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Improvement of microstructure and corrosion properties of friction stir welded AA5754 by adding Zn interlayer

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Abstract: This study investigated the effect of Zn foil layers on the microstructure and corrosion characteristics of friction stir welded aluminum alloy 5754. Samples of various joints were prepared by applying different rotational and welding speeds, and their microstructures were evaluated via a metallographic technique and scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy elemental analysis. The anticorrosion behavior of joints in the absence and presence of a Zn interlayer was studied by cyclic potentiodynamic polarization test in 3.5wt% NaCl aqueous solution, and sound welds were obtained in the presence of the Zn interlayer foil. The results revealed that the joint made at a rotational speed of 800 r/min and traveling speed of 15 mm/min achieved a chemical composition identical to that of aluminum alloy 7xxx series, and as such, it showed the best resistance to corrosion.

Keywords: friction stir welding; AA5754 aluminum alloy; zinc interlayer; microstructure; corrosion resistance

1. Introduction

Aluminum alloys, as prominent engineering materials, have many industrial applications because of their light weight and high strength [1]. For example, the 2xxx and 7xxx series can be used in aircraft structural parts, and 5xxx and 6xxx series can be used to fabricate automotive parts [2–3]. However, many challenges of manufacturing with aluminum parts, particularly joining methods, need to be well addressed.

Over the recent years, there has been an increasing interest on novel welding techniques. For instance, friction stir welding (FSW), introduced by The Welding Institute (TWI), UK in 1991, is a solid state joining method that eliminates conventional welding defects such as cracking, gas porosity, and distortion [4]. Meanwhile, much attention has also been focused on joining materials with adherent oxide layers, particularly aluminum alloys [5−7] as well as structural materials with high melting temperatures, such as steels [8−13]. In addition, various studies have been conducted on the joining of various dissimilar metals and alloys by FSW [14].

Several investigations have been dedicated to welding parameters (pin geometry, rotational and traveling speeds, etc.) in FSW of Al alloys [15−24], and it has been discovered that the ratio of rotational speed to traveling speed is important in determining the mechanical properties of a joint [25–26]. In addition, many attempts have been implemented to add precipitating particles to the joint gaps to achieve composite weld zones [27–29].

More recently, the joint properties have been improved by adding an intermediate layer, which can be in forms of sheet, filler, or foil [30–32]. In a study by Xu *et al.* [33] on dissimilar joint between aluminum 5754 and high strength steel, a layer was coated on steel to control the formation of brittle intermetallic compounds. They fabricated a friction stir spot welded Mg–Zn−Al alloy by adding zinc (Zn) interlayer and reported a reduction of weld defects. In this study, we investigated the effect of Zn foil insert on friction stir welded 5754 joints and assess the microstructural changes and corrosion behaviors.

2. Experimental

A 4 mm aluminum H114-5754 sheet of 100 mm × 200 mm with the chemical composition presented in Table 1 was

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used as the base material. A butt joint was prepared for joining, and the mating surfaces were carefully cleaned via wire brushing to remove oxide layers. A heat-treated H13 steel was machined (as shown in Fig. 1) to a truncated cone-like pin to fabricate the welding tool. Samples were welded based on various rotational and traveling speeds. Furthermore, identical samples were prepared by preplacing a 100 μm zinc foil of 99% purity in the gap joint, as shown in Fig. 2. Welding conditions for all samples together with their labels are illustrated in Table 2.

Table 1. Chemical composition of aluminum alloy 5754 wt%

Al	Si	Fe	Cu	Mn	Mg	Cr	Zn	Others
Base	0.16	0.14	0.05	0.23	2.9	0.12	0.09	0.03

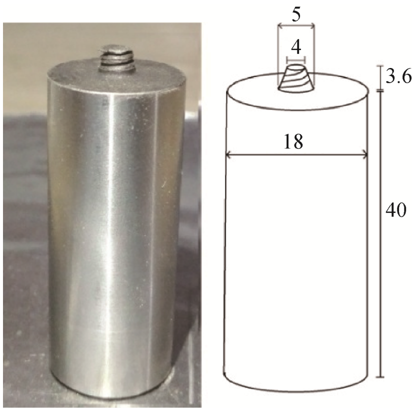

Fig. 1. Friction stir welding pin (unit: mm).

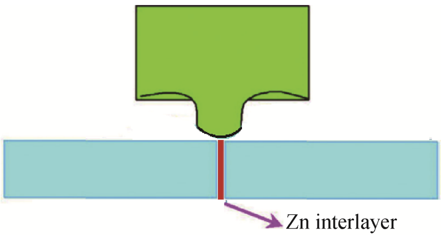

Fig. 2. Schematic presentation of utilized sheets for addition of Zn interlayer.

Table 2. FSW conditions for each specimen

Specimen No.	Rotational speed / ($r\cdot min^{-1}$)	Traveling speed / ($mm\cdot min^{-1}$)
S1	800	15
S2	800	25
S3	800	50
S4	600	25
S5	1000	25
F1	800	15
F2	800	25
F3	800	50
F4	600	25
F5	1000	25

The joints were cut into cross sections and then polished by 80–3000 grade SiC papers. Keller solution (2.5 mL HNO_3 + 1.5 mL HCl + 1 mL HF + 95 mL H_2O) was used as an etchant for macrostructural observations. Metallographic etching was accomplished by Barker's reagent. Grain size measurement was performed via MIP 4 Student Material software in accordance with ASTM E-112-96 standard. Further microstructural assessment was studied by scanning electron microscopy (SEM) equipped with an energy-dispersive X-ray spectroscopy (EDS) analyzer. The effects of Zn foil on corrosion behavior of welded joints were characterized by potentiodynamic polarization test implemented in aqueous 3.5wt% NaCl solution.

3. Results and discussion

Fig. 3 shows the cross sectional macrographs of welded joints without insertion of Zn foil. As seen, all samples were successfully bonded, excluding sample S1 which suffered from tunnel defect in the stir zone (SZ). It has been stated that inadequate material flow can lead to such defects in SZ [34]. For example, if the traveling speed is not enough, there

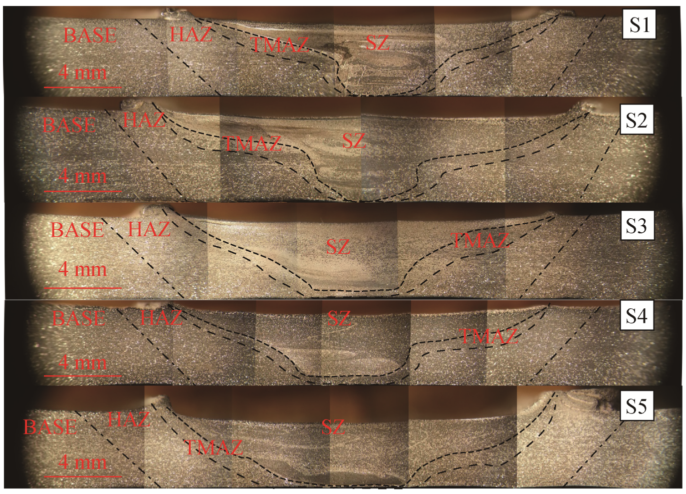

Fig. 3. Macrographic cross sections of joints without Zn foil addition.

would be possibility of porosity formation [18]. In stir zone, the layered structure is detectable. Samples S1 and S5 gained the highest heat input because of the high rotational speed/traveling speed ratio (ω/v); thus, considerable penetration was achieved; however, penetration in samples S3 and S4 was not sufficient to reach the lower surface of the sheet. A proper combination of rotational and traveling speeds was obtained in S2 with a bowl-like stir zone featuring uniformity throughout the thickness.

The macrographic view of each friction stir welded specimen with the addition of zinc foil is depicted in Fig. 4. All specimens are defect-free, indicating sufficient material flow in joint areas. The most uniform distribution of Zn interlayer is obvious in specimen F1, in which the low traveling speed allows proper stirring. The distribution uniformity of F2 is also acceptable; however, in F3, the increase in traveling speed to 50 mm/min led to stirring only in the upper layer. Thereafter, the traveling speed seems to shift stirring to relatively near surface areas. The impact of rotational speed can be observed in F4, F2, and F5 by the increased depth of distribution with increasing rotation speed from 600 to 1000 r/min. Nonetheless, the latter specimen lacked distribution in mid-area of the joint.

Optical micrographs from different areas of the welded joints are shown in Fig. 5. For both welding conditions, the

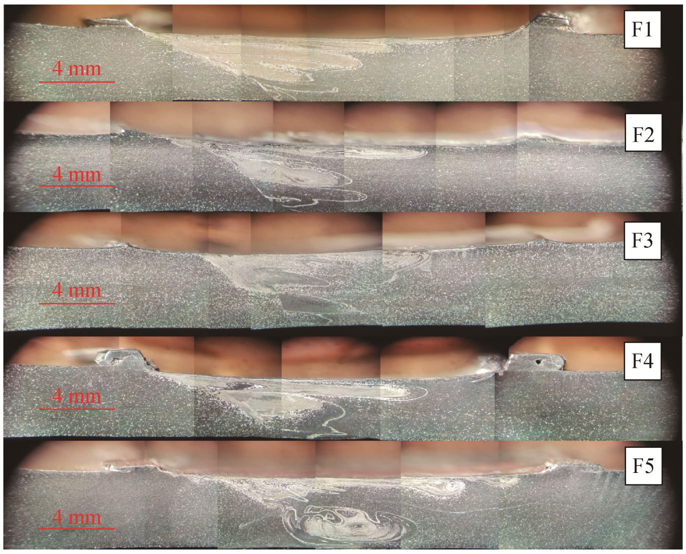

Fig. 4. Macrostructure of samples bonded with Zn interlayer.

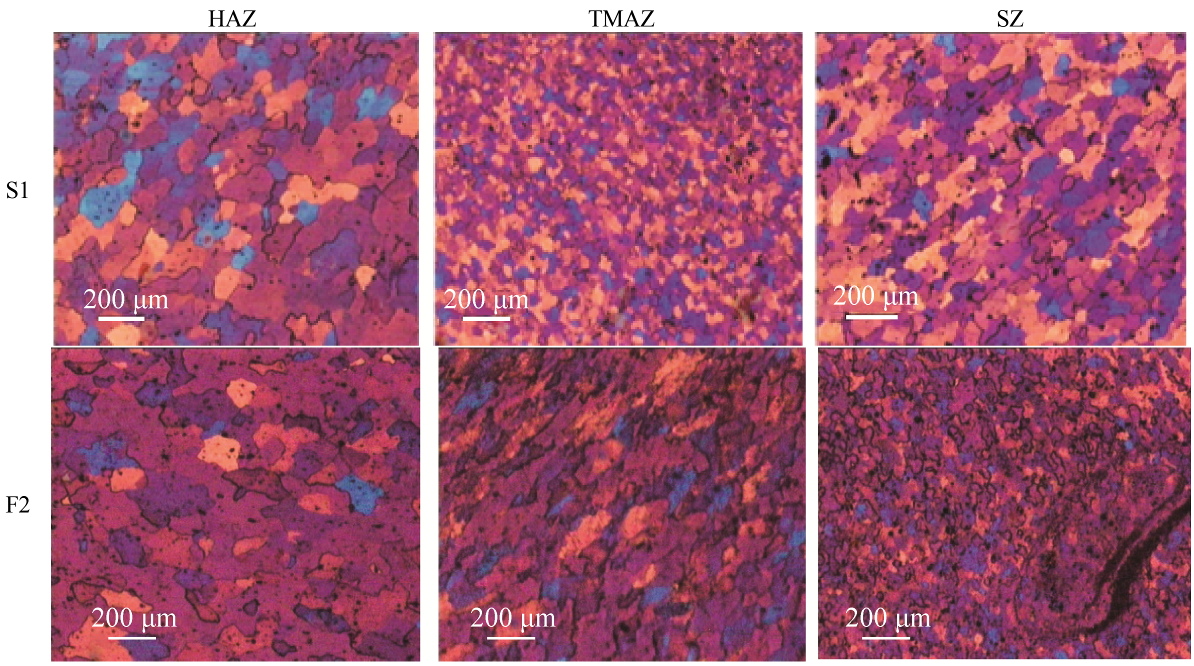

Fig. 5. Metallographic illustrations of various areas of friction stir welded joints.

specimens clearly contained identical morphology, featuring the finest grain size in SZ, deformed grains in thermo mechanically affected zone (TMAZ), and coarse grains in heat affected zone (HAZ). The reduced size of SZ grains can be attributed to recrystallization [5] as a result of high heat input and plastic deformation [4,23,32]. In addition, as the degree of deformation increases, the grains of the base metal become finer [6]. However, temperature was not sufficient to provide recrystallization in TMAZ. Instead, plastic deformation, which stemmed from pin rotation, and deformed grain structure were observed. In the heat affected zone, the occurrence of grain growth can be related to the friction heat induced by rotation of pin [35].

The grain size measurement was conducted based on ASTM E-112-96 standard. Fig. 6 shows the grain size of SZ and HAZ and the corresponding ω/v value as a function of heat input [36]. The grain size of the base metal was measured as 59 μm. The grains of HAZ were coarser compared to SZ grains because of the abovementioned grain growth phenomenon. The increase in heat input, which was shown as the ratio of rotational to traveling speed, revealed that larger grain size is proportional to higher heat input. There was also no considerable change between the grain size of the conventional sample and that of the sample welded with Zn foil, indicating an identical microstructure for both welding conditions.

To further assess the effect of zinc layer on the microstructural facets of the joint, SEM and EDS analyses were carried out. According to micrographic observations, specimens F1 and F4 were selected as the best and worst welded joints, respectively. According to Fig. 7, In F4, the amount

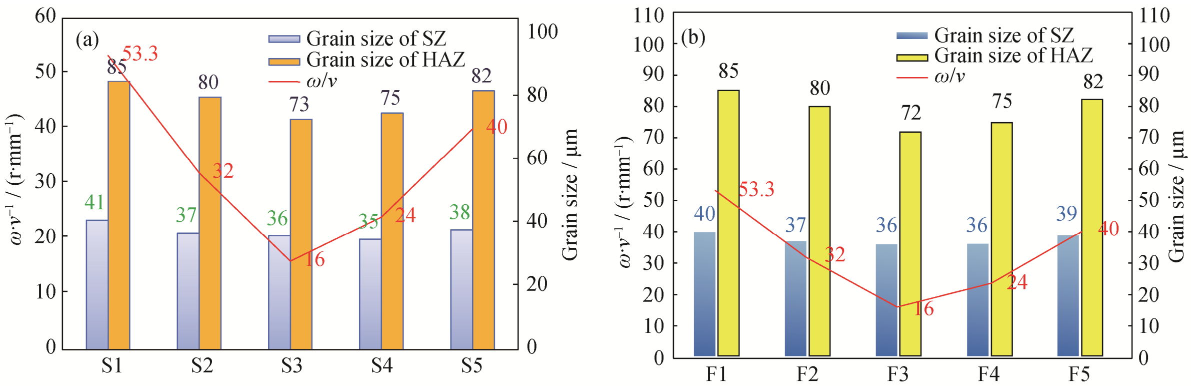

Fig. 6. Grain size values of SZ and HAZ and the corresponding ω/v of samples prior to zinc foil addition (a) and Zn added samples (b).

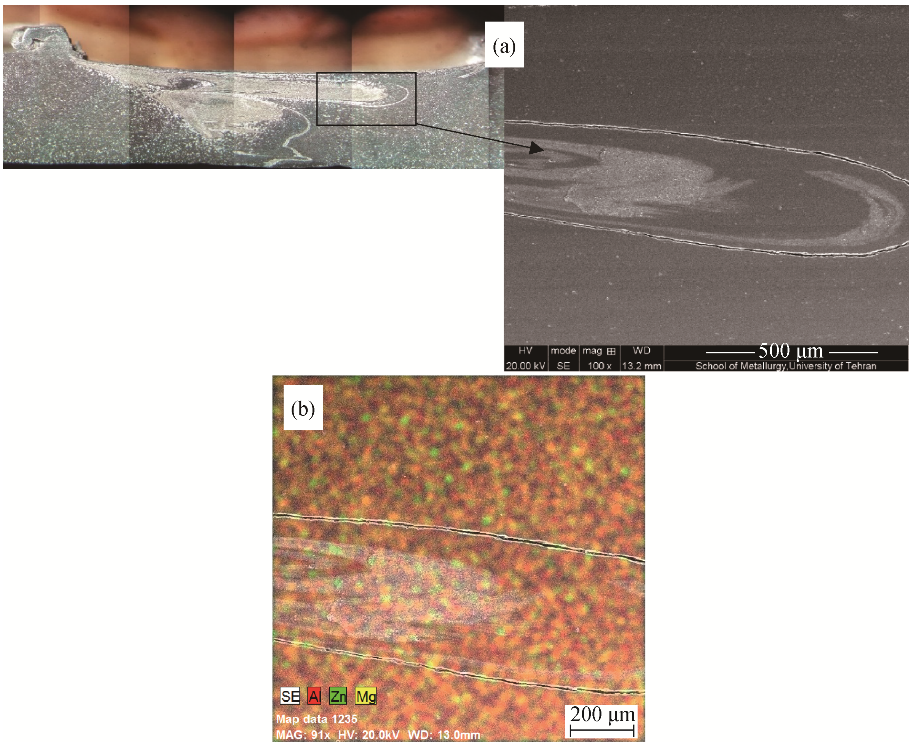

Fig. 7. SEM image (a) and elemental distribution map (b) of stir zone for F4.

of Zn element was not noticeable, indicating the insufficient distribution of zinc interlayer in the joint area. However, the presence of Zn was obvious in the cross section area of joint F1 (Fig. 8). A linear elemental analysis was also conducted across the joint area with elimination of Al concentration to enhance the visibility of Zn, Mg, and Cu elements. Evidently, the amount of Zn appeared in F4 was negligible (Fig. 9); therefore, the compositional state of F4 was not satisfactorily enough to obtain the concentration of 7xxx series. In contrast, the level of Zn across F1 specimen remarkably increased and provided a chemical composition identical to that of 7051 and 7039 Al alloys (Fig. 10). As a result, the joint area produced in F1 was similar to 7xxx series, while the base metal was 5754.

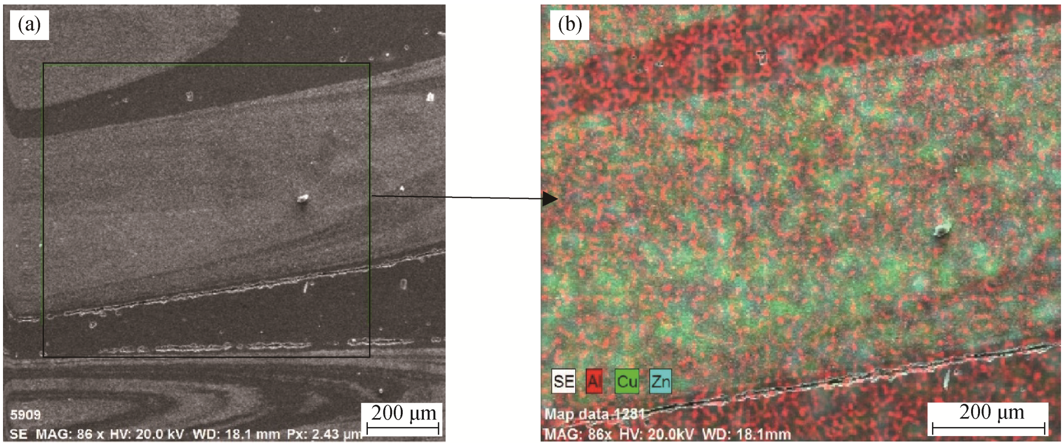

Fig. 8. SEM image (a) and elemental distribution map (b) of stir zone for F1.

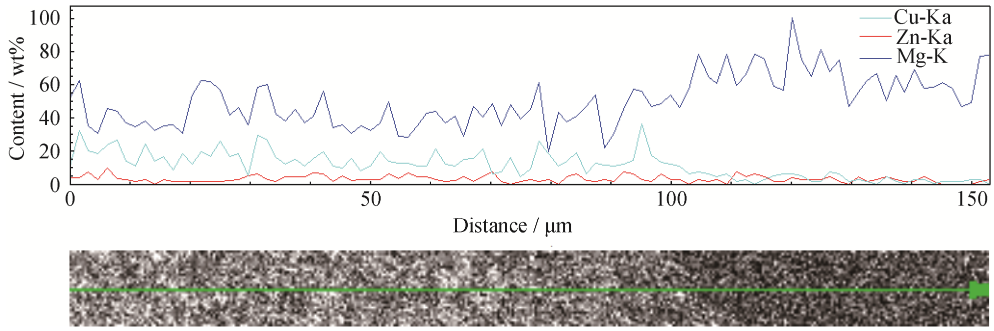

Fig. 9. EDS elemental profiles across the joint area of F4.

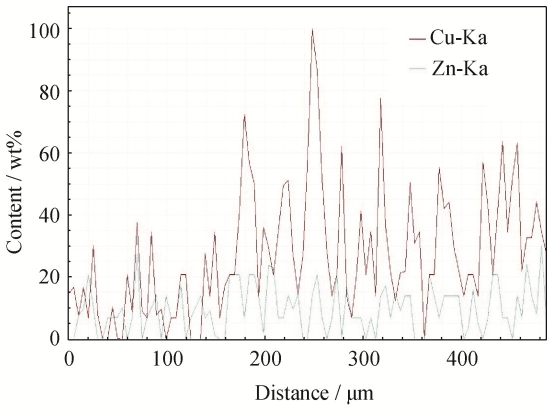

Fig. 10. EDS elemental profiles across the joint area of F1.

The impact of Zn foil addition on corrosion behavior of friction stir welds was studied by a cyclic polarization test. Specimens F1 and F4 were selected based on the results on microstructure. Moreover, specimen S2 was also exposed to corrosion test to thoroughly compare the Zn foil-free and Zn foil-added joints. Potentiodynamic polarization curve for each sample was sketched in Fig. 11 and their corresponding corrosion current (I_{corr}) and corrosion potential (E_{corr}), calculated by tangent method, are indicated in Table 3. As seen, the lowest I_{corr} is related to F1 with a value of 1.78 µA, implying the slowest corrosion rate of foil-added F1 specimen. However, specimen F4 showed the highest I_{corr} of 10.42 µA, which means its corrosion resistance was weaker than the S2 weld with I_{corr} of 3.77 µA. Friction stir welding has been reported to have a localized heterogeneous effect on microstructure, which makes a welded material susceptible to corrosion [37]. Thus, besides the insertion of Zn foil, the welding parameters, traveling and rotational speeds play a crucial role in determining corrosion properties [38].

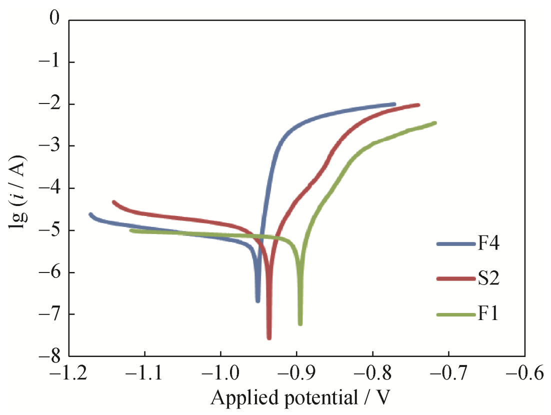

Fig. 11. Polarization curves of stir zone in three different joints.

Table 3. Results of potentiodynamic test

Specimen No.	E_{corr} / mV	I_{corr} / μA
F1	−896.3	1.78
S2	−951.1	3.77
F4	−937.8	10.42

4. Conclusions

In this study, 5754 aluminum alloy was friction stir welded, and the effect of Zn interlayer on microstructure and corrosion properties was investigated. The findings are as follows.

(1) Process parameters should be optimized to ensure a defect-free joint. In other words, the ratio of rotational speed to welding speed determines the uniformity of the welded joint.

(2) No defect was detected in the specimen welded with the help of Zn interlayer; therefore, the addition of Zn foil to the gap facilitated the removal of defects in the joint area.

(3) Although the impact of Zn on grain size was faint, the presence of zinc foil improved the chemical composition of the stir zone and led to a composition identical to that of 7xxx series.

(4) The highest corrosion resistance was achieved in the specimen with fully distributed Zn foil. In other words, a proper combination of welding parameters along with the addition of zinc layer increased resistivity to corrosive environment.

(5) The best sample was found to be F1, with SZ having a chemical composition of 7xxx aluminum alloy and highly improved corrosion properties. This sample was friction stir welded at a rotational speed of 800 r/min and traveling speed of 15 mm/min with an inserted Zn foil.

References

[1] J. Hirsch, Recent development in aluminum for automotive applications, *Trans. Nonferrous Met. Soc. China*, 24(2014), p. 1995.

[2] S.C. Ma, Y. Zhao, J.S. Zou, K. Yan, and C. Liu, The effect of laser surface melting on microstructure and corrosion behavior of friction stir welded aluminum alloy 2219, *Opt. Laser Technol.*, 96(2017), p. 299.

[3] T. Dursun and C. Soutis, Recent developments in advanced aircraft aluminum alloys, *Mater. Des.*, 56(2014), p. 862.

[4] R.S. Mishra and Z.Y. Mab, Friction stir welding and processing, *Mater. Sci. Eng. R*, 50(2005), No. 1-2, p. 1.

[5] G. Çam and G. İpekoğlu, Recent developments in joining of aluminium alloys, *Int. J. Adv. Manuf. Technol.*, 91(2017), No. 5-8, p. 1851.

[6] G. Çam and S. Mıstıkoğlu, Recent developments in friction stir welding of Al-alloys, *J. Mater. Eng. Perform.*, 23(2014), No. 6, p. 1936.

[7] G. Çam, Friction stir welded structural materials: beyond Al-alloys, *Int. Mater. Rev.*, 56(2011), No. 1, p. 1.

[8] G. Çam, G. İpekoğlu, T. Küçükömeroğlu, and S.M. Aktarer, Applicability of friction stir welding to steels, *J. Achiev. Mater. Manuf. Eng.*, 80(2017), No. 2, p. 65.

[9] G. Çam, H.T. Serindağ, A. Çakan, S. Mıstıkoğlu, and H. Yavuz, The effect of weld parameters on friction stir welding of brass plates, *Materialwiss. Werkstofftech.*, 39(2008), No. 6, p. 394.

[10] G. Çam, S. Mistikoglu, and M. Pakdil, Microstructural and mechanical characterization of friction stir butt joint welded 63%Cu–37%Zn brass plate, *Weld. J.*, 88(2009), No. 11, p. 225.

[11] T. Küçükömeroğlu, E. Şentürk, L. Kara, G. İpekoğlu, and G. Çam, Microstructural and mechanical properties of friction stir welded nickel–aluminum bronze (NAB) alloy, *J. Mater. Eng. Perform.*, 25(2016), No. 1, p. 320.

[12] S. Karami, H.R. Jafarian, A.R. Eivani, and S. Kheirandish, Engineering tensile properties by controlling welding parameters and microstructure in a mild steel processed by friction stir welding, *Mater. Sci. Eng. A*, 670(2016), p. 68.

[13] A.S. Zoeram, S.H.M. Anijdan, H.R. Jafarian, and T. Bhattacharjee, Welding parameters analysis and microstructural evolution of dissimilar joints in Al/Bronze processed by friction stir welding and their effect on engineering tensile behavior, *Mater. Sci. Eng. A*, 687(2017), p. 288.

[14] M.L. Saremi, S.E. Mirsalehi, and A. Shamsipur, Investigation on metallurgical structure and mechanical properties of dissimilar Al 2024/Cu FSW T-joints, *Trans. Indian Inst. Met.*, 70(2017), No. 7, p. 1869.

[15] H. Doude, J. Schneider, B. Patton, S. Stafford, T. Waters, and C. Varner, Optimizing weld quality of a friction stir welded aluminum alloy, *J. Mater. Process. Technol.*, 222(2015), p. 188.

[16] M. Bahrami, K. Dehghani, and M.K. Besharati Givi, A novel approach to develop aluminum matrix nano-composite em-

ploying friction stir welding technique, *Mater. Des.*, 53(2014), p. 217.

[17] G.H. Kumar, B. Vishwanath, R. Purohit, R.S. Ranad, and S.S. Rajpurohit, Mechanical behavior of friction stir welding on aluminum based composite material, *Mater. Today Proc.*, 4(2017), No. 4, p. 5336.

[18] G. Çam, S. Güçlüer, A. Çakan, and H.T. Serindağ, Mechanical properties of friction stir butt-welded Al-5086 H32 plate, *Materialwiss. Werkstofftech.*, 40(2009), No. 8, p. 638.

[19] Y. Bozkurt, S. Salman, and G. Çam, Effect of welding parameters on lap shear tensile properties of dissimilar friction stir spot welded AA 5754-H22/2024-T3 joints, *Sci. Technol. Weld. Joining*, 18(2013), No. 4, p. 337.

[20] G. İpekoğlu, S. Erim, and G. Çam, Investigation into the influence of post-weld heat treatment on the friction stir welded AA6061 Al-alloy plates with different temper conditions, *Metall. Mater. Trans. A*, 45(2014), No. 2, p. 864.

[21] G. İpekoğlu, S. Erim, B. Gören-Kıral, and G. Çam, Investigation into the effect of temper condition on friction stir weldability of AA6061 Al-alloy plates, *Kovove Mater.*, 51(2013), No. 3, p. 155.

[22] G. İpekoğlu and G. Çam, Effects of initial temper condition and postweld heat treatment on the properties of dissimilar friction-stir-welded joints between AA7075 and AA6061 aluminum alloys, *Metall. Mater. Trans. A*, 45(2014), No. 7, p. 3074.

[23] G. İpekoğlu, S. Erim, and G. Çam, Effects of temper condition and post weld heat treatment on the microstructure and mechanical properties of friction stir butt-welded AA7075 Al-alloy plates, *Int. J. Adv. Manuf. Technol.*, 70(2014), No. 1-4, p. 201.

[24] G. İpekoğlu, B. Gören-Kıral, S. Erim, and G. Çam, Investigation of the effect of temper condition friction-stir weldability of AA7075 Al-alloy plates, *Mater. Tehnol.*, 46(2012), No. 6, p. 627.

[25] D. Khayyamin, A. Mostafapour, and R. Keshmiri, The effect of process parameters on microstructural characteristics of AZ91/SiO_2 composite fabricated by FSP, *Mater. Sci. Eng. A*, 559(2013), p. 217.

[26] M. Abbasi Gharacheh, A.H. Kokabi, G.H. Daneshi, B. Shalchi, and R. Sarrafi, The influence of the ratio of ''rotational speed/traverse speed'' (ω/υ) on mechanical properties of AZ31 friction stir welds, *Int. J. Mach. Tools Manuf.*, 46(2006), p. 1983.

[27] M. Bahrami, M. Farahmand Nikoo, and M.K. Besharati Givi, Microstructural and mechanical behaviors of nano-SiC-reinforced AA7075-O FSW joints prepared through two passes, *Mater. Sci. Eng. A*, 626(2015), p. 220.

[28] V.K. Patel, S.D. Bhole, and D.L. Chen, Influence of ultrasonic spot welding on microstructure in a magnesium alloy, *Scripta Mater.*, 65(2011), No. 10, p. 911.

[29] M. Haghshenas, A. Abdel-Gwad, A.M. Omran, B. Gökçe, S. Sahraeinejad, and A.P. Gerlich, Friction stir weld assisted diffusion bonding of 5754 aluminum alloy to coated high strength steels, *Mater. Des.*, 55(2014), p. 442.

[30] F. Liu, D.X. Ren, and L.M. Liu, Effect of Al foils interlayer on microstructures and mechanical properties of Mg-Al butt joints welded by gas tungsten arc welding filling with Zn filler metal, *Mater. Des.*, 46(2013), p. 419.

[31] S.L. Yang, J. Zhang, J. Lian, and Y.P. Lei, Welding of aluminum alloy to zinc coated steel by cold metal transfer, *Mater. Des.*, 49(2013), p. 602.

[32] G. Çam, G. İpekoğlu, and H. Tarık Serindağ, Effects of use of higher strength interlayer and external cooling on properties of friction stir welded AA6061-T6 joints, *Sci. Technol. Weld. Joining*, 19(2014), No. 8, p. 715.

[33] R.Z. Xu, D.R. Ni, Q. Yang, C.Z. Liu, and Z.Y. Ma, Influencing mechanism of Zn interlayer addition on hook defects of friction stir spot welded Mg–Al–Zn alloy joints, *Mater. Des.*, 69(2015), p. 163.

[34] A. Abdolahzadeh, H. Omidvar, M.A. Safarkhanian, and M. Bahrami, Studying microstructure and mechanical properties of SiC-incorporated AZ31 joints fabricated through FSW: the effects of rotational and traveling speeds, *Int. J. Adv. Manuf. Technol.*, 75(2014), No. 5-8, p. 1189.

[35] M.W. Mahoney, C.G. Rhodes, J.G. Flintoff, W.H. Bingel, and R.A. Spurling, Properties of friction-stir-welded 7075 T651 aluminum, *Metall. Mater. Trans. A*, 29(1998), No. 7, p. 1955.

[36] B. Abnar, M. Kazeminezhad, and A.H. Kokabi, Effects of heat input in friction stir welding on microstructure and mechanical properties of AA3003-H18 plates, *Trans. Nonferrous Met. Soc. China*, 25(2015), No. 7, p. 2147.

[37] F. Gharavi, K.A. Matori, R. Yunus, N.K. Othman, and F. Fadaeifard, Corrosion behavior of Al6061 alloy weldments produced by friction stir welding process, *J. Mater. Res. Technol.*, 4(2015), No. 3, p. 314.

[38] U. Donatus, Corrosion behaviour of friction stir welded aluminium alloys: unpredictability and protection, *Res. Dev. Mater. Sci.*, 2(2017), No. 2, p. 532.