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Mechanical properties and microstructural evolution of Al 6061 alloy processed by multidirectional forging at liquid nitrogen temperature

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ABSTRACT

Al–Mg–Si alloy was subjected to multidirectional forging (MDF) at liquid nitrogen temperature (LNT), to cumulative strains of 1.8, 3.6 and 5.4. The deformed microstructures were examined by optical microscopy under polarized light and transmission electron microscopy (TEM). The deformed samples showed the formation of dislocation cells structure with high dislocation density at lower strains. Composite structure consisting of lamellar morphology at deformation bands and equiaxed grain morphology was observed. Significant differences in microstructure of the deformed samples were observed with increasing strain at LNT. At cumulative strain of 5.4, the microstructure shows nearly equiaxed subgrain structure with an average size of 250 nm with high angle grain boundaries. The mechanical properties were studied through Vickers hardness testing machine and tensile tester. The hardness value of MDFed alloy at LNT has increased from 50 Hv to 115 Hv for cumulative strain of 5.4. Tensile strength has increased from 180 MPa to 388 MPa with 4.5% percentage of elongation to failure. The improvement in hardness and tensile strength of forged alloy is attributed to the formation of equiaxed sub-grain structures and the presence of high dislocation density.

1. Introduction

Metals and alloys with ultrafine/nanostructured grain morphology are emerging as potential structural materials due to their superior mechanical properties such as strength, ductility, toughness, and fatigue strength as compared to their bulk alloys. Several severe plastic deformation techniques are employed to produce UFG/nanostructure in the metals and alloys by inducing severe plastic strain in the material at room temperature or above room temperature. The typical SPD techniques are Equal channel angular pressing (ECAP) [1–3], High pressure torsion (HPT) [4–6], and Accumulative roll bonding (ARB) [7–9]. The limited utility of the above methods are due to design complexities and high tooling cost (ECAP), unable to produce in bulk quantities (HPT), and tedious processing procedures (ARB). However, these methods are very popular as it enables significant grain refinement in the material to produce thermally stable ultrafine/nanostructured grain morphology with enhanced mechanical properties, which are not realizable in the conventionally processed materials [10,11].

Multiple forging also known as multidirectional forging, developed in the year 1990, has been used to refine the microstructures of bulk billets of titanium alloys, magnesium alloys and high strength nickel base alloys [12]. It is the simplest method to achieve larger strains with minimum distraction from its original shape and allows processing of bulk products [13]. Multidirectional forging involves repeated setting-drawing in three orthogonal directions. Along with deformation strain, deformation temperature also plays a critical role in refining the grain structure. MDF with high processing temperature is used to produce fine grain structure in brittle materials. Magnesium and its alloys have been successfully MDFed at room temperature to elevated temperatures [14–17]. Cherukuri et al. [18] have performed multi-axial forging (MAF) on Al 6061 alloy and reported that MAFed material has shown similar trends in mechanical properties with ECAPed material. It has also been reported that nonuniform distribution of micro hardness observed at initial deformations, has decreased with increasing accumulated strain [18,19].

Cryorolling is one of the deformation processing techniques used to develop fine grain structure in the material by deforming near or at liquid nitrogen temperature (77 K). It is used to produce ultrafine grain structure in high stacking fault energy (SFE) metals. Recent studies have shown the role of SFE in refining the grain structure at cryogenic deformation [20]. It has also been reported that there is an optimum stacking fault energy where the cryogenic deformation is very effective [20]. UFG microstructure has been successfully realized through cryogenic deformation in several

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FCC metals and alloys [21–24]. Cryogenically deformed metals with high stacking fault energy, for example, Al alloys exhibit ultrafine grains with low angle grain boundaries, due to lower induced strains (∼2.6) [25]. Since the material shape gets changed during rolling, it is difficult to induce high strain to the deformed material. The cryogenically deformed Al alloys show superior mechanical properties compared to room temperature deformed Al alloy due to effective suppression of dynamic recovery [26,27,29,30]. Micro structural evolution and mechanical behavior of aluminium alloys subjected to very large deformation strain at cryogenic temperature is scarce in literature. Hence, aim of the present work is to investigate the effect of MDF at LNT on microstructural evolution and mechanical properties of Al–Mg–Si alloys. The influence of cumulative strain on hardness and tensile properties of the Al alloys at intermediate steps was examined through Vickers hardness testing and tensile testing. A systematic study on micro structural evolution with increasing strain at mesoscale and microscale by using optical microscopy and transmission electron microscopy was made in the present work. The role of deformation strains and temperature for the grain refinement in the Al alloys was substantiated through detailed TEM analysis of the forged samples.

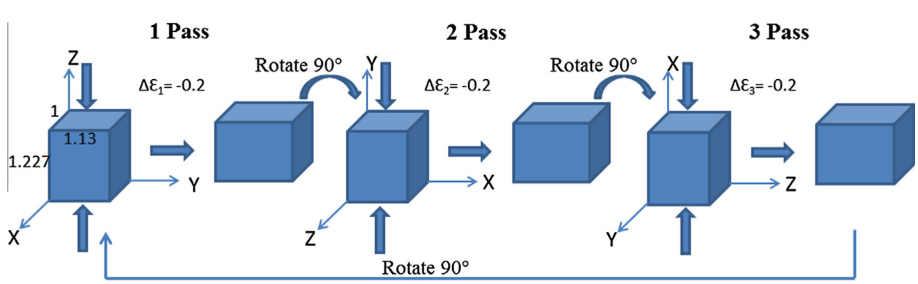
2. Experimental details

Al 6061 alloy was received in the T6 condition in the form of 25 mm thick plate. Chemical composition of the alloy is 0.67 Si, 0.28 Fe, 0.2 Cu,1.01 Mg, 0.04 Mn, 0.05 Cr, 0.06 Zn balance Al. Rectangular samples with 27 × 30.5 × 33 mm^3 were machined from the as received plate and solution treated at 520 °C for 2 h and water quenched at room temperature. The samples were subjected to MDF at 77 K using friction screw forging machine at a strain rate of 10 s^{-1}. The first forging axis was parallel to the rolling direction of the starting sample. The direction of the sample is changed for every pass at an angle of 90°. The sample dimension ratio 1:1.13:1.22 was maintained constant throughout the processing. MDF at cryogenic temperature was performed by filling the die with liquid nitrogen and its level was maintained up to sample height during forging. After every pass, the sample is allowed to attain thermal equilibrium with liquid nitrogen by giving 5–10 min soaking time. Fig. 1 shows the schematic diagram of MDF for a single cycle. Strain per pass is fixed as $\Delta\varepsilon_i = 0.2$ (where '*i*' number of passes) and maintained constant throughout all passes. The cumulative strain after one cycle of MDF was $\sum\Delta\varepsilon_{n=1} = 0.6$ (where '*n*' is number of cycles). In the present study, MDF was carried out to cumulative strains of 1.8, 3.6, 5.4, i.e., 3, 6, 9 cycles, respectively. After 4 cycles, the samples are reshaped by maintaining sample dimension ratio constant. Samples were successfully forged up to 9 cycles without any cracking. For comparative study, samples were forged at room temperature (RT) (at 300 K) for same strains. After MDF, samples were prepared by sectioning along the plane perpendicular to the last forging axis i.e., the highest dimension side. Microstructure evolution and mechanical properties were studied after MDF at different cumulative strains ($\sum\Delta\varepsilon_{n=3} = 1.8$, $\sum\Delta\varepsilon_{n=6} = 3.6$, $\sum\Delta\varepsilon_{n=9} = 5.4$). Optical microscopic studies were performed, under polarized light, using Poultons reagent to etch the sample. Transmission election microscopy (TEM) characterizations were performed under FEI-Techai 20 G2 S-Twin TEM operated at 200 kV. TEM samples were prepared by thinning down the samples to 100 μm through mechanical polishing with emery papers and punching the sample of 3 mm dia using GATAN disc punch. 80:20 methanols: perchloric acid was used at −38 °C in Tenopol twinjet polisher. Hardness measurements were carried out on FEI-VM50 PC Vickers hardness testing machine at room temperature using 5 kg load with 15 s dwell time. Minimum 6 readings were taken to get the average hardness value. Tensile tests were performed at room temperature on H25K-S Tinius Oslen tensile testing machine by preparing small size samples with 8 mm gauge length. The total length of the sample is 27 mm and width and thickness of gauge length section are 3 mm and 1.5 mm, respectively. At least four samples were tested to check the reproducibility. Scanning electron microscopy (SEM) was used to examine the fractured surface of tensile samples.

3. Results and discussion

3.1. Microstructure

Optical micrographs of Al 6061 alloy after solution treatment is shown in Fig. 2. The initial microstructure shows elongated grains with an average length of 300 μm. Fig. 3 shows a series of distinctive optical microstructures evolved after subjecting to MDF at 77 K. Fig. 3a shows 3 cycle's ($\sum\Delta\varepsilon_{n=3} = 1.8$) MDFed sample, where the distinction between grain interiors and grain boundaries was vague. It has shown deformed microstructure consisting of deformation bands in various directions which are clearly visible under polarized light. With increasing number of MDF cycles up to 6 ($\sum\Delta\varepsilon_{n=6} = 3.6$), the density of deformation bands has further increased and S-band formation was observed (marked with arrows in Fig. 3b) [13]. These observations are in agreement with the Kobayashi et al. [13] reported on MDF of pure copper at 195 K. It is evident from Fig. 3c that after 9 cycles MDF, the microstructure got fully perturbed and filled with deformation bands. It can be noticed that with increasing MDF strain, the density of deformation bands has increased with the reduction of the band gap. In Fig. 3c, d areas marked with oval shows development of cross deformation bands, observed in 9 cycles MDFed sample. These



Equivalent strain in one pass : $\Delta\varepsilon_i = \ln(1/1.227) = -0.2$
Cumulative strain in one cycle: $\sum\Delta\varepsilon_{n=1} = |\Delta\varepsilon_1 + \Delta\varepsilon_2 + \Delta\varepsilon_3| = 0.6$

Fig. 1. Schematic view of multidirectional forging (MDF) applied in this work. This is corresponding to one cycle.