili precipitati di dimensioni ridotte, più o meno equiassici, con lati dritti, e spigoli vivi, una morfologia tipicamente associata ai carburi di Nb o Ti nelle leghe Inconel.



Figura II: Immagini da OM del campione da fusione a cera persa senza trattamenti termici. (a) 100x. (b) 200x.

Il campione è anche stato osservato al SEM (Figura III), e analizzato chimicamente per EDS (Tabella I), per permettere una più accurata individuazione delle fasi. Si conferma la presenza della fase di Laves, della fase δ e di carburi ricchi in Nb e Ti. La presenza della fase di Laves, sopprattuto, una fase dura e fragile, evidenzia la necessità di trattamenti termici. La durezza è stata misurata pari a 205 ± 13 HV_{0.5}.



Figura III: Immagini da SEM del campione da fusione a cera persa senza trattamenti termici. I punti indicati con 1, 2 e 3 sono quelli analizzati per EDS.

Punto EDS	Nb	Ni	Ti	Cr	Fe	Mo	Si	Al	0	Zr	Mn	Fase sospetta
1	71.42	12.08	4.60	4.14	3.94	3.13	0.50	0.19				Carburi Nb/Ti
2	20.43	43.95	1.16	10.91	10.85	6.13	0.79		4.13	1.65		Laves
3	7.11	50.95	0.99	17.47	16.89	5.23	0.70	0.62			0.04	δ

 Tabella I – Risultati EDS per il campione prodotto per fusione a cera persa senza trattamenti termici (%massica).

3.2.2 Solubilizzazione

Dopo il trattamento di solubilizzazione, il campione prodotto per fusione a cera persa ha ancora presentato le fase di Laves, δ e anche carburi (Figura IV). Questo dimostra che il trattamento di solubilizzazione a 980 °C per 1 ora non è in grado di portare la fase di Laves a solubilizzarsi. La non solubilizzazione della fase di Laves fa sì che ci sia un minor contenuto di Nb e Ti disponibile nella matrice per poi originare le fasei indurenti γ " e γ '. Inoltre, la fase di Laves è fragile, grossolana e continua, in modo che un componente in Inconel con un contenuto così alto di Laves tende ad avere una bassa duttilità.



Figura IV: Microstruttura del campione da fusione a cera persa dopo trattamento di solubilizzazione a 980 °C per 1 ora. (a) OM, 100x. (b) SEM, 5000x.

Questi risultati erano aspettati, visto che i precipitati in Inconel 718 prodotto per fusione sono significativamente più grossi di quelli presenti in componenti forgiati (i precipitati si frantumano durante la lavorazione meccanica, e precipitati minori sono più facilmente sciolti, poiché vi è meno massa da diffondere), per i quali questo trattamento termico è stato sviluppato. Si ha deciso di eseguire questo trattamento su dei campioni prodotti per fusione a cera persa per avere un *benchmark* (con una microstruttura iniziale più convenzionale e vicina allo stato di equilibrio) contro il quale si potesse paragonare i risultati dei trattamenti sui campioni AM. L'inefficacia del trattamento di solubilizzazione è ulteriormente confermata dal fatto che la durezza del campione non solo non si è ridotta, anzi si è innalzata a 226 ± 24 HV_{0.5}. L'aumento della durezza può essere spiegato dalla possibile formazione di δ a 980 °C (come riportato da Liu et al.) [87].

3.3.3 Solubilizzazione e invecchiamento singolo

Procedendo al trattamento di invecchiamento semplice (a 720 °C per 8 ore, finendo con tempra in acqua) aggiunto dopo quello di solubilizzazione, la microstruttura del campione non sembra essere cambiata, secondo le osservazioni per OM. Nonostante ciò, la durezza è cresciuta a 387 ± 36 HV_{0.5}. Anche se le fasi indurenti γ " e γ ' non sono osservate neanche al SEM, a cause delle sue ridotte dimensioni (nanometriche, di fatto), l'aumento di durezza evidenzia che la precipitazione è avvenuta. Quindi, anche se le fasi di Laves e δ non si sono sciolte con successo, il contenuto di Nb è sufficiente per possibilitare la formazione di γ ", il precipitato indurente principale.

3.3.4 Trattamento completo

Dopo il trattamento completo, cioè solubilizzazione a 980 °C per 1 ora, tempra in acqua, invecchiamento a 720 °C per 8 ore, poi raffreddamento in forno fino a 620 °C, poi invecchiamento a 620 °C per 8 ore e raffreddamento in aria, il campione è stato osservato per OM e SEM e, di nuovo, dalle osservazioni OM, non sembra che la microstruttura abbia subito modificazioni importanti. Le micrografie si incontrano nella sessione 4.2.4.

In questo caso, nemmeno la durezza è cambiata significativamente. La microdurezza media è stata 369 ± 32 HV_{0.5}. Dunque, il secondo step di invecchiamento (quello a 620 °C) si mostra non necessario. Probabilmente non si ha verificato un ulteriore aumento nella durezza perché gli atomi di Nb inizialmente disponibili nella matrice sono stati consumati nel primo step di invecchiamento per la formazione di γ ". Il leggero calo nella durezza può essere parzialmente spiegato dalla trasformazione γ " $\rightarrow \delta$, la quale comporta una diminuzione di durezza poiché si passa da una fase coerente (e metastabile) a una fase incoerente (e stabile).

3.3 LPBF

3.3.1 Campione non trattato

Il campione prodotto per LPBF, analizzato per OM (Figura V), mostra una tipica microstruttura da AM, con pozze di fusione di formato ad arco, e grani colonnari orientati lungo la direzione di costruzione (BD, per building direction), attraversando molteplici strati e pozze di fusione, evidenziando l'accrescimento epitassiale dei grani. L'accrescimento epitassiale sostanzialmente è l'accrescimento di un grano a partire da un altro grano sottostante, mantenendo la stessa struttura cristallina e orientazione, senza lo step di nucleazione. Inoltre, i grani hanno una specie di pattern interno, dovuto all'esistenza di sottostrutture. Infatti, l'osservazione per SEM permette l'individuazione di queste sottostrutture, di morfologia cellullare dendritica (Figura VI).



Figura V: Osservazioni per OM della microstruttura del campione LPBF senza trattamenti termici. Sono visibili le pozze di fusione e granni colunnari che attraversano diversi strati (a) 100x. (b) 200x.

Le immagini di SEM consentono l'osservazione delle dendriti cellulari dentro i grani, orientate in modo parallelo, e con precipitati nella zona interdendritica, indicando l'occorrenza di microsegregazione, nonostante l'elevato tasso di raffreddamento. Dendriti cellulari sono dendriti che non sviluppano le braccia secondarie, a causa di un tasso di raffreddamento molto alto. Le dendriti hanno una larghezza dell'ordine di 1 µm.



Figura VI: Osservazioni per SEM della microstruttura del campione LPBF senza trattamenti termici. (a) 3000x. Grani allungati con sottostrutture allungate (b) 2000x. Sottostrutture cellulari dendritiche, con segni di segregazioni e fasi secondarie nelle zone interdendritiche.

Il punto 1 sulla Figura VIb è stato analizzato per EDS, e i risultati sono riportati in Tabella II. L'alto contenuto di Nb e Mo evidenziano l'occorrenza di segregazione, e indicano che la fase in questione è probabilmente la fase di Laves. L'alto contenuto di C può suggerire la presenza di carburi. L'avvenimento di microsegregazione richiede la esecuzione di trattamenti termici per possibilitare la solubilizzazione della fase di Laves e la successiva precipitazione di γ " e γ '. La microdurezza del campione LPBF è pari a $327 \pm 7 \text{ HV}_{0.5}$, molto più alta rispetto a quella del campione da fusione, in funzione soprattutto della microstruttura molto più fine, in conseguenza dell'alto tasso di raffreddamento caratteristico del processo LPBF.

Tabella II – Risultati EDS per il campione prodotto per LPBF, senza trattamenti termici (%massica).

Punto EDS	Nb	С	Ni	Cr	Fe	Мо	Ti	Al	Si	Zr	Fase sospetta
1	9.60	5.60	46.79	15.35	15.14	4.50	1.12	0.50	0.42	0.97	Carburi / Laves / y

3.3.2 Solubilizzazione

Il campione LPBF solubilizzato (980 °C, 1 ora, tempra in acqua) non sembra aver subito modifiche importanti nella microstruttura (rispetto al campione non trattato). Nell'immagine OM (Figura VIIa), sono ancora presenti le pozze di fusione ed i granni colonnari. Nell'immagine SEM (Figura VIIb), si possono apprezare ancora le sottostrutture cellulari dendritiche dentro i grani, così come le fasi secondarie nelle zone interdendritiche. Pertanto, anche in questo caso il trattamento di solubilizzazione non ha avuto successo. Qui, invece, il risultato è sorprendente, visto che il principio dietro l'utilizzazione del trattamento per componenti forgiati anche per pezzi prodotti tramite AM si basa nel fatto che i precipitati in AM sono fini (addirittura più fini di quelli visti in pezzi forgiati), e quindi si spera che questi precipitati sarebbero sciolti con più facilità rispetto a quelli dei pezzi forgiati. Invece, si verifica il contrario. La misura di microdurezza, 332 ± 16 HV_{0.5}, è un'ulteriore indicazione dell'inefficacia del trattamento a 980 °C.



Figura VII: Microstruttura del campione LPBF dopo trattamento di solubilizzazione a 980 °C per 1 ora. (a) OM, 100x. (b) SEM, 5000x.

3.3.3 Solubilizzazione e invecchiamento singolo

Il campione solubilizzato (980 °C, 1 ora) e invecchiato (720 °C, 8 ore) non ha subito modificazioni microstrutturali facilmente visibili per OM. In Figura VIII, si vede la micrografia ottica del campione, con le pozze di fusione e grani colonnari, a un ingrandimento di 500x. Si vedono ancora le sottostrutture dentro i grani. I grani colonnari possono essere allineati lungo la direzione di costruzione, oppure in direzioni perpendicolari al bordo della pozza di fusione, come visto in questo caso, in conseguenza dei flussi locali di calore.

In questo campione, sembra che i bordi dei grani siano più definiti. Questo può accadere come conseguenza della formazione di precipitati a bordo grano. I grani hanno una larghezza dell'ordine di 10 μ m. La durezza misurata del campione è giunta a 468 ± 21 HV_{0.5}. Come per il campione da fusione, anche se le fasi ricche in Nb non si sono sciolte nel trattamento di solubilizzazione, e anche se le fasi indurenti non sono state osservate, il campione ha subito un importante aumento di durezza, indicando che la precipitazione di γ " e γ ' è avvenuta.



Figura VIII: Microstruttura del campione LPBF dopo trattamento di solubilizzazione a 980 °C per 1 ora più invecchiamento a 720 °C per 8 ore. OM, 500x.

3.3.4 Trattamento completo

Dopo il secondo step di invecchiamento, la microstruttura, come osservata per OM, è rimasta molto simile a quella vista per gli altri campioni LPBF, sempre con pozze di fusione e grani colonnari con sottostrutture allungate (Figura IX). Se si osservano invece le immagini ottenute per SEM (Figura X), si percepisce una quantità apparentemente maggiore di precipitati a bordo grano, in paragone alle immagini SEM dei campioni prima degli step di invecchiamento. Tali precipitati sono le ancora presenti fasi di Laves, con morfologia irregolare, e soprattutto, precipitati δ (dennunciati dalla morfologia ad ago). I precipitati δ possono formarsi durante il trattamento termico, precipitando dalla matrice, oppure dalla trasformazione $\gamma^{"} \rightarrow \delta$. Un precipitato irregolare è stato analizzato per EDS (Tabella III) e, essendo arrichito in Nb e Ti, trattasi molto probabilmente della fase di Laves.



Figura IX: Osservazioni per OM della microstruttura del campione LPBF dopo il trattamento termico AMS 5662 completo. Sono visibili le pozze di fusione e granni colunnari che attraversano diversi strati, con sottostrutture. (a) 100x. (b) 200x. BD=building direction.

Nonostante possa sembrare che le sottostruttura dei grani siano state eliminate nelle immagini SEM, le sottostrutture sono viste nelle immagini OM. Il mottivo per il quale le sottostrutture non sono facilmente visibili al SEM è che la soluzione utilizzata per l'atacco chimico svela più intensamente i precipitati, e siccome essi si trovano più che altro ai bordi di grano, queste regioni diventano più evidenti. Comunque, le sottostrutture sono viste al microscopio ottico.



Figura X: Osservazioni per SEM della microstruttura del campione LPBF dopo il ciclo completo AMS 5662. I precipitati a bordo grano sono in evidenza. (a) 3000x. (b) 10000x.

|--|

Punto EDS	Nb	Ni	Cr	Fe	Mo	Ti	Al	Si	Fase sospetta
1	23.72	58.67	5.74	6.44	2.78	1.92	0.30	0.44	Laves / δ

La microdurezza di questo campione, dopo il trattamento termico completo AMS 5662, è stata 482 \pm 52 HV_{0.5}, che non rappresenta un aumento statisticamente significativo dalla condizione di invecchiamento singolo a 720 °C, evidenziando che il secondo step di invecchiamento non contribuisce significativamente con un indurimento del materiale. L'inefficacia del secondo step di invecchiamento è una conseguenza diretta dell'inefficacia del trattamento di solubilizzazione, il quale è stato sorprendentemente inadeguato. Senza la solubilizzazione della fase di Laves, non si ha disponibilità sufficiente di Nb per un secondo invecchiamento e ulteriore precipitazione di γ ". La logica per la fase γ ' è la stessa, però con Ti al posto di Nb.

Inoltre, Gallmeyer et al. [68], ha dimostrato che la temperatura di 720 °C è in grado di promuovere la formazione sia di γ " che di γ ', mentre la temperatura di 620 °C favorisce la precipitazione di γ " di più, rispetto a γ ', che ha una cinetica più lenta. Quindi, nel secondo step di invecchiamento, la formazione di γ ' è limitata, e se non vi è un contenuto di Nb abbastanza alto (attorno al 4% secondo Qi et al. [83]) per la formazione di più γ ", il trattamento diventa infatti inefficace. Comunque, il risultato finale di durezza è molto superiore a quello dei campioni prodotti per fusione a cera persa, grazie alla microstrutture molto fine dei campioni LPBF a causa dell'elevato tasso di raffreddamento tipico dei processi AM.

3.4 LDED

3.4.1 Campione non trattato

I campioni prodotti per LDED sono stati studiati con gli stessi metodi, per verificare l'effetto dei trattamenti termici in un altro processo AM con un tasso di raffreddamento minore e quindi una microstruttura diversa, più grossolana rispetto a quella dei campioni LPBF. L'immagine OM (Figura XIa) mostra la microstruttura del campione come prodotto, senza alcun trattamento termico. Il processo LDED produce pozze di fusione più ampie rispetto al processo LPBF, a causa del maggiore diametro del laser, maggiori dimensioni delle polveri, tipicamente maggior potenza del laser e minor velocità di scansione. Di conseguenza, i grani sono anche più grossolani (si nota che in Figura XIa, l'ingrandimento è di 50x, meno di quello usato per le micrografie ottiche dei campioni LPBF). Sono comunque grani colonnari che subiscono accrescimento epitassiale, attraverso molteplici strati.

Osservazioni per SEM (Figura XIb) permettono la visione delle sottostrutture dei grani, con morfologia cellulare dendritica. Nelle zone interdendritiche, giace una fase secondarie che sembra la fase di Laves, visto che

ha una morfologia irregolare e continua. Oltre alla fase di Laves, si vedono elementi microstrutturali neri e sferici, che sono stati investigati per analisi EDS.



Figura XI: Microstruttura del campione LDED come prodotto. (a) OM, 50x. (b) SEM, 10000x. I punti indicati com 1, 2 e 3 sono stati analizzati per EDS.

Tabella IV - Risultati EDS per il campione LDED senza trattamenti termici (% massica).

Punto EDS	Nb	Ni	Cr	Fe	Mo	Ti	Al	Si	Fase sospetta
1	13.00	50.34	15.07	14.83	4.65	1.15	0.52	0.44	Laves / y
2	11.06	51.43	15.56	15.07	4.52	1.23	0.71	0.42	Laves / y
3	3.34	54.35	18.00	19.14	3.60	0.59	0.64	0.34	γ

Le fasi secondarie viste nella zona interdendritica sono ricche in Nb, Mo e Ti, il che suggerisce che siano le fasi di Laves, anche per la morfologia irregolare. I risultati EDS evidenziando l'occorrenza di segregazione di Nb e Mo nella zona interdendritica. Nei punti 1 e 2, localizzati in queste zone, la concentrazione di Nb è superiore ai 10% in massa, mentre nel punto 3, piazzato sul corpo della dendrite, il contenuto di Nb è inferiore al 4%, al di sotto della concentrazione nominale (4.75%-5.50%) [24].

Il campione è stato sottoposto a un'analisi EDS di area (Figura XII). Nella figura, un colore più forte o più scuro significa una concentrazione maggiore dell'elemento analizzato, e un colore più chiaro o meno intenso indica una concentrazione minore. L'area analizzata è riportata in alto nella figura, vista con ingrandimento 20000x. Questa analisi espone in modo più evidente l'occorrenza della microsegregazione. La concentrazione di Nb e Mo è più elevata nelle zone interdendritiche, mentre le dendriti stesse ne sono impoverite. La segregazione si verifica per questi elementi soprattutto perché sono elementi con atomi grandi e pesanti, e quindi con limitata diffusività. La bassa concentrazione di Nb nelle dendrite limita la formazione di γ " nel trattamento di invecchiamento. Dunque, è necessario un trattamento di solubilizzazione.

Oltre alla segregazione di Nb e Mo, si notano regioni sferiche con elevato contenuto di Al e Ti. Questi punti hanno anche una notevole concentrazione di ossigeno. Quindi, gli elementi microstrutturali rotondi visti al SEM sono in realtà fasi secondarie, e non porosità, come può sembrare a principio. Infatti, dall'analisi chimica, si conclude che siano ossidi di Al e Ti. La presenza di questi ossidi è menzionata in letteratura [68], e può essere spiegata dall'ossidazione avvenuta durante il processo LDED, che non avviene in una camera chiusa con atmosfera controllata o sotto vuoto, ma invece sfrutta soltanto il shielding gas (Ar) soffiato dall'ugello, che non è in grado di fornire una protezione così effettiva come quella ottenuta nel processo LPBF.

In Figura XII, si notano anche punti con una concentrazione più elevata di Ti e che non sono accompagnati da un'alta concentrazione di Al. Questi punti probabilmente corrispondono a carburi di Ti.



Figura XII: Analisi EDS di area. In alto, la micrografia dell'area analizzata.

La microdurezza del campione LDED nello stato come prodotto è stata 253 ± 18 HV_{0.5}. Questo numero è più alto di quello del campione prodotto per fusione a cera persa, ma più basso di quello del campione prodotto per LPBF, in corrispondenza dei tassi di raffreddamento tipichi dei processi. Il processo LDED, avendo un tasso di raffreddamento intermedio, ha presentato una durezza intermedia.

3.4.2 Solubilizzazione

Le osservazioni del campione LDED dopo solubilizzazione a 980 °C per 1 ora sono riportate in Figura XIII. L'immagine OM (a) consente la visualizzazione di grani colonnari con una direzione di accrescimento approssimativamente parallela alla direzione di deposizione (indicata con "BD"). I grani hanno sottostrutture, come evidenziato dai pattern ai suoi interni. L'immagine SEM (b) mostra appunto le sottostrutture (dendriti cellulari), orientate parallelamente, in modo prossimo alla direzione di deposizione (verticale). Nonostante il trattamento di solubilizzazione, si verifica ancora la presenza di fasi secondarie nelle zone interdendritiche, e anche degli ossidi.

Si nota come i grani e le sue sottostrutture sono di fatto più grossolani rispetto a quelli trovati nei campioni LPBF. Qui, i grani hanno una larghezza attorno ai 50 μ m (contro 10 μ m per i grani LPBF) e le dendriti sono larghe 5 μ m, indicativamente (contro 1 μ m per le dendriti LPBF).



Figura XIII: Microstruttura del campione LDED dopo trattamento di solubilizzazione. (a) OM, 100x. (b) SEM, 5000x.

La presenza delle fasi secondarie evidenzia che il trattamento di solubilizzazione non ha avuto successo, similmente a quanto si è verificato per i campioni LPBF. Le fasi maggiormente presenti dopo il trattamento termico sono la fase di Laves (come suggerito da analisi EDS e dalle morfologie dei precipitati) e la fase δ (come suggerito dalla morfologia dei precipitati fini a forma d'ago). La fase δ non era vista nella microstruttura del campione LDED non trattato, e quindi si è formata durante il trattamento a 980 °C. Liu et al. ha mostrato, infatti, che questa temperatura è in grado di promuovere la formazione di δ [87].

La microdurezza del campione è stata misurata 262 ± 12 HV_{0.5}, quindi praticamente non cambiata dallo stato non trattato, indicando ancora una volta l'inefficacia del trattamento di solubilizzazione.

3.4.3 Solubilizzazione e invecchiamento singolo

Dopo il primo step di invecchiamento, la microstruttura, vista per OM, non sembra aver subito modificazioni importanti (Figura XIV). Si vedono ancora le pozze di fusione ed i grani colonnari con sottostrutture. In questa immagine, si nota come i grani colonnari prodotti per LDED possano deviare della direzione di deposizione. Qui, i grani sono perpendicolari al bordo delle pozze di fusione, a causa dei flussi di calore localizzati. Nel processo LDED, la pozza di fusione è tendenzialmente più profonda, grazie alla più alta potenza del laser e alla minor velocità di scansione, e pozze più profonde originano grani più deviati dalla direzione di deposizione, visto che i flussi di calore sono massimi in direzioni perpendicolari al bordo della pozza, e se il bordo è più ampio e profondo, la direzione perpendicolare ad esso si allontana dalla direzione di costruzione. Comunque, anche in questo caso si verifica l'accrescimento epitassiale, e i grani attraversano diversi pozze di fusione.

Nonostante la micrografia ottica non evidenzi cambiamenti microstrutturali importanti, la microdurezza del campione dopo l'invecchiamento singolo si è innalzata a $455 \pm 22 \text{ HV}_{0.5}$. Quindi, nonostante il trattamento di solubilizzazione non sia stato completamente efficace, il trattamento di invecchiamento a 720 °C ha avuto successo nella promozione della precipitazione delle fasi indurenti. Le fasi γ " e γ ' non sono viste per OM perché hanno di solito dimensione in scala nanometrica [49].



Figura XIV: Microstruttura del campione LDED dopo trattamento di solubilizzazione. OM, 100x. BD=building direction (direzione di deposizione).

3.4.4 Trattamento completo

Le micrografie ottiche del campione che ha subito il trattamento termico completo, con solubilizzazione e doppio invecchiamento, sono riportate in Figura XV. Si osservano le tipiche caratteristiche microstrutturali dei campioni prodotti per LDED, inclusi pozze di fusione, grani colonnari con sottostrutture allungate e l'accrescimento epitassiale.



Figura XV: Microstruttura del campione LDED dopo il trattamento completo AMS 5662. OM. (a) 100x. (b) 200x. BD=building direction (direzione di deposizione).

Le immagini ottenute per SEM (Figura XVI) consentono una visualizzazione più accurata delle sottostrutture dei grani, composte sempre da dendriti cellulari. Nelle zone interdendritiche, si osservano precipitati di Laves e, soprattuto, la fase δ , che non era vista nel campione non trattato. Gli ossidi sono ancora presenti. La microstruttura osservata è simile a quella del campione solubilizzato, con un elevato contenuto di δ . La quantificazione accurata delle fasi richiederebbe un'analisi XRD. I precipitati γ " e γ ' non sono facilmente osservabili per SEM, e quindi non sono visti nella Figura XVI. Per consentire la loro osservazione, sarebbe necessario eseguire analisi TEM. I precipitati δ possono essere stati formati nel trattamento di solubilizzazione a 980 °C (come mostrato da Liu et al.), ma anche dalla trasformazione γ " $\rightarrow \delta$ durante il secondo step di invecchiamento [87].



Figura XVI: Osservazioni per SEM della microstruttura del campione LDED dopo il ciclo completo AMS 5662. (a) 5000x. (b) 10000x. Il numero 2 indica un ponto che è stato analizzato per EDS, con un contenuto di Nb pari a 18.34% in massa. La versione completa dei risultati è disponibile nella sessione 4.4.4.

La microdurezza del campione è stata misurata a 457 ± 7 HV_{0.5}, praticamente lo stesso valore del campione solubilizzato e invecchiato solo a 720 °C, indicando che il secondo step di invecchiamento non ha contribuito significativamente a un aumento della durezza. Questo risultato segue la stessa tendenza generale verificata per i campioni da fusione a cera persa e LPBF, legato alla bassa disponibilità di Nb nella matrice, a causa dell'inefficacia del trattamento di solubilizzazione. Comunque, per il campione LDED, la deviazione standard è crollata da 22 HV a soli 7 HV dopo il secondo step a 620 °C, il che suggerisce che il secondo invecchiamento può aver contribuito con una omogeneizzazione della durezza per il campione LDED (mentre per gli altri campioni, l'aggiunta di più step di trattamento termico hanno portato a un aumento della deviazione standard, in modo generale, evidenziando anche che i trattamenti termici non hanno un effetto omogeneo su tutta la microstruttura, fatto che può essere legato all'occorrenza di segregazione).

3.5 Microdurezza

I risultati di microdurezza sono stati riportati nelle sessione dei rispettivi campioni. Comunque, si fornisce una compilazione comparativa dei risultati in Figura XVII. In modo generale, i trattamenti di solubilizzazione non hanno portato a una diminuzione della durezza, essendo poco efficaci. In modo generale, il trattamento a 980 °C ha occasionato una solubilizzazione molto timida della fase di Laves e, anche se le fasi di Laves sono stati parzialmente sciolte, questo trattamento ha promosso la precipitazione della fase δ , annullando un'eventuale riduzione di durezza. Nonostante ciò, il primo invecchiamento a 720 °C ha causato un notevole aumento nella durezza di tutti i campioni, indicando che la precipitazione delle fasi indurenti è avvenuta. Al contrario, il secondo step di invecchiamento, a 620 °C, non ha portato avanti nessuna modificazione importante nella microstruttura né nella durezza, perché, dovuto all'inefficace solubilizzazione, il contenuto di Nb nella matrice non era sufficiente per ulteriore formazione di γ ", e la precipitazione di γ " è molto lenta a 620 °C.

I campioni prodotti per LPBF presentano i valori più elevati di durezza, in paragone agli altri campioni, sia nello stato non trattato, che in ogni tipologia di trattamento. Questo si deve alla microstruttura più fine dei campioni LPBF rispetto agli altri, in conseguenza dell'alto tasso di raffreddamento tipico del processo LPBF (dell'ordine di 10⁶ °C/s [58]). Analogamente, i campioni ottenuti per fusione a cera persa hanno i valori più bassi di durezza, legati alla microstruttura più grossolana, di conseguenza dei bassi tassi di raffreddamento (dell'ordine di 1 °C/s [75]). I campioni LDED presentano sempre valori intermedi, in conseguenza dei tassi intermedi di raffreddamento (all'ordine di 10³-10⁴ °C/s [75]). Comunque, i campioni prodotti per LDED hanno subito il più espressivo aumento di durezza media (pari a 80%) dopo il trattamento completo, e se i campioni LDED non trattati presentavano una durezza pari a 77% di quella dei campioni LPBF, dopo il trattamento completo, la durezza del campione LDED è pari a 95% della durezza cel campioni LPBF. Questo evidenzia come lo stesso trattamento termico possa influenzare in maniera diversa campioni prodotti per metodi differenti. Nonostante ciò, i campioni LPBF hanno avuto sempre i valori più alti di durezza, il che espone anche l'importanza della microstruttura iniziale.



Figura XVII: Microdurezza dei campioni senza trattamenti e dopo ogni tipologia di trattamento termico.

4. Conclusioni

In questo studio, si ha esplorato campioni in Inconel 718 prodotti per fusione a cera persa, LPBF e LDED sottoposti a differenti trattamenti termici basati sulla norma AMS 5662. Si ha analizzato la microstruttura dei campioni tramite OM e SEM, e si ha misurato la microdurezza di ogni provino.

Processi differenti producono microstrutture diverse, in funzione, tra l'altro, del tasso di raffreddamento. La microstruttura prodotta per fusione a cera persa è piuttosto grossolana in paragone con quelle prodotte per AM. Di conseguenza, la durezza del campione da fusione a cera persa è inferiore a quelle dei campioni AM. D'altra parte, gli elevati tassi di raffreddamento del processo LPBF danno origine a una microstruttura molto fine, con grani colonnari con sottostruture dendritiche cellulari allungate. Di conseguenza, la durezza del provino LPBF è più elevata. Il campione LDED, che subisce un tasso di raffreddamento intermedio, ha una durezza intermedia.

In genere, i campioni non trattati hanno presentato notevole contenuti di fase di Laves, e il trattamento di solubilizzazione non ha avuto successo a promuovere la sua solubilizzazione. Questo risultato era sperato per il campione prodotto per fusione a cera persa, visto che i suoi precipitati sono più grossi di quelli presenti in campioni forgiati, per i quali il trattamento è stato sviluppato. Però, siccome i precipitati nei campioni prodotti per AM sono più fini di quelli presenti in pezzi forgiati, si sperava che la loro solubilizzazione fosse facilitata da una cinetica di diffusione più favorevole. Invece, i risultati hanno mostrato che la solubilizzazione non ha avuto pieno successo. Nonostante ciò, il primo invecchiamento ha portato alla precipitazione delle fasi indurenti, come indicato dal notevole aumento di durezza. Il secondo step di invecchiamento, invece, non ha causato modificazioni notevoli nella microstrutture né nella durezza, fatto spiegato dalla bassa disponibilità di Nb nella matrice per ulteriore formazione di γ ", poiché la fase di Laves, ricca in Nb, non è stata solubilizzata a sufficienza. Questi risultati mostrano che microstrutture diverse, ottenute da processi di fabbricazione diversi, rispondono in modi diversi a uno stesso trattamento termico. Il trattamento termico disegnato per componenti forgiati non ha lo stesso effetto in pezzi prodotti per AM, così come non ha lo stesso effetto su pezzi ottenuti per fusione o colata. Quindi, non è efficace replicare un trattamento termico sviluppato per una determinata tecnica di fabbricazione su pezzi prodotti per altri metodi sperando di raggiungere gli stessi risultati.

Nonostante l'applicazione del trattamento standard per componenti in Inconel 718 forgiati anche per pezzi prodotti per AM sia comune, non è un modo efficace di ottimizzare la complessa microstruttura conseguente di processi AM. Invece, questo studio mostra che lo sviluppo di un nuovo processo di manifattura richiede lo sviluppo di un trattamento termico adatto e disegnato su misura per quella applicazione, tenendo conto delle particolarità del processo produttivo. Infatti, anche i campioni prodotti per due diverse tecniche AM rispondo in modo diverso allo stesso trattamento termico, come visto per i campioni LPBF e LDED. Questi risultati evidenziano anche l'importanza della microstruttura iniziale nella determinazione delle proprietà finali dei componenti a prescindere dal trattamento termico, poiché i campioni LPBF, pur non avendo una soddisfacente risposta al trattamento termico, ha ancora avuto la durezza più elevata, sin dall'inizio dei cicli termici.

Per il trattamento termico di solubilizzazione e invecchiamento di Inconel 718 prodotto per AM, si suggere impiegare una temperatura di solubilizzazione più elevata. Ulteriori esperimenti sarebbero necessari per la determinazione di tale temperatura, che può anche essere diversa per LPBF e LDED. Inoltre, avendo un trattamento di solubilizzazione efficace, si procederebbe a verificare se di fatto il secondo step di invecchiamento sia necessario per Inconel 718 da AM.

LIST OF ABBREVIATIONS

AM	 Additive Manufacturing
3DP	 3 Dimensional Printing
CAD	 Computer Aided Design
RP	 Rapid Prototyping
LPBF	 Laser Powder Bed Fusion
SLM	 Selective Laser Melting
DMLS	 Direct Metal Laser Sintering
SLS	 Selective Laser Sintering
DED	 Directed Energy Deposition
LDED	 Laser Directed Energy Deposition
LAM	 Laser Additive Manufacturing
LENS	 Laser Engineering Net Shaping
RT	 Room Temperature
BD	 Building Direction
ОМ	 Optical Microscopy
SEM	 Scanning Electron Microscopy
EDS	 Energy Dispersive X-Ray Spectroscopy
ISO	 International Standardization Organization
ASTM	 American Society for Testing and Materials
%wt.	 Weight percentage
%vol.	 Volume fraction
POLITO	 Politecnico di Torino
USP	 Universidade de São Paulo (University of São Paulo)
DISAT	 Dipartimento di Scienza Applicata e Tecnologia (Department of Applied Science and Technology)

LIST OF DEFINITIONS

A short list of definitions of terms relevant to the full comprehension of this study is provided, as stated by the international standard ISO/ASTM 52900:2015(E) (Additive manufacturing – General principles – Terminology). The following list is a selection from the extensive list presented on ISO/ASTM 52900:2015(E) [1]. More terms and definitions are presented in Appendix A [1-3].

additive manufacturing (AM): process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies.

additive manufacturing system: machine and auxiliary equipment used for *additive* manufacturing.

as built: refers to the state of *parts* made by an additive process before any post processing, besides, if necessary, the removal from a *build platform* as well as the removal of support and/or processed *feedstock*.

build chamber: enclosed location within the *additive manufacturing system* where the *parts* are fabricated.

build platform: base which provides a surface upon which the building of the *part*, is started and supported throughout the build process.

directed energy deposition: *additive manufacturing* process in which focused thermal energy is used to fuse materials by melting as they are being deposited.

feedstock: bulk raw material supplied to the *additive manufacturing* building process. Synonyms: source material, starting material, base material, original material.

part: joined material forming a functional element that could constitute all or a section of an intended product.

post-processing: process steps taken after the completion of an *additive manufacturing build cycle* in order to achieve the desired properties in the final product.

powder bed: part bed build area in an *additive manufacturing system* in which *feedstock* is deposited and selectively fused by means of a heat source or bonded by means of an adhesive to build up *parts*.

powder bed fusion: *additive manufacturing* process in which thermal energy selectively fuses regions of a *powder bed*.

process parameters: set of operating parameters and system settings used during a *build* cycle.

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1. Introduction

As mankind continues to evolve, new technologies are developed in order to fulfill its needs and provide greater comfort and quality of life. In a world where the population grew from just over 2.5 million people in 1950 to over 6 million in 2000, and is expected to grow further into almost 10 million people in 2050 [4], on a planet with limited resources, it is absolutely necessary for humans to develop and employ new technologies with greater efficiency, better outcome, lower consumption of resources and lower waste generation.

In order to satisfy demand from the growing populace, industrialization continues to spread [4]. At the heart of industrialization, lies metallurgy. Mankind has been trying and mastering metals since the Stone Age [5]. As centuries go by, new metallurgical processes and techniques are developed to achieve greater success, efficacy and efficiency. From manual hammering of native gold in the Stone Age to the advent of rolling in the 18th century; from copper smelting in the Bronze Age, to electrodeposition of copper in the 19th century; from the development of wrought iron in the Iron Age, to the invention of the Linz-Donawitz converter and modern steelmaking in mid-20th century, metallurgical processes and techniques have caused major impacts on the *modus vivendi* of humans [5-7]. There is reference to metallurgy even on the names and definitions of historical periods (Bronze Age, Iron Age). Cooking, sanitation, healthcare, transportation and electricity are examples of common concepts embedded in human daily life that have been profoundly shaped by developments in metallurgy and might otherwise not be possible in the way they are known today [6].

A recent disruptive development in metallurgy is the invention of metal additive manufacturing (in the 1990s) [8]. This is a breakthrough technology that brings with it new, exciting possibilities, and that is in line with the need for humankind to move to more efficient processes that consume less material, yield superior results and generate less waste. However, this technology is still in its infancy, and there is much yet to be understood in order to master it. This work has additive manufacturing at its core. It studies additive manufacturing of a nickel alloy used mostly in the aerospace industry. More specifically, the main focus of the thesis at hand is the effect of thermal treatments on microstructure and mechanical properties of such material.

1.1.Objective

The objective of this work is to characterize the microstructure and hardness of Inconel 718 parts produced by different additive manufacturing techniques (namely LPBF and LDED) and also through the traditional process (casting), submitted through different thermal treatments (no heat treatment, solubilization, solubilization plus aging and solubilization plus double aging) based on the AMS 5662 (standard heat treatment for wrought Inconel 718 parts) and establish a comparison between the results, and ultimately assess whether the standard heat treatment for wrought Inconel 718 is successful and applicable to additively manufactured parts as well, or a novel heat treatment specifically tailored to additive manufacturing processes should be developed.

This is done mainly through optical microscopy (OM), scanning electron microscopy (SEM) and microhardness measurements, all of which were conducted at the Politecnico di Torino, in Italy, after parts were produced by partners and thermally treated at the Politecnico.

Experimental procedure is thoroughly explained in this text, and in order to provide the reader with sufficient information to understand the importance and the results of this work, an extensive (but not exhaustive) review of the literature on additive manufacturing of Inconel 718 is conducted.

The author hopes, as a secondary objective of this work, to inform and entice the readers as to the potential of the technology hereby presented and the benefits it can bring to humankind, with unique advantages and in line with the UN Sustainable Development Goals. However, it should be clear, at the end of the reading, that such benefits require knowledge and dedication to be fully exploited, and with unique advantages come unique challenges. Additive manufacturing is a field that is advancing quickly and has yet so much to be explained and understood, and the author hopes to contribute, at least to some extent, to the expansion of the knowledge frontier on this topic.

1.2.Motivation

According to the ASTM/ISO definition (international standard ISO/ASTM 52900:2015(E)), additive manufacturing is "a process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies" [1]. In contrast, subtractive manufacturing is based on the removal of material (as in machining processes, for instance), and formative manufacturing is based on the application of pressure (forging and casting are some examples) [1].

Additive manufacturing (AM) may be also know as "3D printing" (3DP) or even "rapid prototyping" (RP). "3D printing" is a term commonly used outside of academia interchangeably with additive manufacturing, especially when referring to low-cost additive manufacturing systems or machines, often called "desktop machines" [9]. These systems usually work with polymeric feedstock only and are used, in many occasions, by independent users, enthusiasts or for educational purposes. These systems are also used in industry, but mostly for prototyping. In fact, prototyping was historically the first use for additive manufacturing, hence the name "rapid prototyping" [1].

Nowadays, additive manufacturing has left its status of being a prototyping technology behind, and it is already used for end part production, especially metal additive manufacturing [10]. In fact, annual expenditure on final part production (Figure 1.1) has been rising year after year, especially since 2016, reaching almost 1.5 billion US dollars in 2019 [10]. As shown in Figure 1.2, in 2018, 28.4% of all items produced by additive manufacturing were end-parts suitable for functional and operational applications [9].



Figure 1.1 – Global annual expenditure on the production of end parts through AM. Source: Wohlers Report 2020 [10].



Figure 1.2 – Applications of additive manufacturing in 2018. Source: Wohlers Report 2019 [9].

For prototyping or not, interest on additive manufacturing is on the rise, and certainly not limited to academia or R&D laboratories. The total additive manufacturing industry value has been increasing at a double digit annual rate since 2010. Since 2013, it has grown about 1 billion dollars per year. In 1995, the additive manufacturing industry was valued at 295 million dollars [11]. In 2019, it reached 11.867 billion dollars [10]. Wohlers Associates releases yearly a report on the additive manufacturing market. The total market size (including products and services) is reported in Figure 1.3 (data from Wohlers Reports 2010-2020) [8-18]. In the period 2009-2019, the equivalent annual growth is more than 27%.



Figure 1.3- Total AM market value.

Source: Elaborated by the author based on data from Wohlers Reports 2010-2020 [8-18].

Additive manufacturing was developed initially for prototyping, and for polymers at first. While polymer additive manufacturing continues to be very much a prototyping technology, metals AM, which was developed later, and involves higher costs and is less widespread, finds greater usage in the industrial scenery, including final part production. Metal AM began later, but it is growing at a faster pace than polymer AM. This trend can be linked with the use of metal AM in industry for final part production (Figure 1.4). In 2012, little under 200 metals AM machine were sold. In 2017, this number jumped to over 1700 [18]. The same trend is seen for feedstock: the revenue from sales of metals as feedstock for additive manufacturing went from just under 40 million US dollars in 2013 to over 250 million in 2018 (Figure 1.5). If Figure 1.1 is analyzed again, the increase in final part production through AM seems to be directly correlated with the growth in metal AM (dramatic increase from 2013 onwards), when sales of systems for metal AM started to really take off). In fact, the market for metal AM has grown more than 40% yearly from 2013 to 2018, while the total market has grown at a lower but still impressive rate of just over 27% in the same period [9]. Looking forward, market for metal AM is expected to grow at a CAGR (compound annual growth rate) of 20% through 2028, while the non-metal AM market should see a 11% CAGR in the period [19].

A few examples of companies that are already employing metal additive manufacturing in their production processes are Airbus, Audi, BMW, Boeing, Bugatti, Deutsche Bahn, General Electric, Honeywell Aerospace, Jabil, Lockheed Martin, NASA, Siemens, SpaceX and Volskwagen [9, 20]. In spite of high costs, companies turn their heads to AM because of the many advantages it offers.

Additive manufacturing allows the production of parts with very complex shaped and intricate designs with no additional cost or process complexity. Through AM, the difficulty of producing a simple cube or a turbine blade with internal channels is virtually the same. With this technology, it is possible to produce advanced shapes and hollow parts that would otherwise be very difficult or impossible to produce. With subtractive manufacturing techniques, such as machining, it is necessary that the region of the component to be machined is accessible to the tool. In AM, this is not a concern [21].



Figure 1.4 – Number of AM systems for metals sold per year. Source: Wohlers Report 2018 [18].



Figure 1.5 – Earnings from sales of metallic feedstock for AM. Source: Wohlers Report 2019 [9].

With additive manufacturing, the design of the parts can be optimized in such a way that material is added only where it is necessary, leading to lower material consumption, less waste generation, weight reduction, enhanced performance and efficiency and possibly cost reduction. These benefits are appealing to industries and applications where weight reduction is deeply valued and material cost is elevated, so lower waste generation and material consumption are appreciated. AM becomes even more attractive in sectors in which cost is not a strong constraint. Examples of such industries are aerospace, medical and automotive (especially sports cars and luxury cars). Not by chance, the industry that leads the additive manufacturing application scene is aerospace, with 29% of all metal AM system sales revenue in 2019 (Figure 1.6) [22].



Figure 1.6 – Income from sales of metal AM systems by industry in 2019. Source: AMPOWER Report 2020 - Metal Additive Manufacturing [22].

In addition, additive manufacturing may prove useful when processing a material that is difficult to process with traditional methods such as casting, forging and machining. Metals with high liquidus temperature (hard to cast), high yield strength (hard to forge), high hardness and low thermal conductivity (hard to machine) and high costs, as well as metals that oxidize easily, are therefore prime candidates for additive manufacturing process instead of traditional processes [21]. These metals include titanium alloys and nickel alloys. Indeed, titanium alloys and nickel alloys are the top 2 most used materials in metal AM processes (**Figure 1.7**), with about 30% and 20% of all feedstock consumption (in mass) for metal AM in 2018 adding up to approximately half of feedstock consumption [23]. Coherently, these metals are widely used on the aerospace industry, in turbine blades, landing gear, springs, discs, exhaust and engine parts. The thesis at hand is based on the study of nickel alloy Inconel 718, one of the top used alloys for AM, and employed greatly in the aerospace sector, mostly for turbine blade production, due to its high temperature resistance and outstanding creep resistance [24].

Additive manufacturing is a family of processes, and it comprises different categories and different technologies. There are additive manufacturing processes for polymers, metals, ceramics and composites. Polymer AM is the oldest and more well established branch of processes, followed by metal AM, which is experiencing a rapid development. Ceramic and composites additive manufacturing processes are still in their early days [25]. In this work, the focus will be placed on metal additive manufacturing, since the experimental work was done with a nickel alloy.



Figure 1.7 – Material consumption as feedstock for AM, per alloy type. Source: Elaborated by the author based on data contained in AMPOWER Report 2019 - Metal Additive Manufacturing [23] (approximate values).

For metallic materials, the main process categories are powder bed fusion (PBF) – either with a laser beam or with an electron beam as the heat source –, directed energy deposition (DED) – either from powder or from wire –, binder jetting (BJ or BJT), material extrusion (or fused deposition modeling, FDM), material jetting (MJ). PBF and DED are the two most established and used technologies. Figure 1.8 illustrates key aspects of each technology and provides a schematic comparison between the main processes for metal AM [26].

Part of the reason why PBF and DED processes are the most used currently in industry is the superior mechanical properties (mainly yield strength) of parts produced by these methods (in comparison to other AM technologies). **Figure 1.9** illustrates a comparison between the main metal AM processes in terms of part performance (mechanical properties, mainly yield strength), cost of part production and lot size [27].



Figure 1.8 - Schematic comparison of the main processes for metal AM. Source: Roland Berger [26].



Figure 1.9 – Comparison of the main metal AM processes in terms of part performance. Source: Roland Berger [27].

Another reason why PBF and DED are the top two most employed AM process categories in industry is that they have the highest technological maturity among AM methods (they have been around longer and were more investigated in academia and through practice experimentation). **Figure 1.10** shows the "Maturity Index" for each technology of metal AM in 2020. The maturity index is a concept elaborated by AMPOWER, a German consulting firm specialized in additive manufacturing, and it takes into account the technological maturity of each process (technology maturity index), as well as the level of industrialization or effective application in industry (industrialization index). Based on that information, the firm predicts how long until the technology is well established within the industry. So far, only DED and PBF technologies have reached the maturity index, i.e. are well established and used in industry. Other than DED or PBF, the technologies that are closest to real application in industry are fused deposition modeling and binder jetting, both in the stage of first commercial industrial applications. **Figure 1.10** also portrays the current state of upcoming processes still in development (in bold), giving a good panorama of the state of the art of metal AM technologies [22].



Figure 1.10 – Maturity index of AM technologies. The names in bold refer to new technologies currently being developed, with no industrial use yet (in 2019).

Source: AMPOWER Report 2020 Additive Manufacturing [22].

For the reasons above mentioned and others, PBF and DED are the most currently used technologies for metal AM in industry, with PBF being the most common, accounting for 85% of metal AM systems sales revenue in 2019, and DED being the second most common, with 8% of metal AM systems sales revenue in 2019 (**Figure 1.11**) [22]. As a matter of fact, this work studies Inconel 718 parts made precisely through PBF and DED (as well as casting, the traditional method for forming Inconel 718 components), the most established technologies. More specifically, samples analyzed in this work were produced by laser based PBF and laser based DED (in addition to casting). In this thesis, microstructure and properties of Inconel 718 parts produced by these different techniques are compared.



Figure 1.11 – Earnings from sales of metal AM systems by technology in 2019. Source: AMPOWER Report 2020 Metal Additive Manufacturing [22].

Analyzing once more **Figure 1.8**, it is worth noticing that most metal AM techniques, including laser PBF and laser DED, require post processing operations in order to achieve optimal mechanical and metallurgical properties. This is also the case for traditionally cast Inconel 718. One of the most important and impactful parts of post processing of metal components is the thermal treatment, which can completely change the mechanical properties of the part, through optimization of its microstructure (present phases, precipitate concentration and morphology and grain structure and size) [24]. Other than the functional importance, post-processing also has a high financial importance, as it has a significant impact on the part final cost: a survey by Wohlers Associates aimed at companies that use additive manufacturing industrially (for metals and polymers as well) concluded that over one quarter of the cost of an additively manufactured part is associated with post-processing operations (**Figure 1.12**) [9]. This thesis, at last, studies specifically the impact of thermal treatments on the microstructure and mechanical properties of Inconel 718 parts produced by laser powder bed fusion, laser directed energy deposition and casting.



Figure 1.12 – Distribution of the cost of parts built by AM among the different stages of the fabrication (pre-processing, building and post-processing).

Source: Wohlers Report 2019 [9].

As expected, the relevance and interest in the topic of additive manufacturing has been rising exponentially in the past few years in academia as well. A quick search on Web of Science [28] shows that the number of results for a search on additive manufacturing rose from 938 documents in 2010 to 8015 documents in 2020. That is a growth of more than 750%.

More specifically, results for additive manufacturing of Inconel 718, grew from only 5 in 2010, to 230 in 2020. This is a growth of 4500%. As a consequence, the percentage of results for additive manufacturing of Inconel 718 has risen since 2013, from 0.2% to 2.9% in 2020 (in comparison to the total number of results for additive manufacturing in general). These numbers illustrate well the rise of interest on the topic of this work.

The research was done through the "Advanced Search" resource on the Web of Science website, using the parameter TS=((additive manufacturing OR SLM OR selective laser melting OR DED OR directed energy deposition) AND (Inconel 718 OR IN718)) for the results on additive manufacturing of Inconel 718 and the parameter TS=(additive manufacturing OR SLM OR selective laser melting OR DED OR directed energy deposition) for the results on general additive manufacturing. The results were generated on February 20th 2021 and are shown in **Figure 1.13**.

To sum up, the relevance of this work is evidenced by the increasing interest both in academia and in industry, the growing economic importance and the lack of complete understanding of the topic (natural to a newly developed technology).


Figure 1.13 - Number of scientific papers on on additive manufacturing of Inconel 718, as a result of a search on the Web of Science database, as of 02/20/2021. The number of papers on AM of IN718 is compared to the total number of AM papers, showing a relative increase in the interest for AM of IN718.

Source: Elaborated by the author based on data from the Web of Science database [28].

1.3.Inconel 718

1.3.1. Composition

Inconel 718 (UNS N07718) is a nickel-based alloy with substantial amounts of chromium, iron and niobium in its composition. Titanium, molybdenum and aluminum are also importantly present, although in lower concentrations. The matrix, an austenitic Ni-Fe-Cr solid solution with a face centered cubic (FCC) structure, is labeled γ . The matrix is strengthened through solid solution primarily by Mo, Mn and C atoms. Nonetheless, the main strengthening mechanism is precipitation hardening, through secondary phases γ' (Ni₃(Al, Ti, Nb)) and γ " Ni₃(Nb, Ti) [24]. Table 1.1 shows the typical composition of the alloy:

Table 1.1 – Nominal composition of Inconel718 (weight percentage).

Element	Ni	Cr	Nb	Мо	Ti	Al	Co	С	Fe
Content (% wt.)	50.00-55.00	17.00-21.00	4.75-5.50	2.80-3.30	0.65-1.15	0.20-0.80	≤ 1.00	≤ 0.08	Balance

Source: Elaborated by the author based on Yi et al.and AMS 5663 [29, 30].

Nickel and iron, together with chromium, are the main components of the matrix. Nickel stabilizes the austenitic γ phase (FCC) – the matrix itself, and also forms strengthening

precipitates γ' and γ'' . Chromium has the role of providing oxidation and hot corrosion resistance, as well as forming carbides to strengthen grain boundaries. Niobium plays the key role of forming strengthening precipitates γ' and γ'' alongside nickel. It also forms carbides. Titanium and aluminum originate γ' with nickel (and possibly niobium). Titanium can also form carbides, as well as iron. The role of carbon is evidently to form promote carbide formation. In addition, all these alloying elements, especially those with greater atom radius, such as niobium and molybdenum, contribute to solution hardening [31].

1.3.2. Microstructure

Inconel 718 is heat treatable and precipitation hardenable. Through adequate thermal treatments, strengthening precipitates γ' and γ'' can be formed. The precipitate γ' is composed of ordered cubic Ni₃(Al, Ti, Nb) and is a stable and desirable hardening phase. The precipitate γ'' is composed of body centered tetragonal Ni₃(Nb, Ti) and is metastable at room temperature, but highly desirable. In fact, in the alloy Inconel 718, γ'' is the main hardening phase. The precipitates γ' and γ'' have the greatest impact on the mechanical properties (hardness, yield strength, ultimate tensile strength) of the alloy because they are both semicoherent with the matrix, and usually nanometric and finely dispersed. The phase γ'' has a greater effect as a hardening phase because it typically has a disc-like morphology, and its elongated shape causes greater coherency strain on the interface zone with the matrix [24].

If the metastable phase γ " is exposed to heat for prolonged periods of time, it transforms into stable δ . The δ phase is composed of orthorhombic Ni₃(Nb, Ti) and is incoherent, partly losing its hardening effect. This phase has typically an acicular morphology, and is coarser than its metastable counterpart γ ", which also helps explaining the lesser contribution to strengthening the alloy. Moreover, the δ phase often forms at grain boundaries, which can prevent or hinder grain boundary sliding. This effect is beneficial to creep resistance – an important characteristic of Inconel 718 –, but detrimental to ductility [24].

In addition to δ , there is the incoherent η phase, composed of hexagonal Ni₃(Ti, Al), with similar effects to those of the δ phase. Other than these intermetallic phases, carbides MC and M₂₃C₆ are also found in the Inconel 718 typical microstructure. These incoherent phases offer smaller contribution to hardening of the alloy, since they are not sheared by dislocations as their coherent or semi-coherent counterparts. Their effect on hardness and tensile properties depends on the size and distribution of the particles. Coarse and poorly dispersed particles contribute less to hardening. Fine and dispersed particles can grant a finer microstructure, and precipitates located at grain boundaries can benefit creep resistance (through hindering grain boundary slipping) and harm ductility [24].

Another phase of major importance is Laves, composed of hexagonal (Ni, Cr, Fe)₂(Nb, Ti). This phase usually has a detrimental effect on the mechanical properties, as it is a brittle phase with an often continuous morphology that appears frequently at grain boundaries, decreasing ductility and impact toughness of the alloy [24].

Besides their direct less-than-ideal effect on mechanical properties, the secondary phases δ , η , carbides and Laves consume atoms of important alloying elements (Al, Ti and Nb) that could be forming the most desirable γ' and γ'' precipitates. For those reasons, these phases are usually undesired. **Table 1.2**, elaborated by Ferreri et al [24], summarizes the main phases and their key characteristics. Typical contents of γ' , γ'' and δ are around 4%, 16% and 5% respectively (volumetric fraction) [32].

	Phase	Crystal System	Primary Composition
Solid Solution Matrix	γ	Cubic	Ni,Cr,Fe – Based
Intermetallic	γ'	Cubic	Ni₃(Ti,Al,Nb)
	γ''	Tetragonal	Ni ₃ (Nb,Ti)
	δ	Orthorhombic	Ni ₃ (Nb,Ti)
Topographically Close- Packed (Intermetallic)	η	Hexagonal	Ni ₃ (Ti,Al)
	Laves	Hexagonal	(Ni,Cr,
			Fe) ₂ (Nb,Ti)
Carbide	MC	Cubic	(Nb,Ti) (C,N)
	$M_{23}C_6$	Cubic	(Cr,Fe) ₂₃ C ₆

Table 1.2 – Main phases in Inconel 718

Source: Ferreri et al. [24].

1.3.3. Properties

Inconel 718 is a nickel superalloy. Superalloys are alloys with outstanding mechanical and thermal properties. One of such properties is creep resistance. Most metals are subject to creep when exposed to a homologous temperature of 0.4 (40% of their melting temperature or solidus temperature). Superalloys can withstand homologous temperatures as high as 0.6 (60% of their melting or solidus temperature) or greater. Inconel 718 is one such material, as it can be used up to a homologous temperature of 0.7 (approximately 880 °C). If the alloy is exposed to this temperature for long periods of time, the microstructure does degrade (overaging), but very slowly, allowing an acceptable life even in such adverse conditions [31].

Inconel 718 has a density of 8190 kg/m³, and its solidus and liquidus temperatures are 1260 °C (1533 K) and 1336 °C (1609 K), respectively [33]. When properly heat treated (successful precipitation of strengthening phases γ ' and γ ''), the alloy shows attractive mechanical properties. At room temperature, the yield strength is 1035 MPa (150 ksi), the ultimate tensile strength is 1240 MPa (180 ksi), elongation is 12%, the hardness is 36 RC (344 HV), and the Young's Modulus is 200 GPa (29.000 ksi) [AMS 5596]. Inconel 718 has a relatively low thermal conductivity of 11.386 W/m·K (79 BTU·in/h·ft^{2.°}F) [34].

Inconel 718 retains good mechanical strength at elevated temperatures. Although the alloy can be used up to 880 °C in certain cases, the usual range of maximum temperatures at which it is commonly used is 650-700 °C. This range is enough for the typical applications and protect the alloy from overaging (or at least it occurs at a slower rate) [31]. At 650 °C (1200 °F), yield strength is 825 MPa (120 ksi), ultimate tensile strength is 1000 MPa (140 ksi) and elongation is 5%. In stress rupture tests (accelerated creep tests with greater stresses), failure occurs at 690 MPa (100 ksi) after 23 hours, with 4% elongation [34]. These properties are valid for heat treated wrought Inconel 718 bars in accordance with AMS 5596 [35].

These properties come at a cost, though. Inconel 718 is not a cheap material. The cost of the alloy is around 50-60 USD/kg in March/2021 [36]. This is natural, since the cost of its main component, nickel, is relatively high as well. The price for nickel in March/2021 is set at 16.425 USD/ton by the London Metals Exchange [37]. For reference, at the same date, aluminium is negotiated at 2.254 USD/ton [37].

1.3.4. Applications

With this extraordinary set of properties, Inconel 718 is chosen for applications that require high mechanical strength and oxidation and corrosion resistance, especially at high temperatures. Perhaps the most outstanding properties of this alloy are its high temperature resistance (or high mechanical strength at elevated temperatures) and its high creep resistance. For this reason, the main use for Inconel 718 is in the aerospace industry, primarily as turbine blades, and also as jet engine parts and exhaust. The high prices make it more suitable for high-end industries in which cost is not a strong constraint – precisely the case of the aerospace sector. Additionally, Inconel 718 is also used on stationary gas turbines for energy generation, with similar operation conditions. To a lesser extent, the alloy is used in dies and extruders [38].

In a typical jet turbine (Figure 1.14), peak temperature can exceed 1500 °C. The temperature on the turbine metal blades, however, is usually within 600-800 °C, thanks to cooling mechanisms. Most commonly, the cooling mechanism is comprised of internal channels in the blade, through which cool air flows (it is also possible to use a liquid coolant or water, but in this case, corrosion problems may arise). This intricate design increases complexity and difficulty of manufacturing the part through conventional methods. It is still done, however, because it allows the turbine to operate at a higher temperature, yielding greater efficiency for the engine. In addition to the channels, a ceramic coating may be applied through physical vapor deposition (PVD) [31].



Figure 1.14- Schematic illustrations of jet turbines and the materials commonly used for each component. Source: Srivastava [39] (left); Kushan et al. [31] (right)

1.3.5. Processing

Traditionally, Inconel 718 parts are cast or wrought. In the case of turbine blades, casting is the preferred method. More specifically, investment casting. Often, Inconel 718 turbine blades are cast as a monocrystal, enhancing creep resistance. To obtain the internal channels for cooling, a ceramic core is used. Investment casting of turbine blades is a highly complex and time consuming process. Other than the casting operation itself, the wax pattern must be prepared, the ceramic mold must be produced; the ceramic cores must be fabricated and heat treated for over 24 hours; after casting, the ceramic cores must be dissolved; auxiliary features for casting must be removed through machining; additional holes for cooling must be formed through electrical discharge machining; at last, the blades receive the ceramic coating for thermal insulation [40].

Some of these operations include handmade steps, which increase process time and overall cost, and decrease consistency and quality uniformity. In addition, not all industrial

facilities have the capacity of undertaking all these operations on the same site. This can lead to extra transportation operations, implying great logistical complexity, increasing both costs and time consumption even further. For instance, Rolls-Royce single crystal turbine blades are cast in Coventry, UK, then sent out to Crosspointe, Virginia, USA, for machining, and back to UK for coating in Annesley, Nottinghamshire [41].

Mechanical processing of Inconel 718 is no easy task either. Its high yield strength makes it difficult to forge, press or roll, even at elevated temperatures, since the alloy retains good mechanical resistance up until 0.7 Tm (around 880 °C). As the alloy shows high hardness and low thermal conductivity, it is also difficult to machine. An alternative processing technique that may be promising for Inconel 718 is additive manufacturing [42].

1.4.Additive manufacturing

As stated before, additive manufacturing (AM) is a family of processes in which parts are fabricated through the addition of material where necessary in order to form a part with a design specified by a CAD file. The geometry is built up in a bottom-up fashion, usually layer by layer [21].

This strategy of fabrication yields several advantages over traditional manufacturing methods (forging, rolling, pressing, machining, casting). It allows the production of parts with intricate and complex designs and geometric features that would otherwise be very difficult or impossible to obtain, including hollow parts or internal channels. AM is a near net-shape process, so the produced part requires little or no machining. The process does not depend on mechanical efforts or stresses, so the processing of materials with high mechanical strength is not a problem; yet, the resulting mechanical properties are superior to those of cast parts [42]. In addition, additive manufacturing can be virtually a single step process, potentially saving time and money when compared to multi step processes like investment casting, especially when logistics is taken into consideration.

1.4.1. Process Categories

There are many types and categories of additive manufacturing processes, for metals, polymers and ceramic materials. This work is centered on the nickel alloy Inconel 718, so the literature review is mainly limited to metal additive manufacturing processes.

ASTM and ISO jointly define 7 categories of additive manufacturing: binder jetting, directed energy deposition, material extrusion, material jetting, powder bed fusion, sheet lamination and vat polymerization [1]. All of these techniques can be applied to metallic materials, with exception of vat polymerization, which is used only for polymers. However, material jetting and sheet lamination are still in the development stage and find very few and limited applications. Material extrusion (better known as fused deposition modeling, FDM) and binder jetting are processes that come from polymer AM and were adapted to metallic materials recently. These processes are currently being used in their first industrial applications. In 2019, they represented 2% and 3%, respectively, of AM systems by sales revenue [22]. In addition, the resulting mechanical properties of metal parts produced by FDM or binder jetting are less than ideal, and components produced through these methods cannot yet be used in critical applications [27].

On the other hand, directed energy deposition (DED) and powder bed fusion (PBF) are the two most used processes, and the processes that yield the best mechanical properties among AM technologies, fit for end part manufacturing, even in some critical applications [27]. PBF is the dominant technology, accounting for 85% of AM systems sales revenue in 2019. Additionally, it is the process category that produces the best mechanical properties (mainly yield strength and ultimate tensile strength). DED is the runner up process, with 8% of AM systems sales revenue in 2019 [22]. It is currently the top candidate to compete against PBF, an older and more established technology.

AM processes are classified into single-step and multi-step processes. Single-step processes are those in which the basic geometry, as well as mechanical properties, are achieved in one operation. Multi-step processes are those that require more than one operation to consolidate the basic desired shape and mechanical properties. Usually, the basic shape is obtained first, and a second operation is needed to promote greater strength of the part. Typically, the second operation is some form of curing or sintering. For metallic materials, these multi step processes involve a polymer that act as an intermediate to attain the desired shape, and then this polymer must be eliminated and the metal part consolidated, usually through sintering. Multi-step processes are commonly more time consuming and produce lower mechanical properties (mainly yield strength, ultimate tensile strength and hardness) than single-step processes. FDM and binder jetting are examples of multi-step processes. Both DED and PBF are single-step processes, which partially explains the superior mechanical strength they yield and why their use is more widespread in industry [1].

This work investigates Inconel 718 parts produced by DED and PBF. For this reason, these two processes categories are the focus of the literature review on AM. Although they are very similar in many ways, DED and PBF present serious differences that yield different advantages and disadvantages.

1.4.2. Powder Bed Fusion

For metallic materials, powder bed fusion is a category of AM processes that are based on melting specified areas of a layer of powder in order to form a consolidated part upon solidification of molten areas. After the specified areas on a layer of powder are successfully molten and solidified, the platform where the powder layer is laid is lowered and another layer of powder is spread onto the previous one. On the new layer of powder, selected areas are molten and solidified, after which another layer is spread, and so on [21]. ISO/ASTM 52900 defines powder bed fusion as an "additive manufacturing process in which thermal energy selectively fuses regions of a powder bed" [1]. Figure 1.15 shows a schematic illustration of the LPBF process [21].

The thermal energy only melts regions that add up to be the desired part design (and possibly some support structures). The thermal energy comes from a heat source. The heat source differentiates the two technologies that are categorized under the umbrella of powder bed fusion. The heat source can be a laser beam or an electron beam. If a laser beam is used, the process is called Laser Beam Powder Bed Fusion (LB-PBF), or one of its many variations. If the energy is originated from an electron beam, the technology is labeled Electron Beam Powder Bed Fusion (EB-PBF) – again, there are many variations of this name [21].

Common names for LB-PBF include: Laser Powder Bed Fusion (LPBF, L-PBF, or even PBF-L), Selective Laser Melting (SLM, its most famous commercial name), Selective Laser Sintering (SLS, a primitive name for the technology that is no longer used, since it is now known that the metal is in fact molten, and not only sintered), Direct Metal Laser Sintering (DMLS, also in disuse due to the misconception that the metal is sintered) and Direct Metal Laser Melting (DMLM), Laser Cusing (name coined by system manufacturer Concept Laser), among others [21].



Figure 1.15 – Illustrative depiction of the LPBF process. Source: DebRoy et al. [21].

For EB-PBF, alternative names are Electron Beam based Powder Bed Fusion (EBPBF, PBF-EB) and Electron Beam Melting (EBM, its most common commercial name, coined by the company Arcam, which first invented the technology and was recently acquired by GE) [18].

The main difference between these two processes is the heat source itself, but the change in the heat source implies other different characteristics. LB-PBF is by far the most established and used PBF technology [22]. In LB-PBF, the powder bed is kept in a chamber with an inert atmosphere (mostly argon). The laser directly melts the powder on the regions that form the part design [21].

In EB-PBF, the electron gun must work on vacuum, so the build chamber is kept under vacuum. Moreover, in EB-PBF, the powder bed must be preheated, to avoid electrostatic charging and repulsion of powder particles (which can lead to defects on the built part). With this purpose, a defocused electron beam scans the whole powder bed prior to selective melting, each time a new layer of powder is spread. This can make EB-PBF more time consuming than LB-PBF. Moreover, the preheating lowers the heat extraction rate, so EB-PBF often has lower cooling rates than its laser based counterpart. As a consequence, parts produced by EB-PBF tend to show lower residual stresses than parts obtained by LB-PBF [43].

In addition, LB-PBF processes typically have a spot size (the diameter of the beam as it hits the powder bed) of 50-100 μ m, while EB-PBF processes usually operate in the 100-200 μ m range (due to differences in the technology used for focusing the beam – optical lenses for laser beams and magnetic lenses for electron beams). As a consequence, LB-PBF tends to have a finer resolution. Also contributing to that effect is the powder size distribution: powders for LB-PBF usually have diameters of 10-60 μ m, while powders for EB-PBF usually stay in the 60-105 μ m range [21].

These differences might partially explain the triumph of LB-PBF over EB-PBF in industry in terms of usage.

1.4.3. Directed Energy Deposition

Directed energy deposition is a category of additive manufacturing processes for metals that is defined by ISO/ASTM 52900 as an "additive manufacturing process in which focused thermal energy is used to fuse materials by melting as they are being deposited" [1]. DED works by adding metallic feedstock to a molten pool located on the substrate where new

material is being deposited onto or added to. This way, metal is added where needed to form a part with the desired shape in a bottom-up fashion. As the new material is added, it goes into the molten pool and melts. Once it solidifies, it is consolidated within the part that is being formed. DED is similar to welding, in some ways [21].

DED is, just as PBF, a category of processes. In this category, there are two types of processes, which differ by the form of the metallic feedstock. One technology is powder DED, in which the feedstock is a metallic powder and the heat source is a laser beam. The other technology is wire DED, in which the feedstock is a metallic wire and the heat source can be an electron beam, an electric arc, a plasma arc, or a laser beam [26].

Powder DED far outweighs wire DED in usage. Powder DED additive manufacturing is more developed and applied than its wire based counterpart [22]. In powder DED, the metallic powder is fed through a nozzle and projected into the molten pool, which is created by the laser beam. The powder jet melts as it enters the molten pool. In many pieces of literature, powder DED is referred to as simply DED, since the wire based variation of the process is less known, less developed and less used. Another common label is L-DED or DED-L, meaning laser based DED, since the wire form of the technology is more commonly seen associated with an electron beam as the heat source. Other names used for powder DED include Laser Additive Manufacturing (LAM), Laser Engineering Net Shaping (LENS), Direct Metal Deposition (DMD), Laser Direct Metal Deposition (LDMD), Direct Laser Fabrication (DLF), Laser Powder Deposition (LPD) and Laser Solid Forming (LSF) [21]. Figure 1.16 shows an illustration of a LDED system [21].



Figure 1.16 – Illustrative depiction of the LDED process. Source: DebRoy et al. [21].

In wire DED, the heat source – be it an electron beam, an electric arc, a plasma arc or a laser beam – melts the substrate, creating a molten pool, and the wire is constantly fed into the molten pool, where it melts. Once it solidifies, it integrates the formed part. Perhaps the most known wire DED technology is Sciaky's Electron Beam Additive Manufacturing (EBAM). Wire DED technologies are greatly based on or inspired by welding techniques [21]. Figure 1.17a shows an illustration of an EBAM system [44], and Figure 1.17b displays a schematic drawing of am arc-based wire-DED process (also known as WAAM, for Wire Arc Additive Manufacturing) [21].

In powder DED, with a laser beam as the heat source, the powder is projected from a nozzle into the melting pool. The nozzle also releases a shielding gas (typically argon) that not only protects the molten metal from oxidation, but also carries the powder into the melting pool, for a more directed flow. In this technology, there is no building chamber, so the dimensions of parts that can be deposited are not limited by it [45].



Figure 1.17 – Schematic illustrations of EBAM (a) and WAAM (b). Souce: Sciaky [44] (a); DebRoy [21] (b).

In wire DED with an electron beam as the heat source, the process must be undertaken in vacuum (in a vacuum chamber), and the wire is fed directly into the molten pool created by the electron beam. The vacuum chamber limits the size of parts and features that can be built using this technique. Wire DED with a laser beam occurs in an analogous way, except the heat source is a laser beam, so there is no need for vacuum [45].

In wire DED with plasma or electric arc as the heat source (also known as WAAM – wire arc additive manufacturing), the process resembles welding: an arc is created between the wire and the substrate (or the part currently being deposited), and the metal is transferred into the molten pool [21].

Powder DED provides better surface finish, lower surface roughness and greater accuracy and finer resolution, since powder particles are smaller than metal wires. The downside is that this process is more time consuming, for the same reason, so it is typically used for deposition of smaller parts with more intricate design features. On the other hand, wire DED techniques can be used for large parts, even greater than 10 kg. The deposition rate achieved with wire as feedstock is higher, since the wires, that are larger than powder particles, allow for higher mass flow rate (feedstock wires have diameters typically greater than 0.8 mm). The flip side is that wire DED processes yield poor surface finish and resolution, and usually require machining operations. Wire feedstock is cheaper and easier to find than powders fit for AM. However, the savings may be compensated by greater machining costs with parts produced by wire DED [21].

1.4.4. PBF x DED

PBF and DED are two different technologies that work in different ways. The most blatant difference is the mechanism of the process itself. In PBF, a stationary layer of powder is selectively molten, then another layer of powder is spread on top of the previous one, and so on and so forth. In DED, the powder is projected and deposited onto the substrate, into a melting pool. For DED, the feedstock can also be a wire, in which case the wire is fed continuously into the melting pool, or an arc is created between the wire and the substrate, similarly to welding techniques [46].

Both PBF and DED have variations of their own (LB-PBF, EB-PBF, powder DED, wire DED), which were briefly covered in the previous section. In this work, Inconel 718 parts produced by LB-PBF (also known as SLM) and by powder DED are investigated. For this reason, these two technologies are the focus of the literature review. Moreover, these two are

the top two most used technologies in industry, so it makes sense to explore them further. In this section, a comparison between PBF and DED will be established, first in general aspects that apply for all their variations, and then, a comparison specifically between LB-PBF and powder DED will be set.

PBF processes happen inside a build chamber, defined by the AM machine. This imposes a limit to the size of parts that can be manufactured. In DED processes, there is no chamber (except for wire DED with an electron beam as heat source), so there is virtually no limitation to the dimension of parts that can be build, except for operational limitations like the size of the room where the machine is and such [21].

The spot sizes of the heat sources in PBF processes is finer than those in DED processes. In the first, it is in the order of tens to hundreds of microns. In the latter, it is around 1 mm. This, coupled with the fact that feedstock for DED is often coarser than feedstock for PBF (especially in the case of wire DED, but also for powder DED), gives the DED processes a coarser resolution when compared to PBF. The level of detail attainable with PBF is greater, smaller and more intricate features are possible, and the surface finish is better than with DED. On the other hand, coarser spot sizes and feedstock allow a higher build rate (deposition rate) for DED processes when compared to PBF. The lower build rates, as well as the fact that a new powder layer must be spread after each selective melting stage, makes PBF more time consuming than DED. DED processes generally have lower cooling than PBF. As a consequence, parts build by DED usually show lower residual stresses than those made with PBF [21].

At last, using DED processes is overall cheaper than choosing PBF. DED systems are more financially economic to acquire, the feedstock for DED (including powders) is more financially accessible than powders for PBF, and even when hiring a service provider to build the desired part via AM, doing it by DED leads to a lower bill than opting for PBF [47].

One must not forget, however, that DED provides lower resolution, higher dimensional tolerances, lower accuracy, and poorer surface finish. Depending on the application and design, parts produced by DED may require non negligible machining efforts, which may partially offset economic advantages over PBF [47].

To sum up, it is reasonable to state that DED is more suitable to make larger and less detailed parts, with simpler design, or when budget is tighter, while PBF is a more adequate choice to fabricate smaller parts with intricate design and complex geometric features, if greater accuracy is desired [47].

1.4.5. Process Parameters

In order to accurately describe the processes, some process parameters, common to most AM technologies, are established. Process parameters, in a broader sense, include the type of heat source (laser beam, electron beam, electric arc, plasma arc), the number of heat sources, the type of feedstock (powder or wire), the heat source power (in Watts), the speed with which the heat source moves while melting the feedstock (scanning speed, usually given in mm/s), layer thickness (the thickness of each added layer, regardless if the feedstock was molten in a powder bed or deposited onto a molten pool), build rate (rate at which material is added, regardless if it is deposited or molten in a powder bed; for DED processes, build rate is often replaced by deposition rate; build rate or deposition rate are given in units of mass per unit of time, or units of volume per unit of time, i.e. kg/h, cm³/h), hatch spacing (the distance between two neighboring tracks of added material, usually in mm), spot size (diameter of the heat source beam as it impinges the powder bed or the part being built), scanning strategy (the

order and direction of scanning or deposition for each layer), build volume dimensions (dimensions of the volume within which parts can be built), part orientation within the build volume, build atmosphere, accuracy and resolution. In addition, feedstock characteristics are important and affect the outcome of the process. For powders, important characteristics are morphology, size distribution and flowability, among others [48].

In a stricter sense, the main process parameters are scanning speed, heat source power, powder flow rate (for DED) or layer thickness (for PBF) and hatch spacing. These are parameters that can be controlled and greatly influence the outcome of the process. These variables directly influence the heating and cooling rate, temperature gradient, solidification rate, and undercooling, which ultimately determine solidification structure, phases present, grain size, texture, microstructure and mechanical properties in general, as well as defects and residual stresses (generated due to temperature gradient and thermal expansion) [48]. **Table 1.3** summarizes the process parameters for the main AM technologies as reported by DebRoy et al. [21].

PBF-L	DED-L			
Beam power, W	Beam power, W			
Energy Density, J/mm ²	Energy Density, J/mm ²			
Beam focal offset, spot size	Beam focal offset, spot size			
Hatch spacing (line offset), µm	Hatch spacing (line offset), μm			
Scan speed, m/s	Travel speed, cm/s			
Scan strategy	Powder flow rate			
Powder, PSD, morphology	Powder, PSD, morphology			
Powder layer thickness, µm	Powder layer thickness, µm			
Powder moisture level	Powder moisture level			
Deposit layer, Z step, µm	Deposit layer, Z step, µm			
Build plate preheat, °C	Inert gas flow			
Powder bed preheat, °C				
Chamber gas, ppm O ₂ , H ₂ O				
Additional PBF-EB parameters	Additional DED-EB, DED-PA parameters			
Chamber vacuum	Volts, Amps, wire feed rate			
Sinter pass parameters	-			

Source: DebRoy et al. [21].

1.5. Thermal treatments

Regardless of the processing method, Inconel 718 parts must be heat treated. Parts in the as-produced state have less than ideal mechanical and thermal properties, due to a suboptimal microstructure. The thermal cycles experienced during forming processes are projected for the sake of the shaping technique, and not for the part microstructure optimization. As-cast IN718 parts have significant amounts of Laves phase, which is brittle and detrimental for mechanical properties, and have no γ ' or γ ''. As-built AM parts show no γ ' or γ '' as well, and instead, have noticeable quantities of acicular delta phase [24].

The primary goal of thermal treatments for Inconel 718 is to optimize its microstructure. Since Inconel 718 is a heat treatable alloys that takes advantage of precipitation hardening, the principal way of optimizing its microstructure is by promoting the precipitation of the strengthening phases γ ' and γ ''. However, in order to do so, it is first necessary to solubilize previously existing secondary phases into the matrix, so that undesired phases (such as Laves) are eliminated and the concentration of atoms needed to form the desired precipitates increases, making the process more favorable [49].

Thus, the first step of standard heat treatments for Inconel 718 is a solution treatment. After solubilization, aging treatments are necessary in order to promote the formation of precipitates γ' and γ'' . The SAE (Society of Automotive Engineers) AMS (Aerospace Material Specification) standard for wrought Inconel 718 parts for aerospace applications, AMS 5662, recommends heat treatment as per AMS 2774 (standard heat treatment for wrought Inconel 718) [50]. This includes a solution treatment at 980°C for 1 hour with water quenching, plus a double aging treatment, first at 720°C for 8 hours, then furnace cooling at 50 °C per hour to 620°C, then holding for 8 hours at 620°C, finishing with air cooling [51]. The SAE AMS standard for investment cast Inconel 718 parts, AMS 5383, encourages heat treatment as per AMS 2773 (standard heat treatment for cast Inconel 718), which is the same heat treatment for wrought Inconel 718, with the addition of a homogenization treatment beforehand, at 1080 °C for 1 hour, followed by water quenching [52, 53].

ASTM (American Society for Testing and Materials) presents a similar approach. The ASTM standard for precipitation-hardening Inconel 718 parts for high temperature service (ASTM B670) recommends a precipitation heat treatment consisting of a double aging treatment, first at 718°C for 8 hours, then furnace cooling to 621°C, then holding at 621°C until total heat treatment time reaches 18 hours, finishing with air cooling [54].

As additive manufacturing is still a relatively novel technology, it is still being standardized, and it is still being studied, as it is a complex process that involve many phenomena with effects, possibilities and limitations that are still being understood. There are already some ASTM standards for AM, but many are still to be developed. ASTM standard F3055 sets the best practices for Inconel 718 parts produced by PBF. This standard recommends the same thermal treatment as for wrought Inconel 718, AMS 2774. ASTM standard F3301, which states best practices for heat treating different alloys produced by PBF, also recommends the same heat treatment as for wrought Inconel 718, AMS 2774 [55, 56]. At the time of the undertaking of the work at hand, there are no ASTM standards for the heat treatment of Inconel 718 parts produced by DED.

In addition to promoting precipitation hardening and solubilizing undesired phases, thermal treatments can also relief residual stresses and regulate grain size and morphology [57]. Since Inconel 718 parts produced either by casting or AM do not show optimal phase distribution and mechanical properties, and AM parts have high residual stresses due to the high cooling rates experienced during building, thermal treatments prove absolutely necessary for critical applications such as turbine blades [58]. This work assesses the effect of standard heat treatment for wrought Inconel 718 (AMS 2774) on the microstructure and hardness of parts produced by AM - both SLM and DED – to see if the results are satisfactory and ultimately, if it is reasonable to apply a thermal treatment developed for wrought parts on additively manufactured ones. For comparison, the same treatment is also applied on cast parts.

2. LITERATURE REVIEW

Both casting and additive manufacturing are thermal-based processes that rely on melting and solidification to form parts. As such, their outcome is dependent on variables that are temperature-related. The key variables that ultimately determine the microstructure and mechanical properties of parts produced by additive manufacturing are temperature gradient (G), solidification rate (R) and undercooling (ΔT). These variables, in turn, are dependent on process parameters. For AM technologies, the chief process parameters are heat source power, scanning speed, beam spot size, powder size distribution and powder flow rate (for DED) or layer thickness (for PBF), and hatch spacing and scanning strategy. For casting, the principal process parameters are superheating, pouring temperature, cast volume, mold surface area, mold material (mainly because of its thermal conductivity, which governs the cooling rate [21].

In terms of process parameters, the main difference between casting and AM is that, in AM technologies, since the heat source is localized and has great power, local heating and cooling rates are very high, as well as temperature gradients (in the order of 500 K/mm [61]), much higher than in casting processes. For LB-PBF, typical cooling rates are on the order of 10^5 to 10^6 K/s; for powder DED with laser, cooling rates usually range from 10^3 to 10^4 K/s; in traditional casting processes (including investment casting), cooling rates stay within 10^0 to 10^2 K/s (cooling rates determine between the liquidus and solidus temperatures of the alloys processed) [21].

These elevated cooling rates give additively manufactured parts a rather fine microstructure, which translates into high yield strength, ultimate tensile strength and hardness. On the other hand, high cooling rates result in significant residual stresses on parts built by AM, which harms ductility and toughness. Since the cooling rate in PBF is higher than that of DED, parts produced by PBF have a finer microstructure, with generally superior tensile strength but higher residual stresses [65].

While the microstructure of cast Inconel 718 parts is fairly well studied and understood (although very complex), these features give AM parts a distinguished microstructure and unusual characteristics. The two most important factors governing the solidification and the microstructure of AM parts are the temperature gradient (G) and the solidification rate (R) at the solid-liquid interface. The multiplication of G and R, gives birth to the cooling rate (GR), which governs the microstructure scale. The ratio G/R directly influences the morphology of grains during solidification [21].

In an attempt to better understand, explain and predict the microstructure of parts built by AM, solidification maps (Figure 2.1) are used (an idea extracted from welding). Depending on the ratio G/R, grain morphology can be equiaxed dentritic, columnar dendritic, cellular or planar (with increasing G/R). For each of these morphologies, and increasing product GR makes the microstructure finer, whereas decreasing GR yields a coarser microstructure [21, 59].

The most usual grain morphologies found in additively manufactured metal parts are columnar and equiaxed structures [21]. However, due to the high temperature gradient experienced in AM processes (in the order of 10^6 K/m), equiaxed grains are less common. Equiaxed grain structures normally have equiaxed dendritic substructures, while columnar grains can present cellular, columnar dendritic or planar substructures. Equiaxed grains are usually finer and result in isotropic properties and greater ductility and toughness. Columnar

grains are normally coarser and align closely in the directions of greater heat extraction, leading to intense anisotropy. In spite of this, columnar grains are beneficial for enhanced creep resistance, a key attribute of Inconel 718 [59]



Figure 2.1 – Schematic solidification map. The microstructure can be predicted based on the temperature gradient (G) and on the grain growth rate (R). Source: DebRoy et al. [21].

Other major difference between AM and casting is that in AM each region of the part experiences multiple thermal cycles and temperature peaks. The first heating happens when the feedstock is first molten. When the next layer is added, the immediate previous layer is partially remelted to ensure proper part consolidation and cohesion between the layers. After this, for each new layer that is deposited or selectively molten, all the previously existing layers are reheated, at least to some extent, by the energy (heat) provided to melt the layer currently being built. This adds to the complexity of AM microstructures, as the solidification microstructure can be altered by following thermal cycles during the build [21].

In AM, as a new layer of material is created, heat flows from the newly added layer (where the heat source impinges and the highest temperatures are seen) to the original substrate (the coolest part of the build, furthest from the heat source). The direction of greatest heat extraction, coupled with crystallographic orientation (preferential solidification direction), determine the grain growth direction. For this reason, columnar grains in AM parts are typically aligned along a direction that is close to the build direction [21].

As new layers are added, the last added layer is further and further away from the substrate (the coolest part of the system), so heat extraction is hindered with every new layer. Because of this, layers on the upper region of a part built by AM experience higher temperatures for longer periods of time and lower cooling rate and G/R ratio (G/R decreases with higher scanning speeds and with distance from the substrate), which generates larger melt pools and coarser microstructure. This way, not only are AM parts anisotropic due to columnar grain structures, but the microstructure is also heterogeneous within the part, which

evidently leads to anisotropic and heterogeneous mechanical properties. This is part of the reason why thermal treatments are almost always necessary for AM parts [21].

Another key difference between AM and casting is that, in most AM processes, nucleation is not necessary during solidification. This is the case when the metal being added has is the same (chemically) as the substrate or the previous layer. In these circumstances, since both the newly added material and the previously existing material have the same crystal structure, there is no need for nucleation, and the grains in the previously existing layer can simply grow into the melt pool and into the newly added layer. This is called epitaxial growth, and is a chief concept in AM microstructure [21, 59]. Through epitaxial growth, grains can grow across several subsequent layers.

The direction of growth is determined jointly by the direction of greatest heat extraction (or temperature gradient) and the preferential growth crystallographic directions. For Inconel 718 (and other metals with cubic crystal structure), the preferential growth direction is <100> [59]. At the solid-liquid interface, dendrites with the <100> crystallographic direction closely aligned with the direction of maximum heat extraction are favored with higher growth rates, outgrowing dendrites otherwise oriented (competitive growth). The maximum heat flow direction is, locally, generally perpendicular to the melt pool boundary [59]. However, macroscopically, the maximum heat extraction occurs along the build direction. All these factors contribute to the generation of a widely observed columnar grain structure aligned somewhat closely with the build direction (angular variations may happen due to crystallographic orientation and heat source motion).

Lower cooling rates lead to coarser structures, since there is more time for them to grow. Moreover, lower cooling rates produce lower undercooling, which also favors coarser structures, since coarser structures are associated with lower specific surface area and surface energy. Analogously, higher cooling rates lead to finer structures. This is the main reason behind the transition from equiaxed dendritic, to columnar dendritic, to cellular and finally planar structures [59].

To add to the complexity, different AM processes result in different microstructural features. Since DED and PBF have different process principles and parameter range, the outcome, including microstructure and texture, is fairly different. PBF yields columnar grains more closely aligned with the build direction. This is related to the melt pool geometry, which is directly affected by process parameters and is different for PBF and DED. In PBF, the lower power and higher scanning speeds produce a shallow and long melt pool. As a result, grains grow epitaxially in a direction very close to the build direction. In DED, the melt pool is deeper, with comparable length and depth. As a consequence, grains are more deviated from the build direction, tilted towards the direction of motion of the heat source. If the scanning direction is switched from one layer to the next, a zig-zag grain structure may be formed. The main reason behind the different melt pool shapes is the remarkable difference in scanning speed between PBF and DED. While the first uses speeds typically around 1 m/s, the latter has usual values of around 10 mm/s [21]. Table 2.1 shows typical ranges of process parameters and characteristics for DED and PBF [21].

Figure 2.2 portrays a schematic illustration of melt pool shapes for PBF and DED [21].



Figure 2.2 – Schematic drawing of the typical melt pool shapes for PBF and DED. Source: DebRoy et al. [21]

Table 2.1 – Comparison of the typical range of process parameters of the main categories of additive manufacturing processes for metallic components (DED and PBF). L stands for "laser", EB for "electron beam", PA for "plasma arc" and GMA for "gas metal arc".

Process	DED	PBF			
Feedstock	Powder	Wire	Wire		
Heat source	Laser	E-beam	Electric arc	Laser	E-beam
Nomenclature	DED-L	DED-EB	DED-PA/DED-GMA	PBF-L	PBF-EB
Power (W)	100-3000	500-2000	1000-3000	50-	1000
Speed (mm/s)	5–20	1-10	5-15	10-	1000
Max. feed rate (g/s)	0.1-1.0	0.1-2.0	0.2-2.8		-
Max. build size $(mm \times mm \times mm)$	$2000\times1500\times750$	$2000\times1500\times750$	$5000\times 3000\times 1000$	500 × 2	80 × 320
Production time	High	Medium	Low	Н	igh
Dimensional accuracy (mm)	0.5-1.0	1.0-1.5	Intricate features are not possible	0.04	4-0.2
Surface roughness (µm)	4-10	8-15	Needs machining	7-	-20
Post processing	HIP and surface grinding are seldom required	Surface grinding and machining is required to achieve better finish	Machining is essential to produce final parts	HIP is required por	s rarely to reduce osity

Source: DebRoy et al. [21]

Parts with the same geometry and mass, made from the same material, can have significantly different microstructure, texture and properties when fabricated by different processes or using different process parameters. A classic texture in AM parts is <001> fiber texture, made up from columnar grains that grew epitaxially through several layers, aligned closely with the build direction, which is the macroscopic direction of maximum heat extraction over many layers. Generally, the grain growth direction and the resulting texture are determined by preferred growth direction and the heat extraction direction. When these two directions coincide or are closely aligned, strong fiber texture is witnessed. However, the scanning strategy can greatly alter the texture, as it affects the heat flow. Columnar grains aligned with the build direction are especially common in PBF. In DED, another widely observed possibility is a zig-zag structure, in which the grain growth direction is tilted in the direction of motion of the heat source (alternated), as shown in Figure 2.3 [62].

Texture can be tuned through process parameters. Columnar fiber texture is formed through epitaxial growth. For grains to grow epitaxially from previous layers, during the addition of each layer, the layer underneath must be remelted (at least partially remelted). Thus, epitaxial growth and consequently columnar fiber texture are favored by higher heat power, lower spot size, reduced layer thickness and shorter hatch spacing. If process conditions do not allow sufficient remelting of previous layers, epitaxial growth is not possible, and fine equiaxed grains are formed through heterogeneous nucleation at partially molten powder particles, at the melt pool boundary. Low heat source power, large hatch spacing and elevated layer thickness, as well as high deposition rate for DED processes, favor this kind of solidification. Moreover, when the previous layer is not sufficiently remelted, porosities with sharp edges known as "lack of fusion defects" may be formed in between layers. When processing Inconel 718, it may be of interest to obtain columnar grains through competitive growth, since this leads to fewer and larger grains, which can be beneficial for creep resistance [62].



Figure 2.3 – Optical micrograph of a cross section of a IN718 sample fabricated by LDED, showing the zigzag solidification texture due to alternate scanning direction. Source: Dinda et al. [62].

To sum up, AM microstructure is highly complex, but it is mostly governed by a few variables, namely the temperature gradient (G), the solidification rate (R) and the undercooling (Δ T). These variables, in turn, are consequence of the process parameters. In order to understand and possibly to control the microstructure of parts produced by AM, several studies have been conducted with the objective of establishing the effect of process parameter variation on the microstructure and mechanical properties of AM parts [42, 60].

Besides grain structure, another key factor in Inconel 718 microstructure is the presence of precipitates. When produced by AM, IN718 does not show the strengthening phases γ' or γ'' in the as built state. Consequently, thermal treatments are necessary in order to optimize the microstructure and properties of the alloy [43]. Solidification occurs in non-equilibrium conditions, and parts produced by AM are subject to high cooling rates and experience several thermal cycles during the process. Particles precipitated in the lower portion of the part are submitted to more thermal cycles, so they can be coarser than those found in the upper portion of the part. The microstructure and consequently the properties of parts produced by AM is heterogeneous – yet another reason why post process heat treatments are necessary [64].

Eutectic Laves, δ (Ni₃Nb) and Nb-rich carbides are frequently seen at grain boundaries or at interdendritic or intercellular regions, in parts produced both by DED and by PBF. Due to the high cooling rates, there is not much time for diffusion, especially for large and heavy atoms such as Nb ad Mo, so these metals can remain segregated into the liquid until the final stages of solidification, when their concentration becomes high enough to form precipitates. Laves phase is especially undesirable, because it is brittle and often formed in continuous morphologies that can make the part more susceptible to hot cracking. Laves morphology is linked to the grain morphology: small equiaxed dendrites (with small interdendritic space, formed at high cooling rates GR and low G/R ratio,) contribute to having discrete, noncontinuous Laves morphology, while large columnar dendrites (with wider interdendritic space, formed at lower cooling rates and higher G/R ratio) favor continuous and coarse Laves particles [63]. The dissolution of Laves and promotion of γ ' or γ " is one more motivation for thermal treating IN718 parts built by AM. Post process heat treatments can also be useful to promote recrystallization, turning the columnar structure into a fine equiaxed one (residual stresses as driving force

2.1.LPBF

2.1.1. As-built microstructure

Some of the earliest, most well known and most cited works on AM of IN718 are those made by Amato et al. in 2012 [43] and by Jia et al. in 2014 [48] (as verified by a simple search on Web o Science [28]). Amato and colleagues fabricated IN718 samples by LPBF, with a laser power of 200 W and scanning speeds of 800 mm/s and 1200 mm/s, using a X-Y scanning strategy. Resulting microstructures were analyzed. As-built samples showed clear melt pool boundaries (**Figure 2.4**), and a microstructure consisting of columnar grains aligned with the build direction [43].

The melt pool boundaries mark the laser tracks on each added layer, and are observed in most SLM parts, as exemplified by other works [58, 67, 68, 69]. The melt pools are a feature resulting from the process, and melt pools are not individual grains. Within the melt pools, there are several grains, and a single grain can span across multiple melt pools, a phenomenon kn own as epitaxial growth [48, 68, 69, 70, 71]. Typical SLM microstructure consists of columnar grains approximately parallel to the build direction [24, 43, 48, 58, 67, 68, 69, 71, 73, 77], as exemplified by Gallmeyer and colleagues [68].



Figure 2.4 – Optical microscopy image showing clear melt pools in an IN718 sample manufactured by LPBF (B indicates the building direction).

Source: Amato et al. [43].

Gallmeyer et al. fabricated IN718 samples by LPBF using a laser power of 160 W, a scanning speed of 800 mms, a spot size of 80 µm, a hatch spacing of 160 µm, a layer thickness of 50 µm and an island scanning strategy with bidirectional scanning within each island and a 90° rotation between each added layer. The powder size was 10-45 µm and the build occurred under Ar atmosphere. Samples were built along the diagonal of a cube. The asbuilt sample shows clear melt pool boundaries, and its microstructure consists of columnar grains growing along the build direction, across multiple layers. Grains grow epitaxially, nucleating at or growing from the unmelted grains at the liquid-solid interface at the melt pool boundary through competitive growth. Some grains have misorientations among them. Average grain length was 96.1 \pm 72.9 μm and average width, 13.4 \pm 9.4 $\mu m.$ This sample has a strong fiber texture with {100} aligned with the build direction, with two other fiber components with {100} oriented in directions transverse to the build direction [68]. Figure 2.5 shows the microstructure of the as-built IN718 sample produced by LPBF by Gallmeyer et al. [68]. In Figure 2.5a, an optical microscopy image shows clear melt pool boundaries, and Figure 2.5b shows an EBSD image (inverse pole figure), evidencing the columnar grains and the texture of the sample.



Figure 2.5 – OM micropgraph with clear melt pool boundaries (a). Inverse pole figure/EBSD showing the columnar grains and texture of the sample (b). Souce: Gallmeyer et al. [68].

The columnar grains are not always strictly parallel to the build direction [67, 69, 71]. Chlebus et al. fabricated IN718 samples by SLM with a laser power of 100 W, a spot size of 180 μ m, a layer thickness of 50 μ m, hatch spacing of 160 μ m, a scanning speed of 85.7 mm/s and an atmosphere of Argon The powder used has a size distribution between 5 μ m and 50 μ m. Each layer was scanned in a zigzag pattern rotated 90° for each new layer. Each layer was rescanned after melting in a perpendicular direction in order to guarantee densification and mitigate residual stresses. A constructed image with optical microscopy images of the xy, xz and yz planes is shown in **Figure 2.6** [71]. It is possible to appreciate the laser tracks on the xy plane. The microstructure is composed of columnar grains growing epitaxially through several layers, aligned close to the build direction (z), but tilted towards the scanning direction, determining the direction of greatest heat extraction [71].

Depending on process parameters and the scanning strategy, the columnar grains can have an even greater misorientation among themselves [67, 69]. Deng et al. produced IN718 samples by LPBF with a laser power of 400 W, a layer thickness of 40 μ m and a standard scanning speed recommended by the system manufacturer (EOS, model M290). The unetched

microstructure of as-built parts showed a columnar structure somewhat oriented along the build direction, but often tilted away from it, due to differences in local heat flow [69].



Figure 2.6 – Constructed image with optical microscopy micrographs from three different perpendicular planes (xy, xz and yz) showing the microstructure of Inconel718 fabricated by LPBF in the as-built condition. The building direction is parallel to the z axis. On the xz and yz planes, it I spossible to see the columnar grains and the layer structure of the sampls. On the xy plane, the laser tracks are in evidence.

Source: Chlebus et al. [71].

In metal parts built by LPBF, the microstructure often contains a subgrain structure within the grains, as a consequence of the high cooling rates. In the study by Deng et al, the grains presented such substructures. In some grains, the subgrain structures were columnar, and in other grains, they seemed more equiaxed, and the authors labeled them a "mosaic structure" at first. The microstructures of IN718 parts produced by LPBF, as observed by Deng et al. through SEM, are reported in Figure 2.7 [69]. Figure 2.7a shows a SEM micrograph of a plane parallel to the build direction. Some grains are columnar and align with the building direction, while other grains are tilted in order to accompany local heat flows, affected by the shape of the melt pool. Figure 2.7b shows the microstructure on a section perpendicular to the build direction. On this plane, the grains seem randomly oriented. Figure 2.7c is a cropped region of Figure 2.7a, seen at a greater magnification, allowing the observation of the subgrain structures. Similarly, Figure 2.7d is a cropped region of Figure 2.7b, and in it, columnar and "mosaic"-like subgrain structures are also seen.



Figure 2.7 – SEM images of an IN718 sample produced by LPBF. BD stands for "build direction". (c) and (d) are parts of (a) and (b), respectively, seen at grater magnification (the cropped regions are indicated in (a) and (b)).

Source: Deng et al. [69].

Upon etching and further examination through SEM, Deng and colleagues concluded that the microstructure was in fact cellular dendritic (**Figure 2.8**) [69]. Cellular dendrites are dendrites that only develop primary arms, with secondary arms being completely suppressed. The aforementioned "columnar" and "mosaic" structures are actually cellular dendrites growing in different directions. Primary arm spacing was found to be around 200-500 nm. Cellular dendrites are not all parallel to the build direction (yellow arrows mark their growth direction). It is possible to observe epitaxial growth of the cellular structures across different melt pools, and within the same melt pool, arrays of cellular dendrites growing in two different directions can appear. Furthermore, it is possible to observe that while many cellular dendrites retain the growth direction across multiple melt pools, some cellular dendrites rotate 90 when advancing through a melt pool boundary [69].

In the fcc γ phase, the preferential growth directions are the <100> family of directions, including [100], [010] and [001]. The cellular dendrites that have one of these crystallographic directions aligned closely with the heat extraction direction will outgrow other dendrites. Eventually, that direction may not be aligned with the heat flow direction anymore. In this case, another of the three preferential directions may be favored. A secondary dendrite arm grows (orthogonal to the primary arm) in the newly favored direction. Since those directions are orthogonal among them, the effect is that dendrites appear to rotate 90° as described before. The preferential crystallographic growth direction does not have to be exactly parallel to the heat flow direction; it just has to be close enough. Moreover, the local heat extraction direction is not necessarily the build direction; it is affected by process

parameters and scanning strategy. For this reason, some AM parts have strong texture and others not [69].



Figure 2.8 – SEM micrographs after etching of the IN718 samples built by LPBF by Deng et al. (a) shows the microstructure on a plane parallel to the building direction (z). It is possible to see melt pools (yellow dashed contours mark the melt pool boundaries) with cellular dendritic subgrain structures in their interior. (b) shows the microstructure on a plane perpendicular to the building direction, with laser scan tracks in evidenced (marked by yellow dashed contours). (c) is a magnified region of (a), and (d) is a magnified region of (b). Cellular dendrites are seen and their growth direction is indicated by yellow arrows.

Source: Deng et al. [69].

Many other authors reported the occurrence of submicron cellular subgrain structures (sometimes referred to as columnar cells or cellular dendrites) within the columnar grains [57, 58, 67, 68, 70, 71, 77] in microstructure of IN718 fabricated by LPBF. Other authors reported a columnar dendritic subgrain structure [48, 58]. In cellular structures, the secondary dendrite arm growth is suppressed due to higher cooling rates. Among the columnar or cellular dendrites, in the interdendritic zone, fine (nanometric) precipitates can be found, most commonly Laves phase. Deng et al. characterized the present phases in the as-built state as the matrix γ (dendrite cores) and fine Laves precipitates in the interdendritic zone, with no signs of the strengthening phases γ' and γ'' [69]. Chlebus and colleagues also found Laves phase, mostly in the eutectic form Laves + γ , in the interdendritic zones, as well as fine MC carbides, rich in Nb and Ti according to EDS analysis. The carbides are spherical, which indicate that they are primary carbides formed during solidification, prior to Laves phase [71]. These phases are formed in the interdendritic regions due to microsegregation of alloying elements during solidification in the AM process. The high cooling rates (10³ to 10⁸ K/s) can prevent macrosegregation, but not microsegregation. In addition to the rapid cooling during selective

melting of a layer, the melting of the next layer also leads to partial remelting of previously solidified layers, which can further enhance segregation [68]. IN718 is especially subject to microsegregation of Nb and Mo, large atoms with low diffusivity [71]. Microsegregation of Nb, Mo and Ti is reported by several authors [58, 67, 68, 69, 71, 77]. In addition to microsegregation and secondary phases, the interdendritic or intercellular zone is characterized by a high density of dislocations, as noticed upon TEM observations by Deng et al. and Gallmeyer et al., due to the high residual stresses inherent to AM rapid heating and cooling (**Figure 2.9**) [69].

Strong residual stresses are formed during cooling, since the solidification of a layer happens when the previous layer is already solidified. These elevated residual stresses produce a high dislocation density and dislocation forests in cellular arrays. Gallmeyer et al conducted a study in which TEM observations allowed the identification of dislocation cells with cross-sections that are $620 \text{ nm} \pm 180 \text{ nm}$ wide, and a lattice dislocation density inside the cells of $1.6 \pm 0.8 \times 10^{14} \text{ m}^{-2}$. Nanoprecipitates (about 4 nm) were seen inside the cells (arrows), but were too small to be identified with SAED. STEM-EDX analysis was performed, and it was found that the intercellular boundaries were enriched in Nb and Ti and slightly depleted in Ni (**Figure 2.10**) [68]. In addition, bright spots indicate the presence of nanoparticles rich in Nb and Ti (MC carbides and Laves phase) and nanoparticles rich in Al and O (Al₂O₃ formed due to O entrapment during building). TEM and SAED analysis provided further evidence that some of those nanoparticles (the Nb-rich ones) are Laves phase. The Laves particles measure 214 nm \pm 62 nm, with an interparticle distance of 316 \pm 103 nm [68].



Figure 2.9 – BF TEM micrograph showing grain boundaries and submicron cellular substructures within the grains of IN718 manufactured by LPBF. Source: Gallmever et al. [68].



Figure 2.10 – (a, b) BF TEM images of IN718 samples produced by LPBF in the as-built state, showing dislocation cells. (c) High-resolution TEM image of nanoprecipitates inside the dislocation cells. (d) HAADF-STEM image and (e–i) corresponding STEM-EDX maps highlighting segregation of alloying elements at cell boundaries.

Source: Gallmeyer et al. [68].

Jiang et al. [77] studied IN718 samples fabricated by LPBF with a laser power of 285 W, a layer thickness of 40 μ m, a scanning speed of 960 mm/s, a hatch spacing of 110 μ m and a rotation of 67 in scanning direction between each layer, and observed different characteristics in different regions of the sample. During the AM process, when a layer cools down, it cannot fully shrink due to the restriction of the underlying layer, so residual stresses are formed, along with dislocations (arranged in cellular structures). The bottom layers are more restricted, due to the substrate. Residual stresses are tensile on the outer regions of the part and compression in the inner zones. In addition, bottom layers suffer more thermal cycles and may have more precipitates. Besides, since it sits closer to the substrate that acts a heat sink, cooling rates are higher in the bottom, so the microstructure is finer, leading to higher hardness. In fact, SEM observations of cell sizes show that coarser cells in the upper parts of the sample are coarser than cells in the lower part of the sample. In addition, it is noticed that within the same melt pool, there are finer cells in its interior, and coarser cells by the pool boundary, due to difference in cooling rate, so there is also anisotropy within each melt pool (**Figure 2.11**) [77].

In the as-built state, IN718 produced by LPBF shows relatively high amounts of Laves precipitates at interdendritic zones and at grain boundaries, as well as MC carbides (mostly NbC and TiC). Laves phase is brittle, and in the as-built state its morphology is irregular, elongated and continuous, providing a preferential site for crack nucleation and propagation and microvoid formation. Its presence is detrimental to tensile properties, toughness, creep and fatigue resistance. Moreover, the formation of Laves phase – (Ni, Cr, Fe)₂(Nb, Mo, Ti) – consumes Nb and Ti atoms that could otherwise be used to form the strengthening precipitates $\gamma^{"}$ – Ni₃Nb – and $\gamma^{'}$ – Ni₃(Al, Ti) –, respectively [24, 69].

In addition, strong Nb microsegregation at interdendritic zones is widely reported, and this leads to inhomogeneous Nb concentration, formation of coarse precipitates and uneven distribution of secondary phases and, consequently, heterogeneous and suboptimal mechanical properties [57, 58, 69]. Besides, microstructure (and thus mechanical properties) are heterogeneous even within the same part, as shown by Jiang et al. [77].



Figure 2.11 – SEM (backscattered electron detector) micrographs of LPBF IN718 in the as-built state, showing the xz plane, parallel to the building direction. (a1) corresponds to a top region of the sample with relatively coarse cells, and (a2) corresponds to a top region of the sample with fine cells. (b1) is a bottom region of the sample with relatively coarse cells, and (b2) is a bottom region with fine cells. In the intercellular zones, signs of microsegregation are seen. The yellow lines mark a zone with coarse cells.

Source: Jiang e t al. [77].

Furthermore, IN718 parts produced by LPBF normally do not contain the strengthening precipitates γ ' and γ " in the as-built state [68], although a few authors have reported γ ' and γ " precipitation in some cases [67]. The microstructure of LPFB manufactured IN718 varies greatly in the literature, mainly as consequence of different process parameters adopted by different authors [48].

Therefore, heat treatments are necessary in order to optimize the microstructure and mechanical properties and reduce segregation [57, 69]. Thermal treatments are a strategy to allow the obtention of more consistent results and eliminate or mitigate disparities and inhomogeneities within the parts. Also, Inconel 718 is a heat treatable alloy that relies mainly in precipitation hardening as its primary strengthening mechanism.

2.1.2. Heat-treated microstructure

As of today, standard heat treatments designed for wrought and cast IN718 are recommended and used for AM IN718. Standard heat treatments for wrought IN718 consist of a solution treatment followed by a double aging [50]. Standard heat treatments for cast IN718 include an additional homogenization treatment prior to solution treatment and double aging [52]. The goal of these treatments is to first homogenize microstructure and chemical composition and dissolve unwanted phases (mainly Laves phase), diffusing Nb, Al and Ti back into the matrix, so that these alloying elements are available to then form strengthening precipitates γ ' and γ '' during aging.

The main idea behind applying the standard heat treatments for AM IN718 is to use already known strategies, in hopes that the solution or homogenization treatment would normalize the AM microstructure and make it similar to microstructures of wrought or cast IN718 parts, so that the aging treatments would then lead to analogous results as for traditionally manufactured Inconel 718. However, parts produced by AM show distinguished microstructural features such as columnar grains with epitaxial growth, subgrain structures, dislocation cells and microsegregation, that were not accounted for when designing standard heat treatments for traditionally manufactured parts that do not exhibit such features. And the result of the heat treatments can be very different when these AM-specific characteristics are present [68].

Most authors use either precisely the heat treatments specified by AMS standards 5662 (standard heat treatment for wrought IN718) or 5383 (standard heat treatment for cast IN718), or variations of those [50, 52]. Custom heat treatments based on AMS 5662 or AMS 5383 may include one or more of the following steps: homogenization treatment (aiming at normalizing the microstructure and homogenizing grain size and morphology and concentrations of alloying elements, eliminating segregation); solution treatment (aimed at solubilizing secondary phases such as Laves and MC carbides, promoting diffusion of important alloying elements back into the matrix); stress-relief annealing (with the goal of relieving residual stresses and possibly promoting recrystallization, eliminating dislocation forests and changing grain size and morphology to equiaxed grains); aging treatments (designed to promote precipitation of strengthening phases γ " and γ) [50, 52].

The heat treatment recommended in AMS 5662 (as per AMS 2774) [50] comprises a solution treatment at 980 °C for 1 hour with water quenching, plus a double aging treatment, first at 720 °C for 8 hours, then furnace cooling at 50 °C per hour to 620°C, then holding for 8 hours at 620°C, finishing with air cooling [51]. The heat treatment recommended in AMS 5383 (as per AMS 2773) is the same heat treatment as AMS 5662 with the addition of a homogenization treatment beforehand, at 1080 °C for 1 hour, followed by water quenching [52, 53]. The heat treatment recommended by ASTM (ASTM B670) is similar: double aging treatment, first at 718°C for 8 hours, then furnace cooling to 621°C, then holding at 621°C until total heat treatment time reaches 18 hours, finishing with air cooling [54]. ASTM 3301 [56] which is AM-specific, recommends a heat treatment for Inconel 718 in the aged condition that is precisely the same as in AMS 5662/AMS 2774.

Deng et al. [69] submitted LPBF IN718 samples to different thermal treatments based on AMS 5662 and 5383, that were combinations of a homogenization treatment at 1080 °C for 1 hour with water quenching, a solution treatment at 980 °C for 1 hour with water quenching, and a double aging treatment, first at 720 °C for 8 hours with furnace cooling at 50 °C /h to 620 °C, then holding at 620 °C for 8 hours and finishing with air cooling. One sample was directly double aged (with no solution or homogenization treatment); one sample was solution treated and then double aged; one sample was homogenized and then double aged; and one

sample went through homogenization then solution treatment, and then double aging. The resulting microstructures are shown in Figure 2.12 [69].



Figure 2.12 –SEM images showing the microstructure of etched IN718 samples produced by LPBF after heat treatments.(a) double aging, (c) solution treatment plus double aging, (e) homogenization plus double aging (g) homogenization plus solubilization plus double aging. (b), (d), (f), (h) are the indicated regions in (a), (c), (e), (g), respectively, seen at greater magnification. BD=building direction.

Source: Deng et al. [69].

The samples directly aged still showed clear melt pool boundaries and retained the cellular dendritic microstructure, as well as the Laves phase (the aging temperature is too low to dissolve it). The samples that went through solution treatment plus aging barely showed

melt pool boundaries and the cellular dendritic microstructure. Moreover, although Laves phase was still present, Laves particles were finer, indicating partial dissolution. In addition, a fair amount of acicular δ precipitates (a few hundred nm wide and 1-2 µm long) appeared, both inside the grains and at grain boundaries. The sample homogenized at 1080 °C and aged showed and even lower fraction of Laves phase (precipitates became finer, being partially dissolved) and showed no δ particles (according to the authors, 1080 °C is above the solvus temperature of δ). The homogenization treatment at 1080 °C dissolves the Laves phase to a greater extent and promotes a greater homogeneity of Nb concentration. For this reason, when the sample is subject to both homogenization and solution treatment prior to aging, Laves phase dissolves during homogenization, but, since Nb is more homogeneously distributed, delta particles form almost exclusively at grain boundaries during solution treatment [69].

Direct aging leads to the precipitation of strengthening phases γ " and γ ', but do not promote dissolution of Laves phase, which consumes Nb atoms necessary to form the strengthening phases, and may lead to brittle behavior and compromised ductility and toughness. It is clear then that a solution treatment is necessary. As shown by several studies, the classic solution treatment at 980 °C for 1 hour is not enough either to dissolved Laves phase [57, 71, 77, 78]. Moreover, solution treatment at this temperature leads to the precipitation of δ , which is not typically seen in wrought IN718 that goes through the same heat treatment. The formation of δ is not necessarily detrimental, but this phase consumes Nb atoms that could be available to form the strengthening phases γ '' and γ ' [68, 69, 77]. Other authors tried different solution treatment strategies. Farber et al. used the same temperature of 980 °C, but a dwelling time of 4 hours, which was not enough either to dissolve Laves phase [67]. Chlebus et al. tried using higher temperatures of 1040 °C and 1100 °C with a dwelling time of 1 hour still, but both temperatures led to recrystallization, with twin boundaries and grain coarsening. The grain coarsening partly defeats the purpose and benefits of using AM in the first place, since the fine microstructure is eliminated, which harms yield strength, ultimate tensile strength and hardness, and possibly toughness and ductility [71]. However, different authors may reach different results even using similar heat treatments, mostly due to different process parameters (laser power, scanning speed, layer thickness, among others).

Gallmeyer et al. [68] reached full Laves phase dissolution with the solution treatment at 980 °C for 1 hour. In spite of this, that temperature promoted the formation of δ (the authors state that this phase precipitates in the 700-1000 °C range). The authors also promoted separate aging treatments at 620 °C for 24 hours and at 720 °C for 24 hours to assess the difference in results. It was found that heat treating at 720 °C leads to higher volume fraction of strengthening phases γ " and γ '. Gallmeyer et al. investigated the effects of various heat treatments. These treatments do not annihilate the dislocation cells, which are beneficial to mechanical properties. According to the authors, the dislocation cells are annihilated at temperatures above 1100 °C, and the solvus temperature of the δ phase is approximately 1010 °C, so a solution treatment should be designed above 1010 °C and below 1100 °C [68].

Based on these results, the authors came up with a novel heat treatment that includes a solution step at 1020 °C for 15 min, water quenching, then aging at 720 °C for 24 hours and air cooling. The sample treated this way showed superior properties when compared to standard wrought heat treated sample. Compared to the standard heat treatment (AMS 5662), the proposed heat treatment differs by a higher solution treatment temperature (above δ solvus to avoid this phase and to ensure dissolution of Laves and homogenization of Nb and Ti segregation); a lower solution treatment time, just enough to dissolve Laves phase and promote diffusion of Nb and Ti, without annihilating the dislocation cells; a single aging step at 720 °C, since aging at 620 °C or double aging showed no improvements, ang aging at 720 °C resulted the desired precipitates [68].

Ferreri et al. [24] conducted a study aimed at quantifying the phases existing in IN718 samples fabricated by LPBF. Samples were produced with a laser power of 285 W, a scanning speed of 960 mm/s, layer thickness of 40 μ m and a scanning strategy including a rotation of 67° in the scanning direction between each layer. LPBF samples were heat treated as per AMS 5663: solution treatment at 954 °C for 1 hour, Ar fan cooling to 120 °C, plus double aging first at 718 °C for 8 hours, furnace cooling at 50 °C/h to 621 °C, holding for 8 hours, then air cooling to room temperature [24]. For comparison purposes, a wrought IN718 was also studied after going through the same heat treatment.

Present phases and their characteristics were determined by neutron diffraction (Table 2.2) and also by SEM area-based image analysis (Table 2.3), reaching similar results [24]. The solutionized and aged LPBF showed γ , γ' , γ'' , δ and MC carbides. The matrix did not show significant texture nor subgrain structures. δ and MC particles were submicrometric in the heat treated LPBF sample and γ' and γ'' were nanometric. The percentage of δ and Laves phase in the LPBF sample is higher than in the wrought material. Conversely, the γ'' content in the LPBF is lower than in the wrought IN718 sample.

Table 2.2 – Phase volume fraction determined by neutron diffraction (%vol.)

	γ	γ'	+/-	γ"	+/-	δ	+/-	МС	+/-
LPBF	bal	6.5	1.4	8.4	0.3	5.9	0.4	1.9	0.5
Wrought	bal	6.5	0.8	17.8	0.5	1.3	0.1	0.9	0.3

Source: I	Ferreri et	al. [24].
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Table 2.3 – Phase fraction (area-based) determined by SEM image analysis (units of area %).

	γ, γ', γ"	δ	MC, Laves
LPBF	balance	6.5 (1.5)	1.8 (0.8)
Wrought	balance	1.0 (0.5)	0.9 (0.6)

Source: Ferreri et al. [24].

2.1.3. Influence of heat treatments on hardness

The heat treatments greatly affect the microstructure and, consequently, mechanical and metallurgical properties of the alloy. Hardness is an indication of mechanical strength that is easier to be measured, since it does not require a test specimen with a large volume or shape, and it hardness assessment is a non-destructive test method. Several authors measured hardness (usually Vickers microhardness) and its variation upon heat treatment. Hardness measurements can also be used as an indicator of success of the thermal treatments, especially the aging treatments, since the strengthening phases γ " and γ ' are nanometric and very difficult to observe through conventional microscopy techniques [24, 49].

Generally, when compared to the as-built condition, solution treatments decrease hardness, due to dissolution of hard Laves phase, and possibly elimination of fine cellular or columnar subgrain structures and associated dislocation forests depending on the temperature and time of the treatment. However, a solution treatment may lead to an increase in hardness due to precipitation of hard δ phase. Homogenization treatments normally lead to a lower hardness – especially if recrystallization takes place – due to the Hall Petch effect, whereby

coarser grains are associated with easier dislocation motion (due to a lower number of grain boundaries, which are an important obstacle to dislocation glide) lower tensile strength. On the other hand, aging treatments increase hardness, as a consequence of the formation of strengthening precipitates γ " and γ '. The combination of a solution treatment with an aging treatment boosts this effect, since the solution treatment dissolves Nb-rich phases and mitigate Nb segregation, providing more Nb available to form the strengthening precipitates [57, 73].

Chlebus et al. [71] assessed microhardness of LPBF IN718 samples that followed different heat treatments. Directly aged samples (double aging first at 720 °C for 8 hours, then furnace cooling to 620 °C at 100 °C/h and holding for 10 hours, then air cooling) showed an increase of 48% in hardness when compared to the as-built state, from 312 HV₁ to 461 HV₁, thanks to the precipitation of strengthening phases during aging, indicating that the matrix was supersaturated in Nb. The columnar structure was maintained. δ needle-like precipitates are seen in interdendritic regions through SEM, as well as Laves phase and carbides (**Figure 2.13**) [71].



Figure 2.13 – Microstructure of IN718 fabricated by LPBF, in a plane parallel to the building direction. (a) OM image showing columnar grains with elongated subgrain structures. (b) SEM image showing cellular dendritic substructures with secondary phases at inderdendritic zones. 1 indicates a δ precipitate, 2 indicates a carbide particle, and 3 indicates the Laves phase.

Source: Chlebus et al. [71].

The sample that was solutionized at 980 °C for 1 hour and double aged, and the sample that was solutionized at 1040 °C for 1 hour and double aged showed similar results. The solution treatments did not prove to be very effective in homogenizing the Nb segregation compared to the as-built condition. The sample solutionized at 980 °C had an increase of 49% in hardness, from 312 HV1 to 465 HV, and the sample solutionized at 1040 °C has an increase of 52%, from 312 HV1 to 474 HV1. The resulting hardness was not much higher than the hardness of the directly aged sample, indicating that the solution treatment did not lead to an amount of strengthening phases much higher. SEM observations showed that these solution treatments did not eliminate the Laves phase, that was still seen at grain and subgrain boundaries and in the interlayer region, confirmed by EDS. In addition, the δ phase was also observed in acicular morphology, as it can precipitate in Nb-rich regions between 750 °C and 1000 °C, so mostly in the solution treatment. The Laves phase is more present in the interlayer regions, because these areas are subject to a higher degree of microsegregation, since they are melted twice. The sample solutionized at 1040 °C showed signs of recrystallization, presenting twin boundaries (**Figure 2.14**) [71].



Figure 2.14 – SEM micrograph (xz plane, parallel to the build direction) of the LPBF IN718 sample after solution treatment at 1040 $^{\circ}$ C for 1 h and water quenching. The microstructure was partially recrystallized, as evidenced by the presence of twin boundaries. Recrystallyzed grains are indicated with R.

Source: Chlebus et al.[71].

The sample solutionized at 1100 °C and double aged was successfully homogenized, as interdendritic regions with segregation are not distinguishable. Carbides are still seen at grain boundaries, with a pinning effect. The hardness increase compared to the as-built condition was 48%, from 312 HV1 to 463 HV1. The solution treatment at 1100 °C was enough to cause recrystallization, apparently, or at least, grain boundary motion with a driving force of reducing grain boundary energy. Carbides seem to pin moving grain boundaries [71].

The author of the present work collected data from multiple studies data from [24, 43, 45, 50, 57, 58, 67-69, 71-77, 86] and reported results in terms of microstructure and hardness as consequence of thermal treatments. This extensive collection of results is presented in Table B.1 in Appendix B. This list provides valuable information in order to create a reference set of results and allow thoughtful comparison and analysis of the results further presented in the work at hand. The table contains many different heat treatment strategies and their effect on microstructure and hardness, for IN718 produced by LPBF and also a few results for wrought and cast IN718 for comparative purposes.

2.2.LDED

2.2.1. As-built microstructure

Since LDED is a more recently developed technology currently with a lower usage in industry when compared to LPBF, there are less studies on LDED in general, and also on LDED of IN718 and especially on the effect of thermal treatments on LDED of IN718. Even so, there are still many interesting results. For the most part, LDED is similar to LPBF (there are more similarities than differences). In LDED-made IN718, the typical microstructure is also characterized by columnar grains growing epitaxially across multiple layers, with melt pool boundaries visible (although less clear than in LPBF) and subgrain structures, usually columnar dendrites. The columnar grains are often not exactly parallel to the build direction, but rather inclined towards the scanning direction, due to heat flow. This is demonstrated by Xiao et al. on their 2017 work [78].

In a 2017 work by Xiao and colleagues, IN718 samples were produced by DED, using a laser power of 600 W, a scanning speed of 6 mm/s, a powder feed rate of 9.05 g/min and a spot size of 0.5 mm. Observations per OM showed that the sample has melt pool boundaries, and a columnar structure (Figure 2.15a). Columnar dendrites grow epitaxially across several layers in the direction of greatest temperature gradient (heat flows to the substrate through previous layers), which is approximately perpendicular to the melt pool boundary, so the dendrites are tilted towards the laser scanning direction (Figure 2.15b). SEM observations show that primary dendrite arm spacing ranges from 8 to 15 μ m (Figure 2.16a). In the interdendritic regions (Figure 2.16), Laves precipitates exist in a typical chain morphology that can lead to poor ductility and toughness, and hot cracking. The Laves precipitates (confirmed by TEM and SAED analysis) were continuous, elongated and relatively coarse, with a volume fraction of 11.6 ± 1.4% [78].



Figure 2.15 – Optical microscopy micrographs of IN718 samples manufactured by LDED. (a) Columnar grains growing across multiple layers. BD=building direction; SD=scanning direction. (b) Columnar dendrites within the grains.

Source: Xiao et al.[78].



Figure 2.16 - (a) Secondary electron SEM image showing the morphology of columnar dendrites of IN718 produced by LDED. (b) Backscattered electron SEM image showing the presence of secondary phases at interdendritic zones.

The epitaxial growth of columnar grains is shown by Wang et al. [79], with IN718 samples fabricated by LDED, with a laser power of 1200 W, a scanning speed of 10 mm/s, a powder feed rate of 20 g/min, a hatch spacing of 0.8 mm. The powder size ranged from 50 μ m to 220 μ m, with a mean diameter of 110 μ m. The grain morphology was assessed by EBSD (**Figure 2.17**). The as-built microstructure comprises mostly coarse columnar grains that grow epitaxially across several layers, aligned with the built direction but tilted towards the direction of laser scanning. Within each grain, it is possible to observe color variation, due to the presence of subgrain structures that have low angle misorientations among them. Grains per se are well over 300 μ m long [79].

Similarly to what is observed for LPBF IN718, in LDED IN718 parts, microsegregation also takes place, due to the high cooling rates, and it plays a key role in defining the microstructure of the alloy. In fact, microsegregation is thought to be the main driver for the formation of subgrain structures in LDED-manufactured IN718. While both LPBF and LDED give rise to subgrain structures, they have different dominant characteristics. In LPBF IN718, dislocation cells originated by residual stresses during the building process are the main factor determining subgrain structures – that are most often cellular. In LDED, the most common subgrain structures are columnar dendrites, associated with Nb segregation to the interdendritic zone. The segregation of Nb to the interdendritic zone, in turn, leads to the formation of Laves precipitates [78-80].



Figure 2.17 – Microstructure of IN718 produced by LDED. EBSD image showing the columnar grains spanning across multiple layers. Source: Wang et al. [79]

Many authors reported microsegregation in LDED of IN718, especially for Nb and Mo atoms, which are large and heavy atoms, with low diffusivity [78-82]. In a 2020 study, Xiao et al. used quasi-continuous wave laser to fabricate DED IN718 samples with a laser power of 4 kW, a scanning speed of 6 mm/s, a spot size of 0.5 mm, powder feed rate of 9.05 g/min and laser frequency 50 Hz [80]. EDS analysis was made in order to assess the concentration of Nb and Mo (Figure 2.18). It was found, as expected, that these elements were concentrated in the

interdendritic areas (and in the Laves phase). Phases identification was confirmed through TEM and SAED analysis [80].



Figure 2.18 – EDS analysis of an IN718 sample fabricated by LDED, showing Nb and Mo segregation at interdendritic zones. Source: Xiao et al. [80]

The main differences between LPBF and LDED, in terms of the resulting microstructure, derive from the fact that LDED uses higher laser power, larger spot size, coarser powders and higher building rates. The cooling rates in LDED processes are still very high, but are lower than those experienced in LPBF. As a consequence, the microstructure of IN718 parts produced by LDED is coarser that the microstructure of IN718 parts produced by LPBF [21, 46, 68, 82].

Normally, in the as-built state, LDED IN718 parts do not show the strengthening phases γ " and γ ', nor δ . Instead, the phases typically present, besides the matrix γ , are Laves phase and MC carbides, as reported by several authors. However, in some cases, due to the lower cooling rates, the γ ", γ ', and δ phases can be formed. Li et al. produced IN 718 samples by LDED with a laser power of 4000 W, a scanning speed of 1600 mm/min, a spot size of 5 mm, a layer thickness of 1.3 mm, with a deposition rate of 2.2 kg/h. The powder size ranged from 45 µm to 90 µm. The authors assessed the microstructure in different regions of the parts [81].

In all regions of the part (bottom, middle and top), the microstructure is composed of columnar grains epitaxially growing across several layers, aligned with the build direction (the maximum heat extraction direction). Grain are 100-500 μ m wide, and melt pool boundaries are clear (formed due to different microstructures, with planar interface growth in the lower region and dendrite growth in the upper region). Laves phase is identified in the interdendritic zone (by SAED and EDS). The concentration of Nb in the Laves phase is around 25%, while the Nb concentration in the matrix is about 2.5%, indicating severe microsegregation of Nb. In addition to interdendritic Laves phase, present in all parts of the sample, submicron δ needle-like particles were identified through SAED, as well as nanometric γ ' and γ ", in the bottom and middle regions, around Laves precipitate [81].

The formation of δ , γ' and γ'' in the lower regions of the part happens due to the fact the these regions experience multiple thermal cycles (more than the upper regions), since they were deposited first and the heat flows through them to the substract, during the course of the building process. For this reason, the lower zones of the part spend more time in the temperature range that allows the precipitation of δ , γ' and γ'' [81].

This effect observed by Li and colleagues attests to the inhomogeneous cooling rates and temperature gradients within the part, which are inherent to the LDED process, and lead to inhomogeneous metallurgical and mechanical properties. Since parts produced by LDED are typically larger than those produced by LPBF, this effect is more evident in LDED [81].

Bearing in mind that in the as-built state, parts produced by LDED show severe microsegregation, Laves phase formation, heterogeneous microstructure, and usually no strengthening precipitates, thermal treatments are required in order to meet specifications and satisfactory part performance [81, 82].

2.2.2. Heat-treated microstructure

As for LPBF, most authors use standard heat treatments designed for wrought or cast IN718 parts (AMS 5662 and AMS 5383, respectively) [45, 78-80, 82, 87]. However, while ASTM F3301 recommends heat treating as per AMS 5662/AMS 2774 for LPBF IN718, there is no official agreed-upon recommendation for LDED IN718, to the best of the author's knowledge. For this reason, different authors use different heat treatments, and investigation on the effects of such treatments are crucial.

Yu et al. [82] fabricated IN718 samples by LDED with a powder of average size of 102 μ m, a laser power of 2.5 kW, a spot size of 5 mm, scanning speed of 10 mm/s, powder feed rate of 25 g/min and a layer height of 0.6-0.8 mm and an alternate XY scanning strategy, with argon as the protective/carrier gas. The samples went through different thermal treatments. One sample was kept at the as-built state. One sample went through direct aging at 720 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling. One sample went through solution treatment at 980 °C for 1 hour, water quenching, aging at 720 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling. One went through homogenization at 1100 °C for 1.5 hours, water quenching, solution treatment at 980 °C for 1 hour, water quenching, aging at 50 °C/h to 620 °C, holding for 8 hours, air cooling. One went through homogenization at 1100 °C for 1.5 hours, water quenching, solution treatment at 980 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling. One went through homogenization at 1100 °C for 1.5 hours, water quenching, solution treatment at 980 °C for 1 hour, water quenching, aging at 50 °C/h to 620 °C, holding for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C holding for 8 hours, air cooling [82].

The samples were investigated through OM (**Figure 2.19**). The as-built sample shows columnar grains growing epitaxially along the build direction, the direction of greatest temperature gradient. Dendrites with the <100> crystallographic direction (preferential growth direction for IN718) aligned closely with the direction of highest temperature gradient outgrow other dendrites [82]. The grains have a width of 20 to 1000 µm. The columnar grains are still seen in the directly aged and in the solution treated and aged samples. The homogeneized sample shows heterogeneous grains predominantly equiaxed, and multiple twin boundaries, indicating recrystallization. Grains range from 10 to 500 µm, and grains in the interlayer overlapped regions are finer than those within melt pools, due to non-uniform residual stresses within the sample [82].

The samples were characterized through SEM (Figure 2.20) [82]. In the as-built sample, primary dendrite arm spacing is about 5-10 μ m, and granular Laves particles are found in the interdendritic regions, as well as NbC. No γ' or γ'' are seen, maybe due to their very fine size or their sluggish precipitation kinetics (not favorable in the high cooling rates of LDED). In the directly aged sample, Laves precipitates were still seen in the interdendritic regions, as well as NbC, and nanometric (10-50 nm) γ' and γ'' were observed at Nb-rich zones (surrounding Laves particles).



Figure 2.19 – OM micrographs of LDED IN718: (a) as-built, (b) direct aging, (c) solution treatment plus aging, (d) homogenization plus solution treatment plus aging. Source:Yu et al. [82]

According to Qi et al., the concentration of Nb necessary to allow the formation of Laves, δ , γ' and γ'' is, respectively, 10%, 6-8%, 4% and less than 4% [83]. The Nb concentration in the dendrite core region (point 2) measured by EPMA (electron probe microanalysis, Table 2.4) is lower than the required to form any of these phases, and the diffusion of Nb is not fast, since it is a large atom. The solutionized and aged sample still presents Laves phase and strong Nb segregation, indicating that the solution treatment temperature is not high enough. In this sample, γ' and γ'' are present due to the aging treatment, but also δ is observed, in acicular morphology with 1-3 μ m long, around Laves particles. The δ phase is formed because Laves phase was partially dissolved and Nb atoms diffused to the surroundings, increasing the concentration to a point where δ formation is favorable [81, 82].

The δ phase has a preferential crystallographic orientation with regards to the Laves phase (and the matrix γ). For this reason δ needles appear to be oriented and parallel. As to the Laves phase, it has a melting and precipitation temperature that ranges from 650 °C to 1100 °C, and this is the reason why the precipitates are segmented. In the homogeneized sample, Laves is completely dissolved, and γ ' and γ " particles of about 30 nm are found dispersed in the matrix, and δ needles formed at grain boundaries and a few within the grains, due to the γ " $\rightarrow \delta$ transformation during aging. The volume fraction of γ ' and γ " is higher than in the solutionized sample [82].


Figure 2.20 - SEM images of LDED IN718: (a) As-built sample; (b) Zone indicated with a yellow rectangle in (a) seen at a greater magnification; (c) directly aged sample; (d) Nb-rich region in the directly aged sample; (e) solution-treated and aged sample; (f) region indicated with a yellow rectangle in (e) seen at a greater magnification, showing the presence of γ " and γ ' precipitates; (g, h) homogenized, solubilized and aged sample; (i) zone indicated with a yellow rectangle in (h) seen with a greater magnification, showin the existence of γ " and γ ' precipitates. Red dots indicate the points analyzed by EPMA.

Source: Yu et al. [82].

Position	Al	Nb	Ti	Mo	Cr	Fe	Ni
1	0.25	11.45	1.01	3.21	16.94	15.96	51.18
2	0.19	1.24	1.07	1.92	22.09	20.71	52.78
3	0.41	4.30	0.90	2.66	19.09	18.72	53.92
4	0.45	5.06	0.95	3.19	19.13	17.25	53.97
δ	0.23	20.33	2.39	4.04	5.04	5.70	62.27
Laves	0.12	23.29	2.70	4.71	15.80	9.37	44.01

Table 2.4 – EPMA results (wt. %).

Note: EPMA test positions are indicated in Figure 2.20.

Source: Yu et al. [82].

Since the solution treatment at 980 °C did not completely dissolve the Laves phase, other authors attempted solution treatments at higher temperatures. Zhang et al. [84] fabricated IN718 samples by LDED with powder size ranging from 53 μ m to 150 μ m, a laser power of 900 W, scanning speed of 360 mm/min, a powder feed rate of 15 L/h with argon as the carrier/shield gas, layer thickness of 0.64 mm, and a unidirectional scanning strategy. Some of the samples were heat treated with different manners. Some samples went through solution treatment at 1100 °C for 1 hours, followed by a double aging treatment, first at 720 °C for 8

hours, then furnace cooling to 620 °C in 0.5 hour, then holding at 620 °C for 8 hours, and finally air cooling to room temperature. Other sample was directly aged, with no solution treatment. Other samples were left in the as-built state [84].

Samples were investigated through OM, SEM, EDS and XRD. The as-built sample shows a typical DED microstructure, consisting of columnar grains with a subgrain structure consisting of columnar dendrites epitaxially growing across several deposited layers, aligned close to the build direction, as seen in the OM image on Figure 2.21a. The directly aged sample displays a similar microstructure, since the temperature is not high enough to promote recrystallization (Figure 2.21b). The solutionized and aged sample, on the other hand, shows equiaxed grains, result of recrystallization (Figure 2.21c) [84].

A SEM analysis showed that the as-built sample (**Figure 2.21**d) is rich in irregular Laves phase (confirmed by EDS, rich in Nb and Mo), especially at interdendritic zones. The as-built sample also displayed titanium-niobium carbonitrides (MX) with fine globular morphology [84].



Figure 2.21 – (a) OM image of the as-built LDED IN718 sample. (b) OM image of the directly aged sample; (c) OM image of the solution-treated and aged sample. (d) SEM image of the as-built sample; (e) SEM image of the directly aged sample; (f) SEM image of the solution-treated and aged sample.

Source: Zhang et al. [84]

The directly aged sample (**Figure 2.21**e) showed columnar dendrites as well as irregular Laves phase in interdendritic regions. In addition, it contained very fine γ ' and γ " particles (seen in the insert, as bright dots surrounding Laves phase). The heat treatment temperature is not high enough to dissolve Laves, but it allows the diffusion of Nb atoms (concentration gradient from the Laves phase to the matrix), and thus the formation of γ ' and γ " [84].

The solutionized and aged sample suffered recrystallization, so the grains are equiaxed and apparently, coarser (Figure 2.21f). The Laves phase was mostly dissolved, and needle-like δ particles precipitated at grain boundaries [84].

XRD analysis confirmed that the Laves phase was successfully dissolved in the solutionized sample. The δ phase was not detected, meaning its volume fraction is very low

compared to γ and γ " in the solutionized sample. γ " peaks overlap those of γ , because both phases have the same crystal structure. Volume fraction of the phases were determined based on XRD analysis (Table 2.5) [84].

Sample	γ"	γ / γ '	Laves		
As-deposited	0	95.9	4.1		
DA-treated	11.3	84.4	4.3		
1100STA-treated	18.6	81.4	0		

 Table 2.5 – Volume fractions (vol. %) of each phase in as-deposited and heat-treated Inconel 718 alloy specimens.

Source: Zhang et al. [84].

Although the solution treatment at 1100 °C executed by Zhang achieved total Laves solubilization, the high temperatures induced recrystallization and grain growth, which is detrimental to mechanical properties [84]. Other authors tried solution treatments at intermediate temperatures and for shorter periods.

Sui et al. [85] fabricated IN718 samples by DED with a powder size of 45-90 µm, a laser power of 2000 W, a scanning speed of 10 mm/s, a spot size of 2 mm, a layer thickness of 0.2 mm, a powder feeding rate of 5.7 g/min and an overlap of 50%. Different thermal treatments were conducted in order to produce different morphologies of Laves phase. One sample was directly aged first at 720 °C for 8 hours, then furnace cooled at 50 °C /h to 620 °C, holding for 8 hours and finishing with air cooling. Other sample was solutionized at 1050 °C for 15 minutes, water quenched, then aged at 720 °C for 8 hours and furnace cooled at 50 °C/h to 620 °C and holding for 8 hours then air cooling. Another sample was solutionized at 1050 °C for 45 minutes, then double aged at 720 °C for 8 hours, and furnace cooled to 620 °C at 50 °C /h and held for 8 more hours, finishing with air cooling [85].

All samples show columnar epitaxial grains oriented along the build direction due to the heat flow direction during deposition. Columnar grains are 50-300 μ m wide, melt pool boundaries (layer bands) are visible, and no twin boundaries are seen, indicating that recrystallization did not occur. The melt layer bands are visible because the the interface of the melt pool, grain growth happens with a planar structure, that later on becomes dendrites [85].

The Laves phase is formed during solidification, at interdendritic zones due to Nb and Mo segregation. All samples still displayed Laves precipitates. The directly aged sample had long strips of continuous and irregular Laves phase. The sample solutionized for 15 minutes showed Laves precipitates with a granular morphology and smaller sizes. The sample solutionized for 45 minutes showed granular Laves precipitates still, but with even finer sizes and lower volume fraction [85].

The dimensions and fractions of Laves precipitates were quantified by image analysis through the software Image Pro Plus. In the directly aged sample, Laves precipitates are coarse, long and continuous, with a volume fraction of 3.83% and approximately 25% of particles being longer than 3 μ m (area weighted). In the sample solution treated at 1050 °C for 15 min before aging, Laves precipitates were finer and granular rather than elongated. 68% of particles (area weighted) are smaller than 0.8 μ m (84% smaller than 1 μ m), and the volume fraction of Laves dropped to 1.55%. Solution treatment for 45 min produced similar

results to the sample solutionized for 15 min, except Laves precipitates were finer overall (78% smaller than 0.8 μ m / 93% smaller than 1 μ m) and had a lower volume fraction of only 0.82% [85].

Electron Probe Micro Analysis (EPMA) was used to quantify chemical concentration of alloying elements in the samples. It was shown that microsegregation of Nb occurred at interdendritic zones in all samples, but especially in the directly aged one. While the nominal composition of the powder alloy was 5.3% Nb, the interdendritic zone of the as-built sample boasted 12.6% Nb, and γ " precipitation tends to occur at this region. In the solution treated and aged samples, Nb concentration was much more uniform – except for Laves precipitates – leading to a uniform distribution of fine γ " (Ni₃Nb) precipitates. The Ti distribution is not homogeneous in the directly aged sample, but both solutionized samples show uniform Ti and Al concentrations, leading to uniformly dispersed γ ' (Ni₃(Al, Ti) of about 10-40 nm) [85].

Volume fraction of strengthening phases was calculated (**Table 2.6Table 2.6** – Calculated volume fractions of γ " and γ ' phases in heat treated LDED IN718 samples.) based on EPMA results [85]. While the fraction of γ " increased with the solution treatment, the volume fraction of γ ' remains practically unchanged, which is a sign that the presence of Laves phase influences mostly the γ " phase. The γ " phase has an ellipsoidal disc-like morphology. The diameter/length of the disc was measured.

	γ" phase(%)	γ'phase(%)	γ '-Ni ₃ Al phase (%)	γ '-Ni ₃ Ti phase (%)
DA sample	13.9	5.13	3.64	1.49
S-15 sample	16.2	5.31	3.65	1.66
S-45 sample	16.9	5.37	3.65	1.72

Table 2.6 – Calculated volume fractions of γ " and γ ' phases in heat treated LDED IN718 samples. DA=directlyaged; S-15=solution-treated for 15min; S-45=solution-treated for 45min.

Source: Sui et al. [85]

The solutionized samples showed smaller diameters for γ " phase than the directly aged sample. The average size of γ " precipitates in the directly aged sample was 64.6 nm. The mean diameter of γ " particles in the solutionized samples was similar: 33.8 nm for the sample held for 15 min, and 34.8 nm for the sample held for 45 min [85].

Laves phase formation consumes Nb, Mo, Ti and Al, and Nb is crucial to γ " (Ni₃Nb) formation, and Ti and Al are essential to γ ' (Ni₃(Al, Ti)) formation. So the formation of Laves diminishes the amount of Nb, Al and Ti available to form γ " and γ '. This way, Laves phase formation affects the volume fraction of γ " and γ ', the main strengthening phases. In addition, microsegregation of Nb occurs around Laves particles, in the interdendritic zones (especially in the directly aged sample, with no solution treatment, the formation of γ " is more likely in those areas, near Laves phase (especially in the non-solutioned sample). This way, Laves phase formation affects the distribution of γ ". The γ ' appears to be evenly dispersed, based on EPMA results for Al and Ti. Moreover, the existance of microsegregation of Nb in certain areas makes the Nb concentration higher in those regions, which in turn leads to coarser γ " precipitates. This is why γ " particles are larger in the directly aged sample. In the solutionized samples, Nb concentration is more uniform. The sizes of γ ' precipitates should be approximately the same on all samples, since Al and Ti are somewhat homogeneously

distributed. The Nb microsegregation is shown to be detrimental to yield strength improvement [85].

2.2.3. Influence of heat treatments on hardness

Hardness can be another indication of phases present and microstructural features that is easily assessed. In general, aged IN718 samples show higher hardness than as-built samples, due to the precipitation of strengthening phases γ " and γ '. These phases are usually nanometric, and difficult to observe through traditional microscopy methods, so hardness increments can be a reasonable indicator of their presence. Solution treatments alone normally reduce hardness, since they solubilize hard phases such as Laves precipitates. However, when coupled with aging treatments in sequence, the resulting hardness can be higher than in directly aged samples, because the solution treatment beforehand provides more Nb atoms available to form the strengthening precipitates during the aging treatment. Solution and homogenization treatments can benefit or hurt hardness, depending on the temperature. If the temperature is too high, recrystallization and grain growth can take place, which can partly eliminate the effect of the originally fine microstructure of AM parts [68, 69, 77, 82, 83].

Liu et al. [86] measured Vickers hardness (1 kgf) of IN718 samples produced by LDED. Hardness values varies from 233 HV₁ to 257 HV₁, with an average of 241.6 \pm 12.1 HV₁. The hardness of the samples did not show a clear trend, but it was lower than the hardness of wrought IN718 (362 HV₁), due to strong segregation and lack of hardening phases γ' and γ'' in the as-built state. In order to optimize hardness and the microstructure, thermal treatments are necessary [86].

Li et al. noticed strongly inhomogeneous hardness in IN718 parts produced by LDED, with different values in different regions of the part. The authors measured Vickers hardness (0.5 kgf) on planes perpendicular to the build direction, in the bottom region (at a height of 6 mm), middle region (at a height of 37 mm) and top region (at a height of 69 mm). The mean hardness for the bottom, middle and top regions was 385 HV, 381 HV and 298 HV, respectively. This difference in hardness is a consequence of the different thermal history and cooling rates in different regions of the part. The lower regions experience higher cooling rates during deposition, and they suffer more thermal cycles. These regions presented γ ' and γ " in the bottom and middle regions. This inhomogeneity of microstructure and properties and is a further reason why thermal treatments are required for DED IN718 [81].

LDED produces coarser microstructures than LPBF, so the hardness of parts fabricated by LDED is expected to be lower than that of LPBF-produced parts. In fact, Jinoop et at. demonstrated this to be true. Jinoop and colleagues obtained a hardness of 234 HV (measured with a load of 0.2 kgf) for an IN718 sample manufactured by LDED, and a hardness of 319 HV (measured with a load of 0.2 kgf) for an IN718 sample fabricated by LPBF, both in the as-built state. After solution treatments, the difference was maintained. After solution treatment at 950 °C for 1 hour, and water quenching, the LDED sample had a hardness of 220 HV (0.2 kgf), and the LPBF sample had a hardness of 307 HV (0.2 kgf). After solution treatment at 1050 °C for 1 hours, the hardness was 206 HV (0.2 kgf) for the LDED sample and 260 HV (0.2 kgf) for the LPBF sample [45].

Careri et al. showed an increase in hardness from 260 HV (measured with a load of 0.025 kgf) to 428 HV after direct double aging treatment at 718 °C for 8 hours then furnace cooling to 621 °C and holding for 8 more hours, finishing with air cooling. Careri and colleagues also tried a homogenization treatment at 1093 °C for 2 hours, air cooling to room temperature, then a solution treatment at 968 °C for 1 hour, air cooling to room temperature, then the

double aging treatment at 718 °C for 8 hours then furnace cooling to 621 °C and holding for 8 more hours, finishing with air cooling. This sample had a microhardness of 430 HV, not much higher than the hardness of the directly aged sample. There is a trade-off effect between grain growth and γ " precipitation, both induced by high temperature exposure. Although the homogenization treatment ultimately makes more Nb available to form the strengthening precipitates, it also causes grain growth and possibly recrystallization, which partially offset the increase in hardness due to γ " precipitation [88].

Xiao et al. [80] executed the AMS 5662 standard thermal treatment and verified an increase in hardness from 263.3 HV (measured with a load of 0.5 kgf) in the as-built condition to 413.6 HV in the solution treated and double aged condition.

The author of the present work collected data from multiple studies [45, 50, 69, 75, 76, 78-80, 82, 84-93] and reported results in terms of microstructure and hardness as consequence of thermal treatments. This extensive collection of results is presented in Table C.1 in Appendix C. This list provides valuable information in order to create a reference set of results and allow thoughtful comparison and analysis of the results further presented in the work at hand. The table contains many different heat treatment strategies and their effect on microstructure and hardness, for IN718 produced by LDED and also a few results for wrought and cast IN718 for comparative purposes.

2.3.AM vs. casting

The traditional methods of manufacturing Inconel 718 components are most commonly casting and forging. Mechanical processing, to the likes of forging, result in superior mechanical properties (yield strength, ultimate tensile strength, ductility, toughness, hardness), but it limits the attainable shapes of the part, since machining of IN718 is not easy, as this alloy has a high hardness, high yield strength at high temperatures, and low thermal conductivity, causing tool overheating and excessive wear, and often resulting high surface roughness. Casting presents itself as an alternative that allows the obtention of more complex shapes and more intricate details, such as internal channels needed for aerospace turbine parts. However, this greater flexibility in terms of shape complexity comes at the expense of mechanical properties. Additive manufacturing is a novel alternative that allows the obtention of even more complex and intricate shapes, with the benefit of enhanced mechanical properties, depending on process parameters and heat treatments [40, 46, 58, 76].

Among the plethora of existing casting techniques, the most commonly used method for IN718 is investment casting (also known as "lost wax casting"), which is a precision casting process that allows the fabrication of complex shapes, including hollow parts, with satisfactory level of detail and surface roughness [40]. In short, the investment casting process includes producing first a wax pattern, assembling multiple patterns in a tree, coating the pattern tree in ceramic slurry, drying, melting the wax and pouring it out, in such a way that only the ceramic shell remains, and then pouring the molten metal into the ceramic shell, allowing it to solidify, then essentially breaking the ceramic shell, obtaining thus the metallic part in the same shape as the initial wax pattern. Figure 2.22 illustrates the investment casting process and its steps [94]. As this is the most used method for the production of IN718 parts, this thesis approaches cast IN718 rather than wrought IN718 in greater detail.

Nevertheless, since casting and forging are traditional and well known process routes that have been around longer, there are many studies on cast and wrought Inconel 718, with widespread knowledge, and even industrial standards and regulations (AMS 5662 for wrought IN718 and AMS 5383 for cast IN718). For this reason, this thesis at hand focuses less on cast

IN718 and more in additively manufactured IN718, as AM is yet to be fully understood and explained, using cast IN718 more as a control, in order to compare additively manufactured IN718 to. As noted by Zhang et al. [75], the usual microstructure in LPBF and LDED IN718 is comprised of columnar grains, while wrought IN718 has equiaxed grains. The Laves phase is coarser in cast IN718, medium in AM IN718 and virtually absent in wrought IN718.



Figure 2.22 – Schematic illustration of the investment casting process. Source: Kalpakjian [94].

Up to date, there are few works establishing a direct comparison among AM and cast Inconel 718, and those that do it tend to focus more on LPBF, naturally, as it is the most popular AM process [81, 85].

Zhang et al. [75] fabricated IN718 samples through LPBF, with a powder ranging from 5 to 45 μ m, a spot size of 100 μ m, a laser power of 190 W, a scanning speed of 1200 mm/s, an overlapping ratio of 30%, and a substrate preheating to 80 °C. Additionally, the study also contemplated investment cast IN718 parts for comparison purposes.

The authors observed the microstructure through optical microscopy. As observed in **Figure 2.23**, the cast sample shows an isotropic microstructure, with similar features in the transverse (a) and longitudinal (b) plane [75]. The microstructure consists of coarse dendrites with well developed secondary arms (average secondary dendrite arm spacing of 45 μ m). The LPBF sample, on the other hand, displays anisotropic microstructural features. The melt pools are clearly visible in the longitudinal plane (c), and the transverse plane (d) shows laser scan tracks. Within the melt pools, there are very fine columnar dendrites, with primary arms only (primary dendrite arm spacing of 698 nm), growing across multiple layers [75].

Vieille and colleagues [70] also undertook a comparative study of cast and additively manufactured (by LPBF) Inconel 718 parts. In their work, cast samples were produced by vacuum induction melting (VIM) and subsequent vacuum arc remelting (VAR). AM samples were fabricated by LPBF with a laser power of 400 W, a scanning speed of 1330 mm/s, hatch spacing of 0.1 mm and a rotation of 67° in the laser scanning direction between each layer. Porosity in the AM sample was defined to be 0.27% by density measurements. The cast sample had an equiaxed microstructure with an average grain size of 35 μ m, while the AM

sample had a cellular dendritic microstructure with grains elongated in the build direction growing across multiple layers (epitaxial growth), and an average grain size of 21 μ m (on a plane parallel to the build direction).



Figure 2.23 – OM images of IN718 sample produced by casting on a transverse (a) and vertical (b) cross section, and IN718 sample produced by LPBF on a cross section parallel to the building (vertical) direction (c) and on a cross section perpendicular to the building direction (c).

Source: Zhang et al. [75].

In another study, Cao et al. [49] confirms by observations via TEM and SAED that the γ matrix in LPBF IN718 displays a cellular subgrain structure with submicrometric cell diameter. According to the authors, such microstructure is 10 times finer than wrought or cast conventional microstructures, which leads to greater strength. Moreover, nanometric γ ' and γ '', precipitated after aging thermal treatments, are thought to restrain grain boundary motion, contributing to maintaining the fine cellular structure even after heat treatment.

Zhang et al. [75] also undertook EBSD analysis to assess grain morphology and size (**Figure 2.24**). In the investment cast sample, grains are equiaxed and non-directional, with an average grain size of 1300 μ m and analogous microstructural features in both transverse (a) and longitudinal planes (b). the LPBF sample, on the other hand, shows a highly anisotropic microstructure, with columnar grains in the longitudinal cross-section (c) growing parallel to the build direction (vertical), with an average grain width of 45 μ m and an average grain length of 255 μ m. In the transverse cross-section, grains seem equiaxed (what is seen is the cross section of columnar grains), with an average size of 52 μ m. The difference in grain size between cast and LPBF samples is explained by the higher cooling rates inherent to the LPBF process. Using finite element analysis through the software COMSOL Multiphysics, the authors calculated a cooling rate of 1.4 x 10⁶ K/s for the LPBF process, and a cooling rate of 3.75 K/s for the investment casting process.

Studies comparing LDED and casting are much less numerous. LDED can be used to repair metallic parts that have been damaged, as it does not have the limitation of a vacuum or building chamber. Soffel et al. [76] used LDED to deposit Inconel 718 on top of an investment cast Inconel 718 part. The deposition was executed with a powder that ranged from 44 μ m to 105 μ m, with a powder feed rate of 4.1 g/min, a laser spot size of 3 mm, and Argon as the shielding, protection and carrier gas. The scanning strategy involved a contour scanning at 200 mm/min, and raster scanning at 333 mm/min, with a laser power of 1000 W for most of the process, and 750 W for the upper layers. Prior to the deposition, the cast substrate was solution treated at 1100 °C for 2 hours.



Figure 2.24 –EBSD image of the cast sample on transverse (a) and vertical (b) cross sections, and the LPBF sample on a plane parallel to the building direction (c) and perpendicular to the building direction (d). Source:*Zhang et al.* [75]

The cast microstructure consists of coarse dendrites with primary dendrite arm spacing of about 100-130 μ m, as shown in OM images depicted in **Figure 2.25**. The microstructure of the LDED deposition is much finer, with columnar dendrites with inhibited secondary arms. The LDED microstructure is especially fine near the interface with the cast substrate, due to higher cooling rates in this region, due to the proximity to the cooler substrate. In fact, hardness measurements give an average value of 218 HV (measured with a load of 3 kgf) at the cast region, while the LDED deposit shows an average value of 265 HV at the bottom

region (close to the cast substrate) and 245 HV in the upper regions (2-4 mm above the interface) [76].



Figure 2.25 – (a) Photograph of the deposited part onto the cast substrate. (b) OM image of the DED microstructure. (c) OM image of the interface between the cast substrate and the deposited region. (d) Hardness profile, with indentations indicated on (e).

Source: Soffel et al.[76]

The authors also analyzed the parts through EBSD (Figure 2.26). It is confirmed yet again that the LDED deposit has mostly parallel columnar grains, with an average grain width of 43 μ m, while the cast part has coarser equiaxed grains, with an average grain size of 1584 μ m. The LDED deposit has a porosity level between 0.01% and 0.04%, with a typical pore diameter of 20-30 μ m [76].

Zhao et al. [58] conducted a comparative study on LPBF IN718 versus suction cast IN718. The AM sample was fabricated with a scanning speed of 960 mm/s, a laser power of 285 W and a hatch spacing of 0.11 mm (for each added layer, the scanning direction was rotated by 67°). Samples obtained by AM and suction casting were homogenized at 1180 °C for 20 min, 1 hours or 12 hours. All heat treated samples were quenched in ice-water. An asfabricated sample was kept from both processes.



Figure 2.26 – EBSD analysis of the deposited region (a) and the cast substrate (b), showing grain structure and average grain size.
Source: Soffel et al.[76]

Samples were analyzed at the SEM with a back-scattered electron detector (Figure 2.27) [58]. The as-cast sample showed an equiaxed microstructure, with grain size of about 10 μ m and Laves precipitates along grain boundaries. A few NbC particles are visible as well, embedded in Laves phase. The as-built AM sample (observed in a plane parallel to the building direction), due to the high thermal gradient, showcased a columnar microstructure, with columnar grains growing in directions normal to the melt pool boundaries. Within the grains, there are subgrain structures, especially in the overlapped regions between two consecutive layers. In some cases, subgrains were columnar with a width of 1 μ m, and in other cases, cellular, with a width of 2 μ m. The only secondary phase observed was Laves phase, present along grain and subgrain boundaries, due to Nb microsegregation in these areas during the build. AM parts had a lower fraction of Laves, due to the better Nb homogeneity, a consequence of the fast solidification speed. This is an advantage, since a smaller fraction of Laves means more available Nb to form the strengthening precipitates γ' and γ'' . Identified phases were confirmed through XRD. The cast sample had a volume fraction of Laves phase of 13.13% \pm 1.3%, while the LPBF sample had a volume fraction of 6.52% \pm 0.7%.



Figure 2.27 – SEM images of the suction cast IN718 sample (as-cast, non-treated condition). At a lower magnification (a), the equiaxed grains are seen. At a higher magnification (b), the secondary phases (Laves and carbides) become evident.

Source: Zhao et al. [58]

Given the high volume fraction of Laves phase in the as-cast condition, thermal treatments are necessary in order to dissolve these brittle, coarse, irregular and elongated precipitates [58].

Zhao and colleagues [58] used ThermoCalc to establish the phase diagram of the alloy (**Figure 2.28**), in equilibrium (a) and non-equilibrium conditions (b). It can be seen from the diagrams that NbC and δ are stable even above 1000 °C. Even though the Scheil-Gulliver simulation indicates the formation of σ , this phase is not commonly seen, especially in the asbuilt condition.



Figure 2.28 – Inconel 718 phase diagrams calculated on ThermoCalc. (a) Equilibrium phase diagram; (b) Nonequilibrium phase diagram (suspending the δ phase); (c) Non-equilibrium solidification route calculated by Scheil-Gulliver simulation.

Source: Zhao et al [58].

Zhang et al. [75] submitted investment cast samples through homogenization at 1080 °C for 1.5 hours, air cooling to room temperature, then solution treatment at 980 °C for 1 hour, air cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling at 55 °C /h to 620 °C, holding for 8 more hours and air cooling to room temperature. The same heat treatment was done in the LPBF sample as well. Upon SEM observations (**Figure 2.29**), it was seen that the heat treatment was not sufficient to dissolve the Laves phase in the cast sample. The Laves phase still existed in irregular, coarse and continuous morphology at the interdendritic zone (a). Smaller NbC and δ precipitates were also spotted,

as well as porosities (b). In the LPBF, however, the heat treatment was enough to fully dissolve Laves precipitates. Moreover, the heat treatment cause recrystallization and precipitation of δ platelets (c). Nevertheless, the microstructure of the LPBF sample was still finer than that of the cast sample. The authors measured the hardness (Rockwell C) of the samples, and obtained 28.0 HRC (approximately 280 HV) for the heat treated cast sample and 42.5 HRC (approximately 410 HV) for the LPBF heat treated sample. These values are also compared to the AMS 5662 standard value for wrought IN718, 34 HRC (326 HV).



Figure 2.29 – SEM images showing both the cast and the LPBF samples after the full homogenizationsolubilization-aging treatment. (a) Coarse Laves precipitates lie at interdendritic zones of the cast sample. (b) Porosities in the cast sample. (c) Microstructure of the sample produced by LPBF, which suffered recrystallization, as the subgrain structures were annihilated.

Source: Zhang et al. [75].

Chlebus et al. [71] reached a different conclusion, and stated that the temperature required to homogenize composition and microstructure in LPBF samples was higher than the traditional temperature for cast and wrought IN718 parts. Many other authors support this finding.

Zhao et al. [58] conducted solution treatments at 1180 °C for 20 minutes, 1 hour and 12 hours in LPBF and suction cast IN718 samples. The volume fraction of Laves and NbC was quantified through XRD (**Table 2.7**). The increase in treatment time led to greater dissolution of Laves precipitates in the cast sample, accompanied by intense grain growth. In the LPBF sample, the solution treatment for 20 minutes was enough to fully dissolve Laves phase. Increasing the treatment time from there led to precipitation.

 Table 2.7 – Phase volume fraction for NbC and Laves phase after homogenization at 1180 °C, for different times.

Homogenization time	0	20 min	lh	12h
Suction casting	13.13% ± 1.3%	$0.73\% \pm 0.07\%$	$0.65\% \pm 0.07\%$	$0.47\% \pm 0.05\%$
Additive manufacturing	$6.52\%\pm0.7\%$	~0	$0.05\% \pm 0.005\%$	$0.36\% \pm 0.04\%$

Source: Elaborated by the author based on results by Zhao et al. [58]

Many authors assessed tensile properties of AM IN718 and compared with standard cast or wrought values. Vieille et al. [70] found that yield strength, ultimate tensile strength and ductility of LPBF IN718 was comparable to those of a cast sample. Gallmeyer et al. [68] measured the tensile properties of LPBF and wrought IN718 following different thermal treatments based on AMS 5662. The thermal treatments are reported on Table 2.8, and Table 2.9 shows the measured yield strength, ultimate tensile strength and elongation of each sample after heat treatment (as well as the results for the non-treated sample). The variation of each property in comparison to those of the wrought sample and to those of the as-built sample are also reported.

		I B	
Condition	Designation	Solution Anneal	Aging
As-printed	AP	-	-
Solution amnealed	SA980	980 °C/1 h/WQ	-
Standard heat treatment	SHT-1*	980 °C/1 h/AC	720 °C/8 h/FC 50 °C/h+620 °C/8 h/AC
Standard heat treatment	SHT-2	980 °C/1 h/AC	720 °C/8 h/FC 50 °C/h+620 °C/8 h/AC
Direct age: 620 °C, 24h	DA620	-	620 °C/24 h/AC
Direct age: 720 °C, 24h	DA720	-	720 °C/24 h/AC
Solution amnealed + aged	SA1020 + A720	1020 °C/0.25 h/WQ	720 °C/24 h/AC

 Table 2.8 – Heat treatments employed by Gallmeyer et al. FC=furnace cooling; AC=air cooling; WQ=water quenching.

*Tensile specimen attached to buid plate during treatment.

Source: Gallmeyer et al. [68].

Table 2.9 – Measured values of yield strength (σ_{YS}), ultimate tensile strength (σ_{UTS}) and elongations at failure of IN718 samples produced by LPBF and heat treated according to **Table 2.8**. The relative variation (%) to the wrought, standard heat treated ($\Delta^{W, SHT}$) and as-built samples (Δ^{AP}) IN718 are also reported.

Condition	σγs			συτς			Elongation		
Condition	(MPa)	$\Delta^{W, SHT}$	Δ^{AP}	(MPa)	$\Delta^{W, SHT}$	Δ^{AP}	(MPa)	$\Delta^{W, SHT}$	Δ^{AP}
AP	760	-34.5	-	1335	-17.1	-	21.3	+57.8	-
SAP980	620	-46.6	-18.4	1325	-17.7	-0.75	28.6	+112	+34.3
SHT-1	1135	-2.16	+49.3	1530	-4.97	+14.6	10.6	-21.3	-50.2
SHT-2	1240	+6.90	+63.2	1560	-3.11	+16.9	11.6	-14.1	-45.4
DA620	1120	-3.45	+47.4	1500	-6.83	+12.4	14.5	+7.41	-31.9
DA720	1300	+12.1	+71.0	1580	-1.86	+18.4	9.6	-28.9	-54.9
SA1020 + A720	1245	+7.33	+63.8	1640	+1.86	+22.8	16.6	+23.0	-22.1
Wrought SHT	1160	-	+52.6	1610	-	+20.6	13.5	-	-36.6

Source: Gallmeyer et al. [68].

Yu et al. [82] did the same for LDED IN718 samples and found that the precipitation of acicular δ near Laves phase alters the morphology of Laves particles from continuous to granular, which benefits ductility. The tensile properties reported by Yu et al. for IN718 samples produced by LDED following different heat treatments are shown in (**Figure 2.30**) [82]. One sample (labeled "DA") was directly aged (720 °C for 8 h, cooling at 50 °C/h to 620

^oC, holding for 8 hours, air cooling); one sample (labeled "SA") was solution treated (980 °C, 1 h, water quenching) and then double aged (720 °C for 8 h, cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling); one sample (labeled "HSA") was homogenized (1080 °C, 1 h, water quenching) then solution-treated (980 °C, 1 h, water quenching) and then double aged (720 °C for 8 h, cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling). One sample was kept in the as-built state.



Figure 2.30 – Tensile properties of IN718 samples produced by LDED following different thermal treatments. DA=directly aged; SA=solution treatment plus aging; HSA=homogenization plus solution treatment plus aging. Results for wrought and cast IN718 heat treated as per AMS 5662 and AMS 5383, respectively, are also shown. Source: *Yu et al. [82]*.

For Inconel 718, creep resistance may also be of great relevance. Kuo et al., for instance, found that creep life of IN718 fabricated by LPBF is much lower than that of cast and wrought parts (1200 hours) [64]. As built samples withstood 270 hours, samples that went through solution treatment plus aging lasted 100 hours and samples directly aged (with no solution treatment) lasted 550 hours. Creep resistance is greatly affected by size, concentration and morphology of δ particles. In the study by Kuo et al, δ precipitates were acicular in the sample that was solution treated and aged, acting as a nucleation site for cracks and damage accumulation, while directly aged samples had round δ particles, which explains the difference in creep life [64].

On the other hand, Song et. al found that IN718 parts produced by LPBF and submitted to thermal treatment had superior creep life when compared to cast and wrought parts [65]. The improvement can be explained by the fact that heat treated AM parts had higher amounts of γ ' and γ '', which contain Nb, diminishing available Nb atoms for δ formation. Pröbstle et al obtained similar results, and provided additional explanation: according to them, the subgrain structure consisting of 0.50-0.65 µm cells present in the samples made by LPBF also contributed to the enhanced creep resistance [66]. Different authors may reach different results, as the results are influenced by a plethora of factors that are yet to be fully understood (process parameters, heat treatments, feedstock used, to name a few).

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According to Li et al. [81], the heterogeneous microstructure and mechanical properties, and the lack of strengthening precipitates in the as-built state, as well as the relatively low yield strength, ultimate tensile strength and hardness – lower than cast and wrought values – demonstrate the need for thermal treatments. The authors defend that a heat treatment should be specifically developed for each manufacturing process (including DED), since each process produces different starting microstructures. According to Zhu et al. [92], to the moment, few works established a direct comparison between AM and conventional manufacturing methods for Inconel 718, and the ones that did so, used mostly LPBF. Therefore, a study comparing microstructure and mechanical properties of parts produced by DED and casting is still required, as well as the effect of heat treatments on them. This thesis at hand aims to do just that.

3. MATERIALS AND METHODS

In this work, the microstructure of Inconel 718 samples was assessed by optical microscopy (OM) and scanning electron microscopy (SEM). Inconel 718 samples were fabricated by LPBF, LDED and investment casting, and followed different thermal treatments based on AMS 5662 [50]/AMS 2774[51]. Since the strengthening phases γ " and γ ' are nanometric in additively manufactured Inconel 718, and difficult to observe through OM or even SEM, microhardness measurements were undertaken in order to get an indication of their presence and assess the effect of thermal treatments.

3.1.Sample manufacturing

3.1.1. Casting

Four cast IN718 samples were provided by a partner for comparison purpose. The samples were produced by investment casting, and manufacturing parameters are confidential.

3.1.2. LPBF

The four IN718 samples produced by LPBF were also provided by the same partner, and the process parameters are confidential as well. The samples were fabricated using a gas atomized IN718 powder. The chemical composition of the powder was provided by the manufacturer of the samples and is reported on **Table 3.1**. The powder was processed in a LPBF system equipped with an IPG Photonics Ytterbium YLR-200-SM fiber laser with a maximum output power of 200 W, a an Argon inert gas protective system, an automatic powder delivery apparatus and a computer system for process control. Prior to the building, the sealed building chamber was filled with argon, dropping the oxygen concentration below 10 ppm to avoid oxidation of the powders and the consolidated part during processing. The samples were provided already detached from the build platform.

Element	Al	С	Cr	Fe	Mo	Nb	Ti	Ni
Content (wt.%)	0.80	0.08	21.35	20.47	2.80	4.75	1.15	balance

 $Table \ 3.1 - Powder \ composition \ (\% wt.).$

Source: Sample manufacturer.

3.1.3. LDED

Four LDED samples were produced at partner university SUPSI (Scuola Universitaria Professionale della Svizzera Italiana) in Manno, Switzerland, in a Prima Additive Laserdyne 430 system, with a working volume of 585 mm x 400 mm x 500 mm and a 1kW fiber laser (**Figure 3.1**). This machine has a 3-axis CNC system with one 1.5 L powder hooper, and typically operates with a deposition rate of 20 cm³/h, even though it is capable of attaining a deposition rate of 40-50 cm³/h. The process parameters are shown in **Table 3.2**. The samples were built with a laser power of 600 W, a scanning speed of 900 mm/s, a hatch spacing of 0.3

mm, a layer thickness of 0.2 mm, a laser spot size of 1064 nm, and a rotational scanning strategy, in which for each layer deposited, the scanning direction was rotated. The scanning direction rotated 6 times (one for each new layer) before returning to the original direction. The rotation angles were $0^{\circ}/90^{\circ}/45^{\circ}/135^{\circ}/60^{\circ}/120^{\circ}$. The deposition occurred onto an Inconel 718 cast plate that functioned as the substrate. The samples were provided while still attached to the substrate.



Figure 3.1 – Prima Additive Laserdyne 430, the LDED system used in this work. Source: Prima Additive [95].

Table 3.2 - Process parametesr used in this work.

Laser spot size	Laser power	Scanning speed	Hatching distance	Layer thickness	Rotation angle
1064 nm	600 W	900 mm/s	0.3 mm	0.2 mm	0/90/45/135/60/120°

Source: Elaborated by the author.

The powder used for the LDED process was provided by manufacturer LPW Technology, based in Runcorn, United Kingdom, and part of Carpenter Technology Corporation. The powder is gas atomized, and the chemical composition, as informed by the manufacturer, is shown in **Table 3.3**.

 Table 3.3 – Nominal composition of the LDED powder used in this study, as provided by the manufacturer (%wt.).

Element	Al	В	Bi	С	Ca	Со	Cr	Cu	Fe	Mg
Min	0.20						17.00		Bal	
Max	0.80	0.006	0.00003	0.08	0.01	1.00	21.00	0.30	Bal	0.01
Element	Mn	Мо	Nh+Ta	Ni	Р	Ph	S	Se	C;	Ti
			110.14	1.10	1	10	5	Se	Si	11
Min		2.80	4.75	50.00	1	10	5	5c	51	0.65

Source: LPW Technology [96].

3.2. Powder characterization

Both powders (LPBF feedstock and LDED feedstock) were characterized by with the same procedure, following ASTM F3049 guidelines [97]. Powder characterization experiments were undertook by partners of the 4DHybrid group, and provided as courtesy to complement this study. SEM observations (500x) were carried out according to standard ISO 13322-1 in order to determine powder particle morphology and particle size distribution through image analysis. Moreover, the powder particles were embedded in acrylic resin and grinded and polished in order to reveal the cross-section of powder particles. Powder particles' cross-sections were also observed at the OM (at a 500x magnification for the LPBF powder and 250x magnification for the LDED powder). The flowability of the powder was assessed by pouring 50 g of powder (previously weighted with a digital scale) into a Hall flowmeter funnel and measuring the time required for the powder to flow out of the funnel with a manual chronometer, in accordance with ASTM B213 [99]. Three measurements were made, and the average flowability and average mass flow were determined. After the powder flowed through the funnel and formed a pile, the angle of repose was also assessed (average of three measurements). The apparent density was assessed by pouring the metal powder in a Scott volumeter (a container with a known volume of 25 cm³) and measuring the powder mass, following ASTM B329 [100] (average of three measurements). Afterwards, the tap density was measured also in a 25 cm³ container according to ASTM B527 [101] (average of three measurements).

3.3. Heat treatments

For each manufacturing process (LPBF, LDED and casting), 4 samples were produced, for a total of 12 samples. For each process, one sample was kept in the as-produced condition, while the other three samples were heat treated. Each of the three samples was submitted to a different heat treatment, but all heat treatments were based on the AMS 5662 [50] / AMS 2774 [51] standards for wrought Inconel 718 parts for aerospace applications. The standard heat treatment comprises a solution treatment at 980 °C for 1 hour, water quenching, then a double ageing treatment, first at 720 °C for 8 hours, then furnace cooling to 620 °C and holding for 8 more hours, finishing with air cooling (Figure 3.2). One sample from each manufacturing technique was heat treated with the full standard heat treatment. Another sample (from each manufacturing technique) was only solution treated at 980 °C for 1 hour, and water quenched. Yet another sample (from each manufacturing technique) was the at treatments were carried out at the Laboratory of Heat Treatments, at the Alessandria campus of the Politecnico di Torino, using a Nabertherm RHTH 120-600/16 tubular furnace.



Figure 3.2 – AMS 5662 standard heat treatment for Inconel 718 (schematic). RT=room temperature. Source: Elaborated by the author (data from AMS 5662/AMS 2774 [50,51]).

3.4. Cutting

After heat treatment, the LDED and cast samples were cut approximately in half, in order to obtain solids with every dimension under 25 mm, so that they could be mounted in resin (the dimensions are limited by the size of the chamber of the mounting press) prior to polishing and metallographic observations. LDED and cast samples were cut on a Remet TR 100s machine equipped with a water cooled SiC blade (**Figure 3.3**). The LDED samples were not separated from the substrate, so that the substrate could also be investigated by metallography. LPBF samples were already within desired dimensions.



Figure 3.3 - Remet TR 100s cutting machine used in this study. Source: Remet [102].

After cutting, the samples were cleaned with detergent and water and dried with compressed air. Two samples used in this study are shown in **Figure 3.4**. A LPBF sample is reported in (a) and a cast sample in (b).



Figure 3.4 – IN718 samples used in this study.(a) LPBF sample. The X "mark" indicates the top surface (furthest from the platform) of the LPBF sample, in order to allow identification of the building direction. (b) Cast sample.

Source: Photographs taken by the author.

3.5.Mounting

Afterwards, the cut samples were mounted into transparent polymeric cylinders in order to facilitate handling, polishing, microscopy observations and microhardness assessments. This was done by embedding the metallic samples in acrylic resin, using the Remet IPA 30 mounting press (**Figure 3.5**a and b), following ASTM E3 guidelines [102]. The mounting press has a cylindric chamber (with a diameter of 30 mm) in which the metallic sample is placed. The chamber is then evenly filled with acrylic resin powder until the powder completely covers the metallic sample. Then a piston is lowered into the chamber, sealing it hermetically and compressing the polymeric powder. Once the chamber is sealed, it is heated by an electric resistance up to 170 °C, to allow the polymerization of the resin. After 15 minutes, the chamber is water cooled to room temperature and the sample can be extracted, already embedded in an acrylic cylinder. The samples were mounted in such a way that the exposed plane of the sample (the one that would be polished and observed through microscopy) is a plane parallel to the building direction, so that melt pools could be observed (**Figure 3.5**c and d).



Figure 3.5 – (a) Remet IPA 30 mounting press. (b) Acrylic resin used for mounting the samples. (c) Mounted LPBF samples. (d) Mounted LDED sample.
Source: Photographs taken by the author.

3.6.Polishing

After mounting in acrylic resin, the samples were grinded and polished using a Presi Minitech 233 polishing machine of the rotary disc type, equipped with a 250 W motor (**Figure 3.6**). The rotation speed was set at 240 rpm, and the samples were grinded with SiC grinding papers of #FEPA 240, 400, 800, 1200, 2400, 4000, in sequence. For each grinding paper, the samples were handheld, one at a time, in a fixed position. For each new grinding paper, the sample was rotated by 90° to make the grinding and polishing as homogeneous as possible. The goal of the grinding process is to make the surface as planar and smooth as possible, before actual polishing. During grinding, running water was constantly fed onto the grinding paper, acting both as lubricant and coolant. Grinding with a certain paper was complete when all the abrasion lines are in the same direction. With the next grinding paper, as the sample was rotated by 90°, by the end of the process the abrasion lines should be all rotated by 90° as well.

After grinding, the samples were effectively polished, using polishing pads and diamond paste of 3 μ m, and 1 μ m subsequently, following ASTM E3-11 [103]. The goal of the polishing process is to obtain mirror-like surfaces, apt for microscopy observations (**Figure**

3.7). During polishing the same 90°-rotation strategy was used but, instead of water, these polishing pads require the use of a proper alcohol-based lubricant, which was applied manually at 30 s intervals without interrupting the rotation of the pad. Every time the grinding paper or polishing pad was changed, the sample was cleaned with detergent and very gently rinsed with running water in order to remove debris or abrasive particles eventually attached to the surface of the sample. At the end of the process, samples were analyzed at the optical microscope (Leica DMI 5000) in order to assess whether the polishing was adequate or not. Polishing was considered adequate when abrasive lines were not visible at magnification of 100x. Theoretically, by the end of the process, samples should have a surface roughness Ra of 1 μ m. If the quality of the polishing was not satisfactory, the process was repeated.



Figure 3.6 – Polishing machine Presi Minitech 233. Source:Presi [104].



Figure 3.7 – (a) LDED sample after grinding. (b) LPBF sample after polishing. Source: Photographs taken by the author.

3.7. Etching

Prior to microscopy observations, all samples were etched with Kalling's #2 etchant in order to unveil microstructural features. This etchant was prepared with 10 g of CuCl₂, 200 ml

of HCl_(aq) (37% vol.) and 200 ml of ethanol (100% vol.). Prior to etching, the samples were first gently cleaned with detergent and rinsed with running water, then dried with compressed air. The etchant was poured in a small becker, and each sample was submerged in the Kalling's #2 solution for 20 seconds. Immediately after etching, each sample was individually submerged in another becker containing distilled water. After this the samples were gently cleaned with detergent, rinsed with running water and dried with compressed air. Additionally, samples were cleaned with ethanol (100% vol.) prior to observations on the microscope. After this process, samples were checked on the microscope to assess if etching was successful. In some cases, the etching time was not sufficient to reveal the microstructure. In these cases, the sample was re-etched for 10 seconds. In other cases, etching was too intense, and the samples showed signs of corrosion. In such cases, the sample was re-polished with polishing pads of 3 μ m and 1 μ m and re-etched.

3.8. Optical Microscopy

After polishing and etching, the samples were observed in a Leica DMI 5000 optical microscope (Figure 3.8), with magnifications of 100x, 200x and, in some cases, 500x. All samples were observed.



Figure 3.8 - Leica DMI 5000 optical microscope. Source: Leica [106].

3.9.SEM

The samples in the as-produced state, the soluzionized samples, and the samples that went through the full AMS 5662 heat treatment (solution plus double ageing) were also investigated with Scanning Electron Microscopy (SEM), using a Thermo Fischer Scientific Phenom XL SEM equipped with BSD (backscattered electron detector), SED (secondary electron detector) and EDS (energy dispersive spectroscopy) detectors (**Figure 3.9**). SEM observations were made at the BOOST laboratory at the Politecnico di Torino, main campus.



Figure 3.9 - Thermo Fischer Scientific Phenom XL SEM. Source: Thermo Fischer Scientific [107].

3.10. Microhardness

Lastly, the microhardness of the samples was assessed, using a Leica VMHT microhardness tester (Figure 3.10), with a load of 500 gf and an indentation time of 15 s, within ASTM E384 specifications [105]. All samples were tested, and for each sample, 5 indentations were made, and the average of the five values was considered as the microhardness result. Before registering the five measurements, a first indentation was carried out in order to guarantee that the sample was well accommodated and mechanically stable, so that it would not move during microhardness measurements. Microhardness tests were performed at the Laboratory of Mechanical Characterization at the Alessandria campus of the Politecnico di Torino.



Figure 3.10 - Leica VMHT microhardness tester. Source: Images made by the author;

4. RESULTS AND DISCUSSION

4.1.Powder Characterization

4.1.1. LPBF

The powder was characterized by SEM (500x) and was found to have a spherical morphology (**Figure 4.1**), as expected, since gas atomized powders normally have a spherical morphology as a consequence of the atomizing process: with gas atomization, the cooling rates are lower in comparison to water atomization, so there is more time for the molten metal droplets to shape into the more aerodynamic spherical form during the descent from the atomizer nozzle to the collection chamber [21, 109].



Figure 4.1- SEM images showing the morphology of the powder used for LPBF. Source: Obtained by the author with help from the 4DHybrid group partners.

The SEM images were analyzed by software Image J in order to determine the particle size distribution. The particle size ranges from 5 μ m to 55 μ m. The data acquired was plotted in a histogram, together with the cumulative size distribution. From the cumulative curve, it is possible to identify a median particle size d₅₀ of 17.5 μ m. The particle size distribution is shown in Figure 4.2. The powder particle sizes are comparable to those used in other studies on LPBF IN718 [42, 48].



Figure 4.2 – Particle size distribution, as determined by image analysis from SEM observations. Source: Elaborated by the author with help from the 4DHybrid group partners.

The metallographic analysis of the cross section of powder particles (**Figure 4.3**) shows that they already contain small and round pores, with diameters on the order of 1 μ m. These pores are likely gas porosities originated during the gas atomization process, by entrapped inert gas (usually argon). Pores on the feedstock can lead to porosity in the final processed part, and can potentially hurt mechanical properties [21, 42].



Figure 4.3 – OM image of the cross section of powder particles from the LPBF powder. Source: Elaborated by the author with help from the 4DHybrid group partners.

The apparent density of the powders was measured to be 4.211 ± 0.023 g/cm³, and the tap density resulted 5.035 ± 0.004 g/cm³. These numbers represent 51.42% and 61.48% of the real bulk density of Inconel 718, 8.19 g/cm³ [109]. According to Cordova et al. [110], the layer density in LPBF (before selective melting by the laser) is in the range of 40% to 60% of the material's bulk density, which is in agreement with the results aforementioned, suggesting that the behavior of the powder could possibly relate reasonably well with the behavior of the powder layers during the LPBF process. With this in mind, the Hausner Ratio (ratio between tap density and apparent density) can be used to get an indication of the powder flowability [111, 112]. A Hausner Ratio below 1.25 indicates good flowability, while a value above 1.40 suggests poor flowability [112]. For this IN718 powder, the Hausner Ratio is 1.196 \pm 0.007, indicating a good flowability, which is beneficial for the AM process.

However, Spierings et al. [108] found that this flowability indication based on the Hausner Ratio may not correlate with *de facto* flowability. For this reason, the flowability of the powder was measured with a Hall funnel. The average flowability was 15.50 ± 0.34 s, with an average mass flow of 3.23 ± 0.07 g/s. The angle of repose of the powder pile was $26.00^{\circ} \pm 0.12^{\circ}$. According to Spierings, for Ni-based alloys, an angle of repose below 54° indicates a good (acceptable) flowability [Spierings], and according to Gruber et al., an angle of repose lower than 30° attests to an excellent flowability [Gruber]. In this case, the LPBF IN718 powder has an excellent flowability. This result is in good agreement with those obtained by Gruber ($27.63^{\circ} \pm 0.63^{\circ}$) [109].

The good flowability is further confirmed by visual assessment of the powder pile (as suggested by Spierings [108]), since it shows little agglomeration. In this case, the flowability indicated by the Hausner Ratio correlated well with the repose angle result. **Figure 4.4** shows photographs of the powder pile.



Figure 4.4 – Powder pile of the LPBF powder. (a) Top view. (b) Side view. Source: Obtained by the author with help from the 4DHybrid group partners.

4.1.2. LDED

For the LDED IN718 powder, SEM observations (500x) showed that the powder particles are also spherical, as expected, since the powder was also obtained by gas atomization. Although particles are predominantly spherical, a few particles have irregular or elongated morphologies, and many have satellite particles (Figure 4.5). The LDED powder seems coarser that the powder used for LPBF, which is expected and in agreement with literature. Larger particles are commonly associated with higher surface roughness, satellite particles and irregularities deriving from the atomization process. These defects may lead to a lower

powder flowability, which in turn may cause lack of fusion defects in the built part, excessively high surface roughness or lower accuracy [21, 45, 47, 93].



Figure 4.5- SEM image of LDED powder particles showing their morphology. Source: Obtained by the author with help from the 4DHybrid group partners.

The particle size distribution obtained through image analysis is shown in **Figure 4.6**. The powder particle diameter ranges from 30 μ m to 110 μ m, with a d₅₀ of 57.5 μ m. This confirms that the LDED feedstock is coarser than the LPBF, as pointed in literature [21, 45, 81, 93]. The coarser powder contributes to the higher deposition rates observed in LDED (when compared to LPBF build rates), allowing the fabrication of larger parts in shorter times, at the expense of accuracy and surface quality [81, 93]. The larger particles may also lead to a lower flowability. As for the LPBF powder, the particle size distribution of the LDED feedstock is unimodal and asymmetric, with d₅₀ being closer to the lower end of the size range.



Figure 4.6 – Powder particle size distribution, obtained through SEM image ananlysis. Source: Elaborated by the author with help from the 4DHybrid group partners.

The metallographic observation of powder particle cross sections (**Figure 4.7**) shows that the LDED powder also contained porosities from the atomization process. Pores are round and relatively small, in the order of 1 μ m, likely caused by entrapped gas during the atomization process. There are a few pores with larger size (around 5 μ m) than those observed in the LPBF powder.



Figure 4.7 – OM observations of powder particle cross-section (LDED powder). Source: Obtained by the author with help from the 4DHybrid group partners.

The apparent density was assessed at 4.182 ± 0.000 g/cm³ and the tap density resulted 4.984 ± 0.030 g/cm³. These values are 51.06% and 60.85% of the bulk density of IN718. These numbers are very similar to those obtained for the LPBF powder, and are still in the 40%-60% range. The calculated Hausner Ratio is 1.192 ± 0.007 . Accounting for the error, this result is statistically equal to that of the LPBF powder, and suggests a good flowability (as it is lower than 1.25) [109].

In order to confirm this, the flowability of the powder was assessed using a Hall funnel, and the average flowability was 16.06 ± 0.23 s, with an average mass flow of 3.11 ± 0.05 g/s. The lower mass flow rate and the consequently higher flow time indicate a poorer flowability in comparison to the LPBF powder [108]. This is further confirmed by the angle of repose, which was $30.74^{\circ} \pm 0.46^{\circ}$, and thus higher than that of the LPBF powder ($26.00^{\circ} \pm 0.12^{\circ}$). The angle of repose was right on the line between excellent and acceptable flowability (the lower the angle, the better the flowability [108]), but still within the acceptable range nonetheless.

Visual observation of the powder pile (**Figure 4.8**) indicates very good flowability (seemingly better than the LPBF powder's), as there are no agglomerations [108]. This shows that the many methods for flowability assessment do not always converge to the same conclusion, as pointed out by Spierings et al. [108] and Gruber et al. [109]. The Hall funnel experiment (both flow time and rate and resulting angle of repose) can be influenced by the operator through the filling procedure and the ability with the manual chronometer. The visual assessment is inaccurate and depends heavily on the experience of the researcher. Nevertheless, in short, both powders show good flowability, as indicated by the different approaches explored.



Figure 4.8 - Visual observation of the powder pile for the LDED powder. Source: Obtained by the author with help from the 4DHybrid group partners.

4.2.Cast

4.2.1. As-cast

The as-cast sample was investigated through optical microscopy (OM), first with a 100x magnification (Figure 4.9a), and then with a 200x magnification (Figure 4.9b). The microstructure of the as-cast sample is characterized by coarse equiaxed grains with large, well developed γ dendrites. At the interdendritic zone, coarse, irregular and often continuous precipitates are seen. Based on their morphology and on the thermodynamics of this alloy, such precipitates are predominantly Laves phase. As shown by Zhao et al. [58], the Laves phase forms during solidification of the alloy, accompanied by the δ phase and MC-type

carbides. It is expected in fact, that some of the precipitates are NbC or TiC carbides. Moreover, at 200x magnification, it is possible to observe two different kinds of Laves precipitates. At the bottom right part of the image, the irregular precipitate is the eutectic Laves phase (with a Swiss-cheese-like appearance, due to the eutectic transformation Liquid \rightarrow Laves + γ , originating small γ islets amidst the Laves precipitate). At the upper left region of the image, the irregular precipitates with a clear interior are primary Laves phase, as pointed by Han et al [113]. In addition, δ needle-like precipitates are seen surrounding Laves precipitates. The formation of δ particles is favored around Laves phase due to the higher concentration of Nb in those regions, consequence of segregation during solidification. The δ particles have a preferential crystallographic orientation with respect to the γ matrix, and for this reason they are often parallel to each other [85, 87].



Figure 4.9 – OM images of the cast sample in the as-cast (non-treated) state. (a) 100x magnification. (b) 200x magnification. Source: Obtained by the author.

The sample was also investigated through SEM and EDS analysis. A SEM micrograph with a 5000x magnification is shown in Figure 4.10, with indications of the EDS points analyzed. At this magnification it is easier to see the eutectic Laves morphology, as well as the δ precipitates and their preferential crystallographic orientations. Moreover, it is possible to see a blocky carbide. The identities of the phases were confirmed by the EDS analyses (Table 4.1). The blocky precipitate is a Nb-rich carbide, as shown by EDS analysis (point 1). The carbide is roughly 10 µm wide and also shows a high concentration of Ti (4.60%, versus 0.65-1.15% on the nominal composition). Carbon was not included in the analysis. The Laves phase (EDS point 2) is rich in Nb and Mo (20.43% and 6.13%, respectively), and the EDS point 2 has a relatively high concentration of oxygen, which is likely due to oxidation during the investment casting process. The δ phase (Ni₃Nb) is rich in Nb, but the EDS point analysis is not accurate enough to pinpoint exactly the location of the δ particle, and its resolution also includes the surrounding and underlying matrix, so the composition indicated by point 3 is not exclusively from the δ phase. Nevertheless, the Nb concentration is higher than the nominal concentration (7.11% versus 4.75-5.50%, respectively), and so is the Mo concentration (5.23% measured, versus 2.80-3.30% nominal). This attests to the severe segregation that takes place during solidification, especially for large and heavy atoms such as Nb and Mo, which have limited diffusivity.



Figure 4.10- SEM image of the cast sample (magnification of 5000x), with EDS points indicated by 1, 2 and 3, and indications of the secondary phases present.

Source: Obtained by the author

Table 4.1 – EDS results (%wt.)

EDS Point	Nb	Ni	Ti	Cr	Fe	Мо	Si	Al	0	Zr	Mn	Suspected Phase
1	71.42	12.08	4.60	4.14	3.94	3.13	0.50	0.19				Nb/Ti-rich carbide
2	20.43	43.95	1.16	10.91	10.85	6.13	0.79		4.13	1.65		Laves
3	7.11	50.95	0.99	17.47	16.89	5.23	0.70	0.62			0.04	δ

Source: Elaborated by the author.

The measured microhardness of this sample was $205 \pm 13 \text{ HV}_{0.5}$, comparable to that obtained by Soffel et al. (218 HV₃) [76].

4.2.2. Solution treatment

The microstructure of the solution-treated sample (980 °C, 1h, water quenching) was observed through OM and is reported in **Figure 4.11**, with a magnification of 100x (a) and 200x (b). The microstructure still comprises coarse equiaxed grains with austenite dendrites, and even though the sample went through a solution treatment, intermetallic precipitates Laves and δ are still widely seen at interdendritic zones. This suggests strong segregation at interdendritic zones, as seen in the as-cast condition, and it shows that the solution treatment at 980 °C for 1 hour was not enough to successfully dissolve intermetallic precipitates. This result is in accordance with what was reported by Zhang et al. [75].



Figure 4.11 – OM images of the cast sample after solution treatment at 980 °C for 1 hour. (a) 100x. (b) 200x. Source: Obtained by the author.

The sample was further investigated by SEM and EDS analysis. The SEM micrographs are shown in Figure 4.12a (5000x magnification) and Figure 4.12b (7000x magnification). Table 4.2 reports the chemical composition as assessed by EDS on the points indicated in Figure 4.12a. Carbon was not included in the EDS analysis. A line scan (EDS analysis along a straight line) was performed on the region indicated in Figure 4.12b. The results are reported in Figure 4.13.

The SEM micrographs allow clearer observation of the irregular Laves precipitates (primary Laves on Figure 4.12a and eutectic Laves on Figure 4.12b), as well as δ needle-like particles surrounding Laves precipitates and a blocky carbide (on Figure 4.12a). In IN718, carbides often have straight or planar faces and sharp edges [24], due to crystallographic preferential planes for precipitation with respect to the matrix γ , as seen in Figure 4.12a.



Figure 4.12 – SEM images of the cast sample after solution treatment (980 °C, 1 h). (a) Magnification of 5000x. Acicular δ precipitates are seen, as well as a blocky carbide and irregular Laves precipitates. The cross-marks identified with numbers indicate the points analyzed by EDS. (b) Magnification of 7000x, with a line EDS scan indicated by the dashed horizontal line. Acicular δ phase and eutectic Laves phase are seen.

The identity of the phases, at first hypothesized based on their morphologies, is confirmed by the EDS analysis. For the carbide (EDS point 1), it is clear that the phase is rich in Nb and Ti. Although carbon was not assessed, the low amounts of Ni, Fe, Cr and Mo indicate that this phase is not Laves ((Ni, Cr, Fe)₂(Nb, Mo, Ti)) nor δ (Ni₃Nb). EDS point 2 lies on a δ needle-like precipitate, but the slender morphology makes it difficult to place the electron beam impinging point exactly on the δ particle, so the EDS results for this point are not precise, and include surrounding γ phase. Nevertheless, the Nb concentration (6.17%wt) is above the nominal range (4.75-5.50%wt). Similarly, the EDS point 3 lies on a Laves precipitate, but it appears to include surrounding matrix (in this case, underneath the precipitate). Nonetheless, the Mo concentration (4.15%wt) is higher than the upper limit of the nominal composition of the alloy (3.3%wt).

EDS Point	Nb Ni	Ti	Cr	Fe	Мо	Si	Al	Br	Suspected Phase
1	71.85 11.03	6.71	3.34	3.33	2.97	0.56	0.21		Nb/Ti-rich carbide
2	6.17 53.69	9 0.92	16.99	17.41	4.21	0.60			δ
3	5.73 52.55	5 0.88	17.16	17.60	4.15	0.57		1.36	Laves
3	5.73 52.55	5 0.88	17.16	17.60	4.15	0.57		1.36	Laves

Table 4.2 – EDS analysis executed on the points indicated in Figure 4.12a (%wt.).

Source: Elaborated by the author.

In order to show more assertively the identity of the δ (Ni₃Nb) phase, a line scan was performed (**Figure 4.13**). The results show that when the line scan intersects the δ particles, the Nb concentration rises greatly, accompanied by an increase in Ni concentration, and a corresponding decrease in Cr, Fe and Mo concentrations. Ti appears to follow a trend similar to Nb, indicating that there Ti also suffers segregation.

For the cast samples, these results were expected. The inability to fully solubilize the precipitates formed during solidification in the cast sample was not surprising, since the employed heat treatment was developed for wrought Inconel 718, and mechanically processed Inconel 718 tends to have a finer microstructure, with smaller precipitates that were broken up and segmented by the mechanical processing. Finer precipitates are easier to dissolve, due to facilitated diffusion kinetics when compared to coarser precipitates, which demand more time to solubilize. As the precipitates in cast Inconel 718 are coarser than those of a mechanically processed part, it is only natural that the cast sample demands either a higher temperature or a higher time in order to successfully solubilize its precipitates. In fact, the standard heat treatment for cast IN718 is not the same as that for wrought IN718, as it includes a homogenization step at 1080 °C for 1 hour prior to the solution treatment [52]. To the purposes of this work, the standard heat treatment for wrought IN718 (AMS 5662) is assumed to be effective for wrought IN718 parts, as widely demonstrated in literature and industry alike. With this experiment, the heat treatment is tested on a material with a different starting microstructure - in this case, a coarser microstructure -, and it is shown that the effects of the heat treatment are different too.

The hardness of the sample was measured at 226 ± 24 HV_{0.5}, higher than the hardness of the as-cast sample. This provides further evidence that the solution treatment was not very effective, as it did not promote solubilization of the hard intermetallic phases. In fact, if anything, it seems that the solution treated cast sample shows a higher fraction of δ phase than the as-cast sample. It is possible that the solution treatment led to some (very mild) solubilization of Laves precipitates, and the newly available Nb atoms were then consumed in

the formation of needle-like δ particles. This hypothesis is backed by results obtained by Zhao et al. [58] and Liu et al. [87], that showed that the δ phase is stable up until about 1020 °C, and can precipitate during solution treatment at 980 °C. The δ phase also seems to have precipitated at grain boundaries, on **Figure 4.12**a. The formation of the hard intermetallic δ phase could explain the slight increase in hardness.



Figure 4.13 – Results of the EDS linear scan (indicated in Figure 4.12b). The Nb content (%wt.) increases in correspondence with the δ particles. Source: Obtained by the author.

4.2.3. Solution treatment plus single aging

The micrographs (OM) of the sample that went through solution treatment (980 °C, 1 hour, water quenching) plus single aging (720 °C, 8 hours, air cooling) are shown in Figure **4.14**, with a 100x magnification (a) and 200x magnification (b). The microstructure is very similar to the solution treated microstructure and also to the as-cast microstructure. It is still formed by coarse equiaxed grains with equiaxed dendrites, and there are still signs of segregation at interdendritic zones. The Laves and δ precipitates are still present, as expected, since the aim of this heat treatment is not solubilization and, indeed, the 720 °C temperature is not high enough to cause their dissolution. Rather, the goal of this thermal treatment is to promote the precipitation of the main strengthening phases, $\gamma'' - Ni_3Nb - and \gamma' - Ni_3(Al, Ti)$. However, since the solution treatment was not successful at dissolving Laves and δ phases, the amount of Nb atoms available to form γ " (the key strengthening phase for IN718) is not as high as it could be, so the volume fraction of γ " is expected to be relatively low. In addition to Nb, the Laves phase may contain Ti, so the volume fraction of γ ' after aging should be nonoptimal as well. Nevertheless, precipitation of γ " and γ ' is expected, since there are still Nb and Ti atoms in the matrix. In the images obtained through OM, though, these precipitates cannot be seen, due to their nanometric size. At this temperature, NbC formation is possible [75, 77] and, indeed, there appears to be a higher number of carbides, identified as the blocky precipitates with straight faces and sharp edges.


Figure 4.14 – OM images of the cast IN718 samples after solution treatment (980 °C, 1h) plus single aging (720 °C, 8h). (a) 100x. (b) 200x.
Source: Obtained by the author

In addition to the previously observed phases, a secondary phase with a very particular morphology is seen in a certain region of the sample observed by OM with a magnification of 100x and 200x (**Figure 4.15**a and **Figure 4.15**b, respectively). Based on the morphology, these precipitates are (Nb, Ti)C carbides, with the so-called "Chinese script" morphology. This phase is associated with severe Nb segregation [114].

Furthermore, it seems that no significant grain growth took place (comparing the optical micrographs of this sample with those of the as-cast and solution-treated samples). In the OM image with 200x magnification, it is possible to observe δ needle-like precipitates marking grain boundaries. The δ phase was reported to have a pinning effect on grain boundaries, avoiding their motion during thermal treatments, which ultimately prevents or hinders grain growth [24, 67, 87].



Figure 4.15 – OM images of the solution-treated-and-aged cast sample, showing precipitates with "Chinese script" morphology. (a) 100x. (b) 200x. Source: Obtained by the author.

The solution-treated and single-aged sample showed a microhardness of $387 \pm 36 \text{ HV}_{0.5}$, which is significantly higher than the values of the two previous samples (as-cast and solutionized). In fact, this number is 89% higher than the average microhardness of the asbuilt sample. The main reason for the increase in hardness is the precipitation of strengthening phases γ " and γ '. Even though the solution treatment was not effective, the amount of Nb in the matrix is still sufficient to form γ " (Qi et al. reports that γ " formation may occur with Nb concentrations of about 4% [83]) in many regions. Although the precipitates cannot be seen through OM, this statement is backed by literature. Several authors reported γ " precipitation even after direct ageing treatments, with no preceding solution treatment [69, 77]. Additionally, the γ ' phase also precipitates at this temperature, contributing to the rise in hardness. Moreover, the increased presence of carbides can also promote a growth in hardness, to some extent [58, 75].

The higher hardness is also a further indication that no significant grain growth happened, attesting to the pinning effect of δ precipitates at grain boundaries. Although the microhardness improved, this sample would probably not have an adequate performance under mechanical stresses, since the high volume fraction of brittle Laves phase with coarse and continuous morphologies would harm ductility and toughness, limiting the part life in service [68]. This highlights the importance of an effective solution treatment, beyond increasing the availability of Nb to form γ ".

4.2.4. Full cycle

The sample that went through the complete heat treatment (i.e. solution treatment at 980 °C for 1 hour, water quenching, ageing at 720 °C for 8 hours, furnace cooling to 620 °C, aging at 620 °C for 8 hours, air cooling) was investigated through OM with a magnification of 100x (**Figure 4.16**a), 200x (**Figure 4.16**b) and 500x (**Figure 4.16**c).



Figure 4.16 – OM images of the cast IN718 sample after the full AMS 5662 heat treatment (solution treatment plus double aging). (a) 100x. (b) 200x. (c) 500x. Source: Obtained by the author.

The microstructure is very similar to the previous cast samples. It consists of coarse equiaxed grains with well developed dendrites, and a great amount of secondary phases, especially at interdendritic zones. The Laves phase is still present, as well the δ phase, and a few carbides. At this temperature (620 °C), both γ " and γ ' may be formed. However, γ ' has a sluggish precipitation kinetics, so γ " formation is favored in this treatment, as reported by Gallmeyer et al. [68]. At a magnification of 500x, it is possible to appreciate carbide particles (small precipitates with straight faces and sharp edges), as well as eutectic Laves phase and needle-like δ particles. At 200x magnification, one can notice what seems to be fine precipitates marking a grain boundary contour on the central bottom region of the image.

Analyzing the microstructure with SEM, at 2000x magnification (Figure 4.17a), it becomes clear that the fine precipitates at grain boundaries are the δ phase, with a length of the order of 1 µm. In addition, it is possible to spot small carbide particles (Figure 4.17b, 5000x magnification), with straight faces and sharp edges, and a dark color. Through EDS analysis (Table 4.3), it was established that such particle is in fact a TiC precipitate. Carbon content was not assessed, but the Ti content of nearly 80% in weight is a strong evidence. Given the Nb content, the particle is likely the MC-type carbide (Ti,Nb)C.



Figure 4.17 – OM images of the cast sample after the full AMS 5662 heat treatment (solution treatment plus double aging). (a) 2000x. Irregular Laves precipitates are seen, and acicular δ particles are present surrounding Laves precipitates and at grain boundaries. (b) 5000x. Acicular δ particles and a blocky carbide are seen. The cross-mark indicates the point of EDS analysis.

Source: Obtained by the author.

	Table 4	.3 – EDS	S analys	sis perfo	ormed or	1 point 1	indicat	ed in F	igure 4.17 (%wt.).
EDS Point	Ti	Nb	Cr	Fe	Si	Мо	Al	Ni	Suspected Phase
1	79.99	14.94	1.95	1.46	0.48	0.97	0.21	0.00	Nb/Ti-rich carbide

Source: Elaborated by the author

The measured microhardness of the fully heat treated cast sample was 369 ± 32 HV_{0.5}. This represents a decrease of about 5% from the solution-treated-plus-single-aged sample. When the error is taken into account, both values can be considered statistically equal. These results show that the second step of the ageing treatment (620 °C, 8 hours) does not contribute to improving the hardness and likely the mechanical strength of the alloy (in the case of a cast sample heat treated according to AMS 5662). This can be explained by the fact that the solution treatment was not effective in solubilizing incoherent Nb-rich phases Laves and δ , so there was a relatively low amount of Nb atoms available to form the strengthening coherent precipitate γ " at the ageing stage. The Nb atoms that were available in the matrix to form γ " were consumed during the first ageing step at 720 °C mostly in the precipitation of γ ", but also possibly in the formation of δ and carbides, to a lesser extent. So, when the sample was exposed to the second aging step at 620 °C, the Nb atoms were already consumed in the precipitation of secondary phases in the first aging step at 720 °C, and there was no sufficient Nb content throughout the matrix for significant further γ " precipitation.

The same is valid for γ' . A great amount of Ti atoms are trapped in the Laves phase and in carbides, that were formed during solidification and were not dissolved in the solution treatment. In the first aging step, at 720 °C, the Ti atoms that were available in the matrix, allowed the precipitation of γ' (alongside Al and Ni) and TiC. At the second aging step, at 620 °C, there was not much Ti left to form more γ' . Moreover, the γ' phase contributes less to strengthening Inconel 718 than γ'' , due to a lower coherency strain, and also simply because the concentration of Ti and Al in the alloy is lower than the concentration of Nb, so, as a result, the volume fraction of γ' is lower than the volume fraction of γ'' . Furthermore, at 620 °C, the formation of γ' and carbides is sluggish. Gallmeyer et al. showed that at this temperature, the precipitation of γ'' is favored (over γ'), but anyway the Nb content does not allow further precipitation of the strengthening phase [68].

Additionally, it is possible that during this treatment, part of the coherent and metastable γ " precipitates (formed during the first aging step) transform into the stable and incoherent variant δ , leading to a decrease in hardness as a consequence [24]. This might explain the slight hardness reduction from the single aged condition. Nevertheless, the resulting average hardness after the complete heat treatment is 80% higher than the original hardness of the ascast sample.

The hardness value obtained is higher than those typically found in literature for heat treated cast IN718. Zhang et al. [75] measured a hardness of 280 HV (28 HRC) for a cast sample that was heat treated according to AMS 5383. The higher hardness measured in the thesis at hand may be explained by the persistence of a great amount of hard intermetallic Laves and δ phases even after the solution treatment. The grain size may also play a role. Zhang et al. reported a very coarse average grain size of 1300 µm, which is likely coarser than what is seen in this work [75].

4.3.LPBF

4.3.1. As-built

The LPBF sample in the as-built state had its microstructure investigated through OM at a magnification of 100x (**Figure 4.18**a) and 200x (**Figure 4.18**b). The plane observed is a plane parallel to the building direction. The building direction is the vertical direction in the images. It is possible to observe clear melt pool boundaries, which mark the laser scan tracks in directions that intersect the plane under observation. It is also possible to see columnar grains that grow predominantly along the building direction (or close to it), as this is the direction of the greatest heat extraction and temperature gradient. The columnar grains grow epitaxially across multiple melt pools (and thus across multiple layers). For instance, on the right side of **Figure 4.18**b, it is possible to spot a 20-30 μ m wide columnar grain that grows across at least 5 melt pools. Within the grains, there are subgrain structures, that at low magnifications give a meshed or striped appearance. At these magnifications, through the optical microscope it is not possible to identify secondary phases. The microstructure is finer and overall very different from the microstructure of the cast sample.



Figure 4.18 – OM images of the IN718 sample produced by LPBF in the as-built (non-treated) state. Arc-shaped melt pools are visible, as well as columnar grains that span across multiple layers. (a) 100x. (b) 200x. BD=building direction. Source: Obtained by the author

Upon SEM investigation, at greater magnifications of 3000x (Figure 4.19a) and 20000x (Figure 4.19b), it is easier to see and identify the grains, which are mostly columnar, and the subgrain structures become clear. Within the grain, there are many elongated substructures aligned in the same direction, parallel to each other. Such substructures are cellular dendrites, and the direction of orientation of these cellular dendrites is uniform inside the same grain, but varies from one grain to another (Figure 4.19a).

With a 20000x magnification, one can appreciate the individual cellular dendrites, and notice the interdendritic regions. In this image, there seem to be at least two different grains. One of the grains is on the upper-left portion of the image, with cellular dendrites aligned along the diagonal direction of the image (from the bottom left corner to upper right corner). The other grain is on the bottom-right portion of the image, with cellular dendrites aligned on a direction perpendicular to the page of this document. There is possibly a third grain on the bottom-right corner of the image, with cellular dendrites aligned on the image, from the bottom right to the upper left. At the interdendritic zones, there appears to be a secondary phase (brighter regions). A point in an interdendritic zone was analyzed by EDS (point 1 on **Figure 4.19**b).





Figure 4.19 – SEM images of the LPBF sample in the as-built state. (a) 3000x. It is possible to observe the elongated grains, and within the grains, cellular dendritic substructures. (b) 20000x. With a greater magnification, it is possible to appreciate the subgrain structures and to notice secondary phases at interdendritic zones. Point 1 indicates the spot of the EDS analysis.

Source: Obtained by the author.

The EDS results, reported in **Table 4.4**, show a high concentration of Nb (9.60%wt.), C (5.60%wt.) and Mo (4.50%wt.), way above the upper limits of these elements in the nominal composition of the alloy (5.5%wt. Nb, 0.08%wt. C and 3.3%wt. Mo). This is evidence of microsegregation in the interdendritic zone, with likely formation of Laves phase and carbides, as reported in literature [67-69, 77]. It is clear that even though the cooling rates are very high in the LPBF process, they are high enough to prevent macrosegregation, but not enough to avoid microsegregation. The strengthening phases γ " and γ ' are not seen, as expected and reported in literature, because they have sluggish precipitation kinetics, and the rapid cooling experienced during the LPBF process does not allow sufficient time for their formation. Rather, Laves phase is widely reported in literature to form in as-built LPBF IN718 parts [24, 69], and this is probably the case in this sample too. Additionally, the high amount of C evidences the presence of carbides as well (likely NbC).

	Table 4.4- EDS resulst for point 1 indicated in Figure 4.19b (%wt.).											
EDS Point	Nb	С	Ni	Cr	Fe	Мо	Ti	Al	Si	Zr	Suspected Phase	
1	9.60	5.60	46.79	15.35	15.14	4.50	1.12	0.50	0.42	0.97	Carbides / Laves / γ	
					_							

Source: Elaborated by the author.

The microhardness of the LPBF as-built sample was $327 \pm 7 \text{ HV}_{0.5}$. This is almost 60% higher than the hardness of the cast sample in the as-cast state. Since strengthening precipitates are not usually formed in as-built LPBF IN718, the main reason for the higher hardness is the finer microstructure of the LPBF sample. The LPBF process produces a finer microstructure than casting because the cooling rates in AM are extremely high, much higher

than the cooling rates typically experienced in casting processes. Zhang et al. found a cooling rate of 3.75 K/s for investment casting of IN718, and a cooling rate of 1.4×10^6 K/s for the LPBF processing of IN718 [75]. The cooling rates in AM are elevated mainly due to the strongly localized heating, which generates a very high temperature gradient as well (in the order of 10^6 K/m [21]), much higher than typical temperature gradients in investment casting (especially with the ceramic shell). The temperature gradient also determines the solidification microstructure morphology. In AM, the very high temperature gradients lead to a cellular microstructure, as seen in **Figure 4.20** [21]. In investment casting, the low temperature gradient leads to an equiaxed dendritic microstructure.



Figure 4.20 – Schematic solidification map. Source: DebRoy et al. [21].

In fact, not only are the grains finer in the LPBF sample, but they also have subgrain structures (cellular dendrites), which makes the microstructure overall much finer and more intricate, with more obstacles for dislocation motion when compared to the cast sample. Indeed, Gallmeyer et al. [68] showed that the substructures formed in LPBF are separated by intercellular regions with a high density of dislocations generated due to the thermal stresses experienced during LPBF, especially in solidification and cooling of each layer. When the current layer cools down, it contracts, but the underlying layer is already cooler and contracted, so compression stresses are originated in the underlying (older and cooler) layer, and tensile stresses are generated in the current (newer and warmer) layer, and dislocations are originated as a consequence.

Therefore, while the cast sample has coarse grains, and consequently less grain boundaries to hinder dislocation motion, the LPBF sample has fine grains and subgrain structures, with lots of grain and subgrain boundaries, in addition to dislocation forests in the intercellular zone that can entangle moving dislocations. Since the LPBF sample has more obstacles for dislocation motion, it is more difficult to deform (including microdeformation), resulting a higher yield strength and higher hardness.

The measured microhardness is compatible to other results reported in literature [45, 67, 69]. For instance, Deng et al. found a hardness of $325 \text{ HV}_{0.3}$ for as-built LPBF IN718

samples, with columnar grains and cellular substructures of 200-500 nm, and the presence of Laves precipitates in intercellular zones [69].

4.3.2. Solution treatment

The LPBF samples were also heat treated with variations of the AMS 5662 standard. One sample went only through the solution treatment, at 980 °C for 1 hour, followed by water quenching. It is known that this treatment is effective for wrought IN718, as it is specifically designed for wrought parts, which have relatively fine precipitates. This work showed that such treatment was not effective for cast IN718, which have coarse precipitates (mainly Laves) that require higher temperatures or longer periods of time to dissolve. As seen in the as-built LPBF sample, the microstructure of AM parts is very fine, much finer than the microstructure of cast and even wrought samples. For this reason, the solution treatment at 980 °C for 1 hour is expected to be successful for AM parts, as their precipitates are even finer than those found in wrought parts.

The solution-treated LPBF sample was investigated through OM at 100x (Figure 4.21a), 200x (Figure 4.21b) and 500x (Figure 4.21c) magnifications.



Figure 4.21 – OM micrographs of the solution-treated (980 °C, 1h) IN718 sample produced by LPBF. (a) 100x.
(b) 200x. (c) 500x. Melt pools are still visible, and so are the columnar grains and their subgrain structures. BD=building direction.

Source: Obtained by the author.

It is still possible to see clear arc-shaped melt pool boundaries, as well as the columnar grains growing epitaxially across several melt pools or layers. At 500x magnification, a melt pool is portrayed at the center of the image, and the reader can appreciate the main microstructural features, including columnar grains, epitaxial growth, and the aforementioned meshed or striped appearance, linked to subgrain structures. The slight differences in the meshed regions aspect is due to the cellular dendrites having different orientations from one grain to the next. If the cellular dendrites are perfectly parallel to the plane of observation, the appearance resembles stripes; if the cellular dendrites are perfectly perpendicular to the plane of observation, the appearance resembles dots. Everything in between may look like bands. The grains appear to have a width in the order of 10-30 μ m, so no grain growth or recrystallization have occurred. Most grains are somewhat aligned with the building direction. However, many grains are actually tilted away from it, often aligned perpendicular to the melt pool boundary, as this is the local maximum heat flow direction.

The sample was also observed through SEM, at 5000x (Figure 4.22a) and 10000x (Figure 4.22b and c) magnification. At 5000x magnification (Figure 4.22a), it is possible to observe the grains, as well as their elongated subgrain structures. It is clear then that the solution treatment did not annihilate the cellular structure. In the area between the center and the bottom right corner of the micrograph, there is a grain that is especially visible, with a width of approximately 10 μ m. With a 10000x magnification (Figure 4.22b), the substructures can be observed more closely, and it becomes evident that the intercellular zone still has a distinct aspect, suggesting that the microsegregation was not eliminated by the solution treatment, nor were the secondary phases dissolved (mainly Laves phase).

EDS analysis (**Table 4.5**) was carried out on the points indicated in **Figure 4.22**b. An additional line scan (**Figure 4.23**) was performed along the line 1 indicated on **Figure 4.22**c. The EDS point 1 and 2 targeted the intercellular zones, and the results suggest strong microsegregation, especially for Nb, Mo and C, which are all well above the upper limit of the nominal composition. The assessed chemical composition, as well as the SEM observations, suggests the presence of Laves phase and carbides, based on the high concentration of Nb and Mo, and on the irregular morphology of the precipitates. The high amount of C in point 1 also suggests the existence of carbides in the area (likely NbC). The line scan results (**Figure 4.23**) show that in fact the Nb concentrations rises dramatically when the electron beam crosses the precipitates. It is clear then that although the precipitates are very fine, the solution treatment was not successful.



Figure 4.22 – SEM images of the IN718 sample produced by LPBF, after solution treatment (980 °C, 1 h). The cellular dendritic subgrain structures can be seen. (a) 5000x. Elongated grains can be identified, with subgrain structures within. (b) 10000x. Points 1 and 2 indicate the spots analyzed by EDS. (c) 10000x. The dashed line indicates the linear EDS scan.

Source: Obtained by the author.

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EDS Point	Nb	Ni	Cr	Fe	С	Мо	Ti	Al	Si	Suspected Phase
1	10.50	49.75	16.00	15.83	2.20	4.71	1.01			Carbides / Laves / y
2	6.39	53.30	16.99	17.30		4.11	1.03	0.51	0.36	γ (with microsegregation)

Table 4.5 – EDS analysis results for points 1 and 2 indicated in Figure 4.22b (%wt.).

Source: Elaborated by the author



Figure 4.23 – Linear EDS scan results. The Nb content rises in zones corresponding to the secondary phase. Source: Obtained by the author

The measured microhardness of the solution-treated LPBF sample was 332 ± 16 HV_{0.5}. This number is statistically equal to the microhardness of the as-built sample, which again confirms the inefficacy of this AMS 5662 solution treatment at 980 °C for 1 hour for LPBF IN718. The fact that the average microhardness did not decrease after the solution treatment provides further evidence that the solution treatment at 980 °C for 1 hour did not cause recrystallization or annihilation of the cellular structure, and it confirms that the hard and brittle Laves phase was not solubilized.

This is surprising, since the expectation was that the solution treatment would work, as the precipitates are finer than those of wrought parts. Another detail worth noticing is that the error or standard deviation of the hardness measurements significantly increased (from 7 to 26 HV_{0.5}). This indicates that the effect of the heat treatment was not strictly uniform throughout the sample. It suggests that in some regions, the hardness did decrease, probably due to dissolution of Laves precipitates, to some extent. In other regions, instead, the hardness increased (when compared to the as-built state), possibly due to precipitation of δ phase, which was not seen, but was reported in literature at this temperature [77, 87].

4.3.3. Solution treatment plus single aging

The sample that went through solution treatment (980 °C, 1 hour, water quenching) plus single aging (720 °C, 8 hours, water quenching) had its microstructure investigated by OM, with magnifications of 100x (Figure 4.24a), 200x (Figure 4.24b) and 500x (Figure 4.24c). The melt pool boundaries are still visible and can be observed in Figure 4.24. In Figure 4.24b, it is possible to identify columnar grains growing epitaxially across different layers and melt pools. The columnar grains are approximately aligned in the building direction (vertical direction on the images), but often slightly tilted away from it. These minor deviations from the build direction are caused by the local heat flow at the melt pool boundary, which is maximum in a direction perpendicular to the melt pool boundary [61].

With a 500x magnification (Figure 4.24c), one can notice the meshed or striped patterns within the grains. As stated before, these patterns are actually the cellular dendritic subgrain structures. Therefore, the heat treatment consisting of a solution treatment plus single aging did not eliminate the subgrain structures nor led to recrystallization. Indeed, visible grains appear to be 10-50 μ m wide, about the same as the grains in the as-built or the solution-treated condition.

With this magnification, grain boundaries are clearly seen. In fact, the grain boundaries seem to be more defined in this micrograph (**Figure 4.24**c) than in the 500x OM micrograph of the solution-treated sample. This can be a consequence of secondary phase precipitation at grain boundaries after the aging treatment. At 720 °C, precipitation of γ " and γ ' is expected, and δ phase formation is also possible. Ti-rich and Nb-rich carbides may be formed as well, to some extent. The δ phase and carbides reportedly precipitate preferentially at grain boundaries in IN718 [24, 49], and the etchant used in this experiment (Kalling's solution #2) is known to attack precipitates preferentially. This way, the etching could reveal the grain boundaries to a greater extent if they are decorated with fine precipitates. The strengthening precipitates γ " and γ ' are likely formed, as reported in literature [67-69, 77], but they cannot be observed by OM (not even SEM) due to their very fine size [24, 49].



Figure 4.24 – OM images of the LPBF IN718 sample after solution treatment (980 °C, 1 h) plus single aging (720 °C, 8 h). (a) 100x. (b) 200x. (c) 500. Melt pools are still visible, as well as the columnar grains and their substructures. BD=building direction.

Source: Obtained by the author.

The microhardness of the sample resulted $468 \pm 21 \text{ HV}_{0.5}$. The average microhardness is 43% higher than the average microhardness of the as-built LPBF sample. This significant increase in hardness is strong evidence of the precipitation of γ " and γ ', even though these phases were not observed due to their nanometric dimensions. OM observations lead to believe that no significant grain growth has occurred, which is corroborated by the hardness results, since grain growth tends to be accompanied by a decrease in hardness. The same is valid for recrystallization.

The hardness result shows that although the solution treatment was not effective, the concentrations of alloying elements – namely Nb, Ti and Al – in the matrix is high enough to promote the formation of the strengthening phases at least in some regions of the microstructure, leading to an important rise in microhardness. Indeed, the standard deviation of 21 HV_{0.5} is three times higher than the standard deviation of the as-built sample (7 HV_{0.5}), suggesting that the effect of the heat treatment is not homogeneous throughout the microstructure of the whole sample. This is a direct consequence of the microsegregation that was observed in the as-built (and persisted in the solution-treated sample).

Additionally, it was noted that the microhardness of this heat treated LPBF sample is much higher than the microhardness of the cast sample that went through the same treatment, mainly due to the very fine AM microstructure. The microstructure refining provided by the AM route may counterbalance to some extent, the suboptimal precipitation of γ '' and γ ' caused by the ineffective solution treatment.

4.3.4. Full cycle

One LPBF sample went through the full thermal treatment as per AMS 5662, including the solution treatment (980 °C, 1 hour, water quenching), and a double ageing, first at 720 °C for 8 hours, furnace cooling to 620 °C, then holding at 620 °C for 8 hours and finishing with air cooling. The resulting microstructure was investigated by OM with magnifications of 100x (**Figure 4.25**a) and 200x (**Figure 4.25**b).

The melt pool boundaries are still visible (the building direction is vertical on the images), as well as the columnar grains, that span through multiple melt pools, and are predominantly aligned with the building direction, or close to it. Within the grains, the subgrain structures persist even after the full heat treatment, which is evidenced by the meshed or striped appearance of the microstructures. The grains are 10-50 μ m wide, so no significant grain growth is experienced, and no signs of recrystallization are seen. No precipitates are visually identified at this scale. Nevertheless, precipitation of γ " and γ ' is expected as a consequence of this heat treatment [77]. Also in this case, the grain boundaries seem more defined and discrete (when compared to the as-built sample), which may suggest that precipitation of secondary phases took place at grain boundaries.



Figure 4.25 – OM images of the LPBF IN718 sample after the full AMS 5662 heat treatment (solution treatment plus double aging). (a) 100x. (b) 200x. The typical microstructural features of the as-built sample are still visible (melt pools, columnar grains, subgrain structures). BD=building direction.

Source: Obtained by the author.

To get a better grasp at the microstructural features of the sample, SEM observations were made with magnifications of 3000x (Figure 4.26a), 5000x (Figure 4.26b) and 10000x (Figure 4.26c), and an EDS analysis was carried out on point 1 indicated on Figure 4.26c. The EDS results are presented in **Table 4.6**.

On the SEM micrographs, it is clear that the grains are elongated on the vertical (building) direction, and it is possible to observe many fine precipitates decorating the grain boundaries. The elongated (needle-like) morphology of the precipitates indicate that the majority of the precipitates are the δ phase. However, at a 10000x magnification, precipitates with irregular shapes and curved interfaces are also seen, suggesting that the Laves phase is present as well. Although the precipitates are seen in higher concentrations at grain boundaries, they are also found in grain interiors. The predominance of the precipitates at grain boundaries is a consequence of microsegregation, that happens especially severely in these regions during the LPBF process.

Although the subgrain structures are not easily identified on the SEM images, they are certainly present, as evidenced by the meshed or striped patterns within the grains on OM observations. Indeed, the precipitates that are seen in grain interiors lie likely at intercellular zones, since these regions also suffer significant microsegregation, as shown in the as-built sample and also in the solution-treated LPBF sample, as the solution treatment was not completely successful. Gallmeyer et al. reported through TEM observations that the cellular structures and dislocation cells survived the full AMS 5662 heat treatment in IN718 produced by LPBF [68]. A possible explanation as to why the cellular dendrites are not seen in the SEM images is that the etchant used (Kalling's #2) attacks preferentially secondary phases, and since the grain boundaries are decorated with plenty of precipitates, they were strongly revealed, outshining the intercellular zones. The intense presence of precipitates at grain boundaries elucidate why the grain contours were more defined in the micrographs of the aged samples in comparison to the as-built and to the solutionized samples. Moreover, it confirms that the single aged sample also witnessed precipitation of secondary phases at grain boundaries (and likely in the intercellular zones as well), since it also showed clearer grain contours on the OM images. This is in accordance with what is reported in literature [68, 69, 77].



Figure 4.26 – SEM images of the fully treated (solutionized and double aged) LPBF IN718 sample. (a) 3000x. (b) 5000x. (c) 10000x. The red cross-mark indicates the spot analyzed by EDS. Source: Obtained by the author.

Table 4.6- EDS re	sults for point !	l indicated in	Figure 4.26c (%wt.).
				. /

1 22 72 58 67 5 74 6 44 2 78 1 02 0 20 0 44 Lavas/	hase
1 25.72 38.67 5.74 0.44 2.78 1.92 0.50 0.44 Laves7	5

Source: Elaborated by the author.

The EDS analysis (**Table 4.6**) confirms that the precipitates seen are Nb-rich phases. Based on the morphology, most precipitates seem to be the δ phase (acicular morphology), but the particle that was analyzed is apparently the Laves phase, given the irregular morphology and the relatively high Ti content. However, the identification based on EDS results is not unequivocal. In order to acquire a precise phase recognition, SAED or XRD tests should be performed. Regardless, these results show that Nb-rich precipitates can be formed in the aging treatment, in spite of the poor effectiveness of the solution treatment at 980 °C. This is possible because despite the microsegregation and the undissolved Laves phase, the concentration of Nb in the matrix is still enough to form the δ phase and, quite likely, the strengthening phase γ ", at least in some areas of the microstructure. The same is valid for the γ ' phase, which instead of Nb boasts Ti and Al. As mentioned before, γ " and γ ' are usually only visible through TEM observations, due to their nanometric dimensions [24].

In fact, studies by Gallmeyer et al. and Jiang et al. demonstrated that the strengthening phases γ " and γ ' can be formed even upon direct ageing treatment at 720 °C and 620 °C, with no previous solution treatment at all. However, naturally, if the solution treatment is executed successfully, the following ageing treatment promotes the formation of a higher volume fraction of desired precipitates.

The microhardness test result was 482 ± 52 HV_{0.5}. In a strict manner, it is a 3% increase from single-aged sample and a 47% rise from the as-built LPBF sample. However, this number represents statistically no increase from the single-aged sample, when the error is considered. Moreover, the standard deviation is much higher than the standard deviation of the as-built sample (7 $HV_{0.5}$), and higher still than the standard deviation of the solutiontreated sample (16 HV_{0.5}) and the solution-treated and single-aged sample (21 HV_{0.5}). This evidences a cascading effect of the hardness standard deviation, that increases after every step of the heat treatment. This can be explained by the microsegregation that happens upon solidification during the LPBF process, which generates regions with fairly different concentrations of alloying elements. As a consequence, the chemical composition is inhomogeneous, and so is the hardness. Ultimately, the heat treatment affects these different regions of the microstructure in different manners. In some areas, where there is a higher availability of Nb, the γ " phase can be formed to a greater extent, with a higher volume fraction, leading to a hardness increase. In other regions, for instance, the metastable and coherent y" phase that was formed in the first aging step at 720 °C, can be transformed into the stable and incoherent phase δ , leading to a local decrease in hardness. In fact, while the γ " phase is nanometric and cannot be observed in the SEM micrographs, the submicron δ precipitates that can be seen may be the result of the γ " $\rightarrow \delta$ transformation.

The hardness results evidence that the strengthening precipitates can be precipitated (to some extent) even if the solution treatment is not ideal, achieving high microhardness values, comparable to values found in literature for aged LPBF IN718 [43, 45, 71, 72]. Naturally, the hardness could be even higher if the Nb-rich Laves phase was successfully solubilized. In addition, it is clear that the second step of the aging treatment did not prove very useful, since it did not lead to a significant increase in hardness. This is in accordance with what was found by Gallmeyer et al. [68], and can be explained by the fact that the strengthening phases precipitated during the first aging step, as indicated by the outstanding hardness increase (43%) from the as-built condition. Since the solution treatment was not fully effective in promoting the solubilization of the Laves phase, there was not much Nb or Ti left in the matrix for a second aging step.

Furthermore, experiments by Gallmeyer et al. [68] show that direct aging (with no solution treatment) at 720 °C for 24 hours on LPBF IN718 led to precipitation of both γ " and γ ', as seen through TEM observations, while direct aging at 620 °C for 24 hours led to formation of γ " only. This shows that at the lower temperature of the second aging step, the γ " phase is mainly formed, and not so much the γ ' phase. This observation aligns with the results

obtained in the thesis at hand: since the second aging is aimed at γ " (Ni₃Nb) precipitation, and most of the Nb atoms are already employed in the undissolved Laves phase, the δ phase and in γ " precipitates formed during the first aging step, indeed there is relatively few Nb atoms available to take γ " precipitation further on the second aging step at 620 °C. Moreover, at this temperature, the formation of the other strengthening phase γ ' is sluggish, so it does not boost hardness to a great extent either. Therefore, the microhardness does not rise as a consequence of the second aging step.

The obtained hardness measurement was comparable to results in literature. Deng et al., for instance, used the same heat treatment (AMS 5662) on LPBF-produced IN718 and reached a hardness of 490 HV_{0.3}, with a microstructure very similar to what is seen in this study (partsially dissolved Laves phase, δ particles at interdendritic zones and grain boundaries, and likely the presence of strengthening precipitates γ " and γ ', although not seen due to their reduced size) [69].

4.4.LDED

4.4.1. As-built

The LDED IN718 sample in the as-built condition was analyzed first through OM, to allow microstructure observation, with a magnification of 50x (Figure 4.27). At a glance, the micrograph seems fairly similar to that of the as-built LPBF sample. Indeed, the key microstructural features coincide: the melt pool boundaries are clearly visible, as well as columnar grains that grow epitaxially across multiple melt pools. Additionally, the meshed or striped appearance is found in the LDED microstructure too, indicating the presence of subgrain structures. Such remarkable similarities derive from the fact that both LPBF and LDED are additive manufacturing processes that fabricate the sample in a layer-wise fashion, with highly localized heating and rapid cooling [21].

However, upon careful consideration, it is possible to identify significant differences. The noticeable differences arise mostly from size and scale of the microstructural features (it is worth noticing that magnification – and thus the scale – in this micrograph is not the same as in the LPBF samples). In this LDED sample, the melt pools are much wider when compared to those in the LPBF samples, and the sample applies to the columnar grains. Moreover, the meshed or striped patterns within the grains (i.e. the subgrain structures) seem more distinguishable. All of these major differences derive from the disparity in cooling rates during the AM processes. As reported in literature [21, 81], although both LPBF and LDED processes boast very high cooling rates, the LDED process experience a lower cooling rate than LPBF. This is mainly due to the larger laser spot size and the lower scanning speed used in LDED (in comparison with LPBF) [81]. The powder particle size, which is larger for LDED powders, as confirmed by powder characterization in this study, also contribute towards coarser microstructural features in the LDED sample when compared to the LPBF samples, which use a finer powder. The higher laser power, coupled with lower scanning speed, also give birth to larger and deeper melt pools [78, 80].



Figure 4.27 – OM image (50x) of the LDED IN718 sample in the as-built condition. Melt pools are seen clearly, as well as columnar grains spanning across several layers. The meshed pattern within the grains suggests the existence of subgrain structures. BD=building direction.

Source: Obtained by the author.

The sample was also investigated using SEM, in a effort to attain a clearer view of the microstructural features, especially the subgrain structures, and confirm their existence. Observations were made with magnifications of 2000x (Figure 4.28a), 3000x (Figure 4.28b) and 10000x (Figure 4.28c). The building direction is the vertical direction on the images.

At a 2000x magnification (Figure 4.28a), it is possible to appreciate the subgrain structures, with a cellular dendritic morphology, as in the LPBF samples. The substructures are elongated approximately along the building direction (vertical) and have no secondary dendrite arms, or underdeveloped secondary arms in some cases. A brighter phase can be seen in the interdendritic regions, among neighboring cellular dendrites. Judging by the irregular morphology, and by the solidification thermodynamics of the alloy, this phase is likely the Laves phase. The Laves phase was observed in LDED IN718 samples by several authors [78, 80, 81, 87, 93]. The cellular dendrites are not perfectly parallel to the building direction. This can happen due to local heat flow, which is maximum in a direction perpendicular to the melt pool. Additionally, in LDED, it was reported in literature that the columnar grains and their subgrain structures may be tilted towards the direction of motion of the heat source (scanning direction), as it is one of the factors that determine the local heat flow direction [61, 62, 64].

In addition to the Laves phase, it is possible to observe small rounded dark elements in the microstructure that are somewhat evenly distributed. These features are more easily seen with the 3000x magnification (Figure 4.28b). The dark color and the rounded shape raise suspicion that these features could be either a secondary phase or porosities. If the case is that such features are porosities, then they must be gas porosities, given their spherical shape (lack of fusion defects have irregular shape and often sharp edges). The porosities could derive from the gas pores observed in the powder particles (trapped gas from the gas atomization process) or, alternatively, the porosities could be originated by entrapped shielding gas during the LDED process. To determine whether the dark elements are pores or a secondary phase, EDS analysis was carried out. The EDS point are indicated on Figure 4.28c, and the results are reported on Table 4.7.



Figure 4.28 – SEM images of the LDED IN718 sample in the as-built state. The subgrain structures are identified as cellular dendrites. Secondary phases populate the interdendritic zones. (a) 2000x. (b) 3000x. (c) 10000x. The red cross-marks identified with 1, 2 and 3 indicate the spots analyzed by EDS. Source: Obtained by the author.

Most of the Laves phase seen in Figure 4.28a and Figure 4.28b seem to be primary Laves precipitates, with a bulky morphology. However, on Figure 4.28b, it is possible to spot some eutectic Laves particles on the superior portion of the image. In fact, with the 10000x magnification, on Figure 4.28c, such morphology can be seen more clearly. The morphology of Laves phase in this LDED sample is a bit different than the morphology of Laves precipitates of the LPBF sample, which are finer and seem to be more elongated and continuous.

Three point (Figure 4.28c) were chemically investigated through EDS (Table 4.7). Two of them (points 1 and 2) lie on the supposedly Laves precipitates, at interdendritic or intercellular zones, and one of them lie on the spherical dark features (point 3). Points 1 and 2, as expected, return a very similar chemical composition, rich in Nb, Mo and Ti, which is typical of Laves precipitates [87], confirming their suspected identity. The formation of Laves phase at the interdendritic zones is in accordance with lilterature [89, 92, 93] and evidences Nb and Mo segregation in these regions, showing that the rapid cooling experienced during the LDED process is enough to prevent macrosegregation, but not microsegregation. The Laves precipitates are coarser than those seen in the LPBF samples, attesting to the lower cooling rates of the first with respect to the latter. The slower cooling rates in LDED allow the occurrence of even stronger microsegregation (as more time is allowed for the atoms to diffuse) and coarser Laves phase formation when compared to LPBF.

As for point 3, the result of the EDS analysis was not very conclusive, as it was similar to the nominal composition. This is probably due to the analyzed volume, which is greater than the dark region only, so the surrounding matrix, both around and underneath the dark element is taken into consideration. Nevertheless, one important detail is eye-catching: the Nb concentration is below the lower limit of the nominal composition of the alloy (4.75%wt). This shows that the cellular dendrite trunks are depleted in Nb, which makes the precipitation of the strengthening phase γ " during the aging treatment more difficult and scarse. The Nb-depletion on the denrite core is further proof of microsegregation.

EDS Point	Nb	Ni	Cr	Fe	Мо	Ti	Al	Si	Suspected Phase
1	13.00	50.34	15.07	14.83	4.65	1.15	0.52	0.44	Laves / γ
2	11.06	51.43	15.56	15.07	4.52	1.23	0.71	0.42	Laves / γ
3	3.34	54.35	18.00	19.14	3.60	0.59	0.64	0.34	γ

Table 4.7- EDS results for points 1, 2 and 3 indicated in Figure 4.28c (%wt.).

Source: Elaborated by the author.

Since the point EDS analysis was inconclusive for the dark features, and in order to get confirmation of the remaining phases' identity, an area scan EDS analysis was performed, assessing the chemical composition of a selected area of the microstructure. The analyzed area is portrayed in **Figure 4.29**a, with an overall composition map in **Figure 4.29**b. Concentration maps for individual alloying elements are shown in **Figure 4.30**(a-i), including Ni, Cr, Fe, O, Nb, Mo, Al, Ti and Si. The area analyzed is precisely the same as in **Figure 4.29**a.

In the area scan concentration maps, the darker the color, the higher the concentration of the analyzed element. On the Ni map, it is clear that the Ni concentration is somewhat uniform throughout the microstructure, being just a bit lower on the interdendritic zones due to the presence of microsegregated alloying elements and secondary phases (although the key secondary phases also bear Ni in their composition, which explains the subtle difference between the color intensity of the dendrite trunks and the interdendritic zones on the Ni map). There are, however, a few rounded regions where the Ni concentration is significantly lower (brighter regions on the concentration map). These spherical regions coincide with the dark spherical microstructural features seen by SEM. The lower Ni concentration suggests that these regions might be a secondary phase rather than porosities.



Figure 4.29 – (a) SEM image (20000x) of the LDED sample in the as-built state. (b)EDS area scan for the area depicted in (a).

Source: Obtained by the author



Figure 4.30 – Area EDS analysis for the region displayed in Figure 4.29a. The concentration maps (a-i) show the concentration distribution of the analyzed element throughout the analyzed area. Darker colors indicate a higher concentration. (a) Ni content. (b) Cr content. (c) Fe content. (d) O content. (e) Nb content. (f) Mo content. (g) Al content. (h) Ti content. (i) Si content.

Source: Obtained by the author

The Cr map and the Fe map are very similar, with a uniform concentration on the dendrite cores (more intense color) and a depleted zone (brighter color) that coincide with the interdendritic zones. This confirms that the cellular dendrites are indeed the matrix γ , and constitutes further evidence of microsegregation in the interdendritic zone: the concentration (weight percentage) of Cr and Fe is lower on the interdendritic zone because this region is populated with high concentration of other elements due to segregation and secondary phase formation. In addition, the rounded microstructural features are not rich in Cr or Fe.

The oxygen concentration map shows an increase in oxygen concentration on the rounded microstructural features, indicating that these spherical features are likely oxide particles. The oxygen probably comes from the LDED process, which is not as protected from oxidation as the LPBF process. While LPBF is undertaken inside a sealed building chamber with either vacuum or an inert atmosphere (e.g. argon), the LDED process has no building chamber, happening in the atmosphere, with the only protection being the shielding gas stemming from and around the nozzle. This protection is not as effective as a sealed envelope with vacuum or a controlled atmosphere, and oxidation in IN718 produced by LDED is reported in literature [21].

The concentration maps of Nb and Mo are very similar, except that the Nb case is more intense. Both alloying elements are present in greater concentrations at the interdendritic zones, providing definitive proof of the occurrence of microsegregation. These two elements are the most impacted by microsegregation due to their large atom size and atomic weight, limiting their diffusivity. Furthermore, the interdendritic zone witnesses the formation of Laves phase, which is also rich in Nb and Mo, hence the higher concentration of these elements at the regions corresponding to the brigh irregular-shaped precipitates seen in SEM images. Apparently, these elements do not take part in the formation of the spherical oxygenrich features.

The concentration maps of Al and Ti are also very similar among them. Both Al and Ti are mostly uniformly distributed throughout the microstructure, but have disproportionately high concentrations precisely on the round oxygen-rich regions. This leads to the conclusion that such spherical features are in fact Al-rich and Ti-rich oxides, likely Al₂O₃ and TiO₂. The presence of these oxides has been reported in literature by Gallmeyer et al. [68]. On the Ti concentration map, there are a few Ti-rich spots that do not appear on the Al map (i.e. are not Al-rich). Such spots are probably Ti carbides (mostly TiC). In fact, these spots appear in the interdendritic zones (coinciding with the Nb-rich zones on the Nb concentration maps), which makes it even more likely that these spots are Ti-rich carbides, and quite possibly rich in Nb too ((Ti,Nb)C). The Si concentration was also assessed and is uniform throughout the whole analyzed area.

A microhardness measurement was performed with a load of 500 gf and dwelling time of 15 seconds, and the result was an average value of 253 ± 18 HV_{0.5}. This number is approximately 23% higher than the average microhardness of the cast sample with no heat treatment, and roughly 23% lower than the average microhardness of the LPBF sample in the as-built state. The difference in hardness is mainly due to the differences in grain size (and subgrain structures in the case of the AM samples) among the samples. The differences in grain size, in turn, are consequence of the different cooling rates experienced in each process. The casting process has a lower cooling rate (in the order of 1 K/s [58, 84]), leading to a coarser microstructure, and thus a lower hardness, according to the Hall-Petch effect. The LPBF has an extremely high cooling rate (in the order of 10 μ m) and additionally very fine subgrain structures (with a width in the order of 1 μ m), which result in a high hardness.

The LDED process boasts a high cooling rate in the order of 10^3 - 10^4 K/s, orders of magnitude higher than that of the casting process, but considerably lower than the cooling rate of the LPBF process [21, 45, 58, 75]. As a consequence, the microstructure obtained through LDED is much finer than the microstructure of the cast sample, but not as fine as the microstructure of the LPBF sample. Henceforth, the microhardness value is intermediate, greater than the hardness of the cast sample, but lower than the hardness of the LPBF sample. The microhardness value is comparable to other values reported in literature for IN718 parts produced by LDED [88, 90, 92, 93]. Xiao et al., for instance, found a microhardness of 263 HV_{0.5} for as-built LDED IN718 samples, which showed a microstructure similar to the one observed in this study, with columnar grains and fine cellular dendritic subgrain structures [80]. Xiao and colleagues saw continuous Laves precipitates at interdendritic zones, at a volume fraction of 6.8% (assessed by XRD), and an primary dendritic arm spacing of 5 to 8 µm [80].

4.4.2. Solution treatment

The solution treated LDED sample (980 °C, 1 hour, water quenching) was investigated through OM with magnifications of 100x (Figure 4.31a) and 200x (Figure 4.31b). It is worth highlighting that the magnification used for this sample is greater than that used for the asbuilt sample. On the micrographs, the melt pool boundaries are not clearly visible after the solution treatment. On the other hand, the columnar grains are still present and clear to see. The grains are elongated predominantly along the building (vertical) direction, and the meshed patterns are seen within the grains, suggesting that the subgrain structures survived the solution treatment as well.

On **Figure 4.31**b, the grains are slightly tilted away from the building direction. As stated before, this can happen due to local heat flow direction, which is influenced by the melt pool shape and depth and the motion of the heat source. In LDED the melt pool tends to be deeper than in LPBF, due to the typically higher laser power and lower scanning speed, and as a consequence, the grains tend to be more deviated from the building direction when compared to LPBF, since epitaxial grain growth happens from the melt pool boundary into the melt pool interior [21, 61, 62]. The grains are coarser than those found in the LPBF samples, which is expected, since LDED has a lower cooling rate than LPBF. Nevertheless, no grain growth appears to have occurred from the as-built state to the solution-treated condition. Recrystallization also did not take place, since the grains are still columnar and have subgrain structures.

In order to better observe the subgrain structures, SEM investigations were carried out, with 3000x (**Figure 4.32**a) and 5000x (**Figure 4.32**b) magnifications. On the SEM images, it is possible to see the elongated cellular dendrites approximately parallel to the building direction, especially on **Figure 4.32**b. The cellular dendrites are about 5 μ m wide. In the interdendritic zones, a clear, bright phase is seen, which may be either the Laves phase, undissolved during the heat treatment, or the δ phase, which was not observed in the as-built state, but can be formed at 980 °C as reported by Liu et al. [87]. In addition, the spherical dark oxides are also seen.



Figure 4.31 – OM images for the LDED sample after solution treatment (980 °C, 1 h). (a) 100x. (b) 200x. The columnar grains are seen clearly in both images. BD=building direction.

Source: Obtained by the author.



Figure 4.32 – SEM images of the LDED sample after solution treatment (980 °C, 1 h). It is possible to observe the cellular dendritic subgrain structures, aligned along a direction close to the building direction. Secondary phases populate the interdendritic zones. (a) 3000x. (b) 5000x. Source: Obtained by the author.

With the greater magnification of 5000x (Figure 4.32b), it possible to notice that the phases in the interdendritic zones have two distinct morphologies: some precipitates have irregular shape, and others have needle-like geometries. The irregular morphology is typical

of the Laves phase, and the acicular morphology is typical of the δ phase. In addition, when compared to the SEM micrographs of the as-built LDED sample, the solution-treated sample displays an apparently lower volume fraction of Laves phase. This shows that the solution treatment was not completely effective, since the Laves phase is still present after the heat treatment, however, the treatment did achieve a certain degree of solubilization of the Laves phase, as its volume fraction seems to have diminished. Furthermore, the solution treatment caused the formation of needle-like δ precipitates, which were not seen in the as-built state. A reasonable hypothesis is that the solution treated led to the partial dissolution of some Laves precipitates, making more Nb atoms available in the matrix, which were then consumed in the precipitation of δ particles. Another factor that supports this theory is the fact that the δ precipitates are located in the proximity of Laves particles, at the interdendritic zones. Liu et al. showed that in heat treated IN718 samples fabricated by LDED, the precipitation of the δ phase was associated with the dissolution of the Laves phase [87]. Although this tradeoff between the Laves and δ phases may lead to a lower content of Laves phase and thus a less brittle behavior with improved ductility, the δ phase also consumes Nb, so it does not optimize the Nb content available to form the strengthening phase γ " in the subsequent aging treatments.

In order to confirm the identity of the bright precipitates at interdendritic zones, EDS analysis was performed on point 1 indicated on the SEM image in Figure 4.33. The point analyzed lies onto a precipitates, and the result is reported on **Table 4.8**. The concentrations of Nb (6.30% in weight) and Mo (4.20% in weight) are both above the upper limit of the nominal composition of the alloy (5.50%wt. and 3.30%wt., respectively), which suggests that the precipitate is rich in both Nb and Mo, which is consistent with the composition of the Laves phase. The irregular morphology of the precipitate also corroborates this suspicion, as well as the high contents of Cr and Fe. Liu et al. also reported the persistence of Laves phase even after solution treatment at 980 °C [87].



Figure 4.33 – SEM image of the LDED sample after solution treatment (980 °C, 1 h). Bright regions suggest the existence of secondary phases. The red cross-mark indicates the point analyzed by EDS. Source: Obtained by the author

Table 4.8 – EDS resultsfor point 1 indicated in Figure 4.33 (%wt.).

EDS Point	Ni	Cr	Fe	Nb	Мо	Ti	Si	Al	Suspected Phase			
1	52.76	17.18	18.03	6.30	4.20	0.75	0.39	0.38	Laves / y			
Commente Elaboration de la construction												

Source: Elaborated by the author

The microhardness of the sample was assessed and resulted 262 ± 12 HV_{0.5}. This represents an increase of roughly 4% in average hardness from the as-built condition. However, when the error is taken into account, this value is statistically equivalent to the hardness of the as-built LDED sample. Similarly to what was observed for the LPBF sample, the solution treatment was not completely successful in dissolving the hard and brittle Laves phase, so the microhardness of the sample did not decrease significantly. Moreover, as seen on the SEM micrographs, the LDED sample witnessed the formation of very fine δ precipitates, which are also hard and may counterbalance the decrease in hardness due to the solubilization of Laves phase. The precipitation of δ phase may explain the slight increase in average microhardness after the solution treatment. Moreover, the fact the the hardness did not decrease significantly provides further evidence that neither grain growth nor recrystallization took place during the heat treatment.

4.4.3. Solution treatment plus single aging

One sample produced by LDED was submitted to solution treatment (980 °C, 1 hour, water quenching) plus a single aging at 720 °C for 8 hours, finishing with water quenching. The sample had its microstructure investigated by OM, with a magnification of 100x (Figure 4.34a) and 200x (Figure 4.34b). On the micrographs, the arc-shaped melt pool boundaries are visible, especially on Figure 4.34a. Columnar grains are seen clearly, and grow mostly in directions perpendicular to the melt pool boundaries. The grains show clear signs of epitaxial growth during the LDED process, permeating multiple melt pools in subsequent layers. No significant grain growth seems to have occurred during heat treatment, as the grains have widths of tens of microns, as in the non-treated and the solution-treated samples. Moreover, the subgrain structures are still present, evidenced by the meshed patterns within the grains. No significant grain growth or recrystallization took place, similarly to what was seen for the LPBF sample. On Figure 4.34a, a rounded dark microstructural feature can be seen in the bottom-right corner of the image, which is likely an oxide particle, as the ones observed in the as-built state. On Figure 4.34b, three columnar grains are easily identified on the center of the image.

The microhardness of the sample was assessed at $455 \pm 22 \text{ HV}_{0.5}$. This represents an outstanding increase of 80% from the hardness measured in the as-built condition to the solution-treated plus single-aged condition. This remarkable rise in hardness as a consequence of the aging treatment at 720 °C shows that this treatment is capable of provoking the precipitation of strengthening phases γ " and γ ' in the alloy, even though the phases were not identified by OM due to their typically nanometric size. In fact, these phases are not commonly seen not even through SEM, so hardness measurements are an indirect strategy for getting an indication of their presence. And the presence of γ " and γ ' implies that in spite of the remaining undissolved Laves phase after the solution treatment, there was sufficient Nb and Ti (or Al) content in the matrix to allow the formation of the strengthening phases. In fact, the same behavior was seen in the LPBF samples, so even when the solution treatment

was ineffective, precipitation of γ " and γ ' was possible, to some extent, during the first aging step at 720 °C.



Figure 4.34 – OM images for the LDED sample after solution treatment (980 °C, 1 h) plus single aging (720 °C, 8 h). (a) 100x. Arc-shaped melt pools are still visible. (b) 200x. Columnar grains are seen in the central portion of the image. BD=building direction.

Source: Obtained by the author.

For the LDED samples, the solution treatment seems to be more effective than it was for the LPBF samples, as evidenced by the lower amount of Laves phase visible on the SEM micrographs after the solution treatment in comparison to the as-built LDED sample. Indeed, if the solution treatment was more effective (although yet sub-optimal) for the LDED sample, the Nb content available for the formation of γ " during the aging treatment should be higher when compared to the LPBF samples, resulting a higher volume fraction of the strengthening phase, leading to a more substantial hardness increment (and the same applies to the γ ' phase). In fact, while the LPBF witnessed an increase in average microhardness of 43%, the LDED sample saw a growth of 80%. This superior improvement in hardness is a direct consequence of the effectiveness of the solution treatment. The aging treatment is reported to successfully cause precipitation of the strengthening phases γ " and γ ' in IN718 samples manufactured by LDED [80-82, 84]. With the greater rise in hardness, the LDED achieved an average microhardness that is just 3% lower than the microhardness of the LPBF sample in the solution-treated plus single-aged condition. When the standard deviation is taken into account, one might state that the numbers are statistically equivalent. Nonetheless, the hardness of LDED samples are expected to be lower than those of LPBF samples, since the microstructure of the latter is finer. This difference in microhardness due to the microstructure of LPBF-produced parts being finer than the microstructure of LDED-produced parts is supported by literature [45].

4.4.4. Full cycle

The sample built by LDED and heat treated according to the full AMS 5662 thermal treatment was thoroughly investigated in order to assess the effect of the heat treatment as prescribed by the AMS standard on the microstructure of the alloy. The OM micrographs with magnifications of 100x and 200x are shown in **Figure 4.35**a and **Figure 4.35**b, respectively.

On Figure 4.35a, it is possible to observe arc-shaped melt pool boundaries, which survived the thermal treatments. Predominantly columnar grains are seen crossing different

melt pools, evidencing the occurrence of epitaxial growth in the LDED process. The columnar grains are mostly aligned in a direction close to the bulding direction, as expected and reported in literature [82, 89]. Within the grains, the meshed or striped patterns suggest the presence of subgrain structures, as seen in the previously discussed LDED samples. These observations show that the heat treatments did not cause recrystallization, which is corroborated by the absence of twin boundaries, a common sign of recrystallization [79, 84]. The grains are tens of microns wide, which is the same width range as in the other LDED samples, so no significant grain growth seems to have happened either.

On **Figure 4.35**b, with a greater magnification, columnar grains can be seen clearly, aligned closely with the building (vertical) direction, growing across multiple melt pools. A grain boundary is easily identified close to the vertical direction, on the central-right part of the image. Within the grains, the patterns indicate the existence of subgrain structures, which are likely cellular dendrites, as seen in the other LDED samples. In the grain on right side of the image, the pattern is different from the pattern seen on the grain located on the central-left part of the image. On the right, the pattern is dotted or mesh-like, while in the left, the pattern is striped. Both patterns are created by the cellular dendritic substructures, and their different appearance is due to different orientations and growth directions of the cellular dendrites. The meshed pattern in created by cellular dendrites growing in a direction that intersects the observation plane (a direction close to perpendicular to the page). The striped pattern is created by cellular dendrites growing in a direction growing (vertical) direction (approximately parallel to the page).



Figure 4.35 – OM images for the LDED sample after the full AMS 5662 heat treatment (solution treatment plus double aging). (a) 100x. Melt pools persist even after the full heat treatment, and columnar grains are still seen spanning across multiple layers and melt pools. (b) 200x. Columnar grains aligned closely with the building direction. Within the grains, subgrain structures appear to exist. BD=building direction.

Source: Obtained by the author.

In order to get a clear view of the subgrain structures and the phases present, SEM analyses were carried out. Micrographs with magnifications of 3000x and 5000x are portrayed in Figure 4.36a and Figure 4.36b, respectively. Additionally, observations with 10000x (Figure 4.37a and b) and 20000x (Figure 4.37c) magnification were executed, and EDS analysis was performed on three points, indicated on the micrographs (Figure 4.37).

On Figure 4.36a, the cellular dendritic elongated subgrain structures are seen aligned in a direction that is close to the building (vertical) direction. In the interdendritic zones, bright secondary phases are present. In addition, spherical dark oxide particles are seen both in interdendritic zones and dendrite trunks alike. Overall, the microstructure is very similar to the solution-treated-and-single-aged LDED sample, which may suggest that the second aging step at 620 °C for 8 hours does not cause significant changes on the microstructure, similarly to what was observed for the LPBF samples.

On **Figure 4.36**b, with a greater magnification, the microstructural features can be seen more clearly. The cellular dendrites portrayed on the image grow in a diagonal direction, from the bottom-left corner of the image to the top-right corner. As stated before, the cellular dendrites may deviate from the building direction due to local heat flow, which is influenced by the melt pool shape and the direction of motion of the heat source. This deviation is stronger for LDED than what it is for LPBF, because the melt pools in LDED are deeper, due to greater laser power and lower scanning speed [21, 81]. Since the heat extraction is maximum in a direction perpendicular to the melt pool boundary, a deeper melt pool leads to maximum heat flow directions that can differ considerably from the building (vertical) direction, originating grains that grow misaligned with the building direction.



Figure 4.36 – SEM images of the LDED sample after the full AMS 5662 treatment (solution treatment plus double aging). (a) 3000x. Approximately parallel cellular dendritic subgrain structures are seen, as well as fine oxide particles (dark round particles). (b) 5000x. Cellular dendrites are seen in greater detail. Secondary phases populate the interdendritic zone. Irregular Laves phase, acicular δ phase and round oxide particles seem to be present.

Source: Obtained by the author

The observation with 5000x magnification allows the identification of the morphology of the phases in the interdendritic zones. There are two distinct morphologies: some of the bright precipitates have irregular and continuous shapes typical of Laves phase, while others have a needle-like morphology, traditionally associated with the δ phase. Thus, both the Laves phase and the δ phase are present at the interdendritic zones, as seen also in the solution-treated sample. As discussed before, the strengthening phases γ " and γ ' are too small to be seen by OM or SEM, but their presence is expected, since there was evidence of their existence in the single-aged sample, and the aging treatments are reported to cause their formation [78, 80, 90].

The observations with 10000x magnification (Figure 4.37a and b) allow a closer view of the interdendritic precipitates. Once again, irregular precipitates are seen alongside acicular particles, suggesting the presence of Laves and δ phases. Judging by visual observation, the volume fraction of Laves phase appears to be significantly lower than what is seen on the asbuilt state, and the presence of δ precipitates may stem from the partial dissolution of some Laves particles, which provide enough Nb to allow the formation of δ precipitates. Indeed, the solution treatment appears to be more effective on the LDED samples than on the LPBF samples. This shows the importance of the starting microstructure, which greatly influences the outcome of the thermal treatment. The δ particles have a strong presence in interdendritic (or intercellular) zones, as seen on the SEM micrographs, and also likely at grain boundaries, which may be the reason why grain contours are easily visible on the OM micrographs. Some (few) δ particles are also seen within the cellular dendrites. Lastly, some bright precipitates appear to have straight faces and sharp edges (or a blocky morphology), which suggests that carbides may also be present, which is in accordance with literature [81]. In fact, presence of Ti-rich precipitates (dissociated from oxygen) was seen in the concentration maps elaborated for the as-built sample, and such precipitates can possibly be TiC.

The EDS points 1, 2 and 3, indicated in **Figure 4.37**a, b and c, respectively, have the results reported on **Table 4.9**. All three points lie on Nb-rich precipitates. Points 1 and 3 are likely the Laves phase, given the high Nb content coupled with fairly high amounts of Cr, Fe, Mo and Ti. Point 2 appears to lie on either a δ precipitate or a Nb-rich carbide, bearing in mind the very high concentration of Nb (18.34%wt.) and lower amounts of Cr and Fe. However, the precipitates are very fine (on the order of magnitude of 1 µm), and the analyzed volume may include surrounding phases, including the matrix γ . In order to identify the phases unequivocally, XRD or SAED analyses would be required, which were not executed in this work. Nonetheless, it is clear that microsegregation on the interdendritic zones made those regions prone to the precipitation of secondary phases, especially Nb-rich phases. Since the interdendritic zones are enriched in Nb, the dendrite cores must be depleted in Nb, which makes the formation of the strengthening phase γ " more likely to happen at the interdendritic zones.

Oxygen was detected in points 1 and 3, and may have been reported due to the presence of oxides formed during the LDED process, as a consequence of the suboptimal oxidation protection provided by the shielding gas. As seen in the as-built sample, there are spherical Al-based and Ti-based oxides in the microstructure, and the volume analyzed by EDS may have included underlying oxide particles below the surface of the sample. In addition to Al and Ti oxides, other oxides are reported in literature, such as Mo-based, Cr-based and Nb-based oxides [68]. The presence of Br, detected on point 3, is likely due to the substances used on the maintenance of the SEM equipment, and not necessarily due to the presence of Br in the alloy.



Figure 4.37 – SEM images of the fully heat treated LDED sample (solution treatment plus double aging). Red cross-marks indicate the spots of EDS analysis. (a) 10000x. (b) 10000x. (c) 20000x. Source: Obtained by the author.

<Î 13.4

EDS Point	Nb	Ni	Cr	Fe	Мо	0	Ti	Al	Si	Br	Suspected Phase
1	10.28	51.93	13.91	14.15	3.58	3.90	1.16	0.64	0.45		Laves / δ / oxides
2	18.34	57.95	10.00	10.44	3.27						Laves / δ / NbC
3	10.89	49.55	13.44	13.72	3.50	4.73	1.45			2.72	Laves / oxides

Table 4.9- EDS results for the points indicated in Figure 4.37 (%wt.).

Source: Elaborated by the author

The sample had its microhardness measured at 457 ± 7 HV_{0.5}. This is only a 0.4% increase on the average hardness from the solution-treated and single-aged sample and when the error is considered, the numbers are statistically equivalent. This shows that the second aging step, at 620 °C, was not very effective in promoting strengthening of the alloy through the precipitation of γ ' and γ ', similarly to what was observed for the LPBF samples.

On the other hand, the average microhardness value for the fully heat treated sample is 81% higher than the average microhardness of the as-built LDED sample. This evidences that strengthening precipitates were formed indeed. However, the lack of significant increase in hardness after the second aging step (when compared to the single-aged sample) suggests that the precipitation of strengthening phases occurred mostly during the first aging step, at 720 °C. This is similar to what happened with the LPBF sample. In fact, the higher temperature enhances the precipitation kinetics of both γ " and γ ', and the temperature of 720 °C allows the precipitation of both phases [58, 75, 77]. Meanwhile, the temperature of 620 °C may favor the formation of γ " over γ ' in AM parts, as shown by Gallmeyer et al. [68], because the precipitation of γ ' is more sluggish [77, 81]. The inefficacy of the second aging step in promoting further increase in hardness may be explained by the possible consumption of the available Nb atoms for the formation of γ " in the first aging step, leaving few Nb atoms available for further precipitation of γ " in a second aging step, especially since the solution treatment was not very effective in enhancing the availability of Nb. And as the aging at 620 °C favor the formation of γ " over γ ', the Al and Ti atoms that were not trapped in Laves or δ phase cannot promote significant hardness improvement, as the formation of γ is limited.

Furthermore, the fact that the hardness really increased after the first aging treatment (growth of 80% from the as-built hardness), and not really after the solution treatment alone (growth of 4% from the as-built hardness), confirms that the hardness increase is mainly due to the precipitation of γ " and γ ', and not as much due to the formation of δ phase, since the δ phase was already present in the solution-treated sample, which did not show a great hardness improvement. Therefore, the rise in microhardness is a strong evidence of the precipitation of γ " and γ '.

It worth noticing that the standard deviation decreased to only 7 $HV_{0.5}$ after the full cycle (from 22 $HV_{0.5}$ immediately after the first aging step). This suggests that even though the second aging step did not increase substantially the hardness, it led to a more uniform hardness distribution on the sample.

After the full thermal treatment, as per AMS 5662, the hardness of the sample produced by LDED reached 95% of the value of the hardness of the sample fabricated by LPBF (in the as-built state, the average hardness of the LDED sample was only 77% of the hardness of the LPBF sample). Moreover, the average microhardness of the fully treated LDED sample is 24% superior to that of the fully treated cast sample.

The fact that the hardness of the LDED sample is higher than the hardness of the cast sample and lower than the hardness of the LPBF sample is mainly due to grain size and microstructure fineness. Thanks to the high cooling rates inherent to AM technologies, the LDED process produces a fine microstructure, with columnar grains of reduced width and even finer subgrain structures, providing a great amount of obstacles for dislocation motion. Such microstructure is much finer than the microstructure of the cast sample, since the investment casting process has a relatively low cooling rate. Consequently, the hardness of the LDED sample is higher than the hardness of the cast sample. On the other hand, the LPBF process boasts a cooling rate that is even finer than the LDED's, resulting an even finer microstructure, as observed in this study, and ultimately a higher hardness.

Nevertheless, the LDED samples experienced a hardness increase (80%) that was much greater than the rise seen by the LPBF samples (47%). This is linked to the greater efficacy of the solution treatment for the LDED samples when compared to the LPBF samples. The solution treatment was apparently more successful for the LDED samples than it was for the LPBF samples, which is evidenced by the fact that the volume fraction of the Laves phase in the LDED solution-treated sample appears to be noticeably lower when compared to the asbuilt state, whilst for the LPBF samples, one can hardly notice the change in Laves phase content before and after the solution treatment. A more successful solution treatment leads to a greater availability of Nb atoms to form the strengthening phase on the aging treatment, ultimately leading to a higher hardness. These results highlight the importance of the starting microstructure when considering the effects of a heat treatment: different initial microstructures are affected differently by the heat treatment, and therefore, a manufacturing process that has such unique characteristics as AM processes should gain a heat treatment that is specifically tailored towards the distinct characteristics of parts produced by these methods. Indeed, with the AM realm, different technologies can produce considerably different microstructures that are affected in diverse manners by a same thermal treatment, as shown in this study.

The measured hardness for the fully heat treated LDED sample is compatible with results found by other authors. Xiao et al., after the same AMS 5662 heat treatment, reported a microhardness of 414 HV_{0.5} for an IN718 sample built by LDED. The microstructure of their sample contained columnar grains with cellular dendritic substructures, remaining Laves phase at interdendritic zones, as well as needle-like δ precipitates and the nanometric phase γ " [Xiao]. The difference in microhardness among Xiao's sample and the sample studied in the thesis at hand is likely a consequence of different process parameters. In fact, the process parameters greatly influence the resulting microstructure, especially the grain size, which in turns determines different hardness values.

4.5. Microhardness

The microhardness values were already presented and extensively discussed on the sections referent to each sample. Figure 4.38 summarizes all the microhardness results. As a general trend, one can notice that regardless of the heat treatment, the cast samples always show the lowest hardness among the three manufacturing methods, while the LPBF samples have the highest hardness values, with LDED samples displaying intermediate numbers.

This is a direct consequence of the grain size and microstructure fineness, which in turn is a result of the cooling rate. The LPBF process has the highest cooling rate, leading to a finer microstructure and thus higher hardness. Moreover, the extremely rapid cooling results in considerable residual stresses, which may originate dislocation arrays, also contributing to a hardness increase. On the other hand, the casting process has the lowest cooling rate, and thus a coarser microstructure. In fact, the cooling rate of IN718 LPBF is on the order of 10^6 K/s, while the cooling rate of IN718 investment casting is in the order of 1 K/s, as shown by Zhang et al. [75]. The LDED process has intermediate cooling rates (on the order of 10^3 to 10^4 K/s), which explains the intermediate hardness values. The measured microhardness samples were in accordance with hardness values found in literature, presented in Table B.1 and Table C.1.



Figure 4.38 – Microhardness results obtained in this work for IN718 samples produced by LPBF, LDED and casting, following different heat treatments based on AMS 5662.

Source: Elaborated by the author.

Another trend worth mentioning is that the solution treatment was not very effective for neither sample, since it did not decrease the volume fraction of hard and brittle phases (mainly the Laves phase) in a consistent fashion, which is indicated by the fact that the hardness did not decrease substantially after the solution treatment.

In spite of this, the first aging step seems to be able to promote the formation of the strengthening phases, as evidenced by the significant increase in hardness after the said treatment. This is especially true for the LDED sample, which reached 95% of the hardness value of the LPBF sample after the single aging treatment.

However, the second aging step did not seem to cause significant hardness improvement, possibly due to scarce availability of Nb to promote further precipitation of γ ", since the solution treatment was not completely successful. Gallmeyer et al., found that the second aging step indeed did not lead to significant improvements on the microstructure nor tensile properties of IN718 produced by LPBF, which is in accordance with the findings of this study.

In order to guarantee the solubilization of the Laves and provide sufficient Nb atoms to promote optimal the formation of the strengthening precipitate γ ", the solution treatment should be executed at a higher temperature for AM-fabricated IN718. Moreover, the second aging step, at 620 °C, could possibly be eliminated, since it does not appear to contribute significantly to hardness improvements. In fact, with this in mind, Gallmeyer et al. proposed a thermal treatment specifically tailored for LPBF IN718, consisting of a solution treatment at 1020 °C for 15 minutes, water quenching, and aging at 720 °C for 24 hours and air cooling. The microstructure resulting from this treatment comprises columnar grains aligned with the build direction, with cellular subgrain structures (610 nm), MC and Al₂O₃ at intercellular zones (Laves and δ were dissolved), plus homogeneously distributed lenticular γ " (29 nm) and spheroidal γ' (23 nm) and γ''/γ' coprecipitates (18 nm) [68].

To provide other examples, Deng et al. [69] achieved a microhardness of 500 HV_{0.3} for LPBF IN718 following a heat treatment consisting of a homogenization at 1080 °C for 1 hour with water quenching, plus double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 8 hours and air cooling. For LDED, Kong et al. measured a hardness of 475 HV0.1 after a heat treatment consisting of homogenization at 1095 °C for 1 hour, air cooling to room temperature, then solution treatment at 980 °C for 6 hours, then furnace cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C at 55 °C/h and holding for 8 hours, and finally air cooling [93].

These results show that even though the heat treatment prescribed in AMS 5662 is known to work successfully for wrought IN718 parts, one should not blindly apply the same strategy for parts manufactured otherwise, as the starting microstructure may heavily influence the outcome of the thermal treatment.

5. CONCLUSIONS

In the as-built state, IN718 parts produced by AM display a sub-optimal microstructure, with a great amount of the hard and brittle Laves phase and no strengthening (nanometric and coherent) precipitates, so a thermal treatment is necessary in order to improve the microstructure and consequently mechanical properties. Currently, the AMS 5662 is the most commonly used heat treatment for AM IN718 parts. The AMS 5662 thermal treatment is the standard heat treatment for wrought IN718, and it is known to successfully promote the solubilization of detrimental Laves phase and the formation of strengthening precipitates γ " and γ ' in wrought IN718 parts. In this work, the effect of this heat treatment on the microstructure and hardness of IN718 parts produced by AM was assessed, through OM and SEM observations, EDS analysis and microhardness measurements.

In the present study, Inconel 718 parts were produced by two different AM techniques: LPBF and LDED. For benchmarking purposes, IN718 samples produced by investment casting were also investigated (while the AM-fabricated samples experience an extremely high cooling rate, solidifying in a non-equilibrium state, the investment cast samples solidify with a low cooling rate, closer to the equilibrium). The samples were submitted to thermal treatments based on AMS 5662, which comprises a solution treatment at 980 °C for 1 hour, a first aging step at 720 °C for 8 hours, and a second aging step at 620 °C for 8 hours. For each manufacturing process, one sample was kept in the as-produced state (non-heat treated), one sample was solution-treated, one sample went through the solution treatment plus the first aging step, and one sample went through the whole cycle, including the solution treatment and two aging steps.

The non-treated cast sample showed a coarse microstructure, due to the low cooling rate, with large Laves, δ and carbide precipitates at interdendritic zones. In the AM samples, the characteristic columnar grain structure is seen. The columnar grains are typically oriented along the direction of greatest temperature gradient (the direction of greatest heat flow), which is the building direction (from the heat source to the platform or substrate). However, as seen both in LPBF and LDED samples, grains can deviate significantly from the building direction due to localized heat flow (which is maximum in a direction perpendicular to the melt pool boundary) and heat source motion.

The sample produced by LPBF displayed a very fine microstructure due to very high cooling rates, with columnar grains aligned close to the building direction, cellular dendritic subgrain structures and very fine Laves precipitates at intercellular zones. The LDED sample, with an intermediate cooling rate, showed a fine microstructure (but coarser than the one of the sample produced by LPBF) with columnar grains and cellular dendritic subgrain structures, and Laves phase in the interdendritic zones. As a direct consequence of grain size, the cast sample had the lowest hardness ($226 \pm 24 \text{ HV}_{0.5}$), and the LPBF sample had the highest value ($327 \pm 7 \text{ HV}_{0.5}$), with the LDED sample showing an intermediate number ($253 \pm 18 \text{ HV}_{0.5}$). Despite the high cooling rates, both LPBF and LDED showed signs of microsegregation of Nb and Mo (large and heavy atoms) in the interdendritic zones. Additionally, the LDED sample contains oxide particles due to the imperfect protection offered by the shielding gas (less effective than the controlled atmosphere of the LPBF process). The suboptimal microstructures obtained in the as-built state evidence the need for heat treatments.

With regards to the heat treated samples, it was observed that the solution treatment was not effective for the cast sample, as expected, since the precipitates were very coarse (coarser than in wrought parts), demanding more time and higher temperatures to solubilize and allow
its constituting elements to diffuse back into the matrix. For the AM samples, the solution treatment was expected to successfully promote the dissolution of Laves phase, since the precipitates are finer than those found in wrought IN718. However, the heat treatment was not completely successful, as Laves precipitates persisted. In all samples (cast, LPBF and LDED), the Laves precipitates appear to have been partially dissolved (to a very small extent), with most of the volume fraction remaining undissolved. By visual assessment of the micrographs, the LDED sample seems to be the one in which the partial solubilization is most evident. Microsegregation was not eliminated by the solution treatment, and recrystallization was not seen.

Nevertheless, the first aging step successfully promoted the precipitation of strengthening phases, as evidenced by significant hardness increases in all samples. Comparing the average hardness of the solution-treated-and-single-aged sample with the non-treated sample, the cast sample witnessed an increase of 89%, the LPBF saw an increase of 43%, and the LDED sample showed a rise of 80%. In this condition, the LDED sample reached 97% of the average hardness of the LPBF sample

After the full cycle, the microstructures saw no significant changes, and neither did the hardness. Thus, the second aging step provided no valuable contributions to hardness increase, which suggests that there was not sufficient Nb in the matrix to allow further precipitation of γ " in a second aging step, which is a direct consequence of the ineffectiveness of the solution treatment, failing to deliver a greater availability of Nb on the matrix to fuel optimal precipitation of the key strengthening phase. In terms of microstructure, the cast sample, and the two additively manufactured samples (both by LPBF and LDED) still displayed Laves phase in the fully treated condition, as well as δ particles and carbides. The strengthening phases are not observed due to their nanometric size, but their presence is evidenced by the increased hardness.

These results show that samples produced by different processes, with diverse starting microstructures, respond differently to the same heat treatment. It becomes then evident that the strategy of replicating the well-known standard heat treatment for wrought IN718 on additively manufactured parts is not fruitful, just as it is not effective to use a standard heat treatment designed for wrought parts on cast ones. This evidences the need of developing heat treatments specifically tailored to AM. In fact, even parts produced by different AM processes are affected differently by the heat treatment, as seen in this study for LDED and LPBF. Moreover, this study highlights the role of the fine microstructure in attaining satisfactory mechanical properties, as the LPBF samples, although responding most poorly to the heat treatment, still yielded the highest hardness.

Moving forward, the author suggests the employment of a higher temperature for the solution treatment for AM parts. A possible evolution of this work might test different (higher) solution treatment temperatures, and include TEM observations to try and identify the strengthening precipitates γ " and γ '. XRD analyses could be included to quantify the fraction of phases present, and DSC analysis could be employed in order to try and identify the temperatures at which the phase transformations occur, and this information could be used to design more suitable treatments specifically tailored for LPBF and LDED.

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APPENDIX A – Extended list of definitions

Below, there is a list of definitions, or a glossary, of terms relevant to the full comprehension of this study, as stated by the international standard ISO/ASTM 52900:2015(E) (Additive manufacturing – General principles – Terminology). The following list is a selection from the extensive list presented on ISO/ASTM 52900:2015(E) [1].

3D printer: machine used for *3D printing*.

3D printing: fabrication of objects through the deposition of a material using a print head, nozzle, or another printer technology. Term often used in a non-technical context synonymously with *additive manufacturing*. Until present times this term has in particular been associated with machines that are low end in price and/or overall capability.

accuracy: closeness of agreement between an individual result and an accepted reference value.

additive manufacturing (AM): process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies.

additive manufacturing system: machine and auxiliary equipment used for *additive* manufacturing.

additive manufacturing machine: section of the *additive manufacturing system* including hardware, machine control software, required set-up software and peripheral accessories necessary to complete a *build cycle* for producing parts.

as built: refers to the state of *parts* made by an additive process before any post processing, besides, if necessary, the removal from a *build platform* as well as the removal of support and/or processed *feedstock*.

build chamber: enclosed location within the *additive manufacturing system* where the *parts* are fabricated.

build cycle: single process cycle in which one or more components are built up in *layers* in the process chamber of the *additive manufacturing system*.

build envelope: largest external dimensions of the x-, y-, and z-axes within the *build space* where *parts* can be fabricated.

build platform: base which provides a surface upon which the building of the *part*, is started and supported throughout the build process.

build space: location where it is possible for *parts* to be fabricated, typically within the *build chamber* or on a *build platform*.

build surface: area where material is added, normally on the last deposited *layer* which becomes the foundation upon which the next layer is formed. For the first layer, the build surface is often the *build platform*. In the case of *directed energy deposition* processes, the build surface can be an existing part onto which material is added.

build volume: total usable volume available in the machine for building parts.

directed energy deposition: *additive manufacturing* process in which focused thermal energy is used to fuse materials by melting as they are being deposited.

feedstock: bulk raw material supplied to the *additive manufacturing* building process. Synonyms: source material, starting material, base material, original material.

fusion: act of uniting two or more units of material into a single unit of material.

front [of the machine]: side of the machine that the operator faces to access the user interface or primary viewing window, or both.

fully dense: state in which the material of the fabricated part is without significant content of voids. In practice, material completely free of voids is difficult to produce by any manufacturing process and some micro-porosity will generally be present.

layer: material laid out, or spread, to create a surface.

machine coordinate system: three-dimensional coordinate system as defined by a fixed point on the *build platform* with the three principal axes labelled x-, y-, and z-, with rotary axis about each of these axis labelled A, B, and C, respectively, where the angles between x-, yand z- can be Cartesian or defined by the machine manufacturer. Machine coordinate system is fixed relative to the machine, as opposed to coordinate systems associated with the *build surface*, which can be translated or rotated. Machine coordinate system is illustrated in ISO/ASTM 52921 [2].

multi-step process: type of additive manufacturing process in which parts are fabricated in two or more operations where the first typically provides the basic geometric shape and the following consolidates the part to the fundamental properties of the intended material. Removal of support structures is not considered an additional step.

near net shape: condition where the components require little *post-processing* to meet dimensional tolerance.

origin: zero point (0, 0, 0) (when using x-, y-, and z-coordinates). Designated universal reference point at which the three primary axes in a coordinate system intersect. Coordinate system can be Cartesian or as defined by the machine manufacturer. The concept of origin is illustrated in ISO/ASTM 52921 [2].

part: joined material forming a functional element that could constitute all or a section of an intended product.

porosity: presence of small voids in a *part* making it less than *fully dense*.

post-processing: process steps taken after the completion of an *additive manufacturing build cycle* in order to achieve the desired properties in the final product.

powder bed: part bed build area in an *additive manufacturing system* in which *feedstock* is deposited and selectively fused by means of a heat source or bonded by means of an adhesive to build up *parts*.

powder bed fusion: *additive manufacturing* process in which thermal energy selectively fuses regions of a *powder bed*.

process parameters: set of operating parameters and system settings used during a *build* cycle.

prototype: physical representation of all or a component of a product that, although limited in some way, can be used for analysis, design and evaluation.

rapid prototyping: application of *additive manufacturing* intended for reducing the time needed for producing *prototypes*. Historically, rapid prototyping (RP) was the first commercially significant application for additive manufacturing, and have therefore been commonly used as a general term for this type of technology.

single step process: type of additive manufacturing process in which parts are fabricated in a single operation where the basic geometric shape and basic material properties of the intended product are achieved simultaneously. Removal of support structures is not considered an additional step.

STL: file format for model data describing the surface geometry of an object as a tessellation of triangles used to communicate 3D geometries to machines in order to build physical *parts*.

x-axis: axis in the *machine coordinate system* that runs parallel to the *front* of the machine and perpendicular to the *y*-axis and *z*-axis. The positive *x*-direction runs from left to right as viewed from the front of the machine while facing toward the *build volume origin*. It is common that the *x*-axis is horizontal and parallel with one of the edges of the *build platform*.

y-axis: axis in the *machine coordinate system* that runs perpendicular to the *z-axis* and *x-axis*. The positive direction is defined in ISO 841 to make a right hand set of coordinates. In the most common case of an upwards *z*-positive direction, the positive *y*-direction will then run from the front to the back of the machine as viewed from the front of the machine. It is common that the *y*-axis is horizontal and parallel with one of the edges of the *build platform* [3].

z-axis: axis in the *machine coordinate system* that run perpendicular to the *x*-axis and *y*-axis. The positive direction is defined in ISO 841 to make a right hand set of coordinates. For processes employing planar, layerwise addition of material, the positive *z*-direction will then run normal to the *layers*. For processes employing planar layerwise addition of material, the positive *z*-direction, is the direction from the first layer to the subsequent layers. Where addition of material is possible from multiple directions (such as with certain *directed energy deposition* systems), the *z*- axis may be identified according to the principles in ISO 841 [3].

APPENDIX B – Heat treatments, microstructures and hardness of Inconel 718 produced by LPBF (literature compilation)

Table B.1 – Compilation of data from literature regarding the effect of heat treatments on the microstructure and hardness of IN718 samples produced by LPBF. Data for wrought and cast samples are also included for comparison purposes. W=Wrought; IC=Investment casting; N/A=Not available.

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Amato et al. [43]	LPBF	None (as-built state)	Columnar grains aligned in the build direction with cellular subgrain structures. Lenticular nanometric γ " precipitates (250 nm)	387.5 HV (0.025kgf)
Amato et al. [43]	LPBF	Annealing at 1160 °C for 4 hours	Mixture of columnar grains with cellular substructures, and recrystallized equiaxed grains. Ellipsoidal γ " (35 nm), spheroidal γ " or γ ', and δ at grain boundaries	458.9 HV (0.025kgf)
Tucho et al. [72]	LPBF	None (as-built state)	Elongated grains with columnar and cellular substructures (400 - 900 nm). Laves phase (Cr,Fe,Ni)2(Nb, Mo,Ti) (10 - 300 nm), TiC, TiN and Al2O3 in the interdendritic zones.	304 HV (10 kgf)
Tucho et al. [72]	LPBF	Solution treatment at 1100 °C for 1 hour, water quenching	Partial recrystallization, grain coarsening and partial dissolution of seconadry phases. Equiaxed recrystallized grains with no substructures mixed with elongated grains with columnar and cellular substructures. MC carbides, Al2O3 and Laves (< 100μ m).	258 HV (10 kgf)
Tucho et al. [72]	LPBF	Solution treatment at 1100 °C for 7 hours, water quenching	Partial recrystallization, grain coarsening and partial dissolution of seconadry phases. Mostly coarser equiaxed recrystallized grains with no substructures. Twin boundaries. MC carbides (1 - 6 µm), Al2O3 and Laves.	217 HV (5 kgf)
Tucho et al. [72]	LPBF	Solution treatment at 1250 °C for 1 hour, water quenching	Equiaxed recrystallized grains with no substructures. MC carbides (150 - 1000 nm), Al2O3 and Laves phase (<100 μ m)	210 HV (5 kgf)
Tucho et al. [72]	LPBF	Solution treatment at 1250 °C for 7 hours, water quenching	Coarse equiaxed recrystallized grains with no substructures. MC carbides (150 - 10000 nm) and Al2O3 (< 100 μ m). Laves phase was dissolved	207 HV (5 kgf)
Chlebus et al. [71]	LPBF	None (as-built state)	Columnar grains aligned in the building direction, with cellular subgrain structures. Laves phase and MC carbides (NbC and TiC) at intercellular regions	312 HV (1.0 kgf)
Chlebus et al. [71]	LPBF	Double ageing first at 720 °C for 8 hours, then furnace cooling to 620 °C at 100 °C/h and holding for 10 hours, then air cooling	Columnar grains aligned in the building direction, with cellular subgrain structures. Laves phase, MC carbides and δ at intercellular regions, and γ " and γ " at grain boundaries and intercellular regions	461 HV (1.0 kgf)
Chlebus et al.[71]	LPBF	Solution treatment at 980 °C for 1 hour, water quenching, then double ageing first at 720 °C for 8 hours, then furnace cooling to 620 °C at 100 °C/h and holding for 10 hours, then air cooling	Columnar grains aligned in the building direction, with cellular subgrain structures. Laves phase, MC carbides and δ at intercellular regions, and γ'' at grain boundaries and intercellular regions	465 HV (1.0 kgf)

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Chlebus et al.[71]	LPBF	Solution treatment at 1040 °C for 1 hour, water quenching, then double ageing first at 720 °C for 8 hours, then furnace cooling to 620 °C at 100 °C/h and holding for 10 hours, then air cooling	Mixture of columnar grains with cellular subgrain structures and equiaxed recrystallized grains. Laves phase, MC carbides and δ at intercellular regions, and γ'' and γ' at grain boundaries and intercellular regions	474 HV (1.0 kgf)
Chlebus et al.[71]	LPBF	Solution treatment at 1100 °C for 1 hour, water quenching, then double ageing first at 720 °C for 8 hours, then furnace cooling to 620 °C at 100 °C/h and holding for 10 hours, then air cooling	Mostly equiaxed recrystallized grains. Cellular dendrites were eliminated, as well as Laves and δ . MC carbides at grain boundaries and γ " and γ '	463 HV (1.0 kgf)
Jiang et al. [77]	LPBF	None (as-built state)	Fine columnar grains aligned in the building direction, with cellular subgrain structures. MC carbides ((Nb, Ti)C) at intercellular regions	309.7 HV (0.5 kgf)
Jiang et al. [77]	LPBF	Annealing at 1120 °C for 4 hours (Ar atmosphere), water quenching	Coarser equiaxed (recrystallized) grains with twin boundaries mixed with coarser columnar grains. MC carbides at grain boundaries	209.7 HV (0.5 kgf)
Jiang et al. [77]	LPBF	Double aging, first at 720 °C for 8 hours, then furnace cooling at 55 °C/h to 620 °C, holding for 8 hours and air cooling (Ar atmosphere)	Fine columnar grains aligned in the building direction, with cellular subgrain structures. MC carbides ((Nb, Ti)C) at intercellular regions and nanometric γ " and γ '	465.5 HV (0.5 kgf)
Jiang et al. [77]	LPBF	Annealing at 1120 °C for 4 hours, water quenching, plus double aging, first at 720 °C for 8 hours, then furnace cooling at 55 °C/h to 620 °C, holding for 8 hours and air cooling (Ar atmosphere)	Coarser equiaxed (recrystallized) grains with twin boundaries mixed with coarser columnar grains. MC carbides at grain boundaries and nanometric γ " and γ '	434.9 HV (0.5 kgf)
Deng et al. [69]	LPBF	None (as-built state)	Columnar grains with cellular substructures (200 - 500 nm). Laves precipitates in the intercellular zone	325 HV (0.3 kgf)
Deng et al. [69]	LPBF	Double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 8 hours and air cooling	Columnar grains with cellular substructures. Laves precipitates in the intercellular zone. γ " and γ ' (speculation based on literature and mechanical properties)	499 HV (0.3 kgf)
Deng et al. [69]	LPBF	Solution treatment at 980 °C for 1 hour with water quenching + double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 8 hours and air cooling	Columnar grains. Laves (partially dissolved) at grain boundaries and acicular δ (1-2 µm) at grain interior and boundaries. γ " and γ ' (speculation based on literature and mechanical properties)	490 HV (0.3 kgf)
Deng et al. [69]	LPBF	Homogenization at 1080 °C for 1 hour with water quenching + double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 8 hours and air cooling	Coarser grains (substructures were eliminated), Laves phase (partially dissolved). γ " and γ ' (speculation based on literature and mechanical properties)	499 HV (0.3 kgf)
Deng et al. [69]	LPBF	Homogenization at 1080 °C for 1 hour with water quenching + solution treatment at 980 °C for 1 hour with water quenching + double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 8 hours and air cooling	Coarser grains (substructures were eliminated), acicular δ precipitates mostly at grain boundaries (Laves phase was completely dissolved). γ " and γ ' (speculation based on literature and mechanical properties)	490 HV (0.3 kgf)

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Gallmeyer et al. [68]	LPBF	None (as-built state)	Columnar grains aligned with the build direction, with cellular subgrain structures (620 nm). Nanometric (4 nm) Laves phase, MC and Al2O3 at intercellular zones	N/A
Gallmeyer et al. [68]	LPBF	Solution treatment at 980 °C for 1 hour, water quenching	Columnar grains aligned with the build direction, with cellular subgrain structures (650 nm). MC, Al2O3 and acicular δ (500 nm) at intercellular zones (Laves was dissolved)	N/A
Gallmeyer et al. [68]	LPBF	Direct aging at 620 °C for 24 hours, air cooling	Columnar grains aligned with the build direction, with cellular subgrain structures (640 nm). Laves phase (234 nm), MC and Al2O3 at intercellular zones, and γ " (8 nm) in cell interior	N/A
Gallmeyer et al. [68]	LPBF	Direct aging at 720 °C for 24 hours, air cooling	Columnar grains aligned with the build direction, with cellular subgrain structures (620 nm). Laves phase (240 nm), MC, Al2O3 and γ'' (210 nm) at intercellular zones, and γ'' (31 nm), γ' (21 nm) and γ''/γ' (15 nm) in cell interior	N/A
Gallmeyer et al. [68]	LPBF	Solution treatment at 1020 °C for 15 minutes, water quenching, and aging at 720 °C for 24 hours, air cooling	Columnar grains aligned with the build direction, with cellular subgrain structures (610 nm). MC and Al2O3 at intercellular zones (Laves and δ were dissolved). Lenticular γ'' (29 nm) and spheroidal γ' (23 nm) and γ''/γ' coprecipitates (18 nm) homogeneously distributed	N/A
Gallmeyer et al. [68]	LPBF	Solution treatment at 980 °C for 1 hour, air cooling, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 2 hours, and holding at 620 °C for 8 hours, finishing with air cooling	Columnar grains aligned with the build direction, with cellular subgrain structures (610 nm). MC, Al2O3 and acicular δ (700 nm) at intercellular zones (Laves was dissolved). Lenticular γ " (27 nm) and spheroidal γ' (18 nm) and γ''/γ' coprecipitates (16 nm) in cell interior	N/A
Farber et al. [67]	LPBF	None (as-built state)	Columnar grains (100 - 300 μ m) aligned in the build direction with cellular subgrain structures. γ " and γ ' nanometric precipitates at interdendritic zones	295 HV (1 kgf)
Farber et al. [67].	LPBF	Solution treatment at 980 °C for 4 hours, air cooling. Double Aging at 720 °C for 8 hours then furnace cooling to 620 °C and holding for 8 hours, air cooling to room temperature	Columnar grains (100 - 300 μ m) aligned in the build direction with cellular subgrain structures. Acicular δ , γ " and γ ' nanometric precipitates at interdendritic zones, and Laves phase at grain boundaries	416 HV (1 kgf)
Zhao et al. [58]	LPBF	None (as-built state)	Columnar grains with columnar dendrites and cellular strubstructures. Laves phase at grain boundaries and interdenderitic zones	N/A
Zhao et al. [58]	LPBF	Homogenization at 1180 °C for 20 min, water quenching	Coarse equiaxed grains (recrystallized), NbC at grain boundaries. Laves phase was dissolved and columnar and subgrain structures were eliminated	N/A
Zhao et al. [58]	LPBF	Homogenization at 1180 °C for 1 h, water quenching	Coarse equiaxed grains (recrystallized), coarser NbC at grain boundaries.	N/A
Zhao et al. [58]	LPBF	Homogenization at 1180 °C for 12 h, water quenching	Coarse equiaxed grains (recrystallized), even coarser NbC at grain boundaries.	N/A
Schneider et al. [73]	LPBF	None (as-built state)	Columnar microstructure	N/A

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Schneider et al. [73]	LPBF	Ageing at 720 °C for 8 hours, air cooling	Columnar microstructure	N/A
Schneider et al. [73]	LPBF	Double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 10 hours and air cooling	Columnar microstructure	N/A
Schneider et al. [73]	LPBF	Solution treatment at 1010 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 10 hours and air cooling	Columnar microstructure	N/A
Schneider et al. [73]	LPBF	Stress relieving at 1066 °C for 1.5 hours, argon quenching	Equiaxed (recrystallized) microstructure	N/A
Schneider et al. [73]	LPBF	Stress relieving at 1066 °C for 1.5 hours, argon quenching, plus ageing at 720 °C for 8 hours, air cooling	Equiaxed (recrystallized) microstructure	N/A
Schneider et al. [73]	LPBF	Stress relieving at 1066 °C for 1.5 hours, argon quenching, plus double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 10 hours and air cooling	Equiaxed (recrystallized) microstructure	N/A
Schneider et al. [73]	LPBF	Stress relieving at 1066 °C for 1.5 hours, argon quenching, plus solution treatment at 1010 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 10 hours and air cooling	Equiaxed (recrystallized) microstructure	N/A
Cao et al. [57]	LPBF	Solution treatment at 1065 °C for 1 hour, air cooling, plus double aging first at 760 °C for 10 hours, furnace cooling for 2 hours to 650 °C, holding for 8 hours, then air cooling to room temperature	Cellular microstructure with δ platelets (300 - 1000 nm) at grain interiors and boundaries, disc-like (20 - 50 nm) γ " at cell interior, acicular γ " at grain boundaries and interior, and round γ ' (20 nm).	N/A
Ferreri et al. [24]	LPBF	Solution treatment at 954 °C for 1 hour, Ar fan cooling to 120 °C, plus double aging first at 718 °C for 8 hours, furnace cooling at 50 °C/h to 621 °C, holding for 8 hours, then air cooling to room temperature	Columnar grains (with no subgrain structures). Submicron δ and MC carbides and nanometric γ ' and γ ".	N/A
Jinoop et al. [45]	LPBF	None (as-built state)	N/A	319 HV (0.2 kgf
Jinoop et al. [45]	LPBF	Solution treatment at 950 °C for 1 hour, water quenching	N/A	307 HV (0.2 kgf
Jinoop et al.[45]	LPBF	Solution treatment at 1050 °C for 1 hour, water quenching	N/A	260 HV (0.2 kgf
Newell et al.[74]	LPBF	None (as-built state)	Large columnar grains aligned in the build direction.	276.2 HV (0.02 kgf

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Newell et al. [74]	LPBF	Solution treatment at 1160 °C for 1 hour, water quenching	Columnar grains mixed with recrystallized equiaxed grains	280.5 HV (0.02 kgf)
Newell et al. [74]	LPBF	Solution treatment at 1160 °C for 2 hours, water quenching	Columnar grains mixed with coarser recrystallized equiaxed grains	254.9 HV (0.02 kgf)
Newell et al. [74]	LPBF	Solution treatment at 1160 °C for 4 hours, water quenching	Coarse equiaxed recrystallized grains with annealing twins	240.1 HV (0.02 kgf)
Newell et al. [74]	LPBF	Solution treatment at 1160 °C for 8 hours, water quenching	Coarser equiaxed recrystallized grains with annealing twins	243.5 HV (0.02 kgf)
Zhang et al. [75]	LPBF	None (as-built state)	Columnar grains (average width of 45 μ m and average length of 255 μ m) oriented along the build direction with fine columnar dendritic substructures (primary arm spacing of 698 nm)	N/A
Zhang et al. [75]	LPBF	Homogenization at 1080 °C for 1.5 hours, air cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C at 55 °C/h and holding for 8 hours, then air cooling	Recrystallized equiaxed grains with carbides at grain boundaries. Laves phase was fully dissolved. γ " and γ ' were not seen due to very fine size	440 HV (45 HRC)
Zhang et al. [75]	LPBF	Homogenization at 1080 °C for 1.5 hours, air cooling to room temperature, then solution treatment at 980 °C for for 1 hour, air cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C at 55 °C/h and holding for 8 hours, then air cooling	Recrystallized equiaxed grains, with δ platelet-like precipitates (1-5 µm long) at grain boundaries, as well as some carbides. Laves phase was fully dissolved. γ " and γ ' were not seen due to very fine size	410 HV (42.5 HRC)
Deng et al. [69]	W	Solution treatment at 980 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 2 hours, and holding at 620 °C for 8 hours, finishing with air cooling	N/A (traditionally equiaxed grains with carbides, $\gamma^{\prime\prime}$ and γ^{\prime}	350 HV (1 kgf)
Liu et al. [86]	W	Solution treatment at 980 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 2 hours, and holding at 620 °C for 8 hours, finishing with air cooling	N/A (traditionally equiaxed grains with carbides, $\gamma^{\prime\prime}$ and γ^{\prime}	372 HV (1 kgf)
AMS 5662 [50]	W	Solution treatment at 980 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 2 hours, and holding at 620 °C for 8 hours, finishing with air cooling	N/A (traditionally equiaxed grains with carbides, $\gamma^{\prime\prime}$ and γ^{\prime}	326 HV (34 HRC)
Zhang et al. [75]	IC	Homogenization at 1080 °C for 1.5 hours, air cooling to room temperature, then solution treatment at 980 °C for for 1 hour, air cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C at 55 °C/h and holding for 8 hours, then air cooling	Coarse equiaxed dendritic grains with well developed secondary arms (average grain size of 1300 μ m, average secondary dendrite arm spacing of 45 μ m). Laves phase at interdendritic zones, NbC and δ . Strengthening precipitates not observed due to small size	280 HV (28 HRC)

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Soffel et al. [76]	IC	Annealing at 1100 °C for 2 hours	Coarse equiaxed dendritic grains with well developed secondary arms (average grain size of 1584 μ m, primary dendrite arm spacing of about 100-130 μ m)	218 HV (3 kgf)

Source: Elaborated by the author with data from [24, 43, 45, 50, 57, 58, 67-69, 71-77, 86].

APPENDIX C – Heat treatments, microstructures and hardness of Inconel 718 produced by LDED (literature compilation)

Table C.1– Compilation of literature data regarding the effects of different heat treatments on the microstructure and hardness of IN718 samples produced by LDED. Data for cast and wrought samples are also included for comparison purposes. W=Wrought; IC=Investment casting; N/A=not available.

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Jinoop et al. [45]	LDED	None (as-built state)	Fine columnar dendritic microstructure. Phases not assessed	234 HV (0.2 kgf)
Jinoop et al. [45]	LDED	Solution treatment at 950 °C for 1 hour, water quenching	Coarse recrystallized equiaxed grains. Phases not assessed	220 HV (0.2 kgf)
Jinoop et al. [45]	LDED	Solution treatment at 1050 °C for 1 hour, water quenching	Coarse recrystallized equiaxed grains. Phases not assessed	206 HV (0.2 kgf)
Bambach et al.[89]	LDED	None (as-built state)	Columnar grains aligned in the build direction, with columnar dendritic substructures. Laves, MC and TiN at interdendritic zones	N/A
Bambach et al. [89]	LDED	Homogenization at 1065 °C for 1 hour	Partial recrystallization, mostly columnar grains with no subgrain structure. Laves phase was mostly dissolved	N/A
Xiao et al. [78]	LDED	None (as-built state)	Columnar grains with columnar dendritic substructures tilted in the scanning direction (primary dendrite arm spacing: 8-15 μ m). Continuous Laves precipitates (11.6 %vol.) at interdendritic zones.	255.4 HV (0.5 kgf)
Xiao et al. [78]	LDED	Double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C, holding for 8 hours and air cooling	Columnar grains with columnar dendritic substructures tilted in the scanning direction. Continuous Laves precipitates at interdendritic zones and fine γ " (speculation).	390.3 HV (0.5 kgf)
Sui et al. [85]	LDED	None (as-built state)	Columnar epitaxial grains (50-300 µm) oriented along the build direction. Continuous elongated Laves precipitates at interdendritic zones	N/A
Sui et al. [85]	LDED	Direct ageing at 720 °C for 8 hours, then furnace cooling at 50 °C/h to 620 °C, holding for 8 hours and finishing with air cooling	Columnar epitaxial grains oriented along the build direction. Continuous elongated Laves precipitates (3.83 %vol.), fine γ' (5.13 %vol.) and disc-like γ'' (13.9 %vol., 64.6 nm) at interdendritic zones	N/A
Sui et al. [85]	LDED	Solution treatment at 1050 °C for 15 minutes, water quenching, then ageing at 720 °C for 8 hours and furnace cooling at 50 °C/h to 620 °C and holding for 8 hours then air cooling	Columnar epitaxial grains oriented along the build direction. Granular Laves precipitates (1.55 %vol.) at interdendritic zones, and fine γ' (5.31 %vol.) and disc-like γ'' (16.2 %vol., 33.8 nm) uniformly dispersed	N/A
Sui et al. [85]	LDED	Solution treatment at 1050 °C for 45 minutes, water quenching, then ageing at 720 °C for 8 hours and furnace cooling at 50 °C/h to 620 °C and holding for 8 hours then air cooling	Columnar epitaxial grains oriented along the build direction. Fine granular Laves precipitates (0.82 %vol.) at interdendritic zones and fine γ' (5.37 %vol.) and disc-like γ'' (16.9 %vol., 34.8 nm) uniformly dispersed	N/A

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Zhai et al. [90]	LDED	None (as-built state)	Predominantly columnar grains with columnar dendritic substructures (1-3 μ m), mixed with fine equiaxed grains at interlayer zones. Laves phase at interdendritic zones and γ' .	285.3 HV (1 kgf)
Zhai et al. [90]	LDED	Ageing at 732 °C for 4 hours, furnace cooling	Predominantly columnar grains with columnar dendritic substructures, mixed with fine equiaxed grains at interlayer zones. Laves phase at interdendritic zones and γ' (predominantly) and γ''	403.6 HV (1 kgf)
Zhai et al. [90]	LDED	Double ageing, first at 720 °C for 8 hours, then furnace cooling to 620 °C and holding for 10 hours, then air cooling	Predominantly columnar grains with columnar dendritic substructures, mixed with fine equiaxed grains at interlayer zones. Laves phase at interdendritic zones, and γ " (predominantly) and γ '	419.5 HV (1 kgf)
Zhai et al. [90]	LDED	Homogenization at 1093 °C for 1 hour, air cooling to room temperature, then solution treatment at 960 °C for for 1.5 hours, air cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C and holding for 10 hours, then air cooling	Refinement of columnar grains and coarsening of equiaxed grains. Laves phase was partially dissolved. δ at grain boundaries, and γ " and γ '	435.8 HV (1 kgf)
Glerum et al. [91]	LDED	None (as-built state)	Columnar dendrites. Laves phase at interdendritic zones	240 HV (0.5 kgf)
Glerum et al[91]	LDED	Double ageing, first at 720 °C for 8 hours, then furnace cooling to 620 °C and holding for 10 hours, then air cooling	Columnar dendrites. Laves, δ , γ " and γ ' (speculations).	420 HV (0.5 kgf)
Zhang et al. [84]	LDED	None (as-built state)	Columnar grains aligned in the build direction, with columnar dendritic substructures. Irregular Laves (4.1 %vol.) precipitates at interdendritic zones, fine globular Ti/Nb carbonitrides ((Ti,Nb)(C,N))	N/A
Zhang et al. [84]	LDED	Double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 0.5 hour, then holding at 620 °C for 8 hours, and air cooling	Columnar grains aligned in the build direction, with columnar dendritic substructures. Irregular Laves (4.3 %vol.) precipitates and nanometric γ " (11.3 %vol.) and γ ' at interdendritic zones	N/A
Zhang et al. [84]	LDED	Solution treatment at 1100 °C for 1 hour, followed by double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 0.5 hour, then holding at 620 °C for 8 hours, and air cooling	Coarser equiaxed recrystallized grains with annealing twins. Acicular δ at grain boundaries, plus nanometric γ " (18.6 %vol.) and γ '. Laves phase was dissolved	N/A
Yu et al. [82]	LDED	None (as-built state)	Columnar grains (20 - 1000 μ m) aligned in the build direction, with columnar dendritic substructures (primary dendrite arm spacing of 5-10 μ m). Continuous Laves phase and NbC at interdendritic zones.	N/A
Yu et al. [82]	LDED	Ageing at 720 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling	Columnar grains aligned in the build direction, with columnar dendritic substructures. Continuous Laves phase, NbC, and nanometric (10-50 nm) γ " and γ ' at interdendritic zones	N/A

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Yu et al. [82]	LDED	Solution treatment at 980 °C for 1 hour, water quenching, ageing at 720 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling	Columnar grains aligned in the build direction, with columnar dendritic substructures. Granular Laves, NbC, nanometric (10-50 nm) γ " and γ ', and acicular δ (1-3 μ m) at interdendritic zones	N/A
Yu et al. [82]	LDED	Homogenization at 1100 °C for 1.5 hours, water quenching, solution treatment at 980 °C for 1 hour, water quenching, ageing at 720 °C for 8 hours, furnace cooling at 50 °C/h to 620 °C, holding for 8 hours, air cooling	Recrystallized equiaxed grains (10 - 500 μ m), with twin boundaries and no subgrain structures. Acicular δ mostly at grain boundaries, and γ ' and γ " particles (30 nm) dispersed in the matrix. Laves phase is dissolved.	N/A
Zhu et al. [92]	LDED	None (as-built state)	Columnar dendrites growing epitaxially along the build direction, and a few equiaxed dendrites. Laves phase and TiC and NbC at interdendritic zones	N/A
Zhu et al. [92]	LDED	Solution treatment at 1100 °C for 1 hour with air cooling	Coarse equiaxed recrystallized grains. Spheroidal TiC and NbC (Laves phase was dissolved)	239 HV (Load N/A)
Zhu et al. [92]	LDED	Homogenization at 1100 °C for 1.5 hours with air cooling + solution treatment at 980 °C for 1 hour with air cooling + double aging, first at 720 °C for 8 hours, then furnace cooling at 55 °C/h to 620 °C, holding for 8 hours and air cooling	Equiaxed grains with fine acicular δ at grain boundaries, spherical γ' (15 nm) and lenticular γ'' (20 nm). Laves phase was not present	496 HV (Load N/A)
Careri et al. [88]	LDED	None (as-built state)	Columnar dendritic microstructure with Laves phase at interdendritic zones	260 HV (0.025 kgf)
Careri et al. [88]	LDED	Double aging, first at 718 °C for 8 hours, then furnace cooling to 621 °C and holding for 8 hours, then air cooling	Columnar dendritic microstructure with Laves phase at interdendritic zones and nanometric γ " and γ ' (speculation).	428 HV (0.025 kgf)
Careri et al. [88]	LDED	Homogenization at 1093 °C for 2 hours, air cooling to room temperature, then solution treatment at 968 °C for for 1 hour, air cooling to room temperature, then double aging, first at 718 °C for 8 hours, then furnace cooling to 621 °C and holding for 8 hours, then air cooling	Equiaxed grains with nanometric γ" and γ' (speculation). Laves phase was mostly dissolved.	430 HV (0.025 kgf)
Xiao et al. [80]	LDED	None (as-built state)	Columnar grains aligned in the build direction, with columnar dendritic substructures (primary dendrite arm spacing: 5-8 μ m). Continuous Laves (6.8 %vol) at interdendritic zones	263.3 HV (0.5 kgf)
Xiao et al. [80]	LDED	Solution treatment at 980 °C for 1 hour with air cooling + double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C, holding for 8 hours and air cooling	Columnar grains aligned in the build direction, with columnar dendritic substructures. Laves phase was partially dissolved and acicular δ precipitated at interdendritic zones, as well as nanometric γ "	413.6 HV (0.5 kgf)
Wang et al. [79]	LDED	None (as-built state)	Coarse columnar grains aligned in the build direction but tilted towards the scanning direction, with columnar dendrites (primary dendrite arm spacing of 5-10 μ m). Laves phase at interdendritic zones.	N/A

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Wang et al. [79]	LDED	Solution treatment at 1020 °C for 1 hour, followed by double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C, then holding at 620 °C for 8 hours, and air cooling	Coarse columnar grains aligned in the build direction but tilted towards the scanning direction, with columnar dendrites. Laves phase at interdendritic zones. Plate-like γ " and γ ' (< 100 µm).	N/A
Wang et al. [79]	LDED	Solution treatment at 1100 °C for 1 hour, followed by double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C, then holding at 620 °C for 8 hours, and air cooling	Fine, partially recrystallized equiaxed grains with twin boundaries, and remnant columnar dendritic grains. Plate-like γ " and γ' (< 100 µm). Laves phase was partially dissolved.	N/A
Wang et al. [79]	LDED	Solution treatment at 1180 °C for 1 hour, followed by double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C, then holding at 620 °C for 8 hours, and air cooling	Coarse (250 μ m), fully recrystalized equiaxed grains with no subgrain structures. Plate-like γ " and γ ' (< 100 μ m). Laves phase fully dissolved.	N/A
Kong et al.[93]	LDED	None (as-built state)	Columnar grains. Irregular Laves precipitates (24% wt) and Al/Ti oxides	240 HV (0.1 kgf)
Kong et al. [93]	LDED	Homogenization at 1095 °C for 1 hour, air cooling to room temperature, then solution treatment at 980 °C for for 1 hour, air cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C at 55 °C/h and holding for 8 hours, then air cooling	Columnar grains mixed with equiaxed grains. Al/Ti oxides, acicular δ precipitates, disc-like γ " (25 nm), and γ '.	475 HV (0.1 kgf)
Liu et al. [87]	LDED	None (as-built state)	Columnar dendritic microstructure. Continuous elongated Laves precipitates (10 μ m, 5.21 %vol.) at interdendritic zones	301 HV (0.2 kgf)
Liu et al. [87]	LDED	Solution treatment at 1020 °C for 30 min, water quenching	Columnar dendritic microstructure. Partially dissolved coarse Laves precipitates (4.5 %vol.)	240 HV (0.2 kgf)
Liu et al. [87]	LDED	Solution treatment at 1020 °C for 60 min, water quenching	Columnar dendritic microstructure. Partially dissolved Laves precipitates	205 HV (0.2 kgf)
Liu et al. [87]	LDED	Solution treatment at 1020 °C for 120 min, water quenching	Columnar dendritic microstructure. Partially dissolved Laves precipitates	200 HV (0.2 kgf)
Liu et al. [87]	LDED	Solution treatment at 1020 °C for 240 min, water quenching	Columnar dendritic microstructure. Partially dissolved granular Laves precipitates (1.77 %vol.)	198 HV (0.2 kgf)
Liu et al. [87]	LDED	Solution treatment at 1020 °C for 60 min, water quenching, then double ageing first at 720 °C for 8 hours, then furnace cooling to 620 °C and holding for 8 hours, then air cooling	Columnar dendritic microstructure. Remnant Laves and γ "	N/A
Liu et al. [87]	LDED	δ ageing at 890 °C for 12 hours, water quenching	Columnar dendritic microstructure. Laves precipitates (4.48 %vol.) intertwined with acicular δ precipitates (33 %vol.)	291 HV (0.2 kgf)
Liu et al. [87]	LDED	δ ageing at 890 °C for 12 hours, water quenching, solution treatment at 1020 °C for 30 min, water quenching	Columnar dendritic microstructure. Laves precipitates (2.25 %vol.) intertwined with acicular δ precipitates	239 HV (0.2 kgf)

Author	Pro- cess	Treatment	Microstructure	Hardness (Vickers)
Liu et al. [87]	LDED	δ ageing at 890 °C for 12 hours, water quenching, solution treatment at 1020 °C for 60 min, water quenching	Columnar dendritic microstructure. Partially dissolved fine granular Laves precipitates	234 HV (0.2 kgf)
Liu et al. [87]	LDED	δ ageing at 890 °C for 12 hours, water quenching, solution treatment at 1020 °C for 120 min, water quenching	Columnar dendritic microstructure. Partially dissolved fine granular Laves precipitates	212 HV (0.2 kgf)
Liu et al. [87]	LDED	δ ageing at 890 °C for 12 hours, water quenching, solution treatment at 1020 °C for 240 min, water quenching	Columnar dendritic microstructure. Partially dissolved fine granular Laves precipitates (0.98 %vol.)	204 HV (0.2 kgf)
Liu et al. [87]	LDED	δ ageing at 890 °C for 12 hours, water quenching, double ageing first at 720 °C for 8 hours, then furnace cooling to 620 °C and holding for 8 hours, then air cooling	Columnar dendritic microstructure. Laves precipitates intertwined with acicular δ precipitates, and γ "	N/A
Liu et al. [87]	LDED	δ ageing at 890 °C for 12 hours, water quenching, solution treatment at 1020 °C for 60 min, water quenching, then double ageing first at 720 °C for 8 hours, then furnace cooling to 620 °C and holding for 8 hours, then air cooling	Columnar dendritic microstructure. Remnant Laves and γ"	N/A
Deng et al.[69]	W	Solution treatment at 980 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 2 hours, and holding at 620 °C for 8 hours, finishing with air cooling	N/A (traditionally equiaxed grains with carbides, $\gamma^{\prime\prime}$ and γ^{\prime}	350 HV (1 kgf)
Liu et al. [86]	W	Solution treatment at 980 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 2 hours, and holding at 620 °C for 8 hours, finishing with air cooling	N/A (traditionally equiaxed grains with carbides, $\gamma^{\prime\prime}$ and γ^{\prime}	372 HV (1 kgf)
AMS 5662 [50]	W	Solution treatment at 980 °C for 1 hour, water quenching, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C in 2 hours, and holding at 620 °C for 8 hours, finishing with air cooling	N/A (traditionally equiaxed grains with carbides, $\gamma^{\prime\prime}$ and γ^{\prime}	326 HV (34 HRC)
Zhang et al.[75]	IC	Homogenization at 1080 °C for 1.5 hours, air cooling to room temperature, then solution treatment at 980 °C for for 1 hour, air cooling to room temperature, then double aging, first at 720 °C for 8 hours, then furnace cooling to 620 °C at 55 °C/h and holding for 8 hours, then air cooling	Coarse equiaxed dendritic grains with well developed secondary arms (average grain size of 1300 μ m, average secondary dendrite arm spacing of 45 μ m). Laves phase at interdendritic zones, NbC and δ . Strengthening precipitates not observed due to small size	280 HV (28 HRC)
Soffel et al.[76]	IC	Annealing at 1100 °C for 2 hours	Coarse equiaxed dendritic grains with well developed secondary arms (average grain size of 1584 µm, primary dendrite arm spacing of about 100-130 µm)	218 HV (3 kgf)

Source: Elaborated by the author based on data from [45, 50, 69, 75, 76, 78-80, 82, 84-93]

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