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### DEVELOPMENT OF IRON ALUMINIDE ON STEEL SUBSTRATES

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**Abstract.** Iron aluminides are intermetallic alloys regarded as potential materials for high temperature applications because of their low production cost, elevated strength to weight ratio, wear resistance and excellent high temperature oxidation and corrosion resistance. The low ductility and susceptibility to environmental embrittlement has limited the options for manufacturing of components and inhibited the widespread use of iron aluminides. For these reasons, several processes for developing iron aluminide coatings on conventional steel substrates have been developed, such as: thermal spraying, electrodeposition, laser and plasma alloying, as well as aluminizing followed by diffusion treatments. All these manufacturing processes have their peculiarities both in terms of the final results obtained and regarding the manufacturing process parameters. In the present contribution, a critical survey on the different manufacturing processes applied for the deposition of iron aluminide coatings is presented. The main characteristics (thickness, mechanical properties, phase composition) of current iron aluminide coatings are compared with a novel friction based solid-state deposition process.

**Keywords:** Iron Aluminides; Coatings; Friction surfacing; Thermal spraying .

#### 1. INTRODUCTION

Iron aluminides are alloys with potential for high temperature applications because of their low density, excellent oxidation resistance, and relatively low cost. Materials based on the Fe-Al system (bulk or as coatings) have thus been investigated for applications involving super-critical nuclear reactors (Sun *et al.*, 2009), concentrating solar power (Fetzer *et al.*, 2012; Cionea *et al.*, 2016), high temperature KCl environments found in biomass plants (Li and Spiegel, 2004; Pan *et al.*, 2011), among others. In recent years, iron aluminides have also been considered as potential substitutes of conventional stainless steels at room temperature, mainly because of lower production costs since Fe-Al alloys do not involve large quantities of Ni and Cr.

The high temperature oxidation resistance of iron aluminides is due to the formation of a stable  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> oxide layer on the metal surface, which acts as a diffusion barrier limiting the interactions between the reactive underlying metallic substrate and the environment. The formation of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> is in many alloys preceded by the formation of less stable Al<sub>2</sub>O<sub>3</sub> polymorphs, such as  $\theta$ -Al<sub>2</sub>O<sub>3</sub> or  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> which are eventually converted to  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> causing elevated tensile strains because of volume incompatibility which are detrimental to oxidation behavior (Huang *et al.*, 2011). The formation of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> as a protective layer is important for various metallic systems in high temperature applications, but is particularly interesting in iron aluminides, since the oxidation of Fe produces template oxides that allow the formation of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> is

avored at lower temperatures and at shorter oxidation times in comparison to other  $\text{Al}_2\text{O}_3$ -forming alloys (Asteman and Spiegel, 2009). The process of protective  $\alpha\text{-Al}_2\text{O}_3$  oxide scales in iron aluminides is presented schematically in Figure 1.

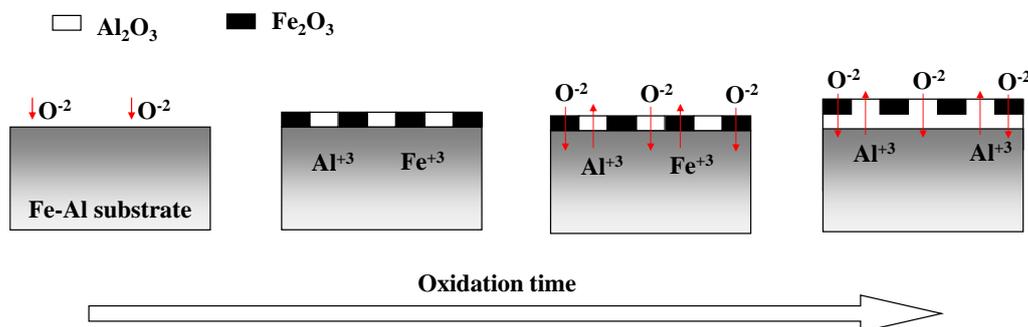


Figure 1. Mechanism of protective  $\alpha\text{-Al}_2\text{O}_3$  formation in iron aluminides. Adapted from Pöter *et al.* (2005) and Asteman and Spiegel (2009).

Iron aluminides do, however, face some challenges regarding mechanical properties in order to achieve widespread utilization. These materials have low room temperature ductility, which is reduced with the addition of the Al necessary for passivation at high temperatures. Indeed, it has been reported that alloys with Al wt.% have brittle behavior at room temperature (Prescott and Graham, 1992). These alloys may also experience losses in mechanical strength at high temperatures, which results in low creep resistance above 500 °C (Palm, 2005; Morris *et al.*, 2006). Attempts to counter these problems include dispersion strengthening and grain refinement, among others (Cinca *et al.*, 2019; Sarma *et al.*, 2019; Durga *et al.*, 2020).

The difficulties related to mechanical properties of iron aluminides have prompted the development of Fe-Al coatings on conventional steel substrates (Cinca and Guilemany, 2012). Most techniques employed for producing the coating involve fusion-based technologies such as thermal spraying or aluminizing. A possible alternative to the conventional deposition methods is the friction surfacing process that allows joining of similar and/or dissimilar materials in the solid state, thus avoiding some of the problems associated with solidification such as pore formation, hot cracking, dilution, residual stresses, among others. The friction surfacing process was effectively developed from the 1990s and has drawn growing interest from the scientific community given the increased demand for high performance coatings in critical applications (Gandra *et al.*, 2014).

In the friction surfacing process, a rotating consumable rod (with rotation speed  $R$ ) is pressed against a metallic substrate, as illustrated in Figure 2(a). With the initial contact, friction between the faying interfaces causes temperatures to rise and which causes plastic deformation and the formation of flash at the consumable rod tip, as seen in Figures 2(b) and 2(c). Once a critical plunge depth is achieved, the rotating consumable rod is moved forward with speed  $V$  along the substrate producing a longitudinal deposit, Figure 2(d). Since heat generation is caused by friction, the process takes place entirely in the solid state (Gandra *et al.*, 2014).

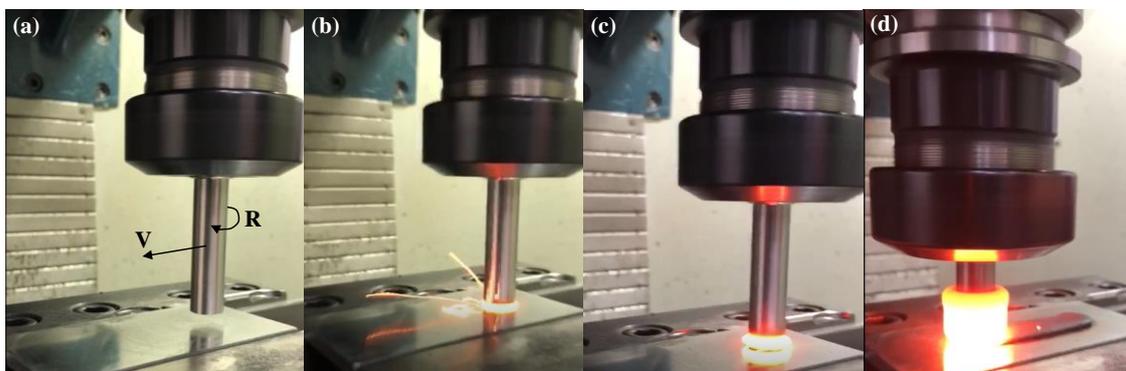


Figure 2. Stages of coating deposition by friction surfacing: (a) pre-contact setup, (b) initial contact, (c) flash formation at the consumable rod tip and (d) coating deposition.

In recent years, the friction surfacing process has been utilized for producing several coatings on conventional carbon steel substrates, such as: stainless steel, tool steel, Al-alloys, among others. The process involves intense plastic deformation which combined with elevated deposition temperatures leads to dynamic recrystallization and the formation of a fine-grained microstructure. In this review, a literature survey on current technologies applied for producing iron

aluminide coatings is presented followed by an analysis of results concerning iron aluminide coatings obtained from friction surfacing.

## 2. CURRENT PROCESSES IRON ALUMINIDE COATING PROCESSES

### 2.1 Thermal spraying

Thermal spraying encompasses a group of processes in which materials (in the form of powders, sticks or wires) are heated and melted by a heat source, usually flame, electric arc or plasma. After fusion, metal droplets are precipitated in a gas flow and directed towards the substrate to be covered. With the impact, the metal droplets solidify rapidly after potentially limited wetting and adhere to the surface. An example of a typical thermal sprayed coating (cross-section) is presented in Figure 3. The presence of pores caused by incomplete wetting can be identified, as well as the layered structure formed by the individual metal “splats”.

Currently, thermal spraying is one of the methods of choice for producing iron aluminide coatings and because of its inherent productivity, allows relatively thick (>1mm) coatings to be produced at relatively low cost (Cinca and Guilenmany, 2012; Deevi, 2021). In spite of many advantages and generally good results, it has been observed that the iron aluminide coatings produced by thermal spraying are sensitive to deposition parameters, tending to develop gas porosities (Haušild *et al.*, 2012; Porcayo-Calderon *et al.*, 2013) and/or chemical heterogeneities along the deposited layer (Canarim *et al.*, 2013), which may lead to a reduction in corrosion, oxidation and/or wear resistance.

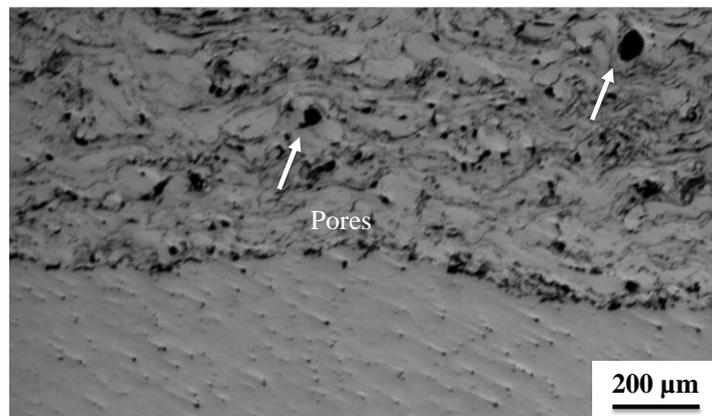


Figure 3. Thermal sprayed coating of an AISI 420 martensitic stainless-steel coating on 1045 carbon steel substrate (Optical micrograph, no etching).

Variations in chemical composition present in thermal sprayed Fe-Al coatings induce the formation of different intermetallic phases. For instance, Chmielewski *et al.* (2018) observed both intermetallic constituents and Fe(Al) solid solutions. Similarly, Senderowski (2014) identified heterogeneous phases in the thermal sprayed Fe-Al coatings, with significant variations in aluminium content and the presence of oxides inclusions, particularly  $Al_2O_3$ . The variations in chemical and phase composition may cause heterogenous mechanical properties in the coating (see properties of Fe-Al intermetallics in Table 1).

Table 1. Hardness of Fe-Al intermetallic phases (Potesser, 2006).

Phase	Crystal Structure	Vickers Hardness
Fe	BCC	180 – 480
FeAl	BCC	491 – 667
Fe <sub>3</sub> Al	DO <sub>3</sub>	344 – 368
FeAl <sub>2</sub>	Triclinic	1058 – 1070
Fe <sub>2</sub> Al <sub>5</sub>	Orthorhombic	1000 – 1158
FeAl <sub>3</sub>	Monoclinic	772 – 1017
Al	FCC	35 – 150

### 2.2 Laser assisted deposition

More recently, laser deposition has been used for the production of iron aluminide coatings. Sharma, Awasthi and Chandra (2010) employed laser deposition to obtain Fe-Al coatings on carbon steel substrates. Using a 300 W power source and scan speed of 200 mm/min, the investigators obtained intermetallic phases FeAl<sub>3</sub> and Fe<sub>2</sub>Al<sub>5</sub>. Because of the

brittle nature of some of these intermetallics, cracks were found to develop at the interface with the substrate. It was possible, however, to modify coating phase composition by reducing scan speed to 100 mm/min, whereupon less brittle iron-rich intermetallics Fe<sub>3</sub>Al and FeAl were formed. Bax *et al.* (2013) also developed intermetallic coatings by using laser deposition and were able to obtain single phase Fe<sub>3</sub>Al and crack-free coatings on steel. Cracks were found to develop in coatings deposited on Al substrates.

### 2.3 Aluminizing

Several Fe-Al coatings can be obtained by aluminizing followed by heat treatment to promote diffusion. In general, it is possible to obtain homogenous coatings, but with comparatively lower thickness compared to thermal sprayed coatings. In addition, it was shown that it is possible to control intermetallic phase formation by varying aluminizing and heat treatment parameters.

The process was investigated in detail by Kobayashi and Yakou (2002), who varied heat treatments time and temperature to control intermetallic growth on aluminized coatings deposited on carbon steel substrates. For lower diffusion temperatures, Al-rich intermetallics are formed, namely Fe<sub>2</sub>Al<sub>5</sub>, with reduced thickness values. By increasing diffusion temperature and time, intermetallic layer thicknesses increased from approximately 100 to 200 μm, and conversion of the initially Al-rich intermetallics to Fe-rich FeAl and Fe<sub>3</sub>Al structures. The maximum reported intermetallic layer in this study was 600 μm (Kobayashi and Yakou, 2002). Similar results were obtained by Awan and Hasan (2008), who developed Fe<sub>2</sub>Al<sub>5</sub> with approximately 150 μm intermetallic coatings by hot immersion between 700 and 750 °C. More recently, Arabi Jeshvahani *et al.* (2014) analysed the formation of iron aluminide coatings on 9Cr-1Mo steel substrates by aluminizing with different laser sources. The investigators revealed that by varying laser pulse energy in the range of 7 to 10 J it was possible to alter intermetallic constituents from Fe<sub>2</sub>Al<sub>5</sub> to FeAl.

### 2.4 Summary

A summary of current technologies currently investigated for producing Fe-Al coatings is presented schematically in Figure 4. The diagram was arbitrarily developed relating the main Fe-Al intermetallic constituent with coating thickness. It is worth noticing that most of the coatings developed are formed by Al-rich iron aluminides, of superior hardness but limited ductility. It is considered that Fe-rich intermetallics exhibit a superior trade-off between strength and toughness (Mohammadi *et al.*, 2018).

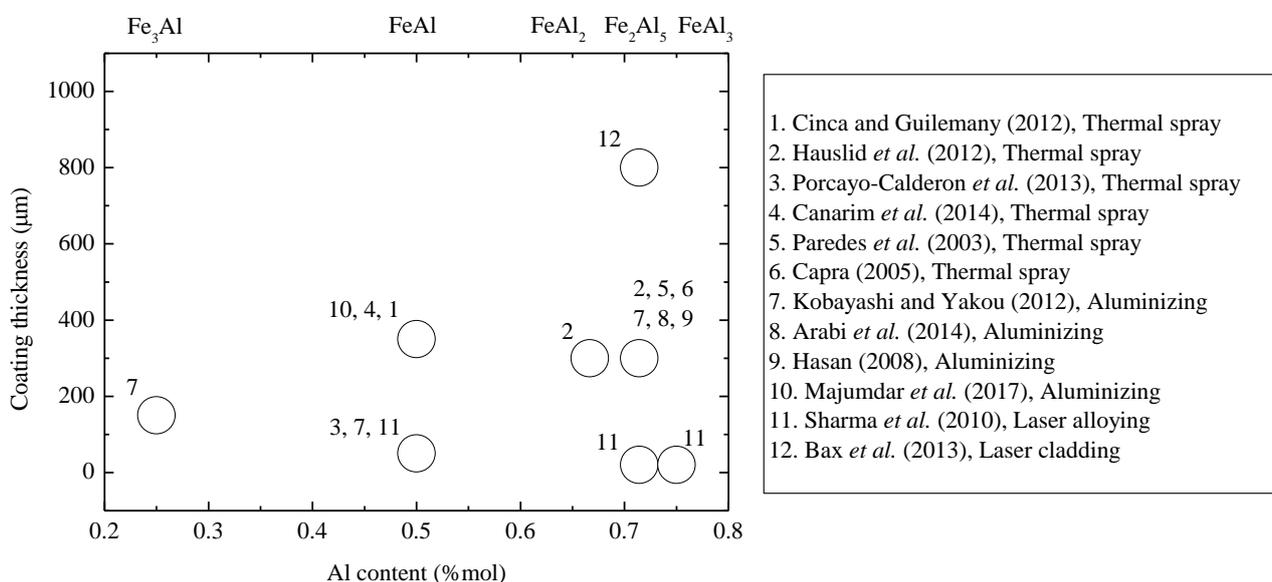


Figure 4. Comparison of different Fe-Al coating technologies as a function of thickness and Al-content.

## 3. IRON ALUMINIDE COATINGS BY FRICTION SURFACING

There are two current technologies based on friction surfacing proposed for the production of iron aluminide coatings, which involve the deposition of Al-alloy followed by diffusion heat treatment (Troysi and Brito, 2020) or the direct deposition of the desired Fe-Al alloy.

An analysis of the microstructure of diffusion-based coatings is presented in Figure 5. Initially, an AA6351 Al-alloy coating was deposited onto a ABNT 1020 carbon steel substrate with the deposition conditions reported previously by

Carvalho Filho and Brito (2020), which is shown in Figure 5(a). It is possible to notice distortion of the ferrite grains from the substrate, which become elongated parallel to coating interface (as indicated by the dark arrows). After 72 hours heat treatment at 600°C, the microstructure of the coating is modified, and the results presented in Figure 5(b) are obtained. The coating is not free from defects, as Kirkendall porosities are formed due to the difference in Fe and Al diffusivities (practically no Al diffusion towards the substrate is observed, as presented in Figure 6). The interface between substrate and coating also is altered: whereas a planar interface was formed directly after deposition, after heat treatment the coating exhibits protrusions towards the substrate, which are typical of intermetallic formation.

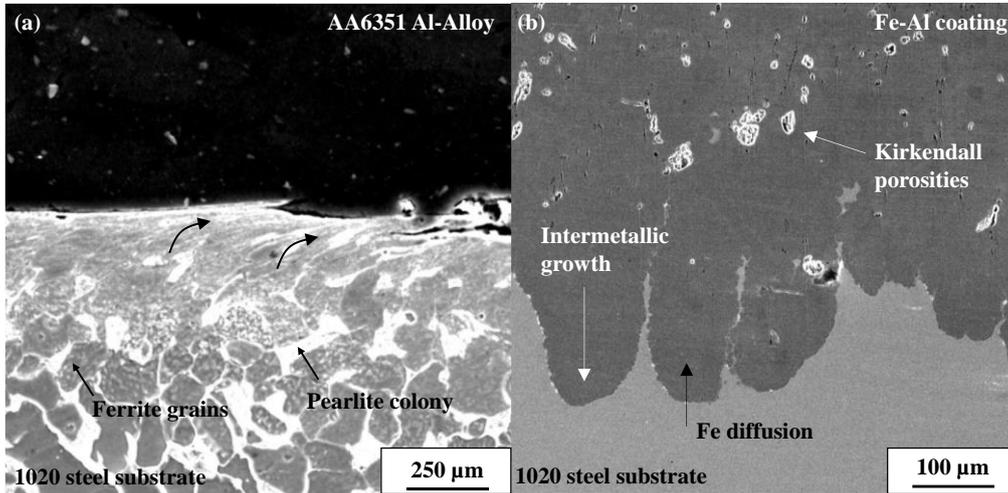


Figure 5. Microstructure of friction surface coatings: (a) AA6351 alloy deposited onto 1020 carbon steel substrate prior to heat treatment and (b) formation of Fe-Al intermetallics in the coating as a result of 700 °C heat treatment for 72 h.

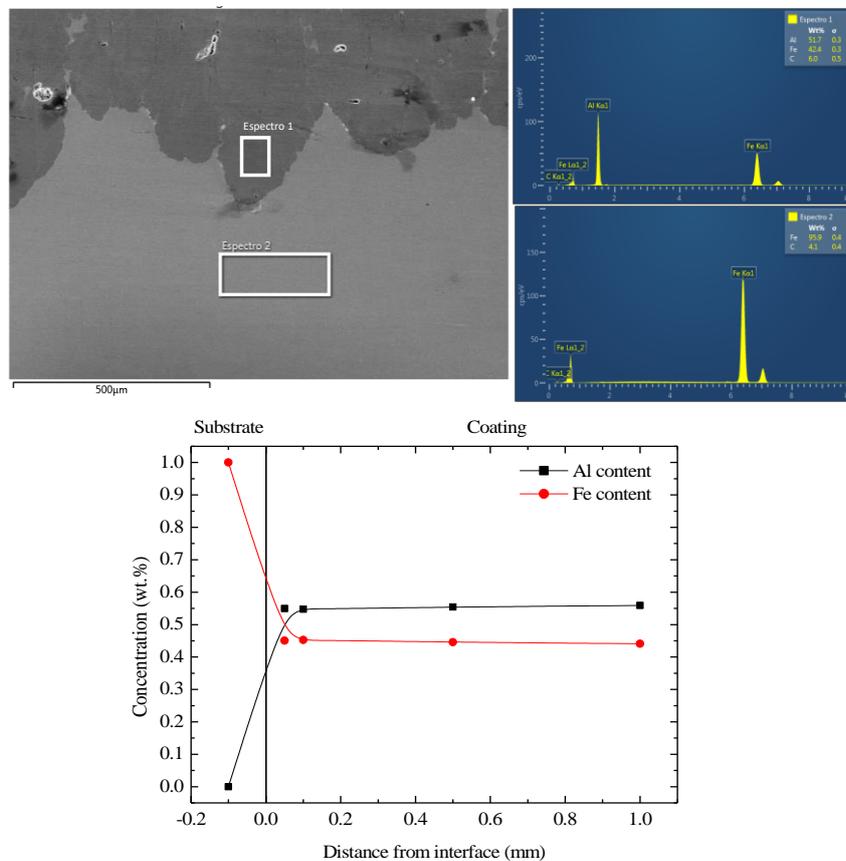


Figure 6. Coating/substrate interface of Fe-Al diffusion coating obtained by prior Al-alloy deposition analysed by SEM/EDS: (a) microstructure indicating EDS measurement spots, (b) corresponding EDS spectra and (c) Fe/Al relative concentration profile in-depth of the coating.

The analysis presented in Figure 6 shows that it is possible to obtain coatings with homogenous composition throughout the entire thickness (which in this case was of approximately 1.6 mm), with approximately 55 wt.%Al. The chemical composition indicates that possible intermetallic phases, based on the Fe-Al equilibrium diagram, are  $\text{FeAl}_2$  and  $\text{Fe}_2\text{Al}_5$ . The intermetallic protrusions towards the substrate also indicate that good adhesion can be expected, assisted by mechanical interlocking, although this still needs to be determined experimentally.

The advantage of diffusion coatings is that they can be obtained from Al-alloy consumable rods, which are readily available. The disadvantage is that the diffusion process occurs continuously at high temperature, which means that composition changes can take place in the coating in service depending on the operation temperature. In addition, the ongoing diffusion means that Kirkendall porosities will continue to be formed and coalesce, leading to an increase in the quantity and size of voids in the coating. For relatively lower operation temperatures, however, the coatings can be considered to be stable. A second alternative for the production of Fe-Al coatings assisted by friction surfacing is to deposit the desired Fe-Al alloy directly on the metallic substrate. This requires casting and preparing the intermetallic alloy, but it eliminates the necessity of heat treatment and in principal allows for better control of coating composition and properties.

An example of an iron aluminide coating obtained directly by friction surfacing is presented in Figure 7. The advancing and retreating sides of the coating reveal signs of the plastic flow observed during deposition. At the advancing side, Figure 7(a), near the outer edges of the coating, it is possible to notice the presence of discontinuities which are caused by heterogenous pressure distribution during friction surfacing. The presence of “undercut” at the edges of the coating is characteristic of the friction surfacing process (Gandra *et al.*, 2014). At the centre of the coating and most of the cross-section, however, the coating is continuous, cracks and pores being absent. It is worth noticing that the temperatures during deposition of bulk iron aluminide alloys are significantly higher than those observed during the deposition of Al-alloys, which are close to 400 °C according to Silva *et al.* (2018). As such, no signs of plastic deformation are observed in the substrate shown in Figure 7(a-c), since in this case deposition takes place above the steels austenitizing temperature.

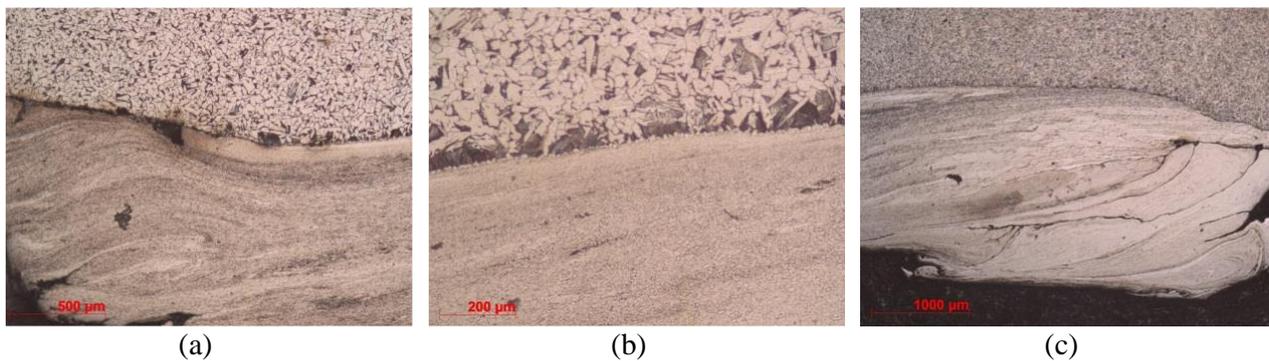


Figure 7 – Cross-section optical micrographs of a Fe-15Al coating (inferior portion) deposited on 1020 carbon steel: (a) advancing side, (b) centre and (c) retreating side.

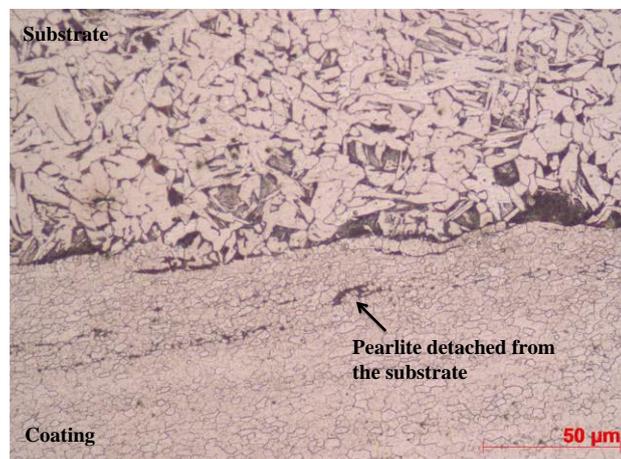


Figure 8 – Cross-section micrograph of the central region of an Fe-15Al coating deposited on 1020 carbon steel by friction surfacing.

The microstructure of the Fe-15Al coating reported in Figure 7 is analysed in more details in Figure 8. It is possible to notice that a fine-grained structure is obtained with the friction surfacing process which is attributed to dynamic recrystallization made possible by the elevated strain rates at high temperature. In the substrate (inferior portion of Figure 8) it is possible to notice the presence of Widmanstätten ferrite, probably caused by rapid cooling from the deposition temperature. Finally, it is worth noticing that the coating process does not cause dilution. However, detached pearlite particles from the substrate were found in the coating (as indicated). The effect of mechanical mixing is known to happen in solid state joining processes (Gandra *et al.*, 2014).

#### 4. OUTLOOK

In the present work, different deposition methods for producing iron aluminide coatings were analyzed. It was possible to identify opportunities for further development, seeking higher levels chemical and microstructure uniformity, lower levels of discontinuities, and homogeneous thickness. The production of Fe-Al coatings by friction surfacing (or assisted by friction surfacing) have the potential to increase coating thickness. Because the process takes place in the solid-state, coatings are less prone to formation of porosities and exhibit good mechanical integrity. As such, it is possible to consider that friction surfacing is a promising new technology for developing iron aluminide coatings on conventional steel substrates.

#### 5. ACKNOWLEDGEMENTS

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