Thermal stability and superplastic behaviour of an Al-Mg-Sc alloy processed by ECAP and HPT at different temperatures

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Abstract. An Al-3%Mg-0.2%Sc alloy was processed by ECAP and HPT at different temperatures. Afterwards, samples subjected to 10 turns of HPT at 300 and 450 K, 8 passes of ECAP at 300 K and 10 passes of ECAP at 600 K were annealed for 1 hour at 523 K and their mechanical properties and microstructure were examined using microhardness measurements and EBSD analysis. In addition, tensile specimens with similar dimensions were machined from the HPT and ECAP-processed materials and further tensile tested at 523 K. The results demonstrate that the Al alloy processed by HPT at 450 K exhibits higher microhardness values (~138 Hv) and a smaller average grain size (~0.28 μm) after annealing at 523 K among all SPD processing conditions. Accordingly, the material subjected to HPT at an elevated temperature displays superior superplastic properties such that an elongation of ~1020 % was attained after testing at 523 K at 1.0×10^{-3} s^{-1}. Furthermore, detailed EBSD analysis revealed a significant fraction of low-angle grain boundaries (LAGBs > 33 %) in the ECAP-processed material after annealing which may be responsible for the inferior superplastic behaviour by comparison with the HPT-processed samples (LAGBs < 13 %) tested at 523 K.

1. Introduction

There is a growing interest in the application of Al alloys in the transportation industry for replacing heavier structural components usually fabricated with steel and reduce the energy consumption and, thereby the pollutant emissions from motor vehicles [1]. Accordingly, in order to satisfy this demand, processing by severe plastic deformation (SPD) [2,3], as in high-pressure torsion (HPT) [4] and equal-channel angular pressing (ECAP) [5], has been successfully used to produce ultrafine-grained (UFG) metals having remarkably enhanced properties by comparison with materials subjected to conventional thermo-mechanical processing.

Among the various Al alloys already processed through SPD procedures, notable attention has been dedicated to Al-Mg-Sc alloys because of their excellent mechanical strength [6,7] and superplastic properties [8,9]. The addition of Mg solutes in aluminium delays its recovery kinetics and promotes further strain hardening and grain refinement during processing by ECAP [10,11] and HPT [12,13]. Furthermore, the Al_3Sc precipitates are effective in pinning the grain boundaries and retaining ultrafine grains even at elevated temperatures [14-16].

There are numerous studies showing the outstanding superplastic properties of Al-Mg-Sc alloys processed by ECAP [8,9,14,15]. The Al-3Mg-0.2Sc alloy subjected to 8 passes of ECAP at room temperature achieves an elongation of ~2580 % after tensile testing at 723 K at 3.3×10^{-3} s^{-1} [8].

Conversely, a record elongation of ~4100 % was attained in the Al-5Mg-0.2Sc-0.08Zr alloy processed through 10 passes of ECAP at 598 K and further tested in tension at 5.6 × 10^{-2} s^{-1} at 723 K [9]. It is worth mentioning that, although finer grain sizes are obtained after ECAP at ambient temperature [8], the Al alloy processed by ECAP at an elevated temperature displays an improved thermal stability and a larger proportion of high-angle grain boundaries [9,16].

Several investigations have demonstrated that HPT processing at ~300 K leads to additional grain refinement and improved strength in Al-Mg-Sc alloys by comparison with ECAP [17-23]. The Al-3Mg-0.2Sc alloy processed through 10 turns of HPT exhibits exceptional superplastic elongations at low homologous temperatures [19,20,24], although there are only few and isolated studies evaluating the microstructural changes in this metal when exposed to high temperatures [18,22,24]. Furthermore, it was revealed in recent studies that the microstructural stability in pure Ni [25] and the superplastic properties in Mg alloys [26,27] are significantly enhanced by conducting HPT processing at high temperatures and, to date, there have been no studies dedicated to assess the superplastic properties of Al-Mg-Sc alloys processed by HPT at an elevated temperature.

Therefore, the present study was initiated to evaluate the effect of the nature of the SPD procedure and the processing temperature on the thermal stability and low temperature superplasticity of an Al-3Mg-0.2Sc alloy subjected to ECAP and HPT at different temperatures.

2. Experimental material and procedures

The material used in this study was an Al-3% Mg-0.2% Sc (in wt. %) alloy supplied by China Rare Metal Material Corporation (Jiangxi Province, China) as forged bars with ~130 mm length and 10 mm diameter. These bars were solution treated at 880 ± 2 K for 1 h and quenched in water. Afterwards, billets with lengths of ~65 mm were cut from the bars and then processed through 8 passes of ECAP at room temperature (~300 K) or through 10 passes of ECAP at 600 ± 5 K using route B_c wherein each billet is rotated by 90° in the same sense between passes [28]. The processing was conducted using a pressing speed of ~2 mm s^{-1} and a solid die having an internal channel angle of 90° and an outer arc of curvature of ~20°. These angles impose an equivalent strain of ~1 on each ECAP pass [29].

Discs with ~1 mm thickness were cut from the solution treated bars and then ground to a thickness of ~0.8 mm. The discs were processed by HPT under quasi-constrained conditions [30] either at ambient temperature (~300 K) or 450 ± 5 K. The elevated processing temperatures were attained by using small heating elements around the anvils and the temperature was controlled by a thermocouple placed within the upper anvil at a position of ~10 mm from the HPT sample as described in earlier studies [26,27]. Initially, in the compression stage of HPT [31], the discs were compressed within the anvils under a nominal pressure of 6.0 GPa and then held at the HPT facility for ~1 and ~10 min for the procedures conducted at 300 and 450 K, respectively. Thereafter, the lower anvil was rotated at a constant rate of 1 rpm for 10 turns imposing high torsional straining within the HPT sample.

Following ECAP processing, discs with ~1 mm thickness were cut from the processed billets and then annealed for 1 h at 523 K and cooled in air. The same annealing procedure was performed in the HPT-processed material. Afterwards, both the SPD-processed metal and the annealed samples were ground and polished to obtain mirror-like surfaces and hardness measurements were taken at the middle-sections of the discs using the same method described as in earlier studies [23,32]. The microhardness was recorded using an FM300 microhardness tester equipped with a Vickers indenter under a load of 200 gf and a dwell time of 15 s. Since there is axial symmetry around the central axis in HPT processing, an area-weighted average microhardness was estimated in the HPT samples using the hardness values recorded along the diameters of the discs. Conversely, a normal average microhardness was calculated in the ECAP-processed samples.

The microstructures of the ECAP and HPT-processed materials were analysed using a JSM6500F thermal field emission scanning electron microscope. Samples were ground, final polished using 0.06 μm colloidal silica and then etched using an aqueous solution of 5 % HBF_4. The grain structures of the Al alloy were examined by scanning electron microscopy (SEM) and electron backscattered diffraction (EBSD) and the average grain size, *d*, was estimated using the linear intercept method.

EBSD patterns were collected for the samples annealed at 523 K using step sizes as small as 0.03 µm for the material processed by HPT at 450 K. A cleaning procedure, including grain dilatation, was performed after data collection such that the total number of modified points was <20 %. Low-angle grain boundaries (LAGBs) were defined as having misorientation differences between adjacent points from 2° to 15° and high-angle grain boundaries (HAGBs) had misorientations higher than 15°.

For tensile testing, miniature specimens with the same dimensions as in earlier investigations [7,24] were machined from the discs previously processed by ECAP and HPT at different temperatures. These specimens were tested in tension at 523 ± 2 K using a Zwick Z030 testing machine operating at a constant rate of cross-head displacement in order to provide an initial strain rate of 1.0×10^{-3} s^{-1}. All samples were held at the testing temperature for ~10 min prior to the application of the load.

3. Experimental results

Figure 1 shows the variation of the Vickers microhardness either along the diameter of the HPT discs or along the cross-section of the ECAP billets of the Al-3Mg-0.2Sc alloy processed by SPD at different temperatures and further annealed for 1 h at 523 K. It is clearly noted from these plots that the microhardness distribution along the diameter of discs is fairly uniform after post-SPD annealing, except for the metal processed by ECAP at 300 K which displays higher hardness values at the centre of the sample. In addition, the HPT-processed alloy exhibits superior microhardness compared with the material processed by ECAP and subsequently annealed at identical conditions, especially for the metal processed by HPT at 450 K in which the average microhardness is ~138 Hv after annealing.

Typical OIM images are shown in Fig. 2 for the Al-3Mg-0.2Sc alloy processed through 10 turns of HPT at (a) 300 and (b) 450 K or subjected to either (c) 8 passes of ECAP at 300 K or (d) 10 passes of ECAP at 600 K and further annealed for 1 h at 523 K. It follows from Fig. 2 that the microstructure of the Al alloy processed either by ECAP or HPT consists of a homogeneous array of ultrafine grains after post-SPD annealing, regardless of the processing temperature. The samples originally subjected to HPT at 300 and 450 K have nearly equiaxed grains mostly formed by HAGBs, as evident from Fig. 2(a) and (b). By contrast, inspection of Fig. 2 (c) and (d) reveals that the microstructure of the metals processed by ECAP displays a significant proportion of LAGBs after annealing at 523 K and average grain sizes of ~0.34 and ~0.94 µm for the Al alloy processed by ECAP at 300 and 600 K, respectively.

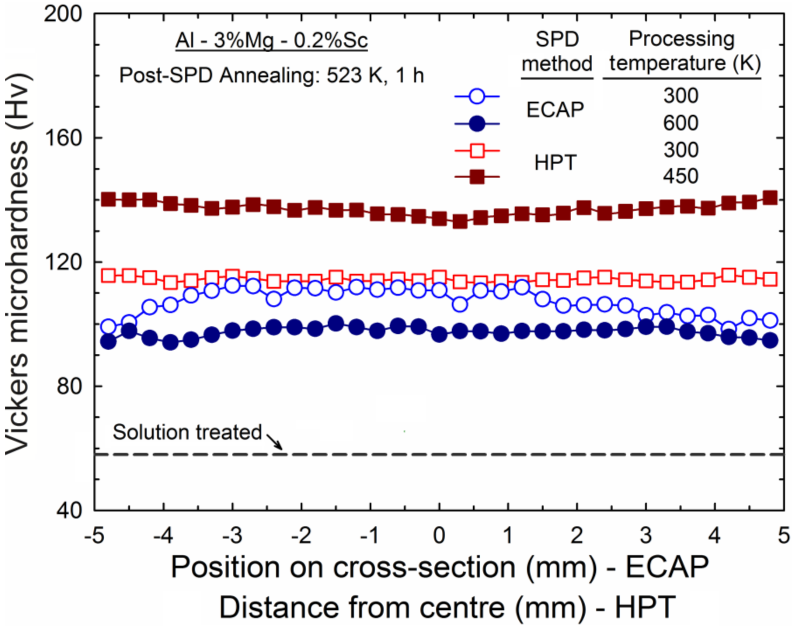

Fig. 1. Variation of the average microhardness recorded along the cross-section of the ECAP billets and the diameter of the HPT discs of the Al-3Mg-0.2Sc alloy subjected to SPD processing at different temperatures and further annealed for 1 h at 523 K.

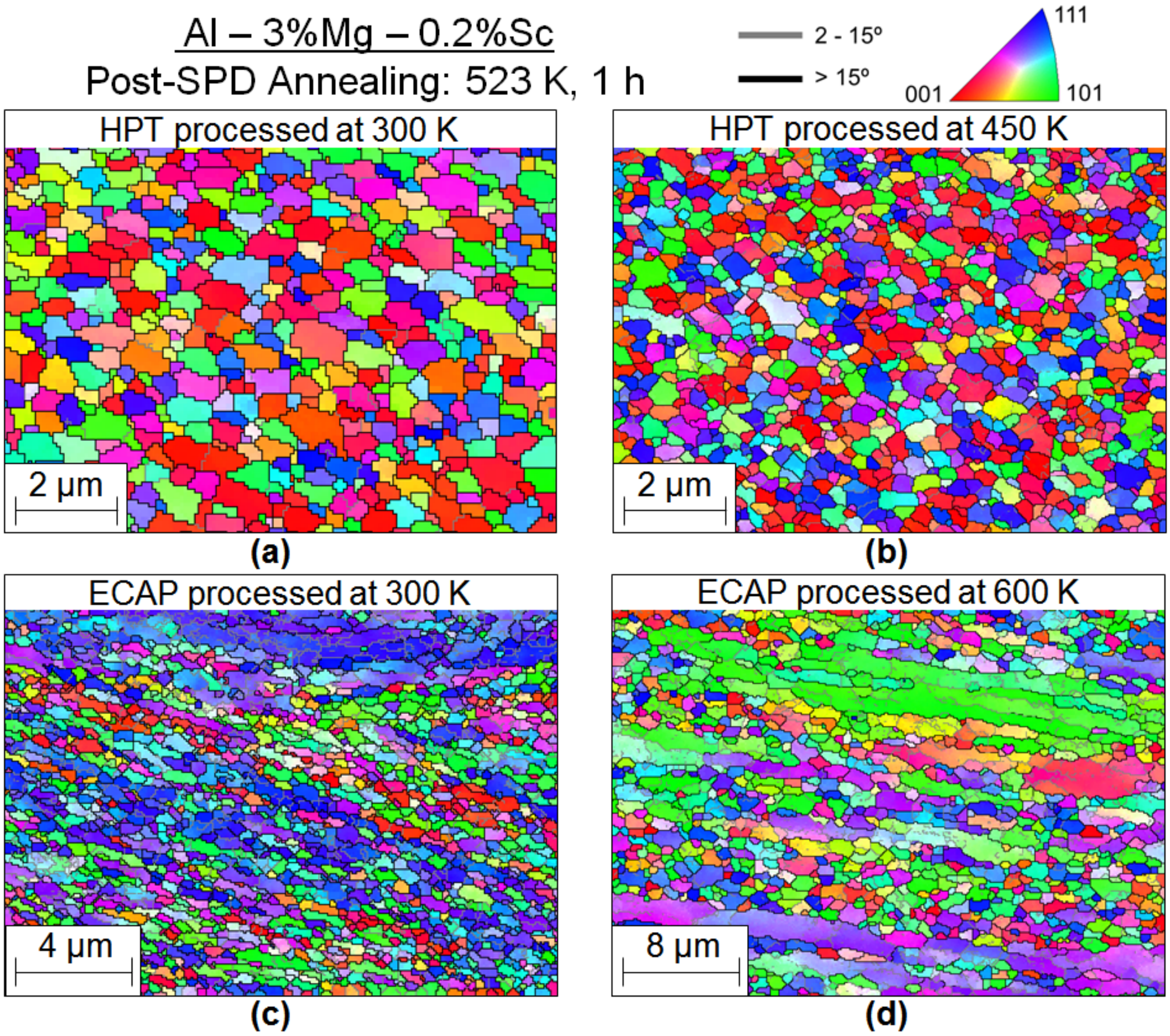

Fig. 2. OIM images of the Al-3Mg-0.2Sc alloy processed through 10 turns of HPT at (a) 300 and (b) 450 K or subjected to either (c) 8 passes of ECAP at 300 K or (d) 10 passes of ECAP at 600 K and further annealed at 523 K for 1 h.

The grain sizes of the Al-3Mg-0.2Sc alloy immediately after ECAP and HPT were estimated using SEM images obtained at positions located at distances of ~3 mm from the centres of the discs. Accordingly, in order to provide information on the coarsening kinetics and superplastic behaviour of this material, Table 1 shows the average microhardness and the average grain size in Al-3Mg-0.2Sc samples immediately after SPD processing and after annealing for 1 h at 523 K. The elongation and the average grain size are also listed for the Al alloy after tensile testing at 1×10^{-3} s^{-1} at 523 K.

It is clearly demonstrated in Table 1 that higher hardness values and notably smaller grain sizes are attained in the Al-Mg-Sc alloy immediately after HPT processing by comparison with ECAP. The thermal stability in this material is significantly enhanced by conducting HPT at 450 K as finer grains ($d \approx 0.28$ μm) are retained after annealing at 523 K. Furthermore, it is interesting to note that the samples processed by ECAP and HPT at 300 K have similar microhardness and grain sizes after post-SPD annealing, although a larger fraction of HAGBs was achieved through HPT.

It also follows from Table 1 that the Al-3Mg-0.2Sc alloy processed by HPT exhibits superior superplastic properties during tensile testing at 523 K at 1.0×10^{-3} s^{-1}. An exceptional elongation of ~1020 % was attained in the material processed by HPT at 450 K whilst the elongations in the Al alloy processed by ECAP at 300 and 600 K were ~540 and ~560 % after straining under identical conditions.

Table 1. Average microhardness and average grain size of Al-3Mg-0.2Sc samples measured immediately after SPD processing and after post-SPD annealing for 1 h at 523 K. The elongation and the average grain size are also measured for the Al alloy after testing at 1×10^{-3} s^{-1} at 523 K.

SPD method	*Processing temperature (K)*	*Average microhardness (Hv)*		*Average grain size (μm)*			*Elongation (%)*
		After SPD processing	*After post-SPD annealing for 1 h at 523 K*	*After SPD processing*	*After post-SPD annealing for 1 h at 523 K*	*After Tensile testing at 523 K at 1×10^{-3} s^{-1}*	*After Tensile testing at 523 K at 1×10^{-3} s^{-1}*
HPT	300	194	115	0.14	0.40	1.1	620
	450	179	138	0.15	0.28	1.0	1020
ECAP	300	138	106	0.25	0.37	1.1	540
	600	96	93	0.60	0.94	1.4	560

4. Discussion

This investigation reveals that processing through 10 turns of HPT leads to a superior average microhardness and smaller grain sizes in the Al-3Mg-0.2Sc alloy compared with ECAP processing. In addition, the material processed by ECAP at 600 K depicts significantly larger grain sizes than the Al alloy processed at 300 K. These results are consistent with earlier studies with Al-Mg-Sc alloys [7-9,17-23] and are associated with the higher strain rates in the UFG metal during HPT processing as well as with the increasing action of dynamic softening mechanisms during deformation at higher homologous temperatures, T/T_m, as reported for various fcc metals [25,33].

It is also clearly demonstrated in Fig. 2 and Table 1 that the microstructural stability in Al-Mg-Sc alloys is notably improved by conducting HPT at an elevated temperature. Although higher hardness values were attained in the material processed by HPT at 300 K, the grain sizes after HPT processing are essentially identical, regardless of the processing temperature. Nevertheless, finer grain structures are distinguished in the material processed by HPT at 450 K after subsequent annealing for 1 h at 523 K. This is attributed to the contribution of dynamic recovery on reducing the strain energy stored in the form of dislocations in Al-Mg-Sc alloys during HPT at elevated temperatures. It is worth noting that the temperature rise in the disc was estimated as ~30 K using an empirical relationship [34,35] and therefore there is no significant change in T/T_m during processing by HPT.

The Al-3Mg-0.2Sc alloy processed by HPT at 450 K and further tensile tested at 523 K at 1.0×10^{-3} s^{-1} exhibits finer grains and a remarkably superior elongation among all SPD processing conditions. This excellent superplastic behaviour is a direct consequence of the extremely small grain sizes and the larger proportion of grains with HAGBs in the HPT-processed metal. By contrast, although true superplastic elongations of >400 % [36] were also achieved in the material processed by ECAP at 300 K, the larger grain sizes and the presence of a significant fraction of substructures with LAGBs lead to an earlier failure in the specimens of the Al alloy pulled to failure in tension at 523 K at 1.0×10^{-3} s^{-1}.

It is therefore concluded the Al-3Mg-0.2Sc alloy processed by HPT at 450 K is a potential candidate for applications demanding materials with high strength to density ratio fabricated through superplastic forming at low temperatures.

5. Summary and conclusions

1. An Al-3% Mg-0.2% Sc alloy was processed through 10 turns of HPT at 300 and 450 K, 8 passes of ECAP at 300 K and 10 passes of ECAP at 600 K to produce grain sizes in the range of ~140-600 nm. Thereafter, samples were annealed for 1 h at 523 K or tensile tested at 523 K at 1.0×10^{-3} s^{-1}.

2. The Al alloy processed by HPT at 450 K displays finer grain structures (~0.28 μm) after annealing at 523 K among all SPD processing conditions. This is attributed to the increasing action of dynamic recovery during HPT at high temperatures which delays its grain coarsening kinetics.

3. Excellent superplastic properties were attained in the Al-3Mg-0.2Sc alloy processed by HPT. A maximum elongation of ~1020% was achieved in the material processed by HPT at 450 K and further tensile tested at 523 K at 1.0×10^{-3} s^{-1}. This is associated to the presence of a uniform array of equiaxed grains with a lower fraction of substructures by comparison with the ECAP-processed metal.

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