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New insight into the phenomenon of the abnormal grain growth in friction-stir welded aluminum

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ABSTRACT

Keywords: Aluminium alloys Friction-stir welding Abnormal grain growth Electron backscatter diffraction (EBSD) Microstructure Texture In this work, the annealing behavior of friction-stir welded aluminum alloy 6061 was studied. Due to essential grain refinement and dissolution of secondary particles, this material usually exhibits relatively low stability against abnormal grain growth during post-weld solutionizing treatment. In the present study, it was found that the abnormal microstructural coarsening actually develops during the heating stage of the tempering process, i. e., during material warming from the ambient condition to the solutionizing temperature.

1. Introduction

A relatively low microstructural stability against abnormal grain growth is an essential issue in friction-stir welding (FSW) of heattreatable aluminum alloys [1]. This phenomenon involves a catastrophic coarsening of a few grains, which eventually consume almost the entire weld zone. This undesirable effect has been observed to occur during post-weld heat-treatment of 2xxx [2], 6xxx [3], and 7xxx [4,5] series of aluminum alloys thus perhaps being an intrinsic characteristic of FSW. The abnormal grain growth in friction-stir welded metals is often explained in terms of the Humphreys cellular model [6], thus being virtually attributed to the combined effect of the drastic grain refinement and the dissolution of the constituent second-phase particles both occurring during FSW [2,4,5].

In the previous work [7], it has been suggested that the abnormal grain growth in the friction-stir welded heat-treatable aluminum alloys basically occurs during the *heating stage* of the post-weld solutionizing treatment, i.e., during warming of the welded material from the ambient condition to the solutionizing temperature. If this is indeed the case, this peculiarity may provide a new insight into the abnormal grain growth phenomenon and thus may be helpful to elucidate its fundamental mechanism(s). Therefore, the present study was undertaken in order to explore this hypothesis.

2. Experimental

The material used in the present investigation was a commercial 6061 aluminum alloy supplied as a hot extruded bar. To obtain the precipitation-hardened condition, the received material was undergone the T6 tempering, i.e., solutionized at 540°C, water quenched and then artificially aged at 160°C for 8 h. The produced material was denoted as *base material* throughout this manuscript.

The sheets of the base material of 3 mm in thickness were but-welded using a commercial AccurStir FSW machine. The welding tool had a conventional design including a concave-shaped shoulder of 12.5 mm in diameter, and a threaded M5 cylindrical probe of 2.7 mm in length. To examine a possible influence of FSW variables, two different welding trials were conducted at low- and high-heat-input conditions, as indicated in Table 1. The typical convention for FSW geometry was used with WD, ND, and TD being the welding-, normal-, and transverse directions of the welded plates, respectively. Further details of FSW procedure have been described elsewhere [8].

To simulate the heating stage of the solutionizing treatment, the following procedure was adopted. The welded specimens with attached K-type thermocouples were placed in a muffle furnace heated to the solutionizing temperature (i.e., 540° C), and their thermal history was continuously recorded, as shown in supplementary Fig. S1. Some scattering in the heating rate seen in the supplementary figure was associated with an imprecision of the procedure of placement of specimens into the furnace. When a particular specimen was heated to a desired

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Table 1

FSW conditions studied in the present work.

Weld definition	Tool rotation rate, rpm	Tool travel speed, mm/min
Low-heat-input weld	500	380
High-heat-input weld	1100	125

temperature, (i.e., 350° C, 400° C, 425° C, 450° C, 500° C, 525° C, or 540° C), it was immediately quenched in water to preserve the evolved microstructure.

Following the heat treatment, the produced microstructures were studied by electron backscatter diffraction (EBSD) technique. The suitable surface finish was obtained by mechanical polishing in a conventional fashion followed by electro-polishing in a 25% solution of HNO₃ in CH₃OH. To provide a thorough insight into microstructural changes, the sample-scale EBSD maps were acquired across the entire weld zone using a FEI Quanta 600 field-emission-gun scanning electron microscope equipped with TSL OIMTM EBSD system and operating at an accelerating voltage of 20 kV. The scan step sizes of 1 µm and 5 µm were used in the

as-welded and annealed material conditions, respectively. To enhance reliability of the collected data, the fine grains comprising two or one pixels were automatically removed from the EBSD maps using a standard grain-dilation option. Considering the limited angular accuracy of EBSD, a lower-limit misorientation cut-off of 2° was employed. A 15° criterion was used to differentiate low-angle boundaries (LABs) from high-angle boundaries (HABs).

3. Results and discussion

The sample-scale EBSD maps taken from the low- and high-heatinput welds are summarized in Figs. 1 and 2, respectively. Grain size distributions measured in the central section of the low-heat-input weld are shown in supplementary Fig. S2. In the as-welded condition, a distinct basin-shaped stir zone with drastically refined microstructure was clearly seen in both joints (Figs. 1a and 2a). The mean grain size in the stir zone was measured to be $\approx 2 \,\mu\text{m}$ and $\approx 9 \,\mu\text{m}$ in the low- and highheat-input welds, respectively [8]. The essentially inhomogeneous texture distribution within the stir zone is also worthy of remark.



Fig. 1. Sample-scale EBSD maps showing microstructure of the low-heat-input weld (a) in the as-FSWed condition, and after subsequent heating to (b) 450°C, (c) 500°C, (d) 540°C, or (e) after 1-hour static storage at 540°C. In the maps, individual grains are colored according to their crystallographic orientations relatively welding direction (the color-code triangle is given in the top right corner). In all cases, the retreating side is on the left and the advancing side is on the right.



Fig. 2. Sample-scale EBSD maps showing microstructure of the high-heat-input weld (a) in the as-FSWed condition, and after subsequent heating to (b) 500°C, (c) 525°C, (d) 540°C, or (e) after 1-hour static storage at 540°C. In the maps, individual grains are colored according to their crystallographic orientations relatively welding direction (the color-code triangle is given in the top right corner). In all cases, the retreating side is on the left and the advancing side is on the right.



Fig. 3. High-magnification EBSD maps showing abnormal grain growth at the (a) advancing side of the low-heat-input weld heated to 450°C, and (b) upper surface of the high-heat-input weld heated to 500°C. In both maps, individual grains are colored according to their crystallographic orientations relatively welding direction; LABs and HABs are depicted as white- and black lines, respectively. The common color-code triangle and reference frame for both maps are given in the bottom right corner of (a) and (b), respectively.

In the *low-heat-input weld*, optical observations revealed no substantial changes in grain structure during heating up to 425°C provided no substantial changes in grain structure. However, an increase of the heating temperature to 450°C gave rise to the abrupt microstructural coarsening which encompassed the almost entire stir zone (Fig. 1b). A small fraction of the original fine-grained structure retained only at its advancing side (Fig. 3a). Considering a characteristic bimodal microstructural morphology in this area, it was deduced that the grain growth was governed by the abnormal mechanism. After heating to 500°C, the abnormal grains eventually consumed the stir zone (Fig. 1c). The further heating to 540°C (Fig. 1d) as well as the subsequent 1-hour storage at this temperature (Fig. 1e) resulted in relatively small microstructural changes.

In the high-heat-input weld, the first evidences of the microstructural coarsening were observed only after heating to 500°C (Fig. 2b). It is important to emphasize, however, that the grain growth developed only locally, at the upper surface of the stir zone, and also had an abnormal character (Fig. 2b, 3b). With the increasing of the heating temperature to 540°C, the abnormal grains tended to propagate in a downward direction, thus gradually consuming the stir zone (Fig. 2c and d). At this temperature, the abnormal grains were also observed to nucleate at the weld root, whereas the microstructure of the central (nugget) region of the stir zone remained relatively stable (Fig. 2d). Therefore, the abnormal coarse grains preferentially nucleated at the stir zone periphery, in good agreement with scientific literature [2,5]. After reaching the solutionizing temperature, the process of the abnormal grain growth in the stir zone has nearly completed (Fig. 2d). The further 1-hour static storage at this temperature resulted in only minor microstructural changes (Fig. 2e).

From comparison of the annealing behavior of the low- and highheat-input welds, it was clear that the latter joint exhibited superior thermal stability. This agreed well with literature data [2,5]. On the other hand, it was also worth noting that the fully-annealed high-heatinput weld was characterized by the markedly coarser abnormal grains than the low-heat-input one (compare Figs. 1 and 2e and e). The origin of this effect is not clear, however.

Therefore, the abnormal grain growth in both studied welding conditions has indeed completed during the heating stage of the solutionizing treatment, in full accordance with the initial suggestion of this study. This effect is thought to be associated with the fine-grained nature of the stir zone microstructure. According to Humphreys et al [9], the gross grain boundary migration in fine-grained aluminum initiates above 250°C, i.e., well below the solutionizing temperature. On the other hand, as the studied material is a *heat-treatable* alloy, it is quite likely that its microstructural coarsening may be additionally influenced by the precipitation phenomena (which presumably also occur during the heating stage). This issue obviously warrants further study.

4. Conclusions

- (1) The abnormal grain growth, which is often observed to occur in friction-stir welded 6061 aluminum alloy during post-weld solutionizing annealing, actually develops during the heating stage of the tempering treatment, i.e., during material warming from the ambient condition to the solutionizing temperature. The subsequent 1-hour storage at the solutionizing temperature results in only minor microstructural changes.
- (2) In the *low-heat-input* welds, the stir zone microstructure was characterized by the relatively low resistance against the abnormal microstructural coarsening, but the final size of the abnormal grains was relatively small. In contrast, the *high-heat-input* welds exhibited superior thermal stability. In this case, the abnormal grains nucleated at comparatively high temperatures and only locally, preferentially at the upper surface of the stir zone. Then, those propagated in a downward direction, thus gradually consuming the remaining stir zone. As a result, the final size of the abnormal grains achieved a mm-scale.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matlet.2021.130407.

References

R.S. Mishra, Z.Y. Ma, Friction stir welding and processing, Mater. Sci. Eng. R 50 (1-2) (2005) 1–78.

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- [2] M.M. Attallah, H.G. Salem, Friction stir welding parameters: A tool for controlling abnormal grain growth during subsequent heat treatment, Mater. Sci. Eng. A 391 (2005) 51–59.
- [3] I. Vysotskiy, S. Malopheyev, S. Mironov, R. Kaibyshev, Pre-strain rolling as an effective tool for suppression of abnormal grain growth in friction-stir welded 6061 aluminum alloy, Mater. Sci. Eng. A 733 (2018) 39–42.
- [4] K.A.A. Hassan, A.F. Norman, D.A. Price, P.B. Prangnell, Stability of nugget zone grain structure in high strength Al-alloy friction stir welds during solution treatment, Acta Mater. 57 (2003) 1923–1936.
- [5] I. Charit, R.S. Mishra, Abnormal grain growth in friction stir processed alloys, Scripta Mater. 58 (5) (2008) 367–371.
- [6] F.J. Humphreys, A unified theory of recovery, recrystallization and grain growth, based on the stability and growth of cellular microstructures—II, The effect of second-phase particles, Acta Mater. 45 (12) (1997) 5031–5039.
- [7] I.S. Zuiko, S. Mironov, S. Betsofen, R. Kaibyshev, Suppression of abnormal grain growth in friction-stir welded Al–Cu–Mg alloy by lowering of welding temperature, Scripta Mater. 196 (2021), 113765.
- [8] A. Kalinenko, K. Kim, I. Vysotskiy, I. Zuiko, S. Malopheyev, S. Mironov, R. Kaibyshev, Microstructure-strength relationship in friction-stir welded 6061–T6 aluminum alloy, Mater. Sci. Eng. A 793 (2020), 139858.
- [9] F.J. Humphreys, P.B. Prangnell, J.R. Bowen, A. Gholinia, C. Harris, Developing stable fine-grain microstructures by large strain deformation, Phil. Trans. R. Soc. Lond. A 357 (1756) (1999) 1663–1681.