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Research on the Brazing Technology and Interfacial Characterization of Al6016/Zinc- Coated Steel Joints

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Abstract

The growing demand for lightweight structures in the automotive sector has increased the interest in multi-material systems combining aluminium alloys and steel, where low density, corrosion resistance, and structural strength can be exploited simultaneously. However, joining aluminium to steel remains a major challenge because of the large thermo-physical mismatch between the two materials, the presence of a stable oxide layer on aluminium, and the tendency to form brittle Fe–Al intermetallic compounds at the interface. In this context, brazing represents a promising joining route, especially when zinc-coated steel is used, since zinc can act as a low-melting interfacial medium that promotes wetting, modifies local interfacial reaction, and reduces direct Al–Fe interaction.

The aim of this thesis is to investigate the brazing of aluminium alloy to zinc-coated steel, with particular focus on the role of zinc in interfacial bonding and on the effect of aluminium surface preparation on joint formation. More specifically, the work compares different experimental trials carried out on the Al / Zn-coated steel system, using chemical surface treatments on the aluminium side and varying brazing temperature while keeping the dwell time constant. The purpose is to evaluate how these conditions influence wetting, zinc redistribution, interfacial morphology, and the formation of Zn-rich or mixed reaction layers. To achieve these objectives, the joints are characterised by optical microscopy, SEM-EDS and backscattered electron imaging (BSE), which are used to examine interfacial continuity, local morphology, compositional difference and phase distribution across the joining region. Through this approach, the thesis aims to provide a clearer understanding of the mechanisms governing brazing between aluminium and zinc-coated steel and to identify the processing conditions that are more favourable for stable interfacial bonding.

The results show that bonding in the Al / Zn-coated steel system is governed by the formation of Zn-rich and Al–Zn mixed interfacial regions rather than by direct aluminium–steel contact. At the same time, the quality of the joint was found to be highly sensitive to local processing conditions. In several cases, the interface was non-uniform and affected by excessive gaps, porosity, local discontinuities, and heterogeneous zinc redistribution. The observations further indicate that surface preparation strongly influences interfacial reactivity, while non-uniform contact and experimental setup limitations can significantly affect the development of a continuous bond. For this reason, the study is not limited to verifying whether joining occurs, but also examines why the quality of the interface changes from one trial to another and from one local region to another.

Overall, the thesis shows that brazing is a viable route for joining aluminium to zinc-coated steel, but the stability and reliability of the joint depend on careful control of oxide removal, zinc-mediated wetting, and local interfacial conditions. The work contributes to a better understanding of the mechanisms governing Al / Zn-coated steel brazing and provides a basis for further process optimisation for lightweight multi-material applications.

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1. Introduction

1.1 Lightweight multi-material structures in automotive applications

The continuous search for higher energy efficiency, lower emissions, and improved structural performance has driven the automotive sector toward the increasing use of lightweight multi-material solutions. In modern vehicle design, reducing mass is one of the most effective strategies for decreasing fuel consumption in conventional vehicles and extending driving range in electric vehicles. For this reason, the replacement of heavier monolithic steel components with hybrid structures combining lightweight alloys and high-strength steels has become an important research and industrial objective [1,2].

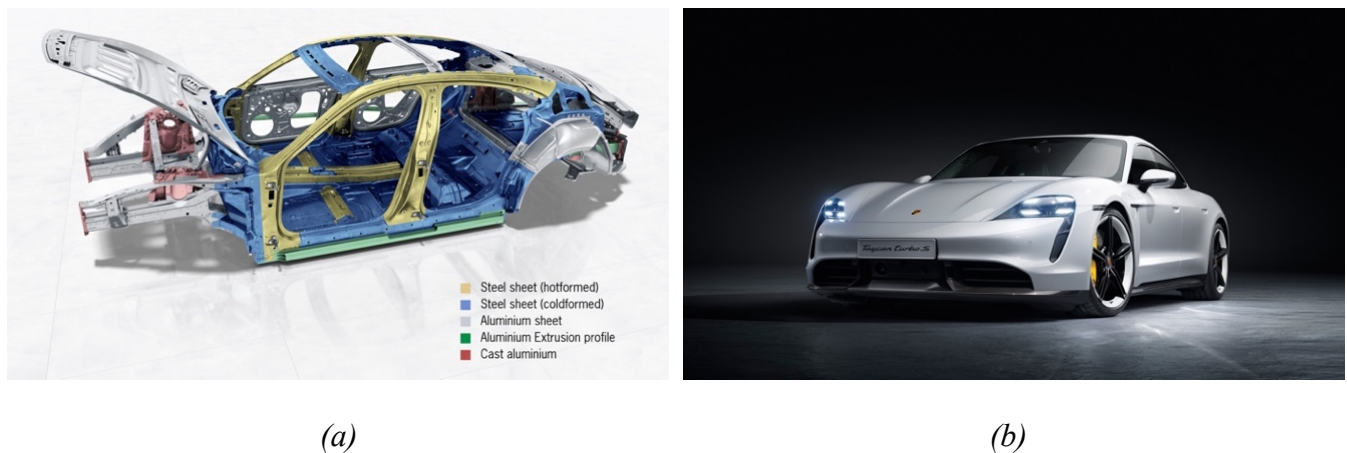


Figure 1. Porsche Taycan body multi-material design: (a) body-in-white multi material structure; (b) complete electric vehicle [5].

Among lightweight structural materials, aluminium alloys are particularly attractive because of their favourable strength-to-weight ratio and good corrosion resistance. Steel, however, remains indispensable in many vehicle components because of its higher strength, lower cost, and established role in forming and joining operations. The difficulty does not lie only in combining two materials with different compositions, but also in managing their very different thermo-physical behaviour during processing. As noted in current thesis work, aluminium-to-steel joining is hindered by differences in melting temperature, thermal conductivity, thermal expansion, and chemical affinity, all of which complicate the formation of a sound and reliable metallurgical bond [1]. The logic behind multi-material design is not simply weight reduction for its own sake, but rather the optimisation of each structural zone according to loading, stiffness, crashworthiness, and durability requirements. In practice, this means that lightweight alloys can be used where mass savings are critical, while steels remain essential in regions requiring higher strength or more robust structural performance.

The development of lightweight automotive body structures increasingly relies on the combined use of advanced high-strength steels (AHSS) and aluminium alloys. Multi-material concepts based on these materials are already being applied in lightweight body-in-white structures, including components such as pillars and seatbacks [1]. In such designs, AHSS is used where high strength and crash resistance are required, while aluminium alloys are introduced in order to reduce mass and improve overall structural efficiency [2]. This

material combination is therefore highly attractive for modern fuel-efficient vehicles, but its broader application depends strongly on the availability of reliable joining technologies for dissimilar metallic systems [2].

A representative example of this material combination was reported for the joining of AA6022-T4 to galvanized zinc-coated DP980 steel [3], showing that this type of aluminium/AHSS combination is directly relevant to automotive lightweighting research. Their study highlights that most automotive body structures use AHSS protected by a thin zinc coating for corrosion resistance, and that the presence of this coating must be considered when designing joining processes because it modifies the interfacial reactions and the resulting microstructure. For this reason, aluminium-to-zinc-coated steel systems represent an important class of multi-material joints in automotive engineering and provide a meaningful basis for further study of dissimilar joining routes [3].

Another representative automotive application of dissimilar aluminium-to-steel joining was reported for the combination of AA6016 aluminium alloy and zinc-coated DP600 steel [4]. Zinc-coated steel sheets are increasingly used in the automotive industry because of their high corrosion resistance, while aluminium alloys are progressively introduced to replace heavier steel components in order to reduce vehicle mass and support the development of lightweight car bodies. In this context, the combination of AA6016 and zinc-coated DP600 steel represents a relevant material system for multi-material automotive structures [4].

1.2 Challenges in Al–Steel joining

Joining aluminium to steel is difficult because the two materials behave very differently during heating and cooling. Their melting temperatures, thermal conductivity, and thermal expansion coefficients are strongly mismatched, so the thermal cycle is not uniform at the interface. This makes it difficult to obtain a stable joining condition without inducing local stress concentration, uneven reaction, or microstructural instability [2]. A second major difficulty is the formation of brittle Fe–Al intermetallic compounds. Because aluminium and iron have limited mutual solubility, direct contact at elevated temperature promotes the formation of interfacial reaction products that are hard and brittle. If these compounds grow excessively, they weaken the joint and increase the risk of cracking or premature failure [3].

A quantitative example from the literature confirms the critical effect of intermetallic growth on Al–steel joint performance. In resistance spot welding of aluminium to hot-dipped galvanised steel, the fracture mode changed from button pull-out to interfacial failure when the intermetallic compound layer thickness exceeded approximately 3 μm . They also cited results showing that, for aluminium joined to galvanized steel, a thin intermetallic layer below about 2 μm was necessary to obtain a strong joint [3]. These findings demonstrate that even small increases in the thickness of the interfacial reaction layer can significantly reduce the mechanical reliability of aluminium–steel joints.

Another critical obstacle is the oxide layer naturally present on aluminium. Aluminium rapidly forms a stable Al_2O_3 film when exposed to air, and this layer strongly reduces surface reactivity [7]. During joining, the oxide acts as a barrier to wetting and metallurgical interaction, making it harder for the intermediate liquid phase to spread uniformly and bond effectively to the aluminium substrate [8]. For this reason, proper surface preparation is

essential before brazing. The present work identifies oxide removal as a key requirement for successful joining.

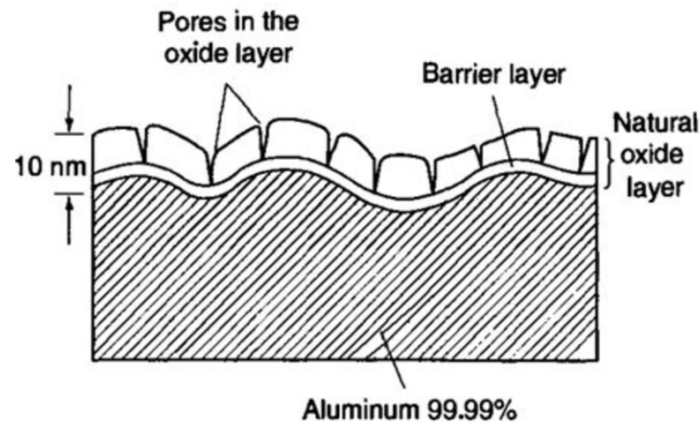


Figure 2. Oxide-layer schematic on aluminium surface [7]

In the case of zinc-coated steel, zinc helps the joining process but also introduces additional complexity. Since zinc melts at a much lower temperature than aluminium, it can promote wetting and reduce direct Al–Fe contact [4]. In addition, the behaviour of the zinc coating during heating may expose fresher metallic steel surface and modify local spreading conditions, which further affects the continuity and morphology of the interfacial layer. However, molten zinc may redistribute unevenly during heating, producing non-uniform interfacial layers and locally different reaction products. The current work shows that this behaviour can lead to heterogeneous bonding, with some regions showing interfacial continuity and others showing gaps, discontinuities, or Zn-rich intermediate layers [4].

Therefore, the main challenges in Al–steel joining can be summarized as follows: thermo-physical mismatch between the two base materials, formation of brittle Fe–Al intermetallic compounds, low reactivity of oxidised aluminium surfaces, and the complex redistribution of zinc during joining. These factors act simultaneously and make the formation of a continuous, defect-free, and mechanically reliable interface particularly difficult.

1.3 Role of zinc-coated steel in dissimilar joining

In dissimilar aluminium–steel joints, zinc-coated steel plays a central role because the zinc layer actively modifies the interfacial reactions that occur during joining. Unlike bare steel, which would come into direct contact with aluminium and promote the formation of brittle Fe–Al intermetallic compounds, zinc-coated steel introduces an intermediate metallic layer that changes both the chemistry and the wetting behaviour of the joint. In this sense, the zinc coating is not only a protective surface layer, but also a functional interfacial medium in the joining process.

The first important function of the zinc coating is to act as a barrier between aluminium and iron. By limiting direct Al–Fe contact, the zinc layer helps reduce the tendency for immediate formation of brittle Fe–Al intermetallic compounds, which are generally detrimental to joint integrity [3]. The zinc-coated surface therefore provides a more favourable condition for the development of an intermediate joining layer. In addition, during heating, the zinc coating

may expose fresher metallic steel surface and improve local wetting conditions, which further influences the continuity and morphology of the interfacial region [6].

A representative literature study confirms that the zinc coating actively controls both phase formation and joint quality in aluminium-to-steel joining. Dharmendra et al. investigated laser welding-brazing of AA6016 to zinc-coated DP600 steel using a Zn–15Al filler and reported that the reaction layer formed along the steel–seam interface had a thickness in the range of 3–23 μm , depending on the brazing conditions. The best joints were obtained for heat inputs between 60 and 110 J/mm and brazing speeds between 0.5 and 0.8 m/min, for which the interfacial layer thickness was approximately 8–12 μm and the mechanical resistance reached about 220 MPa. Their SEM–EDS analysis further showed that the interface layer contained approximately 48% Al, 31% Fe, and 21% Zn, indicating that the zinc coating does not remain inert, but participates directly in the formation of Zn-containing reaction products at the interface [4]. These results show quantitatively that the role of zinc-coated steel is not limited to corrosion protection: the zinc layer melts at relatively low temperature, promotes wetting, modifies local diffusion conditions, and contributes directly to the development of the interfacial phases formed during joining. In this sense, the use of temperatures above the melting point of zinc but below the melting point of aluminium makes it possible to form a Zn-rich liquid interfacial region, which favours Al–Zn interaction before extensive direct Al–Fe reaction occurs [4].

The second major function of zinc-coated steel is related to the low melting temperature of zinc. Since zinc melts at approximately 419 °C, it becomes liquid within the temperature range used for brazing, while both aluminium and steel remain solid. This temperature selection is critical because the brazing process is intentionally performed above the melting point of zinc but below the melting point of aluminium [4]. Under these conditions, zinc can form a liquid interfacial phase that improves wetting and promotes spreading along the aluminium–steel interface without melting the base materials [3]. According to literature studies on aluminium-to-zinc-coated steel joining, temperatures in this intermediate range favour the formation of Zn-rich and Al–Zn interfacial regions while limiting excessive direct Al–Fe interaction and suppressing the growth of thick brittle Fe–Al intermetallic compounds.

If the temperature is too low, zinc spreading and wetting become insufficient, leading to poor interfacial continuity. In contrast, excessively high temperatures increase diffusion between aluminium and iron, accelerating intermetallic growth and increasing the risk of brittle joint behaviour. Therefore, the selected brazing temperature range represents a compromise between sufficient zinc activation and controlled interfacial reaction development. This molten zinc can then spread at the interface and promote wetting, allowing bonding to occur through a Zn-rich liquid phase rather than through direct solid-state contact between aluminium and steel. In this way, the zinc coating supports the formation of a metallurgical bond at temperatures lower than the melting point of the base materials, which is one of the main reasons why zinc-coated steel is particularly suitable for brazed dissimilar joints [3].

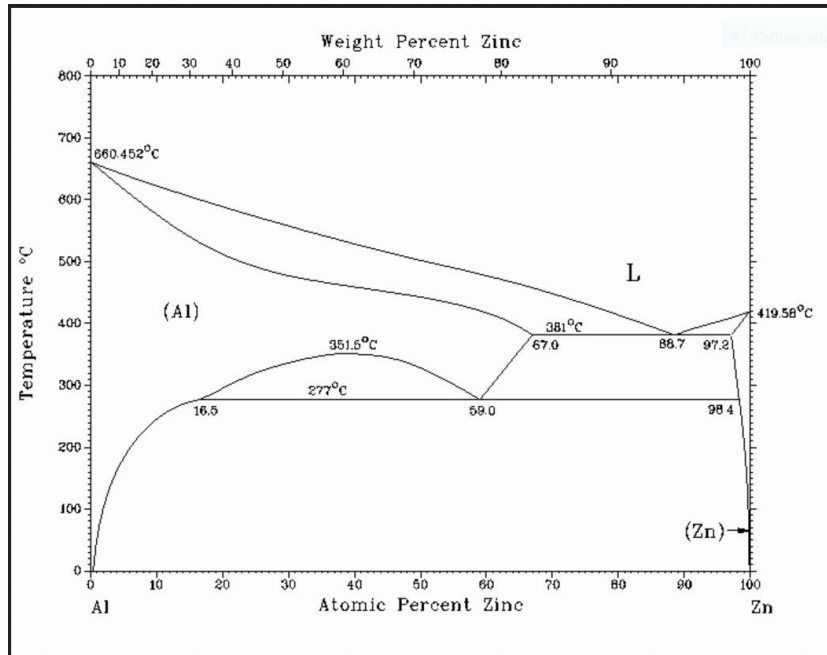


Figure 3. Al-Zn phase diagram [7]

According to the Al–Zn phase diagram, eutectic reactions between aluminium and zinc can occur at relatively low temperatures, with a eutectic point near 381–382 °C, which is significantly lower than the melting point of aluminium. During brazing above the melting temperature of zinc (~ 419 °C) but below the melting temperature of aluminium, a Zn-rich liquid phase can therefore form at the interface and promote local diffusion and wetting between the materials. Literature studies report that this liquid interfacial region favours the formation of Al–Zn mixed phases before extensive direct Al–Fe interaction occurs [4, 6].

Dharmendra et al. observed that Al–Zn phase formation is thermodynamically favoured because aluminium has a stronger affinity for zinc than for iron, while limited heat input suppresses excessive formation of brittle Fe-rich intermetallic compounds [4]. Similarly, Lu et al. reported the formation of Al–Zn diffusion layers and local eutectic structures during joining of aluminium to zinc-coated steel [3].

At the same time, the role of zinc-coated steel is not purely beneficial, because the zinc layer also introduces complexity into the joining process. As zinc melts and redistributes, it may form non-uniform interfacial layers depending on local temperature, oxidation, contact conditions, and pressure distribution. If the liquid phase is not able to spread uniformly, the interface may become discontinuous, with localised Zn-rich regions, gaps, or partially reacted zones [1] [6]. Thus, while zinc-coated steel improves the possibility of joining aluminium to steel, the final quality of the joint still depends on how effectively the zinc layer is able to wet, spread, and interact with the adjacent materials during processing.

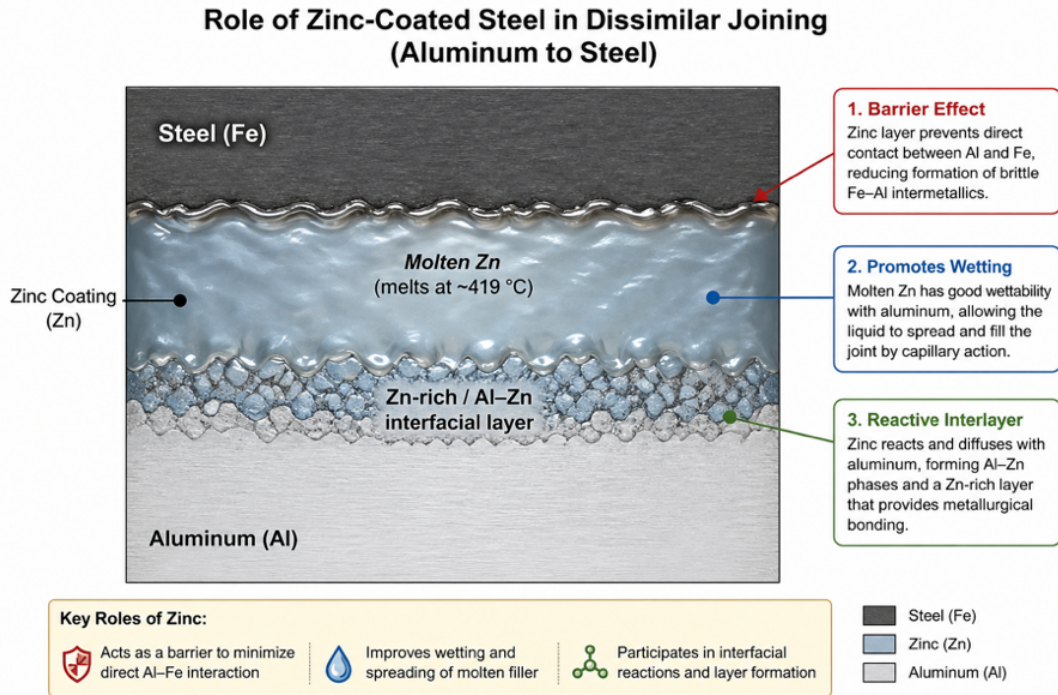


Figure 4. Schematic illustration of the role of zinc-coated steel in dissimilar joining (schematic created by the author based on Refs. [6])

Overall, in dissimilar aluminium–steel joints, zinc-coated steel performs three essential functions: it reduces direct aluminium–iron interaction, it provides a low-melting interfacial phase that promotes wetting during brazing, and it actively participates in the formation of Zn-based reaction layers at the interface. For this reason, its role is fundamental in enabling and controlling the joining of aluminium to steel under brazing conditions.

1.4 Brazing as a joining route for Al / Zn-coated steel

Brazing is a suitable joining route for aluminium and zinc-coated steel because it allows bonding without melting the base materials. This is important in dissimilar joints, where direct fusion joining often causes severe metallurgical problems, especially the formation of brittle Fe–Al intermetallic compounds [2]. In the Al / Zn-coated steel system, brazing is particularly effective because zinc melts at a much lower temperature than aluminium and steel. As discussed before, during heating, the zinc layer becomes liquid and acts as an intermediate joining medium, promoting wetting and interfacial interaction while the base sheets remain solid [3]. In this way, bonding can occur through a Zn-mediated mechanism rather than direct aluminium–steel fusion.

Another important advantage of brazing is the lower thermal input compared with welding processes. Since the process is carried out below the melting point of aluminium and steel, it reduces thermal damage and helps limit excessive interfacial reaction. This makes brazing attractive for lightweight multi-material systems where preserving the integrity of the base materials is essential [4].

A representative literature example demonstrating the suitability of brazing for aluminium-to-zinc-coated steel joining was reported by Dharmendra et al. In their laser welding-brazing study of AA6016 aluminium alloy to zinc-coated DP600 steel, sound metallurgical bonding was achieved without melting the steel substrate. The authors reported that the zinc-containing liquid interfacial layer improved wetting and reduced excessive direct Al–Fe interaction, while controlled heat input promoted stable joint formation and limited brittle intermetallic growth [4]. These observations are consistent with the present work, where brazing is used to promote Zn-mediated bonding and controlled interfacial layer development between aluminium and zinc-coated steel.

However, successful brazing of Al / Zn-coated steel still depends strongly on interfacial conditions. Oxide removal from aluminium, proper wetting of molten zinc, and good local contact between the sheets are all necessary to obtain a continuous and reliable joint. This experimental work shows that even when brazing conditions are similar, the interface may still become discontinuous or non-uniform if these factors are not properly controlled. For these reasons, brazing is used in this thesis as the main joining route for the Al / Zn-coated steel system, since it provides the most suitable condition for studying the formation of Zn-rich interfacial layers and the factors controlling joint quality.

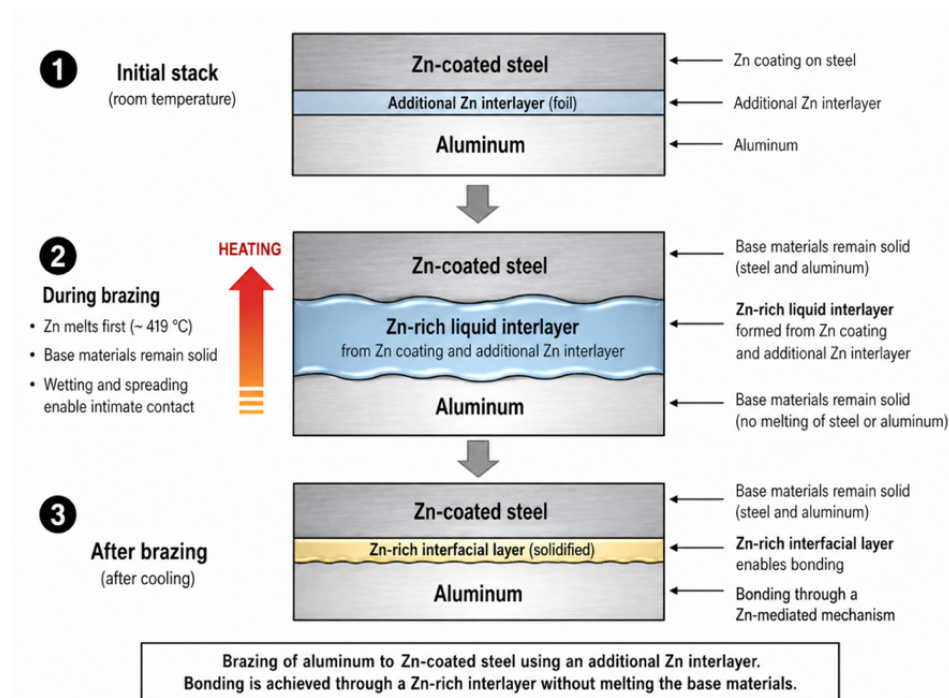


Figure 5. Schematic representation of Zn-mediated brazing in Al / Zn-coated steel system (schematic created by the author based on Refs. [3,6])

2. Technical Background

2.1 Fundamentals of brazing

2.1.1 Definition and principles of brazing

Brazing is conventionally defined as a joining process in which metallic materials are bonded using a filler metal that melts above 450 °C but below the melting temperature of the base materials [30]. During the process, the filler metal melts and flows into the joint, while the base materials remain solid. After cooling, the molten filler solidifies and forms a metallic bond between the substrates. In the present work, zinc melts at approximately 419 °C, which is slightly below the conventional brazing threshold [17]. Therefore, the process is discussed as a brazing-related joining route, because the Zn-rich liquid phase performs the same essential function as a brazing filler: it wets the interface, spreads between aluminium and zinc-coated steel, and promotes metallurgical bonding without melting the base materials [24].

The process is based on the ability of the liquid phase to wet the surfaces of the base materials and to flow into the joint gap by capillary action. For successful brazing, the surfaces must be clean and reactive, the joint clearance must be appropriate, and the thermal conditions must allow sufficient melting and spreading of the filler metal. If these conditions are not satisfied, the joint may remain incomplete or discontinuous [30].

Compared with fusion welding, brazing causes less thermal damage and is particularly suitable for dissimilar materials, since bonding is achieved without melting the substrates. For this reason, brazing is widely used when preservation of the base materials and controlled interfacial reaction are required.

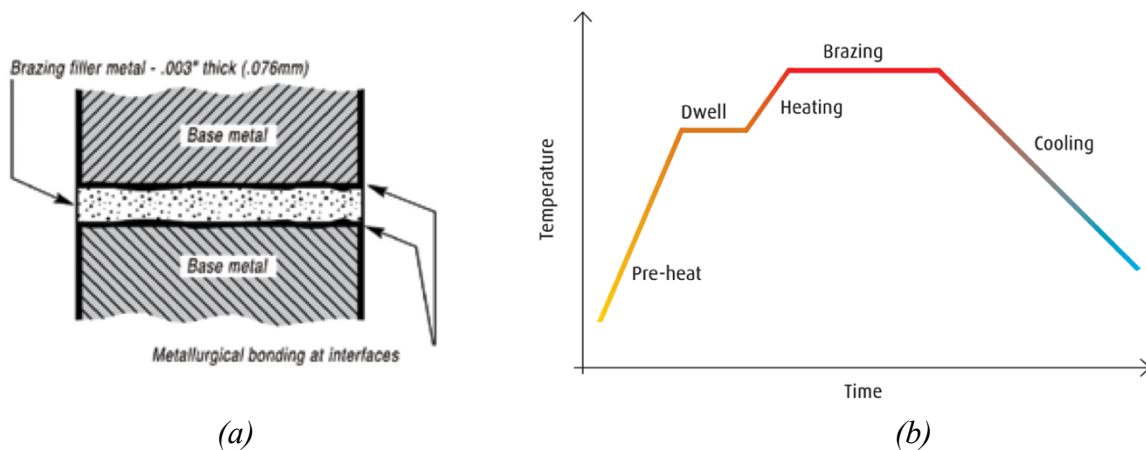


Figure 6. (a) the schematic representation of brazed joint and (b) a typical brazing thermal cycle [30]

2.1.2 Wetting and capillary action

Wetting and capillary action are fundamental to brazing because they control how the molten filler spreads and fills the joint. Wetting describes the ability of the liquid phase to spread over the surface of the base materials and establish intimate contact with them. Good wetting is essential for forming a continuous metallurgical bond, while poor wetting leads to incomplete or weak joining. The degree of wetting is commonly described by the contact angle between the molten filler and the solid surface: a lower contact angle indicates better wetting, while a higher contact angle corresponds to poor spreading [19].

Capillary action is the mechanism by which the molten filler is drawn into the gap between the joined parts. Its effectiveness depends on favourable wetting conditions and suitable joint clearance. If the gap is too large or the surface is poorly wettable, the liquid phase cannot spread uniformly, and the joint may remain discontinuous.

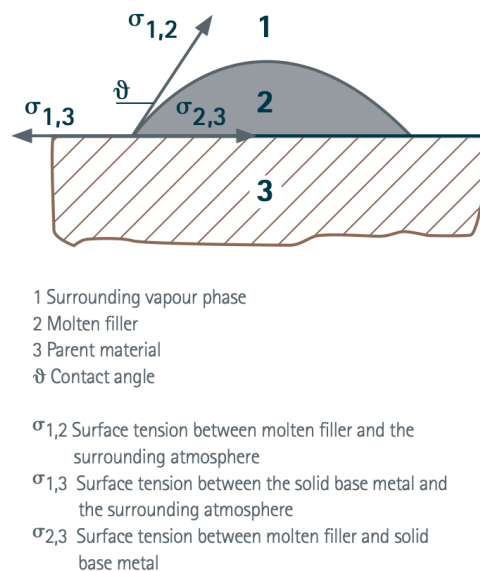


Figure 7. Wetting of a metallic surface with a liquid filler metal [21]

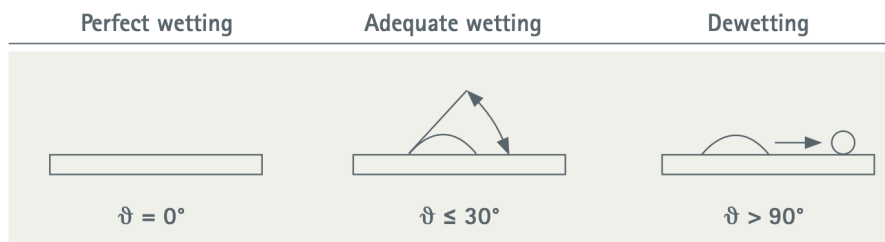


Figure 8. Relationship between contact angle and degree of wetting [19]

In aluminium-containing systems, wetting and capillary flow are strongly affected by the surface condition of the aluminium substrate. Therefore, the presence and stability of the surface oxide layer must be considered separately, since it influences the reactivity of the interface during brazing.

2.1.3 Interfacial reactivity and oxide stability in Al-steel brazing

The reactivity of aluminium–steel systems is one of the main challenges during brazing. Aluminium rapidly forms a stable Al_2O_3 oxide layer when exposed to air. This oxide film is chemically stable and has a very high melting temperature, above 1800°C , which is much higher than the brazing temperature used in this work. As a result, the oxide layer remains solid during brazing and acts as a barrier between the molten Zn-rich phase and the metallic aluminium substrate. Therefore, the main problem is not only geometrical wetting, but mainly the reduced surface reactivity of aluminium, since the oxide layer limits metallurgical interaction and the formation of a continuous interfacial bond. For this reason, surface preparation and oxide removal are essential before brazing, as they increase the availability of fresh reactive aluminium surface and improve the possibility of interfacial bonding [24].

When the oxide layer is locally removed or disrupted, aluminium becomes highly reactive with iron from the steel substrate. This promotes diffusion between Al and Fe atoms and leads to the formation of Fe–Al intermetallic compounds such as Fe_2Al_5 and FeAl_3 at the interface [17]. Although a thin intermetallic layer is necessary for metallurgical bonding, excessive growth of these phases is undesirable because they are hard and brittle and may reduce the mechanical strength of the joint [18].

Temperature and heat input strongly influence interfacial reactions. Higher temperatures and longer holding times accelerate diffusion and increase intermetallic layer thickness. For this reason, thermal control is essential during aluminium–steel brazing. The use of zinc as an interlayer is particularly beneficial because molten zinc improves wetting and spreading while reducing direct Fe–Al interaction. Since zinc melts at approximately 419°C , it becomes liquid before aluminium melting and acts as an intermediate layer that helps control interfacial reactions and limits excessive intermetallic growth.

2.1.4 Brazing temperature range and filler/interlayer behaviour

The brazing temperature range is critical because it must be high enough to melt the filler or interlayer material but remain below the melting point of the base materials. This allows the formation of a liquid phase that can spread along the interface and create bonding without melting the substrates [30].

If the temperature is too low, the filler may not melt completely or may not have sufficient fluidity to wet the surfaces properly. As a result, spreading is limited and the joint may remain incomplete. If the temperature is too high, excessive interfacial reactions, oxidation, porosity, or unstable redistribution of the liquid phase may occur, which can reduce joint quality [17].

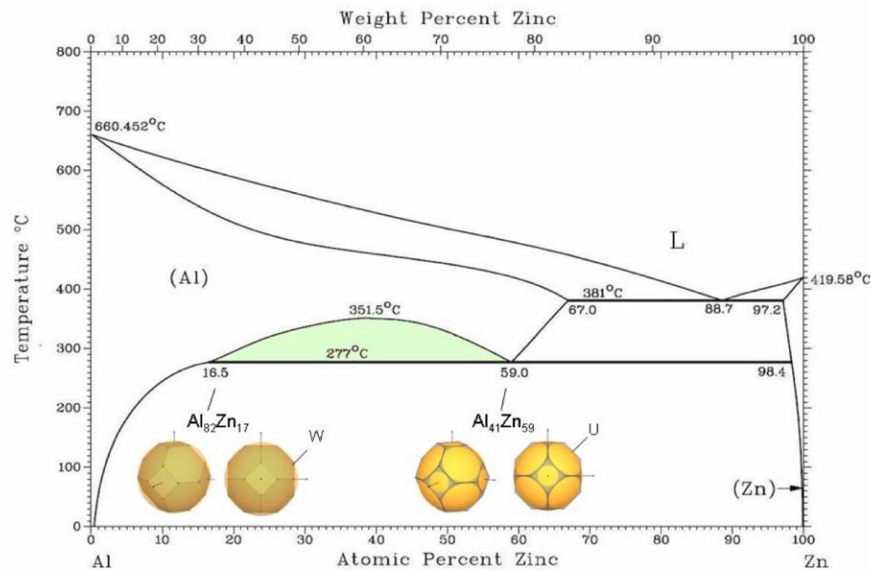


Figure 9. Al-Zn binary phase diagram illustrating the temperature range relevant to the melting and redistribution of zinc during brazing

As shown in Figure 9, zinc melts at a much lower temperature than aluminium, which allows the formation of a Zn-rich liquid phase during brazing while the aluminium substrate remains solid. This behaviour is essential for Zn-mediated joining, since the molten zinc can spread along the interface and promote local diffusion and interfacial interaction [24].

The behaviour of the filler or interlayer during brazing is therefore essential. Once melted, it must spread uniformly, penetrate the joint gap, and interact with the surfaces of the base materials. In dissimilar systems, the filler or interlayer does not only fill the gap but also affects diffusion and interfacial layer formation. For this reason, both the selected temperature range and the behaviour of the liquid phase strongly influence the final morphology and quality of the brazed joint.

2.2 Brazing of aluminium to zinc-coated steel

2.2.1 Role of zinc during brazing

During brazing, zinc acts as the active liquid interlayer between aluminium and zinc-coated steel. Because it melts at a much lower temperature than the base materials, it can spread along the interface while aluminium and steel remain solid. This liquid-phase behaviour is essential for interfacial wetting, gap filling, and the initiation of metallurgical interaction across the joint. In addition, the evaporation and redistribution of zinc during heating can expose fresher metallic steel surface, improve the wettability of liquid aluminium on steel, and modify local spreading conditions at the interface.

As the process proceeds, molten zinc redistributes along the interface and interacts mainly with aluminium, leading to the formation of Zn-rich and Al-Zn transition regions. It should

also be noted that the zinc coating is not always chemically pure Zn, since galvanised or galvanized steel may already contain Zn–Fe intermetallic phases before joining. The extent and uniformity of this redistribution strongly affect joint continuity. Therefore, in the present system, the role of zinc during brazing is mainly to provide the liquid phase through which interfacial spreading, diffusion, and bond formation can occur [25].

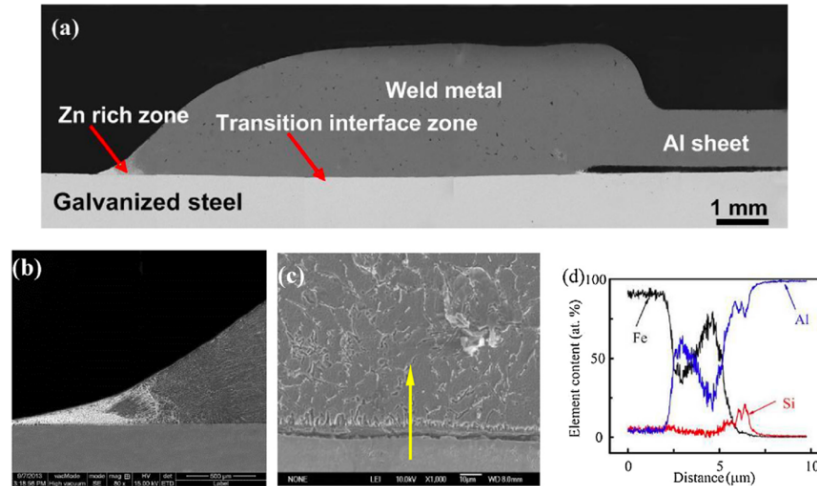


Figure 10. (a) The typical cross sectional structures of a welding – brazing joint between Al and steel, (b) the details of Zn – rich zone at the periphery of weld, (c) the microstructures of the transition interface zone, (d) EDS line scanning of the transition interface zone along yellow line in (c) [24].

2.2.2 Zn-mediated joining mechanism

In the brazing of aluminium to zinc-coated steel, joint formation occurs through a Zn-mediated mechanism rather than by direct contact between aluminium and steel. When the brazing temperature exceeds the melting point of zinc, the molten zinc then spreads along the interface, wets the adjacent surfaces, and creates the conditions for interfacial bonding.

The first stage of this mechanism is the melting and spreading of zinc in the joining region. Through wetting and capillary flow, the liquid phase penetrates the interfacial gap and increases the real contact area between the materials. The second stage involves interaction between molten zinc and the solid substrates, leading mainly to the formation of Zn-rich and Al–Zn transition regions, sometimes with minor Fe contribution. As a result, the joint develops through intermediate interfacial layers rather than through direct fusion of the base materials [21].

The effectiveness of this mechanism depends strongly on how uniformly the liquid zinc can spread and react. According to literature, this redistribution is not always a simple outward flow of zinc but may also involve diffusion from Zn-enriched peripheral regions into the liquid interaction zone. If zinc distribution is homogeneous, a more continuous interfacial layer can form; if not, the interface may remain discontinuous and contain gaps, voids, or isolated Zn-rich regions. Therefore, Zn-mediated joining should be understood as a liquid-phase interfacial process controlled by zinc redistribution, local contact conditions, and interfacial reactions during brazing [20].

2.2.3 Interfacial reactions in Al–Zn–steel systems

Interfacial reactions in Al–Zn–steel systems are controlled by the melting and redistribution of zinc between the two solid substrates. During brazing, molten zinc reacts first with aluminium, leading mainly to the formation of Zn-rich and Al–Zn transition regions near the aluminium side. In some areas, limited iron diffusion from the steel side also occurs, giving rise to local Al–Zn–Fe reaction zones [24].

Previous studies on Zn-based joining of aluminium to zinc-coated steel reported that the steel-side reaction layer may vary from a few micrometres to a few tens of micrometres depending on process conditions and may also be locally non-uniform along the interface.

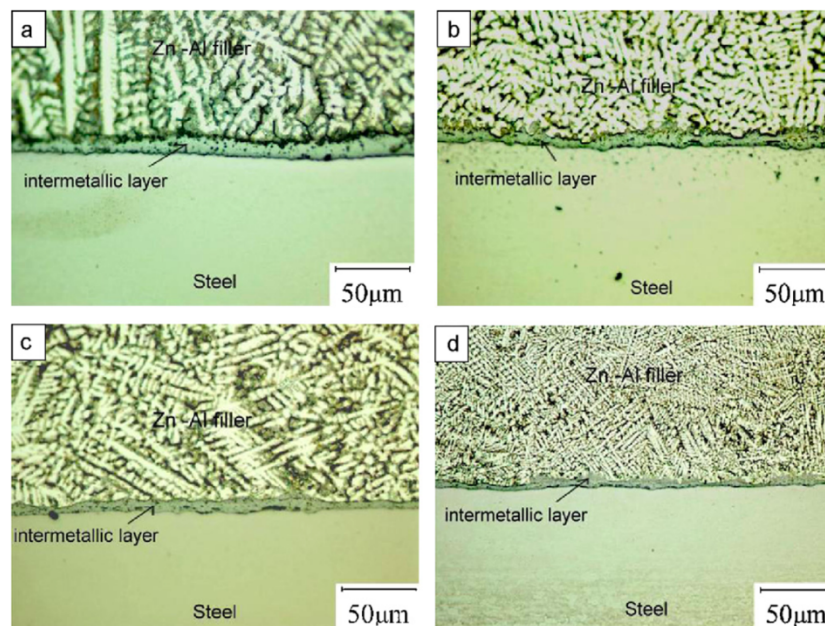


Figure 11. Cross sectional images of the interface region of joints produced under different brazing speeds: (a) 0.2 m/min ($X=19.4m$), (b) 0.3 m/min ($X=10.3m$), (c) 0.5 m/min ($X=9.2m$), (d) 0.7 m/min ($X=6.4m$)[19].

As a result, the interface is not a single sharp boundary, but a heterogeneous reaction layer whose composition and thickness vary locally. Depending on temperature, surface condition, and contact quality, this layer may be continuous in some regions and discontinuous in others, with possible pores, isolated Zn-rich regions, or partially reacted zones. Therefore, bonding in Al–Zn–steel systems is governed by the formation and local evolution of these interfacial reaction layers [25].

2.3 Influence of processing parameters on joint formation

The formation of a brazed joint is strongly influenced by the selected processing parameters, since they control melting of the interlayer, wetting of the substrates, interfacial diffusion, and the final morphology of the reaction zone. In the Al / Zn-coated steel system, temperature, dwell time, and local contact conditions are therefore considered the main

variables governing joint formation. The processing conditions adopted in the present study were selected with reference to previous experimental observations, which showed that brazing at 480 °C with a dwell time of 8 minutes provided more favourable joining behaviour. These conditions therefore served as the basis for the present investigation [24].

2.3.1 Influence of temperature

Temperature is one of the most important processing parameters in brazing because it directly controls melting, fluidity, wetting behaviour, diffusion, and interfacial reaction kinetics. In Al / Zn-coated steel systems, the selected temperature must be high enough to activate the zinc layer and allow the formation of a liquid Zn-rich interfacial phase, but it must remain below the melting point of the aluminium and steel substrates in order to avoid fusion of the base materials [30].

According to the literature, zinc melts at approximately 419 °C, whereas aluminium melts at approximately 660 °C [7]. Therefore, the useful brazing-related temperature range for Al / Zn-coated steel joining is generally located above the melting point of zinc and below the melting point of aluminium. Within this range, molten zinc can spread along the interface, promote wetting, and contribute to the formation of Zn-rich and Al–Zn interfacial regions. At the same time, excessive temperature or excessive heat input can increase diffusion between aluminium and iron, promote the growth of brittle Fe–Al intermetallic compounds, and lead to a thicker or more heterogeneous reaction layer.

Literature data also show that the interfacial reaction layer is strongly affected by thermal conditions. In laser welding–brazing of AA6016 aluminium alloy to zinc-coated DP600 steel, Dharmendra et al. reported reaction layer thicknesses in the range of 3–23 µm depending on the brazing conditions [4]. The best joints were obtained under controlled heat input conditions, where the reaction layer was limited and stable metallurgical bonding was achieved. This confirms that temperature and heat input must be carefully controlled in order to obtain sufficient zinc activation while avoiding excessive interfacial growth.

If the temperature is too low, zinc spreading and capillary flow may be insufficient, resulting in incomplete wetting or discontinuous bonding. If the temperature is too high, interfacial reactions may become excessive, increasing the risk of brittle intermetallic formation, porosity, oxidation, or unstable redistribution of the liquid phase. For this reason, temperature selection in Al / Zn-coated steel brazing represents a compromise between effective Zn-mediated wetting and controlled interfacial reaction development [6].

2.3.2 Influence of dwell time

Dwell time is another important processing parameter because it determines how long the joint remains at the selected brazing temperature. During this period, the molten interlayer can spread along the interface, wet the substrates, and promote diffusion and interfacial reactions [30]. If the dwell time is too short, the liquid phase may not have enough time to fill the joint gap or interact sufficiently with both base materials, resulting in incomplete or discontinuous bonding. If the dwell time is too long, excessive diffusion and reaction layer

growth may occur, increasing the risk of brittle intermetallic formation and local degradation of the joint [2].

In aluminium–steel joining, literature data show that the effect of dwell time is closely linked to heat input and interfacial reaction kinetics. Longer exposure at elevated temperature generally promotes the growth of Fe–Al intermetallic compounds, whereas shorter times may limit bonding if diffusion and spreading are insufficient. For this reason, dwell time must be selected as a compromise between allowing sufficient wetting and metallurgical interaction while avoiding excessive reaction layer thickening [2].

In the case of aluminium to zinc-coated steel joining, the presence of zinc makes the dwell time particularly important because the molten Zn-rich phase needs sufficient time to spread, redistribute, and participate in the formation of interfacial reaction layers. Dharmendra et al. showed that the quality and thickness of the reaction layer in AA6016/zinc-coated DP600 steel joints depended strongly on the thermal conditions of the process, confirming that controlled heat exposure is necessary to obtain stable bonding without excessive interfacial growth. In the study, controlled thermal exposure allowed the reaction layer to remain within the reported range of 3–23 μm , showing that time and heat input together influence the final interfacial thickness and joint quality [4].

2.3.3 Influence of local contact and pressure distribution

Local contact conditions and pressure distribution also play an important role in brazed joint formation. Even when the selected temperature and dwell time are appropriate, bonding cannot occur effectively if the molten interlayer cannot bridge the gap between the two substrates. A sufficiently small and continuous joint clearance favours capillary flow, wetting, and interfacial reaction [30]. In contrast, excessive or non-uniform gaps reduce the ability of the liquid phase to spread continuously, leading to discontinuous bonding, voids, or locally unreacted regions.

In brazing, the joint clearance must therefore be controlled together with the thermal cycle. Literature on dissimilar aluminium–steel joining shows that non-uniform contact can strongly influence the morphology and continuity of the interfacial layer [2]. When the contact pressure is not uniformly distributed, some regions may develop intimate contact and metallurgical bonding, while other areas may remain separated or only partially reacted. This produces heterogeneous interfaces with local variations in reaction layer thickness and bonding quality [6].

For this reason, local contact and pressure distribution should be considered as process-dependent variables, even if they are more difficult to quantify than temperature or dwell time. They directly affect the real contact area between the sheets, the capillary flow of the molten zinc-rich phase, and the final continuity of the brazed interface [6, 30].

2.4 Surface preparation of aluminium prior to brazing

2.4.1 Native oxide layer on aluminium

Aluminium rapidly forms a stable oxide layer when exposed to air. This surface film, mainly Al_2O_3 , has a high melting point and acts as a barrier to wetting and metallurgical interaction during brazing. As a result, even when the interlayer becomes liquid, bonding with the aluminium substrate may remain limited if the oxide is not sufficiently removed or reduced. For this reason, the native oxide layer is one of the main obstacles in brazing aluminium-containing systems [25].

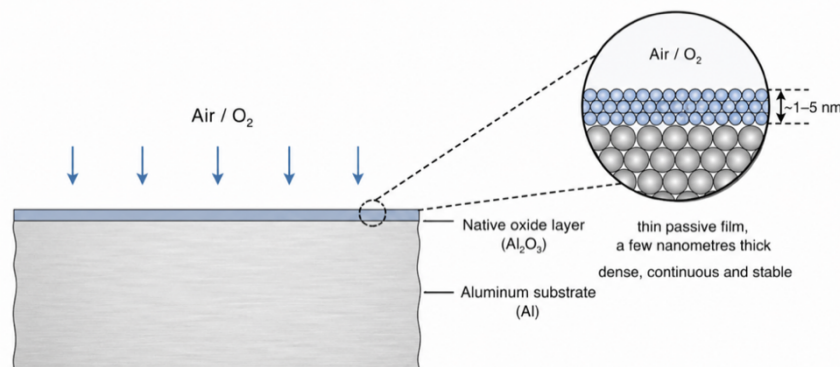


Figure 12. Schematic representation of the native oxide layer naturally formed on the aluminium surface [25]

2.4.2 Chemical treatments: HNO_3 and KOH

Chemical surface treatments are commonly used before brazing of aluminium-containing systems in order to modify the native oxide layer, remove surface contaminants, and increase the reactivity of the aluminium surface [7]. Since aluminium oxide is chemically stable and acts as a barrier to metallurgical interaction, surface preparation is necessary to improve the possibility of bonding between the aluminium substrate and the molten interlayer [25].

Alkaline treatments such as KOH can attack the aluminium surface and partially dissolve the native oxide film. This process may expose fresh aluminium and increase surface roughness, which can improve the interaction with the liquid filler or interlayer during joining. However, excessive alkaline etching may also produce an irregular surface morphology and non-uniform surface activation, which can negatively affect the continuity of the interface.

Acid treatments such as HNO_3 are also used for aluminium surface preparation because they can remove contaminants and modify the surface oxide condition before joining. Compared with strong alkaline etching, acid treatment can provide a cleaner and more uniform surface condition when properly controlled. The effectiveness of this treatment depends on concentration, exposure time, rinsing, and drying conditions, since any remaining oxide, moisture, or contamination may affect the subsequent interfacial reaction.

Therefore, chemical treatments before brazing should be considered not only as cleaning steps, but also as surface activation procedures. Their role is to improve the availability of reactive aluminium surface, support interaction with the molten Zn-rich phase, and reduce the negative effect of the stable native oxide layer during interfacial bonding [24,25].

2.4.3 Effect of surface condition on interfacial reactivity

The surface condition of aluminium directly affects wetting, diffusion, and interfacial layer formation [24]. If the oxide barrier remains too stable, the molten zinc cannot interact effectively with the aluminium substrate, and the resulting bond may remain weak or discontinuous. In contrast, a better-activated surface promotes more effective Al–Zn interaction and more developed transition regions. Both the previous thesis and the present work therefore indicate that surface preparation is a key factor controlling the continuity and quality of brazed Al / Zn-coated steel joints [25].

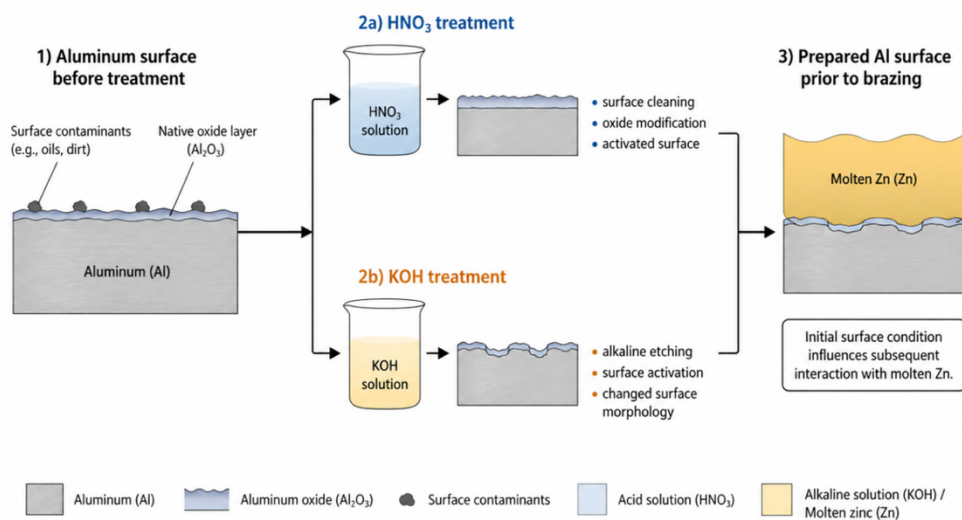


Figure 13. Schematic representation of chemical treatments of aluminium surface prior to brazing (schematic created by the author based on Refs. [7,9,25])

2.5 Summary of literature

The reviewed literature covers several approaches for joining aluminium alloys to steel, including laser welding–brazing, MIG/CMT welding–brazing, ultrasonic plus resistance spot welding, and fluxless soldering approaches using zinc-based interlayers. Although these studies differ in materials, surface conditions and joining processes, they provide useful information about the main factors controlling Al–steel and Al / Zn-coated steel joining. Table 1 summarizes the most relevant works and compares their materials, process conditions, main findings and relevance to the present thesis.

Table 1. Critical comparison of literature relevant to Al/Zn-coated steel brazing

Ref.	Source	Materials / system	Joining method / filler	Surface / coating condition	Key process observations	Main findings	Critical relevance
[1]	Yang et al., 2022	Aluminium alloys / steels	Laser keyhole welding, laser welding–brazing, laser-arc hybrid welding, laser-assisted joining and related processes	Different Al–steel configurations; discussion includes coatings, filler/interlayers and interface control	Review of laser-based joining conditions, wetting/spreading, interfacial IMCs, welding defects and joint strength	Al–steel joining is limited by thermophysical mismatch, low metallurgical compatibility, brittle Fe–Al IMCs, porosity, cracking and heat-input sensitivity.	Strong general background for the introduction/state of the art. It explains why controlled heat input, IMC control and defect limitation are essential.
[2]	Selvamani, 2022	Aluminium alloys / steels	MIG, TIG, laser welding, FSW, FSSW, CMT, explosive welding, hybrid welding and other processes	Different surface/coating conditions depending on the joining method	Summarises the role of process parameters and interfacial phenomena in many Al–steel joining methods	Confirms that Al–steel joining is challenging because of melting-point differences, thermophysical mismatch, IMC formation, defects and sensitivity to process parameters.	Useful as broad comparative background. It supports why the thesis focuses on a low-temperature, Zn-mediated route rather than conventional fusion welding.
[3]	Lu et al., 2020	AA6022-T4 / GA Zn-coated DP980 steel; AA3003 insert	Ultrasonic spot welding followed by resistance spot welding (U+RSW)	Galvannealed Zn coating on DP980 steel; AA3003 insert between Al and steel	1.2 mm AA6022-T4; 1 mm GA DP980; RSW at 17 kA gave peak tensile shear strength 3.7 kN and fracture energy 1.16 J	During USW, multilayer Zn–Fe IMCs from the original coating and a new Al–Zn diffusion layer formed; during RSW these layers melted/diluted and a new Al–Fe IMC layer formed.	Very relevant for showing that Zn coating actively changes the interfacial microstructure and can create Al–Zn/Zn–Fe layers before Al–Fe reactions dominate.
[4]	Dharmendra et al., 2011	AA6016-T4 / hot-dip zinc-coated DP600 steel	Laser welding–brazing with Zn–15Al filler wire	Hot-dip Zn-coated DP600 steel; surfaces cleaned with acetone; no flux used	Best joints: heat input 60–110 J/mm, brazing speed 0.5–0.8 m/min; reaction layer 3–23 μm ; optimal layer about 8–12 μm ; strength about 220 MPa	Shows that AA6016 can be joined to Zn-coated DP600 using a low-melting Zn–Al filler. Layer thickness depends on heat input and brazing speed; optimal joints failed on Al side rather than at the interface.	Closest external literature to this thesis because it uses AA6016, Zn-coated steel and a Zn-based joining medium. It gives quantitative benchmark values for reaction-layer thickness and mechanical resistance.

[6]	Cao et al., 2018	Al alloy / galvanised steel and Mg alloy / galvanised steel	CMT welding–brazing; dynamic observation of Zn coating behaviour	Galvanised steel compared with bare steel	For Al–galvanised steel, contact angle around 35° compared with around 55° for Al–bare steel; Zn-rich triangular zone could reach low contact angle locally	Zn coating improves wettability of liquid Al on steel and affects spreading behaviour. The behaviour of Zn is dynamic and locally complex; Zn may burn off, redistribute, or concentrate near the bead toe.	Important for explaining why Zn is beneficial but not automatically sufficient for a uniform interface. It supports discussion of Zn redistribution and local heterogeneity.
[14]	Zhang et al., 2007	Aluminium / zinc-coated steel	Modified MIG welding–brazing	Zinc-coated steel	Interfacial microstructure and mechanical properties analysed as a function of welding–brazing conditions	Shows that the interfacial layer morphology and joint properties depend strongly on process conditions and the reaction layer formed between aluminium and zinc-coated steel	Very relevant because it concerns aluminium joined to zinc-coated steel by welding–brazing and supports the discussion of reaction-layer control and interface quality
[31]	Wan et al., 2015	Aluminium foam / aluminium face sheets	Fluxless soldering with Zn-based solder and surface abrasion assisted by vibration	Aluminium surfaces mechanically abraded; fluxless joining; Zn-based solder	Ultrasonic vibration improved uniformity, reduced seam defects and increased bending strength up to 571% of aluminium foam reference value	Shows that oxide removal/surface abrasion and mechanical assistance can improve wetting, spreading and metallic bonding in fluxless Zn-based joining of aluminium systems.	Indirect but useful for surface preparation and fluxless Zn-based bonding concepts. Not a direct Al–steel reference.
[32]	Wan et al., 2015	Aluminium foam / aluminium alloy face sheets	Fluxless soldering with self-abrasion using Zn-based alloy	Mechanical self-abrasion used to assist oxide disruption and spreading	Self-abrasion helped remove oxide film, improved wetting/spreading of molten filler and produced excellent metallic bonding	Supports the idea that oxide disruption and surface activation are critical in fluxless joining of aluminium with Zn-based liquid phases.	Indirect relevance. Useful for surface activation/oxide removal theory, but not a direct Al–steel or Zn-coated steel comparison.

Among the reviewed sources, the work of Dharmendra et al. [4] is the closest external reference because it considers AA6016 joined to zinc-coated DP600 steel using a Zn-based filler. Lu et al. [3] and Cao et al. [6] further confirm that zinc actively modifies the joining mechanism and affects the formation of Al–Zn, Zn-rich and Zn–Fe interfacial regions. Other works, such as Wan et al. [31,32], are less directly related to Al–steel joining, but they support the importance of oxide disruption and surface activation in fluxless Zn-based joining systems.

Overall, the literature indicates that Al / Zn-coated steel brazing depends on a combined balance between surface preparation, zinc activation, thermal conditions, local contact and interfacial layer growth. The research gap addressed in this thesis is therefore the need to better understand how these factors influence the continuity, morphology and chemical heterogeneity of the Zn-mediated interfacial region in brazed Al / Zn-coated steel joints.

3. Materials and Sample Preparation

3.1 Materials

3.1.1 Base materials

The aluminium base material used in this work was Al6016 sheet. The specimens were prepared as square plates with dimensions of 1 cm × 1 cm and a thickness of 2 mm. Before brazing, the Al6016 surface was mechanically ground using #320 abrasive paper and then subjected to chemical treatment. The aluminium side was the substrate on which the KOH and HNO₃ treatments were applied to modify the initial surface condition before joining.

Zinc-coated steel sheet was used as the second base material. The specimens were also prepared as square plates with dimensions of 1 cm × 1 cm and a thickness of 1.5 mm. Before assembly, the zinc-coated steel was mechanically ground using #4000 abrasive paper.

Zinc foil was used in this work as the brazing interlayer between Al6016 and zinc-coated steel. The foil was prepared with dimensions of 1 cm × 1 cm, while its thickness was below 1 mm, in the range of approximately 0.5 - 0.7 mm. In the adopted assembly, the zinc foil was placed between the two base materials.

3.1.2 Chemical reagents and consumables

The main chemical reagents used in this work were KOH pellets (molar mass 56.11 g/mol), concentrated HNO₃, ethanol, and deionised water for surface preparation and cleaning.

The chemical treatments were applied independently to different aluminium specimens. In particular, the KOH and HNO₃ treatments were not performed sequentially on the same

sample but were used as separate surface preparation routes to evaluate their individual effects on the Al6016 surface prior to brazing.

Ethanol and deionised water were used as auxiliary liquids during surface preparation and cleaning. Ethanol was employed without dilution as a moisture-displacing solvent to assist rapid drying of the treated surface, while deionised water was used for the preparation of the KOH and HNO₃ solutions and for rinsing between treatment steps.

3.2 Equipment used

The equipment used in the present work served specific functions during sample preparation, chemical treatment, embedding, grinding, and polishing:

3.2.1 Sample preparation setup

- **Precision Cutting Equipment** - a metallographic abrasive cutting machine (Remet TR80 Evolution) was used to cut the metallic specimens into flat and reproducible geometries suitable for metallographic mounting. In this work, each metallic sheet was prepared with 1 cm × 1 cm geometry.
- **Mounting Moulds** - two cylindrical mounting moulds were used to position the metallic samples centrally and to maintain stable orientation during subsequent preparation steps. The mould defines the final shape of the mounted specimen and keeps the sample in a fixed orientation while the resin cures.
- **Analytical balance** was used for accurate mass measurements during epoxy and chemical solution preparation.
- **Polypropylene mixing cups** were used as containers for preparing the epoxy mixture. A clean cup was placed on the analytical balance and tared before adding resin and hardener.
- **Wooden Spatula/Stirrer** was used to mix the epoxy resin and hardener until a homogeneous mixture was obtained.



Figure 14. Laboratory equipment for sample preparation - Cutting (Remet TR80 Evolution) and Grinding/Polishing machines used for metallographic preparation

3.2.2 Chemical treatment setup:

- **Chemical-Resistant Glass Beakers** were used to contain KOH, HNO₃, ethanol, and water during surface preparation procedure. The containers must resist chemical attack and must not contaminate the solutions.
- **Ultrasonic Cleaner/Bath** was used to remove fine particles, residues, and contaminants from the sample surface. Ultrasonic cleaning works through cavitation: high-frequency vibrations generate microscopic bubbles in the liquid, and their collapse helps detach particles from the surface.
- **Compressed Air Gun/Dryer** was used to dry the samples after rinsing, ethanol cleaning, and polishing steps. Drying is critical because remaining water can cause oxidation, staining, or contamination on the surface.

3.2.3 Embedding equipment:

- **Struers EpoFix Cold-Mount Epoxy System** - was used to embed the metallic samples in a rigid resin matrix. It consists of two components:
 - Resin
 - Hardener

Cold mounting is performed at room temperature, which is useful because it avoids thermal damage or microstructural alteration of the sample.



Figure 15. Laboratory chemical preparation area with ventilated hood used for preparation, mixing and handling of the KOH and HNO₃ solutions as well as specimen embedding

3.2.4 Grinding and polishing equipment:

- **Grinding/Polishing Machine with Water supply** – provides controlled rotation and contact between the sample and abrasive paper. Water is used continuously as a lubricant and coolant.

- The subsequent grinding stages employed waterproof SiC abrasive papers from coarse to very fine grain size:
 - P240 (coarse) - removes cutting marks and ensures overall sample flatness
 - P600 (intermediate) - removes P240 scratches and reduces the deformation depth
 - P1200 (fine) - further minimises the deformation layer
 - P2500 (very fine) - final removal of SiC scratches before diamond polishing
- For polishing:
 - Grey polishing cloth was used with 3 μm LAM PLAN BIO DIAMANT diamond suspension
 - Black Akasel cloth was used for the final polishing step with 1 μm diamond suspension.

The 3 μm polishing stage was used to further smooth the surface after grinding, whereas the 1 μm stage was used to obtain a fully reflective, scratch-free surface without a remaining deformation zone.

3.3 Preparation of Chemical Solutions

Before surface treatment, the chemical solutions required for cleaning and activation of the Al6016 surface were prepared in controlled quantities. The preparation of these solutions was carried out using calculated proportions to ensure reproducibility of the chemical treatment conditions.

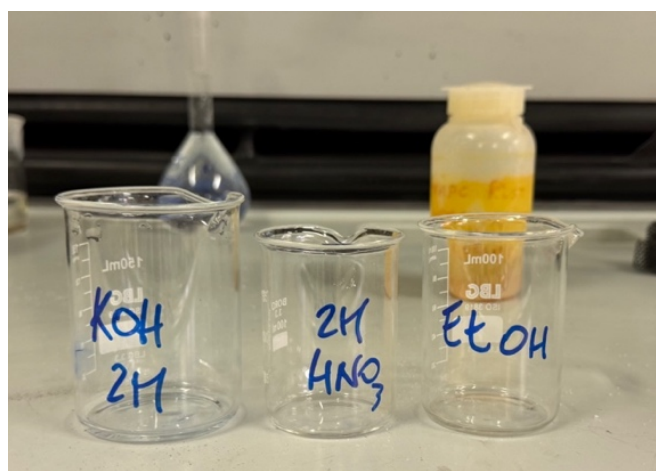


Figure 16. Labelled glass beakers prepared for the chemical treatment sequence, including separate containers for KOH, HNO_3 and ethanol

The solutions were prepared separately and were used as independent surface-treatment routes for different aluminium sheets. The KOH (potassium hydroxide) solution was prepared by dissolving KOH pellets in deionised water, while the HNO_3 solution was prepared by diluting concentrated nitric acid with deionised water.

3.3.1 Preparation of 2M KOH solution

The alkaline solution used in this work was prepared from KOH pellets and deionised water. According to the adopted laboratory procedure, a 2H KOH solution, corresponding approximately to 2M, was prepared. Since KOH is a monobasic base, its normality is equal to its molarity.

Using the molar mass of KOH, 56.11 g/mol, the required mass for 1 L of solution was calculated as:

$$m = C * M * V$$

$$m = 2 \frac{\text{mol}}{\text{L}} * 56,11 \frac{\text{g}}{\text{mol}} * 1\text{L} = 112.22 \text{ g}$$

This value was then scaled to the working laboratory volume of 60 mL:

$$m = 112.22 * 0.060 = 6.7332 \text{ g}$$

Therefore, 6.73 g of KOH was dissolved, and the final volume was adjusted to 60 mL with deionised water.

3.3.2 Preparation of HNO₃ 12% solution

The acidic solution used in this work was prepared from concentrated HNO₃ and deionised water. According to the adopted laboratory reference composition, the standard mixture was:

$$179 \text{ mL HNO}_3 + 821 \text{ mL DI water} = 1000 \text{ mL}$$

To prepare a working volume of 60 mL, the composition was scaled proportionally:

$$V_{\text{HNO}_3} = 179 * 0.06 = 10.74 \text{ mL}$$

$$V_{\text{water}} = 821 * 0.06 = 49.26 \text{ mL}$$

Thus, the final mixture consisted of 10.74 mL of concentrated HNO₃, diluted with deionised water up to a final volume of 60 mL.

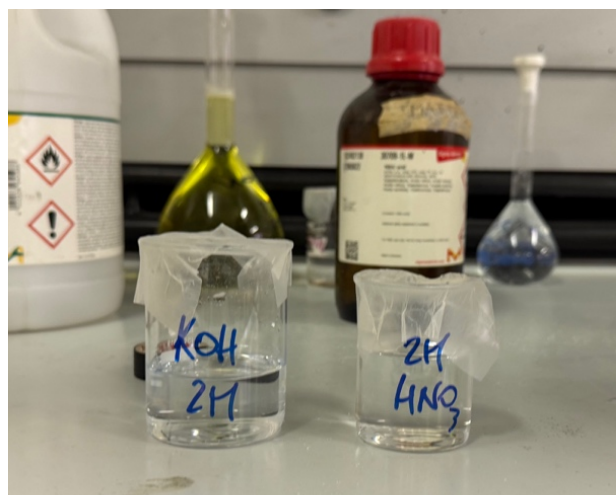


Figure 17. Prepared chemical solutions before application to the Al6016 specimens. Beakers covered with plastic film to minimise contamination and limit evaporation during handling prior to treatment

3.3.3 Use of ethanol and deionised water

Ethanol was used without dilution during the surface preparation procedure. Its main function was to act as a moisture-displacing solvent and to promote rapid drying of the treated aluminium surface. Due to its high volatility, ethanol evaporated quickly and helped reduce the risk of residual moisture remaining on the specimen before the subsequent processing step.

Deionised water was used both for the preparation of the KOH and HNO₃ solutions and for rinsing after each individual chemical treatment. Since the KOH and HNO₃ treatments were applied independently to different aluminium specimens, deionised water was used to remove residual chemicals after either alkaline (KOH) or acid (HNO₃) treatment. The use of deionised water also helped avoid contamination from dissolved ions and ensured that the surface remained clean before further processing.

3.4 Initial preparation of base materials

3.4.1 Cutting and specimen dimensions

The initial preparation of the base materials started with cutting the aluminium and zinc-coated steel sheets into small square specimens suitable for laboratory-scale brazing experiments. The samples were cut from sheet material using a metallographic abrasive cutting machine (Remet TR80 Evolution) equipped with a black alumina blade. This cutting system was used to obtain flat and reproducible specimen geometries before subsequent surface preparation and brazing.

- The Al6016 sheet was prepared as square plates with dimensions: 1 cm × 1 cm and a thickness of approximately 2 mm

- The zinc-coated steel sheet was prepared with the same planar dimensions: $1\text{ cm} \times 1\text{ cm}$, and a thickness of approximately 1.5 mm
- Zinc foil was prepared with dimensions: $1\text{ cm} \times 1\text{ cm}$, while its thickness was below 1 mm, approximately 0.5–0.7 mm

The use of the same in-plane dimensions for both base materials ensured proper overlap and alignment during assembly. The controlled specimen geometry was important for reproducibility, because similar sample dimensions provide a consistent contact area during brazing and allow comparison between different treatment routes.



(a)



(b)

Figure 18. Remet TR80 Evolution metallographic abrasive cutting machine used for preparation of the metallic specimens: (a) with closed protective cover; (b) with open protective cover showing the cutting chamber and specimen holding area

3.4.2 Mechanical grinding of base materials before treatment

Before chemical treatment and brazing, the surfaces of the base materials were mechanically ground to obtain a more uniform and reproducible surface condition. This step was necessary to remove surface irregularities, contamination, weak oxide layers, and marks produced during cutting.

The Al6016 specimens were mechanically ground using #320 abrasive paper. This relatively coarse grinding step was applied to modify the initial aluminium surface and improve the effectiveness of the following chemical treatment.

The zinc-coated steel specimens were mechanically ground using #4000 abrasive paper. A much finer abrasive paper was selected for the steel side to preserve the zinc coating while slightly cleaning and homogenising the surface.

3.4.3 Initial handling and cleaning

After cutting and mechanical grinding, the specimens were cleaned to remove loose metallic particles, abrasive residues, oxide fragments, and surface contamination. This step was necessary to avoid interference with the following chemical treatment and brazing process.

The prepared surfaces were handled carefully to prevent direct contamination. The samples were then placed in ethanol, which acted as a cleaning and moisture-displacing medium. Ultrasonic cleaning was performed for 5 minutes at room temperature to detach fine particles and residues from the surfaces. After ultrasonic cleaning, the specimens were dried using compressed air to remove remaining liquid and reduce the risk of oxidation before brazing. This procedure ensured cleaner surfaces and improved the reproducibility of the Al–Zn–steel joining process.

3.5 Surface preparation before brazing

3.5.1 KOH treatment

The Al6016 specimens assigned to this route were fully immersed in the KOH solution for 5 minutes at room temperature. After the immersion period, the specimens were immediately removed from the solution and rinsed with deionised water to remove remaining alkaline residues from the surface. The samples were then cleaned in ethanol using ultrasonic treatment for 5 minutes. Finally, the specimens were dried using compressed air to reduce oxidation risk and keep the surface clean before brazing.



Figure 19. Immersion of an Al6016 specimen in the KOH solution during alkaline surface treatment before brazing

3.5.2 HNO₃ treatment

The Al6016 specimens assigned to this route were fully immersed in the HNO₃ solution for 5 minutes. After the immersion period, the specimens were immediately removed from the solution and rinsed with deionised water to remove residual acid from the surface. The samples were then cleaned in ethanol using ultrasonic treatment for 5 minutes. Finally, the specimens were dried using compressed air to remove remaining liquid and reduce the risk of oxidation before brazing.



Figure 20. Immersion of an Al6016 specimen in the HNO₃ solution during acid surface treatment before brazing

3.5.3 Water rinsing

Deionised water was used for rinsing after each individual chemical treatment to remove residual chemical species from the specimen surfaces. This step was essential to prevent unwanted chemical interactions and surface contamination.

The use of deionised water was particularly important because it does not contain dissolved ions or impurities that could deposit on the surface. As a result, rinsing ensured that the specimens remained chemically clean and suitable for the following ethanol cleaning and drying prior to brazing.

3.5.4 Ethanol and ultrasonic cleaning

After water rinsing, the specimens were placed in ethanol for final cleaning before brazing. Ethanol was used without dilution and acted as a moisture-displacing solvent, helping to remove residual water from the treated surfaces and promoting faster drying.



Figure 21. *Ultrasonic bath used for ethanol cleaning of the specimens, with a beaker placed inside the bath during the cleaning step*

Ultrasonic cleaning was performed in ethanol for 5 minutes at room temperature. During this step, ultrasonic agitation helped detach fine particles, residues, and small contaminants that could remain on the surface after grinding, chemical treatment, and rinsing.

3.5.5 Drying with compressed air

After ethanol cleaning and ultrasonic treatment, the specimens were dried using compressed air to remove any remaining liquid from the surface. This step was essential to eliminate residual ethanol and prevent moisture retention on the specimens. As a result, the specimens were left clean, dry, and ready for assembly and brazing, ensuring consistent and reproducible surface conditions.

3.5.6 Final surface condition before brazing

The applied surface preparation procedures were intended to modify the initial condition of the Al6016 surface by removing contaminants and reducing the native oxide layer before brazing. Mechanical grinding provided the first level of surface modification by removing surface irregularities and part of the oxide layer formed during exposure to air. Subsequent chemical treatment with either KOH or HNO₃ was used to further clean and activate the aluminium surface according to the selected preparation route. After each treatment, rinsing with deionised water was carried out to remove residual chemical species. Ethanol cleaning, ultrasonic treatment, and drying with compressed air were then used to remove residual moisture and fine surface contaminants before assembly and brazing.

3.5.7 Surface roughness measurement by contact profilometry

In order to characterise the surface topography of the specimens before brazing, surface roughness measurements were performed by contact profilometry. A Taylor Hobson INTRA contact profilometer connected to dedicated acquisition and analysis software was used for this purpose. Measurements were carried out on untreated and chemically treated aluminium and zinc-coated steel surfaces.

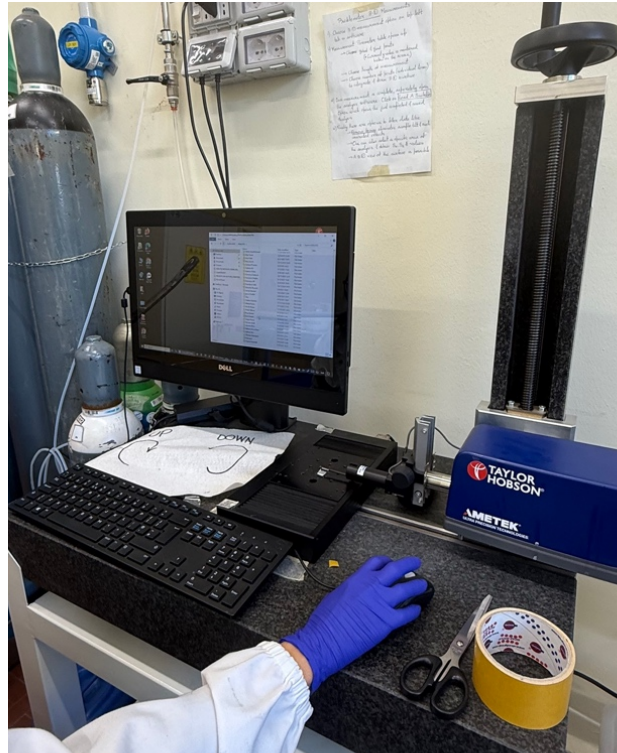


Figure 22. *Taylor Hobson INTRA contact profilometry setup*

Before measurement, the specimens were cleaned, dried, and positioned on the profilometer stage. Roughness measurements were performed on line scans measuring at least 3 lines per sample. During measurement, the stylus moved across the specimen surface and recorded the height variations of the profile.

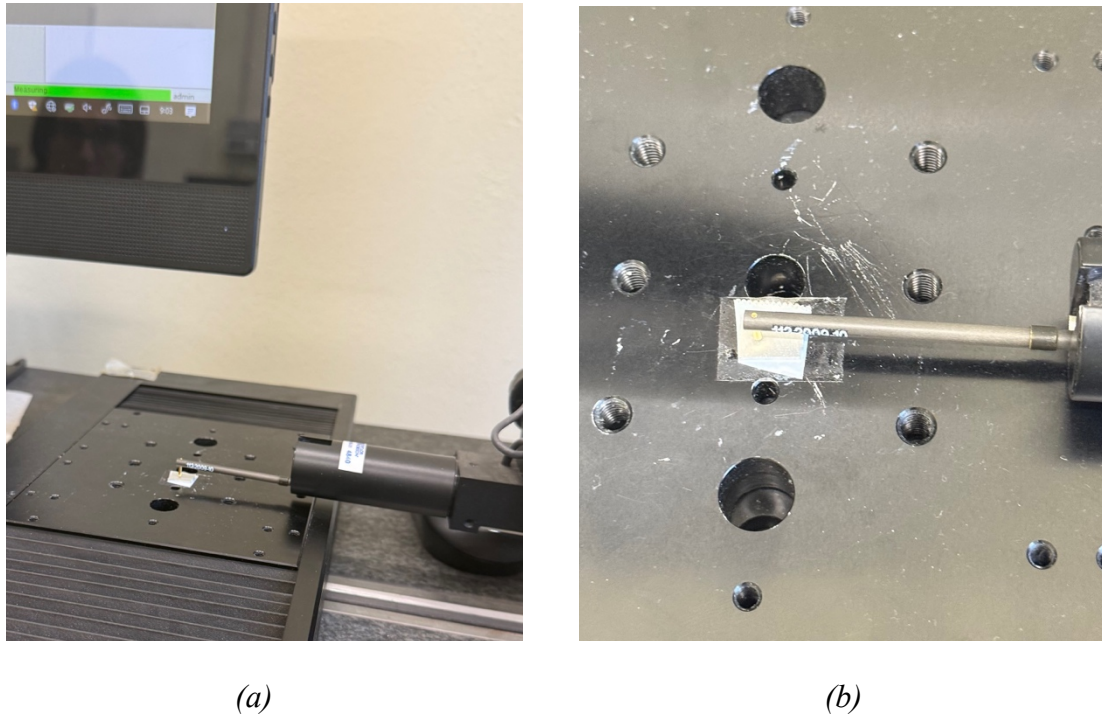


Figure 23. Contact profilometry setup: (a) specimen positioned on the profilometer stage before measurement; (b) close-up view of the stylus in contact with the specimen surface during scanning

After scanning, the acquired data were processed in the analysis software. A form removal operation was applied to eliminate the effect of specimen tilt and other geometric deviations before roughness evaluation. Representative surface regions were then selected for analysis, and the corresponding roughness parameters were calculated.

To improve the reliability of the results, measurements were performed on several representative surface regions. The obtained data were used to characterise the surface condition of the specimens after preparation and before brazing.

3.6 Brazing configuration and sample assembly

Before brazing, the prepared Al6016, zinc foil, and zinc-coated steel specimens were assembled into a multilayer configuration. The purpose of this assembly was to create direct contact between the base materials and the zinc interlayer, allowing zinc to melt during heating and act as the brazing filler.

The brazing configuration was based on a sandwich arrangement. In the adopted setup, the zinc-coated steel specimen and the Al6016 specimen were separated by the zinc foil interlayer. The zinc foil was positioned between the two base materials so that, after melting, it could spread along the interface and promote bonding between aluminium and zinc-coated steel.

3.6.1 Sandwich configuration

In the adopted sandwich configuration, the Al6016 specimen was positioned at the bottom, while the zinc-coated steel specimen was positioned at the top. The zinc foil was placed between them as the brazing interlayer. Thus, the final metallic stack was arranged as:

Zn-coated steel / Zn foil / Al6016

In the furnace assembly, this metallic stack was placed on a graphite plate to maintain alignment and stable positioning during heating.

The graphite plate was used to support the sample assembly, prevent direct sticking to the furnace support, and help reduce local thermal gradients during brazing. This configuration also protected the external surfaces of the specimens and helped maintain the alignment of the stack during heating.

Correct alignment of the sandwich structure was essential. The steel, zinc foil, and aluminium specimens were prepared with similar planar dimensions, allowing the interlayer to remain properly positioned between the two base materials. This ensured a consistent contact area and improved reproducibility between different brazing trials.

3.6.2 Position of aluminium and steel sheets

In the adopted assembly, the Al6016 sheet was positioned at the bottom, while the zinc-coated steel sheet was positioned at the top. The zinc foil was placed directly between the two base materials and acted as the brazing interlayer.

During assembly, particular attention was given to the orientation of the treated aluminium surface. The prepared Al6016 surface was placed in direct contact with the zinc foil, so that molten zinc could interact with the activated aluminium surface during heating. Similarly, the zinc-coated side of the steel was positioned toward the zinc foil to promote contact between the zinc coating and the molten zinc interlayer.

This arrangement ensured that, during brazing, molten zinc was located between the aluminium and zinc-coated steel surfaces. The positioning of steel at the top and aluminium at the bottom also helped keep the stack orientation consistent between different brazing trials.

3.6.3 Use of graphite plate

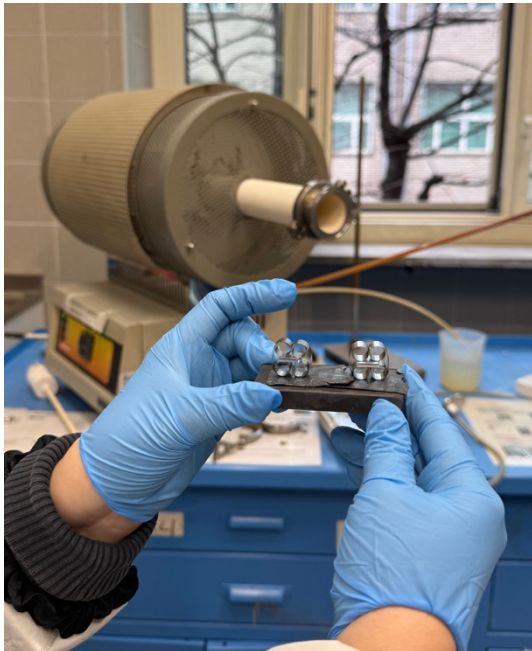
A graphite plate was used in the brazing setup to support the sample assembly during thermal processing. The assembled stack (zinc-coated steel / Zn foil / Al6016) was placed on the graphite plate to maintain alignment and ensure stable positioning during heating.

The use of graphite provided several advantages. Due to its high thermal stability and resistance to high temperatures, graphite remained chemically and structurally stable during the brazing process. Additionally, its relatively low reactivity with the base materials minimised the risk of contamination or unwanted reactions at the specimen surfaces.

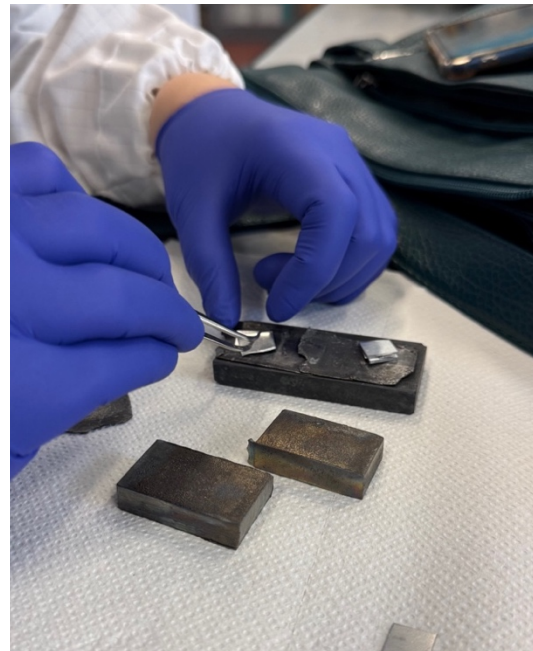
The graphite plate also contributed to a more uniform thermal condition across the sample assembly. This helped reduce temperature gradients and supported consistent melting of the zinc interlayer. Furthermore, the plates assisted in maintaining a controlled contact between the layers, which is important for achieving uniform wetting and bonding during brazing.

Another important function of the graphite plates was to prevent direct contact between the specimens and the furnace support. This reduced the risk of sticking, surface damage, or contamination during heating.

Overall, the use of the graphite plate ensured mechanical stability, thermal uniformity, and clean processing conditions, contributing to improved reproducibility of the brazing experiments.



(a)



(b)

Figure 24. Graphite plate employed in the brazing assembly: (a) graphite support element used during sample preparation; (b) positioning of the metallic stack on the graphite plates prior to clamping and brazing

3.6.4 Use of steel spring clamps

Steel spring clamps were used as mechanical fixing elements to secure the assembled specimens during the brazing process. After arranging the sandwich configuration (zinc-coated steel / Zn foil / Al6016) between graphite plates, the assembly was fixed with these clamps in order to maintain contact between the stacked layers during handling and heating.

The main function of the clamps was to keep the sandwich assembly compressed during heating and to reduce possible displacement or misalignment between the aluminium, zinc foil and zinc-coated steel layers. Maintaining contact between the layers was important because gaps or poor alignment could limit wetting, reduce interfacial interaction and lead to discontinuities in the brazed joint.

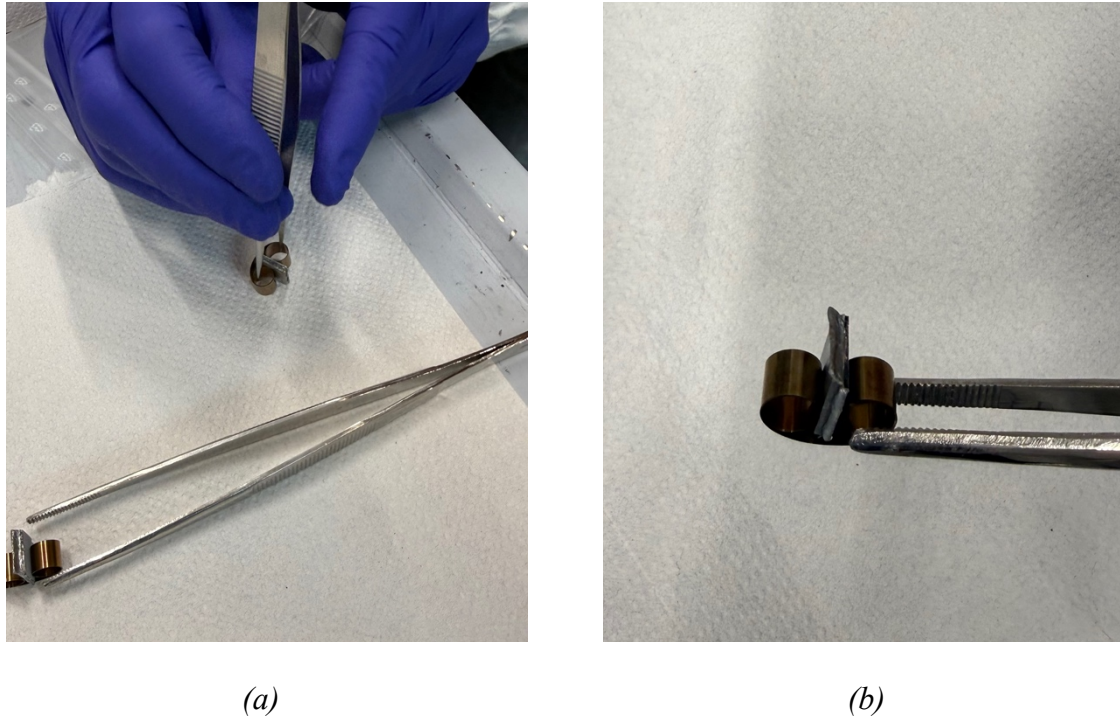


Figure 25. Steel spring clamps employed to secure the sandwich assembly during brazing: (a) clamp handling during sample preparation; (b) detailed view of the clamp used to maintain contact between the stacked layers

However, the clamping system also introduced some limitations. Although the springs helped maintain compression during heating, thermal contraction during cooling could generate tensile stresses at the interface. Since aluminium, zinc and steel have different thermal expansion behaviour, cooling after brazing may produce local stresses and partially compromise the continuity of the joint, especially if the contact pressure is not uniformly distributed.

Therefore, the steel spring clamps were used only as mechanical fixing elements to stabilise the assembly and maintain contact during heating. No specific metallurgical role was attributed to the clamp material. Possible non-uniform pressure distribution and thermal stresses during cooling were considered as potential factors contributing to local defects, variations in joint thickness or partial loss of interfacial continuity.

3.6.5 Possible limitations related to pressure uniformity and alignment

Although the use of steel spring clamps provided a practical method for maintaining contact during brazing, several limitations related to pressure distribution, alignment and cooling stresses must be considered.

Firstly, the applied pressure was not perfectly uniform across the entire specimen surface. Spring clamps generate localised forces depending on their contact points, which can lead to variations in pressure distribution. As a result, some regions of the assembly may experience higher contact pressure, while others may have reduced contact. This non-uniformity can influence the flow and distribution of the molten zinc interlayer, potentially leading to uneven joint thickness or localised defects such as voids.

Secondly, slight misalignment of the aluminium, zinc foil and zinc-coated steel sheets could occur during manual assembly and clamping. Even small deviations in positioning may affect the overlap area, local gap size and continuity of the interface. This can directly influence wetting behaviour and the formation of the brazed joint.

Additionally, after brazing, thermal contraction during cooling may generate tensile stresses at the interface. Since aluminium, zinc and steel have different thermal expansion behaviour, cooling can produce local stresses and slight dimensional mismatch between the joined layers. These stresses may contribute to partial loss of interfacial continuity, especially in regions where the contact pressure was not uniform.

Overall, the clamping system ensured sufficient contact during heating, but its limitations in controlling uniform pressure, precise alignment and cooling-induced stresses should be recognised as possible sources of variability in the experimental results.

3.7 Brazing procedure and process parameters

After completion of surface preparation and assembly, the specimens were subjected to brazing under controlled thermal conditions. The brazing procedure was designed to allow melting of the zinc interlayer while maintaining the aluminium and steel base materials in the solid state. For this purpose, the assembled stacks were heated in a tube furnace under argon atmosphere, and the main process parameters were defined in terms of furnace environment, brazing temperature, and dwell time. Different trial conditions were then applied in order to evaluate the influence of the selected thermal parameters on the brazing process.

3.7.1 Furnace and argon atmosphere

Brazing was carried out in a tubular furnace (Carbolite, Hope Valley, UK) operated under argon atmosphere. The assembled stack was placed at the centre of the furnace in order to ensure more uniform heating, since the temperature near the sides of the furnace may differ from the programmed value. The furnace was connected to an argon cylinder to maintain inert gas flow during the brazing cycle, and the atmosphere inside the chamber was controlled throughout heating to limit exposure to oxygen.

The use of argon was necessary to reduce oxidation of both the activated aluminium surface and the molten zinc interlayer at elevated temperature. In the adopted procedure, argon acted as an inert shielding gas that displaced atmospheric oxygen from the furnace chamber and helped maintain a more stable thermal environment during brazing. The furnace setup was selected for operation at temperatures up to 480 °C, corresponding to the thermal conditions used in the present work.

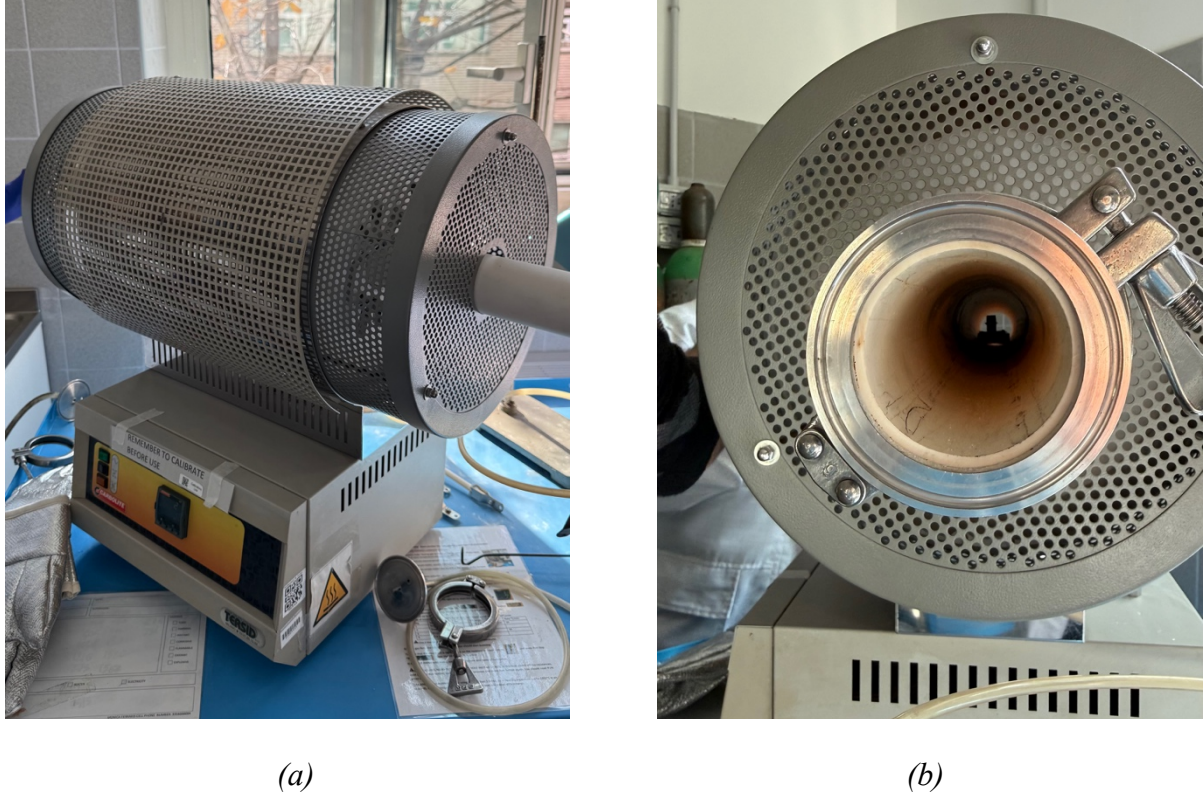


Figure 26. Carbolite tubular furnace used in the brazing experiments: (a) external view of the furnace setup; (b) front view of the furnace chamber showing the sample stack placed inside under the adopted brazing configuration

3.7.2 Brazing temperature and dwell time

The brazing temperature and dwell time were selected so that the zinc interlayer could melt and spread at the interface while the aluminium and zinc-coated steel base materials remained in the solid state. The chosen temperature range was particularly relevant for the Al–Zn–steel system because it was above the melting point of zinc (approximately 419–420 °C) but well below the melting point of aluminium (approximately 660 °C). Under these conditions, molten zinc could act as the brazing interlayer and promote interfacial interaction between Al6016 and zinc-coated steel without melting the substrates.

In the present work, the dwell time at the target temperature was kept constant at 8 minutes for all brazing trials, while temperature was used as the main variable. Trial 1 was carried out at 480 °C, Trial 2 at 470 °C, and Trial 3 again at 480 °C in order to repeat the reference thermal condition. Thus, Trial 2 was introduced to evaluate the effect of reduced thermal

input, whereas Trial 3 was used to assess repeatability under the same nominal conditions as Trial 1.

Thus, the main variable between the trials was the selected thermal condition, while the general assembly route and furnace environment were kept consistent. These controlled conditions were used to enable comparison between the different brazing trials before subsequent microstructural evaluation.

3.7.3 Trial 1

In Trial 1, the assembled Al6016 / Zn foil / zinc-coated steel stack was brazed after the selected surface preparation procedure had been completed. The specimen was positioned in the adopted sandwich configuration and introduced into the furnace under argon atmosphere. The brazing treatment was carried out at 480 °C with a dwell time of 8 minutes. Under these conditions, the zinc interlayer was expected to melt and redistribute along the interface between aluminium and zinc-coated steel while the base materials remained solid.

This trial served as one of the reference experimental conditions for evaluating the effect of the selected processing route on joint formation and interfacial development.

3.7.4 Trial 2

In Trial 2, the assembled Al6016 / Zn foil / zinc-coated steel stack was brazed after completion of the selected surface preparation procedure. The specimen was arranged in the adopted sandwich configuration and placed in the furnace under argon atmosphere. The brazing treatment was carried out at 470 °C with a dwell time of 8 minutes. Under these conditions, the zinc interlayer was expected to melt and spread at the interface, while the lower processing temperature allowed evaluation of the effect of reduced thermal input on joint formation.

This trial was used to compare the influence of brazing temperature with respect to the other experimental conditions and to assess its effect on interfacial development and continuity of the brazed joint.

3.7.5 Trial 3

In Trial 3, the assembled Al6016 / Zn foil / zinc-coated steel stack was brazed after completion of the selected surface preparation procedure. The specimen was arranged in the adopted sandwich configuration and placed in the furnace under argon atmosphere. The brazing treatment was carried out at 480 °C with a dwell time of 8 minutes.

This trial was used as an additional experimental condition to compare the effect of the selected preparation and brazing route on joint formation and interfacial development.

3.8 Metallographic preparation of brazed samples

Prior to microstructural analysis, the brazed specimens were subjected to a controlled metallographic preparation procedure in order to obtain high-quality cross-sections of the Al/Zn/steel joints. The objective of this preparation was to expose the interfacial region with sufficient clarity and surface integrity, enabling reliable characterisation of joint morphology, interlayer distribution, and possible defects.

The preparation sequence included sectioning, epoxy mounting, grinding, and polishing. Each step was carefully performed to minimise the introduction of preparation-induced artefacts such as surface deformation, smearing of the zinc interlayer, or edge rounding. Particular attention was given to preserving the integrity of the dissimilar materials and the relatively soft Zn phase, ensuring that the final prepared surface accurately represented the actual microstructure of the brazed joint.

3.8.1 Sectioning

The brazed specimens were sectioned to obtain cross-sections perpendicular to the joining interface, allowing direct observation of the aluminium, zinc interlayer, and zinc-coated steel across the joint thickness.

Sectioning was carried out using a precision cutting machine equipped with a suitable cutting blade to minimise mechanical deformation and thermal damage. Cooling was applied during the cutting process to prevent overheating, which could alter the microstructure, particularly in the low-melting zinc interlayer.

Special care was taken to ensure that the cutting plane passed through the central region of the joint, providing a representative cross-section for subsequent metallographic preparation and microstructural analysis.

3.8.2 Epoxy mounting

After sectioning and initial cleaning, the specimens were mounted in epoxy resin to facilitate handling and provide mechanical stability during subsequent grinding and polishing. Mounting was necessary because the brazed samples contained dissimilar materials with different hardnesses, including the relatively soft Zn interlayer, which required additional support to preserve the joint edges and interfacial region during mechanical preparation.

Cold mounting was performed using the Struers EpoFix two-component epoxy system, consisting of EpoFix resin and EpoFix hardener. This system was selected because it cures at room temperature, avoiding thermal alteration of the brazed joint, and provides a rigid support with minimal shrinkage.

Before embedding, the specimens were cleaned to remove loose particles and surface debris. The samples were then placed centrally inside cylindrical mounting moulds, with the cross-section of interest properly oriented for later grinding and polishing. Correct positioning was

important to ensure stable orientation and uniform exposure of the Al/Zn/steel interface during preparation.



(a)



(b)

Figure 27. Struers EpoFix cold-mount epoxy system used for specimen mounting during metallographic preparation: (a) EpoFix resin; (b) EpoFix hardener.

The epoxy mixture was prepared using the laboratory ratio of 25 g EpoFix resin and 3 g EpoFix hardener, giving a total mixture mass of 28 g. A clean polypropylene cup was placed on the analytical balance and tared before weighing the resin and hardener. The two components were then mixed carefully for several minutes using a wooden spatula to obtain a homogeneous mixture while minimising air bubble entrapment.

The freshly prepared epoxy mixture was poured into the moulds until the specimens were fully covered and embedded in the resin matrix. The moulds were then left undisturbed at room temperature until complete polymerisation occurred. After curing, the hardened mounts were removed from the moulds and prepared for the following grinding and polishing steps.

This mounting step ensured stable handling, protected the edges of the brazed interface, and helped preserve the integrity of the Al/Zn/steel joint during metallographic preparation.

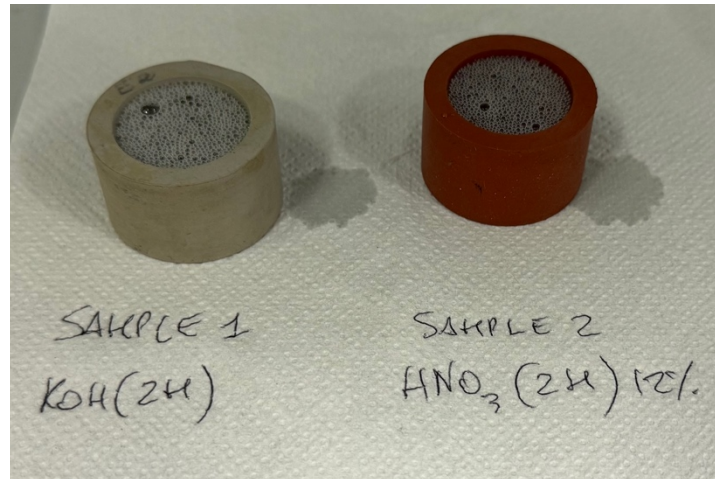


Figure 28. Specimens immediately after pouring of the epoxy mixture into cylindrical moulds
(Ex: Al6016 specimens after KOH and HNO₃ treatment)

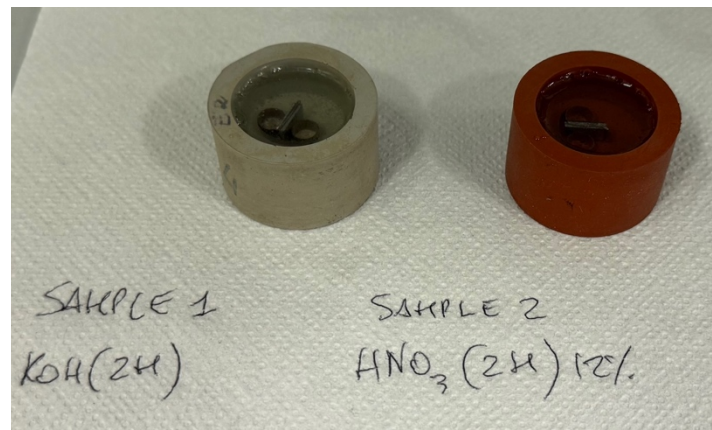


Figure 29. Specimens after curing of the epoxy mixture into cylindrical moulds
(Ex: Al6016 specimens after KOH and HNO₃ treatment)

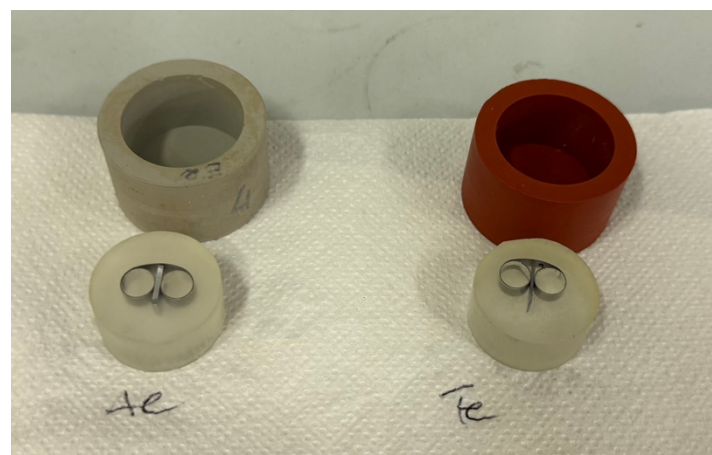


Figure 30. Mounted specimens after removal from the moulds following curing of the epoxy mixture (Ex: pristine specimens of Al6016 and zinc-coated steel)

3.8.3 Grinding

After epoxy mounting, the samples were ground to obtain a flat and uniform cross-sectional surface suitable for further polishing and microscopic observation. The purpose of grinding was to remove cutting marks, reduce surface deformation, and progressively expose the Al–Zn–steel joint region without damaging the interface.

Grinding was performed using waterproof SiC abrasive papers with progressively finer grit sizes: P240, P600, P1200, and P2500. These papers were used sequentially in order to gradually refine the mounted surface and reduce the depth of scratches introduced by the previous step. All grinding stages were carried out under wet conditions using water lubrication, which helped remove debris, reduce frictional heating, and prevent abrasive particles from being embedded in the sample surface.

The first grinding step was performed using P240 abrasive paper. This coarse step was used to remove cutting marks and ensure overall flatness of the mounted sample. It was carried out for approximately 1 minute with continuous water flow and moderate pressure. A figure-eight movement was used during grinding to avoid directional grooves and promote uniform material removal.



Figure 31. P240 waterproof SiC abrasive paper box used in the first grinding step

The second step was performed using P600 abrasive paper for approximately 3 minutes. This stage removed the scratches produced by the P240 paper and reduced the deformation layer. Lower pressure was applied compared to the first step. Before moving to the next grit size, the surface was checked to ensure that the coarse P240 scratches had been fully removed.

The third step used P1200 abrasive paper for approximately 3 minutes. This fine grinding stage further reduced the deformation layer and prepared the surface for the final grinding step. Light pressure and water lubrication were used to minimise additional surface damage.

The final grinding step was performed using P2500 abrasive paper for approximately 3 minutes with very light pressure. This very fine grinding stage was used to remove the remaining SiC scratches before diamond polishing. During this step, the sample was rotated by 90° relative to the previous grinding direction in order to identify and eliminate directional marks.

Between grinding stages, the sample was cleaned carefully to prevent contamination by abrasive particles from the previous step. This included rinsing with water, cleaning with ethanol, drying with an air gun or dryer, and wiping with lint-free tissue when necessary. This cleaning procedure was important because remaining abrasive particles could produce new scratches during the following grinding stages.

Microscopic inspection was used during preparation to check scratch direction, confirm removal of previous scratches, verify surface flatness, and detect possible pull-outs or pits. This ensured that the Al–Zn–steel interface remained clearly visible and that the metallurgical features were preserved before polishing.

3.8.4 Polishing

After completion of the grinding stages, a multi-step polishing procedure was carried out to obtain a deformation-free, mirror-like surface suitable for microstructural characterisation of the Al–Zn–steel interface.

The polishing process was performed using diamond suspensions of decreasing particle size. Initially, a 3 μm diamond suspension was applied on a medium-hard polishing cloth. This step was conducted for approximately 3 minutes using moderate-to-low pressure, ensuring uniform material removal and elimination of fine grinding scratches. Adequate lubrication was maintained to prevent overheating and particle agglomeration.

Following this stage, the samples were thoroughly cleaned to avoid cross-contamination. The cleaning sequence included rinsing with water, followed by ethanol cleaning, ultrasonic treatment for approximately 5 minutes, and drying using compressed air. This procedure was essential to remove residual abrasive particles that could otherwise introduce new surface defects in subsequent steps.

Final polishing was then performed using a 1 μm diamond suspension on a fine polishing cloth (Akasel type). This step was extended to approximately 15 minutes under very low-pressure conditions. The objective of this stage was to completely eliminate any remaining deformation layer and achieve a highly reflective surface finish.

The effectiveness of the polishing procedure was verified through microscopic inspection. A successful result was defined by:

- absence of visible scratches from previous steps
- uniform surface reflectivity
- no pull-outs or embedded particles
- clear visibility of the metallurgical interface

This preparation was critical to ensure accurate observation of interfacial features such as diffusion layers, intermetallic compounds, and possible defects during optical and SEM analysis.

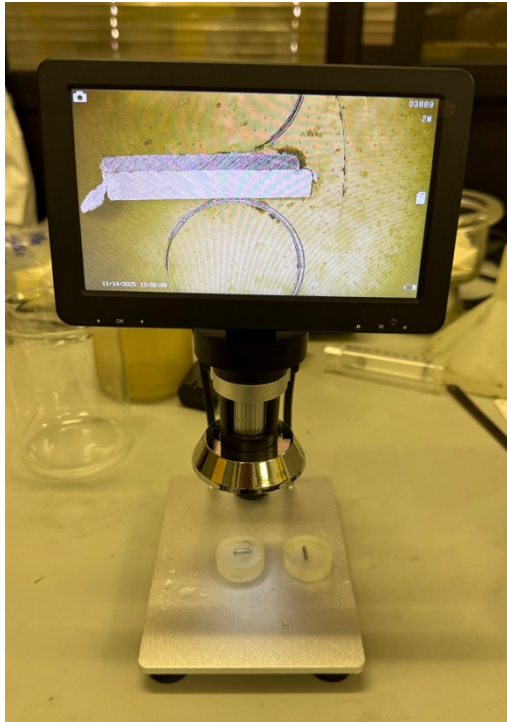


Figure 32. *Portable digital microscope used for preliminary visual inspection of the specimen surface after polishing*

3.8.5 Possible preparation-induced artefacts

During metallographic preparation of the brazed Al–Zn–steel samples, several artefacts may be introduced as a result of mechanical, chemical, or thermal effects occurring during cutting, mounting, grinding, and polishing. These artefacts must be carefully considered, as they can influence the interpretation of the microstructure at the interface.

One of the most common artefacts is surface deformation and smearing, particularly in softer phases such as aluminium and zinc. During grinding and polishing, these materials may plastically deform and spread over adjacent regions, potentially obscuring the true morphology of the interface or thin intermetallic layers.

Another important issue is the formation of pull-outs or void-like features, especially at phase boundaries. Differences in hardness between aluminium, zinc, and steel can lead to preferential removal of softer or weakly bonded regions, creating artificial cavities that may be mistaken for real defects.

Edge rounding can also occur during polishing, particularly at the Al–Zn–steel interface. This effect reduces the sharpness of phase boundaries and may alter the apparent thickness of interfacial layers, making quantitative analysis less accurate.

Additionally, scratches from insufficient grinding or improper cleaning between steps may remain on the surface. If not fully removed, these scratches can interfere with microscopic observations and be misinterpreted as microstructural features.

Contamination artefacts may arise from residual abrasive particles, polishing media, or debris trapped on the surface. These can introduce foreign features or affect contrast during optical or SEM analysis.

Thermal and mechanical stresses introduced during preparation may also lead to microcracking, particularly in brittle intermetallic compounds formed at the interface. Such cracks may not represent the actual brazing condition but rather damage induced during sample preparation.

To minimise these artefacts, careful control of grinding pressure, proper cleaning between stages, appropriate selection of polishing parameters, and continuous microscopic inspection were applied throughout the preparation process. As a result, the observed microstructures can be considered representative of the actual brazed joint with minimal preparation-induced distortion.

3.9 Microstructural characterisation of brazed joints

3.9.1 Optical microscopy

Optical microscopy is a basic microstructural characterisation technique used to examine the general morphology of brazed joints at low to medium magnification. It is particularly useful for observing the overall geometry of the joining region, the continuity or discontinuity of the interface, the approximate thickness of reaction layers, and the presence of visible defects such as gaps, pores, or cracks. Because it provides a relatively broad view of the cross-section, optical microscopy is well suited for identifying local variations along the joint and for selecting representative areas for more detailed analysis [27].

In the present work, optical microscopy was used as the first stage of interfacial characterisation. It allowed a preliminary assessment of bond continuity, local morphology, and general quality of the brazed region before higher-resolution SEM, EDS, and BSE analyses were performed.

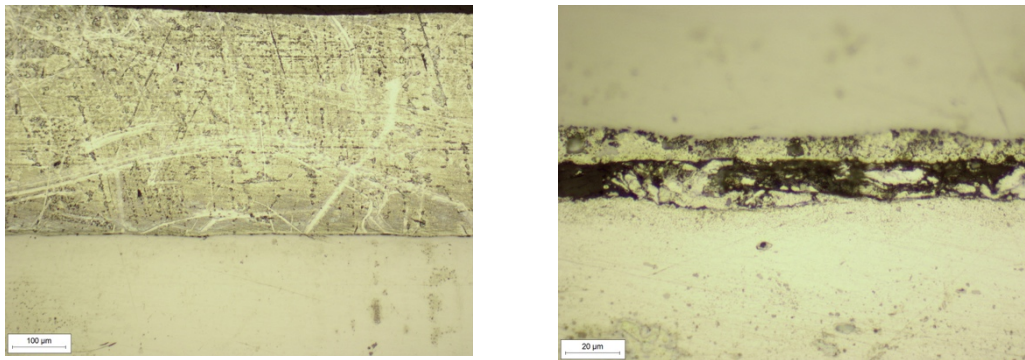


Figure 33. Representative optical micrograph of HNO_3 treated Al-Zn-coated Steel brazed joints at 100 $\times$ and 500 $\times$ magnifications

3.9.2 SEM, EDS and BSE analysis

Scanning electron microscopy (SEM) is used to observe the interfacial microstructure of brazed joints at higher magnification and resolution than optical microscopy. It allows detailed examination of the morphology of the joining zone, including the continuity of the interfacial layer, local irregularities, cracks, pores, and the thickness of reaction regions. In the present work, SEM was used to investigate the local microstructural features of the Al / Zn-coated steel interface in greater detail than was possible by optical microscopy alone [28].

Energy dispersive spectroscopy (EDS) is used together with SEM to determine the elemental composition of selected points or regions of the sample. This technique makes it possible to identify the local distribution of the main elements involved in the joint, especially Al, Zn, Fe, and O. In brazed dissimilar systems, EDS is particularly useful for distinguishing the base materials from the interfacial regions and for identifying diffusion zones or mixed layers formed during joining. However, EDS provides elemental information rather than direct phase identification.

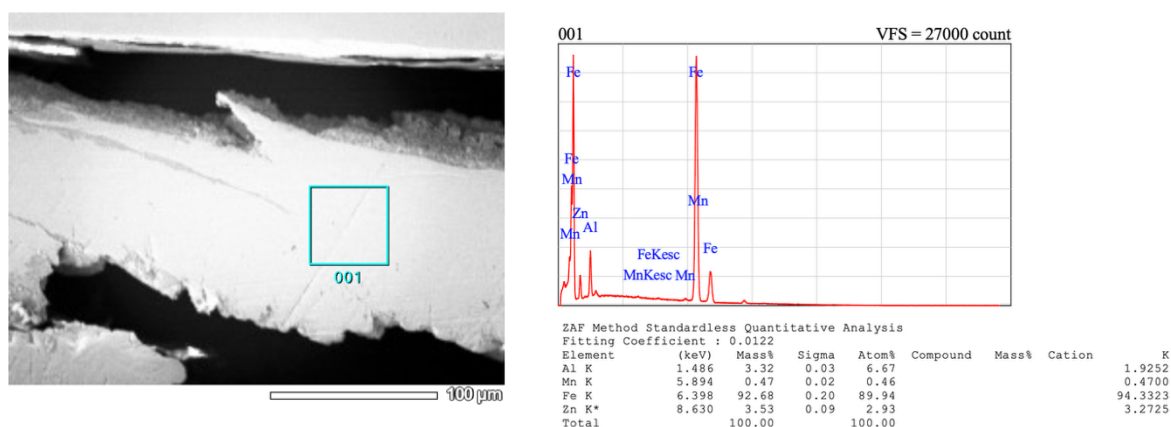


Figure 34. Representative SEM micrograph of the KOH treated Al/ Zn-coated Steel joint with corresponding EDS point analyses

Backscattered electron imaging (BSE) is an SEM imaging mode based on compositional contrast. In BSE images, regions containing heavier elements generally appear brighter, whereas regions containing lighter elements appear darker. This contrast makes BSE especially useful for distinguishing Fe-rich, Zn-rich, and Al-rich regions across the brazed joint. In the present work, BSE imaging was used to support the interpretation of interfacial morphology and compositional variation within the reaction zone [28].

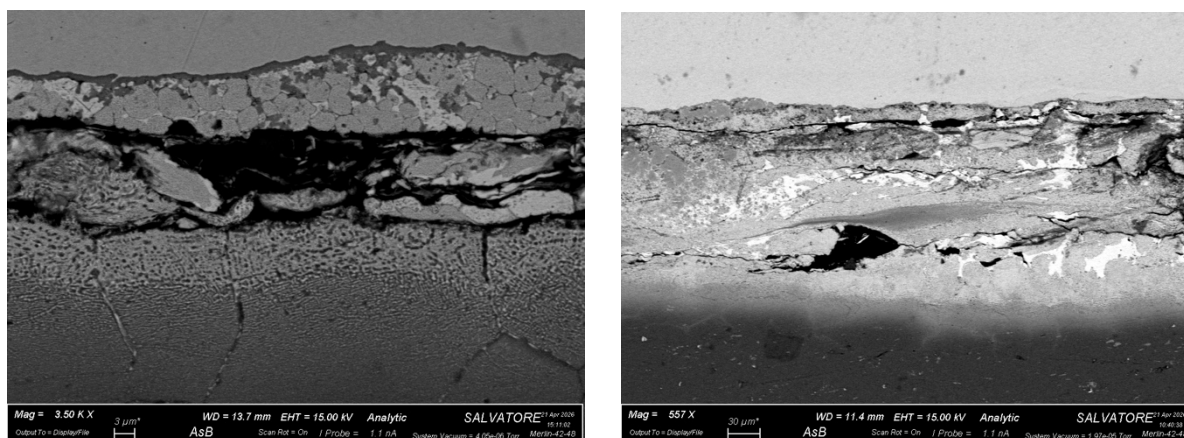


Figure 35. Representative BSE micrograph of the HNO₃ treated Al/ Zn-coated Steel joints

Elemental mapping (EDS mapping) was performed in order to evaluate the spatial distribution of the main elements across the brazed joint and to investigate the development of the interfacial diffusion layer between the Al6016 alloy and the Zn-coated steel substrate. In our case study example, the elemental maps revealed a clear compositional separation between the two base materials while simultaneously confirming the presence of an intermediate reaction region.

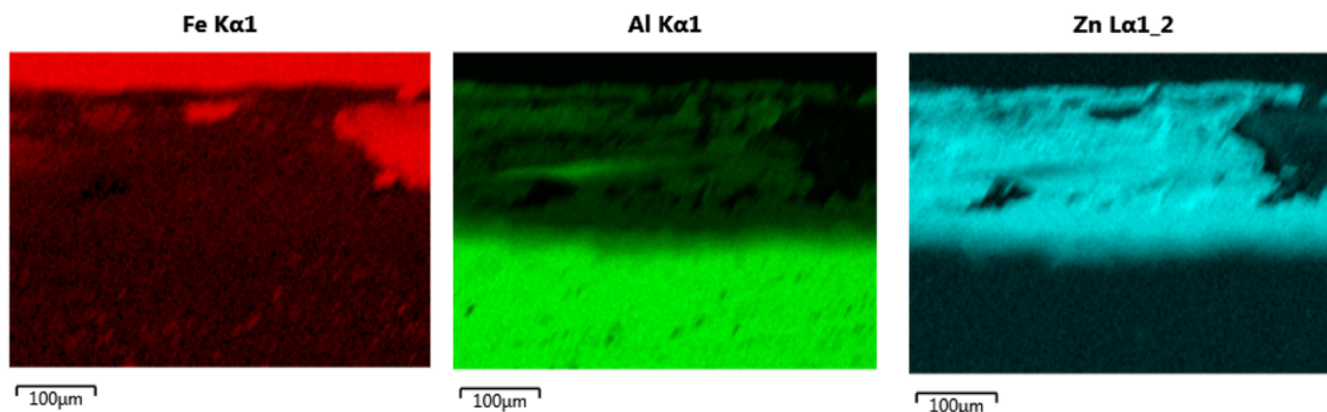


Figure 36. Elemental distribution map of Fe, Al and Zn obtained by EDS mapping

The Fe map (red) showed that iron was concentrated predominantly in the upper region of the cross-section, corresponding to the steel substrate. In contrast, the Al map (green) demonstrated that aluminium was mainly distributed within the lower region, corresponding to the aluminium base material. The Zn map (blue) indicated that zinc was primarily localised along the interfacial region between the two substrates, forming a transition layer between the steel and aluminium sides.

An overlap between the Al and Zn signals was observed at the interface, suggesting the presence of an Al–Zn mixed interfacial region. However, the EDS maps mainly provide qualitative information about the spatial distribution of elements. Therefore, these observations should be compared with the local EDS point/area analyses in order to confirm the chemical composition of the interfacial layer and to better distinguish between Al-rich, Zn-rich, and mixed reaction zones.

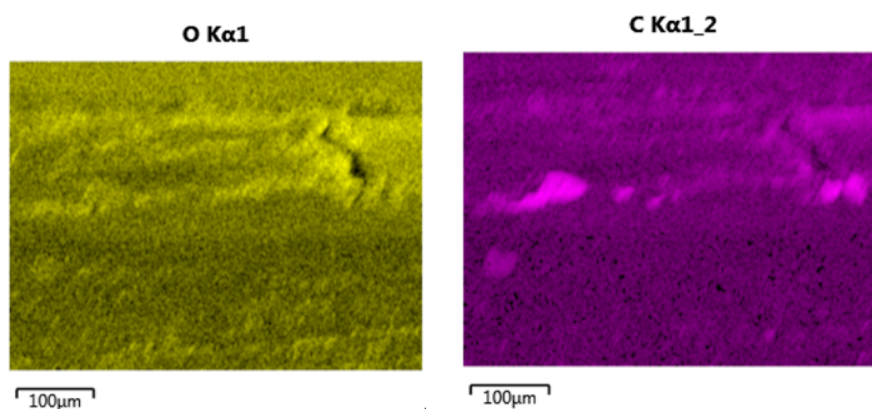


Figure 37. Elemental distribution map of O and C obtained by EDS mapping

Additionally, the O map (yellow) showed the presence of oxygen locally along the interfacial region. This oxygen distribution is likely associated with surface oxidation or residues remaining after the HNO₃ etching process. The C map (purple) revealed carbon-rich localised areas, which are most likely related to contamination, mounting resin, or polishing residues rather than to actual interfacial reaction products.

Overall, SEM, EDS, BSE imaging, and elemental mapping provide complementary information for the characterisation of Al–Zn-coated steel brazed joints. SEM allows detailed observation of the interfacial morphology, including continuity, defects, cracks, pores, and local reaction regions. EDS point analysis provides local elemental composition, while elemental mapping shows the spatial distribution of the main elements across the joint. BSE imaging supports the interpretation of compositional contrast, allowing Fe-rich, Zn-rich, and Al-rich regions to be distinguished more clearly. Therefore, the combined use of these techniques is essential for evaluating interfacial layer formation, elemental diffusion, and local heterogeneity in the brazed joints.

3.9.3 Limits of microstructural interpretation

Although optical microscopy, SEM, EDS, and BSE provide valuable information on interface morphology and composition, their interpretation must be made carefully. Features such as polishing artefacts, local contamination, oxidation, or non-uniform sectioning may influence the observed appearance of the interface [27]. In addition, compositional analysis alone does not always allow definitive identification of all phases, especially in complex Al–Zn–Fe systems. For this reason, microstructural observations should be interpreted as evidence of interfacial behaviour and local reaction trends, rather than as definitive proof of every phase or interfacial mechanism.

3.10 Characterisation procedures

After metallographic preparation, the brazed samples were characterised to evaluate the quality of the Al–Zn–steel joint. The characterisation focused on observing the cross-sectional morphology of the interface, identifying the continuity of the reaction layer, detecting possible defects, and analysing local chemical composition.

The main characterisation methods used in this work were optical microscopy, SEM observation, backscattered electron imaging (BSE), and EDS analysis. Optical microscopy was first used to obtain an overall view of the joint and to locate regions of interest. SEM and BSE imaging were then used to examine the interfacial region in more detail. In particular, BSE imaging was useful because it provides compositional contrast, allowing regions with different average atomic numbers to be distinguished more clearly. Finally, EDS analysis was used to support the identification of local elemental distribution across the joint.

3.10.1 Optical microscopy

Optical microscopy was performed after grinding and polishing of the mounted cross-sections. The purpose of this step was to observe the morphology of the Al–Zn–steel interface, evaluate the continuity of the brazed joint, measure the approximate thickness of interfacial layers, and identify possible defects such as voids, cracks, porosity, or unbonded regions.

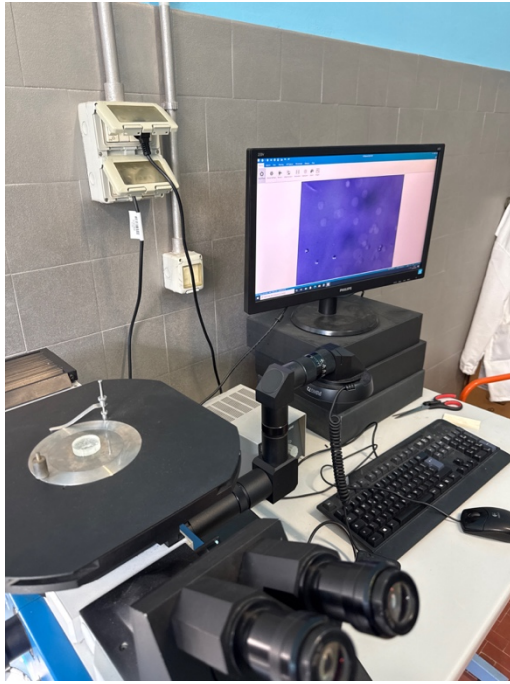
The observations were carried out using a Reichert-Jung optical microscope system equipped with a digital camera and imaging software. The sample was fixed on the rotating stage to ensure stable positioning and accurate focusing across the joint region. Several magnification levels were used during the analysis, including 100 $\times$, 200 $\times$, and 500 $\times$. Lower magnification was useful for observing the full joint line and global morphology, while higher magnifications were used to inspect the interlayer, diffusion region, and local defects in greater detail.



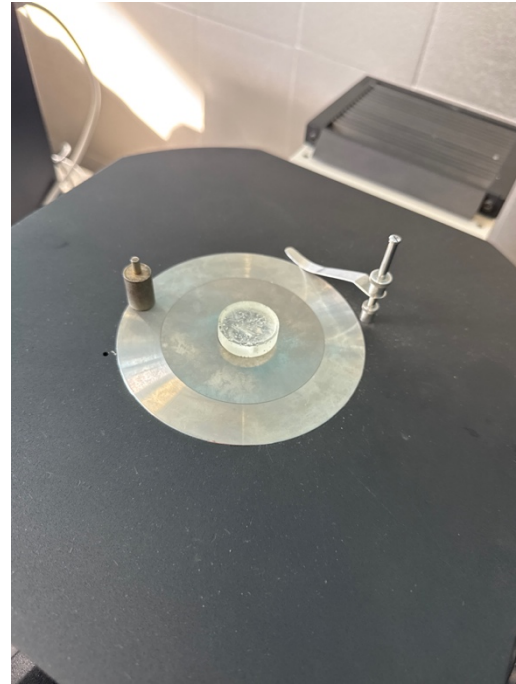
Figure 38. General view of the Reichert-Jung optical microscope system

In order to improve image quality across uneven regions of the mounted cross-sections, multiple partially focused images were captured and later processed using PICOLAY for focus stacking and tile stitching. This procedure made it possible to obtain clearer composite images of the joint region with improved depth of field and wider field coverage.

In the present work, optical microscopy also served as the first screening step before SEM and EDS analysis. This preliminary examination made it possible to identify representative regions of the brazed cross-section, including areas where bonding appeared continuous as well as zones showing discontinuities, gaps, porosity, or irregular interfacial morphology. In this way, optical microscopy supported the selection of regions of interest for subsequent higher-resolution characterisation and provided the first visual assessment of joint quality before SEM-based analysis.



(a)



(b)

Figure 39. Optical microscope setup: (a) general view of microscope together with the connected computer used for image acquisition and visualisation; (b) mounted specimen fixed on the rotating stage

3.10.2 SEM observation

Scanning Electron Microscopy (SEM) was used to perform a detailed analysis of the brazed Al–Zn–steel interface after optical microscopy. While optical microscopy provided a general overview of the joint, SEM enabled higher-resolution observation of the interfacial region, allowing a more precise evaluation of microstructural features.

SEM observation was carried out using a JEOL scanning electron microscope. SEM imaging allowed clear visualisation of different phases due to contrast differences. This made it possible to distinguish regions with different compositions and identify the formation of intermetallic compounds at the interface. Higher magnifications were used to investigate the fine structure of the reaction layer and to detect microstructural details that could not be resolved by optical microscopy. SEM observation also supported the selection of specific regions of interest for further EDS analysis.

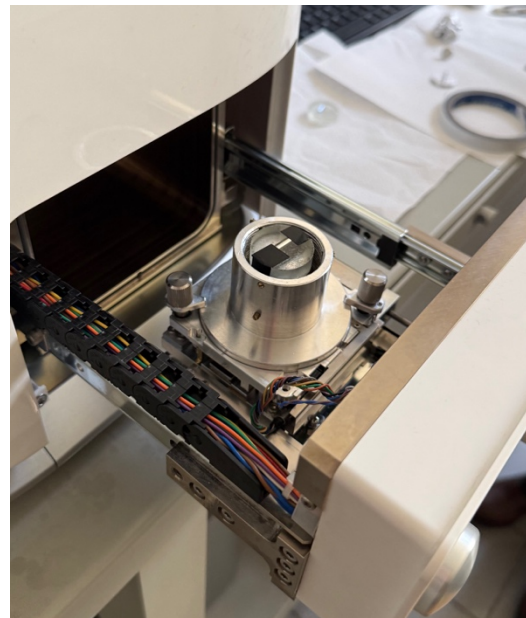


Figure 40. JEOL scanning electron microscope used for SEM observation

Before SEM observation, the polished mounted cross-sections were prepared to ensure stable positioning and reliable imaging conditions. Each specimen was fixed onto an aluminium SEM stub, and conductive adhesive tape was used to provide an electrical path between the sample and the holder. The mounted specimen was then introduced into the SEM chamber, where high vacuum was established before imaging. After insertion, the stage was centred and tilted, and preliminary focusing tests were carried out using both SE and BSE detectors in order to optimise image quality before detailed observation.



(a)



(b)

Figure 41. Preparation of the mounted specimens before SEM observation: (a) mounted specimen attached to an aluminium stub with conductive adhesive tape prior to insertion into the SEM chamber; (b) view of SEM chamber and specimen holder positioned for insertion

Overall, SEM observation provided a deeper understanding of the interfacial structure, complementing optical microscopy and enabling a more accurate interpretation of the brazing behaviour and joint quality.

3.10.3 EDS analysis

Energy Dispersive X-ray Spectroscopy (EDS) was used in combination with SEM to analyse the elemental composition of the brazed Al–Zn–steel interface. This technique allowed identification and mapping of the distribution of key elements such as aluminium (Al), zinc (Zn), and iron (Fe) across the joint. EDS analysis was performed on selected regions of interest identified during SEM observation. Both point analysis and line scans were used to evaluate local composition and elemental gradients across the interface.

The main objective of EDS analysis was to confirm the presence of different phases and to better understand the formation of intermetallic compounds at the interface. By analysing the variation in elemental concentration, it was possible to distinguish between aluminium-rich, zinc-rich, and iron-rich regions, as well as to identify diffusion zones between the base materials.

Special attention was given to the interlayer region, where the interaction between molten zinc and the aluminium and steel substrates occurs. EDS results helped to verify whether the zinc interlayer was uniformly distributed and to assess the extent of diffusion between the materials.

Overall, EDS analysis provided essential compositional information that complemented SEM observations, enabling a more comprehensive interpretation of the interfacial structure and the mechanisms involved in the brazing process.

3.10.4 BSE imaging

Backscattered Electron (BSE) imaging was performed using a ZEISS MERLIN field-emission scanning electron microscope (FE-SEM) equipped with a GEMINI column to obtain compositional contrast across the Al–Zn–steel interface. Unlike secondary electron imaging, BSE mode is sensitive to differences in atomic number, allowing regions with higher average atomic number to appear brighter, while lighter elements appear darker.

BSE imaging was used to clearly distinguish between aluminium, zinc-rich regions, and iron-based steel, as well as to identify interfacial layers and possible intermetallic compounds. This contrast made it easier to observe the morphology, continuity, and thickness variations of the interlayer and reaction zones. In addition, elemental mapping was carried out in some areas to visualise the spatial distribution of elements within the interfacial region.

In addition, BSE imaging supported the interpretation of EDS results by providing a direct visual correlation between compositional contrast and elemental distribution. This was especially useful for selecting representative regions for quantitative analysis.



Figure 42. *Zeiss Merlin field-emission scanning electron microscope equipped with Gemini column, used for backscattered electron imaging*

Overall, BSE imaging provided essential complementary information to SEM and EDS, enabling a clearer understanding of the compositional variations and microstructural features of the brazed joint.

4. Results

4.1 Pristine materials

4.1.1 Aluminium surface condition before brazing

The pristine Al6016 sample was examined after metallographic preparation, including grinding and polishing. Optical microscopy observations showed that the aluminium surface appeared relatively uniform at the selected magnification, with no visible macroscopic defects. Small dark particles and polishing traces were observed across the surface, but no large discontinuities or severe surface damage were detected.

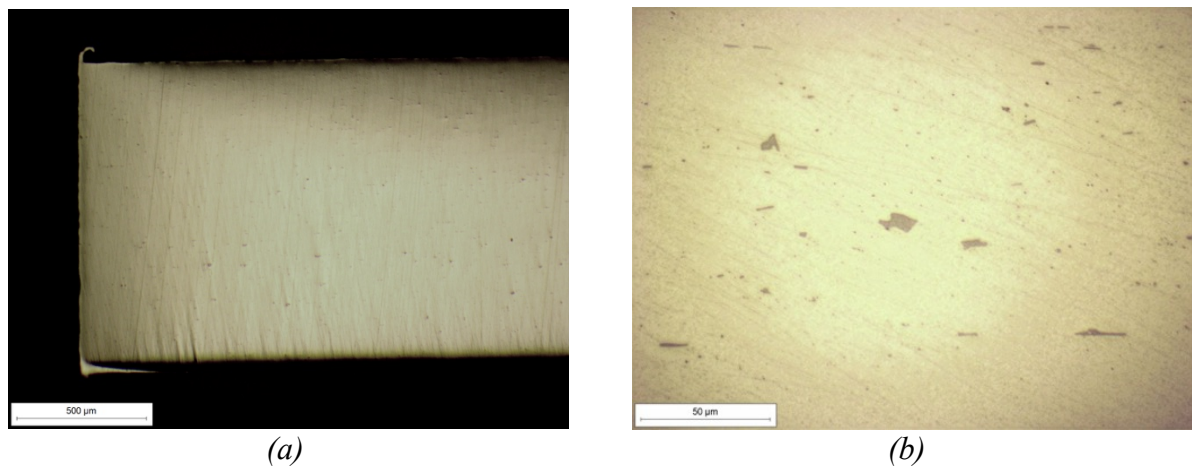


Figure 43. Pristine Al6016 surface: (a) 20× magnification; (b) 200× magnification

However, the grain boundaries were not clearly resolved in the obtained micrographs. This indicates that the optical observations were mainly useful for evaluating the general surface condition, polishing quality and visible surface defects, rather than for detailed grain-structure analysis. As a result, no definitive conclusion regarding the complete microstructural homogeneity of the material can be made from these observations alone.

Overall, the pristine Al6016 surface exhibited a relatively smooth and homogeneous appearance after metallographic preparation, although minor polishing marks and small dark surface particles were still present before brazing.

4.1.2 Zinc-coated steel surface and coating thickness

4.1.2.1 Measurement of Zn coating thickness values

The pristine zinc-coated steel substrate was also examined in order to evaluate the morphology and thickness uniformity of the protective Zn coating before brazing. Optical microscopy observations showed the presence of a continuous zinc layer covering the steel

substrate along the entire observed surface. Measurements performed at different positions indicated that the coating thickness remained relatively uniform, although slight local variations were observed between the analysed regions.

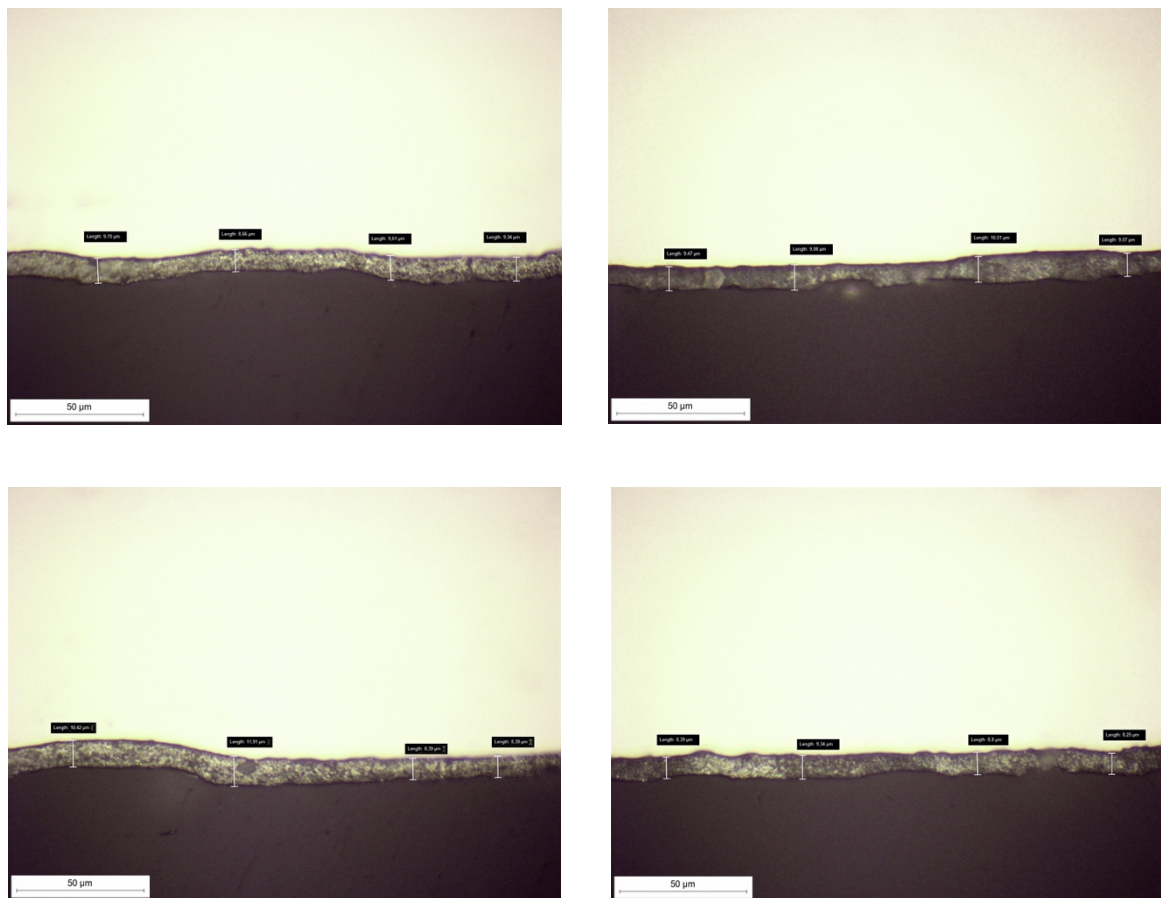


Figure 44. Optical micrographs (200×) of the four pristine zinc-coated steel cross-sections used for Zn coating thickness measurements

	Sample 1	Sample 2	Sample 3	Sample 4
Thickness 1	9,75	9,47	8,39	10,42
Thickness 2	8,66	9,88	9,34	11,91
Thickness 3	9,61	10,01	8,8	8,39
Thickness 4	9,34	9,07	8,25	8,39
Average Thickness	9,34	9,6075	8,695	9,7775

Table 2. Measured Zn coating thickness values and average thickness values for four different zinc-coated steel cross-section areas

Average Thickness of Zn coating - 9,355 μm

The measured coating thickness values ranged approximately between 8 and 12 μm depending on the measurement location. The calculated average zinc coating thickness from all investigated samples was approximately 9.36 μm . These observations confirm that the galvanised steel substrate possessed a stable and continuous Zn-rich surface layer prior to the joining process.

4.1.2.2. SEM and EDS observations of Zn-coated steel surface

SEM observations of the pristine Zn-coated steel surface revealed a relatively rough and heterogeneous morphology characterised by irregular Zn-rich surface features and local surface particles. The EDS spectra obtained from the analysed regions confirmed that the surface was predominantly composed of zinc, with only small amounts of oxygen and aluminium detected.

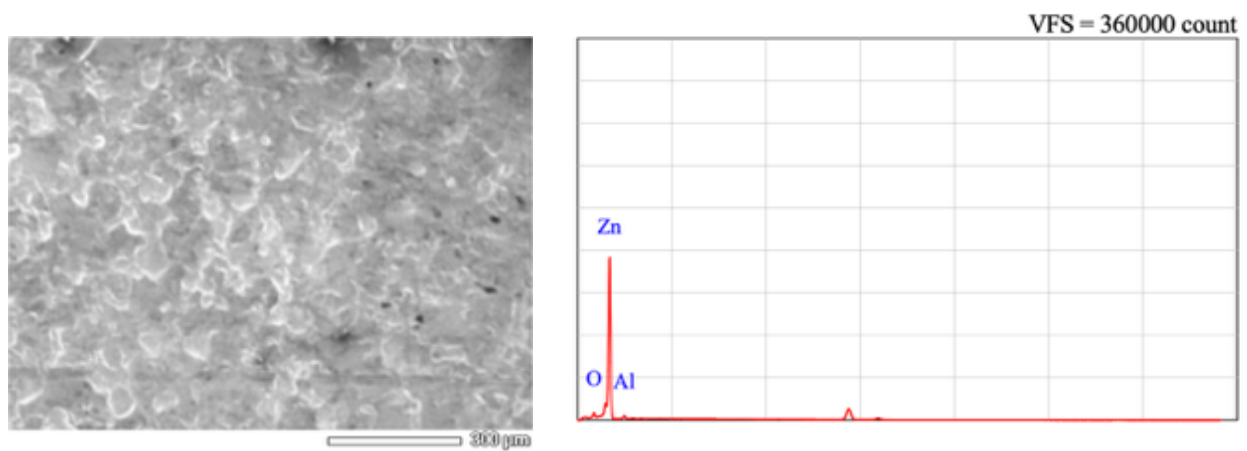


Figure 45. SEM micrograph and EDS spectrum of region S1 of pristine Zn-coated steel surface

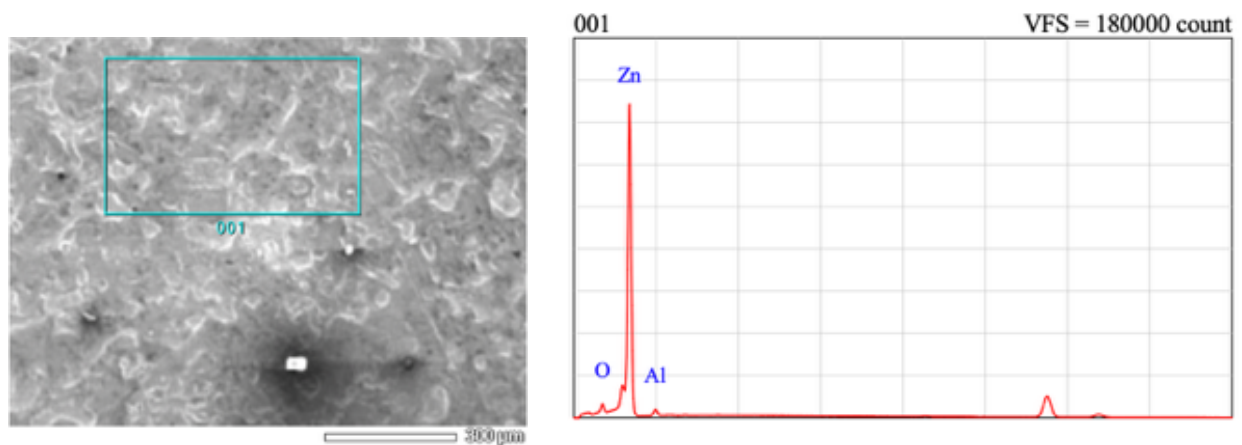


Figure 46. SEM micrograph and EDS spectrum of region S2 of pristine Zn-coated steel surface

	O	Al	Zn
S1 wt%	3.83	2.12	94.05
S2 wt%	3.62	2.08	94.3
AVERAGE wt%	3.725	2.1	94.175
SD	0.105	0.02	0.125
	3,7 ± 0,1	2,1 ± 0,02	94,175 ± 0,13

Table 3. Average elemental composition of pristine Zn-coated steel surface

The average elemental composition was approximately 94.18 wt% Zn, 3.73 wt% O, and 2.10 wt% Al. The presence of oxygen indicates partial surface oxidation of the zinc coating after exposure to air, while the small aluminium content may originate from surface contamination or neighbouring material interaction during preparation.

Overall, the pristine zinc-coated steel exhibited a continuous Zn-rich protective layer with relatively uniform thickness and characteristic galvanised surface morphology. The presence of the Zn coating is expected to play an important role during brazing by improving wetting behaviour and limiting direct Al–Fe interaction at the interface.

4.1.3 Effect of HNO₃ and KOH treatments on Al6016 surface

SEM observations were performed to evaluate the effect of chemical surface treatments on the Al6016 surface morphology and surface oxidation condition. Three conditions were analysed: pristine aluminium, HNO₃-treated aluminium, and KOH-treated aluminium. The SEM images revealed that all samples retained visible grinding and polishing traces originating from the mechanical surface preparation. However, differences in surface appearance and oxygen content were observed after chemical treatment.

4.1.3.1 Pristine Al6016

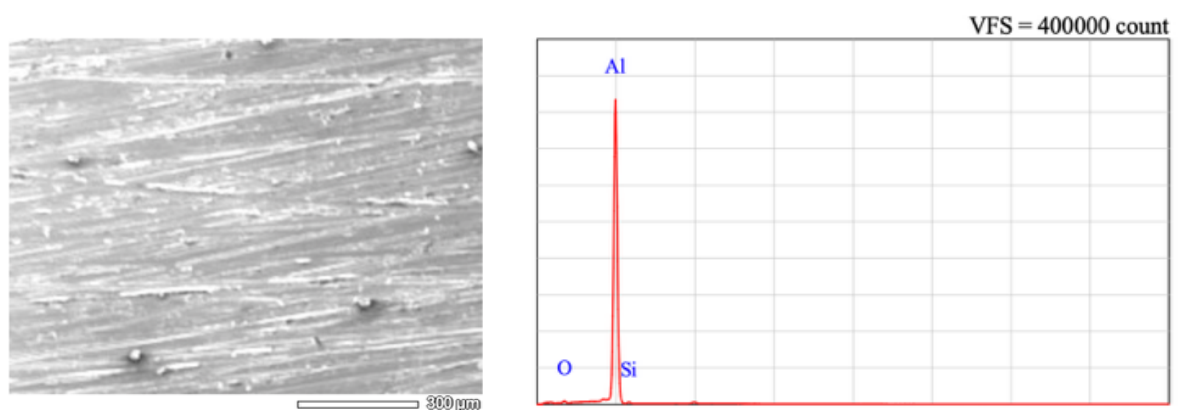


Figure 47. SEM image and EDS spectrum of the pristine Al6016 surface

	O	Al	Si
AVERAGE wt%	3,68	95,40	0,92
SD	0,250	0,291	0,037

Table 4. Average elemental composition of the pristine Al6016 surface and its standard deviation

	Oxygen/Aluminium	Surface Oxidation Indicator (O/Al ratio)
Pristine	0,04	High

Table 5. Oxygen-to-aluminium ratio and surface oxidation for pristine Al6016 surface

The pristine aluminium surface exhibited a relatively rough appearance with visible polishing grooves and scattered surface particles. EDS analysis showed an average composition of approximately 95.40 wt% Al, 3.68 wt% O, and 0.92 wt% Si. The calculated oxygen-to-aluminium ratio (O/Al) was approximately 0.04, indicating the highest level of surface oxidation among the investigated conditions.

4.1.3.2 HNO₃ treatment

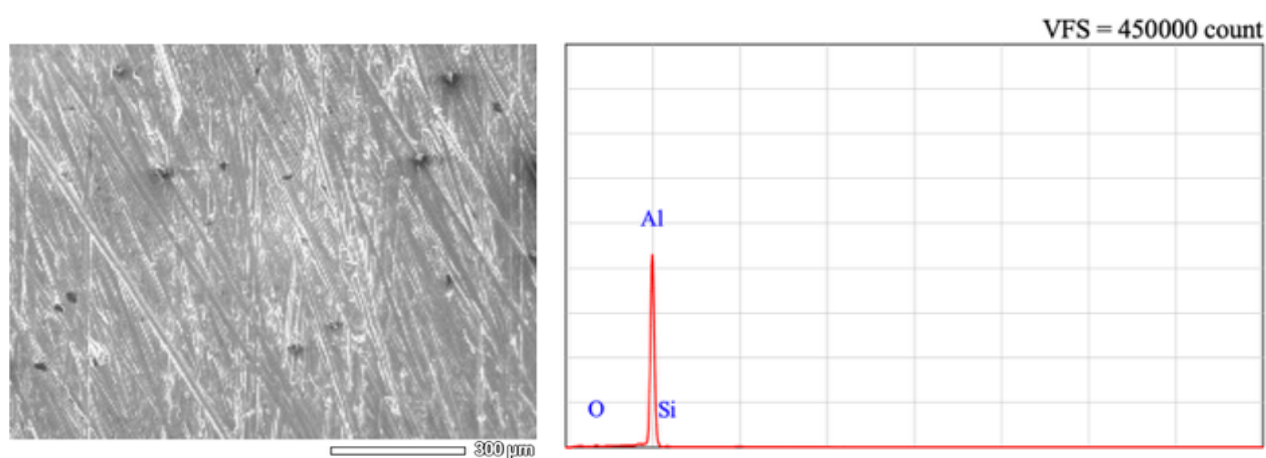


Figure 48. SEM image and EDS spectrum of HNO₃-treated Al6016

	O	Al	Si
AVERAGE wt%	2,56	96,65	0,79
SD	0,139	0,095	0,050

Table 6. Average elemental composition of HNO₃-treated Al6016 and its standard deviation

	Oxygen/Aluminium	Surface Oxidation Indicator (O/Al ratio)
HNO₃	0,03	Medium

Table 7. Oxygen-to-aluminium ratio and surface oxidation for HNO₃-treated Al6016 surface

After HNO₃ treatment, the surface morphology appeared cleaner and more uniform compared to the pristine condition, although polishing lines were still visible. The oxygen concentration decreased to approximately 2.56 wt%, while the aluminium concentration increased to about 96.65 wt%. The O/Al ratio decreased to approximately 0.03, corresponding to a medium oxidation level. This result suggests that the nitric acid treatment partially removed the native oxide layer from the aluminium surface.

4.1.3.3 KOH treatment

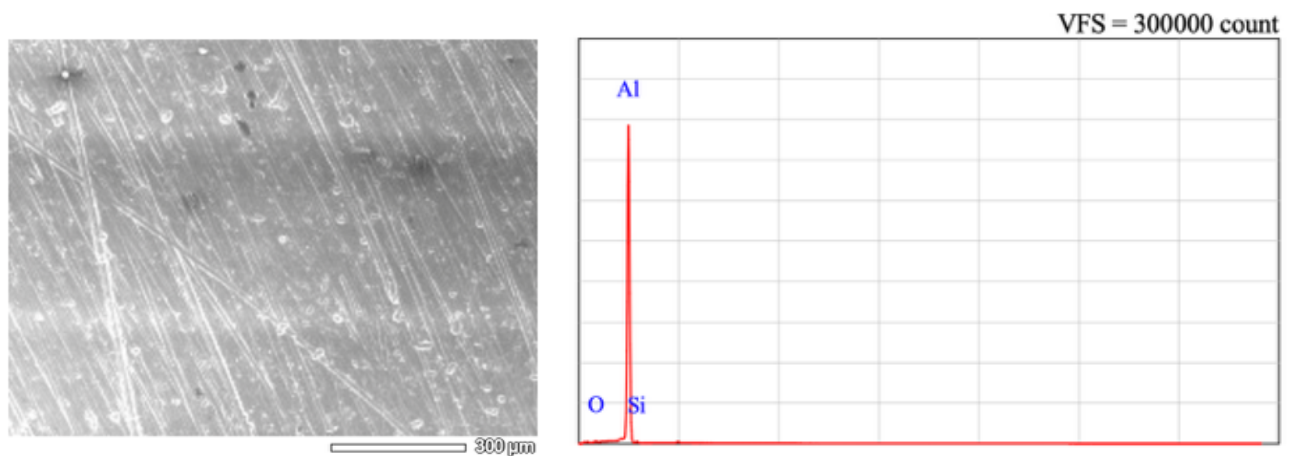


Figure 49. SEM image and EDS spectrum of KOH-treated Al6016

	O	Al	Si
AVERAGE wt%	2,17	96,89	0,94
SD	0,114	0,139	0,037

Table 8. Average elemental composition of KOH-treated Al6016

	Oxygen/Aluminium	Surface Oxidation Indicator (O/Al ratio)
KOH	0,02	Low

Table 9. Oxygen-to-aluminium ratio and surface oxidation for KOH-treated Al6016 surface

The KOH-treated sample exhibited the lowest measured oxygen concentration among all conditions. EDS analysis showed approximately 96.89 wt% Al, 2.17 wt% O, and 0.94 wt% Si. The calculated O/Al ratio was approximately 0.02, corresponding to the lowest surface oxidation indicator. The SEM image also showed a relatively cleaner surface morphology despite the persistence of grinding traces from mechanical preparation.

4.1.3.4. Comparison of surface morphology and oxidation

A comparison of the SEM observations and EDS results indicates that the chemical surface treatments significantly influenced the surface condition and oxidation level of the Al6016 samples. Although all specimens retained visible grinding and polishing traces originating from the mechanical preparation procedure, clear differences in surface morphology and oxygen concentration were observed between the pristine, HNO₃-treated, and KOH-treated conditions.

The pristine aluminium surface exhibited the roughest appearance, characterised by pronounced polishing grooves and scattered surface particles. EDS analysis showed the highest oxygen concentration (3.68 wt%) and the highest oxygen-to-aluminium ratio (O/Al = 0.04), corresponding to a high surface oxidation level. These observations suggest that the untreated aluminium surface retained a relatively thick native oxide layer.

After HNO₃ treatment, the surface appeared cleaner and more homogeneous compared to the pristine condition. The oxygen concentration decreased to approximately 2.56 wt%, while the O/Al ratio decreased to 0.03, corresponding to a medium oxidation level. The nitric acid treatment therefore partially reduced the surface oxide layer and improved the surface condition prior to brazing.

The KOH-treated sample exhibited the lowest measured oxygen concentration (2.17 wt%) and the lowest O/Al ratio (0.02), corresponding to the lowest surface oxidation indicator among the investigated conditions. The SEM observations also revealed a relatively cleaner surface morphology despite the remaining polishing traces. These results indicate that KOH treatment was the most effective method for reducing surface oxidation and improving the aluminium surface condition before brazing.

	O	Al	Si
Pristine	3,7 ± 0,25	95,4 ± 0,29	0,9 ± 0,04
HNO₃	2,6 ± 0,14	96,7 ± 0,10	0,8 ± 0,05
KOH	2,2 ± 0,11	96,9 ± 0,14	0,9 ± 0,04

Table 10. Comparative EDS elemental composition of pristine, HNO₃-treated, and KOH-treated Al6016 surfaces.

	Oxygen/Aluminium	Surface Oxidation Indicator (O/Al ratio)
Pristine	0,04	High
HNO₃	0,03	Medium
KOH	0,02	Low

Table 11. Comparison of oxygen-to-aluminium ratio and surface oxidation indicators for pristine, HNO₃-treated, and KOH-treated Al6016 surfaces.

Overall, both chemical treatments improved the surface condition compared to the pristine aluminium sample. However, the KOH treatment produced the lowest oxidation level and the cleanest overall surface morphology among the investigated conditions.

4.1.4 Roughness comparison after surface treatments

Contact profilometry measurements were performed to evaluate the influence of chemical surface treatments on the roughness characteristics of the Al6016 alloy and to compare them with the pristine Zn-coated steel surface.

The measured roughness parameters included:

- Ra (arithmetic average roughness) - average height deviation of the surface profile from the mean line
- Rz (maximum height of the roughness profile) - average vertical distance between the highest peaks and deepest valleys of the profile
- Rsk (skewness of the roughness profile) - describes the asymmetry of the surface profile.
 - $Rsk > 0 \rightarrow$ surface dominated by peaks
 - $Rsk < 0 \rightarrow$ surface dominated by valleys
 - $Rsk \approx 0 \rightarrow$ symmetric distribution
- Rku (kurtosis of the roughness profile) - describes the sharpness or distribution shape of peaks and valleys.
 - $Rku = 3 \rightarrow$ normal/Gaussian distribution
 - $Rku > 3 \rightarrow$ sharp peaks or deep narrow valleys
 - $Rku < 3 \rightarrow$ flatter and broader features

Characteristics of Al6016:

	Al6016 untreated			
	Ra [μm]	Rz [μm]	Rsk	Rku
AVERAGE	0,95	7,55	-0,43	3,52
SD	0,06	0,61	0,09	0,32

Table 12. Average roughness parameters of untreated Al6016

	Al6016 HNO ₃			
	Ra [μm]	Rz [μm]	Rsk	Rku
AVERAGE	0,94	7,93	-0,46	4,06
SD	0,11	0,89	0,33	0,31

Table 13. Average roughness parameters of HNO₃-treated Al6016

	Al6016 KOH			
	Ra [μm]	Rz [μm]	Rsk	Rku
AVERAGE	1,38	9,08	-0,35	3,03
SD	0,07	0,48	0,04	0,09

Table 14. Average roughness parameters of KOH-treated Al6016

The untreated Al6016 surface exhibited an average roughness of approximately $R_a = 0.95 \mu\text{m}$ and $R_z = 7.55 \mu\text{m}$. After HNO_3 treatment, the roughness remained relatively similar ($R_a = 0.94 \mu\text{m}$), indicating that nitric acid produced only a limited modification of the surface topography. In contrast, the KOH-treated sample showed a noticeable increase in roughness, with R_a increasing to approximately $1.38 \mu\text{m}$ and R_z to $9.08 \mu\text{m}$. This suggests that the alkaline treatment generated a more aggressive surface etching effect, leading to a rougher and more developed surface morphology.

Characteristics of Zn-coated steel:

	Zn-coated steel			
	Ra [μm]	Rz [μm]	Rsk	Rku
AVERAGE	1,14	7,12	-0,82	3,63
SD	0,12	0,21	0,17	0,08

Table 15. Average roughness parameters of pristine Zn-coated steel

The Zn-coated steel surface exhibited intermediate roughness values, with $R_a \approx 1.14 \mu\text{m}$ and $R_z \approx 7.12 \mu\text{m}$, indicating a relatively irregular coating morphology associated with the galvanising process.

All analysed surfaces showed negative skewness values ($R_{sk} < 0$), indicating that the surface profiles were dominated by valleys rather than sharp peaks. Such morphology may be beneficial for retaining molten zinc during brazing and improving local wetting conditions at the interface. The HNO_3 -treated aluminium sample exhibited the highest kurtosis value ($R_{ku} \approx 4.06$), suggesting the presence of sharper localised surface features compared to the other conditions.

Overall, the profilometry results indicate that the HNO_3 treatment had only a minor influence on the average surface roughness, while the KOH treatment significantly increased surface development and roughness. These observations are consistent with the SEM analysis, which showed a more pronounced surface modification after alkaline treatment.

4.2 Trial 1: Brazing at 480 °C

4.2.1 Optical microscopy: HNO₃-treated sample

The micrographs revealed the presence of a continuous but non-uniform interfacial region between the aluminium alloy and the Zn-coated steel substrate. The interface generally appeared as a thin dark layer extending along the bonding zone, with a thickness varying approximately between 2 and 8 μm depending on the local region of observation.

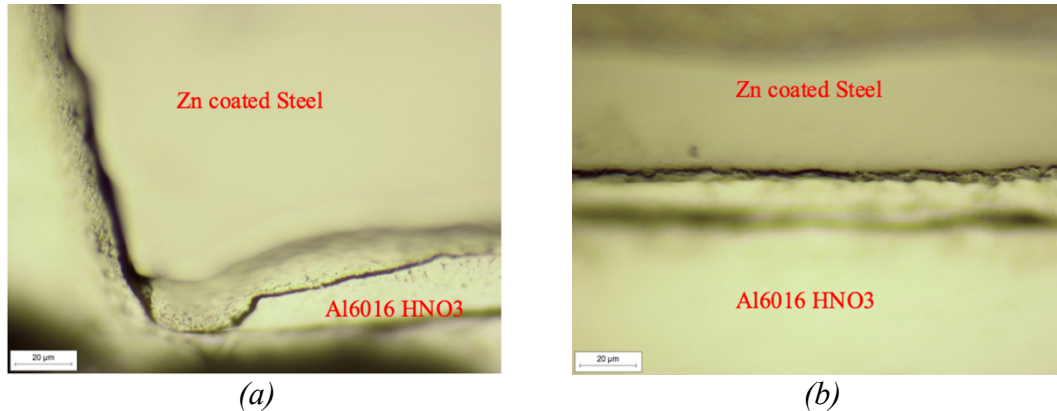


Figure 50. Optical micrograph (500 $\times$) showing: (a) interfacial region near the edge; (b) continuous interfacial layer in central region

The morphology of the interfacial layer was irregular and locally wavy. In several regions, the interface remained relatively thin and continuous, while other areas exhibited localised thickening, roughness, and small protrusions. The dark regions observed along the interface are more likely related to cracks, discontinuities, gaps, or locally unbonded regions rather than directly identifiable reaction products. These features indicate non-uniform contact and local instability of the interface during the brazing process.

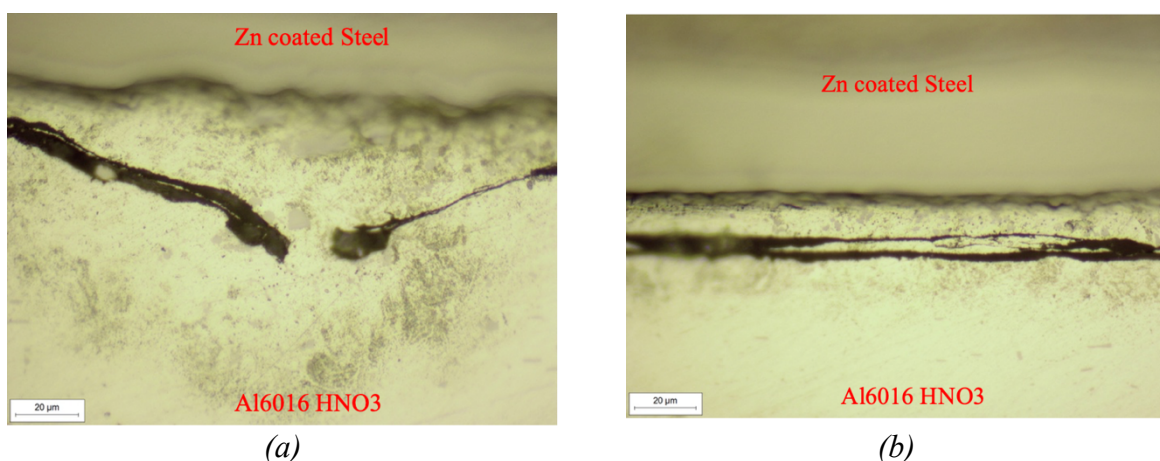


Figure 51. Optical micrograph (500 $\times$) showing: (a) limited localised bonding; (b) non-uniform bonding and crack-like interfacial discontinuities

Several discontinuities and defects were also observed throughout the joint region. In particular, dark rounded regions visible in some micrographs were identified as pores likely formed during heating. These pores were often surrounded by locally thicker interfacial regions, suggesting uneven redistribution of the molten Zn-rich phase during brazing. In addition, thin dark linear features extending along the interface were interpreted as crack-like discontinuities likely caused by localised stress and non-uniform pressure application during heating. The use of mechanical springs for load application may have generated non-homogeneous contact conditions, leading to localised separation and irregular bonding along the interface.

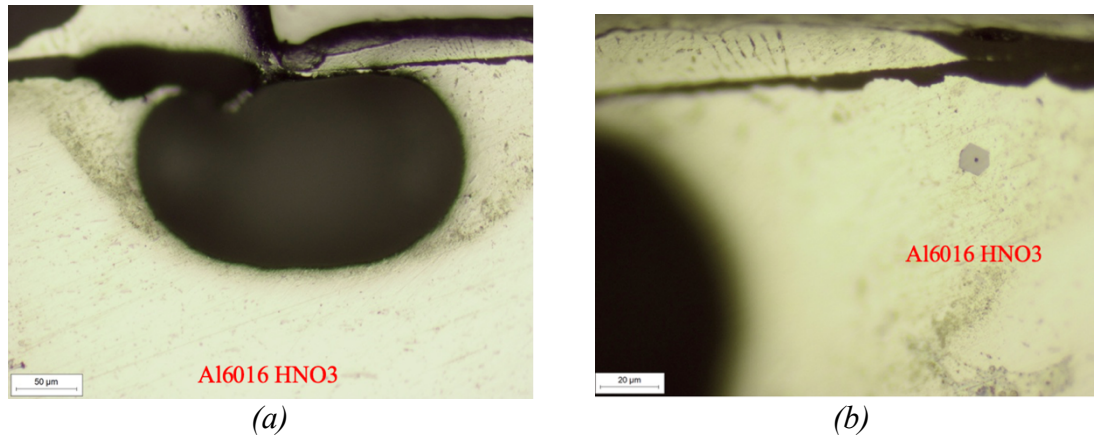


Figure 52. Optical micrograph showing large pore formation and localised interfacial separation (a) 200 $\times$ magnification; (b) 500 $\times$ magnification

Optical microscopy observations performed on the opposite side of the brazed specimen revealed a slightly more regular interfacial morphology compared to the first observation side. In several regions, the interfacial layer appeared relatively continuous and uniform, with fewer pronounced interruptions along the bonding zone.

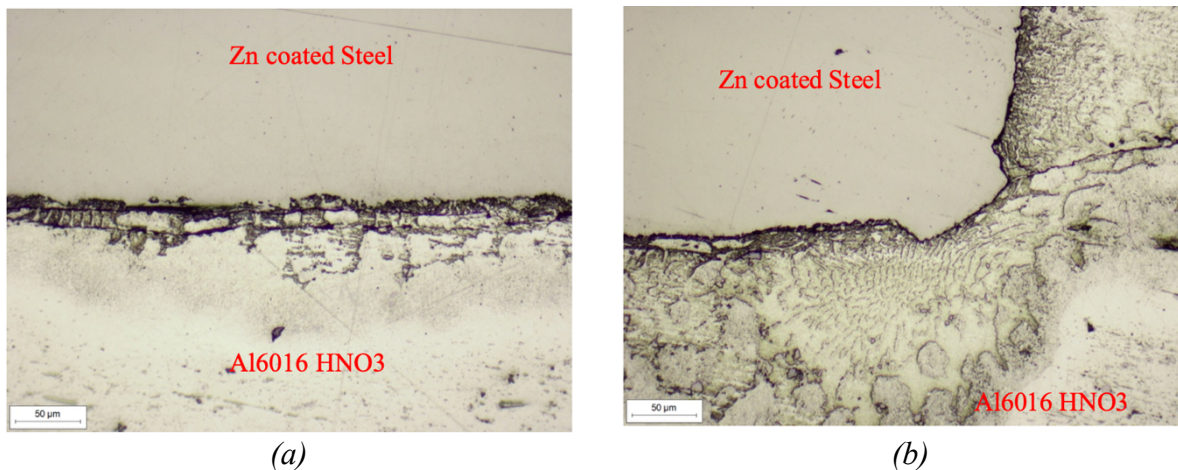


Figure 53. Optical micrograph (200 $\times$) showing: (a) a relatively continuous interfacial layer with local irregularities; (b) localised widening and roughness of the interfacial region

The observed interfacial morphology continued to exhibit local thickness variations, roughness, and isolated discontinuities. Some regions showed a relatively straight and continuous reaction layer, while other areas contained local widening of the interface and irregular surface features. These observations suggest that the interaction between the molten Zn-rich phase and the aluminium substrate remained non-uniform during brazing, resulting in heterogeneous interfacial development.

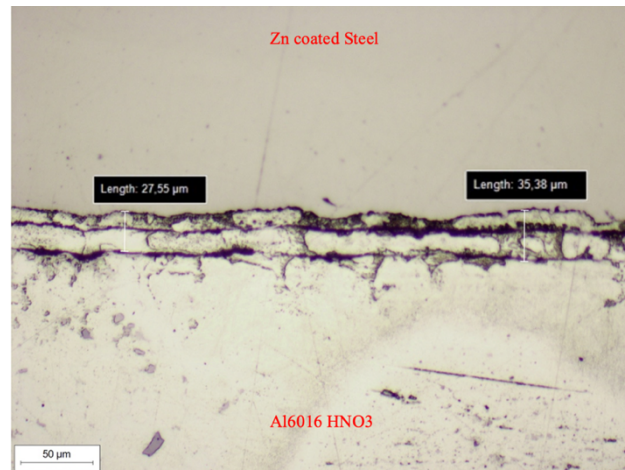


Figure 54. Optical micrograph (200 $\times$) showing localised interfacial thickening with measured regions of approximately 27-35 μm

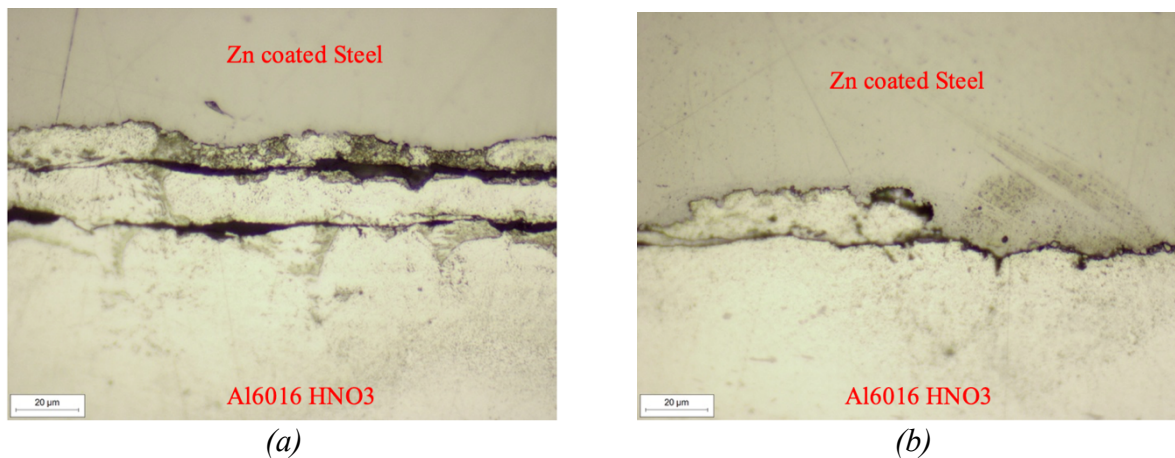


Figure 55. Optical micrograph (500 $\times$) showing: localised interfacial separation and crack-like discontinuities; (b) non-uniform bonding interfacial morphology with localised thickening

The differences observed between the two sides of the specimen are likely associated with metallographic preparation, which may have influenced the apparent morphology of the interface. Since the aluminium phase is softer than the Zn-coated steel substrate, the polishing process can locally remove material from the aluminium side, leading to polishing-induced artefacts and modification of the apparent interfacial continuity.

To minimise preferential material removal, a milder and shorter polishing procedure was applied during preparation of this observation side. As a result, the interface locally appeared more continuous than on the first side of the sample. Nevertheless, the interface still remained

thin, irregular, and locally discontinuous, indicating that the overall bonding quality was not significantly improved across the sample. The overall morphology still demonstrated non-uniform bonding and limited interfacial reactivity.

4.2.2 SEM-EDS: HNO₃-treated sample

SEM observations revealed the interface as a relatively thin reaction layer with irregular morphology and localised thickness variations along the bonding zone. In several regions, partial separation and discontinuities were observed, confirming the limited and heterogeneous nature of the bonding identified during optical microscopy analysis. The observations also revealed localised interfacial thickening and irregular surface morphology near the reaction layer. These variations are likely associated with non-uniform diffusion and uneven redistribution of the molten Zn-rich phase during brazing. The presence of localised voids and discontinuities further suggests that the applied contact pressure was not homogeneous across the sample, resulting in inconsistent bonding conditions.

The interfacial regions showed mixed elemental compositions associated with the formation of a Zn-rich reaction layer. At point s1, located near the interface, the measured composition contained approximately Zn (~50 wt%), Al (~30 wt%), and Fe (~15 wt%), indicating the formation of a mixed Al–Zn–Fe reaction zone.

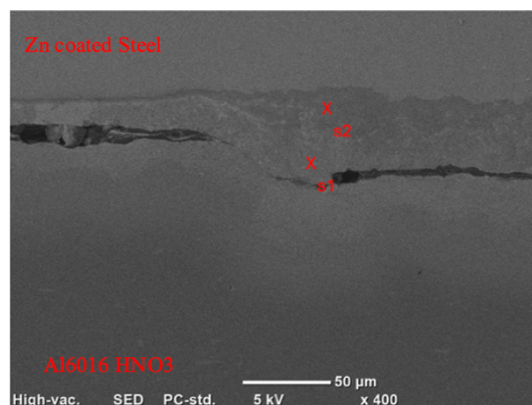


Figure 56. SEM micrograph showing the locations of EDS point analyses s1 and s2

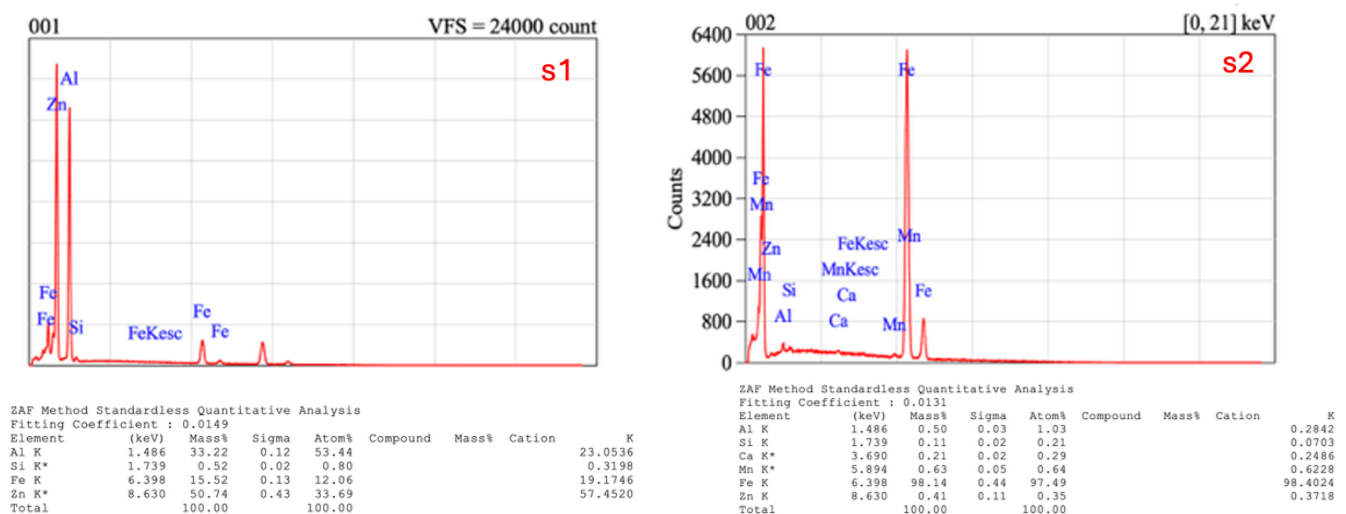


Figure 57. EDS spectra corresponding to point analyses s1 and s2

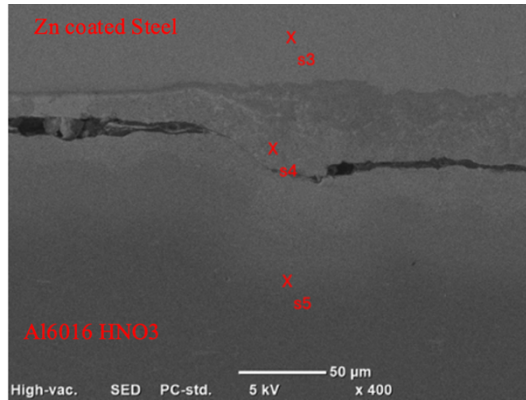


Figure 58. SEM micrograph showing the locations of EDS point analyses s3, s4 and s5

The measurements showed significant compositional differences between the steel side, the interfacial layer, and the aluminium substrate. Points located inside the steel region (s2 and s3) were predominantly composed of Fe (~98–99 wt%), indicating limited diffusion of aluminium into the steel substrate during brazing. In contrast, the aluminium region (s5) exhibited a composition dominated by Al (~97–98 wt%), confirming that the bulk aluminium phase remained chemically distinct from the steel side. Point s4 revealed a Zn-rich interlayer containing approximately Zn (~75 wt%) and Al (~23 wt%), suggesting localised accumulation and redistribution of the molten Zn phase during heating.

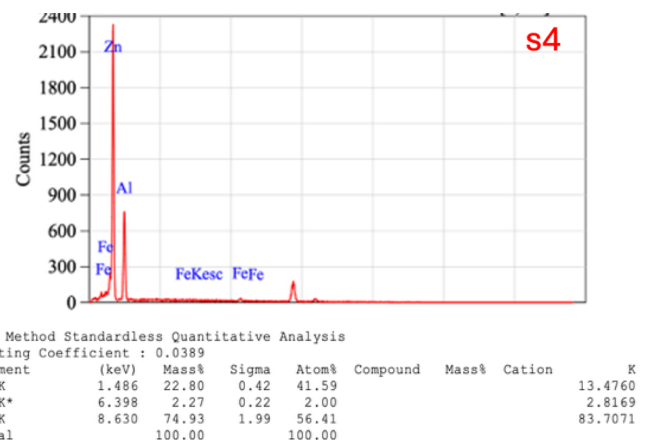
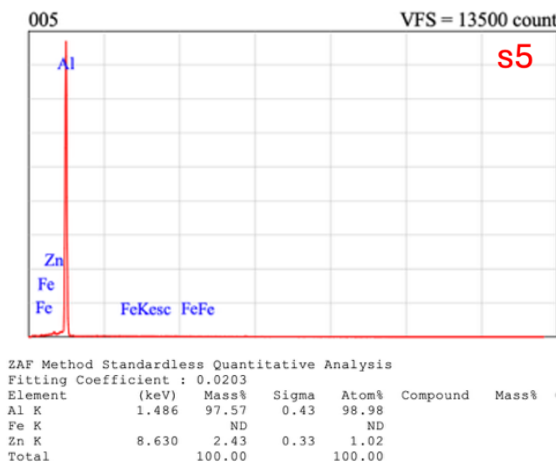
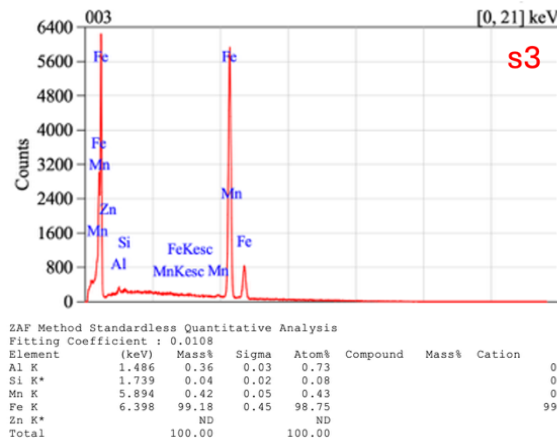
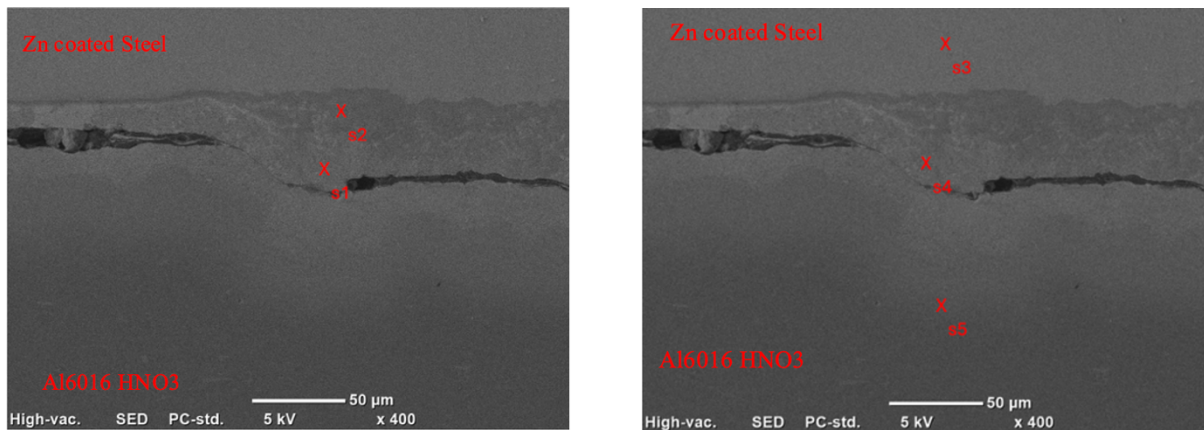


Figure 59. EDS spectra corresponding to point analyses s3, s4 and s5

Similar to point s1, point s4 revealed a Zn-rich interlayer containing approximately Zn (~75 wt%) and Al (~23 wt%), suggesting localised accumulation and redistribution of the molten Zn phase during heating. This composition is consistent with the Al–Zn phase diagram discussed in the literature review. Since the brazing temperature was higher than the melting point of zinc but lower than that of aluminium, the Zn-rich liquid phase could spread along the interface and dissolve a limited amount of aluminium from the Al6016 substrate. The measured Zn-rich composition at point s4 therefore suggests the formation of an Al–Zn interfacial region after cooling, rather than direct melting of the aluminium base material.

Overall, the SEM–EDS analysis confirmed partial metallurgical interaction between the aluminium alloy and the Zn-coated steel substrate. Although a Zn-rich Al–Zn interfacial region was formed, the bonding remained locally discontinuous and chemically heterogeneous. This indicates that joint formation was controlled by molten zinc redistribution, partial aluminium dissolution, and non-uniform local contact conditions during brazing.



Point	Location	Composition (wt%)	Interpretation	Segment direction	Distance between segments (µm)
s1	Interface	Zn (~50), Al (~30), Fe (~15)	Mixed Al-Zn-Fe reaction zone at the interface	From s1 to s2	32
s2	Steel side	Fe (~98-99)	Steel substrate, minimal diffusion		
s3	Steel side	Fe (~98-99)	Steel substrate, minimal diffusion	From s3 to s4	68
s4	Intermediate layer	Zn (~75), Al (~23)	Zn-rich interlayer formed during brazing		
s5	Aluminium side	Al (~97-98)	Aluminium base material	From s4 to s5	77

Table 16. Summary of EDS point analysis (s1-s5) corresponding to SEM micrographs shown above (The reference point taken is the highest point on y axis on the SEM image)

4.2.3 Backscattered electron imaging: HNO₃-treated sample

Backscattered electron (BSE) imaging combined with SEM-EDS analysis was additionally performed on the opposite side of the cross-sectioned brazed specimen in order to further investigate the interfacial morphology and elemental distribution after brazing. Unlike the first analysed side, this specimen surface was prepared using a gentler grinding and polishing procedure to minimise excessive material removal and reduce preparation-induced artefacts, particularly in the softer aluminium region. The use of a milder metallographic preparation method was intended to preserve the original interfacial morphology more accurately and improve the reliability of the compositional observations obtained during SEM examination.

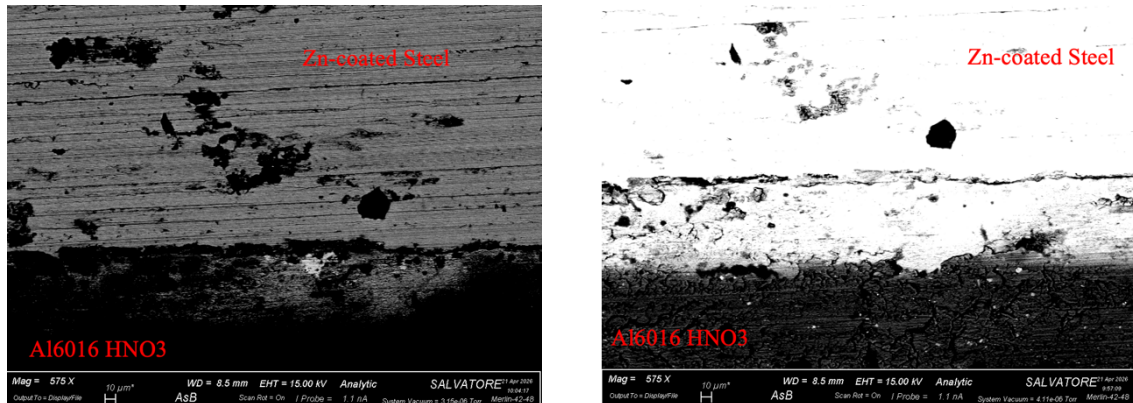


Figure 60. Backscattered electron (BSE) images of HNO₃-treated brazed joint from opposite specimen side

The analysed cross-section corresponded to the opposite side of the cut specimen, allowing comparison between different regions of the same brazed joint. This approach made it possible to evaluate the consistency of the bonding morphology and determine whether the observed interfacial irregularities were localised or representative of the overall joint.

Electron Image 1

In Electron Image 1, the EDS results showed a clear compositional variation across the interfacial region. Point S1, located in the upper region, was characterised by a high Fe content (~92 wt%), while Zn and Al were present only as minor traces. This confirms that this area corresponds to the steel substrate, with limited interfacial diffusion.

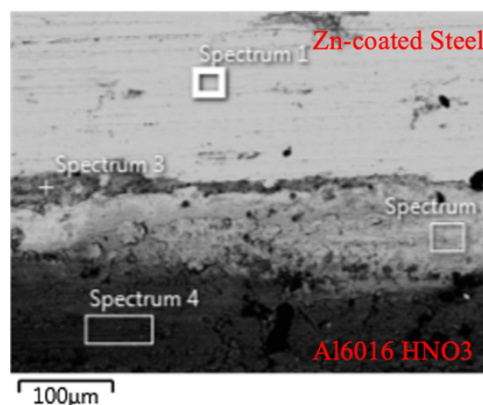


Figure 61. Backscattered electron (BSE) electron image 1 with spectrum from 1 to 4

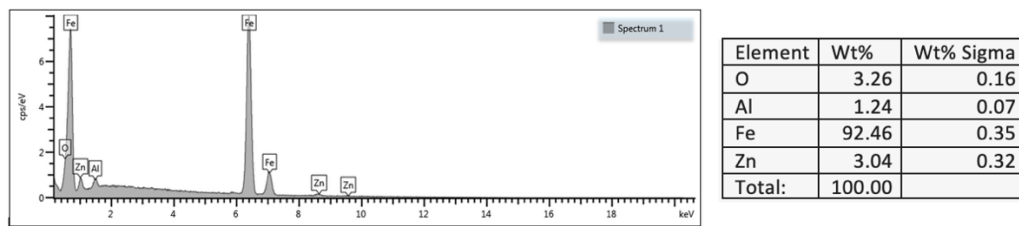


Figure 62. EDS spectrum and elemental composition of spectrum 1

Point S2, located in the intermediate layer, contained a high Zn concentration (~51 wt%) together with Al (~26 wt%) and oxygen. This composition indicates the formation of a Zn-rich interfacial layer during brazing.

Point S3, located in the transition zone, showed a mixed composition of Al (~34 wt%), Zn (~30 wt%), and a small amount of Fe (~4 wt%). This suggests the presence of a local diffusion zone where partial mixing between aluminium and zinc occurred, with limited iron participation.

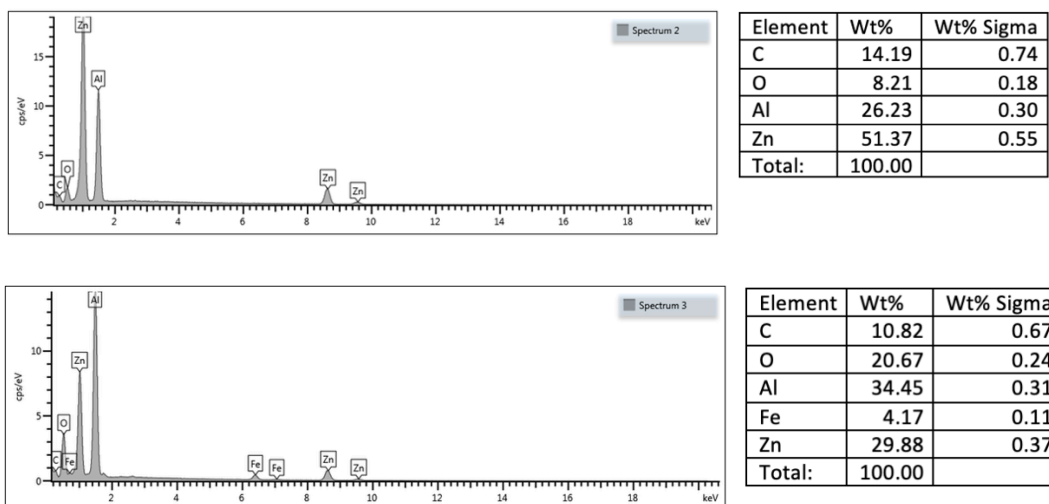


Figure 63. EDS spectrum and elemental composition of spectrum 2 and spectrum 3

Point S4, located in the lower region, was mainly enriched in Al (~81 wt%), confirming that this area corresponds to the aluminium base material. The presence of minor Zn and oxygen indicates limited contribution from the interfacial region and surface oxidation

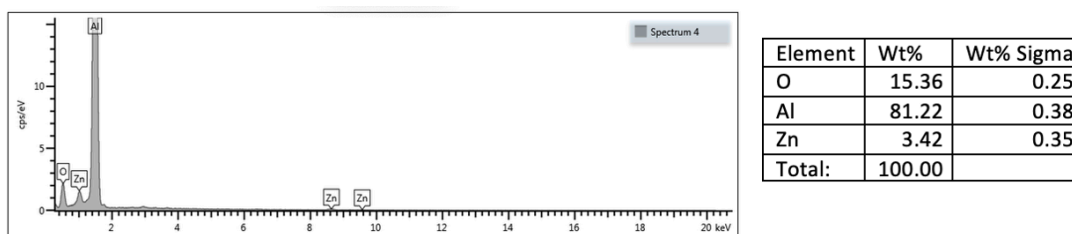


Figure 64. EDS spectrum and elemental composition of spectrum 4

Overall, Electron Image 1 confirms the formation of a chemically heterogeneous interface, with a localised Zn-rich reaction layer between the Fe-rich steel substrate and the Al-rich aluminium material.

Electron Image 2

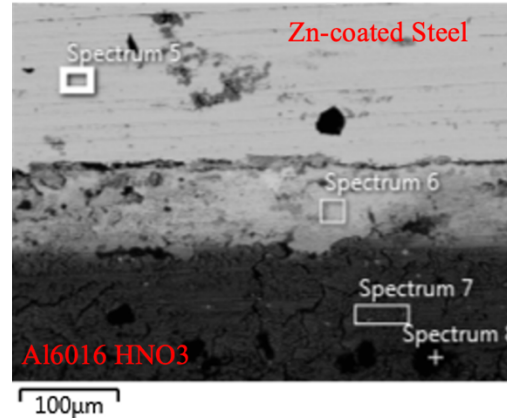


Figure 65. Backscattered electron (BSE) electron image 2 with spectrum from 5 to 8

In Electron Image 2, the EDS results further confirmed the chemical heterogeneity of the HNO₃-treated joint. Point S5, located in the upper region, showed a high Fe content of approximately 96 wt%, indicating that this area corresponds to the steel substrate. The presence of minor Al and Zn signals suggests limited elemental contribution from the adjacent coating or interfacial region.

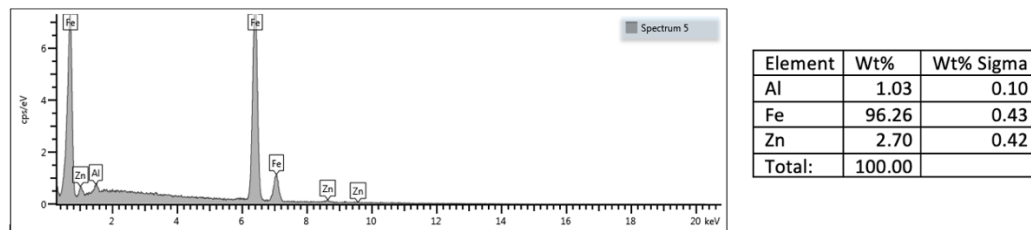


Figure 66. EDS spectrum and elemental composition of spectrum 5

Point S6, positioned within the interfacial layer, was mainly composed of Zn (~67 wt%) and Al (~27 wt%). This composition confirms that the interlayer was Zn-rich and that aluminium participated in the reaction zone during brazing. Therefore, S6 can be interpreted as a localised Zn–Al reaction layer formed between the steel and aluminium substrates.

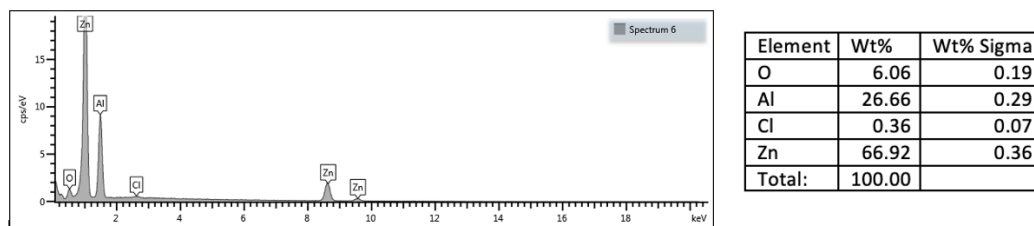


Figure 67. EDS spectrum and elemental composition of spectrum 6

Point S7, located in the aluminium region, contained mainly Al (~89 wt%), with small amounts of oxygen and traces of Fe and Zn. This confirms that the lower region corresponds to the Al6016 base material, while the minor Fe and Zn signals indicate limited interfacial diffusion or residual contribution from the nearby reaction layer.

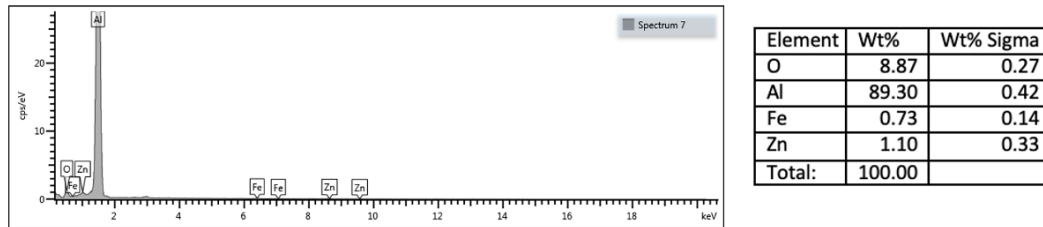


Figure 68. EDS spectrum and elemental composition of spectrum 7

Point S8 showed a very high carbon content (~68 wt%), together with oxygen and minor elements. This composition does not correspond to a metallic reaction layer and is more likely associated with contamination, etching residue, polishing debris, or mounting material. Therefore, S8 was not considered representative of the true metallurgical interface.

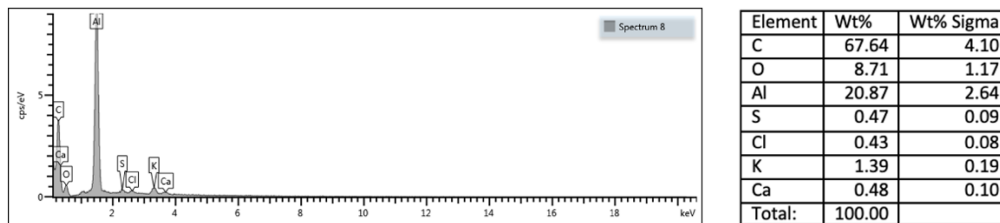


Figure 69. EDS spectrum and elemental composition of spectrum 8

Overall, Electron Image 2 confirms that the joint consisted of clearly separated Fe-rich steel and Al-rich aluminium regions, with a localised Zn-rich interfacial layer between them.

BSE observations combined with SEM-EDS analysis demonstrated that the interfacial region was chemically heterogeneous and morphologically non-uniform. The steel substrate remained predominantly Fe-rich, while the aluminium side preserved its Al-rich composition. Between these two regions, localised Zn-rich interfacial layers and transition zones were identified, indicating partial interaction between the substrates during brazing. However, the reaction products were discontinuous and irregularly distributed along the interface, suggesting that stable metallurgical bonding was achieved only locally. The presence of isolated diffusion regions together with interfacial discontinuities confirms that the selected brazing conditions were insufficient to produce a continuous and homogeneous bonded joint across the entire interface.

4.2.4 Optical microscopy: KOH-treated sample

Optical microscopy observations performed on the KOH-treated specimen revealed a significantly different morphology compared to the HNO₃-treated sample. A large and continuous gap was visible along the joining region between the Al6016 alloy and the Zn-coated steel substrate, indicating the absence of effective metallurgical bonding in the interface.

The observed morphology suggests that the molten Zn-rich phase did not adequately wet or bridge the contact surfaces during brazing at 480 °C. Instead of forming a thin and continuous interfacial layer, the joining zone contained void-like regions and open separations extending along the interface. These features indicate insufficient interfacial interaction and limited diffusion between the aluminium substrate and the steel surface.

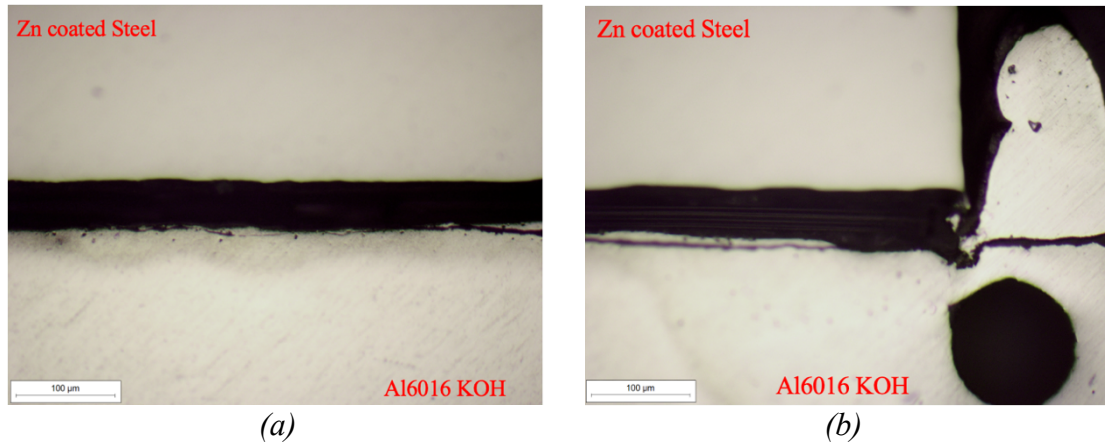


Figure 70. Optical micrograph (100×) showing: (a) an extensive interfacial gap and absence of bonding along the interface; (b) separation at the edge of interface

4.2.5 SEM-EDS: KOH-treated sample

The analysed point located near the interface contained a mixture of Zn, Al, and Fe, indicating localised formation of a reaction region involving material transfer between the two substrates. However, despite the presence of these elements, the overall morphology demonstrated that the reaction products remained spatially isolated and were not sufficient to produce continuous bonding across the joint.

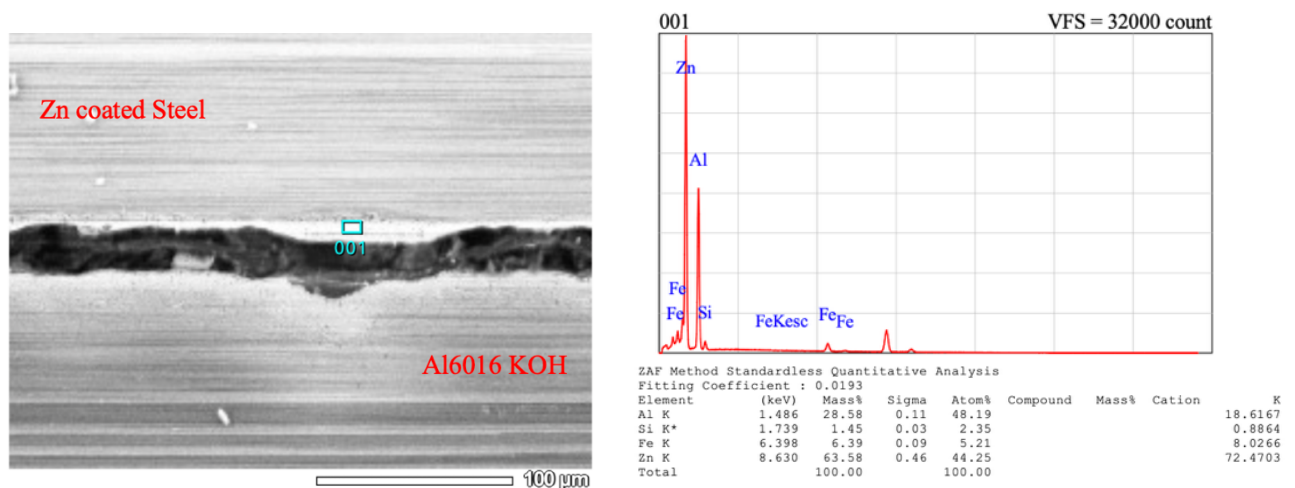


Figure 71. SEM micrograph and corresponding EDS spectrum showing localised Zn-Al-Fe reaction products at the interfacial regions

The optical and SEM observations therefore indicate that the KOH-treated sample did not achieve successful brazing under the selected processing conditions. The excessive interfacial

gap prevented stable contact between the substrates and inhibited the formation of a continuous Zn-rich bonding layer. As a result, the obtained microstructure cannot be considered representative of a properly bonded brazed joint.

4.2.6 Summary of Trial 1

Trial 1 was performed at 480 °C in order to evaluate the brazing behaviour between Al6016 aluminium alloy and Zn-coated steel after different surface treatments. Optical microscopy, SEM-EDS analysis, and BSE imaging revealed that the obtained joint was characterised by heterogeneous interfacial morphology and limited metallurgical bonding.

For the HNO₃-treated sample, localised bonding regions were observed together with the formation of Zn-rich interfacial regions between the aluminium and steel substrates. SEM-EDS and BSE analyses confirmed the existence of chemically heterogeneous transition zones containing mainly Zn and Al, with limited Fe participation. The steel side remained predominantly Fe-rich, while the aluminium side preserved its Al-rich composition. Although localised diffusion and reaction regions were detected, the interfacial layer was discontinuous and non-uniform along the joint.

In contrast, the KOH-treated sample exhibited a large and continuous interfacial gap. Optical microscopy showed no physical contact between the substrates, indicating the absence of effective bonding over most of the interface. SEM-EDS analysis detected only isolated reaction products containing Zn, Al, and Fe, while no continuous Zn-rich bonding layer was formed.

4.3 Trial 2: Brazing at 470 °C

4.3.1 Optical microscopy: HNO₃-treated sample

One side of the analysed cross-section exhibited a large and continuous interfacial gap with an approximate thickness of 120–150 µm. As a result, the obtained optical micrographs from this region were not considered representative of effective metallurgical bonding.

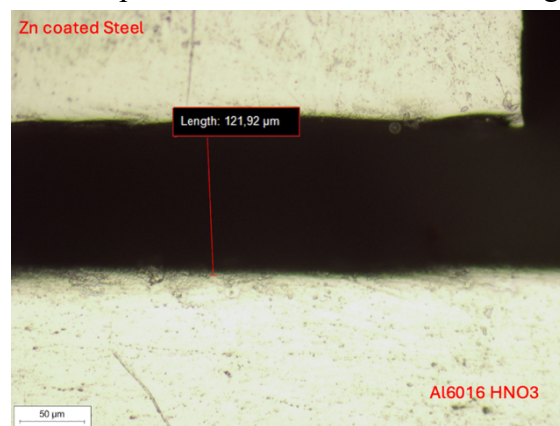


Figure 72. Optical micrograph (200×) showing a large interfacial gap (~120-150 µm) In contrast, observations performed on another side of the same specimen revealed a more continuous and better-defined interface. Unlike the regions exhibiting excessive interfacial

separation, this area showed visible interfacial contact together with the presence of localised reaction products distributed along the bonding zone. The interface appeared morphologically heterogeneous but remained partially connected, suggesting that localised metallurgical interaction occurred during brazing.

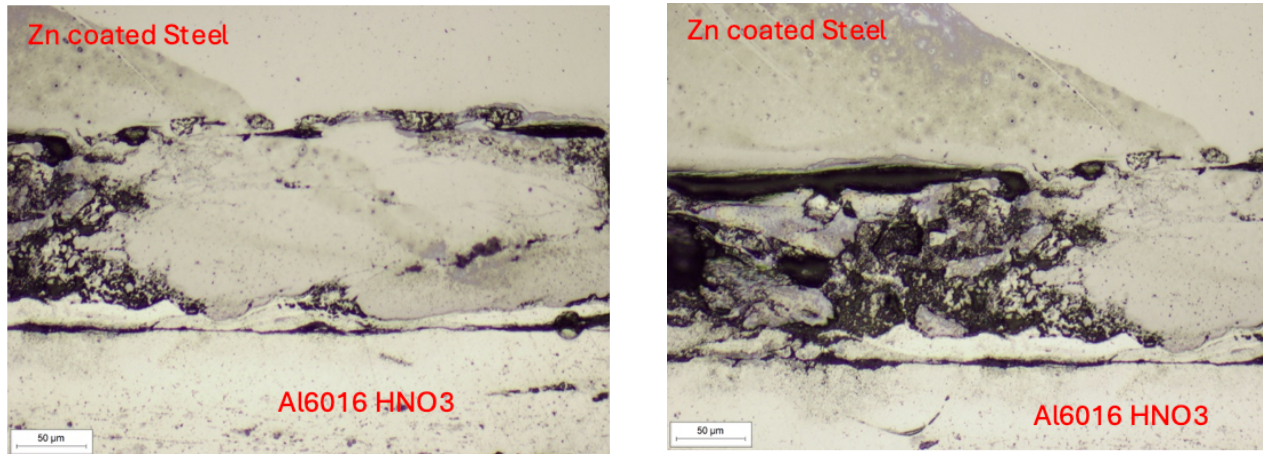


Figure 73. Optical micrograph (200×) on the opposite side of HNO_3 -treated specimen

No major continuous gaps were observed in these regions, although the morphology remained irregular and non-uniform. The observed structure suggests that under locally favourable contact conditions the brazing process was capable of producing partial interfacial bonding and localised reaction layer formation. However, the interfacial continuity remained limited and no fully homogeneous bonding layer was formed across the joint.

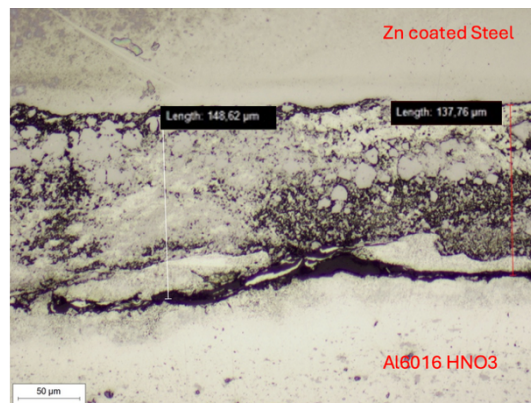


Figure 74. Optical micrograph (200×) showing locally developed reaction zone having thickness of approximately 139-149 µm

It should also be considered that the softer aluminium phase is more susceptible to polishing-induced material removal during metallographic preparation. Excessive polishing may locally deform or remove material from the aluminium side, thereby modifying the apparent morphology of the interface and potentially leading to overestimation of bonding quality. For this reason, a milder and shorter grinding and polishing procedure was applied on this side of the specimen in order to minimise preferential removal of the softer aluminium phase and better preserve the original interfacial morphology.

4.3.2 SEM-EDS: HNO₃-treated sample

Since the two substrates were not continuously joined, the analysis was focused on investigating the local chemical composition at the separated joint region of the aluminium alloy and Zn-coated steel, where possible localised reactions between Al, Zn, and Fe could be detected. Several points (x1–x6) were selected across the interfacial region, extending from the reaction layer toward the aluminium and steel substrates. The obtained results confirmed the formation of a chemically heterogeneous interfacial zone dominated by Zn-rich phases with localised Al and Fe diffusion.

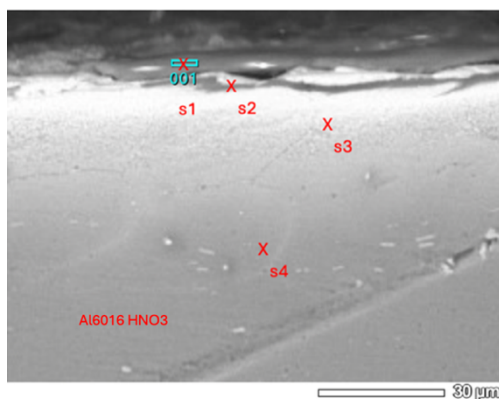


Figure 75. SEM micrograph showing the locations of EDS point analyses x1 to x4

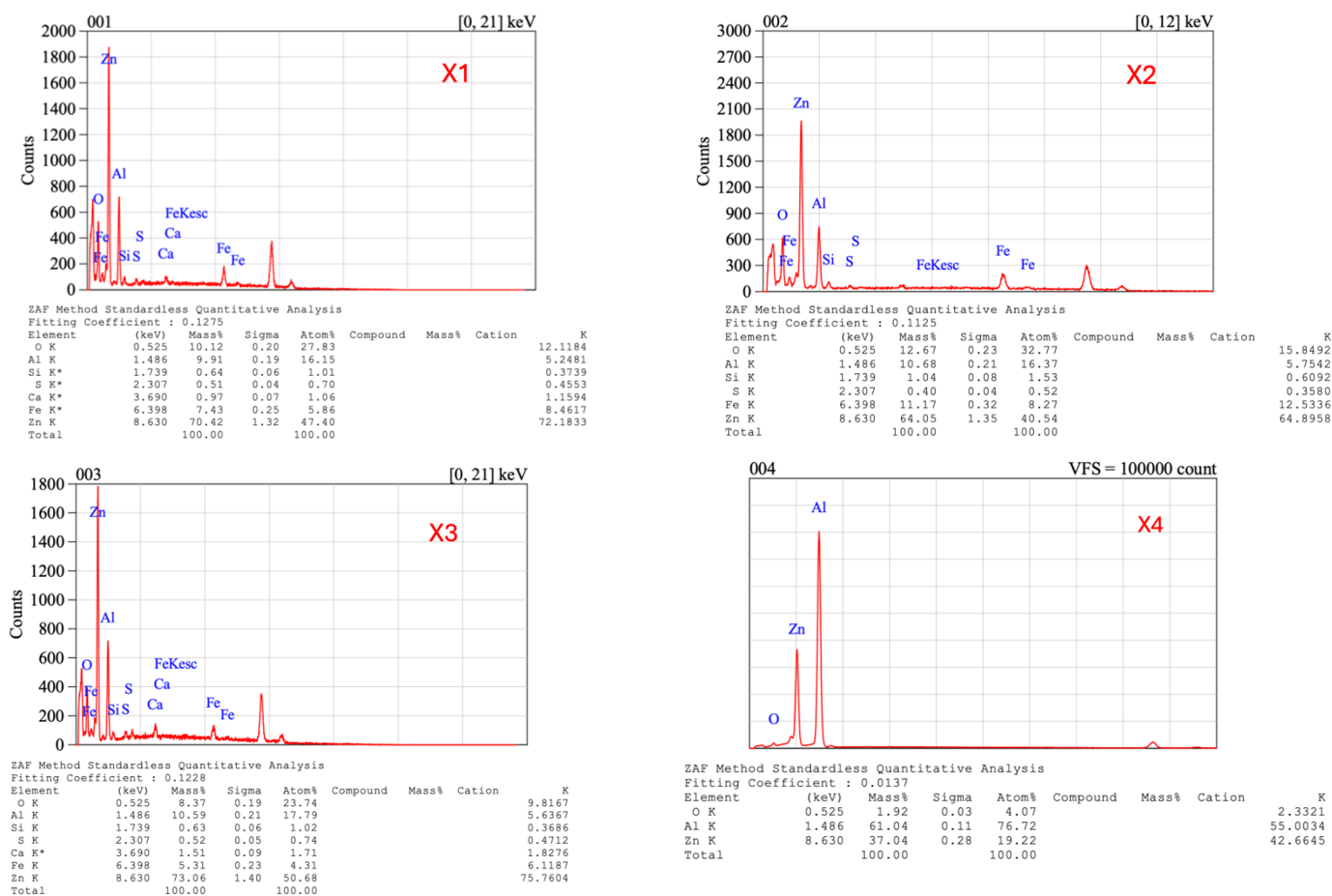


Figure 76. EDS spectra corresponding to point analyses x1 to x4

Points x1, x2, and x3, located directly within the interfacial layer, showed high Zn concentrations ranging from approximately 64 to 73 wt%, confirming that the reaction zone was primarily Zn-rich. At point x1, the composition consisted of approximately 70 wt% Zn, 20 wt% Al, and 7 wt% Fe, indicating the formation of a Zn-dominated reaction phase containing dissolved aluminium and limited iron diffusion.

Point x2 contained approximately 64 wt% Zn, 11 wt% Fe, 10 wt% Al, and about 12 wt% oxygen, suggesting partial oxidation within the interlayer together with localised interaction between Zn and Fe.

Point x3 exhibited approximately 73 wt% Zn, 11 wt% Al, 5 wt% Fe, and 8 wt% oxygen, corresponding to a relatively stable Zn-rich interfacial region with limited Fe diffusion from the steel substrate.

Point x4, positioned closer to the aluminium side, showed approximately 61 wt% Al and 37 wt% Zn, indicating significant Al–Zn mixing within the transition region. This composition suggests the formation of an Al–Zn intermediate zone or eutectic-like structure produced by localised diffusion and partial melting during brazing. The absence of high Fe concentration in this region indicates that iron diffusion remained limited mainly to the immediate vicinity of the steel interface.

The compositional data from points x1–x4 can be interpreted with reference to the Al–Zn phase diagram and the selected brazing temperature. Points x1, x2 and x3 were dominated by zinc, with Zn contents between approximately 64 and 73 wt%. Since the brazing temperature was above the melting point of zinc but below the melting point of aluminium, these compositions suggest that the molten Zn-rich phase spread along the interface and dissolved a limited amount of aluminium from the Al6016 substrate. After cooling, these regions formed Zn-rich Al–Zn interfacial zones rather than regions produced by direct melting of the aluminium base material. The limited Fe content detected in these points indicates only local interaction with the steel side, while the oxygen detected especially at point x2 may be associated with local oxide contribution within the interfacial layer.

Point x4, located closer to the aluminium side, showed a higher aluminium content of approximately 61 wt% Al and 37 wt% Zn. According to the Al–Zn system, this composition indicates an Al-rich transition region where zinc locally mixed with or diffused into the aluminium side during brazing. Therefore, the EDS data show a gradual chemical transition from Zn-rich interfacial regions toward the aluminium-rich substrate, confirming that the joint formation was controlled by molten zinc redistribution, partial aluminium dissolution and limited local Fe contribution.

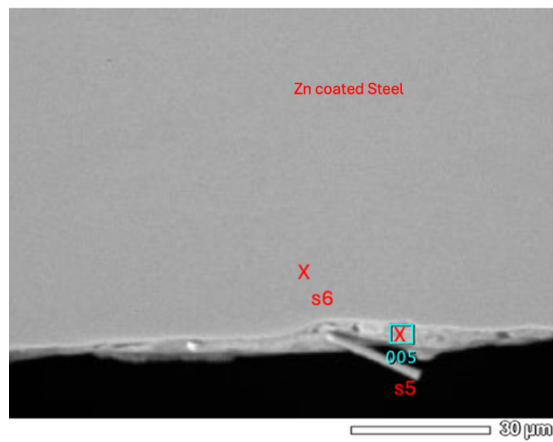


Figure 77. SEM micrograph showing the locations of EDS point analyses x5 and x6

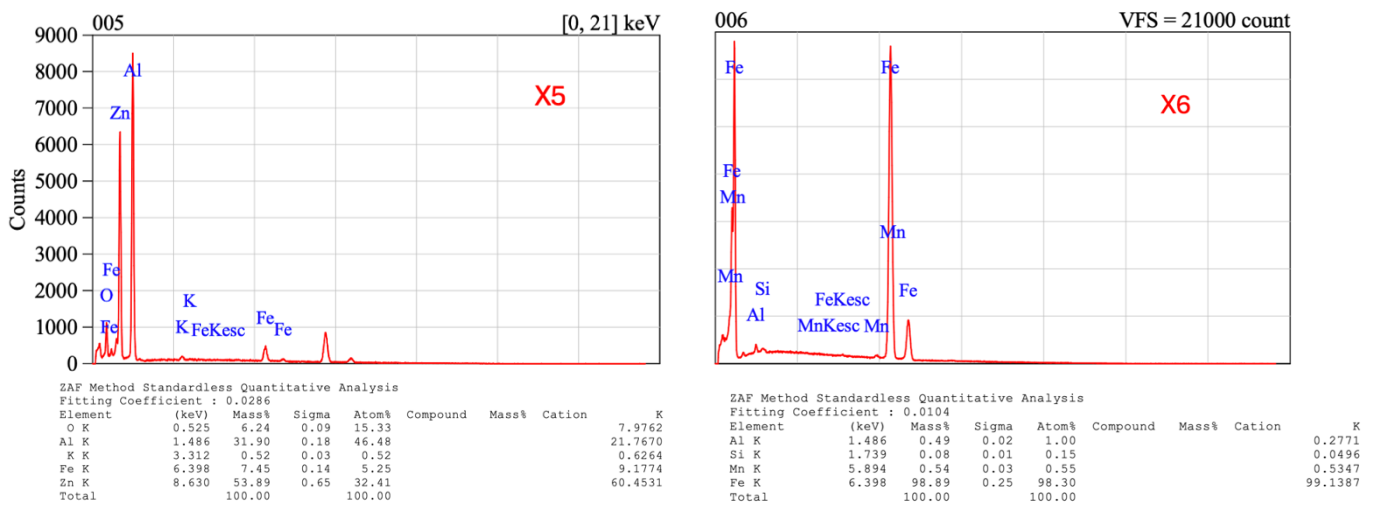
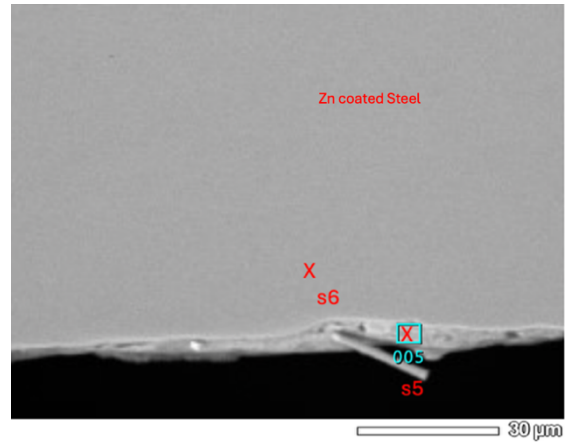
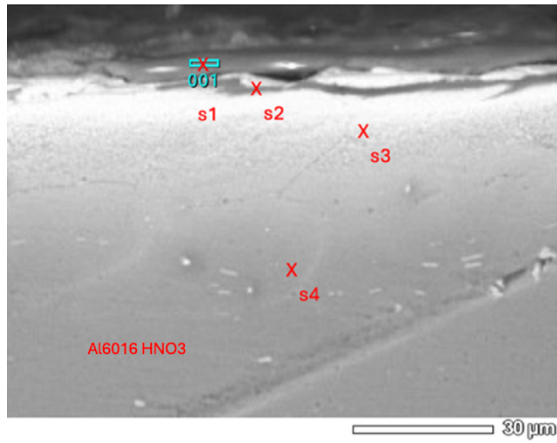


Figure 78. EDS spectra corresponding to point analyses x5 to x6

Additional measurements performed on another interfacial region confirmed similar compositional behaviour. Point x5 contained approximately 54 wt% Zn, 31 wt% Al, and 7 wt% Fe, corresponding to a mixed Zn–Al interfacial layer with minor Fe contribution. In contrast, point x6 showed approximately 99 wt% Fe, confirming the steel substrate composition and indicating that the steel base material remained chemically stable during the brazing process.



Point	Location	Composition (wt%)	Interpretation	Segment direction	Distance between segments (μm)
x1	Interface	Zn (~70), Al(~20), Fe(~7)	Zn-dominated phase with Al and minor Fe → reaction layer(Al,Zn,Fe)	From x1 to x2	5
x2	Interface	Zn (~64), Fe(~11), Al(~10), O (~12)	Zn-rich region with oxidation and Fe → partially reacted interlayer	From x2 to x3	9
x3	Interface	Zn (~73), Al (~11), Fe (~5), O (~8)	Zn-rich phase with slight Fe diffusion → stable Zn interlayer	From x3 to x4	29
x4	Near Al side	Al (~61), Zn (~37)	Strong Al–Zn mixing → Al–Zn eutectic/intermediate zone	From x1 to x4	44
x5	Interface	Zn (~54), Al (~31), Fe (~7)	Mixed Zn–Al with Fe traces → transition layer	From x5 to x6	16
x6	Steel	Fe (~99)	Pure Fe → steel substrate		

Table 17. Summary of EDS point analysis (x1-x6) corresponding to SEM micrographs shown above (The reference point taken is the highest point on y axis on the SEM image)

4.3.3 Backscattered electron imaging: HNO₃-treated sample

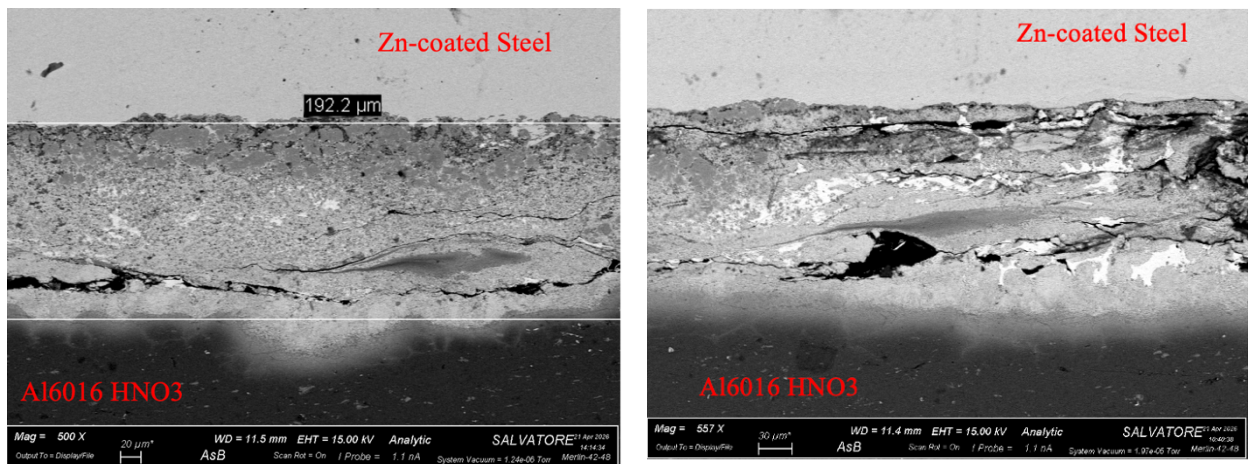


Figure 79. Backscattered electron (BSE) images of HNO₃-treated brazed joint from opposite specimen side with measured layer thickness of 192.2 μm

The BSE-SEM observations revealed the formation of a relatively continuous interfacial layer between the two materials. In several regions, the interface exhibited an approximate diffusion layer thickness of 192.2 μm, indicating significant interaction between the aluminium alloy and the Zn coating during the brazing process. The interfacial region appeared more developed and chemically mixed, suggesting enhanced diffusion and localised metallurgical bonding under the selected processing conditions.

Electron Image 1

In Electron Image 1, point S9, located in the upper region, showed 100 wt% Fe, clearly corresponding to the steel substrate with no significant diffusion of Al or Zn into this region.

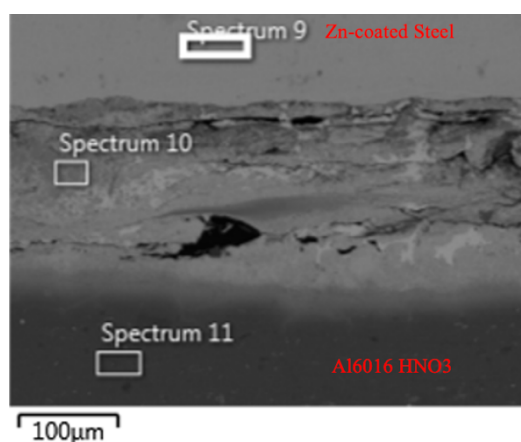


Figure 80. Backscattered electron (BSE) electron image 1 with spectrum from 9 to 11

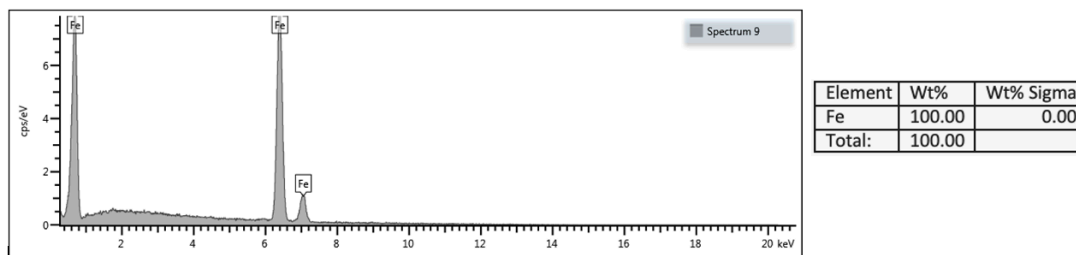


Figure 81. EDS spectrum and elemental composition of spectrum 9

Point S10, located within the joining zone, exhibited a mixed composition consisting of approximately 40.50 wt% Al, 38.46 wt% Zn, 14.69 wt% Fe, 4.79 wt% O, and minor Si contribution. This composition indicates the formation of a multi-component diffusion zone produced by interfacial interaction between aluminium, the Zn coating, and the steel substrate during brazing. The simultaneous presence of Al, Zn, and Fe within the same region suggests enhanced metallurgical interaction and confirms the development of a more chemically mixed interfacial layer.

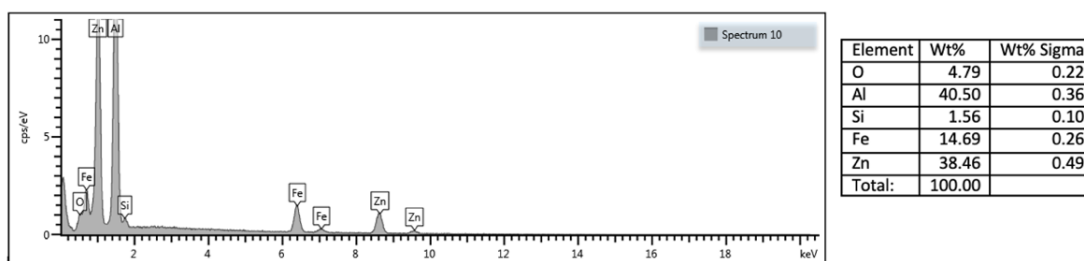


Figure 82. EDS spectrum and elemental composition of spectrum 10

Point S11, located in the lower region corresponding to the aluminium side, was dominated by Al with approximately 99.05 wt% Al and only 0.95 wt% Fe, confirming the aluminium base material with minimal iron diffusion into the substrate.

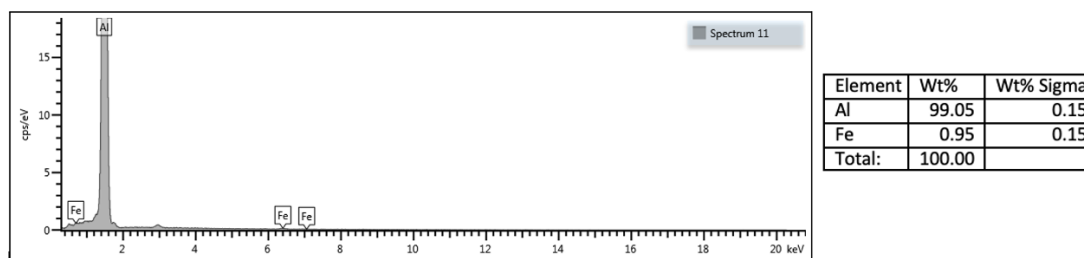


Figure 83. EDS spectrum and elemental composition of spectrum 11

Electron Image 2

In Electron Image 2, point S12, located in the upper region near the Zn-coated steel side, exhibited a high Zn concentration of approximately 89.12 wt%, together with minor amounts of Al, Fe, O, and Si. This composition indicates that the analysed region mainly corresponds to the Zn-rich coating layer, while the presence of small Al and Fe contributions suggests localised diffusion and interfacial interaction during brazing.

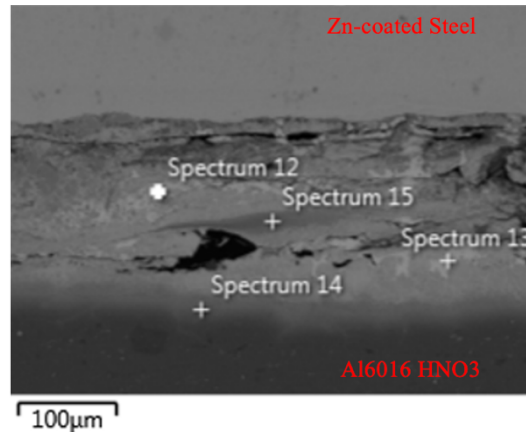


Figure 84. Backscattered electron (BSE) electron image 2 with spectrum from 12 to 15

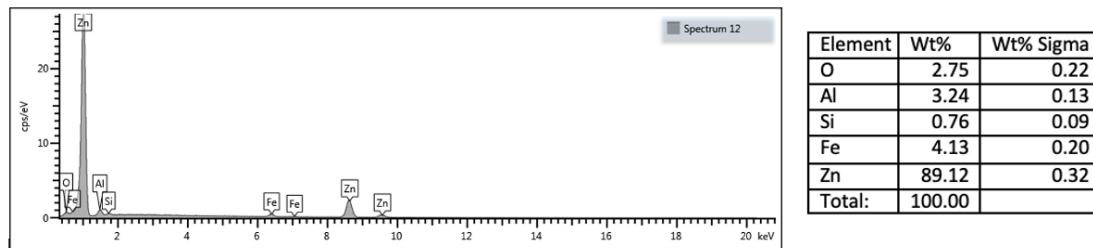


Figure 85. EDS spectrum and elemental composition of spectrum 12

Point S13 is located on the aluminium-side region of the joint, close to the Al–Zn interaction area. The EDS result shows nearly pure Zn composition, with approximately 100 wt% Zn. This indicates a localised Zn-rich region formed mainly by the reaction between the aluminium side and the zinc brazing material. Therefore, S13 should be interpreted as a residual or locally concentrated Zn-rich phase within the aluminium-side joining zone, associated with incomplete homogenisation of molten zinc during brazing at 470 °C.

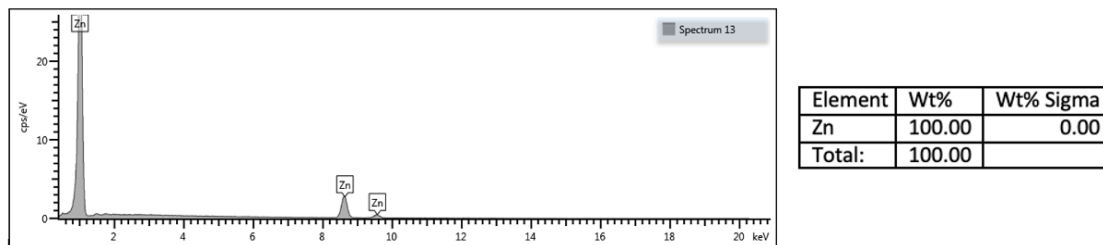


Figure 86. EDS spectrum and elemental composition of spectrum 13

Point S14, corresponding to the reaction at the Al interface, contained approximately 63.90 wt% Al and 36.10 wt% Zn. The absence of significant Fe contribution in this region indicates

the formation of an Al–Zn mixed layer produced by diffusion between the aluminium alloy and the Zn coating. This composition is consistent with the development of an intermediate interfacial region characterised by partial metallurgical interaction and localised chemical mixing.

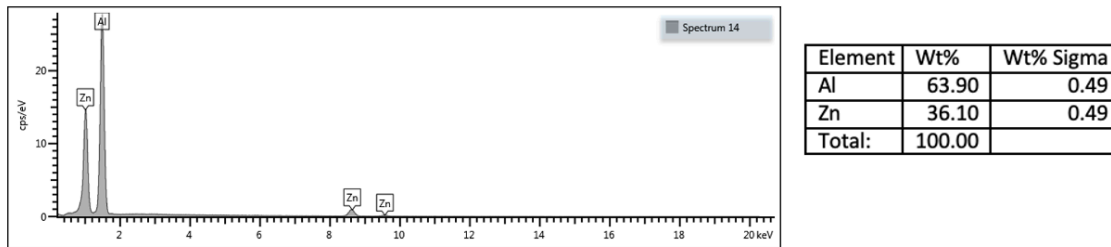


Figure 87. EDS spectrum and elemental composition of spectrum 14

Point S15, located in the joining zone, exhibited nearly balanced concentrations of Al and Zn, with approximately 49.85 wt% Al and 47.20 wt% Zn, together with a minor oxygen contribution. This composition confirms intensive interdiffusion and chemical mixing between aluminium and zinc during brazing. The nearly equivalent Al and Zn contents suggest the formation of a highly mixed interfacial layer, indicating stronger metallurgical interaction compared with the more heterogeneous regions observed in Trial 1.

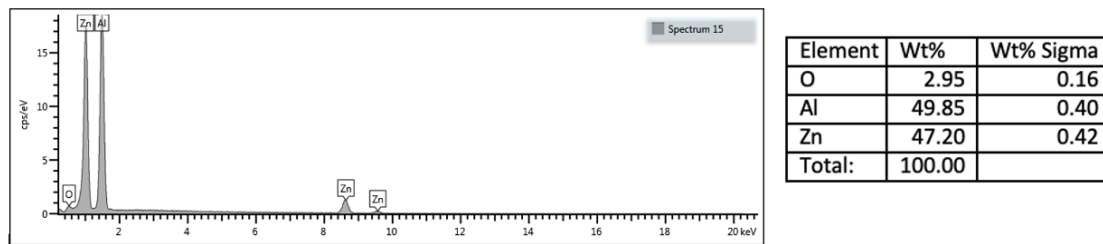


Figure 88. EDS spectrum and elemental composition of spectrum 15

Elemental mapping

Elemental mapping (EDS mapping) was performed in order to evaluate the spatial distribution of the main elements across the brazed joint and to investigate the development of the interfacial diffusion layer between the Al6016 alloy and the Zn-coated steel substrate. Elemental mapping confirmed the spatial distribution of the principal elements across the joint and demonstrated the presence of a developed interfacial diffusion region.

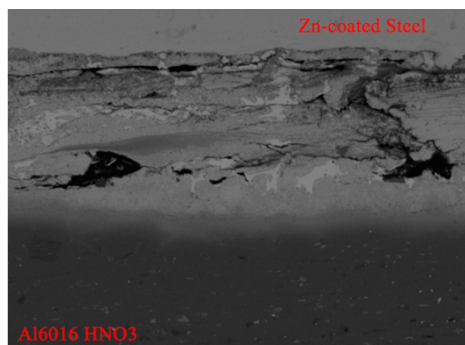


Figure 89. Backscattered electron (BSE) electron image treated for elemental mapping

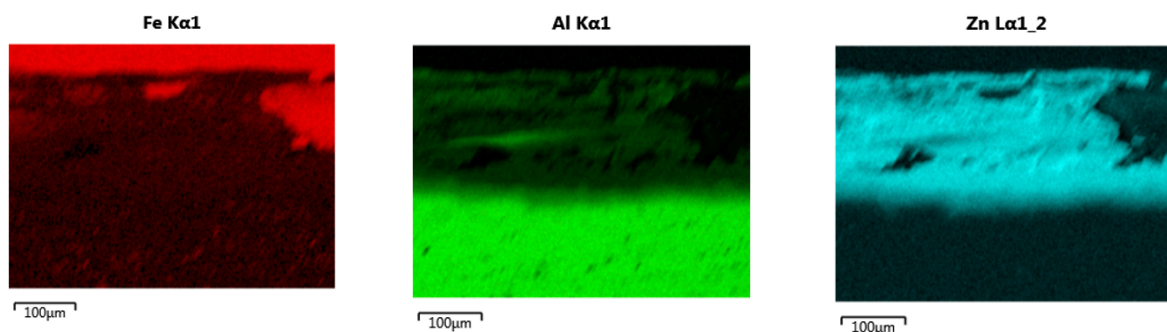


Figure 90. Elemental distribution map of Fe, Al and Zn obtained by EDS mapping

The Fe map (red) showed that iron was concentrated predominantly in the upper region corresponding to the steel substrate. The Al map (green) demonstrated that aluminium was mainly distributed within the lower region corresponding to the aluminium base material. The Zn map (blue) revealed that zinc was localised primarily along the interfacial region, forming a transition layer between the steel and aluminium substrates.

An overlap between the Al (green) and Zn (blue) signals was clearly observed at the interface, indicating the formation of an Al–Zn diffusion layer and confirming strong localised interaction between the aluminium alloy and the Zn coating during brazing. The elemental distribution suggests that partial melting and redistribution of the Zn coating occurred during heating, promoting chemical mixing and diffusion within the reaction zone.

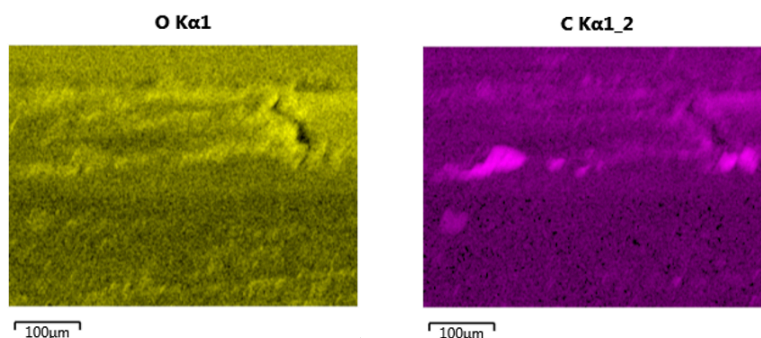


Figure 91. Elemental distribution map of O and C obtained by EDS mapping

Additionally, the O map (yellow) revealed localised oxygen-rich regions along the interface. This oxygen presence is likely associated with surface oxidation or residual products remaining after the HNO₃ etching process. The C map (purple) showed localised carbon-rich areas, which are most likely related to contamination, mounting resin, or polishing residues rather than to primary interfacial reaction products.

Overall, the combined BSE and elemental mapping analyses confirmed the development of a chemically heterogeneous but continuous interfacial diffusion region in Trial 2. The coexistence of Al, Zn, and limited Fe contributions within the reaction zone demonstrated enhanced interfacial interaction and localised metallurgical bonding between the Al6016 alloy and the Zn-coated steel during brazing at 470 °C.

4.3.4 Optical microscopy: KOH-treated sample

Optical microscopy was performed on the KOH-treated cross-section of the brazed Al6016/Zn-coated steel joint in order to evaluate the general morphology of the interface after chemical etching. The observations revealed the presence of a large gap between the aluminium alloy and the Zn-coated steel substrate. Due to this excessive separation, a continuous bonding region was not established across the interface.

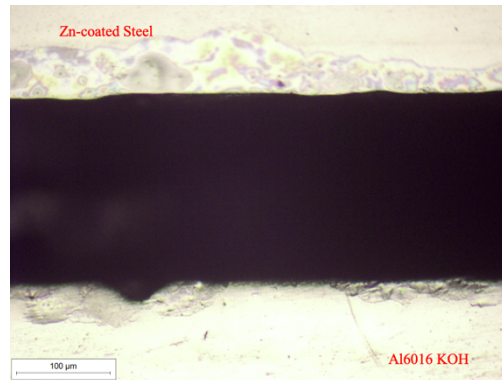


Figure 92. Optical micrograph (100×) showing an extensive interfacial gap over the interface

The optical micrographs showed no contact between the two substrates, indicating that proper metallurgical interaction did not occur during brazing. The interface appeared discontinuous, with non-contact areas extending along the joint line. As a result, the obtained optical images did not provide sufficient evidence of a developed diffusion layer or effective interfacial bonding. Consequently, the optical microscopy results for the KOH-treated sample were considered less representative for detailed interfacial interpretation compared with the HNO₃-treated sample.

4.3.5 SEM-EDS: KOH-treated sample

Since the two substrates were not continuously joined, the analysis was focused on investigating the local chemical composition at the separated joint region of the aluminium alloy and Zn-coated steel, where possible localised reactions between Al, Zn, and Fe could be detected. The analysed points x2–x4 were selected to evaluate the compositional variation within the reaction zone.

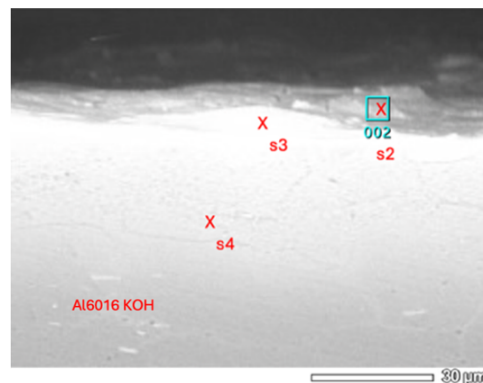


Figure 93. SEM micrograph showing the locations of EDS point analyses x2 to x4

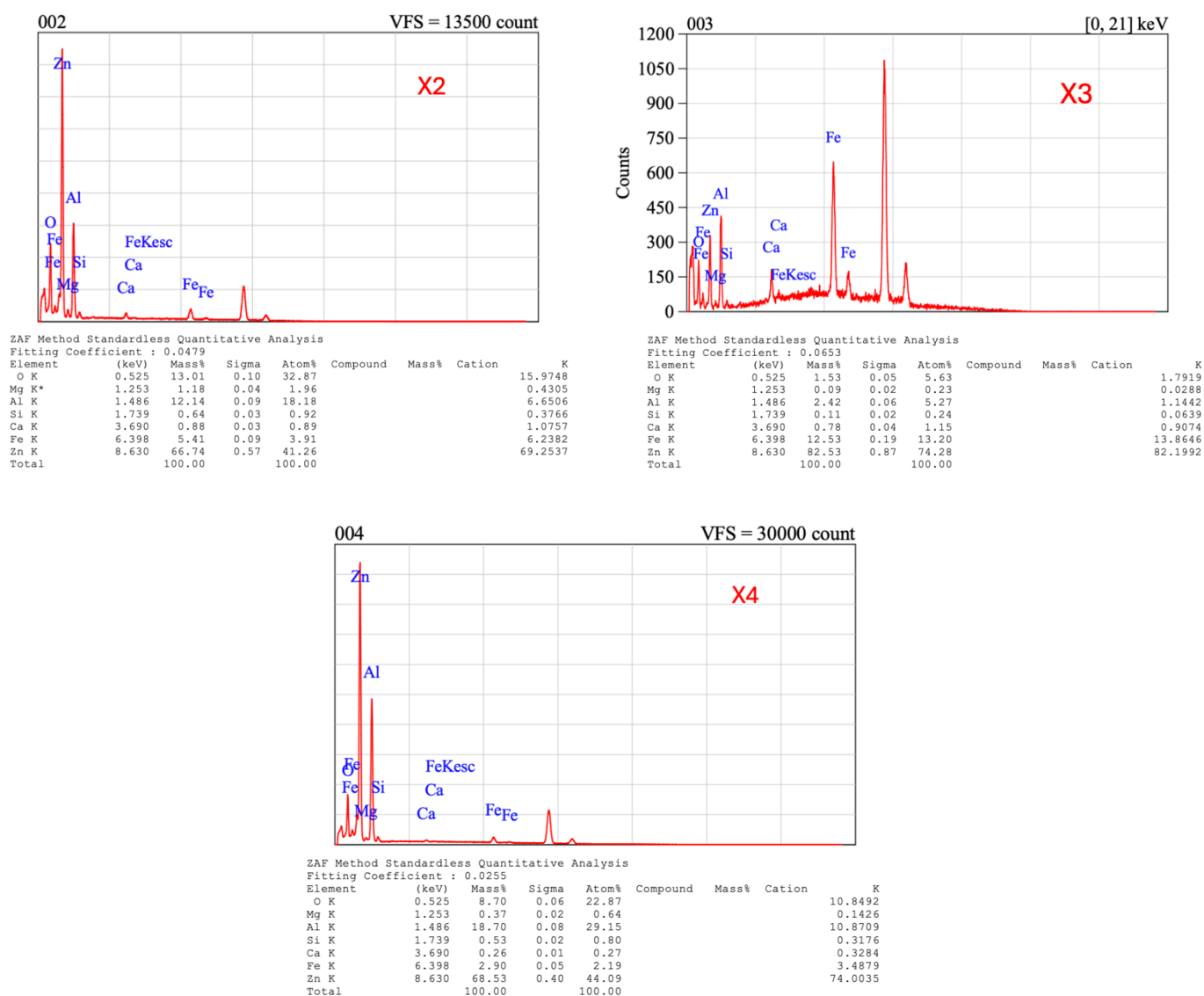


Figure 94. EDS spectra corresponding to point analyses x2 to x4

Point x2, located at the interface, showed a Zn-rich composition containing approximately 66.7 wt% Zn, 12.1 wt% Al, 13.0 wt% O, and 5.4 wt% Fe. The relatively high oxygen content suggests the presence of oxidation products within the interfacial region. This area can therefore be interpreted as an oxidised Zn-rich interlayer with minor Fe contribution.

Point x3, also positioned within the interfacial region, exhibited a strong Zn-rich composition with approximately 82.5 wt% Zn and 12.5 wt% Fe, while the Al content remained relatively low at about 2.4 wt%. The coexistence of Zn and Fe in this region indicates localised interaction between the Zn coating and the steel substrate, forming a Zn–Fe interaction zone during heating.

Point x4, located closer to the aluminium side, contained approximately 68.5 wt% Zn, 18.7 wt% Al, 8.7 wt% O, and 2.9 wt% Fe. The simultaneous presence of Zn and Al suggests the formation of an Al–Zn intermediate region near the aluminium substrate. The detected oxygen content additionally indicates partial surface oxidation within this area.

Point	Location	Composition (wt%)	Interpretation	Segment direction	Distance between segments (μm)
x2	Interface	Zn (~66.7), Al (~12.1), O (~13.0), Fe (~5.4)	Zn-rich phase with oxide and minor Fe $\rightarrow$ oxidised Zn interlayer	From x2 to x3	2
x3	Interface	Zn (~82.5), Fe (~12.5), Al (~2.4)	Strong Zn phase with Fe diffusion $\rightarrow$ Zn–Fe interaction zone	From x3 to x4	25
x4	Near Al side	Zn (~68.5), Al (~18.7), O (~8.7), Fe (~2.9)	Zn-rich layer with Al mixing and oxidation $\rightarrow$ Al–Zn intermediate layer	From x2 to x4	27

Table 18. Summary of EDS point analysis (x2-x4) corresponding to SEM micrograph shown above (The reference point taken is the highest point on y axis on the SEM image)

4.3.6 Summary of Trial 2

Trial 2 demonstrated a significantly more developed interfacial interaction between the Al6016 alloy and the Zn-coated steel compared with the previous trial. The combined optical microscopy, SEM, BSE, EDS point analysis, and elemental mapping results confirmed the formation of a continuous interfacial diffusion region characterised by localised chemical mixing between Al, Zn, and Fe.

The HNO₃-treated sample exhibited a relatively continuous reaction layer with visible redistribution of the Zn coating along the interface. SEM and BSE observations revealed a heterogeneous interfacial morphology consisting of Zn-rich and Al–Zn mixed regions. EDS point analyses confirmed the coexistence of Al, Zn, and limited Fe within the reaction zone, indicating the development of localised metallurgical bonding during brazing at 470 °C.

In contrast, the KOH-treated sample showed a considerably large interfacial gap between the substrates. Optical microscopy indicated no physical contact and absence of continuity of the joint region. Although SEM–EDS analysis still identified Zn-rich interfacial regions with minor Al and Fe interaction, these regions remained localised and did not form a continuous bonded interface.

Overall, Trial 2 confirmed that the brazing conditions promoted interfacial diffusion and reaction layer formation between the aluminium alloy and the Zn-coated steel. Among the investigated surface preparation conditions, the HNO₃-treated sample demonstrated superior interfacial continuity, stronger elemental interaction, and a more developed diffusion layer.

4.4 Trial 3: Brazing at 480 °C (repeatability condition)

4.4.1 Optical microscopy: HNO₃-treated sample

Optical microscopy observations of the HNO₃-treated Al6016/Zn-coated steel joint brazed at 480 °C revealed the formation of a non-uniform dark interfacial layer between the aluminium alloy and the Zn-coated steel substrate. The presence of this intermediate layer indicates that bonding occurred through the development of a reaction or diffusion zone rather than by direct Al–Fe contact. The interfacial morphology appeared irregular along the joint line and exhibited significant local variation in thickness and continuity.

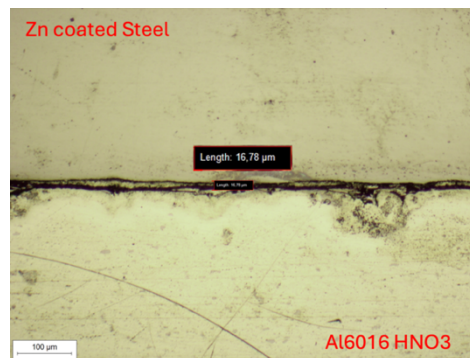


Figure 95. Optical micrograph (100×) showing a thin interfacial layer with an approximate local thickness of 16.78µm

Several regions showed globular and heterogeneous interfacial features, suggesting localised melting and redistribution of the Zn-rich interlayer during heating. The observed morphology indicates that the joining process likely involved partial liquid-phase participation within the reaction zone. However, optical microscopy alone does not allow direct phase identification, and therefore the exact composition of these regions requires further SEM–EDS investigation.

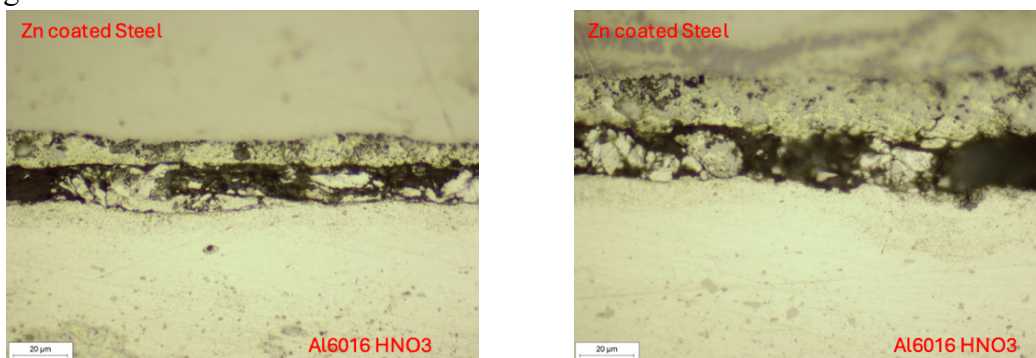


Figure 96. Optical micrograph (200×) showing a thin interfacial layer with localised globular features, porosity, and interfacial discontinuities along the brazed region

In multiple areas, the interface contained visible porosity, voids, and crack-like discontinuities distributed along the joint region. These defects suggest incomplete bonding and possible gas evolution during the brazing process. Localised interfacial separation and discontinuous regions were also observed, indicating that the bonding quality was not uniform throughout the sample.

4.4.2 SEM-EDS: HNO₃-treated sample

The SEM observations confirmed the presence of an irregular diffusion layer with non-uniform morphology, local discontinuities, and partially separated regions distributed along the interface. Several areas exhibited porous and crack-like features, indicating incomplete bonding and localised instability during the brazing process.

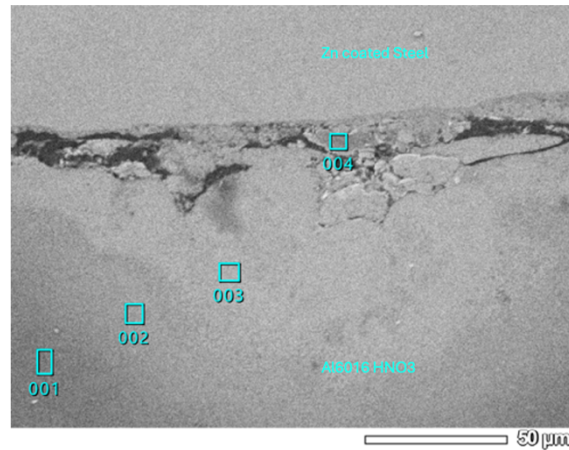


Figure 97. SEM micrograph showing the locations of EDS point analyses 001 to 004

Point EDS analyses performed at different locations across the joint demonstrated a gradual compositional transition from the aluminium substrate toward the steel substrate through a Zn-rich intermediate layer. Point 001, located within the aluminium side, consisted predominantly of Al (~93 wt%) with only minor Zn and oxygen contributions, confirming that this region corresponded to the Al6016 base material. Moving toward the interface, point 002 showed strong Al–Zn interaction, containing approximately 58 wt% Al and 39 wt% Zn, indicating significant diffusion and mixing near the reaction zone.

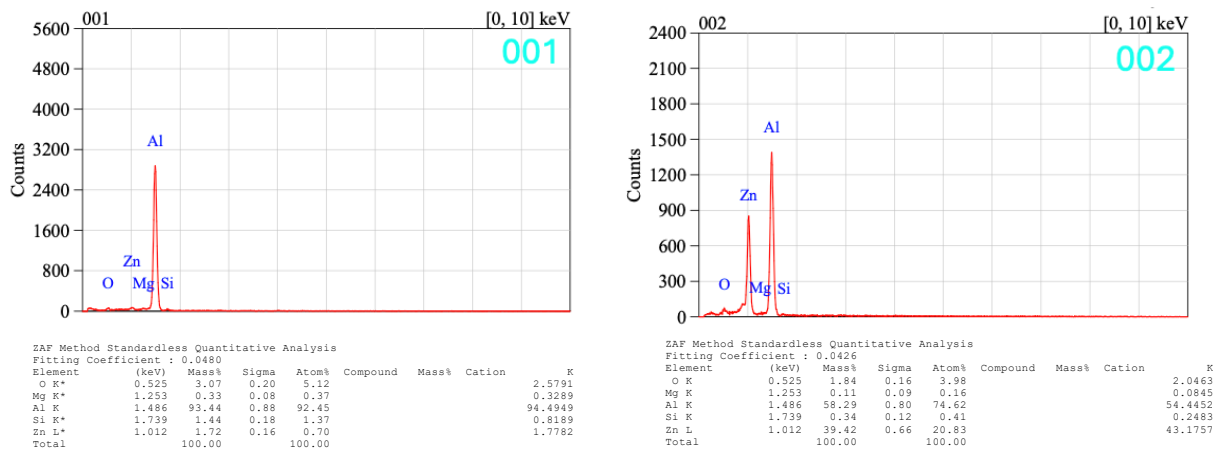


Figure 98. EDS spectra corresponding to point analyses 001 to 002

The interfacial regions represented by points 003, 004, and 005 were characterised by high Zn concentrations ranging from approximately 67–69 wt%, together with varying amounts of Al, oxygen, and minor Fe contributions. These compositions confirm the formation of a Zn-dominated interlayer generated by redistribution of the Zn coating during heating. The presence of Fe within some interfacial points suggests limited diffusion from the steel substrate into the reaction layer and the development of localised Al–Zn–Fe interaction regions.

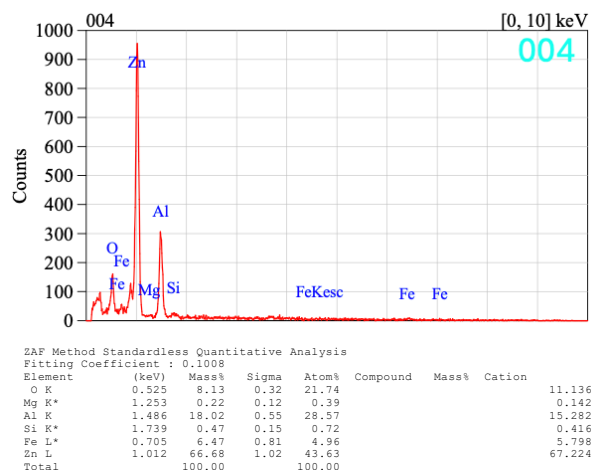
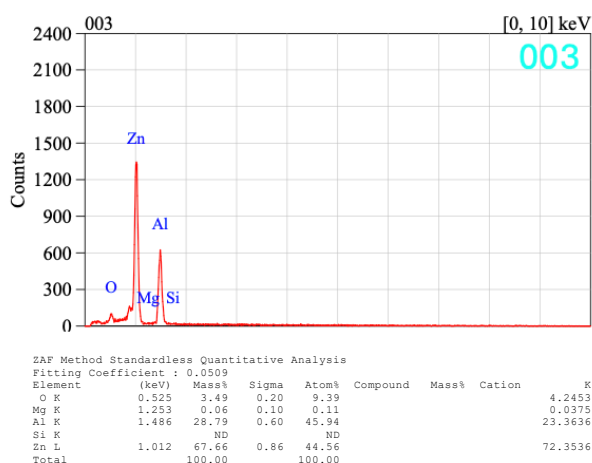


Figure 99. EDS spectra corresponding to point analyses 003 to 004

Closer to the steel side, point 007 exhibited a mixed composition containing Fe (~50 wt%), Zn (~27 wt%), and Al (~16 wt%), indicating the formation of a transition diffusion zone adjacent to the steel substrate. Finally, point 008 contained almost pure Fe (~99 wt%) with only trace amounts of Zn and Al, confirming that this region corresponded to the steel base material.

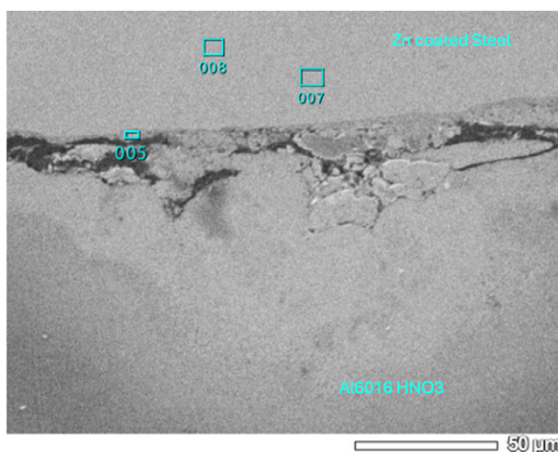
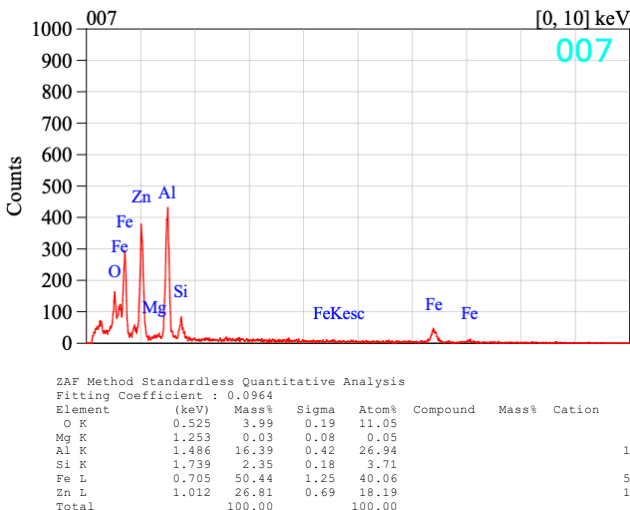
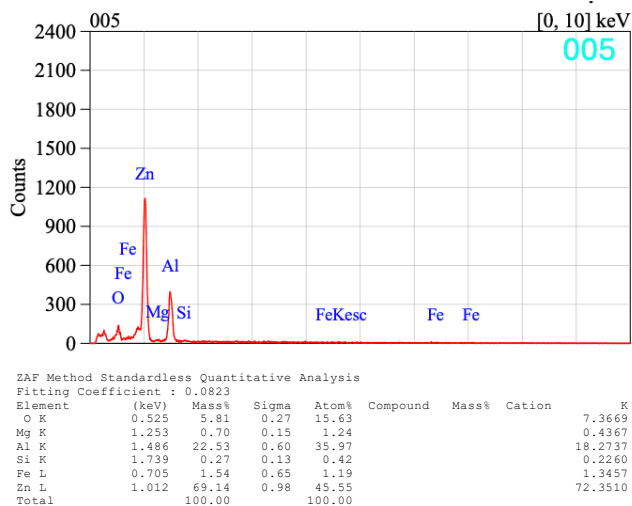


Figure 100. SEM micrograph showing the locations of EDS point analyses 005 to 008



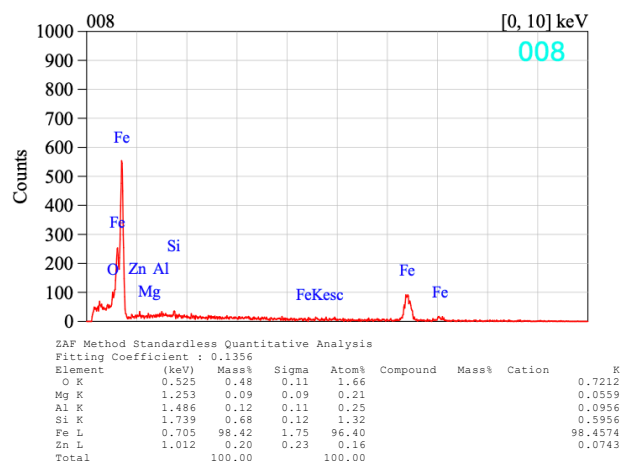


Figure 101. EDS spectra corresponding to point analyses 005 to 008

Additional SEM–EDS point analyses performed at locations 009–012 further confirmed the compositional gradient across the interface. Point 009, positioned within the aluminium substrate, consisted predominantly of Al (~95 wt%) with only minor oxygen and trace Zn/Fe contributions, indicating that the aluminium base material remained largely unaffected by the brazing process except for slight surface oxidation.

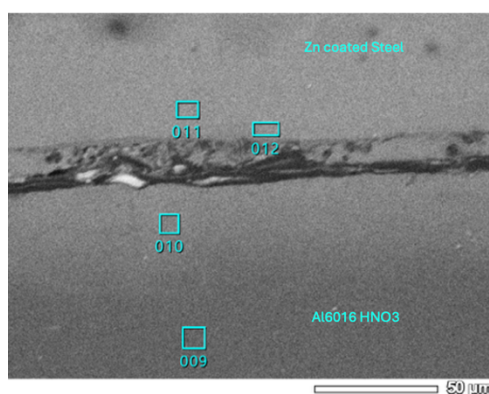


Figure 102. SEM micrograph showing the locations of EDS point analyses 009 to 012

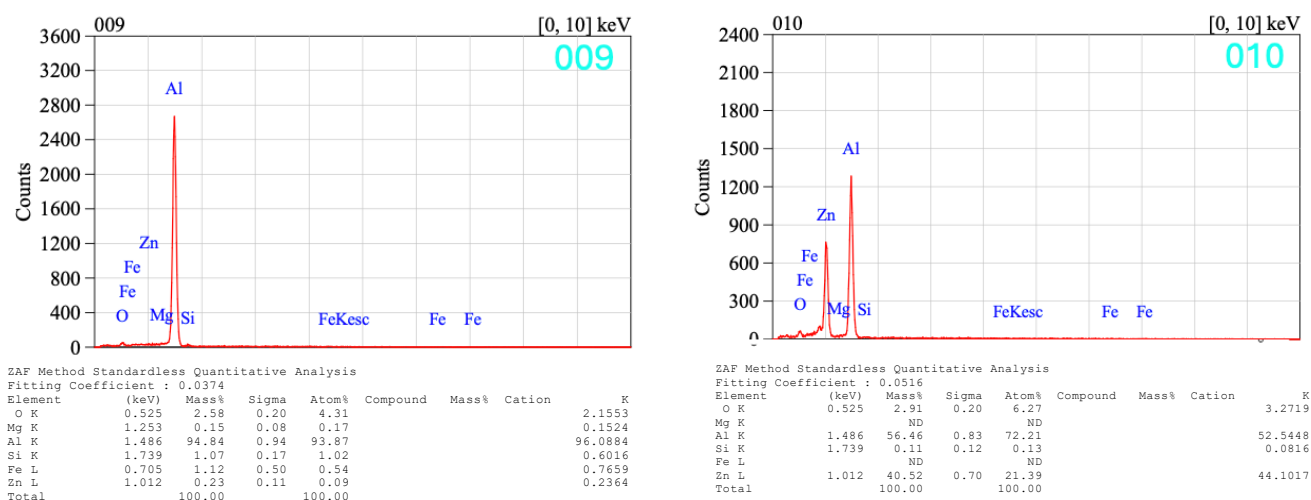


Figure 103. EDS spectra corresponding to point analyses 009 to 010

Closer to the interface, point 010 revealed significant Al–Zn interaction, containing approximately 56 wt% Al and 40 wt% Zn. This composition indicates the formation of an Al–Zn interaction layer caused by diffusion and redistribution of the Zn coating during heating. The relatively high Zn concentration suggests that the interfacial region was strongly influenced by the molten or partially molten Zn-rich phase during brazing.

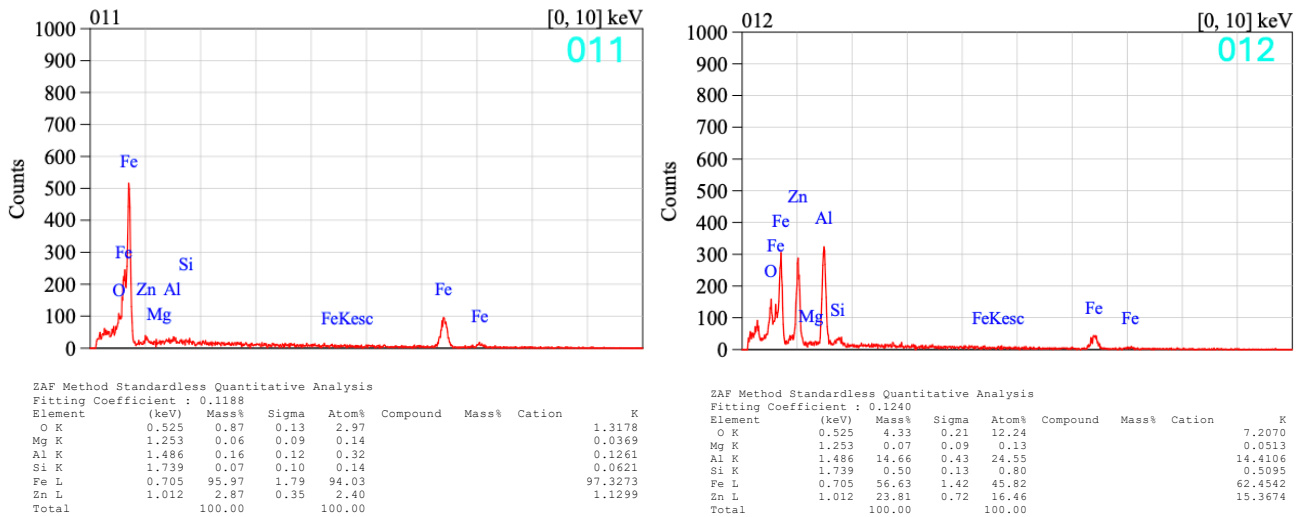
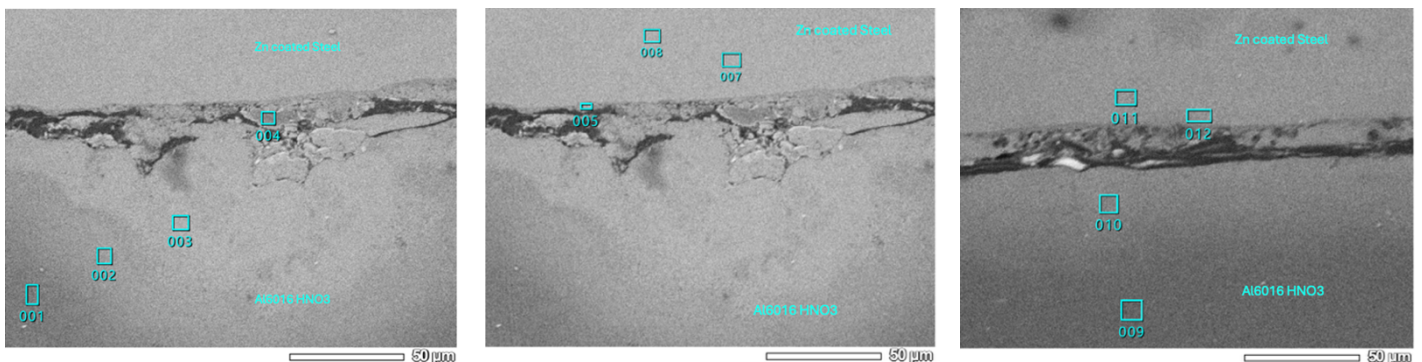


Figure 104. EDS spectra corresponding to point analyses 011 to 012

Point 011, located within the steel region, was composed mainly of Fe (~96 wt%) with only minor Zn traces, confirming that this area corresponded primarily to the steel substrate and that Zn diffusion into the steel remained limited. In contrast, point 012, situated near the steel side of the interface, exhibited a mixed composition containing Fe (~56 wt%), Zn (~24 wt%), Al (~15 wt%), and oxygen (~4 wt%). This composition indicates the presence of a localised Fe–Zn–Al diffusion zone formed through interfacial chemical interaction between the aluminium alloy, Zn coating, and steel substrate.

Overall, the SEM–EDS results demonstrate that the brazed joint contained a heterogeneous reaction region characterised by gradual elemental transition from aluminium to steel through Zn-rich intermediate layers. Although diffusion bonding between the materials occurred, the irregular morphology and localised compositional variations suggest that the interfacial reaction remained non-uniform throughout the joint region.



Point	Location	Composition (wt%)	Interpretation	Segment direction	Distance between segments (μm)
001	Al side	Al (~93), Zn (~1.7), O (~3)	Pure aluminium with slight Zn → Al base material	From 001 to 002	17
002	Near interface (Al side)	Al (~58), Zn (~39), O (~1.8)	Strong Al–Zn mixing → Al–Zn interaction layer	From 002 to 003	14
003	Interface	Zn (~67), Al (~29), O (~3.5)	Zn-rich with Al → Zn-dominated interlayer	From 003 to 004	46
004	Interface	Zn (~66.7), Al (~18.0), O (~8.1), Fe (~2.9)	Zn-rich with Fe traces → reaction layer (Al–Zn–Fe)	From 001 to 004	77
005	Interface	Zn (~69), Al (~23), O (~5.8), Fe (~1.5)	Zn-rich with minor Fe → transition layer	From 005 to 007	19
007	Near steel	Fe (~50), Zn (~27), Al (~16), O (~4)	Mixed Fe–Zn–Al → diffusion zone near steel	From 007 to 008	8
008	Steel	Fe (~99), traces of Zn/Al	Pure Fe → steel substrate	From 005 to 008	27
009	Al side	Al (~95), O (~2.6), minor Zn/Fe	Mostly Al with slight oxidation → Al base material (slightly affected)	From 009 to 010	48
010	Near interface (Al side)	Al (~56), Zn (~40), O (~2.9)	Strong Al–Zn mixing → Al–Zn interaction layer	From 010 to 011	47
011	Steel	Fe (~96), Zn (~2.9), traces	Predominantly Fe → steel substrate (slight Zn diffusion)	From 011 to 012	7
012	Near steel	Fe (~56), Zn (~24), Al (~15), O (~4)	Mixed Fe–Zn–Al → diffusion/reaction zone near steel	From 009 to 012	88

Table 19. Summary of EDS point analysis (001-012) corresponding to SEM micrograph shown above (The reference point taken is the highest point on y axis on the SEM image)

4.4.3 Backscattered electron imaging: HNO₃-treated sample

The BSE micrographs revealed a continuous but highly irregular interfacial layer located between the aluminium alloy and the Zn-coated steel substrate. Significant contrast variation was observed along the joint line, indicating the presence of chemically heterogeneous regions formed during brazing.

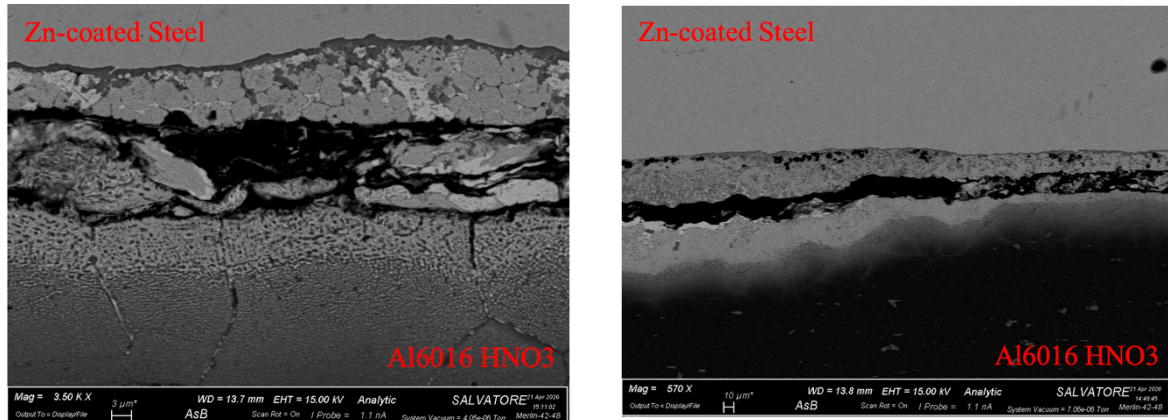


Figure 105. BSE micrograph showing heterogeneous interfacial morphology and localised porosity

Several areas of the interface exhibited porous morphology, localised voids, and discontinuous regions. In some locations, the interfacial layer appeared relatively thin and compact, while in other regions it became thicker and more irregular with globular features and partial separation. These observations suggest non-uniform redistribution of the Zn-rich interlayer during heating and indicate that the reaction zone developed through localised diffusion and partial liquid-phase interaction.

Electron Image 1

Point analyses performed on the BSE image further clarified the elemental distribution across the joint. Spectrum S22, located within the upper region corresponding to the steel substrate, contained nearly pure Fe (~100 wt%), confirming that this area remained largely unaffected by interdiffusion. Spectrum S23, positioned directly at the interface, showed a mixed composition of Zn (~52 wt%), Al (~36 wt%), and Fe (~12 wt%), indicating the formation of a multi-element diffusion zone between the steel and aluminium substrates.

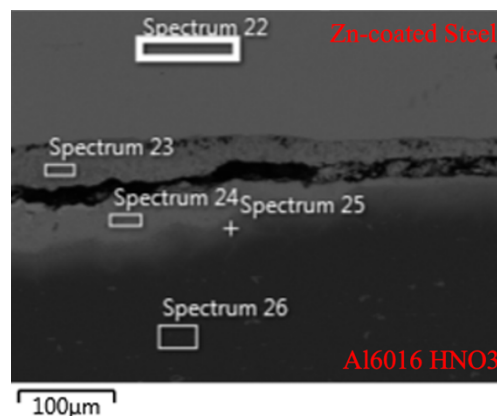
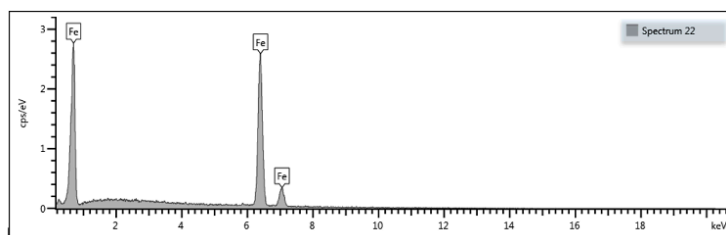
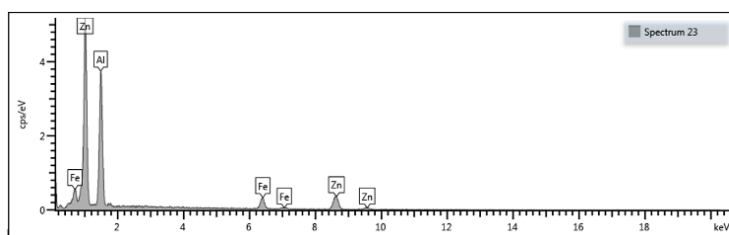


Figure 106. Backscattered electron (BSE) electron image 1 with spectrum from 22 to 26



Element	Wt%	Wt% Sigma
Fe	100.00	0.00
Total:	100.00	

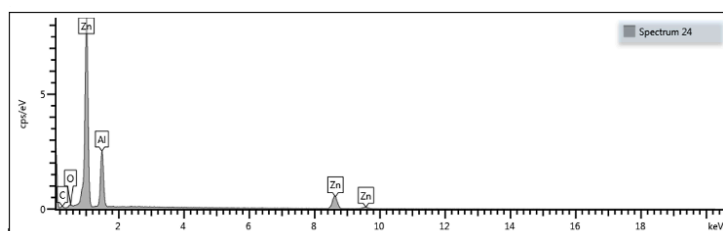
Figure 107. EDS spectrum and elemental composition of spectrum 22



Element	Wt%	Wt% Sigma
Al	36.36	0.68
Fe	11.77	0.47
Zn	51.87	0.86
Total:	100.00	

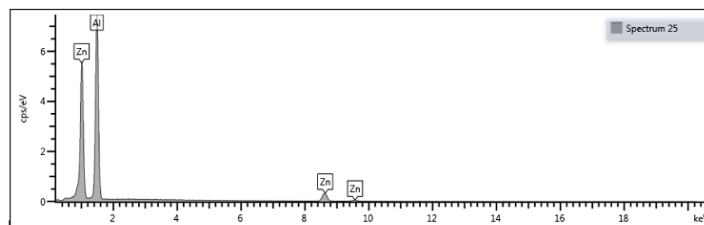
Figure 108. EDS spectrum and elemental composition of spectrum 23

Spectrum S24, located within the intermediate layer, was dominated by Zn (~69 wt%) together with Al (~24 wt%) and minor oxygen and carbon contributions. This composition confirms the existence of a Zn-rich interlayer and suggests localised oxidation or contamination at the interface. Spectrum S25 exhibited approximately 57 wt% Al and 43 wt% Zn, corresponding to an Al–Zn mixed phase formed through interfacial diffusion and chemical interaction during brazing. Finally, Spectrum S26 consisted almost entirely of Al (~100 wt%), confirming that the lower region corresponded to the aluminium base material.



Element	Wt%	Wt% Sigma
C	6.02	0.66
O	1.64	0.18
Al	23.56	0.35
Zn	68.78	0.62
Total:	100.00	

Figure 109. EDS spectrum and elemental composition of spectrum 24



Element	Wt%	Wt% Sigma
Al	56.68	0.53
Zn	43.32	0.53
Total:	100.00	

Figure 110. EDS spectrum and elemental composition of spectrum 25

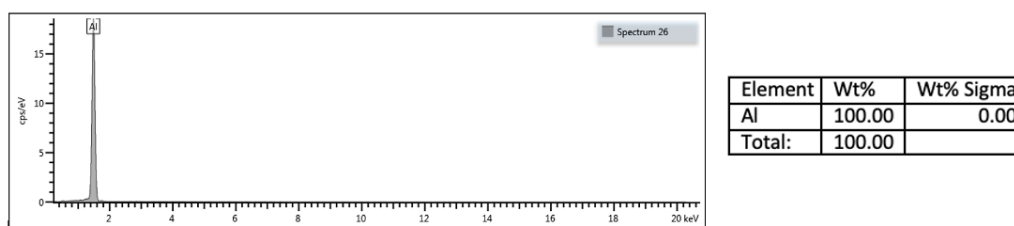


Figure 111. EDS spectrum and elemental composition of spectrum 26

Overall, the combined BSE and EDS analyses demonstrate that the joint formation at 480 °C occurred through the development of a heterogeneous Zn-rich interfacial layer separating the aluminium alloy from the steel substrate. Although elemental diffusion and metallurgical interaction were achieved, the presence of porosity, interfacial discontinuities, and irregular morphology indicates that the bonding quality remained locally non-uniform throughout the brazed region.

Additional BSE–EDS analyses performed on Electron Image 2 further confirmed the heterogeneous nature of the interfacial region in the HNO₃-treated sample brazed at 480 °C. The BSE micrographs revealed a relatively continuous but morphologically irregular reaction layer located between the aluminium alloy and the Zn-coated steel substrate. Variations in contrast and local interfacial thickness indicate compositional inhomogeneity and non-uniform diffusion behaviour across the brazed region.

Electron Image 2

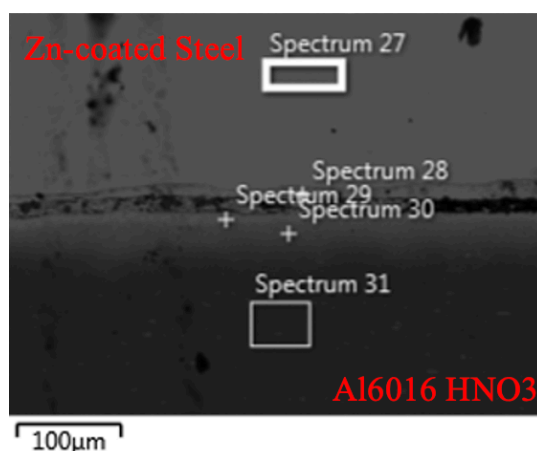
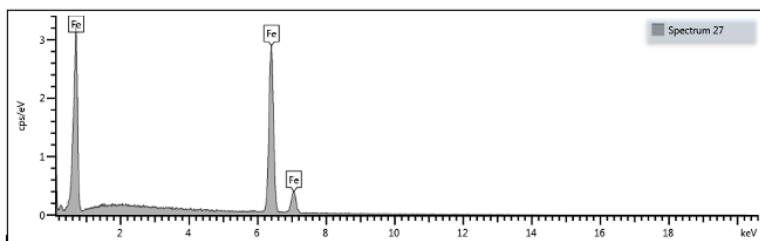


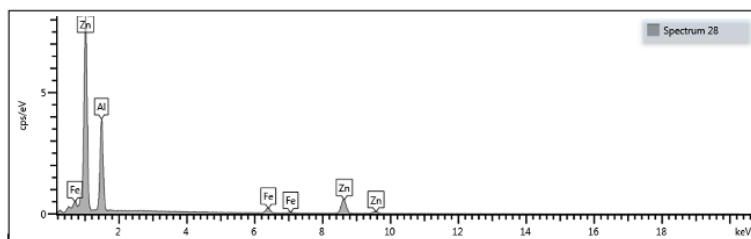
Figure 112. Backscattered electron (BSE) electron image 2 with spectrum from 27 to 31

Spectrum S27, positioned in the upper region corresponding to the Zn-coated steel substrate, consisted entirely of Fe (~100 wt%), confirming that this area remained unaffected by significant Al or Zn diffusion. In contrast, spectra S28 and S29, obtained directly from the interfacial region, showed high Zn concentrations of approximately 63–66 wt% together with Al contents of approximately 31–33 wt%. These compositions indicate the formation of a Zn-rich diffusion layer with partial Al incorporation during brazing.



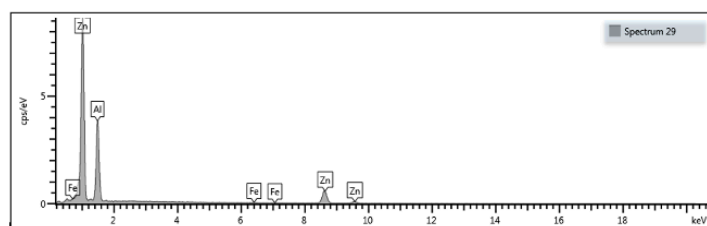
Element	Wt%	Wt% Sigma
Fe	100.00	0.00
Total:	100.00	

Figure 113. EDS spectrum and elemental composition of spectrum 27



Element	Wt%	Wt% Sigma
Al	30.95	0.37
Fe	6.15	0.22
Zn	62.90	0.44
Total:	100.00	

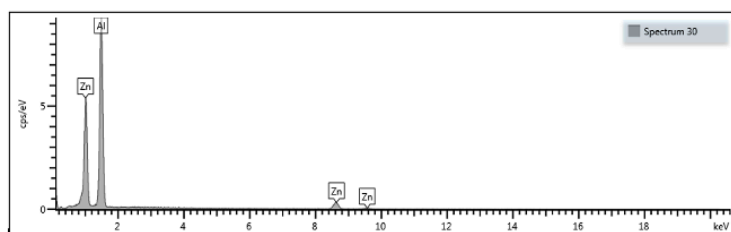
Figure 114. EDS spectrum and elemental composition of spectrum 28



Element	Wt%	Wt% Sigma
Al	33.23	0.47
Fe	0.53	0.17
Zn	66.24	0.48
Total:	100.00	

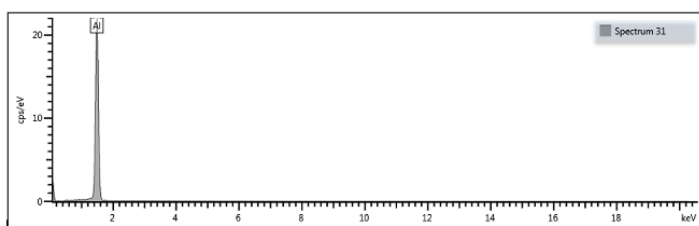
Figure 115. EDS spectrum and elemental composition of spectrum 29

Spectrum S30, located within the transition zone, contained approximately 61 wt% Al and 39 wt% Zn, corresponding to the formation of an Al–Zn mixed phase. This result confirms that substantial interfacial diffusion and chemical interaction occurred between the aluminium alloy and the Zn interlayer during heating. Spectrum S31, taken from the lower region of the sample, consisted almost entirely of Al (~100 wt%), confirming that this region corresponded to the Al6016 base material.



Element	Wt%	Wt% Sigma
Al	61.43	0.78
Zn	38.57	0.78
Total:	100.00	

Figure 116. EDS spectrum and elemental composition of spectrum 30



Element	Wt%	Wt% Sigma
Al	100.00	0.00
Total:	100.00	

Figure 117. EDS spectrum and elemental composition of spectrum 31

Electron Image 3

A localised region analysed at Spectrum S32 exhibited a mixed composition containing Al (~53 wt%), Fe (~27 wt%), Zn (~13 wt%), and Si. This composition suggests the presence of a reaction product formed during interfacial interaction between the aluminium side, zinc brazing material, and limited iron contribution from the steel side. Therefore, S32 should be interpreted as a localised reaction phase within the heterogeneous joining zone, rather than as a defect. The presence of Fe in this region indicates that limited Fe diffusion or local mixing occurred during the development of the Al–Zn–Fe reaction zone.

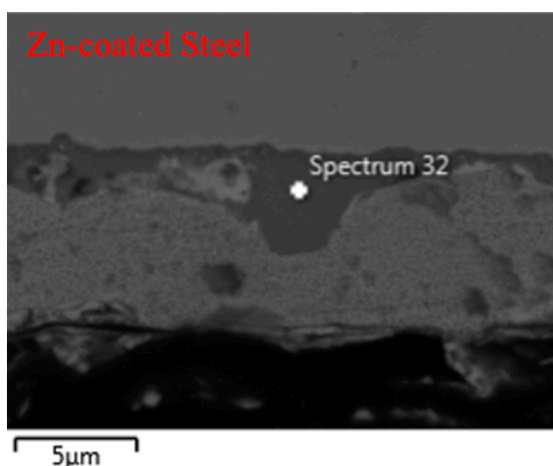
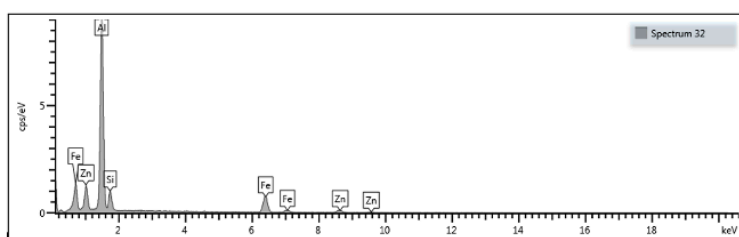


Figure 118. Backscattered electron (BSE) electron image 3 with spectrum 32



Element	Wt%	Wt% Sigma
Al	53.01	0.60
Si	7.24	0.23
Fe	26.64	0.51
Zn	13.11	0.76
Total:	100.00	

Figure 119. EDS spectrum and elemental composition of spectrum 32

Overall, the BSE–EDS analyses of Electron Image 3 demonstrate that the brazed joint was formed through the development of a heterogeneous Al–Zn interfacial layer with localised Fe participation. Although metallurgical interaction between the aluminium alloy and Zn-coated steel was achieved, the observed interfacial irregularities and local compositional inhomogeneities indicate that the bonding process remained non-uniform and susceptible to defect formation.

4.4.4 Optical microscopy: KOH-treated sample

Optical microscopy observations of the KOH-treated Al6016/Zn-coated steel revealed the presence of a highly non-uniform and discontinuous interfacial region between the aluminium alloy and the Zn-coated steel substrate. A dark interfacial line with irregular morphology was observed along the bonding region, indicating the formation of an intermediate reaction layer rather than direct Al–Fe contact. The interface exhibited significant local variations in thickness and continuity, suggesting unstable spreading and redistribution of the molten Zn-rich phase during heating.

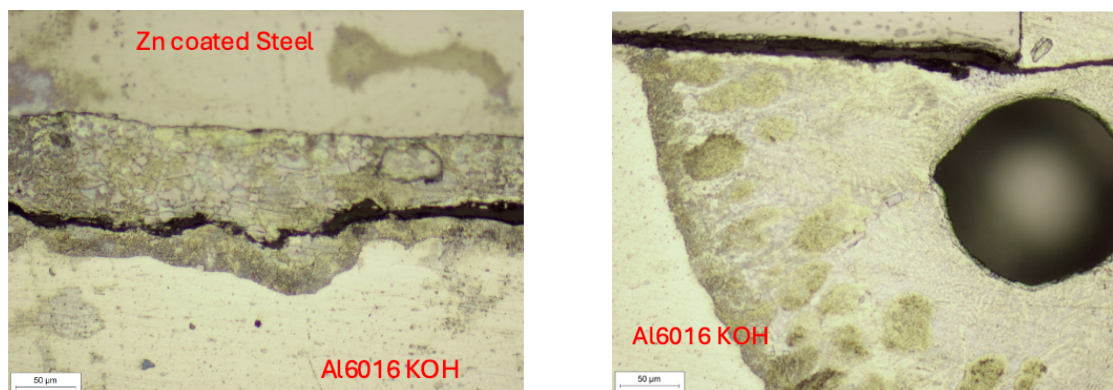


Figure 120. Optical micrograph (200×) showing a discontinuous interfacial reaction layer with localised cracking, interfacial separation and large pore formation

Several regions showed visible cracks, gaps, and interfacial separations distributed along the joint line. These discontinuities indicate incomplete metallurgical bonding and limited effective contact between the substrates. In addition, the aluminium side contained large pores and rounded voids, which are likely associated with localised gas evolution, trapped reaction products, or instability of the liquid phase during brazing. The presence of these defects significantly reduced the continuity of the joint and contributed to local weakening of the interfacial region.

The observed morphology likely suggests that the joining process was governed by the formation of a Zn-rich interfacial layer with highly irregular spreading behaviour. In some regions, the reaction layer appeared relatively thin and continuous, whereas in other areas severe thickening, localised accumulation of material, and crack formation were observed. Such heterogeneous morphology indicates that the brazing conditions did not promote uniform interfacial interaction across the entire bonded region.

4.4.5 SEM-EDS: KOH-treated sample

The SEM micrographs showed a discontinuous reaction layer with local variations in thickness and morphology, confirming the heterogeneous nature of the bonding region observed previously by optical microscopy.

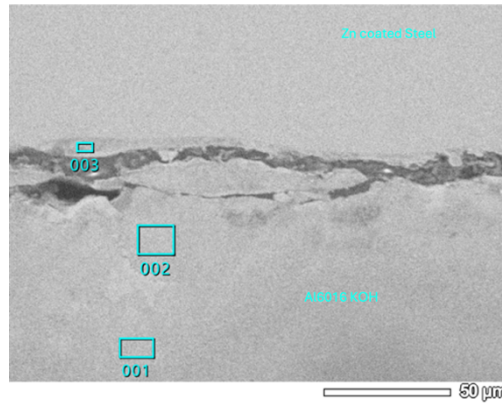


Figure 121. SEM micrograph showing the locations of EDS point analyses 001 to 003

Point analysis performed at different locations across the interface demonstrated that the reaction zone was dominated by Zn with varying amounts of Al and minor oxygen content. Spectrum 001, located within the interfacial region, contained approximately 68.5 wt% Zn and 28 wt% Al, indicating the formation of an Al–Zn interlayer produced by interdiffusion between the molten Zn-rich phase and the aluminium substrate. Spectrum 002 exhibited an even higher Zn concentration of approximately 73 wt% with about 23 wt% Al, suggesting the presence of a relatively stable Zn-rich interfacial phase.

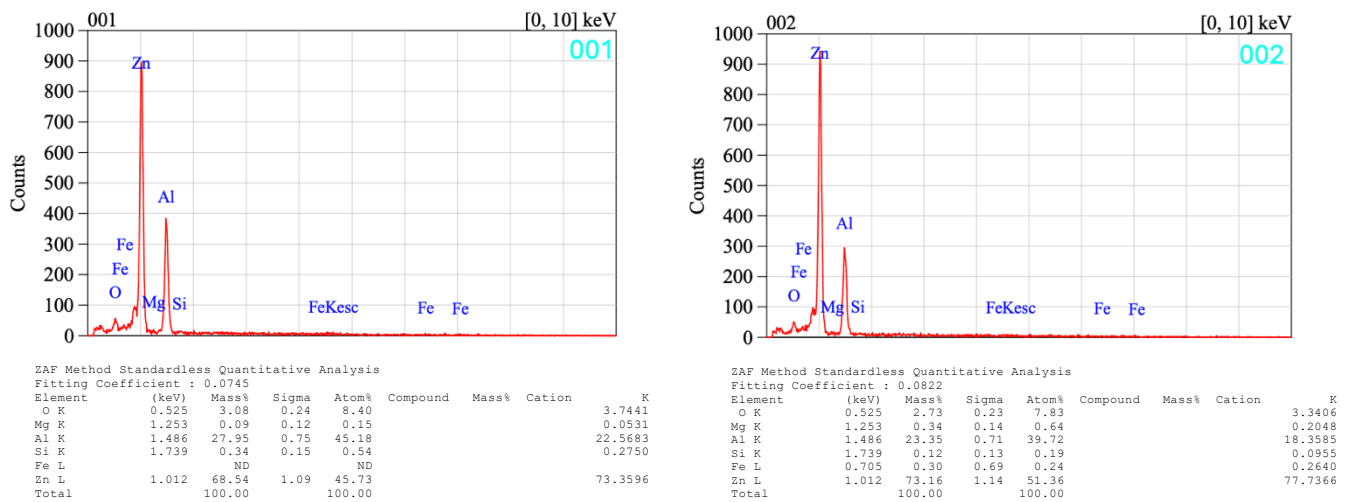


Figure 122. EDS spectra corresponding to point analyses 001 to 002

At spectrum 003, the composition reached approximately 92.6 wt% Zn with only minor Al contribution, corresponding to a nearly pure Zn-rich brazing region. This result indicates localised accumulation and redistribution of Zn during heating. In contrast, spectrum 004, positioned within the steel substrate, consisted predominantly of Fe (~97 wt%) with only traces of Zn and Al, confirming that diffusion into the steel side remained limited under the applied brazing conditions.

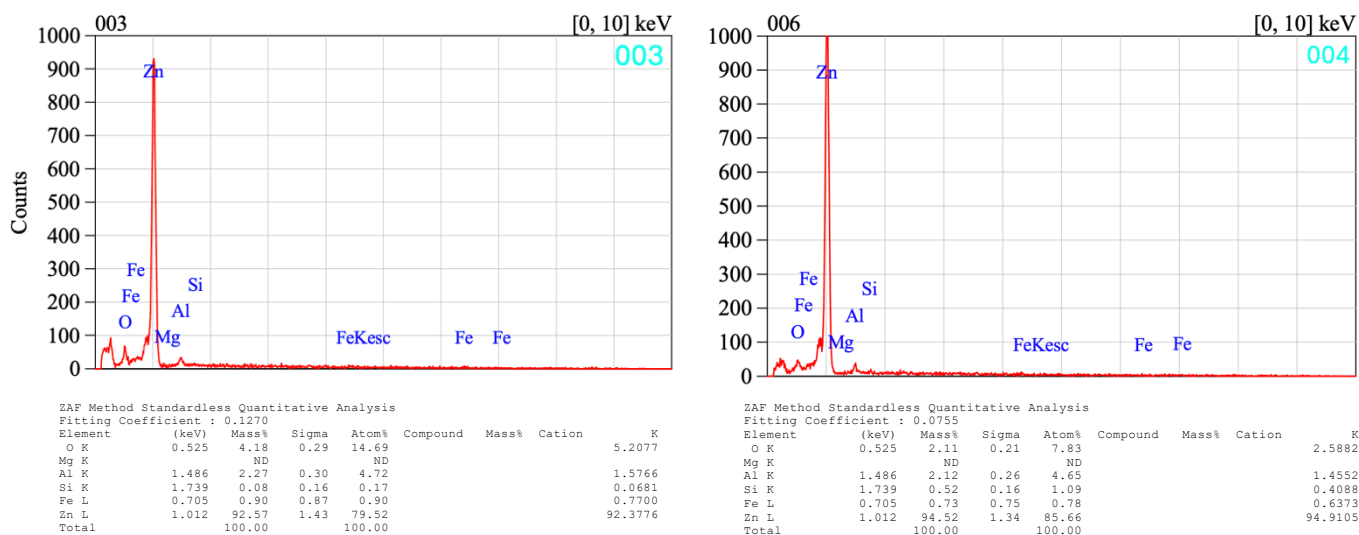


Figure 123. EDS spectra corresponding to point analyses 003 to 004

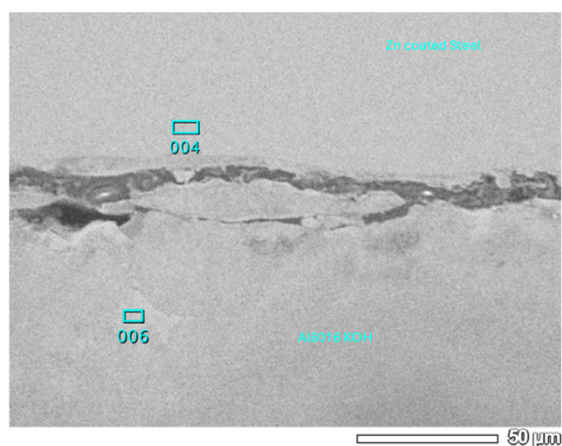


Figure 124. SEM micrograph showing the locations of EDS point analyses 004 and 006

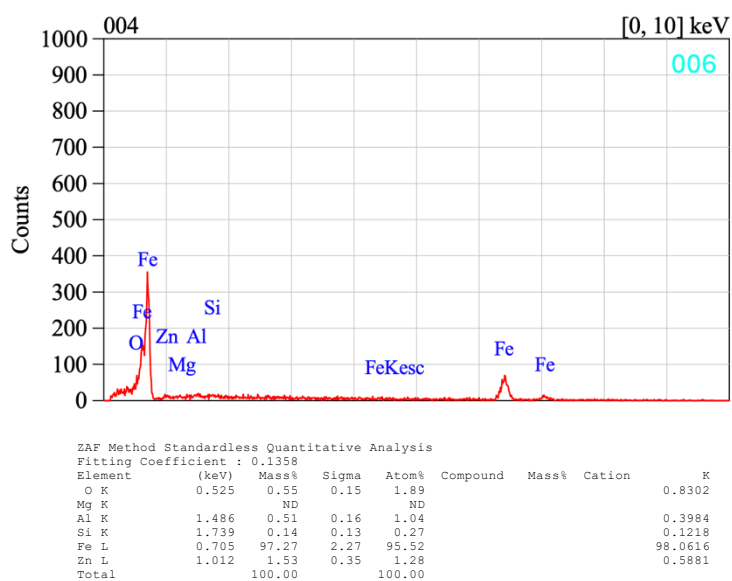
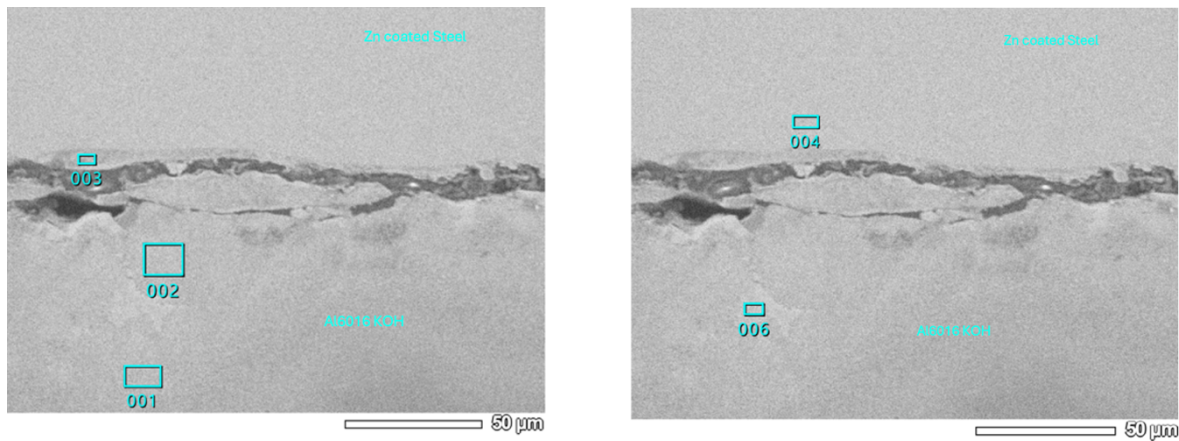


Figure 125. EDS spectra corresponding to point analyses 006

Additional analysis at spectrum 006 also revealed an extremely high Zn concentration (~94.5 wt%), supporting the presence of locally concentrated Zn-rich regions along the interface. The coexistence of nearly pure Zn regions together with mixed Al–Zn phases demonstrates that the reaction layer developed non-uniformly across the joint. Such compositional heterogeneity is consistent with unstable spreading of the molten interlayer and incomplete metallurgical homogenisation during brazing.

Overall, the SEM–EDS investigation confirmed that bonding in the KOH-treated sample occurred mainly through the formation of a Zn-dominated interfacial layer with localised Al participation. However, the strong variation in local composition together with the discontinuous morphology of the interface indicates limited process stability and incomplete bonding quality across the joint region.



Point	Location	Composition (wt%)	Interpretation	Segment direction	Distance between segments (µm)
001	Interface	Zn (~68.5), Al (~28.0), O (~3.1)	Zn-dominated phase with Al → Al–Zn interlayer	From 001 to 002	43
002	Interface	Zn (~73.2), Al (~23.4), O (~2.7)	Strong Zn-rich composition → stable Zn interlayer	From 002 to 003	37
003	Interface	Zn (~92.6), Al (~2.3), O (~4.2)	Nearly pure Zn → Zn-rich layer (brazing phase)	From 001 to 003	80
004	Steel	Fe (~97.3), Zn (~1.5), traces Al/O	Predominantly Fe → steel substrate	From 004 to 006	69
006	Interface	Zn (~94.5), Al (~2.1), O (~2.1)	Very high Zn → pure Zn region		

Table 20. Summary of EDS point analysis (001-006) corresponding to SEM micrograph shown above (The reference point taken is the highest point on y axis on the SEM image)

4.4.6 Summary of Trial 3

Trial 3, performed at a brazing temperature of 480 °C under repeatability conditions, demonstrated that bonding between Al6016 aluminium alloy and Zn-coated steel occurred mainly through the formation of a heterogeneous Zn-rich interfacial layer. Both optical microscopy and SEM–EDS analyses confirmed the presence of an intermediate reaction zone separating the aluminium alloy from the steel substrate, indicating that the joining process was governed by interfacial diffusion and redistribution of the Zn coating during heating.

Optical microscopy observations of both HNO₃-treated and KOH-treated samples revealed a discontinuous and morphologically irregular interface characterised by local variations in thickness, globular features, cracks, pores, and interfacial gaps. These defects indicate unstable spreading of the molten Zn-rich phase and incomplete metallurgical bonding across the joint region. Large pores and voids observed particularly on the aluminium side suggest localised gas evolution and limited continuity of the bonded interface during brazing.

SEM–EDS analysis further demonstrated that the interfacial region was dominated by Zn-rich phases with varying amounts of Al and limited Fe participation. Several analysed locations exhibited compositions corresponding to mixed Al–Zn interlayers, while other regions consisted almost entirely of Zn, indicating localised accumulation and redistribution of the molten Zn phase. Only minor diffusion into the steel substrate was detected, confirming that the interaction remained concentrated mainly within the interfacial region.

The coexistence of Zn-rich regions, mixed Al–Zn phases, and locally heterogeneous reaction products indicates that the interfacial reaction layer developed non-uniformly along the joint. In addition, the presence of compositional inhomogeneities together with porosity and interfacial cracking suggests that the applied brazing conditions did not provide stable wetting and uniform metallurgical interaction across the entire bonded area.

Overall, Trial 3 confirmed that metallurgical bonding between Al6016 and Zn-coated steel can be achieved through the development of a Zn-mediated interfacial layer. However, the significant interfacial discontinuities, porosity, and local reaction heterogeneity demonstrate that the joint quality remained limited under the brazing conditions at 480 °C.

4.5 Cross-trial comparison

4.5.1 Comparison of HNO₃-treated samples

Comparison of the HNO₃-treated samples from Trials 1–3 revealed clear differences in interfacial morphology, diffusion behaviour, and bonding quality between the Al6016 alloy and the Zn-coated steel substrate. Optical microscopy, SEM, BSE imaging, and SEM–EDS analyses showed that the brazing conditions strongly influenced the redistribution of the Zn coating, the formation of Al–Zn reaction products, and the continuity of the interfacial diffusion layer.

In Trial 1, the interfacial region remained relatively thin, discontinuous, and morphologically irregular. Optical microscopy revealed localised dark interfacial regions with partial separation and limited continuity along the joint line. SEM and BSE observations confirmed the presence of a narrow Zn-rich reaction layer with several pores, crack-like defects, and locally separated regions. SEM–EDS point analyses identified mainly Zn-rich interlayers containing moderate Al concentrations and only limited Fe participation. For example, intermediate regions contained approximately 51–67 wt% Zn together with 26–30 wt% Al, while Fe remained very low. These results indicate that only limited redistribution of the molten Zn phase occurred during brazing, resulting in weak Al–Zn interaction and restricted diffusion across the interface. Local carbon-rich areas observed in some analyses were attributed mainly to contamination or mounting residues rather than to actual interfacial phases.

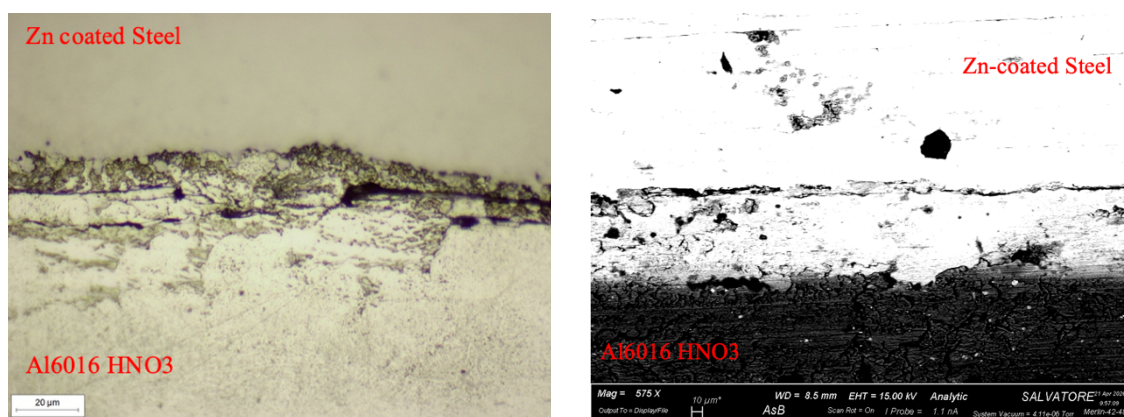


Figure 126. Optical micrograph and BSE image of the HNO₃-treated Trial 1 sample showing a thin and discontinuous Zn-rich interfacial layer with localised pores, crack-like defects and limited interfacial continuity

Trial 2 exhibited the most developed and chemically interactive interfacial structure among the HNO₃-treated samples. Optical microscopy revealed a broader and relatively more continuous interfacial layer extending along larger portions of the joint region. Compared with Trial 1, the reaction layer appeared thicker and showed improved spreading of the Zn-rich liquid phase. BSE imaging demonstrated a more extended diffusion region with fewer sharp discontinuities and a more homogeneous gray-scale contrast distribution. SEM–EDS analyses confirmed significantly stronger elemental mixing between Al and Zn together with increased Fe participation within the diffusion zone. Several analysed regions contained mixed compositions such as Al (~40–50 wt%), Zn (~38–47 wt%), and Fe up to ~15 wt%,

confirming the formation of multi-component Al–Zn–Fe interaction zones. Additional regions consisting almost entirely of Zn indicated local redistribution and accumulation of the molten Zn coating during heating. Although localised pores and void-like defects were still observed, the interfacial layer in Trial 2 showed the highest continuity, the strongest elemental interaction, and the most developed diffusion behaviour among all investigated HNO₃-treated conditions.

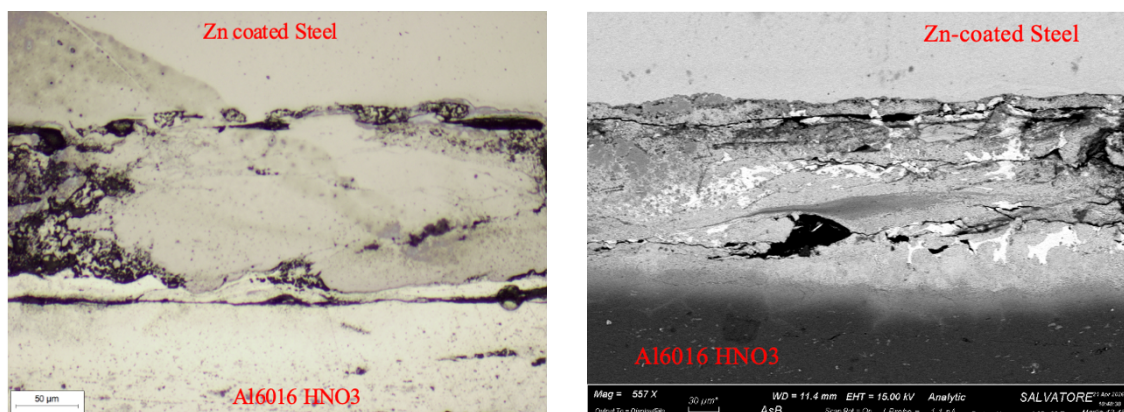


Figure 127. Optical micrograph and BSE image of the HNO₃-treated Trial 2 sample showing a relatively thick and continuous interfacial diffusion layer with enhanced Al–Zn–Fe interaction, improved interfacial continuity and localised pore formation

Trial 3 demonstrated intermediate behaviour between Trials 1 and 2. Optical microscopy showed the presence of a Zn-mediated interfacial layer; however, the interface remained highly heterogeneous with significant local thickness variations and discontinuous regions. Several areas exhibited globular interfacial features, pores, and local cracking, indicating unstable spreading and redistribution of the liquid Zn phase. SEM–EDS analyses identified both mixed Al–Zn regions and nearly pure Zn-rich areas distributed along the interface. In some locations, Zn concentrations exceeded ~90 wt%, while other regions showed partial Al–Zn mixing with only limited Fe diffusion. Compared with Trial 1, Trial 3 exhibited improved interfacial interaction and greater development of the reaction layer, but the diffusion zone remained less continuous and less chemically homogeneous than in Trial 2.

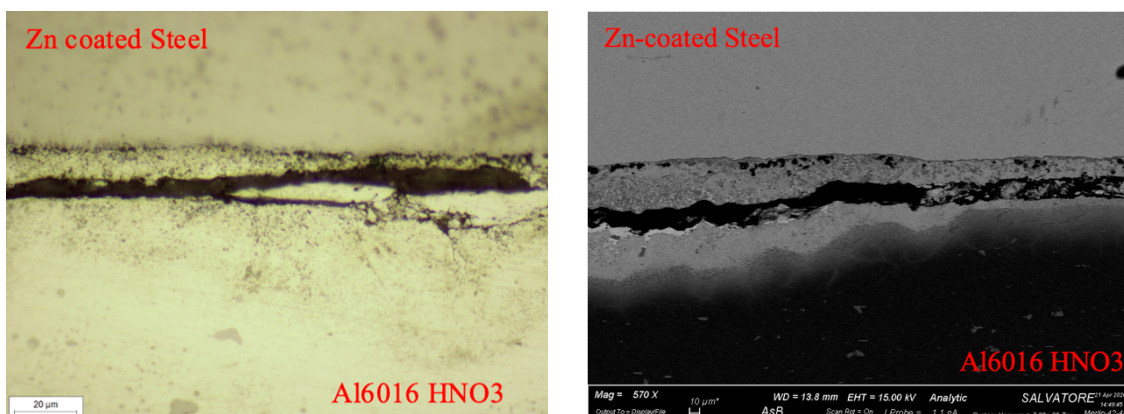


Figure 128. Optical micrograph and BSE image of the HNO₃-treated Trial 3 sample showing a heterogeneous Zn-mediated interfacial layer with partial Al–Zn mixing, localised pores, crack-like defects and discontinuous regions

Overall, the comparison demonstrated that the brazing behaviour of the HNO_3 -treated samples strongly depended on the stability and redistribution of the Zn-rich interlayer during heating. Trial 1 showed limited interfacial development and incomplete bonding, Trial 2 produced the most developed diffusion layer and strongest metallurgical interaction, while Trial 3 showed partial improvement but retained substantial morphological and chemical heterogeneity. None of the investigated conditions produced a completely uniform and defect-free interface; however, Trial 2 provided the most favourable balance between interfacial continuity, elemental mixing, and diffusion-layer development.

4.5.2 Comparison of KOH-treated samples

Comparison of the KOH-treated samples showed that this surface condition did not reliably promote effective bonding. In Trials 1 and 2, the optical micrographs revealed large interfacial gaps between the two substrates, indicating absence of continuous physical contact across most of the joint. Because of this separation, the observed regions cannot be interpreted as a continuous bonded interface.

In Trial 1, the interface was dominated by a large gap and only isolated local contact areas were present. SEM–EDS detected limited Zn-rich reaction products in some regions, but these were not continuous and did not form a stable bonding layer. Therefore, Trial 1 KOH showed poor interfacial contact and ineffective metallurgical bonding.

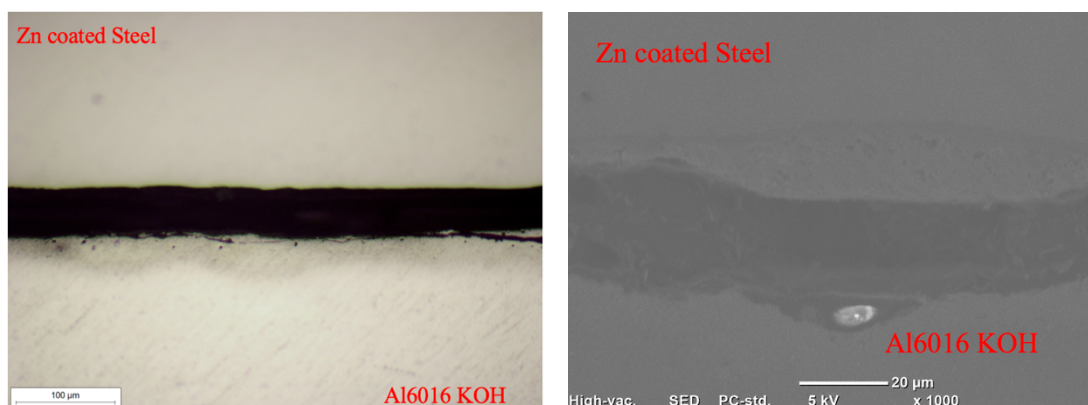


Figure 129. Optical microscopy and SEM image of the KOH-treated sample from Trial 1 showing a large interfacial gap and absence of continuous bonding between Al6016 aluminium alloy and Zn-coated steel.

Trial 2 showed a similar behaviour. A considerable gap was observed between the aluminium alloy and the Zn-coated steel, confirming that the substrates were not in proper contact during brazing. Although localised Zn-rich areas were detected, the lack of continuous contact prevented the formation of a uniform diffusion layer. As a result, Trial 2 KOH cannot be considered representative of a properly bonded joint.

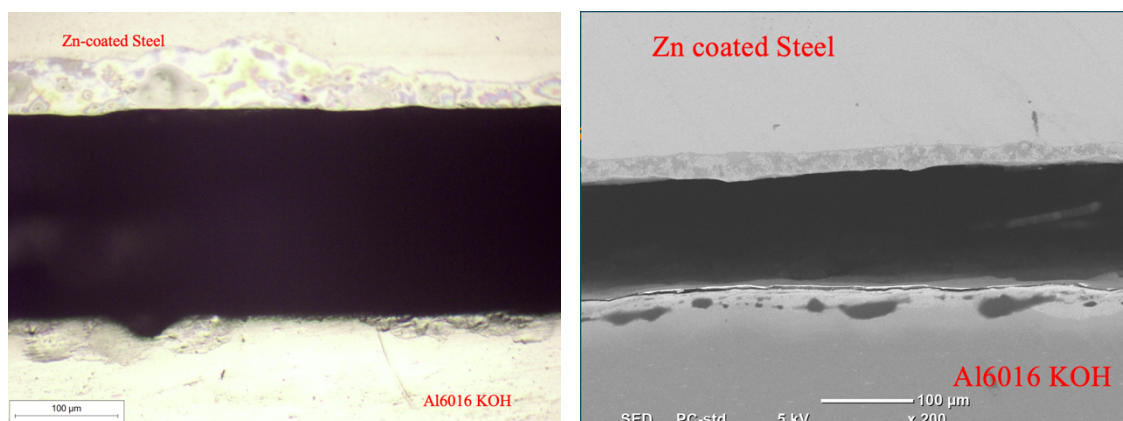


Figure 130. Optical microscopy and SEM image of the KOH-treated sample from Trial 2 showing a large interfacial gap and absence of effective metallurgical bonding

Trial 3 showed the best interfacial development among the KOH-treated samples, although the joint quality remained limited. Unlike Trials 1 and 2, where large interfacial gaps and lack of contact dominated the joint region, Trial 3 exhibited localised formation of a Zn-rich interfacial layer together with partial Al–Zn interaction. However, the interface was still discontinuous and heterogeneous, with visible pores, cracks, and local separations. Therefore, Trial 3 can be considered the most successful KOH-treated condition, but it did not produce a fully continuous or defect-free joint.

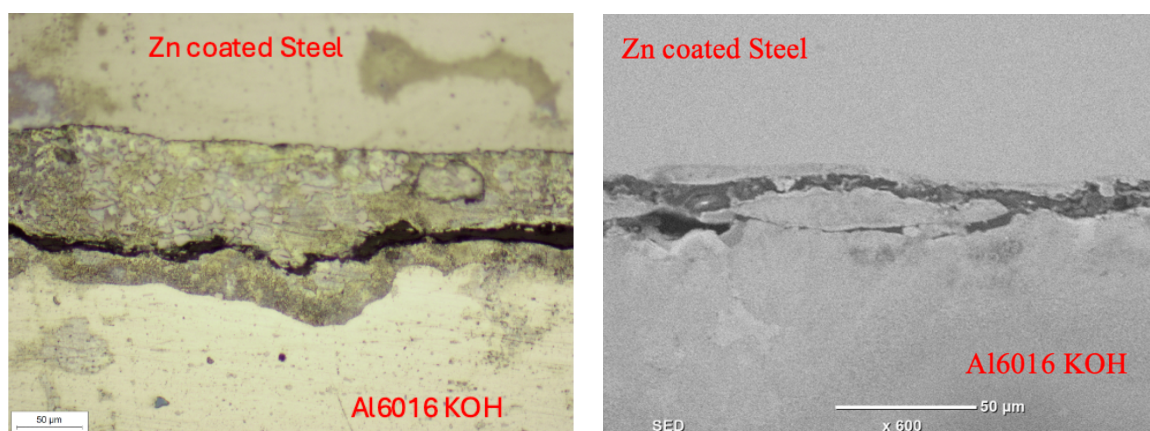


Figure 131. Optical microscopy and SEM image of the KOH-treated sample from Trial 3 showing partial formation of a Zn-rich interfacial layer together with discontinuous bonding regions, pores, cracks, and local interfacial gaps

Overall, the KOH-treated samples showed poor bonding performance compared with the HNO₃-treated samples. Trials 1 and 2 were mainly characterised by large gaps and absence of effective physical contact, which prevented the formation of a continuous diffusion layer. Trial 3 showed partial improvement due to localised Zn-rich layer formation and limited Al–Zn interaction, but the bonding remained non-uniform and defective. These results indicate that, under the investigated brazing conditions, KOH treatment did not provide stable interfacial contact or reliable Zn-mediated diffusion-layer formation.

4.5.3 Comparison of bonding quality across trials

The comparison of bonding quality across Trials 1-3 demonstrated a strong dependence of the brazed joint morphology on both the brazing conditions and the applied surface treatment. Significant differences were observed in the continuity of the interfacial layer, extent of elemental interaction, diffusion-layer development, and defect formation between the investigated conditions.

For the HNO₃-treated samples, Trial 1 showed limited metallurgical bonding characterised by a thin and discontinuous interfacial region with localised Zn-rich reaction products and restricted elemental diffusion. Trial 2 exhibited the highest bonding quality among all investigated conditions. Optical microscopy, SEM, BSE imaging, and SEM-EDS analysis confirmed the formation of the most developed interfacial diffusion region with improved continuity and stronger Al-Zn interaction together with localised Fe participation. Although some pores and local defects were still present, the interface in Trial 2 showed the most stable diffusion behaviour and the highest degree of interfacial development. Trial 3 demonstrated intermediate behaviour. A Zn-mediated reaction layer and partial Al-Zn interaction were observed; however, the interface remained heterogeneous and discontinuous with local pores, cracks, and irregular thickness variations, indicating unstable redistribution of the molten Zn-rich phase during brazing.

In contrast, the KOH-treated samples generally exhibited lower bonding quality and poorer interfacial continuity. Trials 1 and 2 were dominated by large interfacial gaps and lack of continuous contact between the substrates, preventing the formation of a stable diffusion layer across the joint region. Trial 3 showed partial improvement compared with the previous KOH-treated conditions through localised formation of Zn-rich interfacial regions and limited Al-Zn interaction. Nevertheless, the interface remained discontinuous and morphologically heterogeneous, and complete metallurgical bonding was not achieved.

Overall, the results demonstrated that the HNO₃-treated conditions promoted significantly better interfacial interaction and diffusion-layer formation compared with the KOH-treated samples. Among all investigated conditions, the HNO₃-treated sample from Trial 2 produced the most developed and comparatively stable bonding morphology, whereas the KOH-treated conditions remained limited by poor interfacial contact, discontinuous bonding regions, and unstable Zn redistribution during brazing.

4.5.4 Comparison of interfacial continuity, porosity, and gap formation

The comparison of interfacial continuity, porosity, and gap formation across Trials 1-3 revealed significant differences in the stability of the bonding interface and the effectiveness of metallurgical interaction between the Al6016 alloy and the Zn-coated steel substrate. Optical microscopy, SEM observations, and BSE imaging demonstrated that the morphology and continuity of the interfacial region strongly depended on the redistribution behaviour of the molten Zn-rich phase during brazing.

For the HNO₃-treated samples, Trial 1 exhibited a thin and discontinuous interfacial region with several localised pores and crack-like defects distributed along the joint line. Partial separation between the substrates was observed in some regions, indicating unstable

spreading of the molten interlayer and limited interfacial continuity. Trial 2 showed the most continuous and developed interfacial morphology among all investigated conditions. The reaction layer appeared broader and more uniform, with improved continuity and fewer sharp discontinuities compared with the other trials. Although localised pores and void-like defects were still present, the interface exhibited significantly better continuity and more stable diffusion-layer formation. Trial 3 demonstrated intermediate behaviour, characterised by irregular interfacial thickness, localised pores, cracks, globular accumulations, and partially separated regions. The morphology indicated unstable redistribution of the Zn-rich liquid phase and incomplete continuity along the bonding region.

In contrast, the KOH-treated samples showed considerably poorer interfacial continuity. Trials 1 and 2 were dominated by large interfacial gaps extending across significant portions of the joint line, indicating lack of effective contact between the substrates during brazing. In these conditions, continuous diffusion-layer formation was not achieved. Trial 3 exhibited partial improvement, with localised Zn-rich bonding regions and limited interfacial contact; however, pores, cracks, and discontinuous regions remained clearly visible. Large rounded pores and interfacial separations suggested localised gas evolution, instability of the liquid phase, and non-uniform spreading behaviour during heating.

Overall, the results demonstrated that the HNO₃-treated samples, particularly Trial 2, achieved the highest interfacial continuity and the lowest degree of gap formation among the investigated conditions. The KOH-treated samples were characterised by unstable interfacial morphology, poor continuity, and extensive separation between the substrates, indicating insufficient wetting and limited metallurgical bonding during brazing.

5. Discussion

5.1 Interfacial bonding mechanisms in Al/Zn-coated steel brazing

The results obtained from optical microscopy, SEM, BSE imaging, and SEM-EDS analyses demonstrated that the bonding mechanism between the Al6016 alloy and the Zn-coated steel substrate was controlled mainly by the behaviour of the Zn-rich interlayer during heating. Since zinc melts at approximately 419 °C, the Zn coating was already in the liquid state during brazing at 470–480 °C. Consequently, the joining process did not occur through direct Al–Fe contact, but rather through the formation of an intermediate Zn-mediated reaction region between the aluminium alloy and the steel substrate.

During heating, the molten Zn phase spread along the interface and promoted localised wetting between the substrates. SEM-EDS analyses showed that the interfacial regions were mainly dominated by Zn-rich compositions together with mixed Al–Zn regions and locally limited Fe participation. In several regions, Zn contents exceeded 70–90 wt%, while mixed regions contained significant Al participation, indicating partial elemental interaction and redistribution within the liquid interlayer. Direct Fe-rich interfacial regions were limited, suggesting that the Zn layer acted as a diffusion barrier reducing direct Al–Fe interaction.

The morphology of the interface strongly depended on the stability of the molten Zn redistribution during brazing. When spreading was relatively stable, the interface developed into a broader and more continuous diffusion region characterised by improved Al–Zn interaction and better interfacial continuity. However, unstable redistribution of the liquid phase resulted in localised Zn accumulation, discontinuous bonding regions, interfacial gaps, pores, and crack formation. Therefore, the overall bonding quality was controlled primarily by the ability of the Zn-rich liquid phase to maintain stable spreading and continuous interfacial contact during heating.

5.2 Role of zinc in wetting and interfacial layer formation

The experimental observations confirmed that zinc played a critical role in both wetting behaviour and interfacial layer formation during brazing. The Zn coating acted not only as a protective surface layer on the steel substrate, but also as an active liquid interlayer controlling elemental interaction between the aluminium alloy and the steel surface.

Optical microscopy and SEM observations demonstrated that regions with more stable Zn spreading generally showed improved interfacial continuity and reduced separation between the substrates. In the best bonding condition, corresponding to the HNO₃-treated Trial 2 at 470 °C, the interface appeared broader and more continuous, with fewer sharp discontinuities compared with the other investigated conditions. SEM-EDS analyses additionally showed mixed Al–Zn regions together with localised Fe participation, indicating more developed diffusion-layer formation.

The Zn-rich liquid phase also reduced the possibility of direct Al–Fe interaction. This behaviour is important because direct Al–Fe contact can promote the formation of brittle Fe–

Al intermetallic compounds. Instead, the molten Zn interlayer promoted the formation of intermediate Al–Zn-rich transition regions that partially separated the aluminium alloy from the steel substrate.

However, when Zn redistribution became unstable, the liquid phase accumulated locally instead of spreading uniformly across the interface. In these regions, SEM observations revealed localised pores, cracks, discontinuous bonding areas, and large interfacial separations. SEM–EDS analyses frequently detected nearly pure Zn-rich accumulations, indicating that the molten interlayer remained locally concentrated and did not form a continuous diffusion region. Therefore, successful bonding strongly depended on stable wetting and controlled redistribution of the Zn-rich liquid phase during heating.

5.3 Influence of aluminium surface preparation

The obtained results demonstrated that the initial surface condition of the aluminium alloy strongly affected the morphology and continuity of the brazed interface. Significant differences were observed between the HNO_3 -treated and KOH-treated samples in terms of interfacial continuity, diffusion-layer development, and defect formation.

The HNO_3 -treated samples generally showed better bonding behaviour and more stable interfacial morphology. Among all investigated conditions, the HNO_3 -treated Trial 2 produced the most continuous diffusion region and the highest degree of elemental interaction across the interface. Optical microscopy and BSE imaging revealed improved continuity with fewer severe interfacial separations, while SEM–EDS analyses confirmed stronger Al–Zn interaction together with localised Fe participation. These observations suggest that the HNO_3 treatment promoted more stable spreading of the molten Zn-rich phase during brazing.

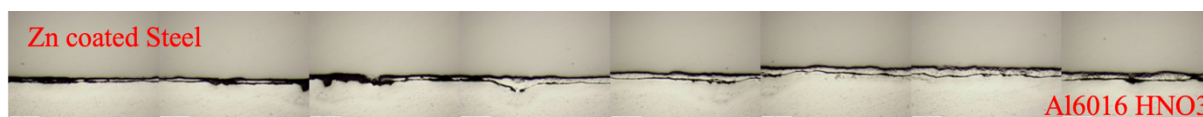


Figure 132. Merged optical micrograph (100×) of the HNO_3 -treated Al6016/Zn-coated steel interface for Trial 1 showing relatively continuous interfacial morphology and localised discontinuities along the brazed region

In contrast, the KOH-treated samples exhibited poorer interfacial continuity and more extensive defect formation. Trials 1 and 2 were characterised by large interfacial gaps and absence of continuous contact between the substrates. Although Zn-rich regions were still detected by EDS analyses, these regions remained localised and did not develop into a stable continuous diffusion layer. Trial 3 showed partial improvement compared with the previous KOH-treated conditions; however, pores, cracks, and discontinuous bonding regions remained clearly visible.

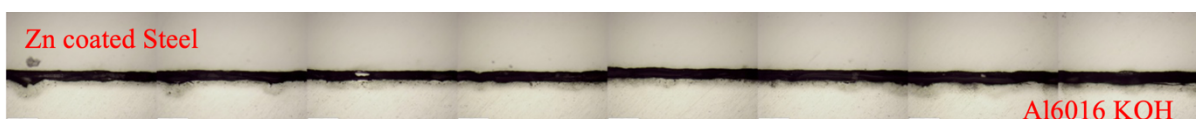


Figure 133. Merged optical micrograph (100 $\times$) of the KOH-treated Al6016/Zn-coated steel interface for Trial 1 showing extensive interfacial gaps between the substrates

These observations indicate that the aluminium surface condition significantly influenced the redistribution behaviour of the molten Zn-rich interlayer. More stable spreading promoted improved contact and diffusion-layer continuity, whereas unstable spreading resulted in localised reaction regions and incomplete bonding.

5.4 Effect of brazing temperature on interfacial morphology

The comparison between brazing conditions at 470 °C and 480 °C demonstrated that brazing temperature strongly influenced the redistribution behaviour of the molten Zn-rich phase and the resulting interfacial morphology.

The best overall bonding condition was observed in Trial 2 performed at 470 °C under HNO₃-treated conditions. In this case, the interface exhibited improved continuity, broader diffusion regions, and stronger elemental interaction compared with the samples brazed at 480 °C. The molten Zn-rich phase appeared to spread more uniformly across the interface, promoting better wetting and more stable diffusion-layer formation.

In contrast, several samples brazed at 480 °C showed unstable interfacial morphology characterised by localised Zn accumulation, irregular layer thickness, pores, cracks, and interfacial separations. The higher temperature likely increased the mobility and redistribution rate of the molten Zn phase, promoting local accumulation and instability instead of uniform spreading. In some regions, excessive liquid-phase redistribution may also have contributed to local separation between the substrates and discontinuous bonding behaviour.

Therefore, the results suggest that the interfacial morphology was highly sensitive to the thermal conditions during brazing. A relatively moderate brazing condition at 470 °C promoted more stable wetting and diffusion-layer development, whereas higher thermal exposure at 480 °C increased the tendency for unstable liquid-phase redistribution and heterogeneous interfacial morphology.

5.5 Local variability of the interface

5.5.1 Non-uniform interfacial gap

One of the most significant features observed in the investigated samples was the strong local variability of the interfacial gap. Optical microscopy and SEM observations demonstrated that the separation between the aluminium alloy and the Zn-coated steel substrate was not constant along the joint region. In some areas, relatively continuous contact was achieved, while in other regions large open gaps were present.

The largest interfacial separations were mainly observed in the KOH-treated samples, particularly in Trials 1 and 2. These gaps prevented continuous diffusion-layer formation and limited elemental interaction between the substrates. The observed morphology indicates unstable wetting and insufficient spreading of the molten Zn-rich phase during heating.

5.5.2 Localised bonding and discontinuities

Several samples showed localised bonding regions surrounded by discontinuous interface areas. In these regions, partial Al–Zn interaction occurred locally, producing isolated bonded zones separated by gaps or crack-like discontinuities.

This behaviour was especially evident in Trial 3, where some portions of the interface exhibited improved continuity while neighbouring regions remained separated. Such morphology indicates that the redistribution of the molten Zn-rich phase was spatially unstable during brazing. Consequently, metallurgical interaction occurred only in localised regions instead of developing uniformly along the entire interface.

5.5.3 Porosity, cracks, and voids

Pores, rounded voids, and crack-like defects were observed in several investigated conditions. These defects were particularly visible in the KOH-treated samples and in the less stable HNO₃-treated regions.

The formation of rounded pores may be associated with localised gas evolution or instability of the liquid Zn-rich phase during heating. Crack-like defects and interfacial separations likely developed due to local shrinkage stresses, unstable spreading behaviour, or insufficient contact between the substrates during brazing. The presence of these defects significantly reduced interfacial continuity and contributed to incomplete metallurgical bonding across the joint region.

<i>Condition</i>	<i>Interface continuity</i>	<i>Gaps</i>	<i>Zn redistribution</i>	<i>Overall bonding quality</i>
<i>HNO₃ Trial 1</i>	Limited	Moderate	Partial	Weak
<i>HNO₃ Trial 2</i>	Most continuous	Low	Stable	Best
<i>HNO₃ Trial 3</i>	Intermediate	Moderate	Non-uniform	Medium
<i>KOH Trial 1</i>	Very poor	Severe	Localised	Very poor
<i>KOH Trial 2</i>	Very poor	Severe	Localised	Very poor
<i>KOH Trial 3</i>	Partial	Moderate	Partial	Limited

Table 21. Comparative summary of interfacial bonding quality, Zn redistribution behaviour, and defect formation for HNO₃ and KOH-treated Al6016/Zn-coated steel brazed samples

5.6 Influence of experimental setup on joint quality

5.6.1 Pressure distribution due to clips

The experimental setup likely influenced the observed variability of the interfacial morphology. The use of steel spring clamps during brazing may have produced non-uniform pressure distribution across the joined substrates. Regions subjected to insufficient local pressure may have experienced incomplete contact, preventing stable spreading of the molten Zn-rich phase.

In addition, the clamping force was not necessarily constant during the full thermal cycle. During heating, thermal expansion of the spring clamps and of the stacked materials could modify the applied pressure and local alignment of the sandwich assembly. During cooling, thermal contraction could further change the stress state at the interface and generate local tensile stresses. These effects may have contributed to variations in interfacial contact, local gaps, discontinuities, or cracks observed in some regions of the brazed joints.

Therefore, the spring clamps should be considered not only as fixing elements, but also as a possible source of local variability in pressure distribution and stress development during heating and cooling.

5.6.2 Sample alignment and contact quality

Accurate alignment between the aluminium alloy and the Zn-coated steel substrate was also important for obtaining stable bonding conditions. Small local misalignments could produce variations in interfacial spacing, leading to localised wetting and discontinuous bonding behaviour.

5.6.3 Time between surface preparation and brazing

Another experimental factor that may have influenced the joint quality was the time interval between surface preparation and introduction of the samples into the furnace. After mechanical or chemical surface preparation, the aluminium surface can rapidly react with air and form a new oxide layer. If the prepared specimens remain exposed to the atmosphere for a long time before brazing, partial surface re-oxidation may occur, reducing the effectiveness of the surface preparation.

This effect is important because the presence of oxide on the aluminium side can limit wetting by the molten Zn-rich phase and reduce metallurgical interaction at the interface. Therefore, minimising the time between surface preparation, assembly and furnace introduction would help preserve the prepared surface condition and improve the repeatability of the brazing process.

5.6.4 Effect of metallographic preparation on interpretation

Metallographic preparation had an important influence on the interpretation of the interfacial morphology. In this work, different sides of the same cut specimen were sometimes analysed, and the obtained optical microscopy and SEM images were not always identical. This difference was especially important because the interface contained soft aluminium, Zn-rich regions, and harder steel, which respond differently during grinding and polishing.

The aluminium side is more susceptible to material removal during polishing, while the steel side remains comparatively stable. As a result, aggressive or prolonged polishing can locally enlarge gaps, expose weakly bonded regions, or modify the apparent continuity of the interface. For this reason, a gentler grinding and polishing procedure was applied on the opposite side of selected specimens.

The observation of different interface appearances from opposite sides of the same specimen indicates that local preparation effects and local joint heterogeneity both contributed to the final micrographs. Therefore, conclusions were not based on a single image only, but on the combined interpretation of optical microscopy, SEM, BSE, and EDS results from different analysed regions. This approach allowed real brazing defects, such as gaps, pores, and discontinuities, to be distinguished more reliably from possible preparation-induced artefacts.

5.7 Proposed interpretation of the joining mechanism

Based on the obtained results, the proposed joining mechanism can be summarized as follows. During heating above the melting point of Zn, the Zn coating transformed into a

liquid interlayer and spread along the interface between the aluminium alloy and the steel substrate. This liquid phase promoted localised wetting and enabled elemental redistribution between Al and Zn.

When spreading remained relatively stable, a broader and more continuous Al–Zn-mediated diffusion region developed, resulting in improved interfacial continuity and stronger metallurgical interaction. However, unstable redistribution of the liquid Zn phase caused localised accumulation, discontinuous bonding, interfacial gaps, pores, and crack formation.

Therefore, successful brazing depended mainly on stable liquid-phase spreading, continuous substrate contact, and controlled interfacial diffusion during heating.

5.8 Main limitations of the present work

Several limitations should be considered in the interpretation of the obtained results. First, the interfacial morphology exhibited strong local variability, meaning that the observed microstructures may not fully represent the entire bonded region. Second, the pressure distribution during brazing was not precisely controlled, which may have contributed to non-uniform interfacial contact.

Additionally, the present investigation was mainly based on microstructural characterisation using optical microscopy, SEM, BSE imaging, and SEM–EDS analyses. Mechanical testing of the joints was not performed; therefore, the direct relationship between microstructure and mechanical performance could not be fully evaluated.

6. Conclusions

The present work investigated the brazing behaviour between Al6016 aluminium alloy and Zn-coated steel using different aluminium surface treatments and brazing conditions. Interfacial morphology and elemental distribution were analysed by optical microscopy, SEM, BSE imaging, and SEM-EDS.

The results demonstrated that the joining process was controlled mainly by the redistribution behaviour of the molten Zn-rich interlayer during heating. Metallurgical interaction occurred through the formation of Zn-rich and Al–Zn transition regions rather than direct Al–Fe bonding.

Among all investigated conditions, the HNO₃-treated sample from Trial 2 produced the most favourable interfacial morphology. This condition showed the highest interfacial continuity, the lowest degree of separation, and the most developed diffusion region. In contrast, the KOH-treated samples generally exhibited unstable bonding behaviour characterised by interfacial gaps, discontinuities, pores, and localised Zn accumulation.

The investigation additionally showed that local pressure distribution, specimen alignment, and metallographic preparation significantly influenced the observed morphology and interpretation of the interface. Differences between opposite analysed sides of the specimens confirmed the strong local variability of the bonded region.

Overall, the study demonstrated that stable redistribution and spreading of the Zn-rich liquid phase are essential for obtaining improved interfacial continuity during Al6016/Zn-coated steel brazing.

6.1 Limitations of the present work

The present investigation was primarily based on microstructural characterisation. Mechanical testing of the joints was not performed; therefore, the relationship between interfacial morphology and mechanical performance could not be directly evaluated.

In addition, local heterogeneity of the interface and variations introduced during metallographic preparation limited the ability to fully represent the entire bonded region using a single cross-sectional observation.

6.2 Recommendations for future work

Future investigations should include mechanical characterisation together with more controlled pressure application and thermal conditions during brazing. The time between surface preparation, assembly and introduction into the furnace should also be minimised in order to reduce possible surface re-oxidation before brazing. Additional studies using higher-resolution characterisation techniques and larger experimental datasets may further improve the understanding of Zn-mediated joining mechanisms and interfacial phase development.

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