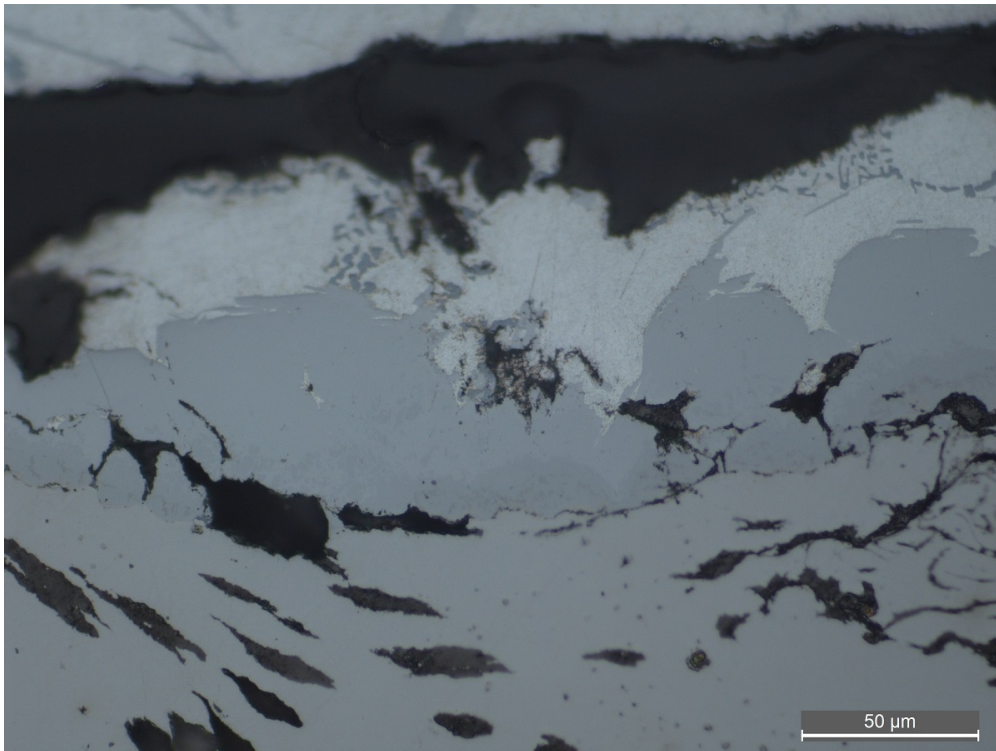




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Effect of cast iron surface treatment on interfacial adhesion of Al/Fe compound cast structure

Master's thesis in Materials Engineering

Nader Heshmati

MASTER'S THESIS 2020:IMSX30

Effect of cast iron surface treatment on interfacial adhesion of Al/Fe compound cast structure

NADER HESHMATI



Department of Industrial and Materials Science
Materials engineering
CHALMERS UNIVERSITY OF TECHNOLOGY
Gothenburg, Sweden 2020

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Supervisor: Aurélien Tricoire, RISE, Research Institutes of Sweden

Examiner: Lars Nyborg, Department of Industrial and Material Science

Master's Thesis 2020:IMSX30

Department of Industrial and Materials Science

Division of Division Materials engineering

Chalmers University of Technology

SE-412 96 Gothenburg

Telephone +46 31 772 1000

Cover: Optical microscope image of formed intermetallic layer during hot dip aluminizing of cast iron substrate.

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Department of Industrial and Materials Science

Chalmers University of Technology

Abstract

Development of the light weight material is necessary for cost optimization and reducing environmental pollution in the automotive industry. However, the aluminum solely does not provide the desired mechanical properties such as wear-resistance and stiffness and it is needed to be combined with ferrous inserts. Compound casting is used as the joining method of Al/Fe components due to its cost-efficiency in which molten aluminum is solidified around a solid ferrous insert to form a metallurgical bonding at the Al/Fe interface. This Metallurgical bonding provides the integrity of the component, and therefore requires to be strong enough to withstand all stresses during the casting process as well as the consumption stage. The formation of a compact reaction layer is restricted by the wetting behaviour of the aluminum and oxide formation on the insert and the aluminum.

In this study, the effect of different cast iron insert surface treatments on the bond formation of the interface between cast iron and AlSi7Mg were investigated. As-cast condition, cleaning, etching, sandblasting, hot-dip galvanizing, acidic zinc plating, hot-dip aluminizing, and thermal spraying samples were pre-heated, followed by gravity casting in a pre-heated mold. The Al/Fe interface was studied by optical and scanning electron microscope (SEM) with a focus on the formation of the oxide and diffusion reaction layer. The results indicated that the diffusion layer formation was suppressed due to the presence of an oxide layer that was formed during the pre-heating stage. The shear strength of the Al/Fe interface was assessed by the push-out test and the result was compared with microstructural changes at the interface. The correlation between the surface roughness of the inserts, their microstructure, and mechanical properties was also discussed. Based on the obtained result, a new cast experiment has been designed by blasting+aluminizing of the cast iron insert, removing the pre-heating, and instant casting after the aluminizing process to ensure the formation of the metallurgical bonding during the compound casting.

Keywords: Cast iron inserts, Interface reaction layer, Interface shear strength, Al/Fe Compound casting

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1

Introduction

1.1 Background

Weight reduction in metallic components without losing safety and functionality can be accomplished by switching from ferrous alloy to aluminum or manganese alloy. In this regard, hybrid materials have attracted extensive attention in developing modern engineering materials for components in the automotive and aerospace industry. Light bimetallic composites consist of an aluminum matrix with cast iron or steel inserts are widely used for lightweight applications. In such a structure, weight reduction, corrosion resistivity, superior castability, high thermal and electrical conductivity of the components is governed by the aluminum matrix while cast iron or steel provides high strength and wear resistance as well as excellent vibration damping. Numerous methods such as friction stir welding, extrusion, accumulative roll bonding, explosive welding etc, are used for Al/Fe bimetallic composites fabrication. However, compound casting has gained paramount importance among all processes.

In compound casting two metal alloys, one in solid-state and another in the liquid state, are combined in order to form a uniform diffusional reaction layer between two materials. This method is believed to be the best process for producing automotive component due to its cost efficiency, high production rate and near net shape castability of complex geometry.

1.2 Bonding type

The strength of a dissimilar metal component strongly depends on the quality of the bonding at the interface. Weak bonding at the interface does not allow for the load to transfer between two metals, causing failure in the whole structure. During the casting of the component two types of bonding between matrix and inserts are possible: mechanical bonding and metallurgical bonding.

1.2.1 Mechanical bonding

Mechanical bonding usually forms in compound casting due to the difference in coefficient of thermal expansion between the solid insert and the molten metal. In the case of aluminum/iron bimetallic structure, aluminum starts cooling at a higher rate compared to the substrate, causing more shrinkage for aluminum during solid-

ification which results in compressive stress on the substrate material. Mechanical bonding is necessary for the structural integrity of compound cast structures.

1.2.2 Metallurgical bonding

In mechanical bonding, there is no chemical interaction between the two materials while in metallurgical bonding there is a diffusion between two metals. In this case, as a result of the interaction between inserts and liquid material, an intermetallic layer forms at the interface which acts as a metallic bridge between the two materials. The composition, morphology and consequently properties of this metallurgical bond depends on the material used.

1.3 Problem statement

This project aimed to achieve a metallurgical bonding by casting molten aluminum around solid cast iron. An excellent continuous metallurgical bond without crack and gaps will provide require mechanical strength of the component to withstand solidification, residual and thermal expansion stresses as well as stresses during usage stage.

1.3.1 Challenges

The large scale application of compound casting is limited owing to oxide formation on cast iron surface and the vast difference in physical and chemical properties of aluminum and cast iron, such as melting point and coefficient of thermal expansion as well as small solid solubility of Al and Fe at contact temperature. Hence, the wettability of molten aluminum diminishes considerably leading to poor metallurgical bonding at the interface between aluminum and cast iron. Since properties of aluminum and cast iron bimetallic composites significantly depend on an excellent metallurgical bonding, controlling oxide formation, improving wettability as well as reducing physicochemical differences between Al and Fe are of great importance in order to obtain sound metallurgical interface.

2

Literature Review

2.1 Interaction between pure Fe and molten Al

The Al/Fe equilibrium binary diagram was shown in Figure 2.1. In this system intermetallic compound (IMC) formation is more preferential owing to small solid solubility of aluminum in iron [1]. Based on the phase diagram six different IMC of Fe_3Al , $FeAl$, $FeAl_2$, Fe_2Al_3 , Fe_2Al_5 and $FeAl_5$ were characterized. Crystal structure and thermodynamic constants of characterized phases of the binary diagram were summarized in Table 2.1 and 2.2, respectively [2].

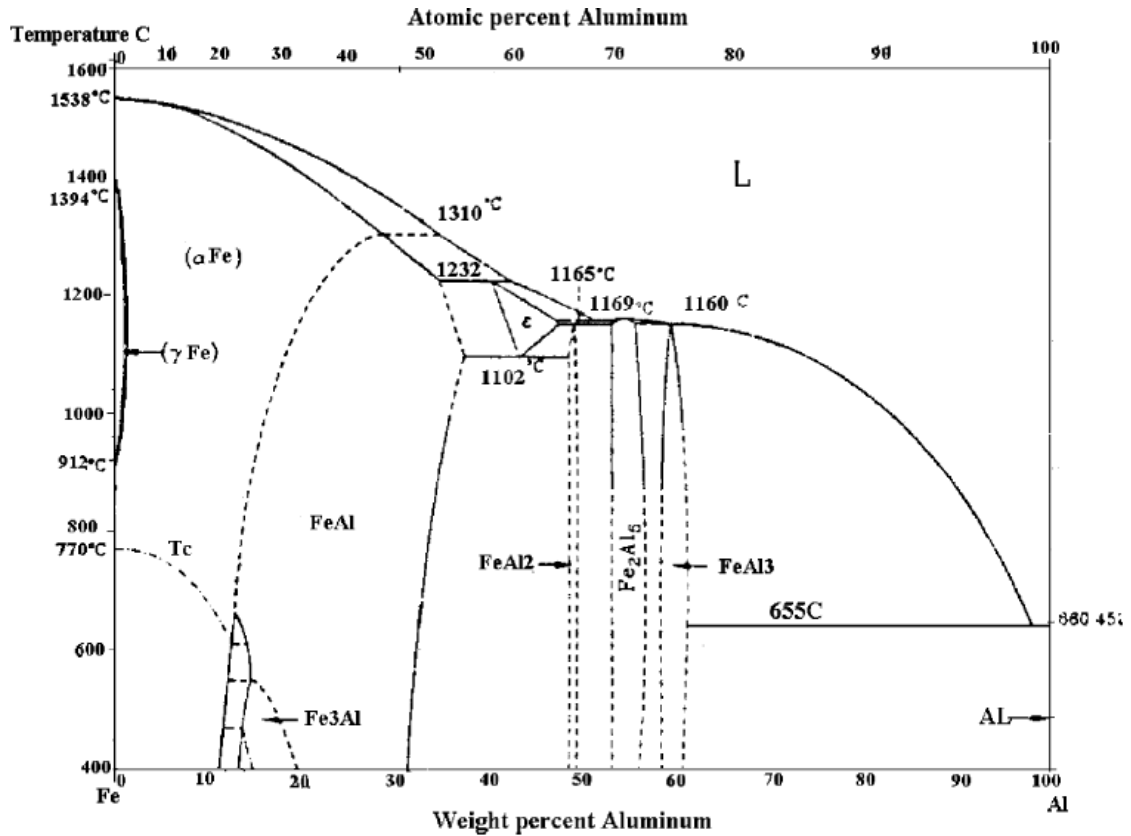


Figure 2.1: Al-Fe equilibrium phase diagram [2].

Table 2.1: Crystal structure and stability range of Fe-Al phase diagram at room temperature [2].

Phases	Crystal structure	Stability range (at. %)
Fe solid solution	BCC	0-45
γ -Fe	FCC	0-1.3
$FeAl(\beta 2)$	BCC order	23-55
$Fe_3Al(\beta 1)$	Do 3	23-34
$Fe_2Al_3(\epsilon)$	Cubic complex	58-63
$FeAl_2(\xi)$	Triclinic	66-66.9
$Fe_2Al_5(\eta)$	Orthorhombic	70-73
$FeAl_3(\theta)$	Monoclinic	74.5-76.5
Al solid solution	FCC	99.998-100

Table 2.2: Thermodynamic constant for Fe-Al binary intermetallic compound [2].

Intermetallic compound	ΔH_{298} ($Jmol^{-1}$)	ΔS_{298} ($K^{-1} mol^{-1}$)	ΔG_{298} ($Jmol^{-1}$)
$FeAl_3(\theta)$	-112560	95.6	-22869
$Fe_2Al_5(\eta)$	-194040	166.7	-19636
$FeAl_2(\xi)$	-81900	73.3	-16999
$FeAl(\beta 2)$	-51240	51	-11090
$Fe_3Al(\beta 1)$	-57372	28	-4827

It has been reported by many research groups that η phase is the major intermetallic compound forming during various Al-Fe joining processes [1, 3, 4, 5, 6]. Although Fe_2Al_5 has higher Gibbs free energy than $FeAl_3$ and hence less thermodynamically favorable phase to be formed, its orthorhombic crystal structure has a more open arrangement of atoms along c-axis which makes Al atoms diffusion more rapidly in that direction, leading to saw-tooth or tongue-like microstructure [5, 2]. As a result, faster growth (kinetic) of Fe_2Al_5 compensates for higher Gibbs free energy of the system.

Takata et. al [7] claimed that this saw-like morphology is owing to the induced strain from anisotropic volume expansion during $\alpha \rightarrow \eta$ phase transformation. In this reaction pure iron with bcc structure should transform to orthorhombic Fe_2Al_5 structure which subsequently requires an increment in volume per atom for unit cell from $11.7 \times 10^{-30} m^3$ to $13.7 \times 10^{-30} m^3$. As a result, the strain from volume expansion can introduce a stress field as shown in Figure 2.2. In order to have the minimum total strain energy of the system, vacancies flow from tip to side towards this compressive stress field which corresponds to Al diffusion in the opposite direction to the grain tip with tensile stress, leading to higher diffusion of Al in the growth direction.

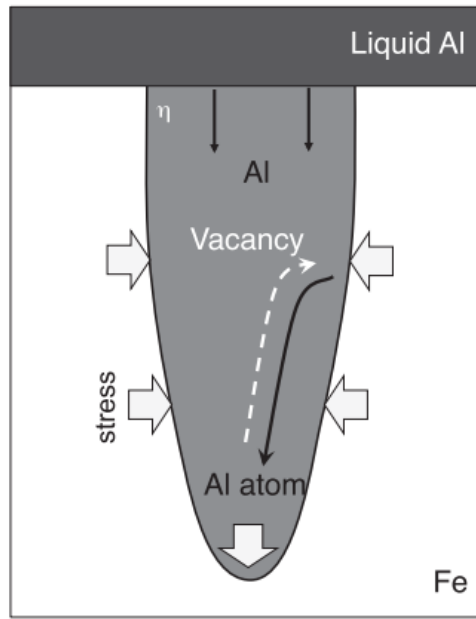


Figure 2.2: Vacancy and Al diffusion path due to the stress field induced by volume expansion according to [7].

2.2 Interaction between Al-Si alloy with ferrous inserts

In many cases, instead of having pure iron and aluminum, there is an Al-Si alloy coming in contact with steel or cast iron insert. Morphology and growth rate of the reaction layer are determined by the chemical composition of the insert and molten material [1]. Hence, a higher amount of alloying elements will alter the type and morphology of the IMC. Due to the fact that mechanical properties of the bimetallic composite mostly rely on the IMC layer, identifying the effect of different alloying elements on both growth rate and morphology of the intermetallic phases is of great importance.

2.2.1 Intermetallic layer sequence

Eggeler et al.[8] studied the interaction layer during a hot-dip experiment of liquid aluminum containing 2 wt.% Si and mild steel substrate at 780°C and 792°C. Similar to the interaction layer for pure aluminum, they identified two major phases of Fe_2Al_5 and $FeAl_3$. The former formed near steel while the latter was found close to the Al side.

In [9], hot-dip coating of aluminum with 0, 4, 8, and 12 wt.% Si were applied on low carbon steel. They reached the conclusion that the coating with an 8 wt.% Si intermetallic compound has a layered structure. In the vicinity of aluminum side, the intermetallic composition corresponded to Al_7Fe_2Si followed by a thin intermediate layer of $Al_{20}Fe_7Si$ and an inner layer of $Al_{19}Fe_8Si$ with dispersed Al_3Fe_2Si

precipitates.

In a study by [10], mild steel was hot-dipped in an aluminum bath containing up to 10 wt.% silicon at 700°C for 180 s. They observed over 2.5 wt.% Si some new ternary (Al-Fe-Si) intermetallic phases such as $\tau_{5(C)} - Al_7(Fe, M)_2Si$, $\tau_{5(H)} - Al_7Fe_2Si$, $\tau_5 - Al_4FeSi$ were formed while the majority of the reaction layer for different silicon content still consists of η and θ phases.

Springer et al. [11] carried out a hot-dip experiment using low carbon steel in Al-5%Si at 670°C followed by air cooling. TEM and EDS analysis revealed that the intermetallic phase contains three layers. The outer layer which was in contact with molten aluminum consisted of Al_8Fe_2Si (τ_5), the intermediate layer of $FeAl_3$, and the inner layer (close to steel) of η phase with dispersed islands of $Al_2Fe_3Si_3$ (τ_1).

Later, Lemmens [12] analyzed the reaction layer formed during hot-dip aluminizing of ferritic steel in molten aluminum with silicon ranging from 0 to 10 wt.% with different dipping time and temperature. Their results showed that the ternary intermetallic phase of τ_1 formed when Si content exceeded 3 wt.%. As it illustrated in Figure 2.3 islands of τ_1 distributed in the Fe_2Al_5 phase adjacent to the steel while θ composed the majority of the reaction layer close to the aluminum. Above 5 wt.% Si, a new ternary phase of τ_5 was characterized between Al and $FeAl_3$ phase which is consistent with [11] data. They also observed that as the Si content rose from 5 wt.% to 10 wt.%, the θ phase adjacent to the aluminum side almost was entirely replaced with τ_5 (Figure 2.3).

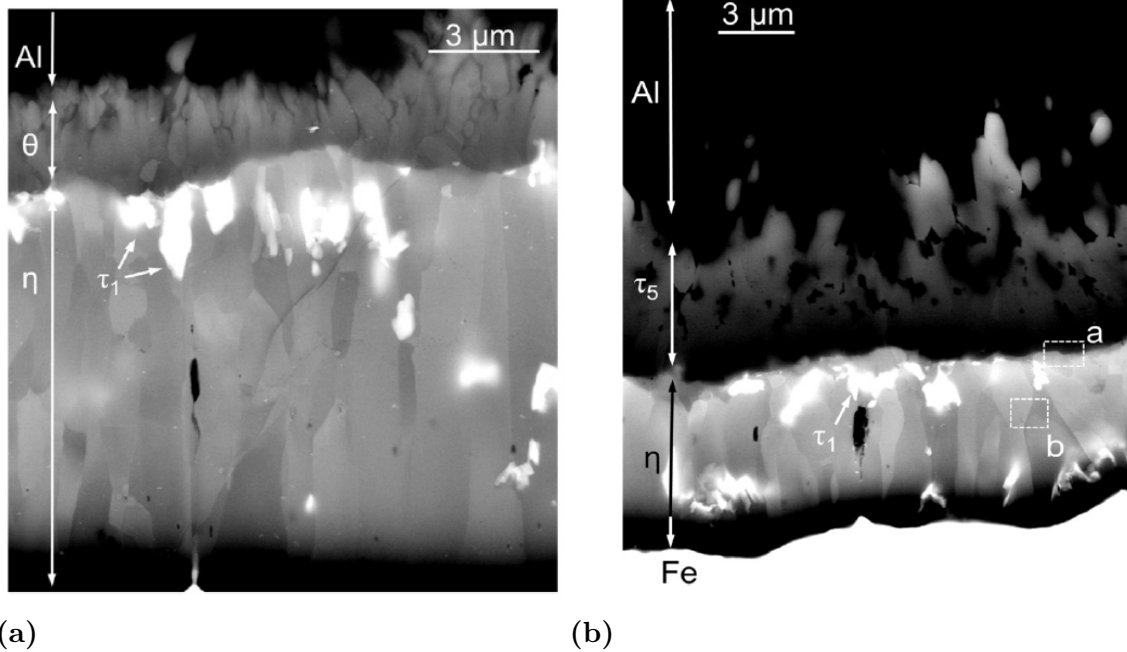


Figure 2.3: BSE image of the reaction layer for: a) Al-3Si and b) Al-10Si [12].

Zhe et al. [13] carried out a hot-dip coating of Al-7Si-3Mg on low carbon steel at 680°C for 40s, followed by quenching in water ($500Ks^{-1}$). The same procedure was

repeated for another sample, but after immersion in aluminum it cooled down in furnace with a much lower cooling rate ($1Ks^{-1}$). They concluded that the latter sample had a much thicker reaction layer which consisted of three different layers. An inner layer of η phase, an intermediary layer of $Al_{7.4}Fe_2Si$, and an outer layer of $Al_{4.5}FeSi$. On the other hand, the quenched sample was made of an only thin layer of $Al_{7.4}Fe_2Si$.

2.2.2 Effect of alloying elements on microstructure and kinetics of intermetallic layer

2.2.2.1 Si

Increasing Si content in molten aluminum is known to reduce the thickness of the reaction layer. In addition, by increasing Si the growth rate of Fe_2Al_5 (η) phase is also diminished considerably which have been confirmed by many research groups [8, 10, 11, 12, 14, 15, 16]. There exists a debate over the retardation growth mechanism of η phase owing to Si addition in the literature. Based on [17], because of the open structure of Fe_2Al_5 there are around 30% vacancies along the c-axis. Hence, there is a preferential diffusion path for Al resulting in an irregular tongue-like morphology as discussed by [2]. These vacant positions in the Fe_2Al_5 phase then will be occupied by Si. Consequently, the easy movement of the Al element along the c-axis is blocked which subsequently hinders the formation of the tongue-like structure. The magnitude of Si in liquid aluminum controls the growth rate and morphology of the Fe_2Al_5 where higher Si concentration results in the thinner reaction layer. A similar explanation was used by others [11, 10]. This mechanism can also be explained with the help of the thermodynamic concept. Since η phase growth is a diffusional process, a smaller reaction layer means smaller diffusion of Al and Fe in Fe_2Al_5 . The interdiffusion coefficient of Al and Fe in Fe_2Al_5 is given by equation (2.1):

$$D_{Al,Fe}^{Fe_2Al_5} = D_0 \exp\left(\frac{-Q}{RT}\right) = \alpha a^2 \nu X_v \exp\left(\frac{\Delta S}{RT}\right) \exp\left(\frac{-Q}{RT}\right) \quad (2.1)$$

In this equation, D_0 is diffusion constant, a jump length, α geometry factor, ν jump attempt frequency, Q activation energy, and ΔS activation entropy. For solid-state diffusion, element should have sufficient energy to open the structure and jump. Increasing Si content induces a strain in the crystal structure resulting in more distortion and consequently lower the activation energy for atomic jumps. Moreover, it increases the entropy of the system since there is more element in the structure. On the other hand, more Si reduces the volume fraction of available vacancies. Hence, based on equation (2.1) the main reason for $D_{Al,Fe}^{Fe_2Al_5}$ is the lower value of vacancy concentration [10, 18].

Lemmens et al. [12] claimed that if growth rate reduction by addition of Si is attributed to the occupation of vacancies in η , there should be a difference in Si

concentration for reaction layers that were grown at different rates. However, they measured almost the same silicon content for various growth rates. In a similar study [10], it was also reported that although growth rate diminished significantly as Si content increased, the magnitude of silicon in Fe_2Al_5 remained unchanged around 2 wt.% which is the solubility limit of Si in Fe.

A study by Kumatso [19] came to the conclusion that increasing Fe dissolution as a result of Si addition is responsible for the lower growth rate of the η phase. In contrast, monitoring Fe concentration in pure aluminum and silicon containing aluminum by Eggeler [8] showed that there is no difference in Fe content in aluminum with or without silicon. They then explained that lower growth rate is owing to solid-state principles rather than the liquid, as opposed to previous work.

Lemmens et al. [12] studied the effect of Si addition on intermetallic formation during hot-dip aluminizing. They concluded that when Si concentration exceeded 3 wt.% there will be a new ternary phase. Due to a much more complex structure, these new constitutes have a slower growth rate than usual Fe-Al phases which act as a diffusion barrier against Al and Fe, leading to a lower growth rate of Fe_2Al_5 . Similarly, [14] showed Si enrichment at grain boundaries between η and θ phases. These areas which contain up to near ten times more silicon compared to aluminum bath could affect the growth rate of η either by its nucleation or acting as a diffusion barrier.

2.2.2.2 Mn

Liu et al. [20] investigated the effect of different Mn concentrations in liquid aluminum up to 5 wt.% on the diffusional reaction layer between Al-7Si and cast iron. They reported that above 1.5 wt.% Mn the amount of detrimental needle-like phase reduced considerably. In addition, in contrast with Si, Mn facilitated the formation of some intermetallic constitute ($\alpha - Al_{15}(Fe_xMn_{1-x})_3Si_2$) and metallurgical reaction layer. The mechanism behind this growth rate enhancement is that Mn counteracts the preventing effect of Si in liquid aluminum by increasing Al atoms mobility.

2.2.2.3 Cu

In a study, Yousaf et al. [21] explored the effect of Cu addition (11 wt.%) in melt on the growth rate and morphology of intermetallic compounds. It was concluded that the reaction layer thickness was reduced from $150\mu m$ in pure aluminum to around $50\mu m$ in Cu containing aluminum bath. EDS and XRD analysis identified new intermetallic phases of Al_2Cu and Al_7Cu_2Fe while the majority of the reaction layer still composed of Fe_2Al_5 . These newly formed phases then were believed to restrain Al atoms diffusion toward the substrate and act as a diffusion barrier, leading to a lower growth rate of reaction layer and subsequently much thinner thickness.

Interface reaction study of different binary Al-alloy coating on steel inserts by [22] confirmed that the addition of 7% Cu resulted in thinner reaction layer while no significant change in wetting angle was observed.

2.3 Effect of insert type

2.3.1 Steel

There have been several studies on producing compound structures by using mild steel substrate and molten aluminum. A variety of different processing procedures have been used by research groups to achieve proper metallurgical bonding. They also evaluated the shear strength of their components by push-out test, however quantitative comparison of their works would not be easy since they used different sample preparation, processing, and testing methods.

Durrant et al. [23] prepared both coated and uncoated bimetallic components using squeeze casting. They showed that hot-dipping of steel inserts in aluminum eased the formation of metallurgical bonding which subsequently resulted in higher shear strength. They also made a connection between surface roughness of the sample and the bonding strength of the component.

Dezellus et al. [24] studied the failure mode in hot-dipped mild steel in Al-Si with a push-out test. They reported that hot-dipping allowed for the formation of a homogeneous diffusion layer at the interface. The push-out test data analysis indicated that crack initiation started from the bottom part of the intermetallic layer, however, no brittle failure was observed in the component.

Choe et al. [25] produced a bimetallic component using an expandable pattern casting experiment at various time and pressure for Zn plated and shot pinned samples. They came up with the conclusion that hot-dip galvanizing was more beneficial for improving bonding adhesion between aluminum and mild steel. They also reported that higher pouring temperature promoted the Fe diffusion in the reaction layer resulting in better bonding at the interface.

Pan et al. [26] used ultrasonic vibration during casting to join pipe steel to molten aluminum. Their results showed that a metallurgical bonding successfully formed. They reached the conclusion that ultrasonic vibration is effective for breaking down the oxide layer on the steel. As a result, the wettability of the aluminum increased significantly, leading to stronger interfacial bonding.

2.3.2 Cast iron

Many research groups also have studied the application of cast iron inserts in compound structures with molten aluminum. In the following mentioned studies cast

iron inserts experienced a hot-dipping process either in aluminum or zinc to ensure the formation of metallurgical bonding. The attention was mostly focused on composition and morphology of the reaction layer.

Viala et al. [1] carried out hot-dipping of granular cast iron inserts in Al-Si followed by pouring aluminum around insert using gravity casting. They observed a continuous diffusion layer at the interface which consisted of three intermetallic phases: $\eta(Al_5Fe_2)$, $\tau_5(Al_{7.4}Fe_2Si)$ and $\tau_6(Al_{4.5}FeSi)$.

Liu et al. [20] tried to bond gray cast iron by dot dipping in silicon containing aluminum with different concentrations of Mn and then placing inserts in a mold and gravity cast of aluminum. They characterized some intermetallic phase of Al-Fe-Mn-Si at diffusion zone.

2.4 Effect of thermal treatment

2.4.1 Insert preheating

Preheating the inserts will reduce the temperature difference between molten aluminum and solid inserts which can prevent the substrate to act as an internal chill during the solidification process.

In [27] the effect of preheating on metallurgical bonding of steel substrates was investigated. Three inserts were pickled in HCl. Then two out of them preheated at 100°C and 300°C while the last one remained at ambient temperature. They reported that no metallurgical bonding occurred and there was no difference between interface shear bond strength of samples. They came to the conclusion that due to preheating a thick oxide layer was developed on the steel substrate which prevented diffusional reaction.

Durrant et al. [23] assessed the effect of pre-heating at 300 and 900°C on bonding strength between uncoated mild steel and Al-7Si. They reported that no metallurgical bonding formed between mild steel and aluminum at both preheating temperatures. They believed that when the hot melt came in contact with a relatively cool insert at 300°C there was not enough time for diffusion of Al and iron resulting in a poor connection. On the other hand, pre-heating the sample up to 900°C triggered the iron oxide formation on the steel surface which also prevented the diffusion reaction between aluminum matrix and steel substrate.

Preheating time and temperature affect the bonding strength of Al/Mg bimetallic. In [28, 29], shear strength rose gradually with an increase in substrate temperature and it reached a maximum due to better metallurgical bonding between Al and Mg. Further increase in temperature caused a reduction in strength owing to the presence of a large proportion of brittle intermetallic in the interface.

2.4.2 Post heat treatment

Jiang et al. [3] studied the influence of heat treatment (aging time and temperature) on microstructure and mechanical properties of hot-dip galvanized steel substrate in liquid aluminum. A continuous reaction layer was observed in the as-cast condition. As heat treatment time and temperature increased there was an increase in reaction layer thickness. They also reported that the size and morphology of the intermetallic compounds changed during heat treatment while no new intermetallic phase formed and the composition of the phases also remained unchanged. They explained that an appropriate heat treatment process altered the morphology of the silicon in reaction layer from plate-like to granular particles which dispersed uniformly in the interaction layer. Moreover, the τ_6 phase showed a more smooth edge, leading to an increment in shear strength from $12.8MPa$ in as-cast condition to $17.86MPa$ for the heat treated specimen. On the other hand, excessive heat treatment had led to excessive growth of the intermetallic phase which induced a crack at the interface. Consequently, bonding strength dropped considerably.

In another study, Jiang et al. [6] instead of steel, carried out a combination of hot-dip galvanizing and aluminizing on cast iron inserts followed by heat treatment at various solution time and temperature. They reported that in as-cast sample reaction layer consisted of Fe_2Al_5 , $\tau_{10} - Al_9Fe_4Si_3$, $FeAl_3$, $\tau_5 - Al_8Fe_2Si$ and $\tau_6 - Al_{4.5}FeSi$ phases. The growth of the interaction layer is governed by diffusion time and temperature as given by (2.2) and (2.3) equations:

$$X = Dt^{0.5} \quad (2.2)$$

$$D = D_0 \exp \frac{-Q}{RT} \quad (2.3)$$

In these equations X represents the thickness of the reaction layer, t is diffusion time, D and D_0 are diffusion coefficient and diffusion constant, respectively. Hence, higher solution time and temperature resulted in obtaining a thicker intermetallic layer. Similar to previous study heat treatment did not change the intermetallic compound but their size and morphology. The nano-hardness test results also indicated that heat treatment only increased hardness by a modest 10% compared to as-cast component

Viala et al. [1] performed a heat treatment on aluminum/cast iron compound structure for 12h at $520^\circ C$ followed by quenching and aging at $170^\circ C$ for 6h. Their results also confirmed that the thickness of the reaction zone increased, however, in contrast with [6] and [3], the composition of intermetallic phases changed. Primarily formed τ_5 and τ_6 phases replaced with a new ternary phase of $\tau_2(Al_5Fe_2Si_2)$ and τ_{10} through solid-state reaction during the thermal treatment process. Besides, the formation of the Kirkendall voids was observed between interface and aluminum which is believed to be due to uneven diffusion of Al atoms toward the intermetallic layer. More diffusion in one direction corresponded to vacancy diffusion in the opposite direction, resulting in vacancy pie-up and subsequently void formation as shown in Figure 2.4.

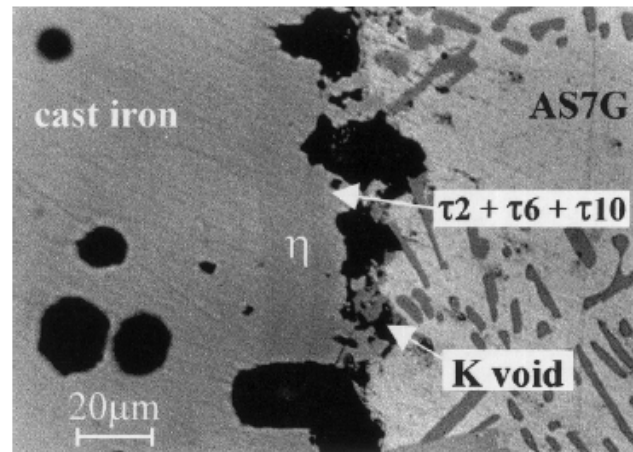


Figure 2.4: Kirkendall voids formation during heat treatment [1].

2.5 Effect of surface treatment

Aguado et al. [27] explored the effect of chemical and mechanical treatments of steel inserts on the shear strength of bimetallic specimens. Cleaning, pickling, hot-dipping, and blasting were applied on steel followed by T6 heat treatment for all samples. They concluded that no metallurgical bonding can be achieved for uncoated samples since diffusion was hindered by a thick oxide layer that formed during mold filling. As a result, no increase in shear strength was seen in pickled samples. However, the sandblasted specimen had the highest shear (31MPa) strength owing to its rougher surface which enables more mechanical grip between aluminum and steel, leading to higher strength.

In the case of hot-dip galvanizing and zinc coating, although a reaction layer has been formed between aluminum and steel, no increase in the push-out test was measured. This is due to the large quantity of brittle intermetallic in the diffusion layer of the hot-dipped sample as well as the thick oxide layer formed in the Zn-coat specimen.

Among coated samples, hot-dip aluminizing showed the best performance (22MPa). The same result also obtained for the combination of blasting and aluminizing while applying grit blasting and Ni/Cu coating resulted in higher strength of 27MPa .

Jiang et al. [4] studied the effect of various surface treatments such as aluminizing, surface modifier (ammonium chloride solution), and a combination of both on microstructure and mechanical properties of aluminum steel bimetallic composites. Upon investigation, they concluded that applying hot-dip aluminizing or surface modifier solely could not grantee a metallurgical bonding. In the former case, oxide layer formed on the substrate prevented chemical diffusion between Al and Fe, resulting in a clear gap at the interface. In the latter case, by using an ammonium chloride solution, the oxide layer at the steel insert was removed which could increase the wettability of the aluminum. However, short interaction time between Al and Fe can only result in a mechanical bonding at the interface. On the other hand,

applying surface modifier and hot-dip aluminizing at the same time combined the beneficial effect of each treatment (increasing wettability and facilitating of diffusion) leading to a sound metallurgical reaction layer which showed 40% higher shear strength in the push-out test in comparison with the untreated specimen.

In another study, Jiang et al. [30] explained how applying a zinc coating can affect mechanical properties and microstructure of the Al/Fe compound structure. They reported that there is no continuous reaction layer in the uncoated sample. They believed it was attributed to the oxide scale formation during the preheating stage which prevents direct diffusion between aluminum and iron as well as poor wetting of steel substrate by aluminum. The application of zinc coating, on the other hand, resulted in sound metallurgical bonding through three mechanisms. Firstly, during zinc hot-dipping, the formed oxide layer on the substrate was removed which led to good adhesion between zinc and steel insert. Secondly, because of the higher melting temperature of liquid aluminum (730°C) than zinc (450°C), the coating is probable to melt upon aluminum pouring that can ease aluminum spreading on the substrate. Finally, zinc coating had a considerable effect on improving the wettability of aluminum on steel. The push-out test for the coated sample also indicated 71% higher strength compared to the as-cast sample.

A study [31] on a combination of hot-dip galvanizing and aluminizing on gray cast iron showed that a compact reaction layer consisting of ternary phases had been formed between Al and Fe. Similar to the previous study, Zn coating prevented surface oxidation of cast iron as well as improved the wettability of aluminum. They explained that during hot-dip aluminizing a reaction layer formed between cast iron and aluminum which was mainly composed of Fe_2Al_5 , $\tau_{10} - Al_9Fe_4Si_3$, $FeAl_3$, $\tau_5 - Al_8Fe_2Si$ and $\tau_6 - Al_{4.5}FeSi$ from cast iron to aluminum side, which is consistent with results obtained by [6]. After surface treatment, molten aluminum was poured around the cast iron insert. They observed that molten aluminum fused through residual aluminum from the hot-dipping process which resulted in the final component. EDS data indicated that oxygen distribution was almost uniform within the whole component while in uncoated specimens an oxygen peak was detected at the interface. They came up with the conclusion that oxide formation at the interface was responsible for poor mechanical bonding in the untreated sample.

The alloying element of aluminum alloy has a great impact on interfacial bonding in the bimetallic composite. In this regard, Liu et al. [20] studied the effect of surface treatment (fluxing and dipping) and addition of Mn, ranging from 0 to 5%, on interfacial diffusion zone of aluminum and gray cast iron. It was concluded that combining both surface treatment methods resulted in a continuous intermediate reaction layer. It is because of fluxing activates the cast iron surface and protects it from oxidization before dipping. Hence, the wettability of the cast iron increased during hot-dipping which effectively improves the metallurgical bonding of the composite at the interface. Furthermore, by increasing Mn content in dipping bath, less harmful needle-like phase was observed at the interface. On the other hand, it eased the formation and growth of intermetallic phases and the metallurgical reac-

tion layer. The maximum effect has been reported at 1.5 wt.% Mn.

Guo et al. [32] developed a new surface treatment method by adding 0.2 wt.% Bi into the zinc bath before hot-dip galvanizing. They reported that this new process increased the shear strength of interface up to 32MPa which is claimed to be the highest value reported in the literature for compound structures up to now. They observed by increasing the depth of Al/Fe at ferrite/graphite interface in cast iron matrix the shear strength of the component reduced. The addition of Bi in zinc bath triggered the formation of a zinc rich area within the cast iron matrix which was accompanied by Al/Fe phases. Consequently, by having Al/Fe phase adjacent to the Zn-rich region less Al/Fe appeared in the ferrite-graphite interface in cast iron, resulting in an improvement in shear strength of Al/Fe composite.

The formation of a stable oxide layer on the solid inserts hinders the required diffusional reaction for metallurgical bonding. Hence, successful removal of this formed oxide layer is a necessity for ensuring a desirable bonded connection. In this regard, Scharf et al. [33] studied the effect of pre-treatments for disrupting the oxide layer using a chemical flux, laser ablation, and a combined treatment. It was found that employing each method solely only led to partial diffusion at the interface while applying both methods together successfully broke up the formed oxide layer on iron inserts, resulting in a complete connection at the reaction layer.

2.6 Interface shear strength

In the literature, the bonding strength of bimetallic castings has been examined mostly by push-out tests. In this experiment, substrate material with aluminum around it was placed on a plate. Then a steel punch pushes the insert out of the bimetallic component, with a continuous measuring of the punch load until failure and its displacement during the test. By having punch load (F) and the surface area of the insert, shear stress at the interface, τ , can be calculated from equation (2.4):

$$\tau = \frac{F}{[\pi D(t - d)]} \quad (2.4)$$

where D represents the diameter of the substrate, t is the thickness of the disc specimen and d equals to the measured displacement of the insert.

Table 2.3 summarized the reported shear strength of different studies in the literature using a push-out test.

Table 2.3: Interface bonding strength reported by different research groups.

Authors	Alloys	Insert	Preparation method	Bond strength (MPa)
Aguado et al. [27]	Al7SiMg	Steel	Cleaned	13
	Al7SiMg	Steel	Etched	12
	Al7SiMg	Steel	Etched+Preheat 100°C	14
	Al7SiMg	Steel	Etched+Preheat 300°C	12
	Al7SiMg	Steel	Blasted	31
	Al7SiMg	Steel	Etched+Aluminized	2
	Al7SiMg	Steel	Hot-dip galvanized	14
	Al7SiMg	Steel	Zinc electroplated	14
	Al7SiMg	Steel	Cu/Ni coating	19
	Al7SiMg	Steel	Blasted+Aluminized	22
	Al7SiMg	Steel	Blasted+Cu/Ni coating	27
Pan et al. [26]	A356	Steel	Ultrasonic vibration	60
Jiang et al. [3]	ZL114A	Steel	Solution treated at 500°C for 2h	18
	ZL114A	Steel	Solution treated at 500°C for 6h	15
Dezellus et al. [24]	AS-13	Steel	Aluminized	10
Durrant et al. [23]	Al-Si7	Steel	Preheat 300°C	30.5
	Al-7Si	Steel	Preheat 900°C	44.5
	Al-7Si	Steel	Aluminized+Preheat 300°C	114
	Al-7Si	Steel	Aluminized+Preheat 900°C	112
Li et al. [34]	Al-2Si	Cast iron	Aluminized+Preheat 700°C	3.6
	Al-6Si	Cast iron	Aluminized+Preheat 700°C	8.8
	Al-11Si	Cast iron	Aluminized+Preheat 700°C	13.6
	Al-16Si	Cast iron	Aluminized+Preheat 700°C	4.8
Guo et al. [32]	ZL101	Cast iron	Hot-dipped in Zn-0.2% Bi	32
Jiang et al. [4]	ZL114 A	Steel	Surface modifier+Aluminized	10.4

Durrant [23] reported that for hot-dipped samples failure occurred near the Al-7Si matrix rather than the steel/aluminum interface. Aguado et al. [27] observed an interfacial failure for samples without metallurgical bonding which they claimed to be due to oxide formation at the interface. On the other hand, the metallurgically bonded samples failed adjacent to the coating/Al interface which is consistent with the previous study. In contrast, Dezellus [24] observed crack initiation at the bottom surface of the formed reaction layer.

As it was observed, the majority of the research question regarding the compound casting of the Al/Fe centered around the steel substrate. Hence, the effect of different surface treatment on the microstructure and mechanical properties of the compound cast Al/Fe structure with cast iron insert remained unexplored.

In the present work, the cast iron inserts were divided into the two groups of coated and uncoated samples. The former set of samples was supposed to explore the effect of surface roughness, and cleanliness on bonding while the latter was focused on the

influence of various coating materials and processes. Also, the effect of the interface morphology of the samples were studied by surface roughness measurement and finally, mechanical properties of specimens were evaluated by the push-out test.

3

Methods

3.1 Surface Treatment Preparation

In order to investigate the effect of coating material, coating process, surface roughness, and surface activation on the diffusion zone and the bonding strength of the interface between aluminum and cast iron, seven types of the surface treatment were applied on the surface of cast iron inserts. For three of them, no coating was used and samples only cleaned, etched, and blasted. Two specimens had a zinc coating with two different processes of hot-dip galvanizing and electroplating. Al coating with hot-dipping and thermal spraying were also applied on two cast iron inserts. In the following sections, there is information about each process in more detail.

3.1.1 Cleaning

For cleaning, cast iron inserts were immersed in Bonderite C-NE 5088 2.5% diluted in water at 60°C. Then samples were flushed with approximately 2L water and dried for 20 – 30min at 60°C. Finally, specimens were stored in plastic bags to prevent them from further oxidation before casting.

3.1.2 Etching

For etching, samples were first cleaned with the above-mentioned procedure. Cleaned inserts then were immersed in H_2SO_4 5% at 50°C for 30s, followed by rinsing in deionized water, then after immersing in $NaOH$ 10% for 30s, and finally rinsing in deionized water again.

3.1.3 Blasting

Blasting was performed manually in a blasting machine in a pressure of 0.3MPa. The distance from the nozzle to the cast iron inserts was around 7cm and the blasting was done twice.

3.1.4 Hot-dip Galvanizing

Cast iron substrates were dipped in a molten zinc bath (around 450°C) using a commercial process, resulting in a zinc coating thickness about 100 μm on the substrates.

3.1.5 Acidic Zinc Electroplating

An available commercial electrodeposition process was used to deposit $10\mu m$ Zinc coating on the cast iron surface. This procedure required an electrolytic cell which consisted of a metal used for plating (anode) which in this case was zinc, an object to be electroplated (cathode), immersed in an electrolytic solution. This aqueous solution contained metals ions (zinc ions). When a direct current was passed through the solution, zinc ions started dissolving into the electrolyte and traveling toward cast iron (cathode). Finally, by attaching zinc ions to the surface of the cathode, a zinc coating was formed [35].

3.1.6 Hot-dip Aluminizing

Cast iron inserts were firstly cleaned and then dipped into Al-7Si melt at $730^{\circ}C$ for 5, 10, and 20min in order to achieve an optimum dipping time with a continuous aluminum coating on samples. Based on the obtained result, a dipping time of 30min was selected for surface treatment before casting.

3.1.7 Thermal Spraying

An Al-12Si alloy was applied on the cast iron substrate using a commercial available wire-arc thermal spraying method. In this process, two Al-12Si wires with electrical opposed charged were fed in a manner that a controlled arc occurred at the intersection, resulting in heating up and subsequent melting of the coating material. Molten material at the wire tip firstly was atomized and then deposited onto a prepared substrate using compressed air [36].

3.2 Surface Roughness Analysis

In order to study the possible correlation between surface roughness of the inserts and the bonding strength of the components, different surface roughness parameters such as S_a , S_{10Z} , and S_{dr} was measured using Sensofar optical profilometer instrument. The S_a (arithmetical mean height of a surface) which usually is reported as an absolute value, demonstrates the height difference of each point compared to the arithmetical mean of the surface. In other words, in this method, the height changes of all surfaces are evaluated. On the other hand, in S_{10Z} (ten-point mean roughness) only indicates the sum of the mean value for the five largest peaks and the mean value for the pit depth of the five deepest valleys of a defined surface. Finally, S_{dr} (developed interfacial area ratio) expresses the percentages of additional surface areas that are associated with the texture of a sample as opposed to the planar area. The S_{dr} of a completely flat surface is considered as zero. The raw data then were analyzed with Mountainsmap commercial software.

3.3 Compound Casting

60 kg Al-7Si ingots were placed in an electrical resistance furnace to make molten aluminum. Refining and degassing with Nitrogen gas was performed as the molten aluminum reached 750°C, then the slag was removed. Each cast iron substrate was pre-heated at 300°C and immediately was placed inside a pre-heated mold at 380°C. Finally, the melt was poured onto the cast iron inserts at 730°C and solidified in ambient temperature for 2 min. The experimental set up for compound casting was shown in Figure 3.1.

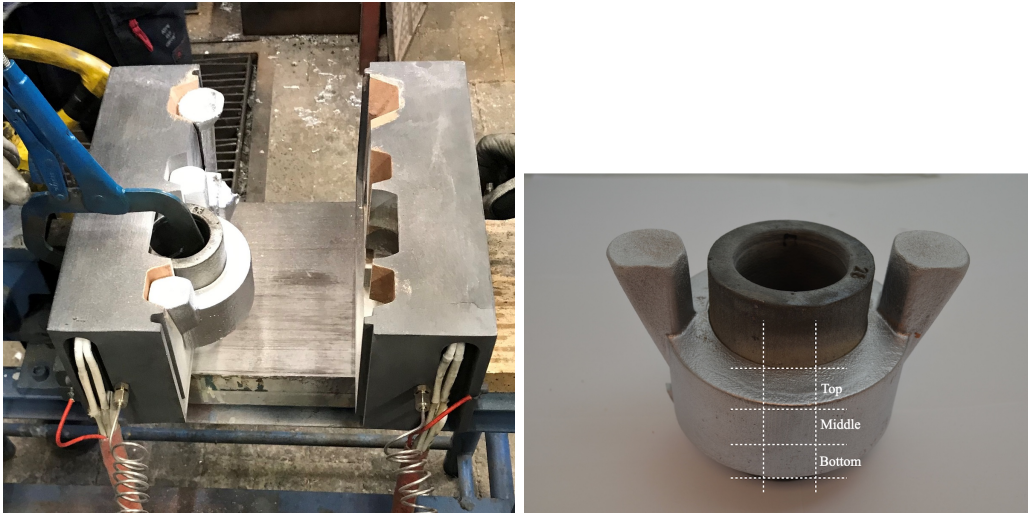


Figure 3.1: Compound casting mold and final cast component.

3.4 Sample Preparation

All samples were cut from the same side of the cast component. Hot mounting was used for the uncoated samples while coated specimens were cold mounted with epoxy not to affect the cast/coating microstructure during hot mounting. Since the sample size for cold mounted specimens was limited to 30 μ m, three samples from the top, middle, and bottom part were mounted to cover the whole interaction zone as shown in Figure 3.1. Sample preparation for optical and SEM microscope was done based on Struers sample preparation guidelines for cast iron and aluminum. In this regard, all specimens were ground with silicon carbide paper starting from 320 grit up to 1000 grit. After that samples were polished with 9 μ m, 3 μ m, and 1 μ m diamond on MD_Largo, MD_Dac, and MD_Nap, respectively. Finally, polished samples were immersed in ethanol and were placed in an ultrasonic bath for removing the residual diamond particles from the polishing stage.

3.5 Optical Microscope Analysis

A Leica DFC420 optical microscope was used to study the interaction between the coating material and the cast iron substrate before and after casting as well as

formed metallurgical bonding at the interface. A set of different magnification from 5x to 500x was applied for taking optical micrographs.

3.6 Scanning Electron Microscope Analysis

The interfacial microstructure of coated samples was analyzed using a JSM-7800F field emission scanning electron microscope. The energy dispersive spectrometer was also used to study and characterize the interface and the formed reaction layer between aluminum and cast iron.

3.7 Push-out testing

The bonding strength of the compound cast structures was examined using a push-out test. The cast components were placed on a steel plate and pushed by the help of a steel punch with a displacement rate of $1\text{mm}/\text{min}$ to achieve a maximum load. Figure 3.2 shows the experimental set up for the push-out test. In order to minimize errors in the experiment three samples were used.



Figure 3.2: Experimental set up for push-out test.

3.8 Micro-Hardness testing

The micro-hardness profile was performed along the reaction zone of the Al/Fe interface by applying a load of 100g using a micro-Vickers indenter and 10s dwell time.

4

Results and Discussion

4.1 Uncoated Samples

In addition to the reference sample, three other specimens were cleaned, etched and blasted as it was described in the method section. Specimens then were pre-heated at 300°C and were placed in a mold for gravity casting. To study the interface between the cast iron and the aluminum after casting, samples were cut for sample preparation, however, it turned out that no metallurgical bonding has occurred between Al and Fe since the aluminum side of the sample easily fell apart after cutting.

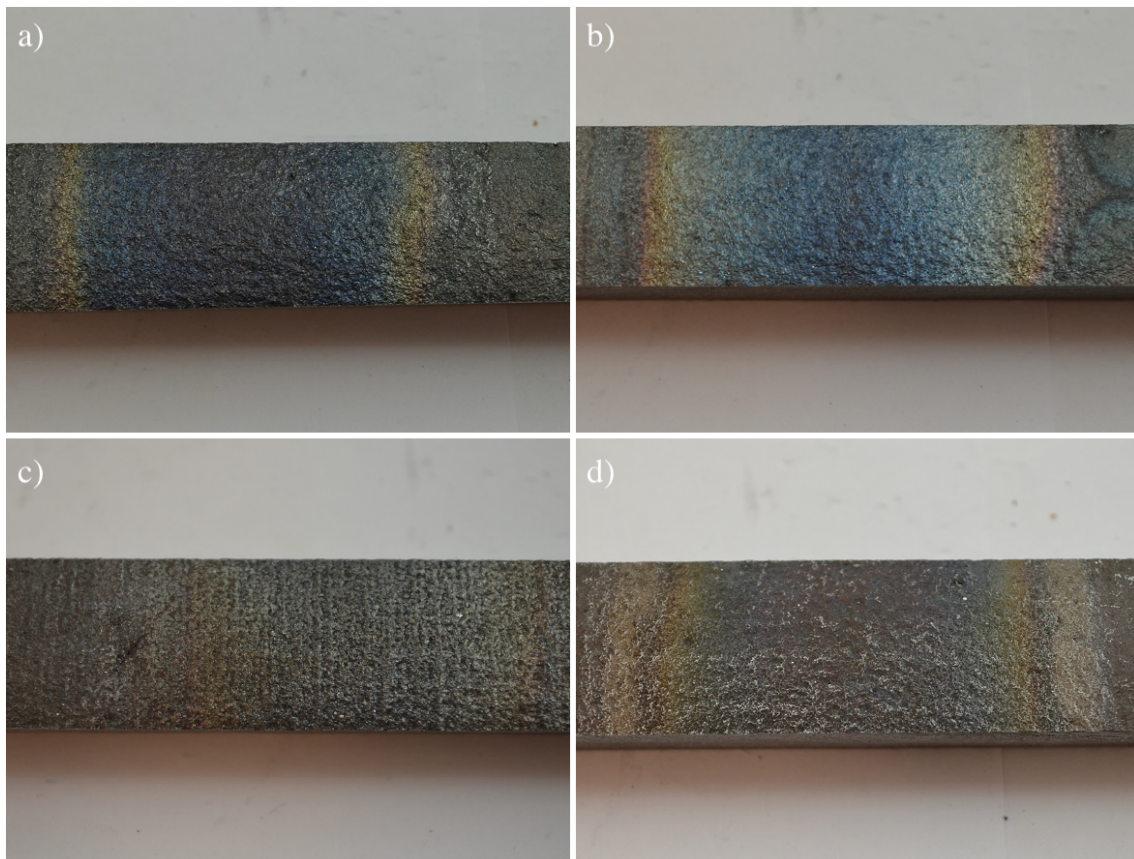


Figure 4.1: The cast iron surface after cutting for : a) reference, b) cleaned, c) etched, and d) blasted samples.

A representative image of the cast iron side of uncoated samples after cutting was depicted in Figure 4.1. It can be seen that a colorful profile was formed at the contact zone of the insert and the liquid aluminum during casting. This colorful profile was probably ascribed to the fact that the inserts were subjected to a high temperature air atmosphere for pre-heating, causing the formation and growth of a thick oxide layer above the cast iron substrate.

The microstructure of the uncoated samples before and after casting were shown in Figure 4.2 to Figure 4.5. It can be observed that the microstructure remained intact during the solidification and there was no difference at the interface for uncoated samples before and after casting. It can be owing to the fact that the thick formed oxide layer above the cast iron insert hindered the aluminum atoms to reach the substrate. Consequently, the Fe and Al atoms could not diffuse along the interface to form the reaction layer and subsequent metallurgical bonding. The result reported by many other research groups [27, 30, 3, 4, 6, 23, 31, 37] also confirmed that without applying proper coating it is not possible to achieve a sound metallurgical bonding at the interface between the base and molten material.

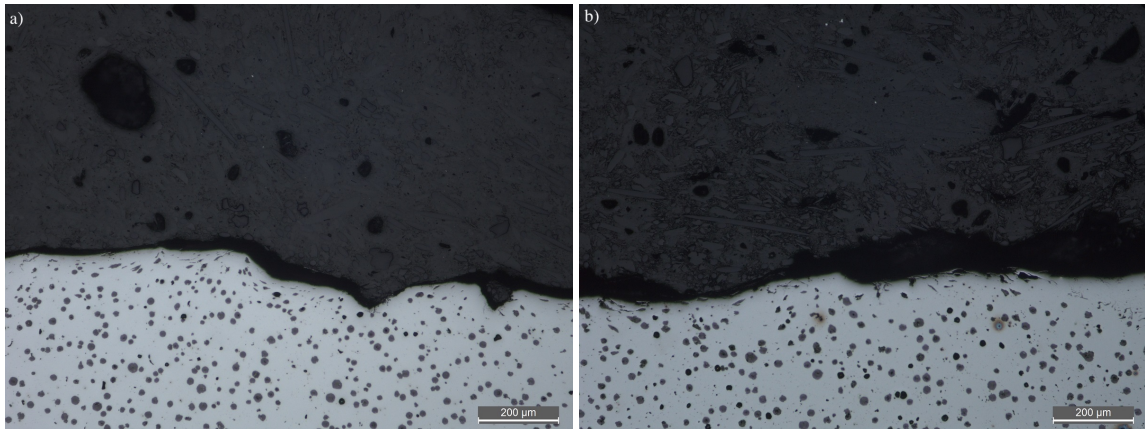


Figure 4.2: Optical micrograph of reference sample: a) before and b) after casting.

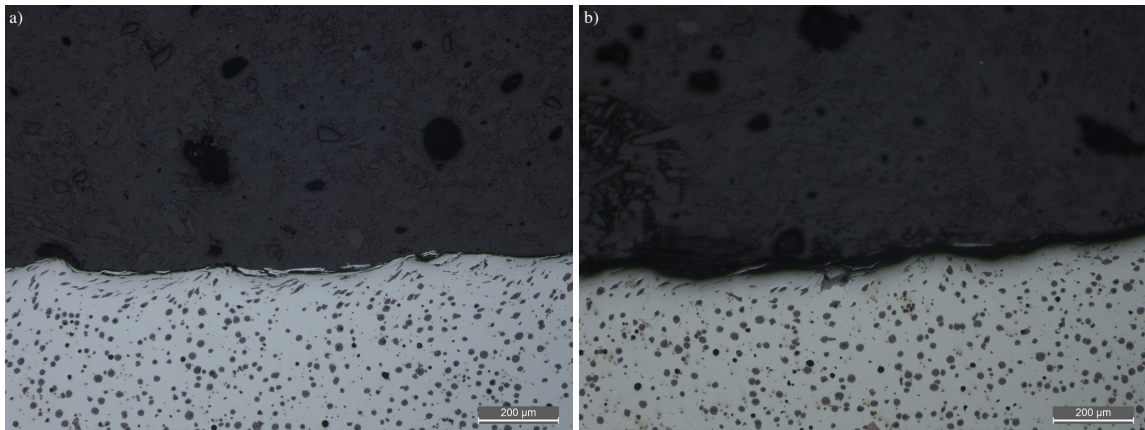


Figure 4.3: Optical micrograph of cleaned sample: a) before and b) after casting.

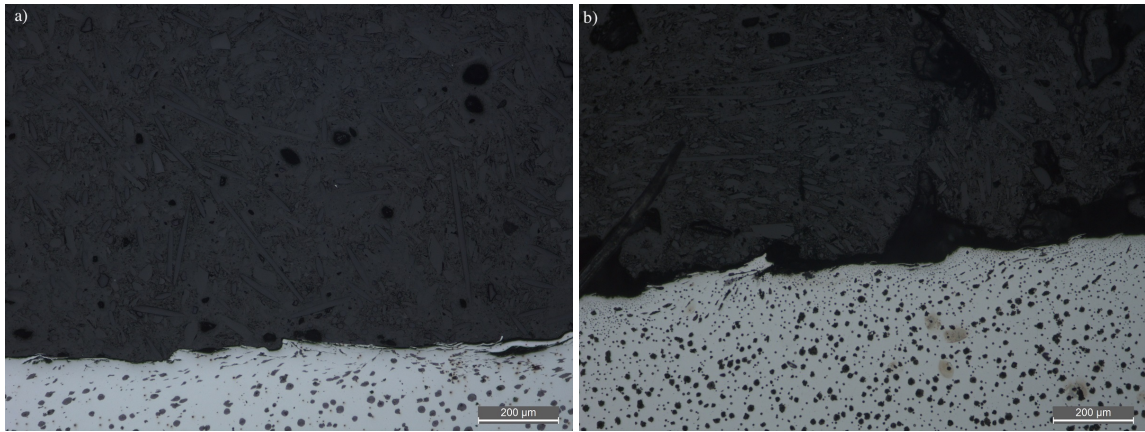


Figure 4.4: Optical micrograph of etched sample: a) before and b) after casting.

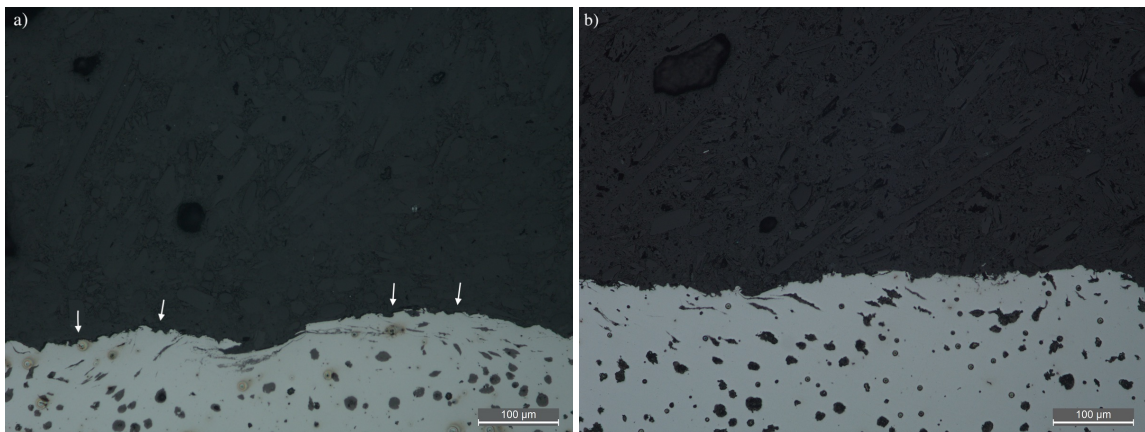


Figure 4.5: Optical micrograph of blasted sample: a) before and b) after casting.

It should be also noted that although all the uncoated samples failed after cutting, there was a difference in the microstructure of the blasted sample compared to other uncoated specimens. As it was shown with white arrows in Figure 4.5.a, the blasted sample had a serrated microstructure as opposed to a rather smoother interface for reference, cleaned, and etched samples. This special microstructure of the blasted samples affected the mechanical properties of the sample as it will be discussed further in the push-out test section.

Based on the visual and the microstructural analysis, it was suggested that the oxide formation during the pre-heating was responsible for the failure of uncoated samples. The aim of this stage was to prolong the solidification time. In more detail, since diffusion is a time and temperature dependant process, by pre-heating the specimens, it takes a longer time for the insert to cool down during solidification, resulting in more time for diffusion reaction and therefore, a higher probability of the reaction layer formation.

As it was observed, the compound casting of the uncoated cast iron insert did not lead to a metallurgical bonding at the interface. Hence, applying a proper

coating material to protect the surface from oxidation seems necessary. Moreover, the coating material is believed to facilitate the wettability of the molten aluminum which is an inevitable requirement for having a compact diffusion zone between the base material and the substrate.

4.2 Coated samples

4.2.1 Hot-dip Galvanizing

Figure 4.6 shows the optical image of the hot-dip galvanized cast iron insert before casting. A zinc coating with a thickness of around $100\mu m$ has been formed during the hot-dipping process. The formation of porosity within the coating material did not allow for a uniform zinc coating above the substrate.

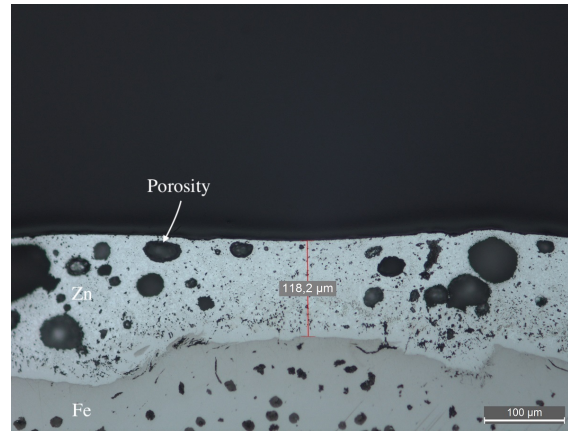


Figure 4.6: Hot-dip galvanized cast iron insert before casting.

To investigate the interaction between the zinc coating and the cast iron, the interface studied by SEM and EDS as it was demonstrated in Figure 4.7. As can be observed, Fe and Zn did not diffuse at the interface between the cast iron and zinc coating which resulted in no intermetallic compound formation at the interface. Hence, it can be concluded that there was not a metallurgical reaction between the substrate and the coating during hot-dip galvanizing of the cast iron which is in good agreement with [30]. They believed that various factors such as alloy composition, the temperature of zinc bath, dipping time, and surface roughness of the insert can affect the formation of the Fe-Zn intermetallic phase.

In addition, Figure 4.7.c indicated that an oxide scale had been built up above zinc coating probably during pre-heating stage where the insert was exposed to elevated temperature for around $45min$.

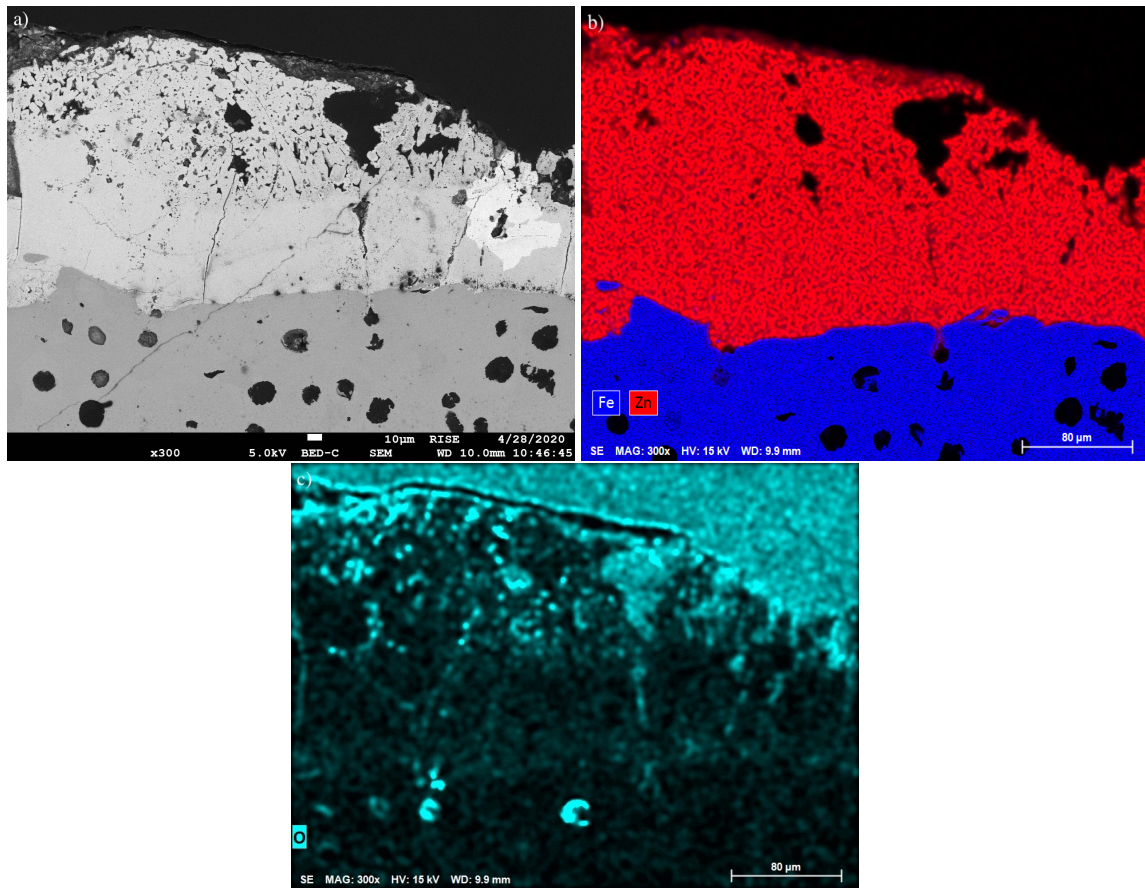


Figure 4.7: BSE image of the interaction between zinc coating and the substrate and its corresponding EDS map.

Figure 4.8 illustrates the interface of the hot-dip galvanized insert after casting. The red dashed region in the bottom part of the sample pointed out to a relatively large gap at the interface between the cast iron and the aluminum. In contrast with other research groups' result for hot-dip galvanized compound cast samples, during cutting of the specimen for sample preparation, the aluminum side fell apart quickly, indicating that there was no bonding between these two materials.

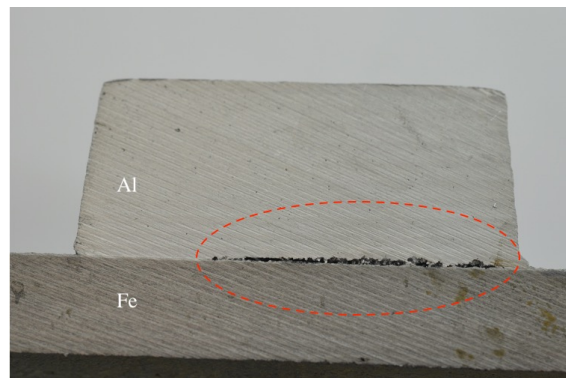


Figure 4.8: Macro-characteristic of the interface between hot-dip galvanized cast iron and the aluminum.

For failure analysis, the cross-section of the hot-dip galvanized sample after casting for the top, middle, and bottom part of the insert was studied by optical microscope, SEM, and EDS. The same procedure was applied to the aluminum side of the sample as well.

4.2.1.1 Cast iron side

The optical images of the substrate after casting were illustrated in Figure 4.9. A comparison between the optical image of the interface before and after casting indicated that the zinc coating in the bottom part of the substrate (Figure 4.9.c) was flushed away during the casting. On the other hand, the coating thickness got thickened in the upper part of the substrate while the middle section remained almost unchanged. Besides, as can be seen in Figure 4.9.a, it seems that a new phase may be formed on top of the zinc coating during the casting process.

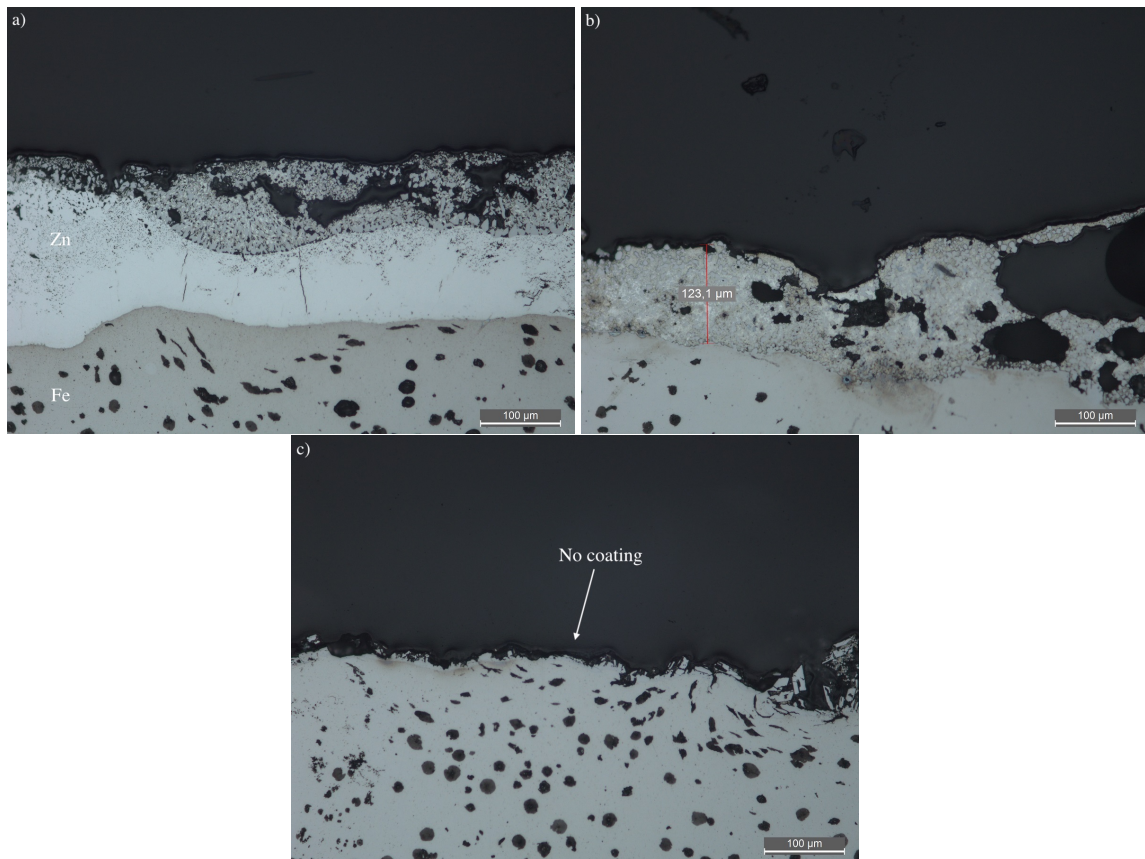


Figure 4.9: Optical micrograph of the hot-dipped cast iron interface after casting: a) top, b) middle, and c) bottom part of the sample.

Figure 4.10 exhibited SEM image and EDS analysis of the upper part of the hot-dip galvanized sample. EDS point scan analysis were presented in Table 4.1. It can be found that two different phase contrasts were present in SEM image which confirmed a newly formed phase during casting. Moreover, EDS point analysis results revealed that both spectrum '1' and '3' in Figure 4.10.b had almost the same composition

4. Results and Discussion

and were made of Zn. Similarly, both spectrum '2' and '4' were also consisted of Al/Fe mixture.

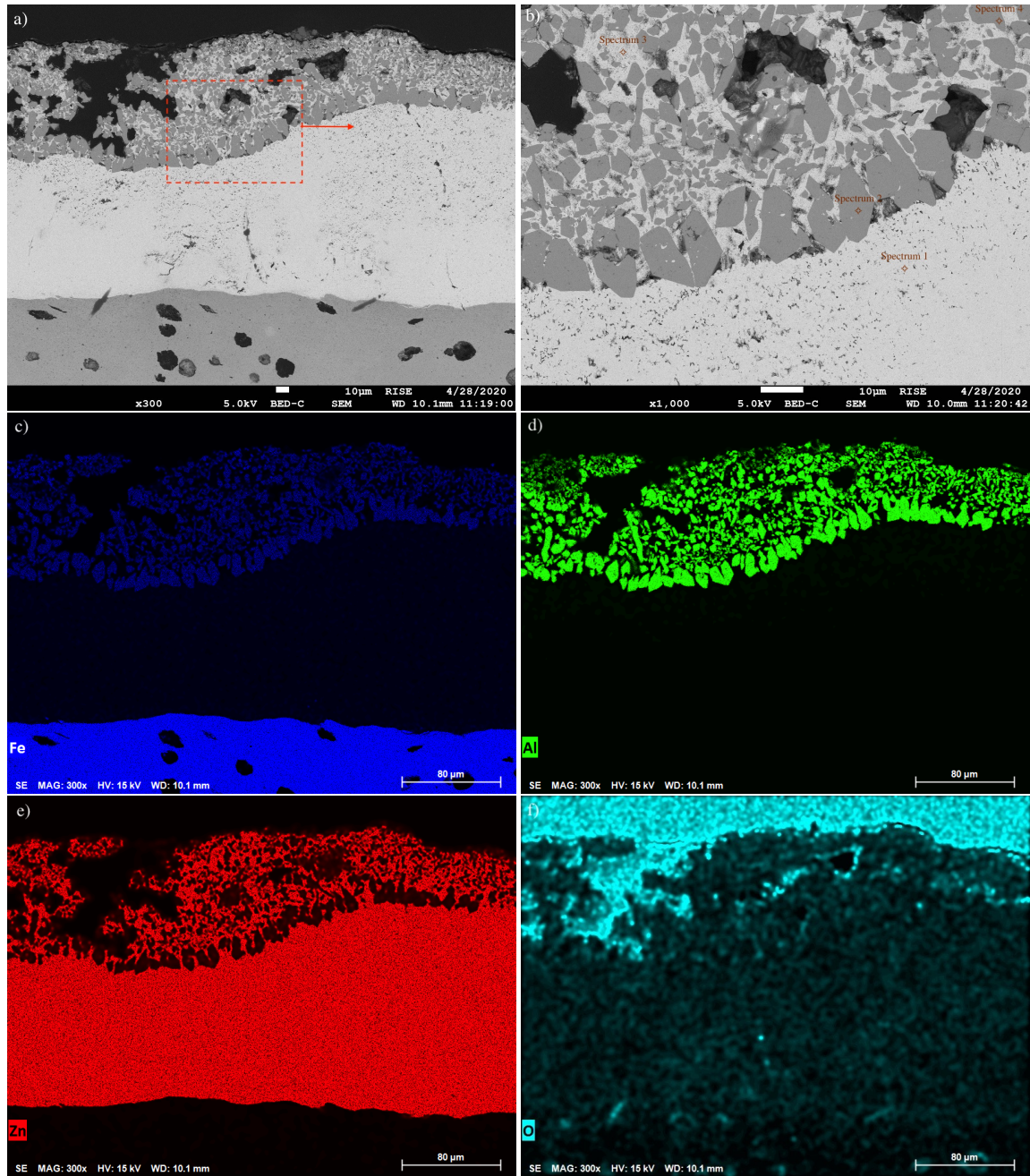


Figure 4.10: a) BSE image of the top section, b) EDS point analysis of the color-coded area, c, d, e, and f) corresponding EDS elemental mapping of (a) for Fe, Al, Zn, and O, respectively.

EDS elemental mapping also confirmed the point analysis results, showing that this new phase consisted of an aluminum/iron in a zinc matrix; however, there was no trace of the Fe diffusion from the base material through the zinc coating up to the newly formed structure. If the iron atoms diffused through the coating material, there should be some iron atoms left in the zinc coating. As a result, this

intermetallic compound was not formed through an interfacial diffusion of the iron and aluminum atoms from the base metal and melt, respectively. Based on Figure 4.7, it should be noted that no iron was found in the zinc coating before casting as well. Consequently, the iron in the newly formed phase could not come from the zinc coating too. Finally, it can be witnessed that the coating material was completely covered by an oxide layer which probably was formed during the pre-heating of the sample.

Table 4.1: EDS analysis of points indicated in Fig 4.10.b.

Number	Elements composition (at.%)		
	Al	Fe	Zn
1	2.6	7.4	88.1
2	59	27.4	12
3	0.4	2.5	96
4	58.5	27.5	12.7

A possible mechanism for Al/Fe intermetallic compound formation in Figure 4.10.a could be explained as follows. During the hot-dip galvanizing process, the cast iron substrate was immersed in a zinc bath at 450°C to reach the proper coating thickness. This temperature was around $0.4T_m$ of the cast iron which was high enough for the stress relaxation, causing a local weak point in the cast iron substrate near the zinc coating. The weak area for this sample was probably located at the bottom part of the cast iron since based on Figure 4.8, a gap was formed in that region where the molten aluminum first came in contact with the substrate at the beginning of the casting process. Consequently, because of the shear stress of the casting, these loose areas with coating on top of them were peeled off. Then, owing to the turbulence of the melt during casting, these regions were moved to the upper part of the sample, leading to an increase in the zinc coating thickness. A small amount of the iron atoms also were subjected to the molten aluminum at 740°C. These relatively cold iron crystals (below 300°C) acted as nucleation sites for the melt aluminum, resulting in a diffusion of Al and Fe and subsequent formation of the Al/Fe equiaxed intermetallic phase above the top part of the insert.

4.2.1.2 Aluminum side

SEM image and EDS point and elemental map scan of the aluminum side of the hot-dip galvanized specimen were depicted in Figure 4.11. EDS point scan of the marked spot in Figure 4.11.b were summarized in Table 4.1. As can be observed in Figure 4.11.a, there was a whitish crystalline material that locally attached to the aluminum. EDS point analysis suggested this phase to be a part of zinc coating that was in good agreement with the EDS elemental mapping data in Figure 4.11.e. These large zinc crystals were formed during the hot-dip galvanizing process and they remained intact during the casting process. A similar structure reported by [30, 37] as well.

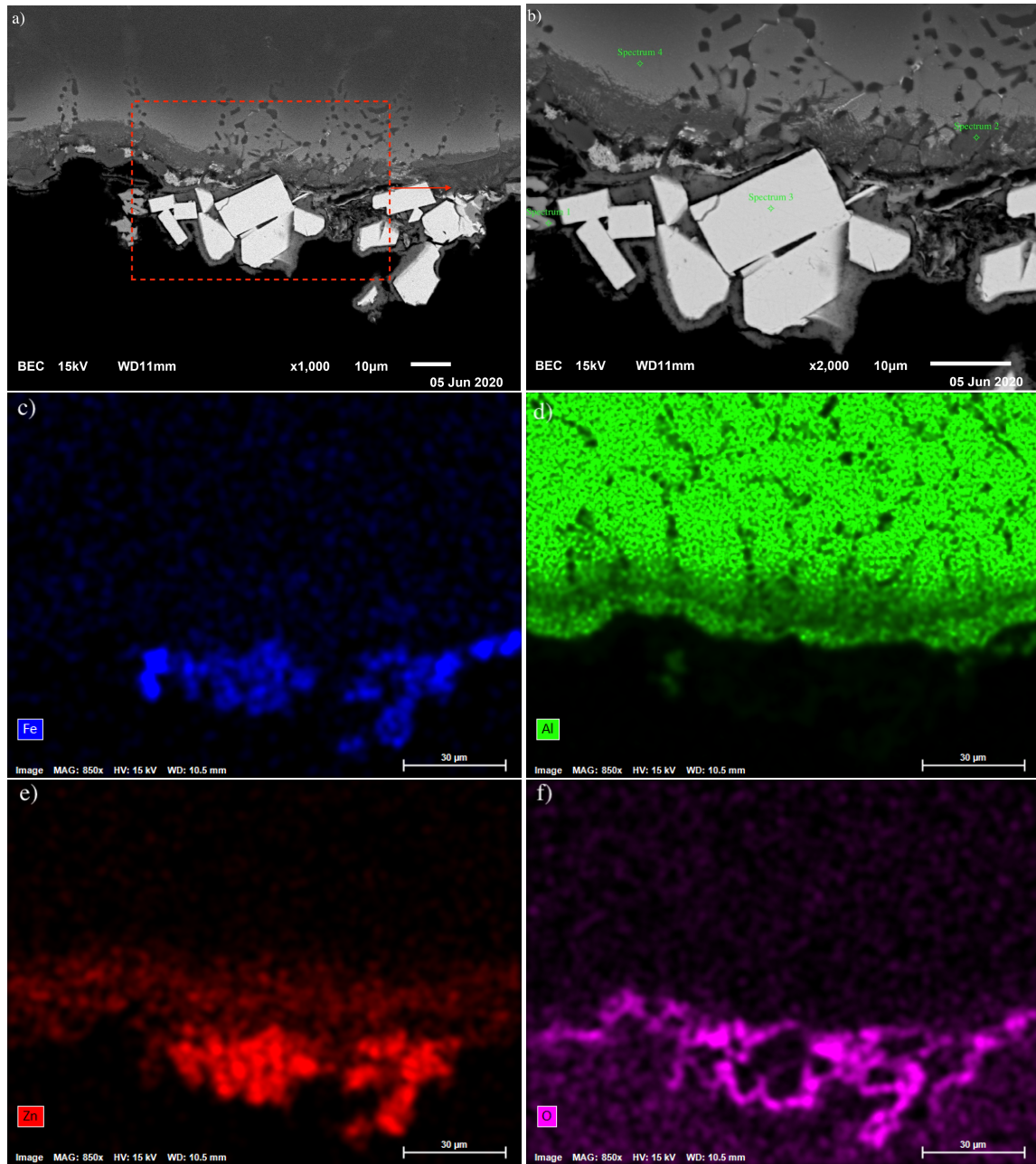


Figure 4.11: a) BSE image of the top section, b) EDS point analysis of the color-coded area, c, d, e, and f) corresponding EDS elemental mapping of (a) for Fe, Al, Zn, and O, respectively.

Furthermore, a slight local diffusion of the zinc into the aluminum can be seen near the interface between Zn crystals and the Al. Moreover, the spectrum "2" in Figure 4.17.b showed almost the same chemical composition as the Al-Fe-Zn intermetallic phase in the upper part of the cast iron side of the sample (spectrum "2" and "4" in Figure 4.10.b), however, there was no trace of the Fe diffusion in the aluminum, indicating that these iron atoms probably reached the interface owing to peeling off the substrate material during casting in a similar manner as the cast iron side of the specimen. Finally, a considerable amount of oxygen (Figure 4.11.f) was detected

at the interface of the aluminum side of the hot-dip galvanized sample which was similar behaviour as the cast iron side.

Table 4.2: EDS analysis of points indicated in Fig 4.11.b.

Number	Elements composition (at.%)		
	Al	Fe	Zn
1	61.7	26.7	11.6
2	93	-	7
3	2.3	7.1	90.5
4	83.7	-	16.1

By analysing the both cast iron and aluminum sides of the hot-dip galvanized sample, it could be suggested that the primary reason for the failure of this specimen was also the formation of the oxide layer on top of the zinc coating. Right before casting, the substrate was subjected to an air atmosphere at elevated temperature for quite a long time (45 min) during pre-heating, causing the development of even thicker oxide layer at the interface, as it was shown in Figures 4.7.c, 4.10.f, and 4.11.f. The zinc coating was supposed to protect the substrate from oxidation and increase the wettability of the aluminum during casting and subsequently the formation of the metallurgical bonding at the interface. However, due to the insufficient energy input of the liquid aluminum, the zinc coating did not melt and therefore it could not play its facilitating role for wettability improvement. Besides, because of the formation of the oxide layer, the molten aluminum could not reach the zinc coating as well as cast iron substrate. Consequently, not only the zinc coating was not beneficial in this case, but it also hindered the diffusion of the Al and Fe, resulting in no metallurgical bonding at the interface.

It seems that for the galvanizing method to be beneficial it is needed to remove the pre-heating stage before casting and also keep the time between galvanizing and casting as short as possible to avoid oxide formation after the surface treatment. In addition, the thickness of the zinc coating might be a bit too thick for this application. As mentioned in the literature, it is better to pre-heat the cast iron inserts before immersing in a zinc bath around 400°C. In this case, the zinc coating on the substrate would be much thinner compared to the thickness of the coating in this experiment. Hence, it would melt easily when aluminum comes in contact with the coating, resulting in better wettability and subsequent adhesion of the cast iron and the molten aluminum.

4.2.2 Zinc Plating

In this surface treatment method, the same zinc material as the hot-dipped sample was used to reach an average zinc coating thickness of $10\mu m$ by electroplating technique. Zinc plating of the cast iron with an alkaline bath is believed to be very difficult or impossible [35]. It is because surface carbon reduces the hydrogen overvoltage, causing hydrogen evolution at the cast iron surface. Consequently,

zinc deposition is delayed or suppressed. Electroplating through a weak acidic electrolyte, on the other hand, keeps hydrogen overvoltage above the standard electrode potential of the zinc, leading to zinc deposition on the cast iron surface [35, 38].

The optical micrograph of the acidic zinc electroplated cast iron before casting was shown in Figure 4.12 at two different magnifications. As can be seen, the zinc material was coated uniformly along with the interface. Moreover, because of the nature of the electroplating method, there was no interaction between the coating and the base material.

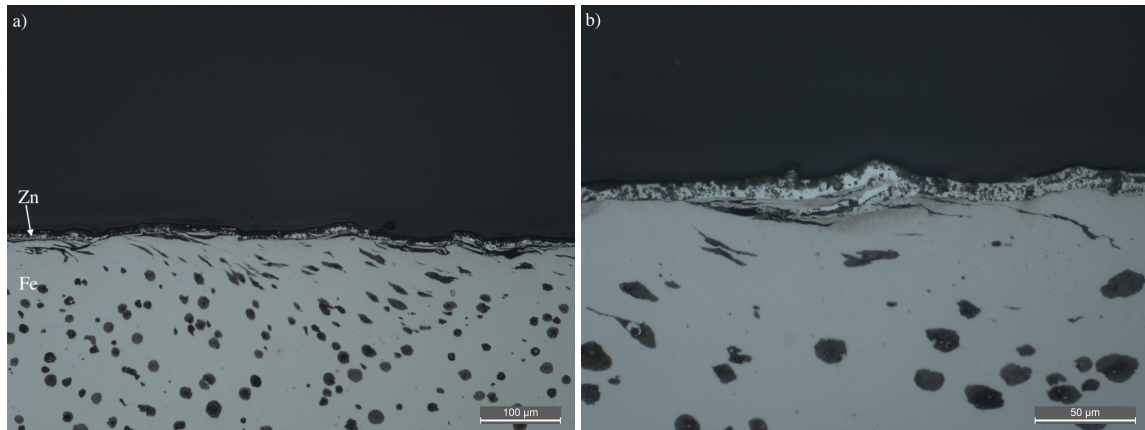


Figure 4.12: Optical micrograph of the acidic zinc electroplated cast iron interface before casting: a) 100x, b) 200x.

The macro image of the interface between the zinc plated cast iron and the aluminum was illustrated in Figure 4.13. It is obvious that in contrast with the interface for hot-dip galvanized samples (Figure 4.8), no visible gap could be detected for this sample. However, after cutting the specimen for the sample preparation, similar to the hot-dipped test, the aluminum side fell apart, showing that no bonding was formed between the melt and the substrate during casting.

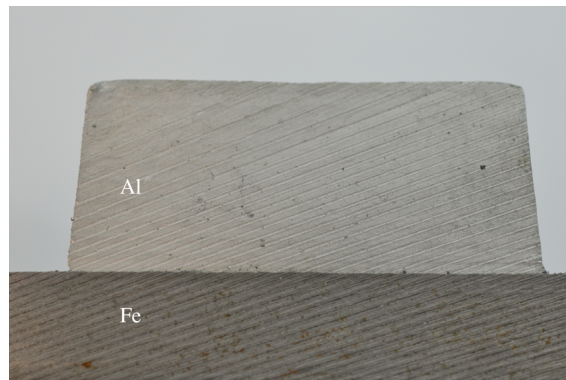


Figure 4.13: Macro-characteristic of interface between acidic zinc electroplated cast iron and the aluminum.

For further failure investigation, the cast iron and aluminum side were studied with the same approach as the hot-dip galvanized sample.

4.2.2.1 Cast iron side

Figure 4.14 shows the optical image of the top, middle, and bottom sections of the zinc plated sample after casting. By comparing the microstructure near the interface with Figure 4.12, it can be seen that the coating material remained almost intact as opposed to the hot-dip galvanized sample. However, some crack initiation within the cast iron insert near the interface can be observed as shown with white arrows in the figure. Similar to the hot-dip galvanizing test, these changes in the microstructure were more pronounced for the middle and bottom part of the sample that can be related to the higher shear stress of the molten aluminum during casting in these areas.

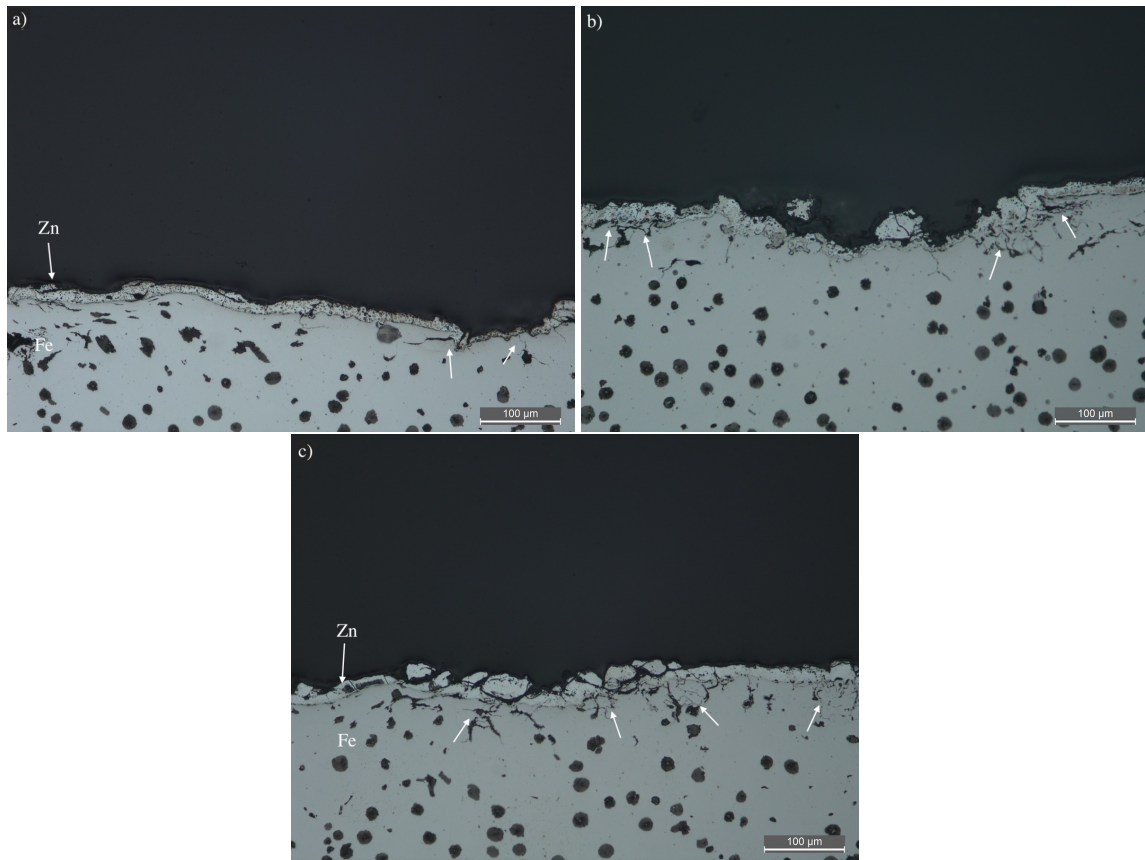


Figure 4.14: Optical micrograph of the zinc electroplated cast iron interface after casting: a) top, b) middle, and c) bottom part of the sample.

The interaction between the zinc plated coating and the substrate after casting was analysed with SEM and EDS as depicted in Figure 4.15. Based on Figure 4.15.a, the zinc coating looks a bit denser than as it was before the casting experiment, however, the thickness remained almost unchanged. Moreover, SEM backscattered image of the red-dashed area at higher magnification and its corresponding EDS elemental mapping (Figure 4.15.a and b) showed that no diffusion of the Fe toward Al occurred during the casting process since there was no trace of iron atoms within zinc coating. The relatively large iron in the zinc coating was probably due to the asperity of the cast iron surface and not related to a diffusion reaction. Also, similar

4. Results and Discussion

to the hot-dip galvanized sample, an oxide layer covered the coating material that probably was developed during the pre-heating stage before casting.

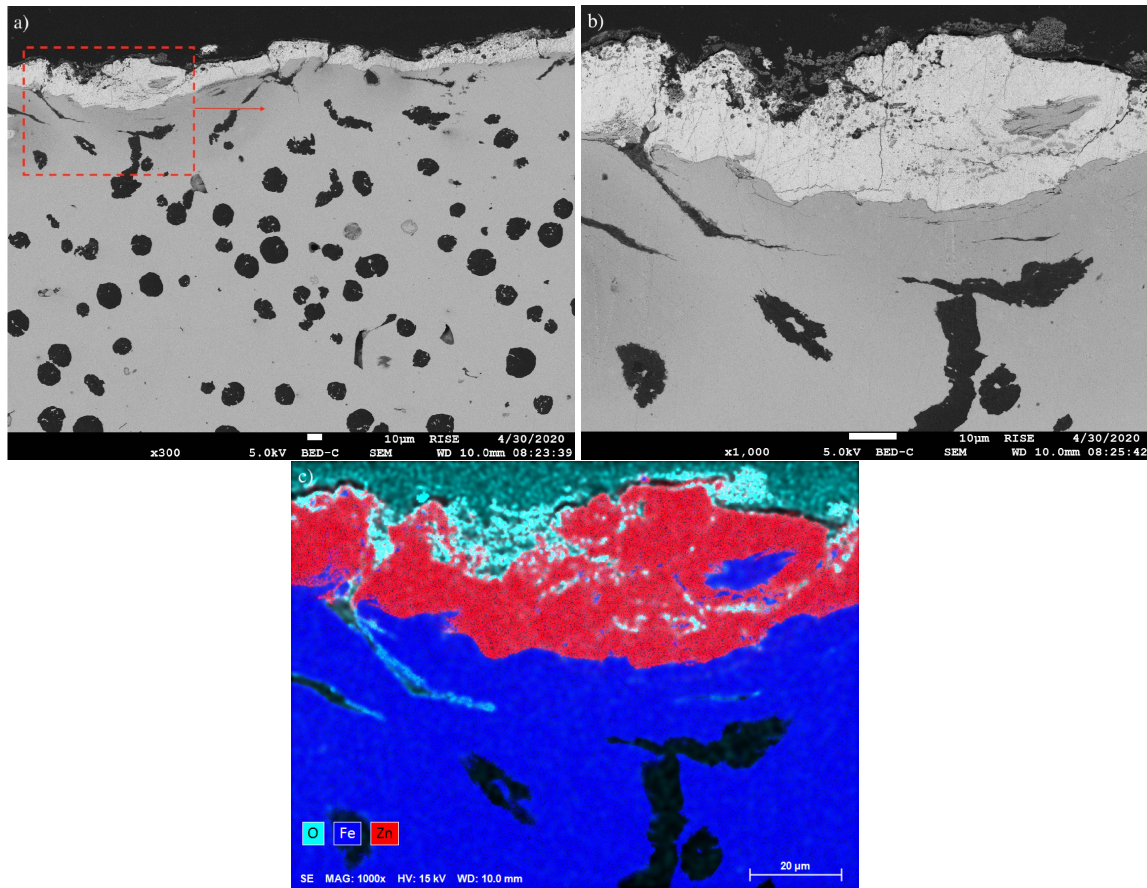


Figure 4.15: a, b) SEM image of the zinc plated cast iron after casting at two magnifications, and c) EDS mapping corresponding to (b).

4.2.2.2 Aluminum side

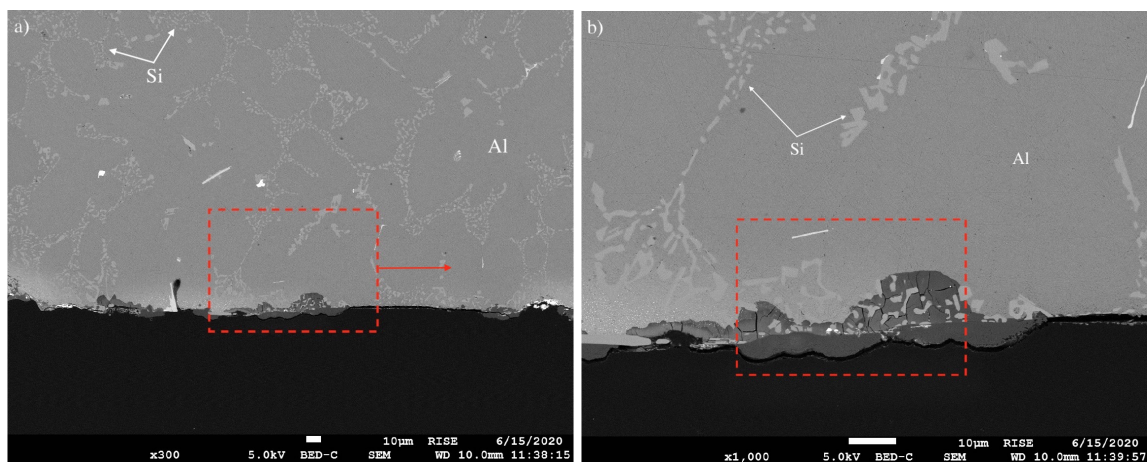


Figure 4.16: BSE image of the aluminum side of the zinc electroplated sample at a) 300x and b) 1000x.

Figure 4.16 represents SEM image of the aluminum side of the zinc electroplated sample at two different magnifications. It can be realized that the majority of the microstructure consisted of an aluminum matrix with dispersed silicon from the eutectic reaction. A very small area at the interface that pointed out with red-dashed rectangular showed a phase contrast in SEM backscattered image which could be a new phase that had been formed during the casting process.

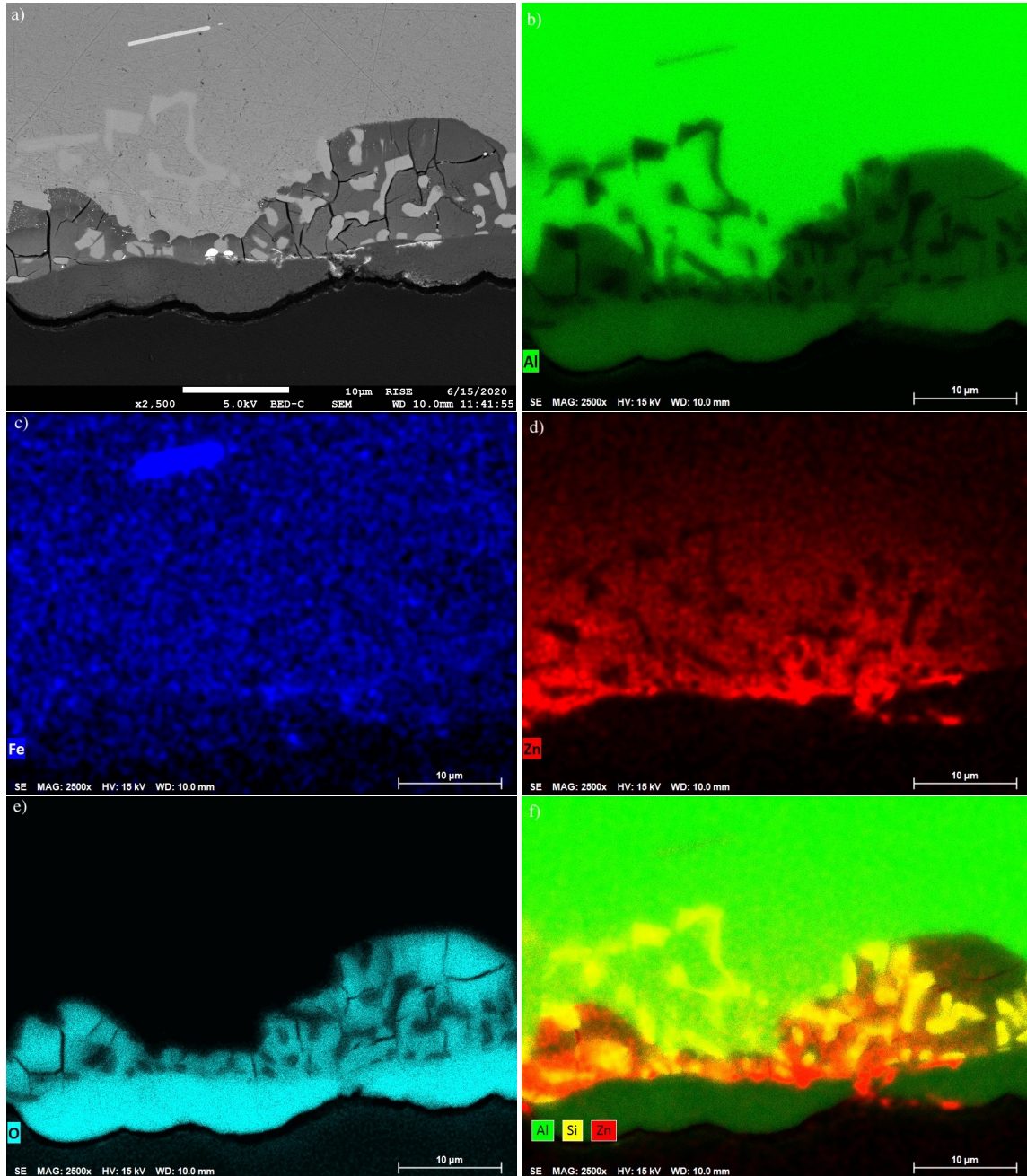


Figure 4.17: a) SEM image of the color-coded area in Figure 4.16.b at 2500, b, c, d, e, and f) corresponding EDS elemental mapping of Al, Fe, Zn, O, and AlSiZn, respectively.

A higher magnification SEM image of the color-coded region in Figure 4.16.b and its corresponding EDS elemental mapping were depicted in Figure 4.17. Based on EDS result, it can be witnessed that the microstructure was composed of a combination of aluminum, zinc, silicon, and aluminum oxide. It seems that the zinc started diffusing to the aluminum locally near the interface between the zinc coating and liquid aluminum (Figure 4.17.d). In addition, by considering both Figures 4.16.b and 4.17.f, it looks that there was a higher concentration of the Si in the determined region in comparison with the surrounding area. This was perhaps due to the diffusion of the Si toward the local contact zone with the substrate down the direction of the chemical concentration gradient. On the other hand, there was no change in the Fe content in this local adhesion point, indicating that Fe atoms could not diffuse to the aluminum side. Thereby, no Al/Fe intermetallic compound was formed at the interface.

Based on the obtained data, it can be observed that zinc electroplating of the cast iron insert did not result in adhesion improvement between the base material and the liquid aluminum. This maybe again owing to the formation of the iron oxide layer on top of the coating during the pre-heating stage as well as developing aluminum oxide during casting (Figures 4.15.c and 4.17.e). These both oxide scales prevented the Al and Fe diffusion which therefore hindered the formation of the metallurgical bonding between the cast iron and the melt which also reported by [27, 39].

The different behaviours of the zinc coating in the hot-dipping experiment compared to electroplating can be related to the nature of these two methods. In contrast with hot-dipping, electroplating was done into an acidic electrolyte by the help of zinc anode and there was no immersing into the melt zinc at high temperature. As a result, the cast iron insert was much less under stress, resulting in no peeling off of the coating during casting, even though the zinc plated sample had much thinner thickness in comparison with hot-dip galvanized specimen.

4.2.3 Hot-dip Aluminizing

To determine the optimum dipping time for the hot-dip aluminized sample, three rings were cut from the un-treated reference specimen, and then were immersed in a molten Al-7Si bath. Afterward, they were retracted from the aluminum bath after 5, 10, and 20min, respectively (Figure A.1). The aluminum coating for the first two samples was easily peeled off. Hence, the dipping time of 20min was selected for hot-dip aluminizing of the cast iron samples for the compound casting experiment.

The optical micrograph of the cross-section of the hot-dip aluminized cast iron interface before casting was shown in Figure 4.18. It can be seen that even after 20min of dipping, the aluminum coating was not formed uniformly on top of the insert and there were some islands of the Al coating on the cast iron. A phase contrast was also observed in the area that the coating was developed, suggesting that a new phase may be formed between the aluminum and the substrate as it was shown with a white arrow in Figure 4.18. In the majority of the interface, however, there was a

clear gap between the coating and the substrate.

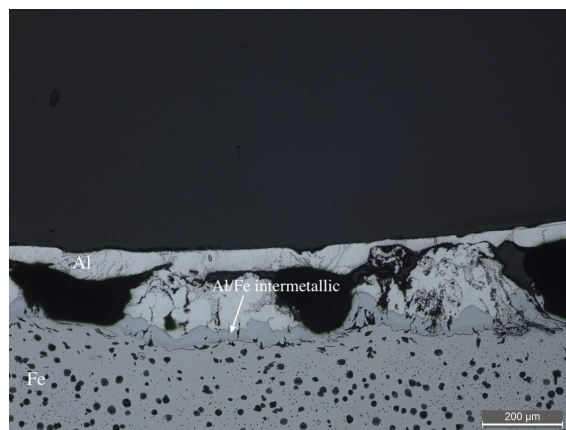


Figure 4.18: Optical micrograph of the hot-dip aluminized cast iron interface before casting.

The aluminum coating was then investigated in more detail with SEM and EDS. Figure 4.19 represents SEM image of the coating layer at two different magnifications and its corresponding EDS elemental mapping. Figure 4.19.b showed that the image contrast in SEM backscattered had changed at the interface between the coating and the cast iron, confirming the formation of a new compound between these two materials. Based on Figures 4.19.c and d, these newly formed phases composed of a ternary AlFeSi structure, indicating that a local metallurgical bonding was developed at the substrate during the hot-dip aluminizing process.

The quantitative results from EDS point analysis of spectrum '1' and '5' in Figure 4.20 showed that the microstructure above the reaction layer mostly was made of an aluminum matrix. Moreover, the highly concentrated silicon area within the aluminum matrix in Figure 4.19.e which corresponds to the spectrum '3' in Figure 4.20, can be related to the eutectic silicon. Spectrum '2' and '4' also were suggesting the formation of a ternary AlFeSi intermetallic phase within the aluminum matrix which was enriched in aluminum. This result was compatible with EDS elemental mapping, showing that the diffusion reaction was not restricted to the interface between the aluminum and cast iron. Finally, there was a gap in the top part of the aluminum coating. This Based on EDS point analysis result for spectrum "6", considerably more oxygen were preset within the gap compared to other marked points. This gap probably was formed during the surface preparation of the sample not because of the hot-dip aluminizing since when the insert was dipped in the Al-7Si bath, no oxide should be present within the melt. EDS elemental mapping for oxygen (Figure 4.19.f) also confirmed that the gap was filled by an oxide scale.

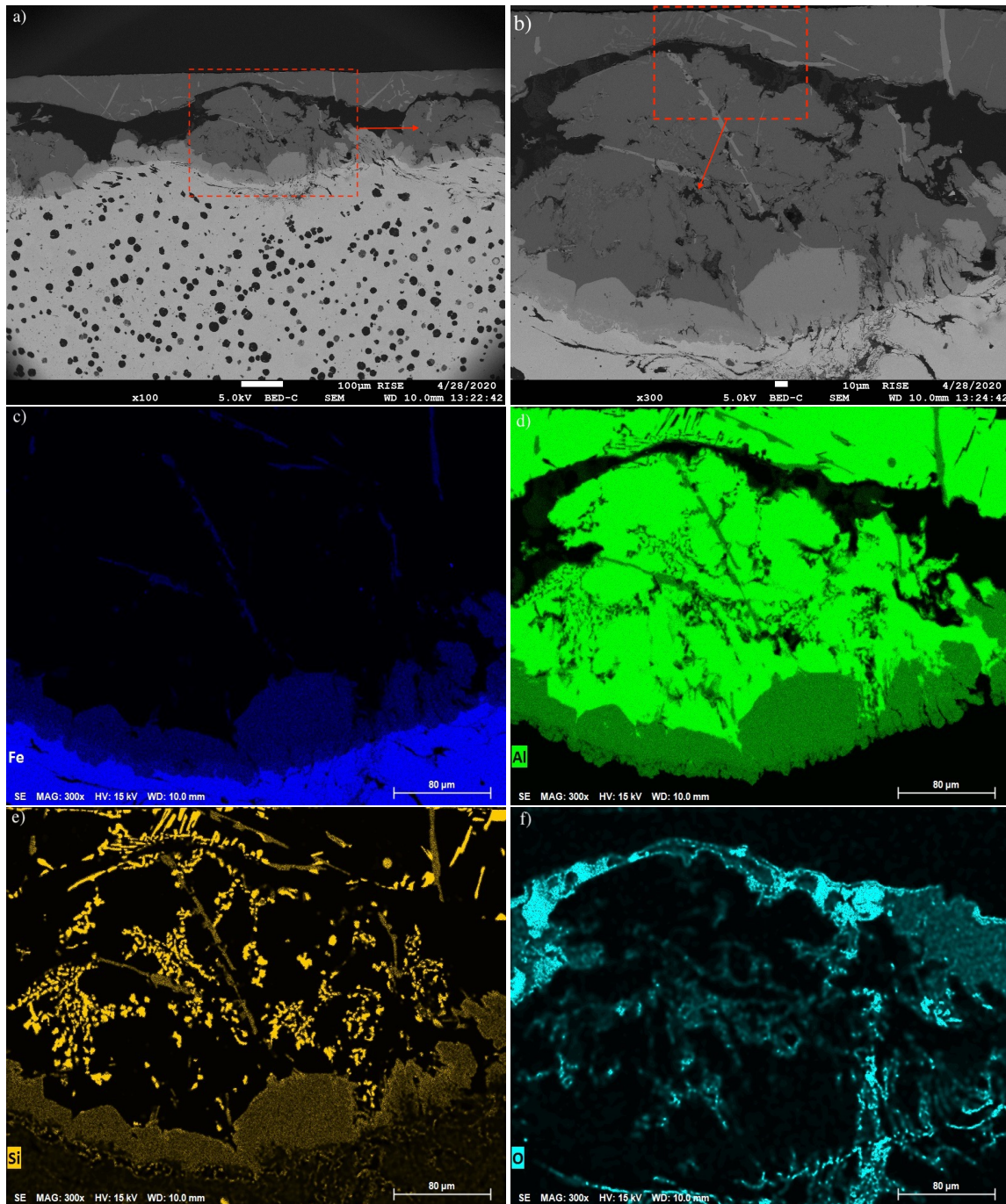


Figure 4.19: a, b) BSE image of the hot-dip aluminized cast iron before casting at 100 and 300x, c, d, e, and f) corresponding EDS elemental mapping of Fe, Al, Si, and O, respectively.

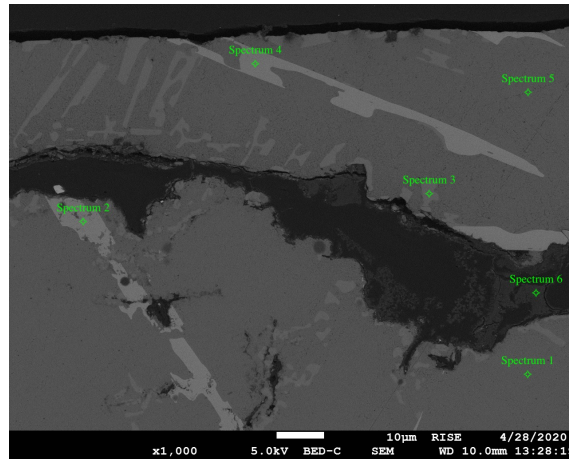


Figure 4.20: point scan analysis corresponding to color-coded region in Figure 4.19.b.

Table 4.3: EDS analysis of points indicated in Fig 4.20.c.

Number	Elements composition (at.%)			
	Al	Fe	Si	O
1	98.5	-	0.9	0.6
2	65.6	16.4	16.2	1.8
3	16.5	-	83.3	0.2
4	63.5	18.6	17	1
5	97.7	-	1.4	0.9
6	40.7	0.5	0.8	58

After hot-dip aluminizing, the sample was pre-heated at 300°C, then was placed in a pre-heated mold and the aluminum was poured around it. Similar to other surface preparation methods, after cutting the cast component for the sample preparation, it was observed that no bonding occurred at the interface of the coating and the substrate and the aluminum fell apart easily.

The microstructure of the hot-dip aluminized cast iron interface after casting for the top, middle, and bottom part of the substrate was depicted in Figure 4.21. It can be found that the previously formed aluminized layer had been removed during the casting. It might be because no compact metallurgical bonding was formed during the hot-dipping experiment. As was observed, there were only some weak local reaction islands between the coating and the substrate while the overwhelming majority of the cast iron insert did not have a coating layer on top. Even in the area where a local adhesion was reached, there was a large gap within the aluminum coating. As a result, during the casting, when the molten aluminum came in contact with the substrate, the weak coating material could not withstand the shear stress of the casting, causing the coating to be flushed away. Hence, the aluminum coating which was supposed to facilitate the wettability of the melt aluminum, and therefore improving the Al/Fe adhesion, could not play its role, resulting in no bonding between the molten aluminum and the cast iron.

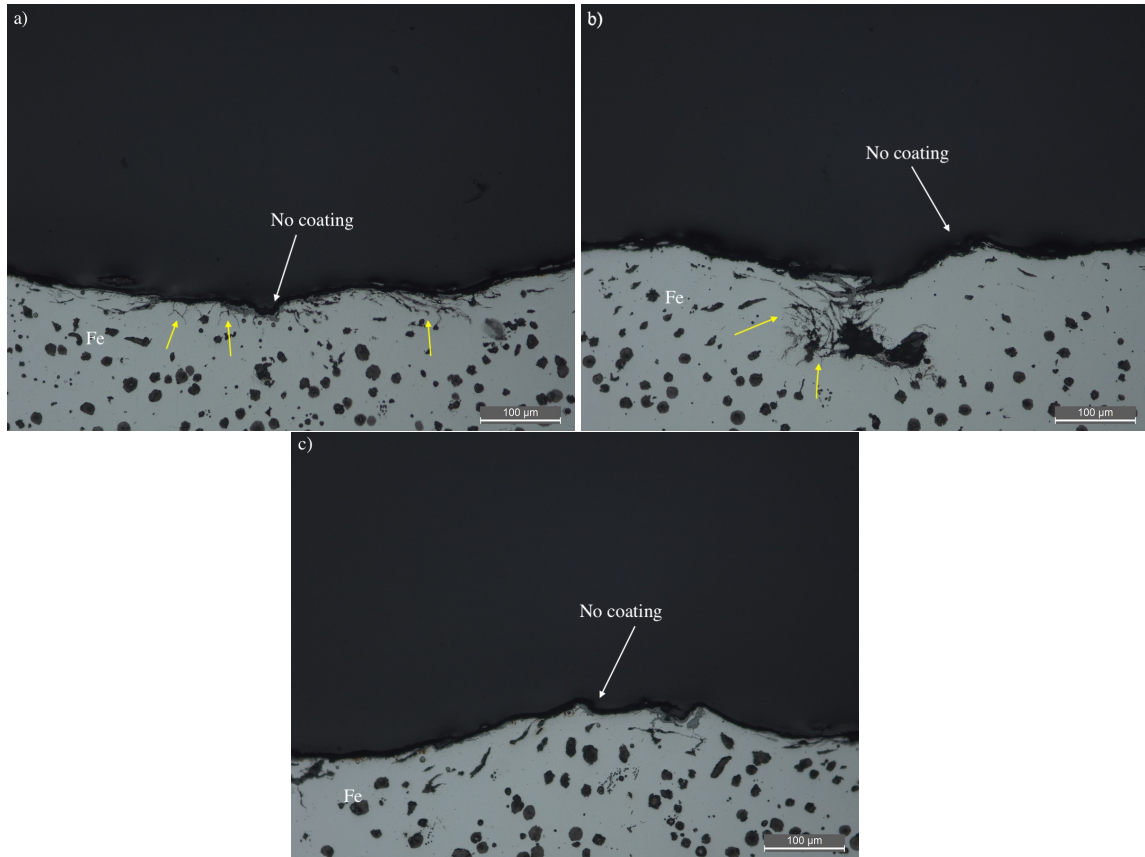


Figure 4.21: Optical micrograph of the hot-dip aluminized cast iron interface after casting: a) top, b) middle, and c) bottom part of the sample.

To address the formation of a poor aluminum coating during the hot-dip aluminizing, the surface condition of the sample should be considered. The reference samples that were used for hot-dip aluminizing were stored in the storage atmosphere for around six months. Consequently, an iron oxide layer had been covered the surface of the samples. Since the aluminum coating layer must form through a diffusion reaction, this oxide layer at the surface hindered the formation of a uniform and compact reaction layer at the interface during the dipping experiment.

Furthermore, during the pre-heating of the coated sample, it was subjected to an air atmosphere at a high temperature which probably resulted in the oxide layer formation above the coating as well. This oxide scale acted as a diffusion barrier, preventing the bonding between Al and Fe. This result was consistent with [40] data. As can be seen from the microstructure after coating, there was completely no interaction between the melt and the cast iron, showing that they did not join at all and the microstructure looks like the microstructure for the uncoated samples.

4.2.4 Thermal Spraying

In this surface treatment technique, the Al-12Si aluminum alloy was deposited on the cast iron throughout a wire arc thermal spraying method. Specimens then were pre-heated and gravity casting was used for pouring molten aluminum around coated samples similar to the previous experiments. After cutting the cast structure

for sample preparation, the aluminum side fell apart from the cast iron, indicating that there was not any sign of adhesion between these two materials.

The micrograph of the interface of the thermal sprayed substrates before and after casting was illustrated in Figure 4.22. Because of the nature of the thermal spraying technique, an irregular interface was formed above the cast iron insert before casting in which two regions were pointed out with red circles (Figure 4.22.a). On the other hand, it can be seen that these irregularities in the interface got flattened during casting in the top, middle and bottom part of the substrate as shown with arrows in Figures 4.22.a to Figure 4.22.d.

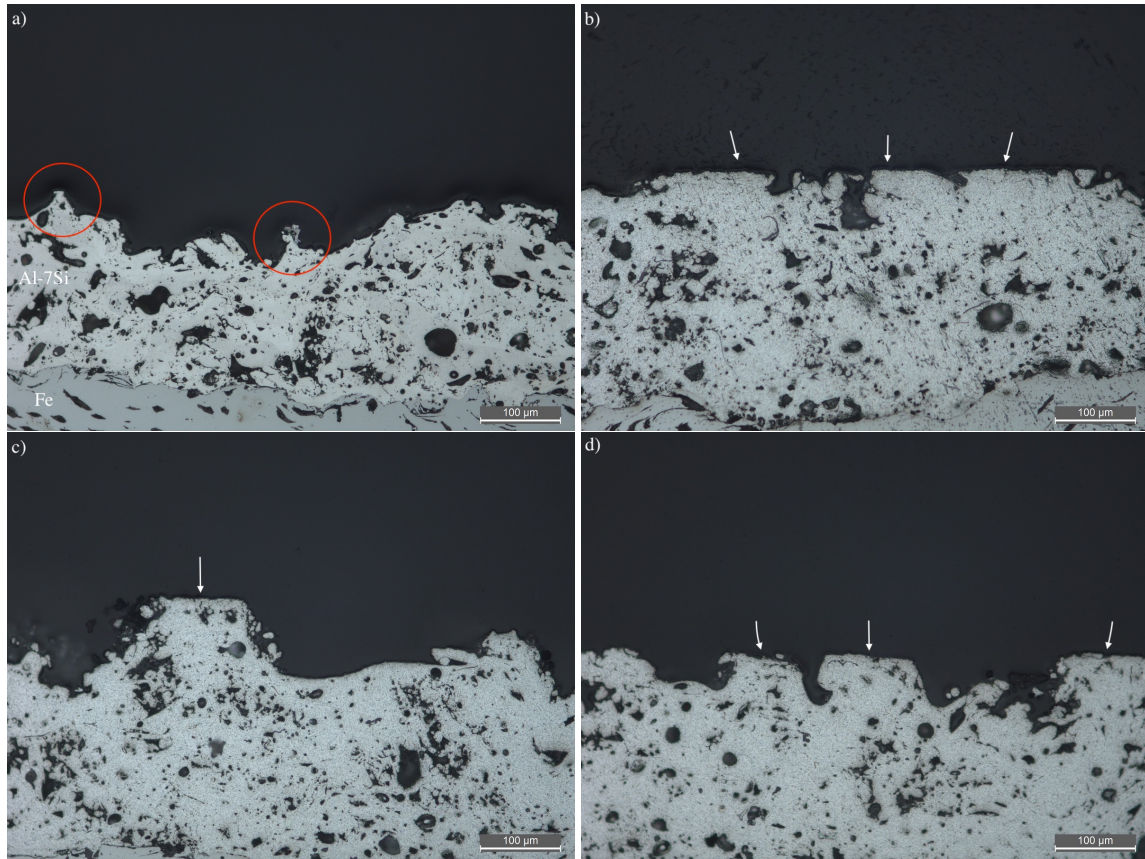


Figure 4.22: Optical micrograph of the thermal sprayed cast iron interface: a) before casting, b) top, c) middle, and d) bottom section after casting.

When the molten aluminum reached the thermal spray coating, the protrusions on the coating were first touched by the melt. The liquid aluminum provided the sufficient energy for the remelting of these regions that stuck out of the coating, resulting in a local adhesion between the molten aluminum and the thermal sprayed coating. This explanation can also be confirmed with the fact that in the vicinity of the flattened regions, the coating became a bit denser in comparison with the rest of the coating which was far from solidification front and therefore contained a large magnitude of porosities. However, these locally bonded regions could not withstand during the cutting of the specimen for sample preparation, leading to breaking apart

of the connection.

Figure 4.23 represents SEM image of the thermal sprayed insert at two different magnifications. It can be observed that no intermetallic compound was formed as a result of Al and Fe diffusion at the interface between the coating and the cast iron. Hence, as it was expected for the thermal sprayed specimens, no metallurgical bonding was formed at the interface. Hence, the substrate and the coating were only mechanically bonded together.

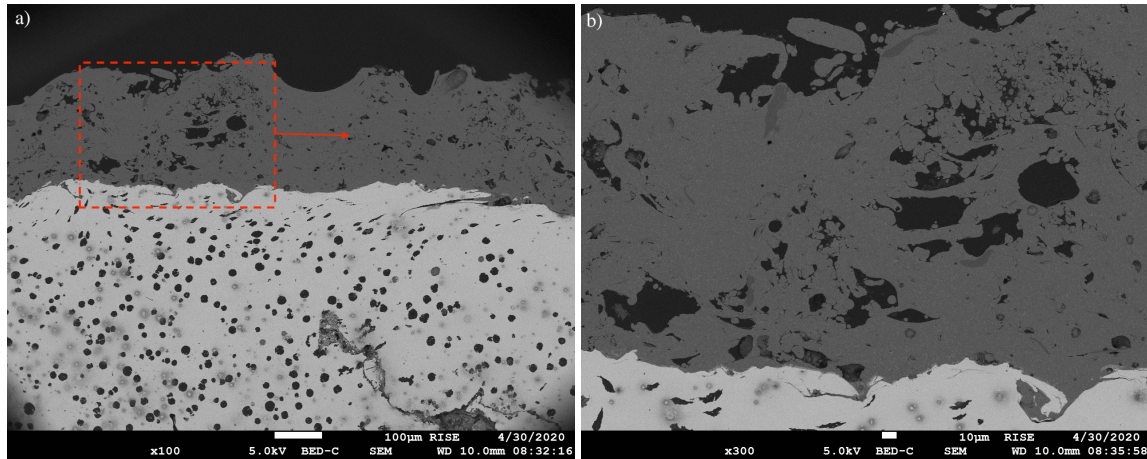


Figure 4.23: BSE image of the thermal sprayed insert at a) 100x and b) 300x.

As it was observed, the thermal sprayed coating was also not efficient for improving the Al/Fe adhesion. It is believed that in a dissimilar joining of the thermally sprayed sample, the strength of the whole component is governed by the mechanical gripping between the coating and the substrate. As a result, unlike other surface treatment methods, the diffusion reaction layer between the thermal spray coating and the melt is only responsible for providing the integrity of the structure. For this experiment, it can be interpreted from the optical image of the microstructure before and after casting that a local adhesion occurred in the protrusion on the coating, however, it was not enough compact and uniform that can tolerate during the stress of cutting.

This failure was perhaps attributed to the fact that, firstly, the Al-Si alloy that was used for thermal spraying had a high affinity for oxidation. Consequently, there was a natural aluminum oxide layer above the coating which could restrict the diffusion between the melt and the coating during solidification. It should be noted that the coated sample was pre-heated before casting which prepared the condition for even more oxide layer growth, causing a severe diffusion block at the interface between the liquid aluminum and the coating.

Secondly, in thermally sprayed specimens, for formation of the diffusion layer, the molten aluminum must be forced to come in contact with the coating. In this experiment, it seems that the gravity casting was not able to provide required pressure for having a reaction layer at the interface. In this regard, High Pressure Die Casting (HPDC) is used in industry for the compound casting of thermally sprayed samples.

4.3 Surface Roughness

To study the effect of surface roughness of the inserts on the interfacial adhesion of the Al/Fe component, different surface parameters such as S_a , S_{10z} , and S_{dr} were measured using a confocal microscope. An example of a surface roughness profile for the thermal sprayed sample before casting was shown in Figure 4.24. The mapping for all other specimens could be found in the appendix (Figure A.2).

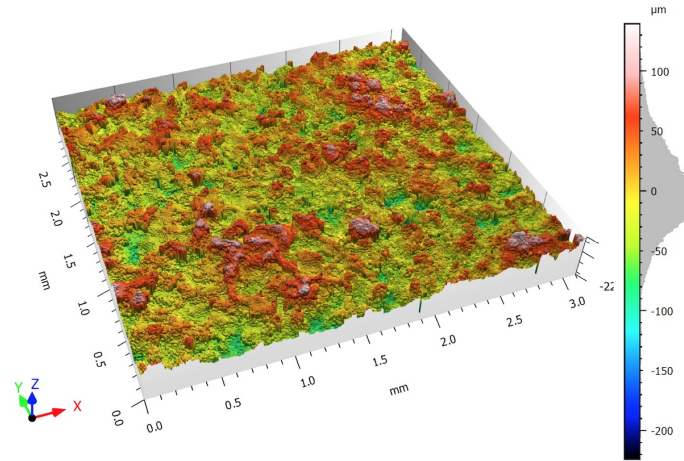


Figure 4.24: Surface roughness mapping for thermal sprayed sample.

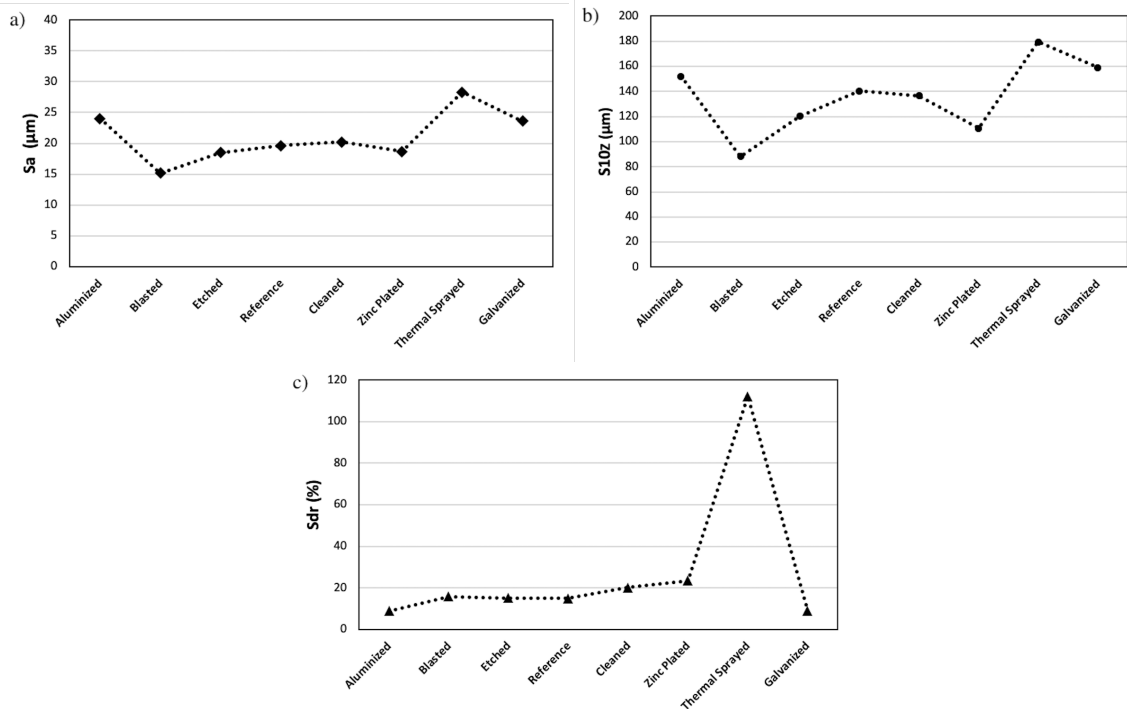


Figure 4.25: Surface roughness parameters of the specimens: a) S_a , b) S_{10z} , and c) S_{dr} .

Figure 4.25 represents three surface roughness parameters of S_a , S_{10_z} , and S_{dr} for all samples which were measured after cast iron surface treatments. Based on Figure 4.25.a and b, both S_a , and S_{10_z} followed the same trend. The blasted sample had the smoothest surface with a minimum value of 18.5 and $88\mu m$ for S_a and S_{10_z} while the roughest surface corresponding to the thermal sprayed specimen with a S_a and S_{10_z} magnitude of 15 and $29\mu m$, respectively. With regard to S_{dr} , it can be found from Figure 4.25.c that the surface area of the thermal sprayed substrate increased by around 112% while the magnitude for all other surface treatment methods stood between 10% to 20%.

4.4 Push-out Test

To evaluate the bonding strength of the interface between the cast iron and aluminum a push-out test was performed. In this test, the maximum load until the failure of the bonding at the interface was measured. Figure 4.26 illustrates shear stress-displacement curves for all samples after the push-out test.

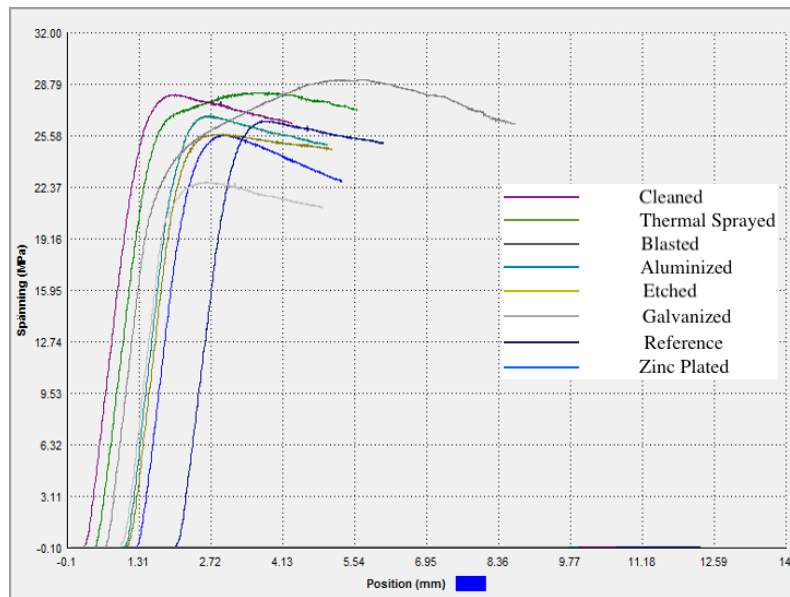


Figure 4.26: Push-out test figure for all specimens.

As can be seen in Figure 4.26, some of the push-out test curves started from zero displacements which were proportional to the first contact of the indenter with the cast iron substrate. Then, the load (stress) increased linearly with the punch displacement since the sample deformed elastically in this stage. After that, a deviation to the linear behaviour occurred, the load reached its maximum value which corresponded to the interface fracture. Afterward, the load dropped gradually, and the resistance to displacement was attributed to the sliding friction.

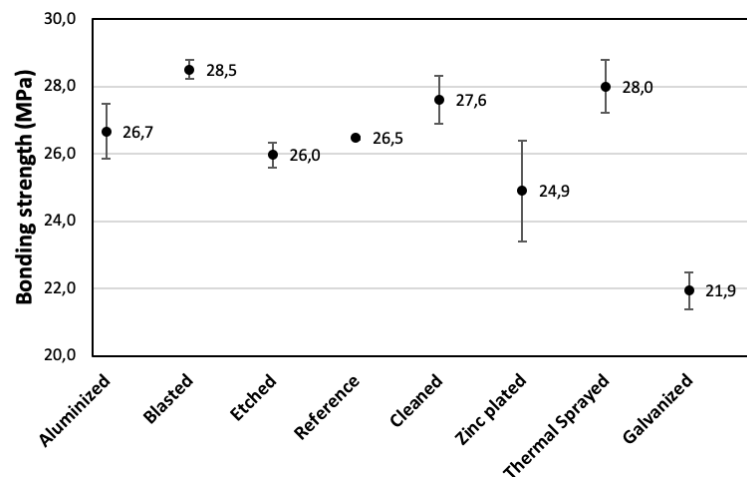


Figure 4.27: Interface shear strength of samples by push out test.

Figure 2.3 plotted the mean value of the bonding strength of the Al/Fe interface which was calculated after three push-out tests for each surface treatment conditions. Based on this figure, there was an overlap in the standard deviation interval of all samples except the hot-dip galvanized. As a result, it was not possible to determine which surface treatment was more beneficial for the adhesion improvement. However, the hot-dip galvanized specimen demonstrated the worst performance followed by the zinc plated sample as the second worst while, there was not a vast differences between the shear strength of the reference sample and all other surface treated substrates. Hence, it can be concluded that the zinc was not a right choice for using as a coating material in this experiment.

This results were consistent with the microstructural analysis of the specimens. As it was observed, no metallurgical bonding was formed during casting for all samples. Hence, there should not be a large variation in the bonding strength as well. In case of the hot-dip galvanized sample, the shear stress of the casting process peeled off a part of the substrate and coating material in the bottom part of the sample, causing in a relatively large gap between the substrate and the aluminum side that can explain the minimum share of the shear strength in this sample.

Furthermore, It can be interpreted from Figure 4.27 that the blasted and thermal sprayed samples showed better performance compared to the reference specimen. Based on Figure 4.25.c, thermal spraying introduced around 90% more interfacial area compared to the reference sample which corresponded to higher possible areas for adhesion between the substrate and molten aluminum. Moreover, the surface roughness profile in Figure 4.24 indicated that roughness was uniformly distributed across the whole area. Hence, there was a large quantity of the local contact zones in the case of the thermal sprayed sample as it was also demonstrated in Figure 4.22.a which needed to be broken before the failure of the interface. Consequently, the push-out test result for the thermal sprayed sample demonstrated a larger shear strength as well as more displacement until failure.

Regarding the blasted sample, there were some irregularities and surface defects after surface treatment which could act as a stress concentration zones during the push-out test, causing the interface failure initiation from these areas. For the blasted sample, these defects on the surface had been removed during sandblasting, leading to a uniform serrated microstructure as it was illustrated in Figure 4.5.a. This was also in good agreement with the results reported in Figure 4.25.a and b, indicating that the blasted sample had the lowest S_a and S_{10Z} values. The serrated microstructure provided a mechanical grip between the substrate and the aluminum. Therefore, during the push-out test for the blasted sample to fail, the whole interface should start sliding which required a higher shear strength as well as more displacement to the failure (Figure 4.26). A similar result also reported by [27].

4.5 New Casting Design

Based on the results obtained from previous surface treatment methods, it was found that the main reason for the failure of the sample was the presence of an oxide layer the cast iron substrate or above the coating during the pre-heating of substrates. Moreover, the push-out test results analysis indicated that there was a relation between the surface roughness of the sample and shear strength of the interface as it was discussed in the previous section. In this regard, a new cast experiment was designed to address these requirements for obtaining a sound adhesion between the cast iron and the aluminum in four steps:

- Sandblasting of the surface
- Removing the remained dust with compressed air followed by rinsing in ethanol
- Hot-dip aluminizing for 30min in the Al-7Si melt at 750°C
- Compound casting of the sample immediately after aluminizing.

In this new casting experiment, a sandblasting surface preparation was used to ensure an oxide-free surface before aluminizing. Also, the pre-heating stage which was believed to be responsible for the oxide formation and growth had been removed. Finally, the casting was performed shortly after the hot-dip aluminizing to minimize the oxide formation as well.

Figure 4.28 demonstrates SEM micrograph of the sample after blasting+aluminizing. As can be seen, a uniform and compact reaction layer was shaped during the hot-dip aluminizing along with the whole interface, indicating that the Al and Fe atoms diffused along the interface, and thereby an intermetallic layer was formed between the iron and aluminum. Moreover, some graphite particles also diffused into the reaction layer as well as the aluminum side (Figure 4.28.a and b) which also reported by [37]. Besides, Figure 4.28.c showed that the reaction layer consisted of three sections as it separated with a white dashed line in the figure. EDS point analysis of the diffusion layer revealed that the outer area, near the aluminum side of the sample, was composed of τ_5 intermetallic compound, an intermediate layer of $FeAl_3$, and the inner layer which consisted of Fe_2Al_5 and eutectic Al-Si. (Figure 4.28.d and

Table 4.4). This intermetallic layer sequence is compatible with the results reported by [10, 11, 12].

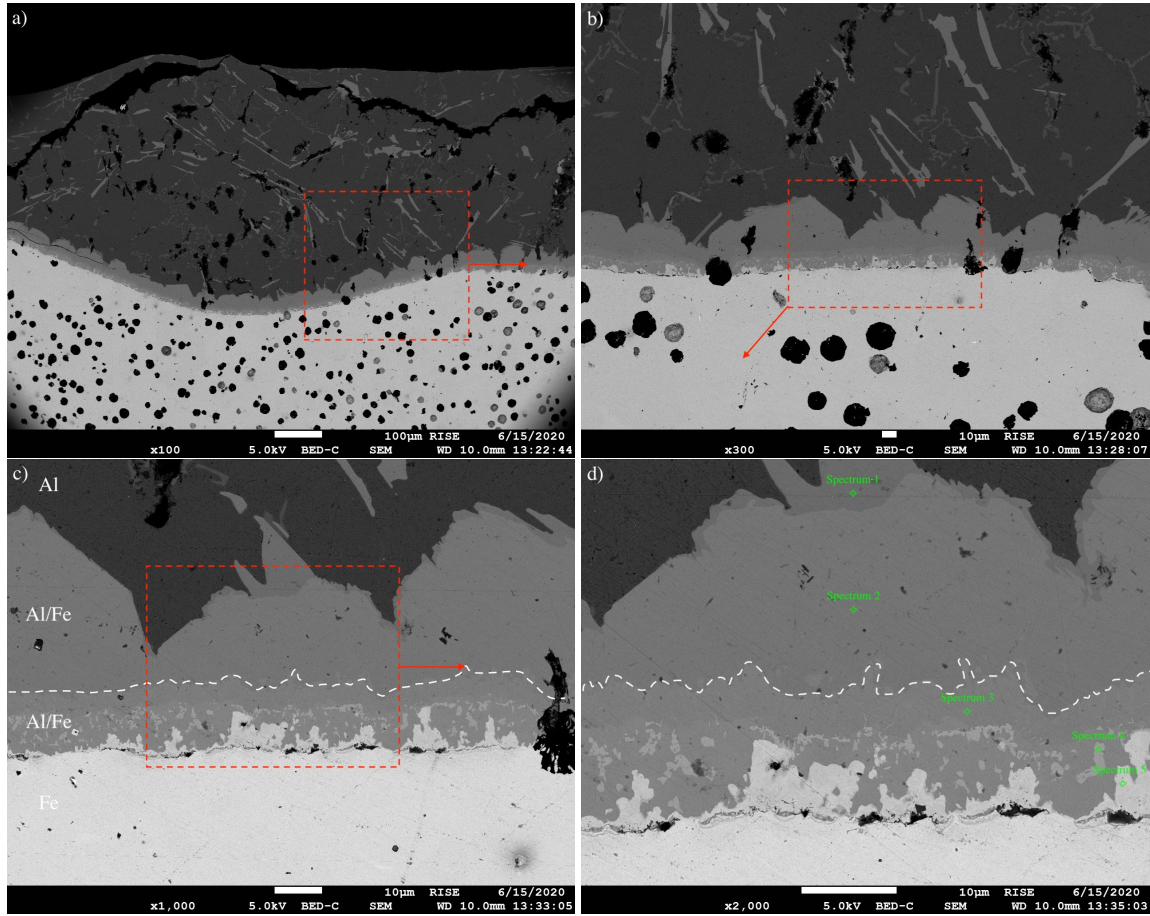


Figure 4.28: BSE image of the cast iron interface after blasting+aluminizing: a) 100x, b) 300x, c) 1000x, and d) 2500x.

Table 4.4: EDS analysis of points indicated in Fig 4.28.d.

Number	Elements composition (at.%)			Inference component
	Al	Fe	Si	
1	64.3	19.1	16.7	$\tau_5 - Al_8Fe_2Si$
2	65.6	23.9	9	$FeAl_3$
3	62.7	4	32	<i>Eutectic Al - Si</i>
4	37.4	41.7	19.4	<i>AlFeSi ternary compound</i>
5	2.3	75	20.3	Fe

After sandblasting and aluminizing of the samples, they were placed in the mold instantly for gravity casting. Then, the cast component was cut for sample preparation and unlike all previous surface treated samples, the substrate, and the solidified aluminum side was stuck together, showing a bonding occurred between these two materials.

The cross-section of the interface at two different regions of the sample was illustrated in Figure 4.29. As can be seen, the microstructure was made of four different sections. The substrate, reaction layer, residual aluminum layer from hot-dip aluminizing, and aluminum layer from casting. To obtain a desirable Al/Fe bonding and integrity of the component, the molten aluminum from the casting should fuse to the residual aluminum on the cast iron insert from the hot-dip aluminizing, forming the whole cast structure. This behaviour can be seen in Figure 4.29.a, while in Figure 4.29.b, there was a gap between the residual and poured aluminum.

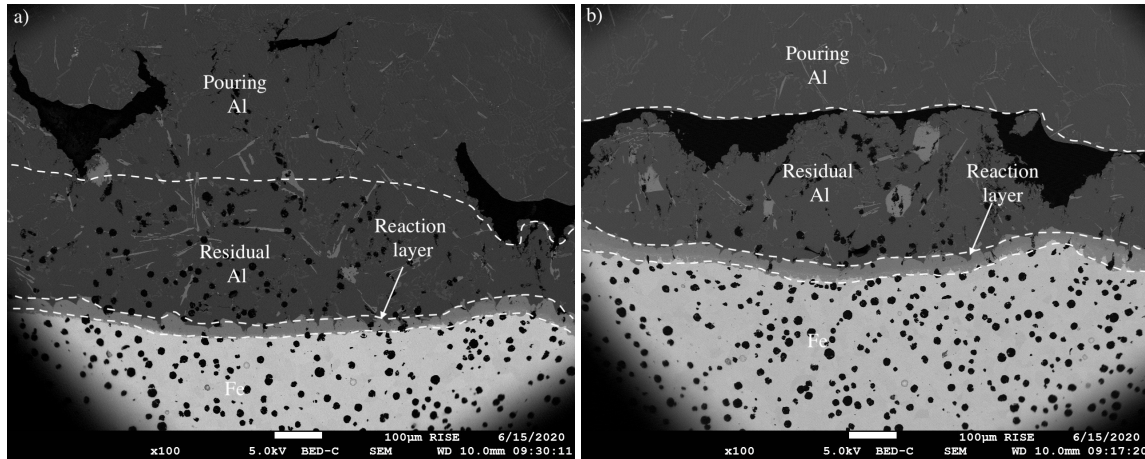


Figure 4.29: BSE image of the blasted+aluminized sample at two different areas.

To address these different behaviours, an EDS line scan was performed across the continuous aluminum layer in Figure 4.29.a as well as the gap in Figure 4.29.b. The results were depicted in Figure 4.30. In the area without a gap, EDS line scan result represented that there was a variation in the mass percentage of the aluminum and silicon which was attributed to the Al-Si eutectic microstructure of the cast component. In more detail, a reduction in the mass percentage of the aluminum corresponded to an increase in the mass quantity of the silicon. The oxygen value, however, remained almost unchanged in the defined line scan, indicating that no oxide was formed within the fusion zone, between the residual aluminum from hot-dip aluminizing and the poured aluminum from casting.

On the other hand, EDS line scan results for the area with gap revealed that the majority of the microstructure consisted of aluminum and a change in the mass percentage of the element only occurred in the vicinity of the gap. It can also be interpreted that at the gap, there was a considerable rise in the oxygen concentration while the aluminum content dropped abruptly, showing the formation of an aluminum oxide layer. As a result, the presence of this oxide layer was prevented the molten and residual aluminum to fuse and create a continuous microstructure. Moreover, because of the interface that was shaped during the hot-dip aluminizing process, it was difficult for Al to wet the residual aluminum during casting. Hence, some air might have been entrapped in the valleys, resulting in a gap between the residual and poured aluminum.

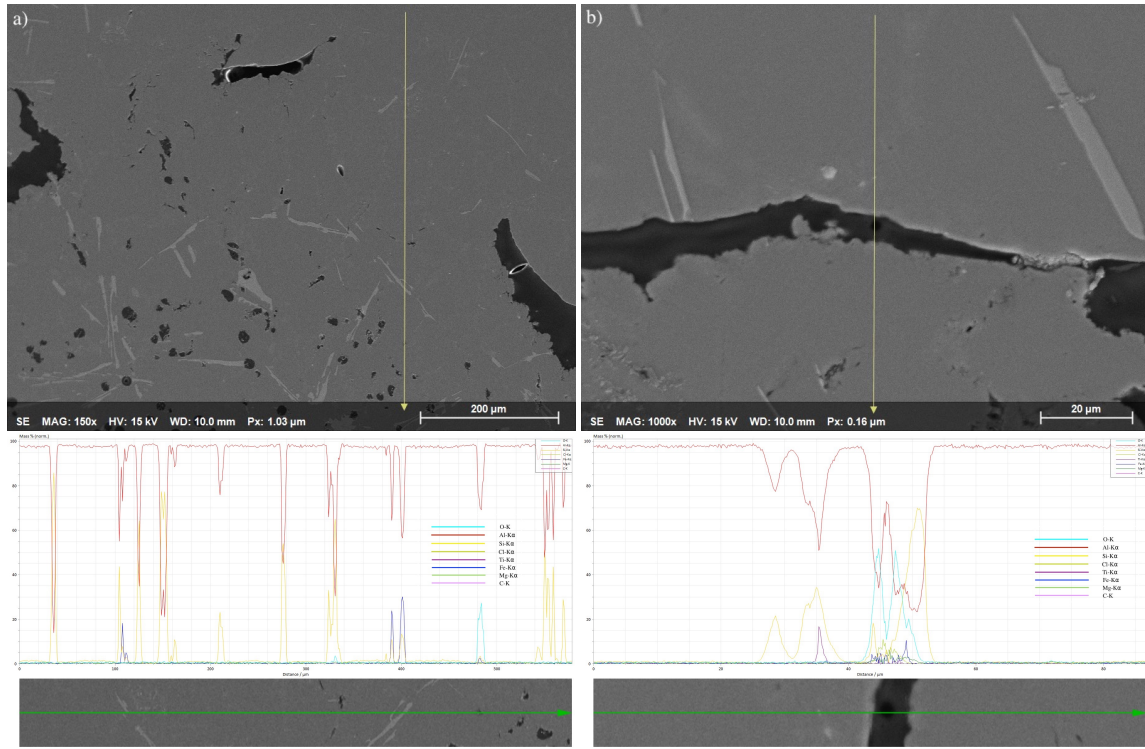


Figure 4.30: BSE image of EDS line scan and its corresponding mass variation result for area: a) without gap and b) with gap.

Figure 4.31 demonstrated SEM images, EDS point, and elemental mapping of the reaction layer of the blasted+aluminized sample after casting. The quantitative results of the point scan EDS analysis for the points marked in Figure 4.31.c were summarized in Table 4.5. It can be seen in Figure 4.31.b that there was an apparent difference in color and morphology of phases that were formed in the Al/Fe reaction layer in different zones, which were consistent with the microstructure in Figure 4.28.d. It was because the intermetallic reaction layer was made during the hot-dip aluminizing and therefore there should not be a difference in the morphology and the chemical composition of the diffusion layer on the cast iron insert before and after casting.

According to Figure 4.31.c and EDS analysis, the inner layer adjacent to the cast iron was suggested to be the Fe_2Al_5 with dispersed islands ternary τ_1 phase. Next to that, there was an intermediate layer of $FeAl_3$, followed by an outer layer of $\tau_5 - Al_8Fe_2Si$ close to the aluminum side which was in good agreement with the result reported by [31]. Hence, these investigations indicated that sequential diffusional reactions occurred at the interface of the cast iron and the aluminum, leading to the formation of a metallurgical reaction layer that included various intermetallic phases of Fe_2Al_5 , $FeAl_3$, and $\tau_5 - Al_8Fe_2Si$.

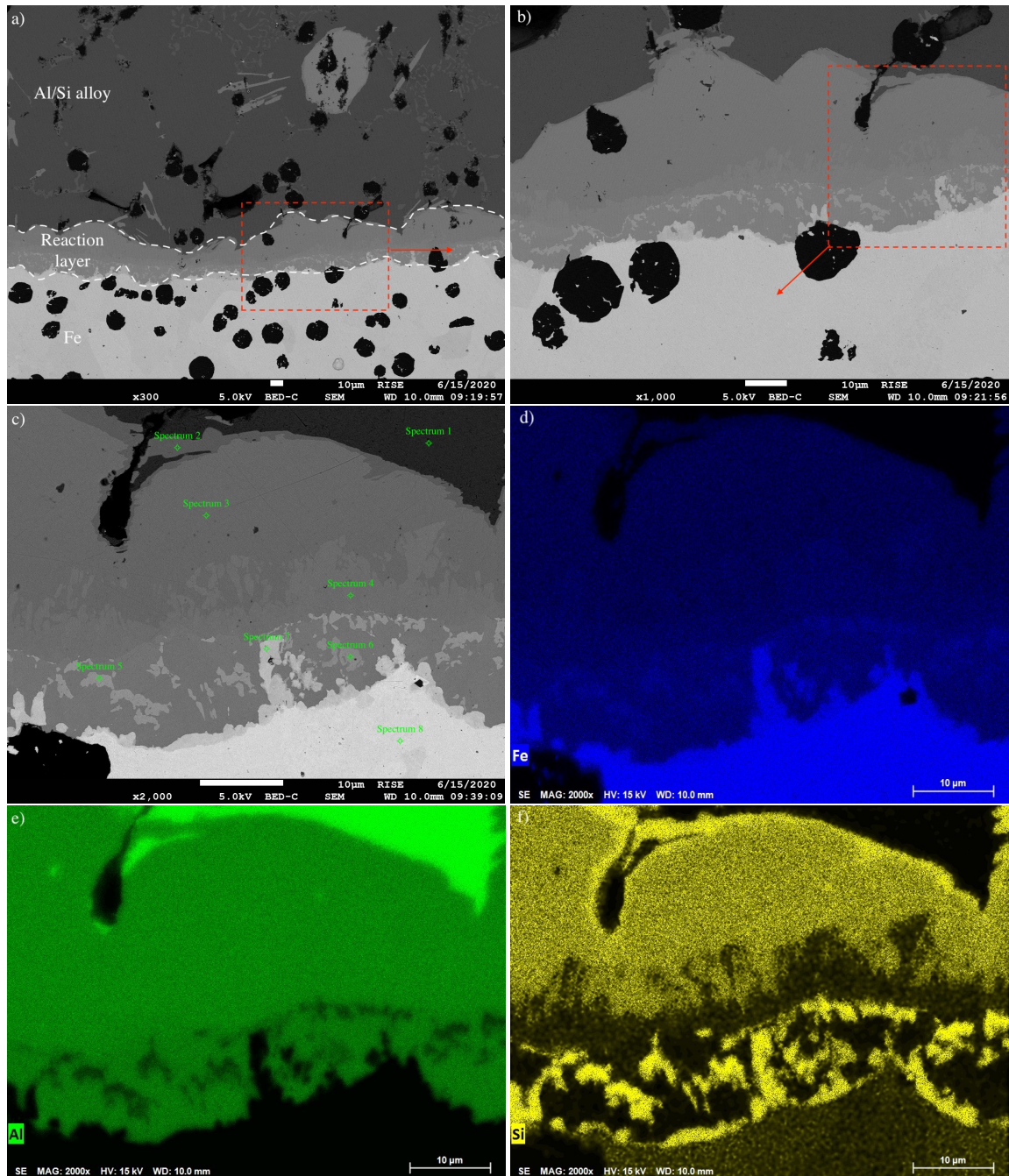


Figure 4.31: a, b) BSE image of the blasted+aluminized insert at two magnifications, and corresponding elemental mapping of c) Fe, d) Al, e) Si, and f) FeAlSi.

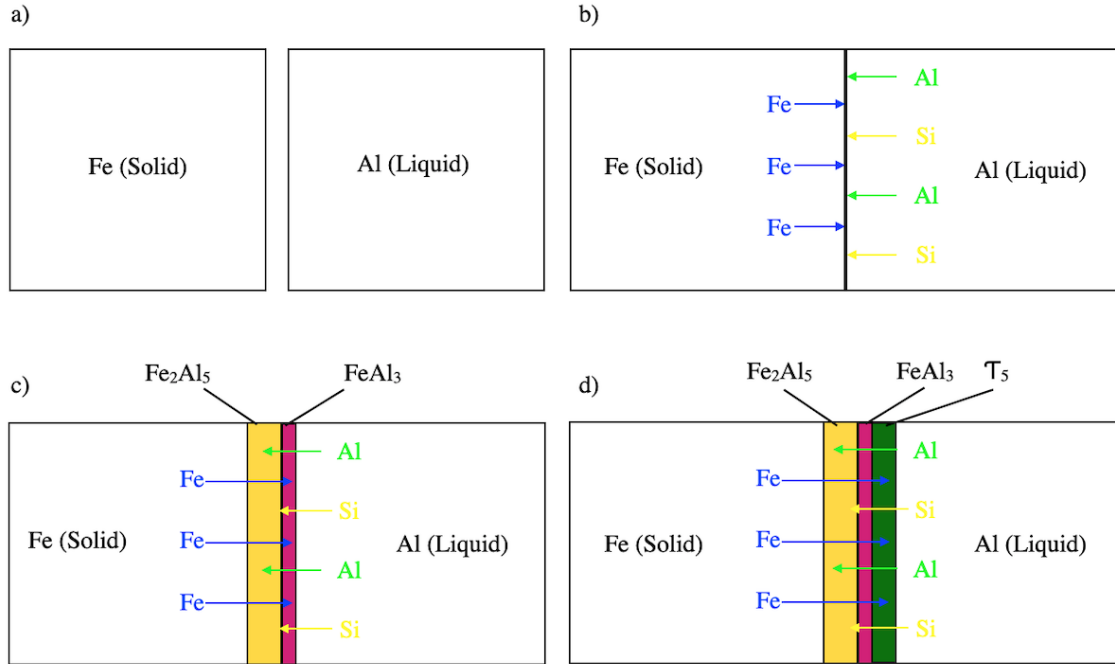
Table 4.5: EDS analysis of points indicated in Fig 4.31.c

Number	Elements composition (at.%)			Inference component
	Al	Fe	Si	
1	97	0.3	0.8	Al
2	64.3	18.6	17	$\tau_5 - Al_8Fe_2Si$
3	64.1	25.7	10.2	$FeAl_3$
4	69.6	27.7	2.7	Fe_2Al_5
5	38.4	41.6	19.7	$AlFeSi$ ternary compound
6	46	41.1	12.6	$\tau_1 - Al_3Fe_2Si$
7	3.3	73.9	22.4	Fe
8		100		Fe

By considering the Al-Si binary phase diagram and Al-Fe-Si ternary system, the following reactions can be suggested for the formation of different intermetallic phases [31]:



Where τ_1 and τ_5 are Al_3Fe_2Si and Al_8Fe_2Si , respectively. A schematic illustration of the formation process of the diffusion layer was depicted in Figure 4.32.

**Figure 4.32:** Schematic illustration of Al/Fe reaction layer formation in blasted+aluminized insert.

4.5.1 Hardness

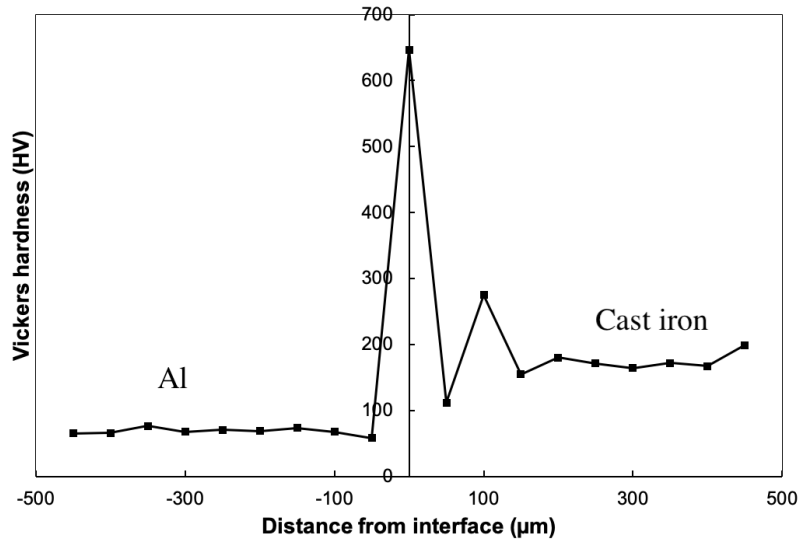


Figure 4.33: Hardness profile for the blasted+aluminized cast iron sample along the interface.

Figure 4.33 plotted the micro-hardness distribution at the interface of the blasted+aluminized cast iron substrate. It can be realized that the micro-hardness magnitude at the interface was significantly higher than the cast iron and aluminum matrix with a maximum value around $650HV$, and it decreased from the interface toward the Al side with an average hardness quantity of $80HV$ while for the cast iron side, there was an abrupt drop followed by an increase in the micro-hardness. It was because the indent was partially within the interface layer (Figure 4.34.c) and therefore the hardness was higher than the cast iron. After that, the hardness value of the cast iron insert remained almost unchanged around $170HV$. These results implied that a diffusional reaction had occurred at the interface of the blasted+aluminized sample, resulting in the formation of a metallurgical bonding between the aluminum and cast iron.

Figure 4.34 demonstrated the micro-hardness indentation size obtained from micro-hardness testing. It can be found that the aluminum matrix had the largest indentation size, indicating a soft matrix that elastically deformed during the hardness test. On the other hand, the interface with the smallest indentation size exhibited a hard and brittle phase that instead of plastic deformation, cracks initiation and growth had happened during the testing.

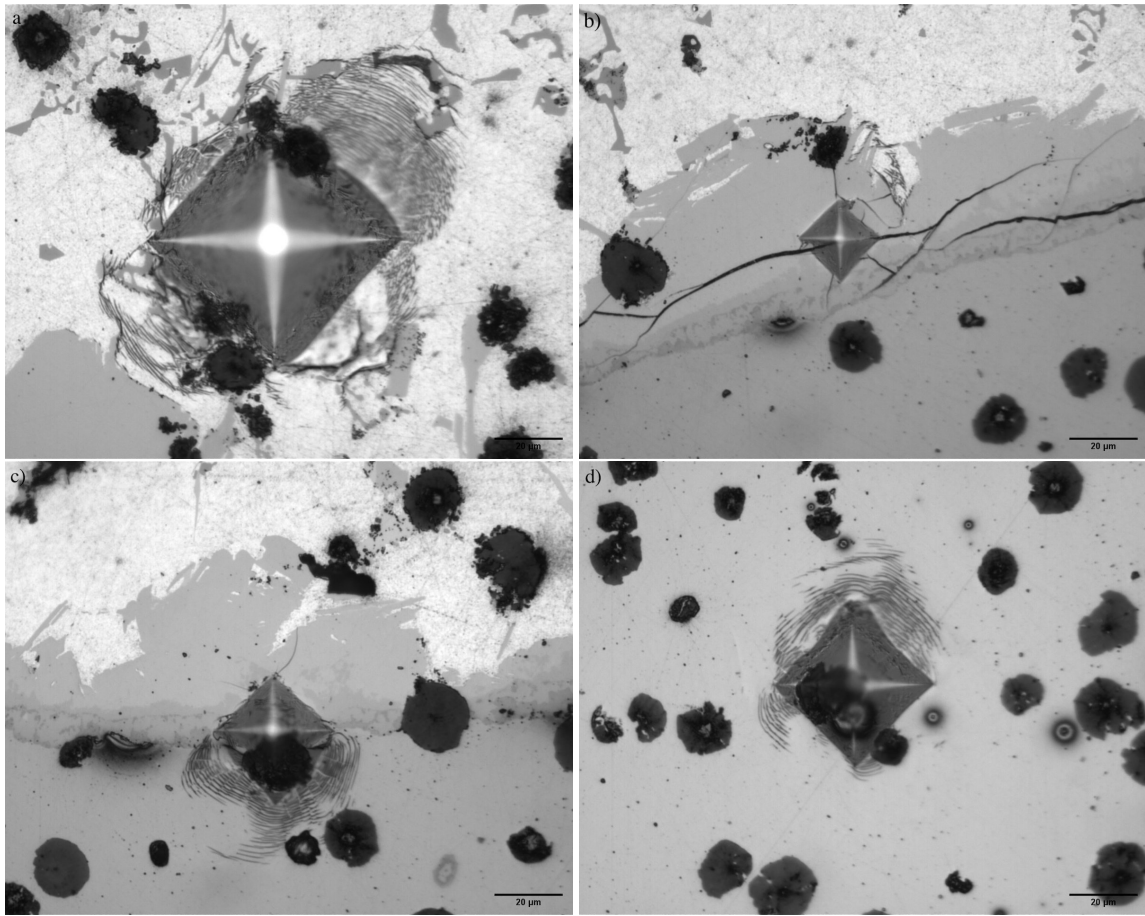


Figure 4.34: Optical image of the micro-hardness indentation size for a) aluminum, b) reaction layer, c) interface between intermetallic layer and cast iron, and d) cast iron.

The microstructural analysis of the blasted+aluminized samples indicated that in contrast with all other surface treatment methods on the cast iron, a continuous and compact metallurgical bonding was formed at the Al/Fe interface which was also compatible with the hardness testing results. This diffusion layer contained a combination of three major complex phases of Fe_2Al_5 , $FeAl_3$, and Al_8Fe_2Si which had been developed through sequential chemical reactions.

There were three main reasons contributing to obtaining a sound intermetallic layer between the cast iron and aluminum. Firstly, the cast iron surface was blasted before casting which provided an oxide-free surface for following hot-dip aluminizing process. Since there was no oxide scale above the cast iron insert, a uniform aluminum coating was shaped on the base material. Moreover, the pre-heating stage also was removed, implying that the cast iron insert would not be exposed to an air atmosphere which can help an oxide formation and/or growth. Therefore, there was not an obstacle for the diffusion of the Al and Fe atoms at the interface. Finally, the time between each surface preparation and casting kept as short as possible to minimize the oxide formation. To elaborate more, the time between the blasting and aluminizing was less than $2min$ and the casting was performed immediately

after aluminizing.

On the other hand, SEM images showed that even by applying these considerations, there are still some areas that a continuous gap has occurred within the microstructure at the interface between residual aluminum from dipping experiment and poured aluminum. Performing the casting experiment under the vacuum to prevent oxidation might be an expedient solution to avoid gap formation, however, it would be a time-consuming method. It is also important that the casting process should be industrially applicable and therefore it may not be a good idea to add more steps.

5

Conclusion and Future Prospects

To investigate the effect of different surface treatments on the interfacial adhesion of the Al/Fe interface, and its corresponding bonding strength seven different types of samples including sandblasting, cleaning, etching, hot-dip galvanizing, acidic zinc electroplating, hot-dip aluminizing, and thermal spraying were prepared. Then, they were pre-heated at 300°C and were placed in a pre-heated mold at 380°C followed by gravity casting of the molten aluminum around the cast iron inserts. The microstructural analysis of the samples indicated that no metallurgical bonding was formed for uncoated samples since the diffusion reaction layer was hindered by the presence of a thick oxide layer above the cast iron substrate during pre-heating and solidification. However, the blasted sample showed around 8% increase in the interface shear strength compared to the reference sample from 26.5MPa to 28.5MPa since its serrated microstructure resulted in mechanical gripping between the cast iron and aluminum.

Regarding the coated sample, optical images, SEM micrographs, and EDS analysis revealed that none of the coating materials and methods had led to the formation of the desired intermetallic reaction layer. The main reason for the failure of all specimens was suggested to be the poor wetting of the aluminum melt and most likely the formation of an oxide layer above the coating materials during the pre-heating which was prohibited the diffusion of the cast iron and aluminum along with the interface. However, some other factors triggered the failure of each sample as well. In the case of hot-dip galvanizing, the thick zinc coating layer did not melt during the casting to facilitate the wetting of the aluminum which was because of insufficient energy input. Instead, a relatively large gap was formed in the component during solidification, resulted in the lowest shear strength among all specimens with a modest value of 22MPa. For the hot-dip aluminized sample, although some local intermetallic islands were shaped after the aluminizing process, they could not tolerate the melt shear stress, and therefore they were flushed away during casting. In association with thermal sprayed sample, some local adhesion between the coating and the molten aluminum was observed which explained its high interface strength in the push-out test (28.5MPa), however, due to the insufficient pressure provided by gravity casting, these points also broke, causing the failure of the specimen.

Based on the obtained result, a new casting experiment was designed with blasting+aluminizing of the cast iron insert, removing the pre-heating stage, and casting immediately after aluminizing to prevent the oxide formation on the surface of the substrate. This procedure successfully led to the formation of a diffusion reaction

layer between the cast iron and the aluminum, confirming that the metallurgical bonding can only be obtained by controlling the oxide scale formation during the casting process.

The formation of the metallurgical bonding was a step forward to obtain the sound reaction layer at the interface between cast iron and aluminum. However, some gap formation was still observed at the interface. Hence, optimizing the blasting+aluminizing process to achieve a defect free interaction layer could be a new research topic. Moreover, the hardness results indicated that the reaction layer was brittle and hard which may cause the failure of the component during the usage stage. Consequently, the effect of heat treatment on the morphology and mechanical properties of the diffusion zone could also be investigated. Finally, after obtaining the optimized solution for having a desired metallurgical bonding, the structure should be characterized regarding the corrosion, fatigue, thermal, and mechanical properties.

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A

Appendix 1



Figure A.1: Hot-dip aluminized rings after 5, 10, and 20 min dipping time.

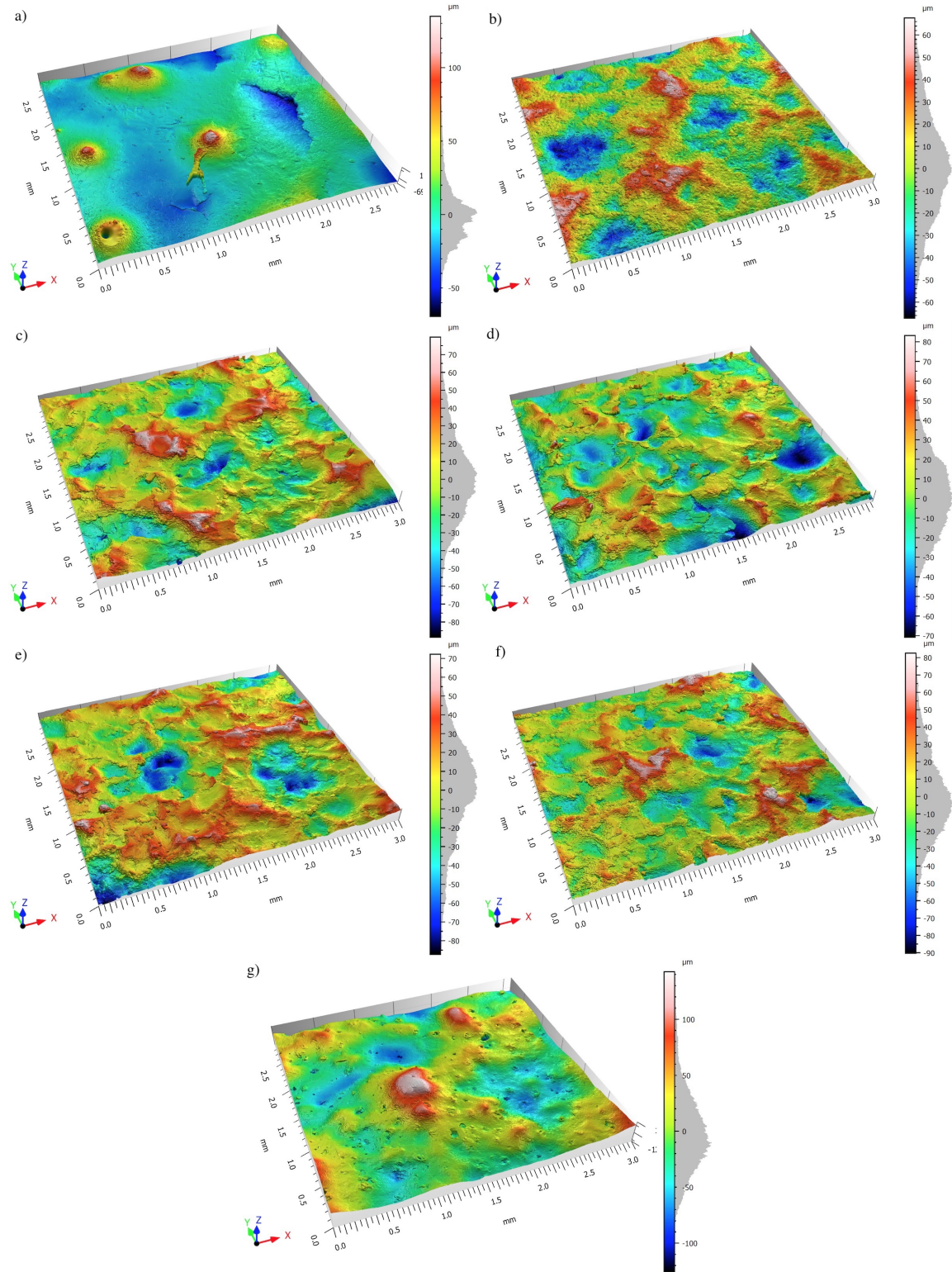


Figure A.2: Surface roughness profile of a)aluminized, b)blasted, c)etched, d)reference, e)cleaned, f)zinc plated, and g)galvanized sample.