# POLITECNICO DI TORINO

College of Computer Engineering, Cinema and Mechatronics Master of science program in Mechatronic Engineering

Master's degree thesis

# Characterization of steel components obtained by means of new print heads for Direct Energy Deposition (DED)



Tutor

Prof. Alberta Aversa

Candidate

Firas Kassem

December 2022

# 1 Table of Contents

2		Abstract						
3	Introduction							
	3.	1	Addi	tive Manufacturing Overview	.9			
		3.1.2	1	Vat Photopolymerization	.9			
		3.1.2	2	Material Jetting	10			
		3.1.3	3	Binder jetting	11			
		3.1.4	4	Material extrusion	11			
		3.1.5	5	Powder Bed Fusion	12			
		3.1.6	5	Sheet Lamination	13			
		3.1.7	7	Directed Energy Deposition	13			
		3.1.8	3	DED Solidification phenomena	17			
		3.1.9		Applications	19			
		3.1.2	10	Advantages and Disadvantages of DED	19			
	3.	2	Stee	ls	20			
		3.2.2	1	Maraging Steels	20			
		3.2.2	2	Maraging Steel by Additive Manufacturing	22			
		3.2.3	3	Heat Treatment for AM Maraging steels	23			
		3.2.4	4	Mechanical Properties	27			
4		Expe	erime	nt	30			
	4.	4.1 Material and Process		erial and Process	30			
	4.	.2 DSC Analys		Analysis	30			
4.3 Heat treatment		Heat	treatment	31				
	4.	4	Porc	sity Analysis	31			
	4.	5	Char	acterization of microstructure	32			
	4.	6	X-Ray Diffraction (XRD) Analysis					
	4.	7	Vick	ers Hardness Test	33			
5		Resu	ults		35			
	5.	1	As-b	uilt Sample	35			
		5.1.3	1	Porosity Analysis	35			
		5.1.2	2	Microhardness Test	36			
		5.1.3	3	Microstructure Characterization	37			
		5.1.4		XRD Analysis	37			

5	5.1.5	DSC Analysis	38
5.2	Hea	treated Samples	39
5	5.2.1	Porosity Analysis	39
5	5.2.2	Microhardness Test	39
5	5.2.3	Microstructure Characterization	39
5	5.2.4	XRD Analysis	41
6 (	Conclusio	۳	42
7 F	Referenc	es	43

# List of Figures

Figure 1 Schematic representation of vat photopolymerization process [3]	10
Figure 2 Schematic representation of material jetting process [4]	10
Figure 3 Schematic representation of Binder jetting process [5]	11
Figure 4 Schematic representation of material extrusion process [6]	12
Figure 5 Schematic representation of PBF variations a) LPBF b) EBM [7] [8]	12
Figure 6 Schematic representation of LOM [9]	13
Figure 7 Schematic representation of a DED process using electron beam [10]	14
Figure 8 Schematic representation of powder laser DED [11]	14
Figure 9 Nozzle types relying on powder distribution [12]	15
Figure 10 Off-axis feeding powder system [12]	15
Figure 11 Coaxial nozzle types [12]	16
Figure 12 Inside beam coaxial nozzle diagram [12]	17
Figure 13 Schematic representation of a melt pool and of the heat affected zone [13]	19
Figure 14 Effect of ageing temperature on the strength and ductility of a maraging steel [17]	21
Figure 15 SEM images showing the microstructure 18Ni-300 samples produced by a) LPBF and b)	
conventional casting [21]	23
Figure 16 DSC curves for maraging steel powder and sample manufactured by LPBF [20]	24
Figure 17 SEM images showing the microstructure of LPBF-formed maraging steel under different he	eat
treatments: (a) as-built; (b) ST780-1 h; (c) ST900-1 h; (d) ST10201 h; (e) ST900-0.5 h; (f) ST900-2 h;	; (g)
ST900-4 h; (h) DAT400-6 h; (i) DAT480-6 h; (j) DAT520-6 h; [20]	26
Figure 18 Micro-Vickers hardness test for 18Ni-300 maraging steel at different heat treatments (a)(b)	ST,
(c)(d) SAT and DAT [20]	27
Figure 19 Stress-strain curves at different heat treatments (a) (b) ST; (c) (d) DAT; (e)(f) SAT [20]	29
Figure 20 ATM cutting machine	31
Figure 21 Furnace used for Heat Treatment	31
Figure 22 Vickers Hardness test indentation example [26]	34
Figure 23 Indenter Geometry of Vickers Hardness test [26]	34
Figure 24 Microscope images under a magnification of x100 for the (a)as-built sample and the sample	s
subjected to (b) DAT480-2h (c) DAT480-4h (d) DAT480-6h (e) DAT550-2h (f) DAT550-4h (g)	
DAT550-6h	35
Figure 25 Mean percentage area of porosity (in %) of the different samples	36
Figure 26 Hardness Values determined from Vickers Hardness Test	36
Figure 27 Microstructure image of the as-built sample at different magnification	37

Figure 28 XRD spectra of the as-built sample and the samples subjected to DAT at various temperature	es
and holding times	.38
Figure 29 DSC curve of M300 maraging steel as-built sample	. 39
Figure 30 Comparison between the microstructure of the (a) as-built sample and the heat treated sample	es
(b) DAT480-2h (c) DAT480-4h (d) DAT480-6h (e) DAT550-2h (f) DAT550-4h (g) DAT550-6h	.40
Figure 31 Comparison between the microstructure of the different samples at a magnification of $x500$ .	.41
Figure 32 Comparison between the microstructure of the different samples at a magnification of $x100$ .	.41

# List of Tables

Table 1 Different maraging steel composition and yield strength [16]	20
Table 2 Chemical Composition of 18 Ni-300 Maraging steel [20]	22
Table 3 Scheme of Heat treatments [20]	24
Table 4 Scheme of Heat Treatments [20]	24
Table 5 Chemical Composition of M300 – Source: Prima Industrie S.P.A	
Table 6 Heat Treatment Scheme	31
Table 7 Specifications of Microscope Lecia DMI 5000 [24]	

# 2 Abstract

When talking about Additive Manufacturing (AM), we refer to a technology that creates parts, components or repairs them through layer-by-layer deposition of material using a sliced CAD model of the desired geometry. There are many types of AM process classes that can be utilized to manufacture components. Maraging steels are a type of ultra-strength steels that are used in tool industry and aerospace applications. These steels are special since they have a very low content of carbon. The aim of this paper is to characterize samples made up of M300 maraging steel, which are manufactured using Directed Energy Deposition (DED), one of the additive manufacturing techniques. These samples are supplied from "Prima Industrie", thanks to the collaboration with Politecnico di Torino. Initially, M300 samples were subjected to Direct aging treatment at 480 °C and 550 °C for 2, 4 and 6 hours. Porosity analysis, microstructure characterization, and Vickers Hardness test for the as-built sample and the heat treated ones were done and compared to each other. This thesis is divided into four chapters. Chapter one introduces additive manufacturing technology with its types and introduces maraging steels with their properties. The second one includes the experimental activities done and the instruments used for achieving these experiments. The third chapter includes the results of the experiments and analysis on these results. The fourth includes conclusions drawn from the whole thesis work.

**Keywords**: Additive Manufacturing, Direct Energy Deposition, Maraging Steel, Direct Aging Treatment, Microstructure, Porosity Analysis, Hardness

# 3 Introduction

# 3.1 Additive Manufacturing Overview

Additive Manufacturing (AM) is a technique that is used to create components by consolidating material, layer by layer. The design of the component to be manufactured is generally prepared on a Computer-Aided Design (CAD) and the information is then transformed in a Standard Triangle Language (STL) file that slices the object into thin layers. This technique involves a computer that controls the AM process, robotic arm, Computer Numerical Control (CNC), nozzle, shielding environment, and systems for scanning and recording. In comparison to the traditional machining process, AM leads to higher material utilization rates, decreased buy to fly ratio, lower processing and finishing costs, decreased manufacturing time, advancement in mechanical properties, and decreased weight of the manufactured parts. Some techniques of additive manufacturing don't require finishing process where it is accurate in manufacturing complex shapes [1].

The produced components are then generally evaluated based on their mechanical properties and strength. The deficiencies such as cracks, thermal stresses, and porosities are studied; and the ways to reduce or eliminate them is the point of interest. Another point of interest is improving the dimensional accuracy and eliminating the necessity of machining the fabricated parts [1].

Additive manufacturing was used to build small models and prototypes, but now it is used to manufacture big components with complex geometries.

There are seven types of Additive Manufacturing process classes that can be utilized to manufacture components, which are: Vat photopolymerization, Material Jetting, Binder Jetting, Material Extrusion, Powder Bed Fusion, Sheet Lamination, and Direct Energy Deposition [2].

#### 3.1.1 Vat Photopolymerization

Another name for Vat Photopolymerization is stereolithography (SLA) that uses liquid photopolymer resin. This type utilizes a laser beam that is directed onto the resin to create the desired layer. The part is created on a building platform that moves downward after printing each layer. The layer thickness is between 0.025 and 0.5 mm. After printing each layer, the resin is cured using ultraviolet light. In stereolithography mirrors are used to direct the ultraviolet light across the resin surface. Vat Photopolymerization manufacturers parts with good accuracy and dimensional tolerance [2]. In Figure 1 a schematic representation of vat photopolymerization is presented.



Figure 1 Schematic representation of vat photopolymerization process [3]

#### 3.1.2 Material Jetting

In Material Jetting the printing head is over the platform and the deposition of material is done on the surface in a droplet form. Many droplets are deposited on the platform according to the design of each layer. After the droplets solidifies, another layer is printed on the top of the previous one until the part is printed completely. This type is accurate, and it allows the use of more than one material while printing [2]. In Figure 2 a schematic representation of material jetting process is shown.



Figure 2 Schematic representation of material jetting process [4]

#### 3.1.3 Binder jetting

This type uses a powder-based material and a binder, in which the powder is spread on the building platform and the binder is deposited on the top of it through the printing head. The binder sticks the layers together. After printing each layer, the platform is lowered, and a new layer of powder is spread on the platform. In this process different materials can be used such as polymers, ceramics, and metals. This method is one of the fastest methods, but the time needed for post processing is high [2]. In Figure 3 a representation of binder jetting process is shown.



Figure 3 Schematic representation of Binder jetting process [5]

#### 3.1.4 Material extrusion

In material extrusion, the material is heated and deposited in continuous stream from the nozzle. The building platform can move up, down and vertically and the nozzle can move horizontally. After printing the first layer, the building platform is lowered, and the successive layer is printed. Each layer fuses with the layer following it.

In this method, plastics and polymers can be used to provide strong structural support. However, this type has some disadvantages where the accuracy is reduced due to the nozzle thickness, and it is relatively slow with respect to other types of additive manufacturing [2]. Figure 4 shows a schematic representation of the material extrusion process.



Figure 4 Schematic representation of material extrusion process [6]

#### 3.1.5 Powder Bed Fusion

In Powder Bed Fusion (PBF), a layer of powder is deposited on the building platform and a thermal energy source such as laser or an electron beam fuses the powder according to the layer geometry. After printing the first layer, another layer of powder is spread using a roller and the process is repeated. This type has two variations, the first is Laser Powder Bed Fusion (LBPF) which is also called Direct Metal Laser Sintering (DMLS) and Selective laser Sintering (SLS). The second is Electron Beam Melting (EBM) [2]. These two variations are shown in Figure 5.

All variants of powder bed fusion must be done inside a chamber that is vacuum, or a chamber filled with inert gas. In this AM processes, metals and polymer powders can partially act as support structures [2].



Figure 5 Schematic representation of PBF variations a) LPBF b) EBM [7] [8]

#### 3.1.6 Sheet Lamination

Sheet lamination uses ultrasonic welding or an adhesive to bind layers. In this type there are two variations which are Laminated Object Manufacturing (LOM), which is shown in Figure 6, and Ultrasonic Additive Manufacturing (UAM). The differences between those two variants are in the bonding process and material used [2].

Sheet lamination is accomplished by putting the material in a sheet form on a cutting bed, then the layer geometry is cut using a knife or a laser. After cutting the first layer, a new sheet is placed above the first one and the process repeats itself. This method is fast and has low cost, but it has reduced accuracy. It is often used for prototypes [2].



Figure 6 Schematic representation of LOM [9]

#### 3.1.7 Directed Energy Deposition

Directed Energy Deposition (DED) is generally used to deposit additional material to an existent part or to repair a component. It can also be used to manufacture parts from scratch. The material is melted as it is deposited from the nozzle using a focused energy source such as an electron beam or a laser [10].

#### 3.1.7.1 How does DED work?

The 3D model is created using a CAD software, which is then sliced into thin layers using a slicer software [10].

The DED process is done by depositing material using a nozzle that is fixed on a four or five axis arm. This deposited material is provided to the nozzle in a form of wire or powder. As the material is being deposited from the nozzle, a heat source melts it. The heat source used is usually laser, electron beam or plasma arc. This process is repeated until the layer is solidified and the part is manufactured or repaired [10].

If an electron beam is used as a heating source, the procedure must be done in vacuum to avoid the electron interaction with air molecules or being defected by it. If a laser is used as a heating source, an inert chamber is required when reactive metals are used. Moreover, a cover of shielding gas can be used to prevent the deposited metal from contamination [10].

Figure 7 and Figure 8 show a schematic representation of a DED process using electron beam and laser respectively.



*Figure 7 Schematic representation of a DED process using electron beam [10]* 



Figure 8 Schematic representation of powder laser DED [11]

#### 3.1.7.2 Materials used in DED

This process generally uses weldable metals, such as titanium and titanium alloys, tantalum, tungsten, stainless steel, Nickel, niobium, and aluminum alloys. The wire diameter ranges between 1 and 3 mm, and the powder particle size ranges between 50 and 150 microns. Although this process is generally used with metals, but it can also be used with polymers and ceramics, such as Polymer DED with a filament of carbon fiber used by AREVO [10].

#### 3.1.7.3 Nozzle design in powder-based DED

The powder is deposited through a nozzle into the molten pool. This nozzle is designed to ensure the optimal deposition of the material, which will affect the characteristics of the manufactured part and the efficiency of the deposition process. The important point in nozzle design is to guarantee the presence of a focus point between the powder flow and the laser beam since the catchment efficiency and the melt properties rely on them. Therefore, several powder delivery systems and deposition nozzle geometries have been established. In Figure 9, five types of powder delivery systems are present. Where "a" is off-axial powder feeding, "b" is continuous coaxial nozzle, "c" is discrete coaxial nozzle, "d" and "e" are inside beam powder [12].



Figure 9 Nozzle types relying on powder distribution [12]

#### 3.1.7.3.1 Off-axial powder feeding

In Off axial powder feeding the deposition process is unidirectional, used to coat shafts and other geometries. In this process, the powder is fed by a system that is separated from the main nozzle. Figure 10 is an example of an off-axial feeding powder [12].



Figure 10 Off-axis feeding powder system [12]

The nozzle geometry establishes several characteristics of the gas powder stream, for example the standoff distance, the vertical axis point, and main and secondary flow gas velocity. These nozzles have an internal geometry that is usually circular and diverging converging one. To avoid choking, the nozzle must be inclined at different angles with respect to the laser beam. The powder is moved into the powder nozzle using a cylindrical inlet on which the powder bounces as they move. These bouncing establish the dispersion in the focus point and the powder concentration distribution. The distance between the laser beam and the off-axial nozzles, the nozzle diameter, and the inclination with respect to the laser beam direction are parameters that effects the process performance. The main disadvantage of this configuration is that this is only suitable for 2D application, and it has a low catchment efficiency [12].

#### 3.1.7.3.2 Coaxial Nozzle

In coaxial Nozzle, the delivery of powder and the laser beam are coaxial to each other. There are two types of nozzle design for coaxial nozzles depending on the delivery of the powder and shielding gas. As shown in Figure 11, the two types are presented, where "a" is continuous coaxial nozzle and "b" is discrete coaxial nozzle [12].



Figure 11 Coaxial nozzle types [12]

In continuous coaxial nozzles, the nozzle is made up of coaxial cones that are spaced apart by a value that is defined according to the annular outlet. The powder flow surrounds the laser beam into a conical shape, and both the laser beam and the cone are coaxial. In this type, the powder particles with the carrier gas are distributed inside a conical annular channel that encloses the laser beam. This system is continuous since the powder particle move everywhere along the annular space. An advantage for this system is that it can improve the nozzle efficiency since the cone angle can be adjusted to adapt the size of laser beam into the deposition area. But their tilting is limited because of the gravity on the powder cone. On the other hand, the main disadvantages of continuous coaxial nozzles are that it has a limited tilting cone angle, the inner shielding gas may force the powder outside the focus point, and it can't be used with reflective material [12].

Discrete Coaxial nozzles were created to accomplish a multidirectional deposition at a satisfactory cost. This process relies on a series of discrete injectors that are placed around the rotational axis of the nozzle having the powder being delivered coaxially to the laser beam. This structure creates a cone coaxial to the laser beam axis. Generally, three or four injectors are used while each one has its own flow control. Moreover, in this configuration, the nozzle can be tilted up to 180 ° to allow a multi axis deposition. However, the main disadvantage for this configuration is that powder flow is not uniform due to its working principle [12].

3.1.7.3.3 Inside Beam powder Nozzle

The design in the "Inside Beam powder nozzle" contains a powder feeding tube that begins outside the main nozzle and it is introduced vertically and coaxially inside the laser nozzle, as shown in Figure 12 [12].



Figure 12 Inside beam coaxial nozzle diagram [12]

Generally, an advantage can be taken from the vertical direction, where the gravity is used in delivering the powder. The system guarantees a low energy loss, and a good powder and laser coupling in the focus point. Moreover, the laser nozzle has a space to add and replace different sizes of tubes, so that it ensures an easier maintenance. However, the disadvantage of this design is that the deposited layer has a thickness less than the thicknesses of another nozzle designs, and it is only suitable for application that has the substrate normal to the nozzle [12].

#### 3.1.8 DED Solidification phenomena

During the DED process a melt pool is formed and then solidifies. The melt pool solidification is controlled by the heat transfer and can be categorized into three main events: heterogenous nucleation, mushy-zone heat transfer, and microstructural evolution by heat treatment. The mushy zone is the region between solid region and the melt pool.

Therefore, the mushy zone is a mixture of two phases: solid particles and molten metal. Moreover, in the mushy zone, the temperature is between the liquidus and solidus temperature. The melt pool and mushy zone are shown in Figure 13. In addition, the heat transfer type in the mushy zone is convection.

The blown powder causes the melt pool to have instable and transient morphology, while the trailing edge of the melt pool provides its latent heat of fusion to generate the solidified layer. This diffusion procedure is coupled with temperature-dependent microstructure development that relies on the material composition, where the initiation and growth of grain boundaries are described by the microstructure development [13].

The melt pool cooling rate is high at the solid/liquid interface and decreases at the center of the of the melt pool. Depending on heat loss and conduction, the melt pool cools rapidly since its temperature is approximately two orders of the magnitude greater than surrounding gas and environment. This rapid cooling can be an advantage since it leads to a production of fine grain structure which is leading to a desirable mechanical property. When the solidification of the melt pool occurs, the prior deposited layer or the substrate acts as a heat sink, and this leads to a directional solidification. The cooling rate is calculated as the multiplication between the laser traverse speed and the liquidus isotherm temperature gradient [13].

The solidification velocity depends on the melt pool morphology and laser travel velocity. Moreover, the melt pool thermal gradient is controlled by the established temperature field in the part and the laser power. For laser processing, the thermal gradient is high, so the liquid/solid interface position can be neglected. Thus, the solidification speed can be calculated as following:

$$|V| = |V_{beam}|.cos\theta$$

Where  $V_{beam}$  is the laser travel speed and  $\theta$  is the angle between solidification front's unit normal vector and the laser beam moving direction [13].

The solidification velocity is maximum at the trailing edge of the melt pool and minimum on the boundaries of the melt. Furthermore, the un-melted powder will act like a nucleation site in the solidified melt pool. Therefore, the melting efficiency will directly affect the solidification phenomenon and microstructure [13].



Figure 13 Schematic representation of a melt pool and of the heat affected zone [13]

#### 3.1.9 Applications

DED can be used in many applications, such as manufacturing of large structures, repairing, and coatings. It can be used to repair huge parts that are made up of high value metals. DED can be used to add material and reduce possible damage or erosion in the future. This procedure is done using a computer controlled deposition head that deposits the material according to a CAD file of the part that is being repaired. First, the part is analyzed to detect the damaged areas, such as wear or thermal degradation. Then another material that is compatible with the base alloy and that has higher hardness or high-temperature resistance will be deposited in the desired location. In some cases, a post heat treatment is used to reduce residual stresses. At last, surface finishing is applied to reach the desired tolerance [14].

#### 3.1.10 Advantages and Disadvantages of DED

Since DED work on the process of melting the metal and depositing it simultaneously, large parts can be created in a short time. Thus, the process is fast and low amount of material will be wasted. However, this process is expensive due to the size of the printers and the complication of the production process. Moreover, it has low build resolution and inconsistent finishes, where the parts need post heat treatment and machining to be harder and denser [15].

#### 3.2 Steels

Steels are alloys that consists of iron with a few amounts of carbon and many other alloying elements. The amount of carbon can range up to 2% in steels [8]. There are different types of steels and one of these types is Maraging Steels.

#### 3.2.1 Maraging Steels

#### 3.2.1.1 What are Maraging Steels?

Maraging steels are a special type of ultra-strength steels that are not hardened by carbon. Carbon is an impurity element in this type of steels, and it is maintained as low as commercially feasible [16]. Therefore, the percentage of carbon in this type of steels is less than 0.03% [17]. The hardness of these steels come from the precipitation of intermetallic compounds instead of depending on the carbide precipitates [16]. The main types of precipitates formed are: Ni<sub>3</sub>Al, Ni<sub>3</sub>Ti, Ni<sub>3</sub>Mo and Fe<sub>2</sub>Mo [17]. Moreover, this type of steel has a better formability, hardenability, and a mixture of strength and toughness due to the absence of carbon in it [16].

Different grade number of maraging steels have been developed to give different properties. The composition of some grade levels that were developed by International Nickel Ltd (Inco) are shown in Table 1 together with the yield strength (Ys) [16].

Alloy designation	Ni	Мо	Со	Ti	AI	Y <sub>S</sub> (MPa)
18Ni (200)	18	3.3	8.5	0.2	0.1	1400
18Ni (250)	18	5.0	8.5	0.4	0.1	1700
18Ni (300)	18	5.0	9.0	0.7	0.1	2000
18Ni (350)	18	4.2	12.5	1.6	0.1	2400
18Ni (cast)	17	4.6	10.0	0.3	0.1	1650

 Table 1 Different maraging steel composition and yield strength [16]

Maraging refers to the ageing of martensite, which is a hard phase that is usually detected in some steels after rapid cooling. In these steels, martensite is easily acquired due to the high amount of nickel present inside them. The hardness and the strength of these steels are increased by subjecting them to heat treatment (ageing) for multiple hours at 480-510 °C because of precipitation. In this stage, the decomposition of the metastable martensite in the steels occurs. The precipitation hardening occurs faster than the reversion reaction that creates ferrite and austenite. Therefore, significant hardening can be made before reversion happens [16].

In Figure 14, the variation of strength and ductility with respect to the ageing temperature is shown. Therefore, to attain the maximum strength, the maraging steel must be subjected to a certain ageing temperature for a specific time. Thus, achieving the yield strength while maintaining good ductility and toughness. However, over-ageing leads to

a loss in strength due to precipitate coarsening and break down of the martensite with a reestablishment of austenite [17].



Figure 14 Effect of ageing temperature on the strength and ductility of a maraging steel [17]

Maraging steels are identified by a huge number of alloying elements. Therefore, they are costly materials in comparison with another engineering alloys. Moreover, the maraging alloy development is generally affected by the presence and the cost of the alloying elements. These steels have good machining properties and are utilized in commercial and military industries such as aerospace, aircraft, and tooling applications [16].

3.2.1.2 Microstructure

Maraging steels are generated by heating the steel in the austenite phase area (about 850 °C) then followed by slow cooling in air to produce a martensite microstructure. The martensite microstructure is formed in these steels after slow cooling due to the presence of high nickel content which prevents the creation of pearlite and ferrite. The resulted martensite microstructure is soft in comparison to the martensite created in plain carbon steels by quenching. This softness is an advantage since it gives high toughness and ductility without the need for tempering. Moreover, this softness allows the maraging steel to be used in structural components, unlike other martensite steels that should first be tempered to prevent cracking [17].

#### 3.2.1.3 Properties of Maraging Steels

Most high-strength steels have low toughness and as their strength increases their toughness decreases. However, maraging steels have the special mixture of high strength and high toughness. This type of steels has high ductility [17], their strength ranges from 1500 to 2400 MPa, and their tensile strength is 2450 MPa. Moreover, these steels have very good weldability, therefore they can be easily joined while having a satisfactory integration level in the welding area. Furthermore, maraging steels have stable physical properties, and they don't undergo significant dimension change or thermal expansion after hardening treatment [18].

#### 3.2.1.4 Application of Maraging Steels

Maraging steels are utilized in aircraft applications which need material with a high strength to weight such as helicopter undercarriages, landing gears, rocket motors and slat tracks [17]. Moreover, it is used in fuel-crucial applications such as missiles and rockets. Additionally, due to their stable physical properties, maraging steels are used for machines that exchanges between hot and cold rapidly. Thus, it can be utilized for weapon firing pins, engine crankshaft, gas centrifuge units and gearboxes. Furthermore, because of its strength, it is used in golf club heads, fencing sport and bicycle frames [18].

#### 3.2.2 Maraging Steel by Additive Manufacturing

There are a lot of maraging steels that are processed by AM such as AMPO maraging steels. AMPO is a commercial name for some maraging steels produced by BÖHLER. There are different examples of AMPO maraging steels that are manufactured using LPBF such as BÖHLER W722 AMPO (X3NiCoMoTi18-9-5), BÖHLER N700 AMPO (X5CrNiCuNb17-4-4) and BÖHLER M789 AMPO. In these samples, an annealed Fe-Ni(Co) martensitic microstructure is observed after solution annealing and aging [19].

Another type of maraging steel that is processed by LPBF is 18Ni-300 maraging steel (AMPOW722). Table 2 shows the chemical composition of 18Ni-300 maraging steel. The microstructure of the as-built structure is presented in Figure 17 (a), where melt boundary is visible (red circle). Moreover, on the different sides of the boundary, different morphologies are present with small angle clusters made up of fine cellular structures (orange arrow) and strips (blue arrow) [20].

	Units	Min(%)	Max(%)	Weight (%)
Al	Aluminium	0	0.1	0.1
Ti	Titanium	0.8	1.2	0.8
Si	Silicon	0	0.10	-
S	Sulphur	0	0.010	-
Р	Phosphorus	0	0.010	-
0	Oxygen	0	0.10	-
Ni	Nickel	17.00	19.00	18.2
Ν	Nitrogen	0	0.10	-
Mo	Molybdenum	4.5	5.2	5.3
Mn	Manganese	0	0.15	-
Fe	Iron	Balance	-	Balance
Cr	Chromium	0	0.25	-
Co	Cobalt	8.5	10.0	9
С	Carbon	0	0.03	<0.2

Table 2 Chemical Composition of 18 Ni-300 Maraging steel [20]

Jaydeep et al. studied the microstructure of 18Ni-300 samples processed by LPBF and reported that due to the LPBF high cooling rate, the microsegregation is obvious and the grain size is very small. Therefore, the resulted microstructure was different than the microstructure of the casting ones (Figure 15) [21].



Figure 15 SEM images showing the microstructure 18Ni-300 samples produced by a) LPBF and b) conventional casting [21]

#### 3.2.3 Heat Treatment for AM Maraging steels

The conventional heat treatments achieved for maraging steels are solution treatment for 1 hour at 840 °C and aging treatment for 6 hours at 480 °C. These treatments were appropriate for wrought and casting maraging steel samples. However, for additive manufacturing, different heat treatments are done. For example, Bai et al. performed solution treatment (ST), direct aging treatment (DAT), and solution + aging treatment (SAT) heat treatments on 18Ni-300 maraging steel samples manufactured by LPBF. To avoid sample oxidation during heating, the samples were placed in a sealed glass tubes loaded with high purity argon gas. The heat treatment temperatures were selected based on the microstructural evolution analyses. A differential scanning calorimeter (DSC) analysis on 18Ni-300 powder and as-built sample were done at a heating rate of 10 °C/min in an argon atmosphere (Figure 16). The peaks of the as-built sample were associated to [20]:

- peak #1 (exothermic) creation of carbide, precipitation of coherent precipitation zones or recovery of martensite
- peak #2 (exothermic) grain growth of retained austenite and the creation of the main intermetallic precipitates, such as Ni<sub>3</sub>(Ti, Mo) phases followed by the decomposition of Fe<sub>2</sub>Mo
- peaks #3 (endothermic) phase change from  $\alpha$ -phase (BCC) to  $\gamma$ -phase (FCC). Where  $\alpha$ -phase is martensite and  $\gamma$ -phase is austenite
- peak #4 (endothermic) related to the decomposition of precipitates



Figure 16 DSC curves for maraging steel powder and sample manufactured by LPBF [20]

# Based on DSC results the heat treatments were selected and are indicated in Table 3 and Table 4 [20].

 Table 3 Scheme of Heat treatments [20]

		-					
Experiment No.	ST		DAT		SAT		Heating rate <sup>°</sup> C/min
	Temperature/°C	Holding time/h	Temperature/°C	Holding time/h	Temperature/°C	Holding time/h	
1	780	1	400	6	900 + 400	6	10
2	840	1	440	6	900 + 440	6	10
3	900	1	480	6	900 + 480	6	10
4	960	1	520	6	900 + 520	6	10
5	1020	1	560	6	900 + 560	6	10

Table 4 Scheme of Heat Treatments [20]

Experiment No.	ST		DAT		SAT		Heating rate°C/min
	Temperature/°C	Holding time/h	Temperature/°C	Holding time/h	Temperature/°C	Holding time/h	
1	900	0.25	520	1	900 + 520	6	10
2	900	0.5	520	3	900 + 520	6	10
3	900	1	520	6	900 + 520	6	10
4	900	2	520	9	900 + 520	6	10
5	900	4	520	12	900 + 520	6	10

Figure 17 shows the microstructure of the as-built and heat treated samples. In Figure 17 (b)-(g), it is clearly obvious that boundaries, cells, and strips vanishes gradually as temperature and holding time increase. Where at low ST temperatures, relatively big size grain boundaries still present with some white particles in the boundaries. As the temperature increases, the microstructure reveals interconnected large slats that are

presented in Figure 17 (c) using orange and white arrows. This occurs due to the elevated temperatures during ST which leads to the growth of austenite grain. The growth of austenite grain causes larger martensite laths after ST. Furthermore, as shown in Figure 17 (e)-(g) small effect on the microstructure is shown when increasing the holding time during ST [20].

Figure 17 (h)-(m) shows the microstructures of the samples subject to Direct Aging Treatment (DAT) at various temperatures and holding times. In these microstructures, it can be observed that the melt boundary, cells, and strips don't vanish. However they become blurred as the temperature and holding time increases. It can be seen that the microstructure at DAT400-6 h, as shown in Figure 17 (h), is similar to the as-built one. However, at DAT520-6 h the melt boundary begins to dissolve. As the temperature increases to 560 °C, the long strips and cells are broken into spherical particles and short strips. The same result is obtained when the holding time increases from 1 hour to 12 hours at 520 °C [20].

Figure 17 (n)-(r) shows the microstructures of the samples subjected to SAT. These microstructures are made up of packets of martensite at SAT400-6 h inside prior-austenite grains as shown in Figure 17 (n), as indicated by the green circles and arrows. The grain boundaries show that the packets of martensite do not extend out of the prior-austenite grain boundary. In Figure 17 (o) and (q), it can be noticed that the martensite laths become wider and longer, but the boundary is still slightly noticeable. However, when the holding time rises to 12 h or the temperature rises to 560 °C, the martensite laths vanish. Instead, a lot of uneven bright bars are inserted in the dark matrix which are indicated by white lines in Figure 17 (p) and (r) [20].

Therefore, the microstructure development of 18Ni-300 maraging steel was significantly affected by Solution Treatment and Solution + Aging Treatment. When the ST temperature is 780 °C, the white particle and the austenite grain boundaries can be seen. Moreover, at 840 °C lathes and melt track boundaries vanish. However, the microstructure of the samples subjected to Direct Aging Treatment is comparable to the microstructure of the as-built sample. Furthermore, microstructure for the samples subjected to SAT is made up of lath martensite. But this lath martensite vanishes and is substituted by white and dark tissues when the holding time reaches 12h or the temperature reaches 560 °C.



Figure 17 SEM images showing the microstructure of LPBF-formed maraging steel under different heat treatments: (a) as-built; (b) ST780-1 h; (c) ST900-1 h; (d) ST10201 h; (e) ST900-0.5 h; (f) ST900-2 h; (g) ST900-4 h; (h) DAT400-6 h; (i) DAT480-6 h; (j) DAT520-6 h; [20]

#### 3.2.4 Mechanical Properties

#### 3.2.4.1 Microhardness

A microhardness test was done on 18Ni-300 maraging steel. This test was done on the as-built, ST, DAT, and SAT samples. In Figure 18(a), it is obvious that the microhardness of the samples subjected to solution treatment for 1 hour, decreases as the temperature increases until it reaches a minimum at 900 °C and then increases slightly. However, in Figure 18(b), the microhardness of the samples subjected to solution treatment at 900 °C decreases sharply, in comparison to the as-built sample, as the holding time increases. This means that solution treatment can decrease the microhardness of 18Ni-300 maraging steel [20].

Figure 18(c) and (d) show the microhardness of SAT and DAT samples, such that SAT-A, SAT-B, DAT-A and DAT-B means solution and aging treatment at various temperature, solution and aging treatment with various holding time, direct aging treatment at various temperature, and direct aging treatment with various holding time respectively. Moreover, Figure 18(c) and (d) show that the microhardness can be enhanced significantly by subjecting the samples to SAT and DAT. The microhardness of the samples subjected to DAT increases first and then decrease slightly as the holding time and aging temperature increases. Furthermore, SAT samples had similar results to that of DAT samples. Therefore, the conduction of solution treatment before the aging treatment has a small effect on microhardness [20].



Figure 18 Micro-Vickers hardness test for 18Ni-300 maraging steel at different heat treatments (a)(b) ST, (c)(d) SAT and DAT [20]

#### 3.2.4.2 Temperature dependent tensile property

Figure 19 presents the tensile curves at room temperature under ST, DAT, and SAT to compare the tensile behavior under several heat treatments. As shown in Figure 19 (a)-(b), the tensile strength of the heat treated samples decreases slightly in comparison to the as-built sample under ST. However, under DAT and SAT, the tensile strength increases sharply in comparison to the as-built sample, as shown in Figure 19 (c)-(f) [20].

In Figure 19(a), the tensile strength of the heat treated samples drops and then increases slightly as the ST temperature increases. However the fracture elongation is approximately the same. On the other side, the tensile strength of the heat treated sample is approximately the same in Figure 19(b), but the fracture elongation increases and then drops as the holding time increases at 900 °C. For samples subjected to DAT, both the tensile strength and the fracture elongation seem to rise and then decrease as the temperature and holding time increase. This is due to the effect of precipitated particles and austenite. With the formation of precipitated particles, the tensile strength increases, however when the particles become too big, the tensile strength will decrease. The big particles will also cause a reduction in the fracture elongation. Moreover, the austenite content can enhance the elongation, but it also decreases the tensile strength. For the samples subjected to SAT, fracture elongation is nearly the same, but the tensile strength increases at the tensile strength is approximately the same the more asses at the tensile strength is approximately the same, but the fracture elongation is nearly the same, but the tensile strength increases and then decreases as the temperature increases (Figure 19(e)). However, as the holding time increases, the tensile strength is approximately the same, but the fracture elongation increases and then decreases as the temperature increases (Figure 19(e)).

Furthermore, it can be noticed that the tensile strength can be enhanced to the same level using DAT or SAT. Therefore, DAT can be used alone to improve the tensile strength of the LPBF manufactured sample, without the need to other solution treatments [20].



Figure 19 Stress-strain curves at different heat treatments (a) (b) ST; (c) (d) DAT; (e)(f) SAT [20]

# 4 Experiment

#### 4.1 Material and Process

The samples of M300 maraging steel were manufactured using DED by Laserdyne 430 AM machine. These samples were provided from Prima Industrie S.p.A. The chemical composition of M300 powder used to build the samples is shown in Table 5. This chemical composition was tested and certified by CARPENTER ADDITIVE on 12/Feb/2020.

	Units	Min (%)	Max (%)	Weight (%)
Al	Al Aluminium		0.1	0.1
Ti	Titanium	0.8	1.2	0.8
Si	Silicon	0	0.10	0.03
S	Sulphur	0	0.010	0.002
Р	Phosphorus	0	0.010	0.005
0	Oxygen	0	0.10	0.01
Ni	Nickel	17.00	19.00	18.38
N	Nitrogen	0	0.10	< 0.01
Мо	Molybdenum	4.5	5.2	4.9
Mn	Manganese	0	0.15	0.02
Fe	Iron	Balance	-	Balance
Cr	Chromium	0	0.25	0.01
Со	Cobalt	8.5	10.0	9.1
С	Carbon	0	0.03	0.02

Table 5 Chemical Composition of M300 – Source: Prima Industrie S.P.A

### 4.2 DSC Analysis

Differential Scanning Calorimeter (DSC) is a thermal analysis device which measures the change of a samples' microstructure. DSC is used to determine the heat flow related to the material variation with respect to time and temperature. During a temperature change, the DSC measures a heat quantity, which is absorbed by the sample according to the temperature change between the reference material and the sample [22].

In order to know at which temperatures the samples must be heat treated, a DSC analysis was done on the as-built sample at a rate of 10 °C/min. Therefore, the cutting machine was used to extract a sample of size 3\*3\*7 mm<sup>3</sup> from the center of the as-built sample. The cutting machine used is ATM Brillant 220 which is shown in Figure 20.



Figure 20 ATM cutting machine

### 4.3 Heat treatment

Heat treatments were done in a furnace of type Nabertherm, shown in Figure 21, in Politecnico di Torino laboratory. The heat treatment method chosen was Direct Ageing Treatment (DAT) in Argon gas atmosphere according to Table 6.



Figure 21 Furnace used for Heat Treatment

Experiment Number	Sample	Temperature (°C)	Holding time(hours)
1	DAT480-2h	480	2
2	DAT480-4h	480	4
3	DAT480-6h	480	6
4	DAT550-2h	550	2
5	DAT550-4h	550	4
6	DAT550-6h	550	6

# 4.4 Porosity Analysis

Porosity is defined as the measure of the void spaces in a sample. These void spaces are called pores and can be found on the surface of the sample or inside it. Generally, the properties of the material can be significantly affected by the presence of porosity [23].

Therefore, porosity analysis was done to assess the effect of heat treatment on the percentage area of porosity found in these samples. The porosity was calculated using ImageJ, which is a software used for image analysis. This software can calculate the percentage area of porosity found in the sample.

After subjecting the samples to the heat treatments and before subjecting the samples to porosity analysis, the samples were grinded at several stages starting from 180 grit size paper till 4000 grit size paper. After grinding, the samples were polished using 6 microns to 1 micron diamond suspension. At last, the samples were polished using diamond paste. After polishing, the microscope, LEICA DMI 5000, was used to observe the samples under a magnification of x100.

For each sample, 50 pictures were taken on different areas of the polished sample. Then the 50 pictures were uploaded to ImageJ software to get the mean value of the percentage area of porosity found in each sample.

# 4.5 Characterization of microstructure

After the porosity analysis, the samples were etched using 2% Nital Etchant solution, and the microstructure was then observed using the microscope at different magnifications. The microscope used was LEICA DMI 5000 which is an inverted light microscope, and its specifications are listed in Table 7.

Manager for a forma in	T
Manufacturer	Lecia
Light Source	Halogen lamp
Magnification Range	(12.5x) 50x-1000x
Lenses	1.2x, 5x, 10x, 20x, 50x, 100x
Additional optical magnification	0.8; 1.25; 1.88
Options	Managing of illumination
	Managing of Contrast
	Personalized function keys
Contrast Techniques	Brightfield
_	Darkfield
	Polarization
	DIC (Nomarsik-Contrast)
Camera	CCD, 1-inch-Chip, 5 Megapixel
Equipment	Image data base
	Image analysis software
	Panoramic image
	Sharpness reconstruction

 Table 7 Specifications of Microscope Lecia DMI 5000 [24]

### 4.6 X-Ray Diffraction (XRD) Analysis

Then XRD analysis was done on the as-built and heat treated samples. This analysis was done to know the phase change of these samples due to the heat treatments done. Thus the cutting machine was used to extract from each sample another sample of maximum height 5 mm. These 5 mm height samples were polished again until 4000 grit size paper and then subjected to XRD analysis.

X-Ray Diffraction (XRD) is a method utilized in material science to know the crystallographic structure of a material. It works by exposing a material with incident X-rays and then measuring the scattering angles and the intensities of the X-rays that are going out from the material [25].

The XRD analysis is a non-destructive procedure that is used to identify the materials according to their diffraction pattern. Moreover, it is used to identify the phase of the material. Also it can give information on how the actual structure changes from the ideal one [25].

### 4.7 Vickers Hardness Test

At last, Vickers hardness test was done to measure the hardness of the samples. Before applying the test, all samples were polished again to get a smooth surface in order to minimize the errors. The hardness test was done in "Politecnico di Torino - Sede di Alessandria", using "Leica VMHT 1200" machine. The test done was microhardness test by applying 500 gf (gram force) for 15 seconds.

Generally, the Vickers hardness test is performed using a diamond indenter that is utilized to exert a force on a sample. The hardness value is concluded from the depth of the indentation on the sample. Usually, as the indentation is smaller as the hardness of the sample is higher. Figure 22 shows an example of the Vickers hardness test indentation, and it shows that the hardness of Sample A is higher than that of Sample B which is also higher than the hardness of Sample C [26].

The hardness test can be divided into two categories, microhardness and macrohardness tests. The hardness test is called microhardness test when the load applied is less than 1 kgf. However it is called macrohardness test when the load applied is greater than 1 kgf [27].



Figure 22 Vickers Hardness test indentation example [26]

The test is performed by pressing the indenter into the surface of the material being tested. The indenter has a pyramid shape, which has a square base and a 136° angle among the opposite faces as shown in Figure 23. The force is usually exerted for 10 to 15 seconds. After applying the force, the diagonals created on the surface of the sample are measured, and the average is utilized to calculate the hardness, having the ratio F/A (Where A (in mm<sup>2</sup>) is the surface area of the indentation, and F (in kgf) is the Force exerted on the surface of the sample). The measurements are generally obtained either by using a microscope or by using a software that gives an accurate result. [26]



Figure 23 Indenter Geometry of Vickers Hardness test [26]

Generally, the Vickers hardness test has some advantages such as [26]:

- The indenter used is small, thus decreasing the risks of potential damage to the test material
- The same indenter is used for any type of material
- Only one test force is applied

# 5 Results

## 5.1 As-built Sample

5.1.1 Porosity Analysis

Figure 24 shows a comparison between the microscope images under a magnification of x100 for the as-built and heat treated samples. Also Figure 25 shows the mean percentage area of porosity found in each sample.

As shown in Figure 24(a) the M300 maraging steel sample printed by DED is a high dense sample since it contains a low percentage of porosity. According to Figure 25, the mean percentage of porosity of the as-built sample is 0.4%. These porosities are produced by unmelted powder particles and capture vapor in liquid pools due to crystallization [28].



*Figure 24 Microscope images under a magnification of x100 for the (a)as-built sample and the samples subjected to (b) DAT480-2h (c) DAT480-4h (d) DAT480-6h (e) DAT550-2h (f) DAT550-4h (g) DAT550-6h* 



Figure 25 Mean percentage area of porosity (in %) of the different samples

#### 5.1.2 Microhardness Test

Figure 26 shows the hardness of the samples understudy. For each sample nine indentations were applied in order to get a mean value for the whole sample. As shown in Figure 26 the hardness of the as-built sample is 347.2 HV



Figure 26 Hardness Values determined from Vickers Hardness Test

#### 5.1.3 Microstructure Characterization

Figure 27 shows the microstructure of the as-built sample at different magnifications. Moreover, in Figure 27 (c) it can be seen that there are several morphologies with clusters consisting of strips (indicated by the orange arrow) and fine cellular structure (indicated by red arrow).



Figure 27 Microstructure image of the as-built sample at different magnification

#### 5.1.4 XRD Analysis

Figure 28 shows XRD spectra of the as-built sample and the samples subjected to DAT at various temperatures and holding time. It is obvious that in the as-built sample the  $\alpha$ -phase (BCC), which is martensite, and  $\gamma$ -phase (FCC), which is austenite, coexist. Therefore, FCC structure has been generated during DED.



Figure 28 XRD spectra of the as-built sample and the samples subjected to DAT at various temperatures and holding times

#### 5.1.5 DSC Analysis

Figure 29 shows the DSC curve generated from the DSC analysis. In Figure 29 the DSC curve of the as-built sample undergoes four peaks at 475 °C, 550 °C, 676 °C and 759 °C. The first peak (exothermic) can be caused due to the formation of carbides or recovery of martensite. The second peak (exothermic) is generally related to the grain growth of retained austenite and the creation of the primary intermetallic precipitates, for example Ni<sub>3</sub>(Ti,Mo) phases followed by Fe<sub>2</sub>Mo decomposition. The third peak (endothermic) is related to the phase change from  $\alpha$ -phase to  $\gamma$ -phase. The fourth peak (endothermic) is produced by the precipitates decomposition [20]. Moreover, certain disturbances appear at high temperatures as shown at 870 °C. These disturbances can be attributed to the presence of impurities such as oxides through the molding procedure [20].

Therefore, it is feasible to deduce that the aging temperature for M300 maraging steel printed by DED is between 400 and 600 °C.



Figure 29 DSC curve of M300 maraging steel as-built sample

#### 5.2 Heat treated Samples

#### 5.2.1 Porosity Analysis

The microscope images in Figure 24(b)-(g) are approximately the same as the microscope image of the as-built sample(Figure 24(a)). Moreover, this is shown in Figure 25, where the minimum percentage area of porosity is for DAT480-4h, indicating 0.13 %, and the maximum is for DAT550-6h, indicating 0.47%. Therefore, it can be concluded that there is no change in the percentage area of porosity after the heat treatments.

5.2.2 Microhardness Test

In Figure 26, it is obvious that the hardness of the heat treated samples are greater than the hardness of the as-built sample. Moreover it can be noticed that as the holding time was increasing from 2 to 6 hours, for the samples that were heat treated at 480 °C, the hardness was increasing. However this is not the same condition for the samples that were heat treated at 550 °C. Where for DAT550-2h the hardness is 526 HV, for DAT550-4h it is 491 HV, and for DAT550-6h it is 504 HV.

The maximum value of hardness was achieved by the sample that was heat treated at 480 °C for 6 hours having a hardness value equal to 546 HV

#### 5.2.3 Microstructure Characterization

In Figure 30, a comparison between the microstructure of the as-built and heat treated samples is shown. As shown in Figure 30, the strips (indicated by the orange arrow), and cellular structure (indicated by the red arrow) don't vanish as temperature and holding time increase. However, they become blurred. It can be seen that at DAT480-2h the morphology is similar to that of the as-built one. However, the long strips breakdown into shorter strips

and the cellular structure change into spherical particles, as the holding time increases from 2 to 6 hours (Figure 30 (b)-(d)). The same is also shown in Figure 30(e)-(g). Furthermore, it is obvious that as holding time is increasing the strips and cell walls become thinner, and the same happens when the temperature increases.

Figure 31 and Figure 32 shows the microstructure of the different samples at a magnification of x500 and x100 respectively.



Figure 30 Comparison between the microstructure of the (a) as-built sample and the heat treated samples (b) DAT480-2h (c) DAT480-4h (d) DAT480-6h (e) DAT550-2h (f) DAT550-4h (g) DAT550-6h



Figure 31 Comparison between the microstructure of the different samples at a magnification of x500



Figure 32 Comparison between the microstructure of the different samples at a magnification of x100

#### 5.2.4 XRD Analysis

In Figure 28, it is obvious that the first and second peaks for  $\gamma$ -phase was absent for both the as-built and DAT480-2h samples. However, the first two peaks for  $\gamma$ -phase are approximately the same for DAT480-4h, DAT480-6h and DAT550-2h. Moreover, it is obvious that the  $\gamma$ -phase peaks have the highest intensity for DAT550-4h and DAT550-6h. This implies that BCC-matrix turns into FCC-matrix during DAT, and high temperatures with long holding time produces austenite phase. Therefore, as the holding time and temperatures increase the content of austenite increases.

# 6 Conclusion

This paper studies the characteristics of M300 maraging steel printed by Direct Energy Deposition (DED). The samples were subjected to Direct Aging treatment (DAT) at different temperatures and holding times. The evolution of porosity, microstructure, and hardness are investigated for the as-built and heat treated samples. The following conclusion are drawn from this study

- 1) The DSC curve of the as-built sample undergoes four peaks at 475 °C, 550 °C, 676 °C and 759 °C, from which the aging temperature can be indicated.
- 2) M300 maraging steel manufactured by DED is characterized by high density
- 3) The porosity of M300 maraging steel does not change with DAT.
- 4) The microstructure of the as-built M300 maraging steel is made up of several morphologies with clusters consisting of strips and fine cellular structure
- 5) DAT causes the breakdown of the long strips into smaller ones, and the cellular structure changes into spherical particles. Moreover, the strips and cell walls become thinner as the temperature and holding time increase.
- 6) Austenite and martensite microstructures are present in the as-built and DAT samples
- 7) Austenite content increases in DAT samples as the temperature and holding time increase.
- 8) DAT enhances the hardness of M300 maraging steel
- 9) The best heat treatment for the highest hardness is DAT at 480 °C for 6 hours having a hardness of 546 HV.

# 7 References

- [1] G. Rasiya, A. Shukla and K. Saran, "Additive Manufacturing-A Review," *Materials Today: Proceedings*, p. 6, 22 May 2021.
- [2] Applied Engineering, "7 Types of Additive Manufacturing," 22 January 2021.
- [3] M. M, R. K, B. N and . P. D. J, Additive Manufacturing: A Tool for Industrial Revolution 4.0, Matthew Deans, 2021.
- [4] 3DEXPERIENCE MAKE, "Introduction to 3D printing additive processes".
- [5] D. Oropeza and J. H. A, "Reactive binder jet additive manufacturing for microstructural control and dimensional stability of ceramic materials," p. 12, 27 October 2021.
- [6] J. Pou, A. Riveiro and P. D. J, Additive Manufacturing, 2021.
- [7] H. RIEDER, A. DILLHÖFER, M. SPIES, J. BAMBERG and T. HIESS, "Online Monitoring of Additive Manufacturing Processes Using Ultrasound," p. 9, 6 october 2014.
- [8] R. Lancaster, G. Davies, H. Illsley, S. Jeffs and G. Baxter, "Structural Integrity of an Electron Beam MeltedTitanium Alloy," p. 19, 14 June 2016.
- [9] A. Razavykia, E. Brusa, C. Delprete and R. Yavari, "An Overview of Additive Manufacturing Technologies—A Review to Technical Synthesis in Numerical Study of Selective Laser Melting," p. 22, 3 September 2020.
- [10] C. V, "The Complete Guide to Directed Energy Deposition (DED) in 3D Printing," 3D natives, 10 September 2019.
- [11] M. Niinomi, Metals for BiomedicalDevices, 2019.
- [12] A. Guner, P. Bidare, A. Jiménez, S. Dimov and K. Essa, "Nozzle Designs in Powder-Based Direct Laser Deposition: A Review," p. 18, 10 August 2022.
- [13] S. M. Thompson , L. Bian, N. Shamsaei and A. Yadollahi, "An overview of Direct Laser Deposition for additive manufacturing; Part I: Transport phenomena, modeling and diagnostics," *Additive Manufacturing*, p. 27, 20 July 2015.
- [14] D. Svetlizky, M. Das, B. Zheng, A. L. Vyatskikh, S. Bose, A. Bandyopadhyay, J. M. Schoenung, E. J. Lavernia and N. Eliaz, "Directed energy deposition (DED) additive manufacturing: Physical characteristics, defects, challenges and applications," *Materials Today*, p. 25, october 2021.
- [15] S. Shepheard, "Directed Energy Deposition (DED): A Complete Guide," *3dsourced,* 26 June 2021.

- [16] W. Sha and Z. Guo, Maraging steels Modelling of microstructure, properties and applications, Woodhead Publishing Limited, Abington Hall, Granta Park, Great Abington, Cambridge CB21 6AH, UK, 2009.
- [17] A. P. Mouritz, Introduction to aerospace materials, Woodhead Publishing materials, 2012.
- [18] B. Toulas, "Maraging Steel: Unique Properties and Uses," engineering clicks, 21 september 2017.
- [19] C. Turk, H. Zunko, C. Aumayr, H. Leitner and M. Kapp, "Advances in Maraging Steels for Additive Manufacturing," p. 5, 18 February 2019.
- [20] Y. Bai, D. Wang, Y. Yang and H. Wang, "Effect of heat treatment on the microstructure and mechanical properties of maraging steel by selective laser melting," *Materials Science & Engineering A*, p. 13, 01 June 2019.
- [21] J. Vishwakarma, K. Chattopadhyay and N. Santhi Srinivas, "Effect of build orientation on microstructure and tensile behaviour of selectively laser melted M300 maraging steel," *Materials Science & Engineering A*, p. 11, 21 August 2020.
- [22] P. Gill, T. T. Moghadam and B. Ranjbar, "Differential Scanning Calorimetry Techniques: Applications in Biology and Nanoscience," 21 December 2010.
- [23] Atria Innovation, "How to measure the materials porosity," 16 February 2021.
- [24] DRESDEN TECHNOLOGY PORTAL, "INVERTED LIGHT MICROSCOPE LEICA DMI 5000".
- [25] TWI, "WHAT IS X-RAY DIFFRACTION ANALYSIS (XRD) AND HOW DOES IT WORK?".
- [26] MATMATCH, "Vickers Hardness Test: What it is and how it's measured".
- [27] Nanoscience Instruments, "Microhardness Testing".
- [28] M. Król, P. Snopiński, J. Hajnyš, M. Pagáč and D. Łukowiec, "Selective Laser Melting of 18NI-300 Maraging Steel," p. 12, 25 September 2020.
- [29] J. Sabhadiya, "What Is Steel?- Properties, Uses | Types Of Steels," 30 January 2021.
- [30] M. Mele, G. Campana and G. Fumelli, "Environmental impact assessment of Arburg plastic freeforming additive manufacturing," p. 14, 9 June 2021.