Gradient micro-structured surface layer on aluminum alloy fabricated by in situ rolling friction stir welding

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Abstract

A gradient micro-structure was formed in the surface layer of 2219 aluminum alloy joint by means of in situ rolling friction stir welding (IRFSW). The micro-structured surface layer is about 200 μm deep, corresponding to a gradient change in microhardness from 86.8 to 59.4 HV in the coarse-grained weld nugget zone (WNZ). Compared with those of the base material, the friction coefficient values are evidently decreased and the wear resistance is obviously enhanced on the surface layer. The corrosion current density decreased and the corrosion potential value was positive with respect to that of the base material. The second-phase particles in the upper surface layer were much more and smaller than those of the base material.

1. Introduction

In most cases, material failures occur on surfaces such as fatigue fracture, fretting fatigue, and wear and corrosion. These failures are very sensitive to the structure and properties of the material surface. To many materials, the low surface hardness and poor wear resistance has restricted their applications in engineering fields [1]. However, optimization of the surface microstructure and properties is an effective approach to enhance the global behavior and service lifetime of materials [2].

Deformation-induced grain refinement in the submicrometer or nanometer regimes has been extensively investigated in various metals and alloys over the past decades, both in bulk forms [3] and in surface layers on bulk materials [4]. Optimizing surface properties by refining grains in surface layers to the nanometer scale (referred to as surface nanocrystallization) provides more promising practical industrial applicability. Surface mechanical attrition treatment (SMAT) is a developed technique that can induce grain refinement into the nanometer regime in the surface layer of bulk materials [5]. The SMAT transforms the original coarse-grains at the surface material into refined-ones [6]. The grains refining mechanisms in SMAT is conducted by the random and repetitive impact of milling balls to the sample’s surface. This technique has been successfully applied in achieving surface nanocrystallization (SNC) in a variety of materials including pure metals, alloys and steels [7–10]. Another technique has been developed based on basically the principle of plastic deformation-induced grain refinement is friction sliding [11]. However, these two processes suffer from either low processing efficiencies or limited nano-structured layer thicknesses, or structural inhomogeneity in the surface layer, all of which have hindered the widespread application of surface nanocrystallization technologies [12].

In recent years, surface mechanical grinding treatment (SMGT) is developed to synthesize a gradient nano-micro-structure in the surface layer of bulk metals [12]. The SMGT technique is based on machining-induced plastic deformation and can achieve surface modification by generation of a nano-structured surface layer so that the overall properties and behavior of the materials are significantly improved. This technique requires only simple procedures and can be readily applied to engineering materials [12]. Friction stir processing technique refines the microstructure in the surface layer during which a large plastic strain is generated together with a substantial mass flow accompanied by a large, localized temperature rise [13]. This results in relatively large grain sizes, typically of the order of several hundred nanometers, via dynamic recrystallization [14]. FSP has proved to be a viable tool for enhancing the mechanical properties of materials, however, the major focus has been upon improving the bulk properties of light metals and their alloys [15,16].

A surface enhancement technology, Low Plasticity Burnishing (LPB), is also developed to produce a deep layer of highly compressive residual stress with a minimum amount of cold working, or plastic deformation [17]. It can provide compression in the surface layer of sufficient depth to effectively eliminate the degradation in corrosion/fatigue life attributed to salt pit corrosion [18]. Unlike LPB, conventional roller and ball burnishing utilize a hard wheel...
tool or fixed lubricated ball pressed into the surface of an axisymmetric workpiece with sufficient force to deform the near surface layers, usually in a lathe. Burnishing is performed with multiple passes, often under increasing load, to improve surface finish and to deliberately cold work the surface [18].

In this work, we develop a novel technique, namely in situ friction stir welding (IRFSW), to synthesize a gradient micro-structure in the surface layer of 2219 aluminum alloy joint. We report the processing of the IRFSW, the microstructure and grain refinement mechanism of the surface layer induced by the IRFSW, as well as properties of the micro-structured surface layer in 2219 aluminum alloy joint.

2. Experimental details

The material used is 2219 aluminum alloy (3 mm × 300 mm × 100 mm) with chemical compositions of (in wt%): 6.48 Cu, 0.32 Mn, 0.23 Fe, 0.06 Ti, 0.08 V, 0.04 Zn, 0.49 Si, 0.02 Zr and balance Al. Tensile strength and elongation of AA2219 rolled plate is 331 MPa and 11.7%, respectively. As shown in Fig. 1a, four rolling balls 8 mm in diameter are fastened to spherical grooves by brazing, and the rolling tool connects with the shoulder through bolt fastening with a rolling diameter of 26 mm. The rolling depth of rolling balls is controlled by the gasket and the thickness of each gasket is 0.05 mm. The welding tool is fabricated from tool steel and consisted of a shoulder with a diameter of 14 mm and a threaded conical pin 2.6 mm in length. The IRFSW process are finished using an FSW machine (FSW-3LM-003). As schematically illustrated in Fig. 1b, the IRFSW processing involves friction, stirring and rolling. The stir tool gets in touch with the interface of aluminum alloy sheet, rubs against the inner interface, and rolls the bonding surfaces which make the surface of weld seam produce severe plastic deformation in the welding processing. During the IRFSW processing, the rolling balls, shoulder and stir pin rotate at a rotation rate of \( \omega \), which slide along the welding line from front to back at a velocity of \( v \). With a preset penetration depth of the shoulder and rolling balls into the plate, \( h \), a plastic deformation zone is induced underneath the IRFSW tool.

To compare the erosion resistance of the base material and micro-structured surface layer, the samples were exposed to 3.5%NaCl solution at the room temperature. The corrosion current and potential were determined by electrochemical analyzer CHI604C which was comprised of electrochemical cell. It is three electrodes system in which the sheet is used as work electrode, the platinum piece is used as the auxiliary electrode and the saturated calomel electrode is used as reference electrode. In the test, cell current readings are taken during a slow sweep of the potential. The sweep was taken from \((-2 \text{ to } +1)\) V and the scan rate was 0.01 V/s. Corrosion potential is the thermodynamic parameter
which reflects the tendency of material corrosion, and corrosion current belongs to dynamic parameter which can reflect the corrosion rate.

The microhardness testing is conducted using a Vickers hardness testing machine (HX-1000) with a test load of 50 g and an indentation time of 10 s and the microhardness values are taken from the cross-section along the thickness direction in the surface layer of the joint. Nano-hardness tests were performed by a nano-indenter G200 with a maximum load of 10 g and peak hold time of 10 s. The tests of wear resistance are performed on ball-plate friction-wear testing machine (QP-1) produced by Harbin Institute of Technology [19], and the specimens were prepared with reference to ASTM: G115-10. The macrographs of joints are characterized by optical microscopy (OM, Olympus-MPG3) and the scanning electron microscope (Hitachi-S4700) is employed to capture a detailed view of the feature.

3. Results and discussion

3.1. Effect of welding parameters

From the previous research, we can know the LPB technique offers the opportunity to induce a layer of compressive residual stress into the weld surface [17]. In the IRFSW processing, the rolling balls are pressed into the sheet for rolling the weld seam. The most relevant process parameter is rolling depth which determines the form of the weld seam [20]. Photo of the surface of the conventional FSW seam is presented in Fig. 2a. It shows that the excessive weld flashes exited on the surface of the weld seam. We can also see that a large amount of plastic material is left on the surface when the rolling depth is more than 0.05 mm. The big rolling depth results in the serious outflow of the plastic material. However, as shown in Fig. 2c, most of the weld flashes are reduced compared with those of the conventional FSW, analogous to the LPB technique which can smooth the weld seam, when the rolling depth is 0.05 mm. As the ball rolls over the sheets, the lateral extrusion pressure from rolling object prevents the outflow of the plastic material and helps smooth surface asperities. When the rolling depth is less than 0.05 mm, the diameter of weld seam is smaller than that in Fig. 2c, as shown in Fig. 2d. This is because the rolling depth is so small that the rolling balls cannot keep in touch with the plates sufficiently. The rolling pressure from the rolling balls is associated with the quantity of the plastic material that flows out the original workpiece surface [20]. Taking into account of the plunge depth of the shoulder, the optimized rolling depth is 0.05 mm.
Fig. 3 shows the variation of depths of upper surface under the condition that the rolling depth increases. The depth of upper surface increases from 83 μm to 111 μm with the increment of rolling depth. This is because the rolling depth is associated with rolling pressure from the rolling balls. When the rolling depth increases, the increased rolling pressure makes the depth of upper surface deeper.

From our previous studies [20], IRFSW joints show superior tensile properties compared to conventional FSW joints. When the tool rotation rate \( \omega = 600 \text{ rev min}^{-1} \), the rolling depth 0.05 mm, and the plunge depth 0.1 mm is fixed, the tensile strength of optimized joint increases 13% and the elongation almost keeps the same compared with those of the conventional FSW. It can be attributed to the rolling balls which make the plastic material flow more fully and the grain refinement so that tensile strength is improved.

### 3.2. Microstructure of the surface layer

The cross-sectional structural characterization of the IRFSW-processed sample was carried out on an optical microscope, as shown in Fig. 4. The IRFSW processing parameters are as follows: rotation rate \( \omega = 600 \text{ rev min}^{-1} \), welding speed \( v = 300 \text{ mm min}^{-1} \), the rolling depth 0.05 mm and the plunge depth 0.1 mm. Plastic deformation is obvious in the surface layer to a depth exceeding 200 μm. Three obvious distinct zones (upper surface, middle

**Table 1**

<table>
<thead>
<tr>
<th>Indentation depth (μm)</th>
<th>Based material (100 s)</th>
<th>Upper surface (100 s)</th>
<th>Based material (200 s)</th>
<th>Upper surface (200 s)</th>
<th>Average depth (μm)</th>
</tr>
</thead>
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<tr>
<td>22.34</td>
<td>20.32</td>
<td>24.72</td>
<td>18.46</td>
<td>25.26</td>
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<td>13.39</td>
<td>25.55</td>
<td>14.98</td>
<td>29.91</td>
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<tr>
<td>29.91</td>
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<td>29.91</td>
<td>20.28</td>
<td>25.25</td>
<td>23.49</td>
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<tr>
<td>23.49</td>
<td>12.33</td>
<td>21.66</td>
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<td>25.46</td>
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surface and lower surface) exist in the surface layer of the joint. In the top 100 μm, the grains are the smallest in the whole joint, implying that this zone undergoes severe plastic deformation at high strain rates. It has recently been shown that, after passage of the tool pin, the upper part of the weld undergoes additional deformation by the rotating tool shoulder [21]. Excepting that, the upper surface layer of IRFSW-processed sample still goes through the severe deformation by rotating rolling balls. The middle layer undergoes the function of the stir pin and rotating tool shoulder, however, the lower layer only undergoes the function of the stir pin.

3.3. Hardness results

Fig. 5 shows the microhardness measurement results from the upper, middle and lower surface layers of the IRFSW-processed sample. Microhardness decreases from about 86.8 HV in the top surface layer to about 59.4 HV in the lower surface layer (the coarse-grained WNZ). The lower surface layer is directly influenced by the tool pin and is subjected to a high level of plastic deformation and frictional heating. The grains are refined in the lower surface layer. This refinement is the result of dynamic recrystallisation and combines action of high rate strain and elevated temperatures [22–24]. However, the upper surface layer is directly under the rolling tool and is plastically deformed by friction, stirring and rolling.

Nano-hardness and elastic modulus across the treated surface layer were also measured by using a nanoindenter with a Berkovich tip. Hardness decreases from about 0.89 GPa in the upper surface layer to about 0.78 GPa in the base material (Fig. 6a). According to the conventional machining process to remove material, severe plastic deformation takes place in the removed layer, analogous to that in equal channel angular pressing (ECAP) [25]. Thereby, similar microstructures are obtained in the removed material compared with the ultrafine structure in the ECAP samples [26]. There is plenty of evidence to indicate that a gradient microstructure composed of ultrafine was developed in a very thin layer on the upper surface layer of the IRFSW-processed sample. The elastic moduli values (about 69.1 GPa) of the base material and the upper surface layer of the IRFSW-processed sample are unchanged, independent of grain size in the present grain size regime, which is consistent with results in the literature [12].

3.4. Friction and wear properties

From the previous investigations [27,28], we can know that deformation twinning plays a key role in refining grains to the nano- and micro-meter scale via plastic deformation at high strain rates and low temperatures. Severe plastic deformation takes place in the upper surface layer of the IRFSW-processed joint due to the friction, stirring and rolling. However, a substantial temperature rise is unavoidable during conventional machining, which might be high enough to induce noticeable recrystallization and even phase transformation [29]. Owing to the high thermal conductivity of 2219 aluminum alloy, most of the transient temperature rise in the upper surface layer induced by friction might be rapidly cooled so that the recovery or recrystallization processes in the deformed layer could be effectively suppressed. Fig. 7 shows comparisons of the indentation width profiles of base material and IRFSW-processed sample. Fig. 7a and b are the indentation width profiles with 100 s function of ball-plate friction-wear testing machine (QP-1) and the indentation width (average 781.72 μm) of the base material is much bigger than that (average 606.43 μm) of the top surface layer. Fig. 7c and d are the indentation width profiles of samples that undergoes 200 s function of the QP-1 machine and the indentation width (average 778.64 μm) of the base material is much bigger than that (average 653.41 μm) of the top surface layer.

According to the pythagorean theorem, as well as the indentation widths (in Fig. 7) and the diameter of the ceramic ball (R = 3 mm), as shown in Fig. 8, we can calculate the indentation depths of the ceramic ball on the surfaces of base material and IRFSW-processed sample, which is shown in Table 1. Friction and wear property measurements indicated a significant increment of the wear resistance in the surface layer with micro-structures after the IRFSW. When base material and IRFSW-processed sample both undergo 100 s function of ball-plate friction-wear testing machine (QP-1), the average indentation depth of IRFSW-processed sample is reduced to 15.60 μm compared with that (25.25 μm) of the base material. Under the condition of 200 s function of QP-1 machine, the average indentation depth of IRFSW-processed sample is reduced to 17.90 μm compared with that (25.46 μm) of the base material.

As shown in Fig. 9, the friction coefficient values of the micro-structured in surface layer of the IRFSW-processed sample are lower than that for the base material. In this test, the test load was 200 g and the rotation speed was 50 rpm. To assure the same surface finish, we polish the surfaces of the base material and the
IRFSW-processed sample with water sandpapers and abrasive papers. It was found that friction coefficient values at different number of wear cycles for the IRFSW-processed sample were evidently smaller than those for the original sample. Especially when the number of wear cycles was 30, the friction coefficient values for the base material suddenly increased. However, the friction coefficient values for the IRFSW-processed sample can remain basically unchanged till the number of wear cycles gets to 500. These observations indicate that the friction and wear properties of the IRFSW-processed sample can be improved by means of the formation of the micro-structured surface layer. The improved wear properties can be attributed to the strong surface layer with micrograins and a gradient variation in the microstructure and properties along the depth from top surface.

3.5. Electrochemical property

Friction stir welds of some aluminum alloys exhibit relatively poor corrosion resistance [30], however, the IRFSW technique enhanced the corrosion properties through imparting additional (secondary) strain into the upper part of the weld. The potentiodynamic polarization curves of micro-structured surface layer in the IRFSW-processed sample and the base material are shown in Fig. 10. The more electro-negative the corrosion potential, the higher the tendency to erosion, and the higher the corrosion current, the more rapid the rate of erosion. The corrosion potential of the IRFSW-processed sample is found to be negative with respect to that of base material. The result also shows that the corrosion current of the IRFSW-processed sample is found to be negative with respect to that of base material. The result also shows that the corrosion current of the IRFSW-processed sample is found to be lower than that of the base material. These observations indicate that the corrosion and wear properties of the IRFSW-processed sample can be improved by means of the formation of the micro-structured surface layer. The improved wear properties can be attributed to the strong surface layer with micrograins and a gradient variation in the microstructure and properties along the depth from top surface.

3.6. SEM observation

The typical patterns of second-phase particle distributions in the upper surface layer and base material are shown in Fig. 11. The second-phase particles were of nearly equiaxed shape and were randomly distributed in the microstructure. The mean second-phase particle size in the base material is much bigger than that in the top surface layer. The second-phase particles can effectively pin grain growth [32], and thus contribute to the formation of a fine-grained microstructure in the surface layer of the IRFSW-processed sample. The improved wear properties of the surface layer can have big improvements.

In the IRFSW processing, the material in the surface layer undergoes the friction, stirring and rolling induced by the tool shoulder and rolling balls. It has been reported that the rotating shoulder entraps the surface layer of the base material during FSW and deposits it far behind the pin [21]. During the deposition process, the rolling balls impart additional (secondary) strain into the upper part of the weld. The material in the surface layer experiences perhaps the largest strain and a rapid heat sink exists in the surface of the joint which can prevent the grain growth. These may give rise to the surface layer with a very fine recrystallized grain structure.

4. Conclusions

In summary, a novel surface treatment technique, IRFSW, is developed to achieve a gradient micro-structured surface layer on 2219 aluminum alloy. The micro-structured surface layer provides a plenty of unique opportunities in both basic scientific research and technological applications. On the basis of present investigation, the following conclusions are reached.

(1) A micro-structured surface layer about 200 μm deep, consisted of upper surface, middle surface and lower surface, is formed in the 2219 aluminum alloy joint. Microhardness decreases from 86.8 HV in the top surface layer to 59.4 HV in the lower surface layer. The mechanical, tribological, chemical and corrosion properties of gradient micro-structured surface layer have been enhanced.

(2) The upper surface layer undergoes a severe plastic deformation and experiences the largest strain induced by the tool shoulder and rolling balls via friction, stirring and rolling. The second-phase particles were randomly distributed in the microstructure and can effectively pin grain growth.
(3) The IRFSW shows an alternative approach to effectively upgrade the global properties of engineering materials without change of the chemical constitution. As the IRFSW is simple and flexible, this new technique is potentially useful in industrial applications.

Acknowledgements

The work was jointly supported by the National Natural Science Foundation of China (No. 50904020), and the Fundamental Research Funds for the Central Universities (No. HIT. NSRIF. 2012007).

References