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Friction stir welding of an Al-Mg-Sc-Zr alloy with ultra-fined grained structure

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Effect of friction stir welding (FSW) on mechanical properties and microstructure of Al-5.4Mg-0.2Sc-0.1Zr sheets with ultra-fined grained (UFG) structure was studied. The UFG-sheets were produced by equal-channel angular pressing (ECAP) followed either by cold or hot rolling. FSW was found to be very effective for retaining the UFG microstructure as well as constituent coherent nano-scale dispersoids in the welded material. Despite the preservation effect, however, the essential material softening was observed in the weld zone. This was attributed to the recrystallization occurring during FSW. The joint efficiency for yield strength of the obtained friction stir welds was found to be 81% in the hot rolled condition and only 55% in the cold rolled state. The relatively low joint efficiency was associated with the recrystallization softening as well as with the formation of a specific "kissing bond" defect in the stir zone. The joint efficiency is believed may be improved by adjusting of welding conditions and/or tool design.

Introduction

Al-Mg alloys are widely used as non-heat-treatable aluminum alloys [1]. Minor additions of Sc and Zr lead to precipitation of coherent nano-scale $Al_3(Sc,Zr)$ dispersoids, which significant raise strength characteristics with retaining ductility at sufficiently high level as well as promote microstructure stabilization [2]. The superior properties of these alloys, however, may further be improved due to the formation of ultra-fine grained (UFG) microstructure by severe plastic deformation (SPD) [3]. Among various SPD methods, equal-channel angular pressing (ECAP) appears to be particularly attractive due to its relative simplicity as well as ability to produce UFG structure in large-scale billets [4]. This enables to combine this technique with conventional rolling for commercial production of sheets with UFG structure [4-6].

It is important to point out however that practical application of Al-Mg-Sc alloys with UFG structure relies heavily on their ability to be successfully welded. The conventional fusion welding technologies cannot retain UFG structure and thus inevitably lead to degradation of the unique properties of these materials. In this context, friction-stir welding (FSW) appears to be particularly attractive for joining of the UFG materials. Due to solid-state nature of the FSW process, it may avoid significant microstructure coarsening (as well as dissolution of the unique coherent dispersoids) and thus preserve high level of service properties [7,8].

The purpose of this work was to examine the feasibility of FSW for joining of Al-Mg-Sc-Zr alloy with UFG microstructure.

Experimental

The commercial alloy with a chemical composition of Al-5.4Mg-0.37Mn-0.2Sc-0.09Zr-0.29Ti-0.07Fe-0.04Si (in wt.%) denoted as 1570C was produced by semi-continuous casting, homogenized at 360°C for 8 hours and then extruded at 380°C with ~75% reduction. The obtained billet was subjected to ECAP via route B_C at 300°C to a true strain ~12 using isothermal die with 90° square channel. After ECAP, the material was finally rolled either at ambient or 300°C to a total thickness reduction of ~80%. Excluding cold rolling, the material was immediately water quenched after each thermo-mechanical processing step.

After rolling, the sheets were but joined using double-side FSW at the tool rotation speed of 500 rpm and the tool travel speed of 75 mm/min. The welding tool was fabricated from a tool steel and consisted of a shoulder with a diameter of 12.5 mm and M5 cylindrical pin of 1.5 mm in length. The angle between sheet normal and tool was 2.5°.

Microstructural observations were performed by optical microscopy, electron backscatter diffraction (EBSD) technique and transmission electron microscopy (TEM). The final surface finish for EBSD and TEM was obtained by electro-polishing in a solution of 25% nitric acid in methanol. The EBSD analysis was conducted using a FEI Quanta 600 field-emission-gun scanning electron microscope (FEG-SEM) equipped with TSL OIM^{TM} software. In the EBSD maps shown, low-angle boundaries (LABs) ($2^o<\theta<15^o$) and high-angle boundaries (HABs) ($\geq 15^o$) were depicted as white and black lines, respectively. The TEM observations were performed by using a JEM-2100EX TEM operating at 200 kV.

Results and Discussion

Microstructure of sheets. Typical microstructures obtained after hot and cold rolling are shown in Figure 1. The microstructure of the *hot* rolled UFG sheet consisted of grains with dimensions of ~1.3 μm in the rolling and ~1 μm in the sheet normal directions (Fig. 1a and Table 1). The grains contained a poorly developed substructure consisting of nearly equiaxed subgrains with a mean size of ~0.5 μm (Fig. 1b and Table 1).The HAB fraction was ~74%. The dislocation density was measured to be high ($\rho \sim 7\times10^{13}$ m^{-2}).

In the *cold* rolled material, the mean grain intercept was measured to be ~1 μm in the rolling direction and ~0.3 μm in the sheet normal direction (Fig. 1c, Table 1). The HAB fraction was 67%. The substructure typically appeared as bands of nearly parallel boundaries with boundary spacing of ~0.1 μm (Fig. 1c, Table 1). The development of such microstructures is well documented in cold rolled aluminum alloys [1,9] and attributed to deformation banding. The dislocation density ($\rho \sim 6\times10^{14}$ m^{-2}) (Table 1) was higher by a factor of 9 in comparison with the *hot* rolled sheets.

In both rolled conditions, the nano-scale (~9 nm) $Al_3(Sc,Zr)$ dispersoids were observed (Figs. 1b and d). These precipitates were often characterized by a specific coffee-bean contrast, which indicated their coherence relationship with matrix as can be seen in encircled dispersoid (Fig.1d). A minor fraction of coarser (~25 nm), round-shaped and incoherent Al_6Mn particles [10] was also found in grain interior (Fig. 1b).

Friction stir welds. Transversal cross sections of the welds produced in both rolled materials are summarized in Fig. 2. The asymmetrical, tear-shaped stir zone was found in both cases; this observation agrees well with previous studies of FSWed Al-Mg-Sc alloys [7,8]. A so-called “kissing bond” defect [7] was also found in both welds (an example is arrowed in Fig. 2b). This defect is relatively rare observed in friction-stir welds being attributable to the formation of oxides at the joining interfaces.

Microstructure of welds. The microstructures developed in the central part of stir zone of both welds are given in Fig. 3. It is clearly seen that FSW led to the formation of fully recrystallized structure with equiaxed grains with a mean grain size of ~0.9 μm (Figs. 3a&c). Density of lattice dislocations in the stir zone and *ho*t rolled sheet was found to be the same (Figs. 3b&d, Table 1). The mean subgrain size was measured to be 0.5 μm and 0.7 μm in hot and cold rolled conditions, respectively (Table 1). The HAB fraction was 77-78%. Therefore, FSW and ECAP followed by *hot* rolling produce essentially the same deformation UFG structure.

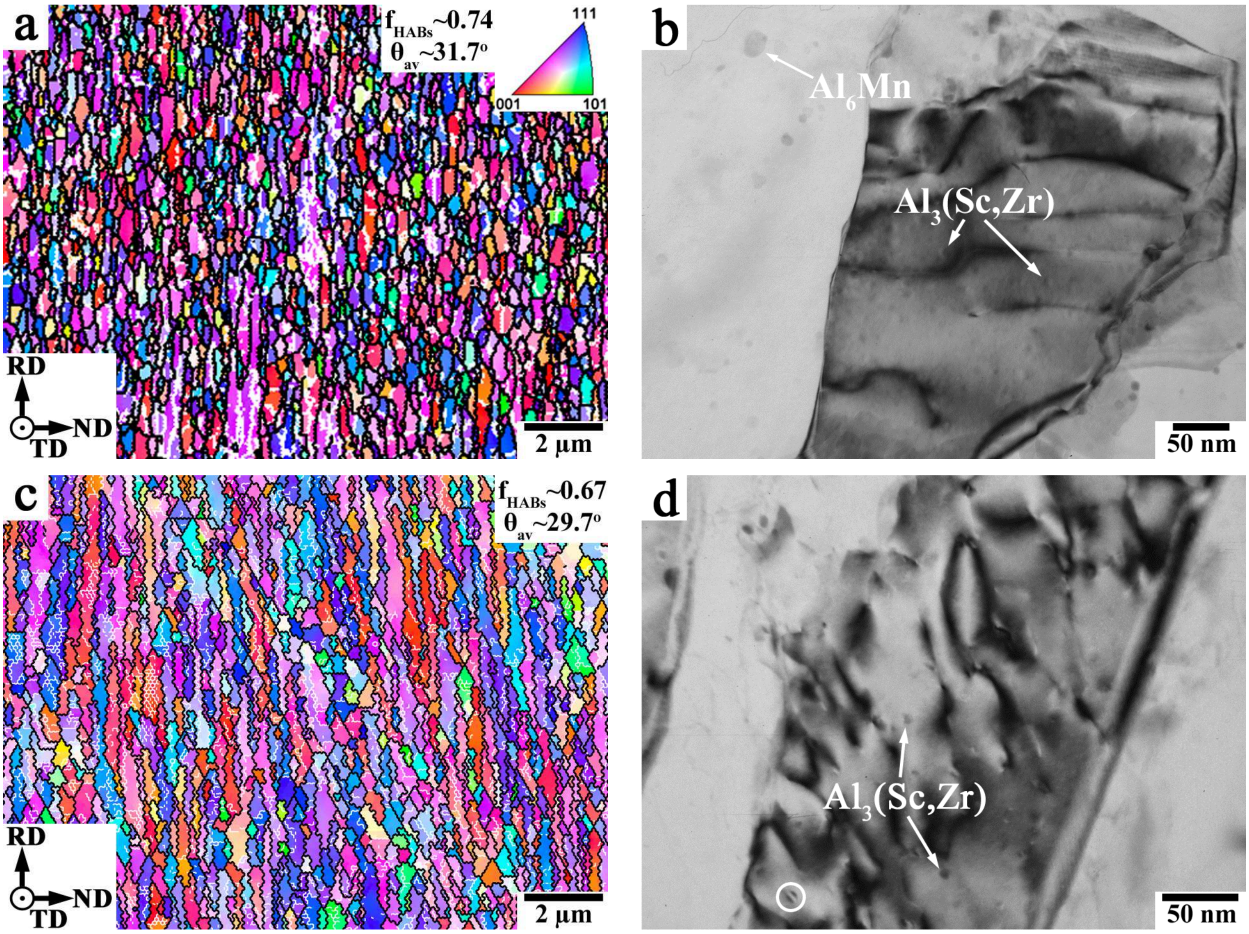

Figure 1. Typical microstructure of hot (a, b) and cold (c, d) rolled UFG sheets. See text for details

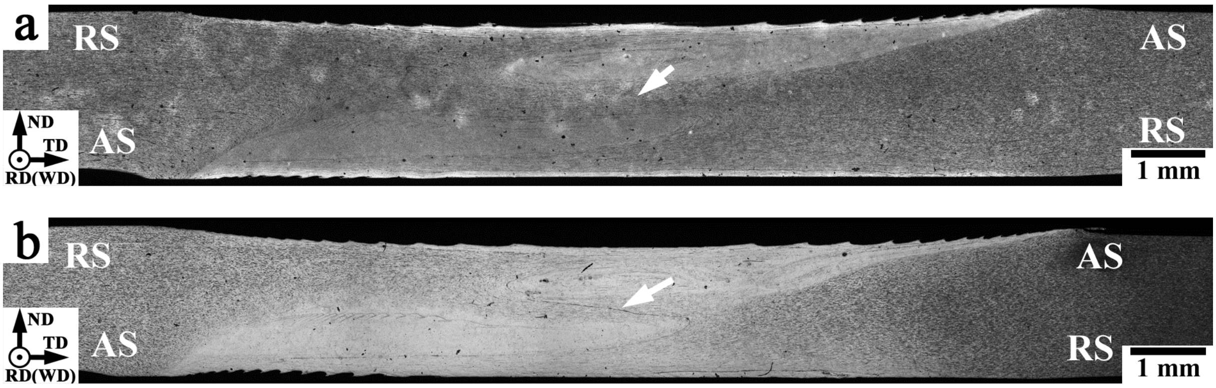

Figure 2. Low-magnification overviews of transversal cross-section welds of hot rolled UFG sheet (a) and cold rolled UFG sheet (b). AS and RS denote advancing and retreating sides, respectively. White arrow indicates “kissing bond” defect.

The volume fraction of the Al_3(Sc, Zr) dispersoids was found to be unchanged in the stir zone of rolled sheet (Table 1). No changes in dimensions of these dispersoids were found in *hot* rolled sheets (Table 1). On the other hand, these particles were found to slightly coarsen up to ~12 nm in the cold rolled condition due to FSW (Fig. 3d and Table 1). The precipitates often exhibited the characteristic coffee-bean contrast (examples are encircled in Figs.3b and d) thus indicating the presence of coherent strain. This observation agrees well with literature data predicting the preservation of the coherent relationship till the particle size of 116 nm [2] or 40 nm [11].

Mechanical properties. The microhardness profiles measured across the welds at the sheet midthickness are shown in Fig. 4. For clarity, the shoulder and pin dimensions are also depicted in the figure. In both preprocessed conditions, a significant material softening was found in weld zone. This effect has been already documented in FSWed Al-Mg alloys [7,8]. Importantly, the softened region was very broad exceeding even shoulder diameter. This indirectly indicates that the observed effect was mainly related with heat input induced during FSW. Considering preservation of fine grained microstructure as well as nano-scale dispersoids in the stir zone (Fig. 3), the material softening is believed to be mainly related with reduction of dislocation density in the rolled materials due to recrystallization during FSW [7,12].

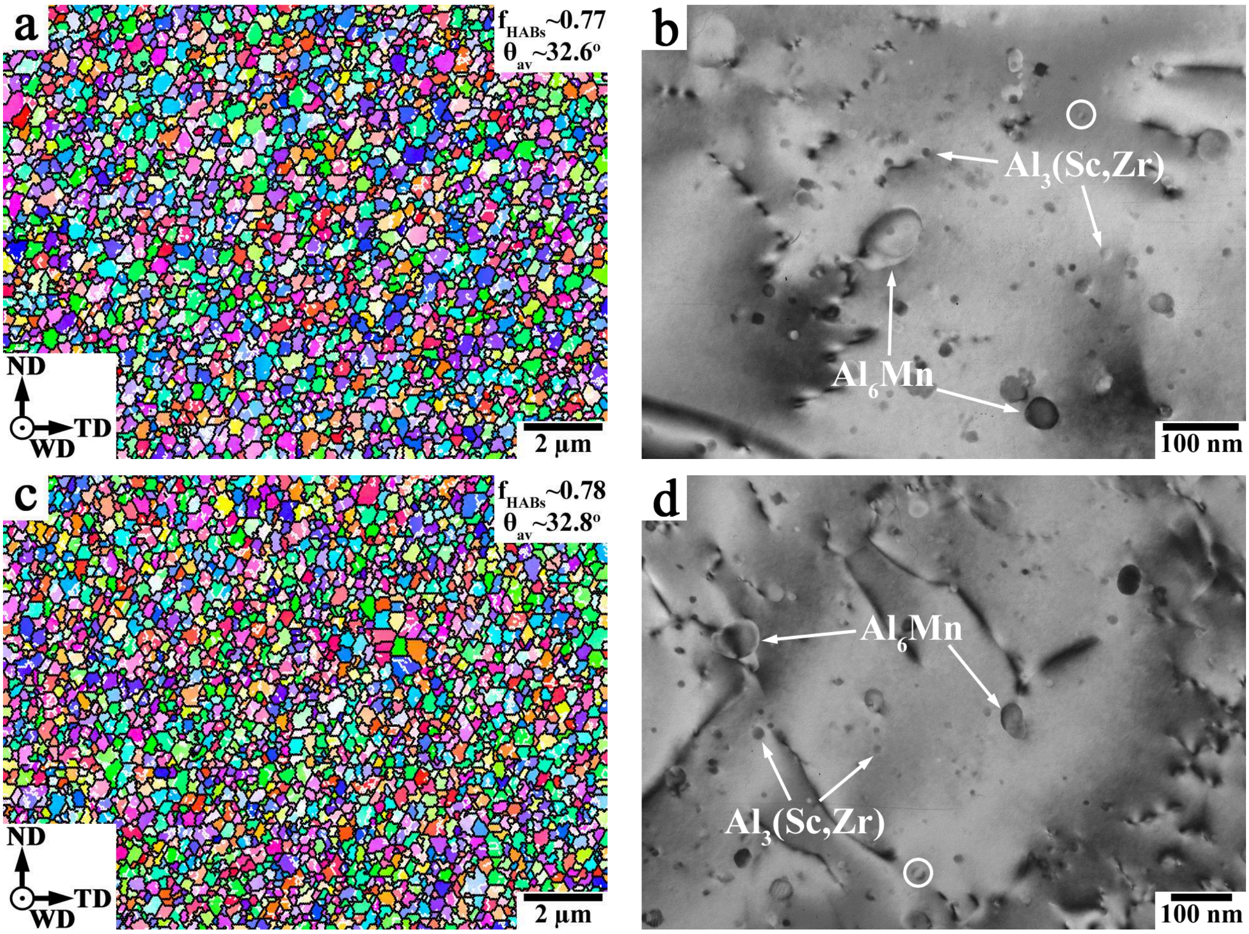

Figure 3. The microstructure in central part of stir zone in the hot rolled condition (a, b) and the cold rolled condition (c, d).

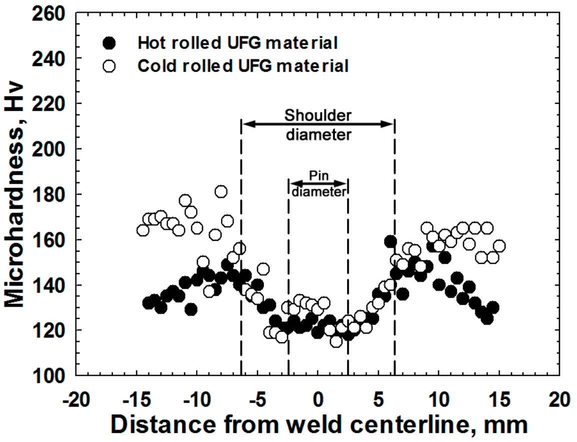

Figure 4. Microhardness profiles of FSWed UFG sheets.

A substantial scattering of microhardness measurements is also worthy of remark (Fig. 4). This may be due to microstructural inhomogeneity induced by the double-side FSW.

Transverse tensile tests showed that joint efficiency for yield strength (E_{YS}) was 81% in the hot rolled state and only 55% in the cold rolled condition (Table 2). All welds were fractured in the stir zone and the crack propagation was substantially influenced by the “kissing bond” defect (Fig. 5).

While the material softening in the stir zone was an expected result, the formation of the “kissing bond“ was rather surprising. The appearance of these defects is thought to be attributable to an insufficient material intermixing in the stir zone. In the present case, this effect may be related with the formation of oxides at the joining interfaces but this idea requires experimental verification. In any case, however, these defects are rather rare in FSW and thereby this problem is thought may be overcome by adjusting of welding conditions and/or tool design. In other words, the joint efficiency of friction stir welds of sheets with UFG structure is believed may be significantly improved.

Table 1. Microstructural parameters of base materials and welds

Material condition	Mean grain size, μm	Mean subgrains size, μm	Dislocation density, m^{-2}	Average size of $Al_3(Sc,Zr)$ dispersoids, nm	Volume fraction of $Al_3(Sc,Zr)$ dispersoids, %
Hot rolled	1.3/1*	0.5	7×10^{13}	9	0.1
Cold rolled	1/0.3*	0.1	6×10^{14}	9	0.1
FSWed hot rolled	0.9	0.5	8×10^{13}	9	0.1
FSWed cold rolled	0.9	0.7	7×10^{13}	12	0.1

*Numerator and denominator show mean grain intercept in the rolling and normal directions, respectively.

Table 2. Mechanical properties of UFG preprocessed materials and friction-stir joints

Material condition	Yield strength, MPa	Ultimate tensile strength, MPa	Ductility, %	E_{YS}, %	E_{UTS}, %	E_{δ}, %	Failure location
Hot rolled	375	440	11.5	-	-	-	-
Cold rolled	555	575	3.5	-	-	-	-
FSWed hot rolled	305	405	6	81	92	52	Stir zone (defect)
FSWed cold rolled	305	380	3.5	55	66	100	Stir zone (defect)

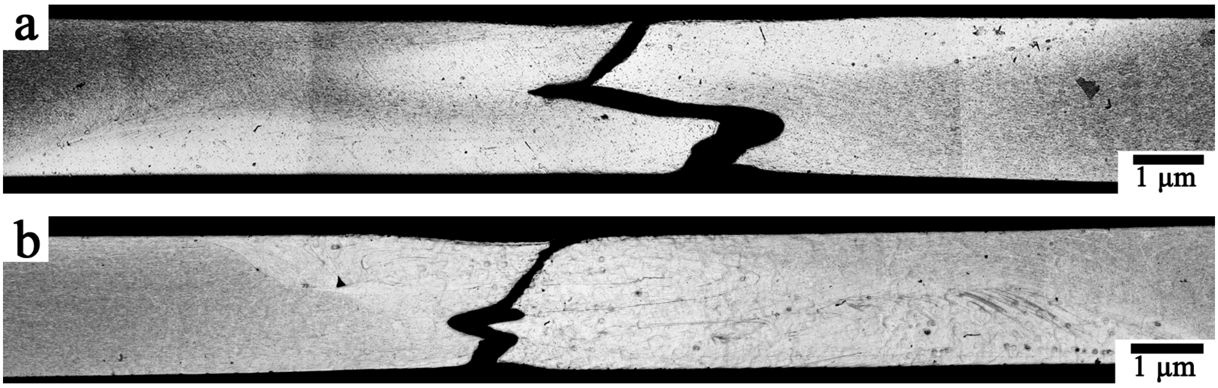

Figure 5. The transversal cross-sections of failure the welds in hot rolled condition (a) and cold rolled condition (b).

Conclusion

The microstructure and mechanical properties of FSWed sheets of Al-5.4Mg-0.2Sc-0.1Zr alloy with UFG structure were studied. The UFG material was obtained by ECAP followed by ether hot or cold rolling. The main conclusions from this work are given below:

1) FSW was demonstrated to be very effective in preservation of fine-grained microstructure and nano-scale coherent dispersoids in UFG Al-Mg-Sc-Zr alloy. Fully recrystallized structure evolved in the stir zone; a mean grain size and HAB fraction were ~0.9 μm and 77-78%, respectively. The volume fraction of constituent $Al_3(Sc,Zr)$ precipitates did not change measurably

whereas the mean dispersoid size increased only slightly from 9 nm to 12 nm in the cold rolled sheets; the coherence relationship between the dispersoids and matrix retains.

2) Despite the preservation of the fine-grained microstructure as well as strengthening precipitates, an essential material softening was observed in the stir zone.

3) The joint efficiency of the obtained friction-stir welds was found to be 81% in the hot rolled condition and only 55% in the cold rolled state. The relatively low joint efficiency is believed to be attributable the recrystallization softening in the cold rolled sheet as well as to the formation of a specific “kissing bond” defect in the stir zone of hot and cold rolled sheets. The weld strength, however, is thought may be improved by adjusting of welding conditions and/or tool design.

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