Arch. Metall. Mater. **70** (2025), 2, 951-956

DOI: https://doi.org/10.24425/amm.2025.153499

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PRODUCING Al-Zn ALLOY COATING ON THE PURE Al SUBSTRATE BY SOLID-STATE FRICTION SURFACE ALLOYING

Using friction-assisted solid-state processes in the surface engineering of metals and alloys addresses the challenges involved in liquid-state methods. In the present work, friction surface alloying (FSA) was used to deposit Al-Zn alloy coating as a layer on pure Al substrate. Zn powder was filled in the holes produced longitudinally in a pure Al rod and was used as the consumable rod. Processing was done at 1100 rpm with 25 mm/min feed rate at a worktable vertical feed of 5 mm/min. Due to the generated heat during the friction surfacing, the zinc reinforcement was melted and a solid solution alloy of Al-Zn was produced on pure Al. The cross-sectional microstructure clearly indicated a strong metallurgical bonding at the alloy-substrate interface. Phase analysis carried out by X-ray diffraction studies confirms the developed Al-Zn alloy in the coated surface. Hardness measurements at the cross-section and at the surface demonstrated improvement (75.2±8.3 HV0.1) compared with the substrate (36.5±7.1 HV0.1) due to the formation of the alloy. Corrosion experiments by polarization studies revealed significant improvement in the corrosion resistance for the produced surface alloy as reflected from the lower corrosion current density (1.39×10^{-4} A/cm^2) compared with the substrate (1.83×10^{-3} A/cm^2). Higher tensile strength (245.5±5.2 MPa) was measured for the developed alloy than the substrate (182.1±6.9 MPa) without losing the ductility significantly. The results demonstrate the potential of FSA to develop surface alloys within the solid-state itself.

Keywords: Aluminum alloys; Surface alloying; Solid state; Friction; Surface hardness

1. Introduction

Surface engineering offers the benefits of improving the surface properties of structures without affecting the core by altering the surface composition or by depositing coatings of suitable phases on the substrate to produce extremely hard and high-performing surfaces [1-4]. On the other hand, alloying has several advantages in engineering applications and aids in the development of metallic structures with better qualities. In materials engineering, it is common practice to produce surface composites by dispersing several phases into the substrate without altering the chemical composition of the core [5]. Centrifugal casting [6], plasma spraying [7], surface laser melting [8], solid solution treatment [9], electron beam irradiation [10], friction surfacing (FS) [11], friction stir processing (FSP) [12], and other specialized techniques are employed to modify the phases at the material surface in order to impart special properties to the surfaces without changing the core. Several surface engineering methods which include melting of the coating surface to deposit the alloy coatings on the substrates of different substrates [13-16]. Solid-state techniques like FS and FSP reduce the limitations associated with liquid-state techniques [17]. Similar alloys or dissimilar alloys can be coated by FS as reported in the earlier literature [18-21].

By adding reinforcements to the surface, FSP enables to production of surface composites in addition to refining the microstructure of the surface [22]. Typically, brittle and hard reinforcing phases are chosen, with a melting point higher than the substrate. As a result, during FSP, the reinforcing phases are stirred and mixed while remaining in the solid state within the processed zone. The FSP tool geometry and the depth and width of the surface grooves or holes that are filled with reinforcements determine the thickness of the composite layer [23]. There are a few reported process modifications for friction-assisted metallic surface modification that impart distinctive features at the surface

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and leave the core unaffected. Among them, FS is one alternative, where the material to be coated is applied to a substrate by using consumable rod [24]. Another method called "friction deposition" was demonstrated by Dilip and Janaki Ram [25]. It is similar to additive manufacturing in that it involves layer-by-layer deposition of material onto a substrate. Recently, B. Lingampalli and S. Dondapati [26] used FSP to produce surface alloy by using ZK60 Mg alloy as the substrate with tin (Sn) within the solid state and improved hardness and corrosion resistance was reported. In order to produce 3D structures, the same group also devised an alternative technique known as "friction-free form" that employs layer-by-layer deposition [27]. The two other versions, known as "friction stir cladding" [28] and "friction stir channelling" [29], were also documented in the literature. They fall within the category of friction-assisted processes that create surface layers on substrates and create blind channels into solids, respectively.

Typically, the heat produced in FSP is insufficient to melt the ceramic or metallic reinforcements during developing a composite. On the other hand, an alloy between the dispersing phase and the substrate may form if FSP causes the dispersed phase to melt locally. It has been demonstrated that, with the correct processing variables, the nugget zone temperature can reach up to 0.7-0.8 times the substrate's melting point during FSP [30]. Therefore, in order to melt the dispersing phase during FSP and to produce a surface alloy, the dispersing phase melting temperature needs to be lower than the substrate. In the earlier study, "friction surface alloying" (FSA) was utilized to successfully create a binary surface alloy of Mg-Zn, and outstanding corrosion resistance against simulated bodily fluids was noted [31]. FSA method also uses consumable rod and coating is done by utilizing the frictional heat similar to that of friction surfacing. However, FS uses a consumable rod called as mechatrode, having the intended chemical composition of the alloy to be coated on a metallic substrate [9]. Whereas FSA does not use the alloy as the consumable rod, the development of alloy is achieved *in-situ* during the process. FSA can be viewed as a variation of FS to develop surface alloy layers. In the present work, FSA was used with the objective of producing Al-Zn surface alloy coating on a pure Al substrate. The surface alloy formation and mechanical and corrosion characteristics of the alloy have been investigated.

2. Materials and methods

Pure Al (99.9%) sheets of size $100 \times 100 \times 10$ mm^3 and rod of 20 mm diameter were purchased from Metro Alloys, India. Pure Zn powder (99.9%) was procured from Merck, India. Holes of 2 mm diameter were longitudinally drilled into the pure Al rod as illustrated in Fig. 1. Then the Zn powder was filled in the holes and the surface was closed with a cellulose tape. The pure Al sheet was then fixed on a vertical milling machine table (Bharat Fritz Werner, India). The Zn powder-filled consumable rod was fixed in the spindle in the inverted direction such that the Zn-filled holes came into contact with the workpiece surface as shown in Fig 1. Based on the preliminary experiments, processing parameters were selected as 1100 rpm (rotational speed of the consumable rod) with 25 mm/min feed rate (work table feed) with a worktable vertical feed of 5 mm/min. After the consumable rod while rotating touched the workpiece, a dwell time of 30 s was allowed and then lateral and vertical feeds were given for the work table. Processing was done for a distance of 30 mm and samples for characterization and performance evaluation were collected from the coated surface alloy as well as from the pure Al.

For microstructural examinations (Leica, Germany), the samples were collected from the pure Al, surface alloy and polished by using emery papers and diamond paste (1-3 μm particle size). The mirror-finished sample surfaces were etched with Picral reagent (5 g picric acid, 5 ml acetic acid, 5 ml water and 100 ml ethanol) for 20 s by immersing the polished samples in the etchant [32]. The base material and the surface alloy were analyzed by using X-ray diffraction (XRD, D8 Bruker, USA) between 20° to 80° range. The hardness of the alloy samples was measured by Vickers indentation method (Omnitech, India, 100 g load for 15 s) and compared with the substrate. Measurements were recorded in the thickness direction and across the produced Al-Zn surface alloy.

Using polarization studies by selecting 3.5% NaCl solution as the electrolyte, corrosion behavior was investigated (IVIUM Soft, Netherlands). In the presence of a saturated calomel electrode (reference electrode), the workpiece was referred to as the "working electrode" and a platinum electrode served as a counter

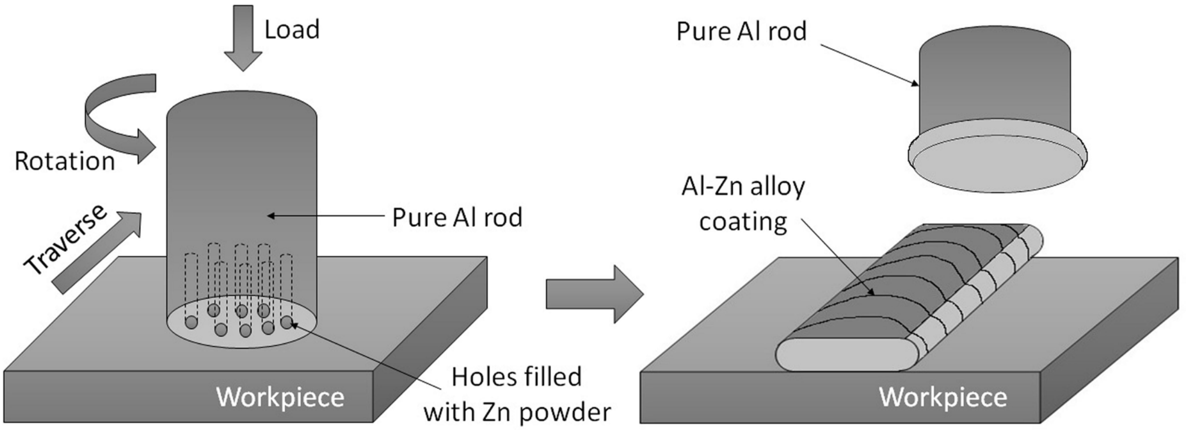


Fig. 1. Schematic illustration of solid-state surface alloy coating process used in the present work

electrode during the experiments. After 30 minutes of cell potential stabilization, polarization studies were conducted at a 5 mV/s scan rate within a fixed potential range of –1000 to 500 mV. Tafel extrapolation of the sample polarization curves was then used to record the electrochemical parameters [33]. In order to record the corrosion parameters, from the polarization curves, the change from anodic polarization to cathodic polarization is noted as corrosion potential (E_{corr}). Then the tangents are drawn from the anodic and cathodic branches from the point where slope is significantly changed. The corresponding value of the intersection point on the horizontal axis gives the corrosion current density (i_{corr}). Tensile experiments (Zwick/Roell, Germany) were carried out by using sub-size sample dimensions as per the ASTM-E8 standards [34]. The specimens ($n = 2$) were machined from the developed surface alloy and also from the substrate by using wire electric discharge machining (EDM). The surfaces of the test samples were polished to remove the surface unevenness and then experiments were carried out at the ambient conditions by employing 0.01/s strain rate. From the stress-strain curves, tensile properties were measured and compared.

3. Results and discussion

The microstructures observed at the cross-section of the developed surface alloy are presented in Fig. 2. It is clearly noticed the formation of a sound bonding at the interface of the surface alloy and the substrate. The corresponding magnified images (Figs. 2(b) and 2(c)) demonstrate the perfect metallurgical bonding without defects in the interface. Fig. 3 presents the XRD patterns of Al substrate and the produced Al-Zn surface alloy. All the peaks in the XRD pattern were identified by referring to the standard XRD data (ICDD, "International Center for Diffraction Data") and accordingly indexed. It is observed that the presence of peaks corresponding to Al and Zn in the XRD pattern of the developed surface alloy. No peak corresponding to any intermetallic was observed in the XRD of the produced surface alloy. By referring to the binary phase diagram of Al-Zn system as presented in Fig. 4, it can be learned that the dissolution of Zn in Al produces solid solution (α) grains of Al-Zn and on the other hand, the dissolution of Al in Zn also produces (η) solid solution grains at the room temperature [35].

Usually in the binary alloys development of intermetallics is inevitable if the alloying element exceeds the solubility limit. In Al-Zn alloy, no intermetallics are observed at room temperature as understood from the binary phase diagram of Al-Zn system. In the present work, no significant indication of intermetallics was observed from the microstructures as well as from the XRD analysis. It is suggested that the produced grains are combination of Al-Zn (α) and Zn-Al (η). Therefore, combination of peaks corresponding to Al and Zn are clearly identified in the XRD of the surface alloy. Producing the surface alloy in the current work can be viewed as a variant of friction surfacing (FS) process. In FS, the consumable rod does not melt but

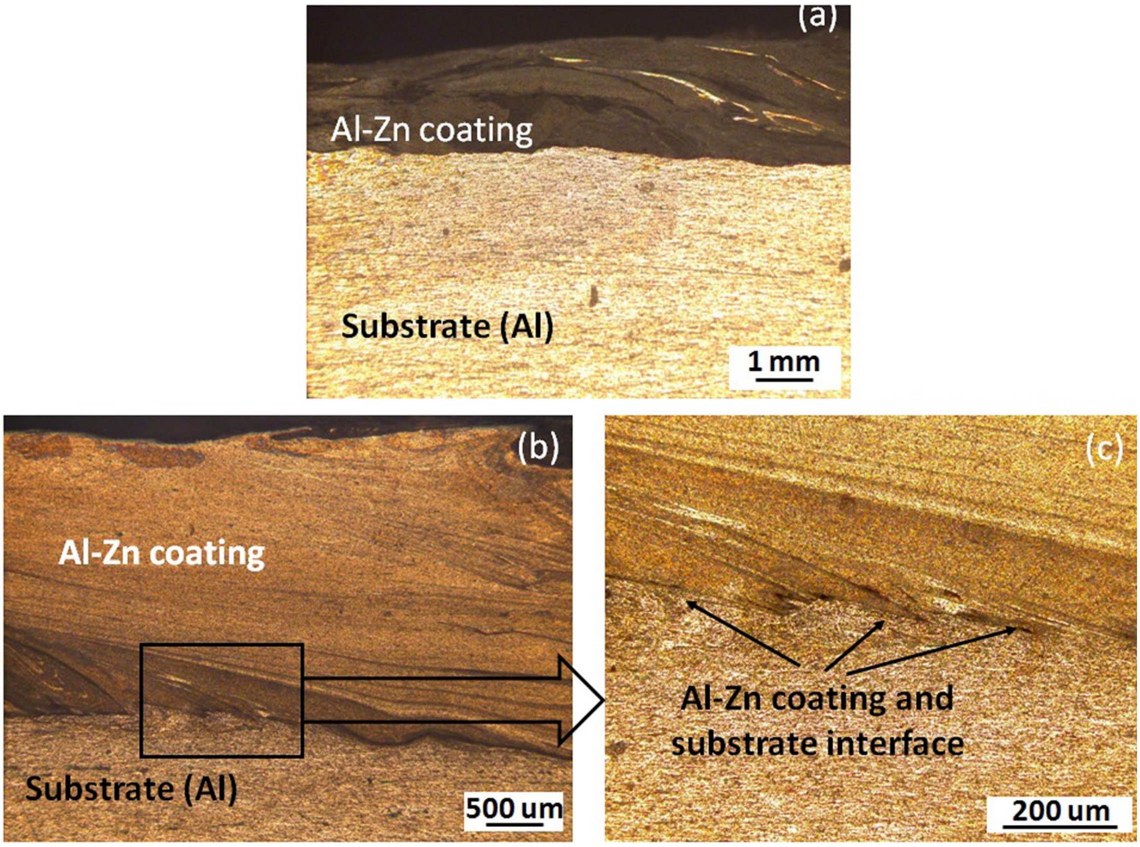


Fig. 2. Cross-sectional microstructures (a) Al-Zn surface alloy coating on the substrate, (b) corresponding magnified image and (c) interface of the coating and the substrate

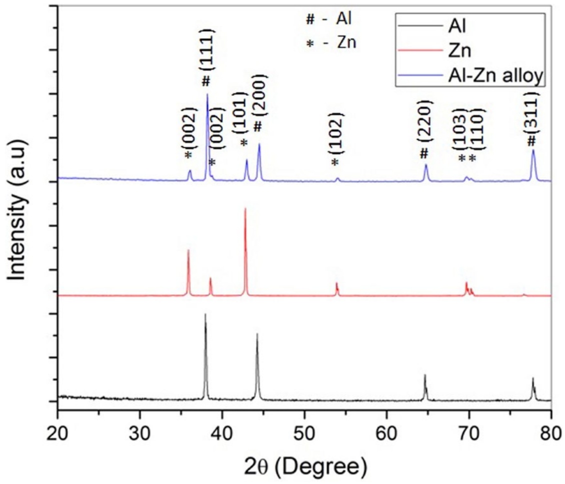


Fig. 3. XRD patterns of the substrate (pure Al), Zn powder and produced Al-Zn alloy

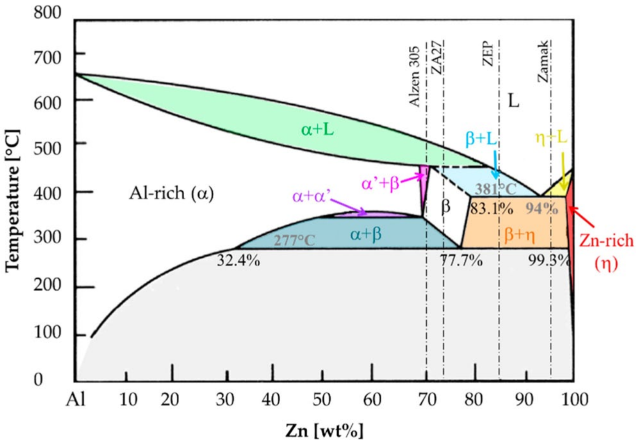
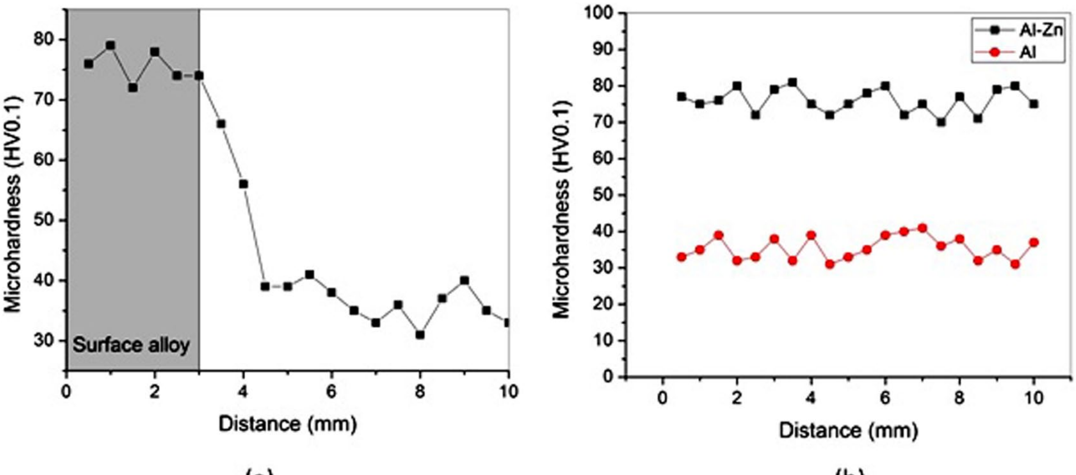


Fig. 4, Phase diagram of Al-Zn. (Source: Pola et al. [23])

undergoes frictional heating. The temperature in the processed zone is reached to 0.7 to 0.8 times of consumable rod melting point and helps to plastically deform at the coating interface [9]. In the present work, the interface of the Zn-filled Al rod and the substrate is reached more than 450°C (since the melting point of pure Al is 660°C). Zn powder in the holes of Al rod undergoes melting during the process as pure Zn has melting point of 420°C. Hence, during the process, the consumable rod does not melt but the Zn powder filled in the consumable rod is subjected to melting and mixed within the plastically deformed coating material and produces alloy.

The microhardness measurements recorded for the substrate and the produced surface alloy across the surface and at the thickness are presented in Fig. 5. It is clearly observed that the developed surface alloy has higher hardness (75.2±8.3 HV0.1) than the substrate (36.5±7.1 HV0.1). The variation of the hardness values can be clearly observed from the coated alloy layer, interface and substrate. Higher hardness values recorded for the surface alloy is attributed to the alloying effect. The ability of the solid solution grains produced in the process to resist the permanent indentation was reflected in the increased hardness compared with the substrate. In alloying, the solid solution grains relatively exhibit higher hardness compared with that of metals in pure state due to the solid solution strengthening effect. As the measurements were carried out across the interface, a steep fall in the hardness was observed from the surface alloy to the substrate. Since, the interface also subjected to plastic deformation, substrate also exhibited higher hardness values beneath the interface.

Fig. 6 shows the potentiodynamic polarization curves and TABLE 1 lists the obtained electrochemical parameters. It is noted that the surface alloy has marginally noble behavior as observed from the increased potential value (E_{corr}) compared with the substrate. From the corrosion current density (i_{corr}), lower value was clearly measured for the surface alloy which gives a clear indication of better corrosion performance of the alloy. Increased corrosion resistance for the surface alloy is attributed to the role of alloying compared with the substrate. Since the alloy grains are of substitutional solid solution grains, the electrochemical reaction in the presence of corroding environment is decreased as reflected from the lower rate of current flow under varying potential [36]. Additionally, absence of intermetal-



(a) (b)

Fig. 5. (a) Microhardness at the cross-section of the developed Al-Zn surface alloy and (b) comparison of the hardness of the surface alloy with the substrate

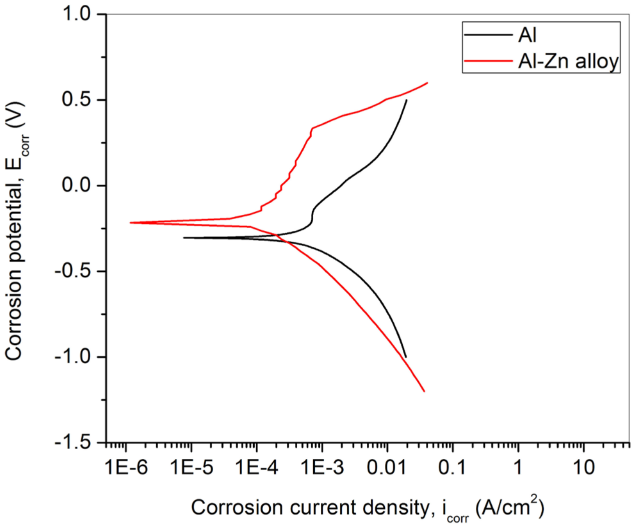


Fig. 6. polarization curves of the substrate and the Al-Zn alloy

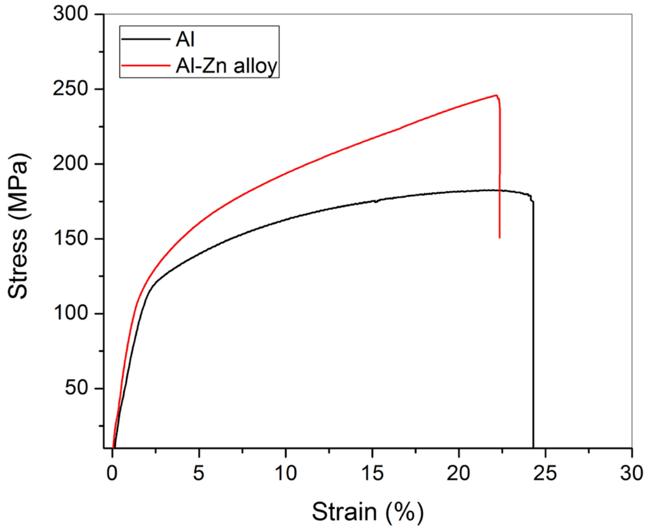


Fig. 7. Comparison of the tensile test results of the samples

TABLE 1

Corrosion parameters recorded from the polarization tests

Sample	Corrosion potential (E_{corr}) (V)	Corrosion current density (i_{corr}) (A/cm^2)
Al substrate	–0.34	1.83×10^{-3}
Al-Zn alloy	–0.21	1.39×10^{-4}

TABLE 2

Mechanical properties recorded from the tensile tests

Sample	Yield strength (MPa)	Ultimate tensile strength (MPa)	% elongation
Al substrate	114.4 ± 5.7	182.1 ± 6.9	24.6 ± 1.7
Al-Zn Alloy	126.3 ± 4.4	245.5 ± 5.2	22.3 ± 1.1

lics also eliminates the occurrence of galvanic corrosion [37]. Therefore, the produced surface alloy exhibited better corrosion performance compared with the Al substrate.

Fig. 7 compares the tensile curves and the properties measured from the test results are presented in TABLE 2. From the data, it can be learnt that the surface alloy has higher yield strength (114.4±5.7 MPa) compared with the substrate (126.3±4.4 MPa). Furthermore, it is also interesting to observe higher ultimate tensile strength for the surface alloy (245.5 ± 5.2 MPa) compared with the substrate (182.1 ± 6.9 MPa) without affecting the ductility as noticed from the similar % of elongation. Without sacrificing the ductility considerably, increasing the strength of the structure is a significant achievement. In alloying, the strength of the metals can be significantly increased along with achieving sustained ductility. By sacrificing some ductility, improving the strength of the metal to a great extent helps engineers to produce modern engineered materials to fabricate high-performing structures. Improving the mechanical and corrosion performance of the surface by developing alloy coatings in a solid state at the surface without affecting the core properties is promising, particularly in the surface engineering of components.

The developed surface alloy in the present work exhibited better corrosion and mechanical performance without affecting the behavior of the core. The alloy was successfully produced with perfect metallurgical interface between the produced surface alloy and the substrate. However, the choice of the alloying elements is crucial to develop the surface alloys. Since the melting point of Zn is lower than the consumable rod (pure Al in the current work), the melting of Zn due to the generation of temperature in the interface during the process helped to produce solid solution grains which resulted in developing the Al-Zn surface alloy. It is not possible with the alloying elements, which exhibit melting temperatures more than the temperature of the processed zone. Hence, availability of only fewer elements to develop the surface alloys in solid state by adopting the proposed method is a limitation. However, other elements with lower melting point can be selected and solid-state surface alloys can be successfully produced by using this method. Surface alloys of dissimilar metals also can be produced by this method. However, the level of solubility of the alloying element in the consumable rod and the substrate governs the quality of the surface alloy. Since the tendency to form intermetallics in dissimilar metals is higher; it is expected to observe intermetallics in the developed surface alloys. The demonstrated FSA method can be used to produce surface alloy layers of thickness up to a few mm to increase the life of the engineering structures and components without altering the chemical composition in automobile, aerospace and marine industries.

4. Conclusions

In the current work, a variation in the friction surfacing process is proposed to produce surface alloys of Al-Zn within the solid state. Holes of 2 mm diameter were drilled in a consumable rod made of pure Al and filled with Zn powder. Then the consumable rod was used in an inverted direction to coat a surface layer on pure Al substrate by adopting the friction surfacing principle. From the cross-sectional microstructures, formation of the surface alloy layer was noticed on the Al substrate with the defect-free interface. XRD analysis confirms the formation of Al-Zn solid solution grains of Al and Zn without the development of intermetallics. Higher hardness was observed in the alloy compared with the substrate. From the polarization studies, better corrosion resistance was demonstrated by the alloy as reflected in the lower i_{corr} value (1.39×10^{-4} A/cm^2) compared with the substrate (1.83×10^{-3} A/cm^2). From the tensile tests, higher strength (245.5±5.2 MPa) was achieved in the surface alloy compared with the substrate (182.1±6.9 MPa) due to solid solution strengthening without sacrificing the ductility significantly. The current study demonstrates the potential of the proposed variation in friction surfacing to produce alloys at the surface itself without affecting the core of the structures to enhance the surface performance of the structures.

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