



Microstructure and mechanical behavior of friction stir processed ultrafine grained Al–Mg–Sc alloy

N. Kumar^a, R.S. Mishra^{a,*}, C.S. Huskamp^b, K.K. Sankaran^b

^a Department of Materials Science and Engineering, Missouri University of Science and Technology, Rolla, MO 65409, USA

^b Boeing Research and Technology, The Boeing Company, St. Louis, MO 63166, USA

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ABSTRACT

Twin-roll cast (TRC) Al–Mg–Sc alloy was friction stir processed (FSP) to obtain ultrafine grained (UFG) microstructure. Average grain size of TRC alloy in as-received (AR) condition was $19.0 \pm 27.2 \mu\text{m}$. The grain size reduced to $0.73 \pm 0.44 \mu\text{m}$ after FSP. About 80% of the grains were smaller than $1 \mu\text{m}$ in FSP condition. FSP resulted into 80% of the grain boundaries to have high angle grain boundary (HAGBs) character. Uniaxial tensile testing of UFG alloy showed an increase in yield strength (YS) and ultimate tensile strength (UTS) (by $\sim 100 \text{ MPa}$ each) of the alloy with a very marginal decrease in total and uniform elongation (total – 27% in AR and 24% in UFG and uniform – 19% in AR and 14% in UFG). A theoretical model predicted that the grain refinement cannot take place via discontinuous dynamic recrystallization. Zener pinning model correctly predicted the grain size distribution for UFG alloy. From work hardening behaviors in both the conditions, it was concluded that grain boundary spacing is more important than the character of grain boundaries for influencing extent of uniform deformation of an alloy.

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1. Introduction

After the pioneering work of Hall [1] and Petch [2] who showed the dependence of strength on grain size, researchers have incessantly tried obtaining very fine grains to improve the strength of metals and alloys. Based on the size scale of grains, materials have been classified as microcrystalline ($>1 \mu\text{m}$), ultrafine grained (UFG) ($100 \text{ nm} - 1 \mu\text{m}$) and nanocrystalline ($<100 \text{ nm}$). UFG alloys have been subject of extensive research in last two decades because of following reasons: (a) relatively easy accessibility of UFG regime by the present processing techniques, (b) Hall–Petch relationship predicts that such materials should show very high strength, (c) low temperature and/or high strain rate superplasticity due to very fine grains.

Traditionally, UFG alloys have been processed using severe plastic deformation (SPD) techniques such as equal channel angular pressing (ECAP) [3,4], high pressure torsion (HPT) [5], accumulative roll bonding (ARB) [6,7]. Although these processing methods have successfully produced UFG alloys, they tend to introduce relatively large fraction of low angle grain boundaries (LAGBs) [5,8–14]. Also, due to the presence of high density of extrinsic grain boundary dislocations, high angle grain boundaries (HAGBs) have non-equilibrium structure [15,16]. Gertsman et al. [17] showed a

considerable change in mechanical behavior of UFG Cu before and after annealing at 473 K for 3 min due to recovery of extrinsic dislocations present at HAGBs.

Friction stir processing (FSP) is a relatively late entrant in the list of SPD processing technique. FSP is a derivative of friction stir welding which was developed at TWI in 1991 by Thomas et al. [18]. FSP was introduced by Mishra et al. [19] in 1999 when they showed high strain rate (HSR) superplasticity in a FSP 7075Al alloy. The details of this process can be found elsewhere [20,21]. This process has been extensively used for microstructural modification including grain refinement by various researchers in last one decade. FSP has shown potential to produce UFG microstructure with more than 90% HAGBs and entirely random texture [22,23]. Also, due to high temperature involved in this processing (usually $400 - 500^\circ\text{C}$ for Al-alloys), HAGBs are expected to have equilibrium structure.

The aim of the present research was two-fold: (a) to produce an UFG alloy having homogeneous, equiaxed grains with a high fraction of HAGBs, (b) to investigate mechanical properties of the UFG alloy containing such a high fraction of HAGBs.

2. Experimental

Twin-roll cast (TRC) Al–Mg–Sc–Zr sheet of $\sim 3.75 \text{ mm}$ thickness (nominal composition – Al–4 wt.%Mg–0.8%Sc–0.08%Zr) produced at CSIRO, Australia, was subjected to a single pass FSP. A FSP tool made of tool steel and having step-spiral, conical pin profile and concave shoulder was used to process the sheet material. The shoul-

* Corresponding author. Tel.: +1 573 341 6361; fax: +1 573 341 6934.

E-mail address: rsmishra@mst.edu (R.S. Mishra).

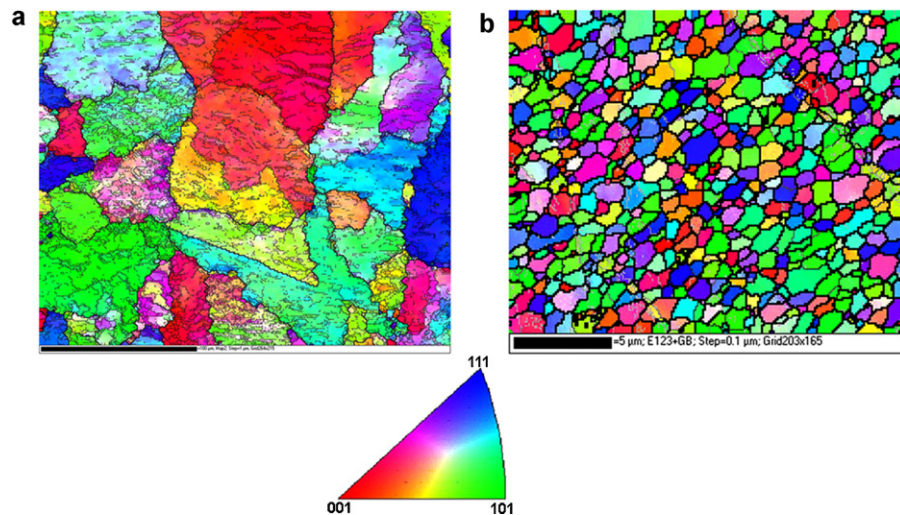


Fig. 1. OIM images of TRC sheets in (a) as-received and (b) FSP conditions. Different colors in the OIM images represent different crystallographic axes parallel to either the twin-roll casting or FSP direction. The crystallographic axes corresponding to various colors can be inferred from the inverse pole figure triangle shown in the figure.

der diameter, pin height, and pin diameter at the tip and root (where pin and shoulder meet) of the pin were 12.0 mm, 2.20 mm, 3.75 mm, and 6.00 mm, respectively. A plunge depth of 2.30 mm was used to process the material. The tool rotational rate and tool traverse speed used were 400 revolutions per minute (rpm) (or 6.6 revolutions per second) and 8 in/min (ipm) (or 3.4 mm/s), respectively. A tool tilt angle of 2.5° was maintained during the processing of the material. Electron backscattered diffraction (EBSD) microscopy was carried out using HKL EBSD system fitted on FEI Helios NanoLab 600 FIB/FESEM to characterize microstructure in as-received (AR) and FSP conditions. A computer controlled custom-built mini-tensile testing machine was used to study the mechanical behavior of the alloy in AR and FSP conditions under uniaxial tensile mode of deformation at the initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ and room temperature. The gage length, width, and thickness of the sample used were 5.0 mm, ~ 0.9 mm, and ~ 0.9 mm, respectively, in AR condition. The gage length, width, and thickness in FSP condition were 5.0 mm, ~ 1.2 mm, and ~ 1.0 mm, respectively.

3. Results

The OIM images corresponding to AR and FSP conditions are shown in Fig. 1. Inverse pole figure (IPF) color coding was used to represent various grains. Different colors represent various crystallographic directions parallel to either twin rolling or FSP direction. Average grain size for TRC alloy was $19 \pm 27 \mu\text{m}$. The reason for standard deviation (S.D.) to be larger than average grain size was large variability in the grain size. The largest grain size was found to be $122 \mu\text{m}$ in TRC condition. After FSP of TRC sheet, the average grain size refined to $0.73 \pm 0.44 \mu\text{m}$. Hence, FSP caused not only a considerable reduction in average grain size but also resulted in a tighter distribution of grains as evident from a smaller S.D. in FSP compared to that of AR condition.

Cumulative frequency (%) vs. grain size plot in Fig. 2 shows the grain size distribution for AR and FSP conditions. It is clear from this plot that about 70% of the grains were smaller than $19 \mu\text{m}$, the average grain size in AR condition. It also shows that about 14% of the grains were larger than $50 \mu\text{m}$. As mentioned earlier, this segment of larger grains resulted into the S.D. to be larger than the average size. The cumulative frequency distribution of grain size for FSP condition has been shown in the same figure. For more clarity by reducing the grain size scale it has been included as an inset in Fig. 2. In this case, the largest grain size was found to be $2.5 \mu\text{m}$.

Also, it can be noted that about 80% of grains were less than $1 \mu\text{m}$ in size.

Misorientation angle distribution (MAD) histograms for misorientations $>2^\circ$ have been shown in Fig. 3. Fig. 3a depicts the MAD of AR alloy. The histogram in Fig. 3b illustrates the distribution of misorientation angles across grains in FSP alloy. Fig. 3a shows a very high fraction of LAGBs. About 87% of the grain boundaries had LAGB character. The average misorientation angle in this condition was 9.0° . For FSP alloy only 20% of the grains had LAGB character. The average misorientation angle was found to be 34.2° . Clearly, FSP of TRC alloy led to the development of a microstructure containing very high fraction of HAGB.

The representative engineering stress–strain curves are given in Fig. 4. The alloy in AR condition showed a yield strength (YS) and ultimate tensile strength (UTS) of $204 \pm 4 \text{ MPa}$ and $293 \pm 4 \text{ MPa}$, respectively. The total elongation to fracture (e_f) and uniform elongation (e_u) were $27.2 \pm 1.2\%$ and $18.6 \pm 0.0\%$, respectively. After FSP, YS and UTS values changed to $319 \pm 1 \text{ MPa}$ and $384 \pm 10 \text{ MPa}$, respectively. Although there was $\sim 100 \text{ MPa}$ change in YS and UTS values after FSP, there was very little decrease in e_f and e_u . After FSP e_f and e_u were $24.4 \pm 0.3\%$ and $14.3 \pm 0.2\%$, respectively.

As can be noted from the stress–strain curves (Fig. 4), the alloy in AR condition showed a gradual transition from elastic to plastic

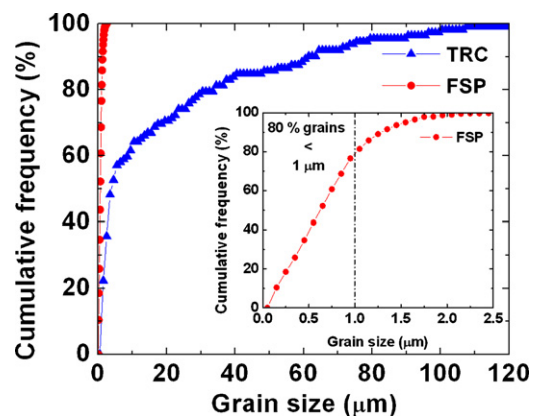


Fig. 2. Cumulative grain size distribution plot showing distribution of grains in TRC and FSP alloys. Inset of the graph shows the expanded view of the distribution of grains in FSP condition.

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