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Original Article

Examining the microhardness evolution and thermal stability of an Al–Mg–Sc alloy processed by high-pressure torsion at a high temperature☆

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ABSTRACT

An Al–3% Mg–0.2% Sc alloy was solution treated and processed through 10 turns of high-pressure torsion (HPT) at 450 K. Afterwards, the HPT-processed alloy was annealed for 1 h at temperatures ranging from 423 to 773 K and its mechanical properties and microstructural evolution were examined using microhardness measurements and electron backscattered diffraction (EBSD) analysis. The results demonstrate that HPT processing at an elevated temperature leads to a more uniform microhardness distribution and to an early saturation in the hardness values in the Al alloy compared with high-pressure torsion at room temperature. In addition, detailed EBSD analysis conducted on the HPT-processed samples immediately after annealing revealed that the Al–Mg–Sc alloy subjected to HPT processing at 450 K exhibits superior thermal stability by comparison with the same material subjected to HPT at 300 K.

1. Introduction

There is an increasing interest in the development of Al alloys with improved mechanical properties in order to substitute heavier materials in vehicle components and reduce the energy consumption in the transportation industry [1]. Severe plastic deformation (SPD) procedures [2,3] are now recognised as effective methods in fabricating Al alloys with exceptionally small grain sizes within the submicrometre range [4]. Although various SPD techniques are now available, major focus has been dedicated to equal-channel angular pressing (ECAP) [5] and high-pressure torsion (HPT) [6] as these procedures require simple facilities and permit the processing of difficult-to-work materials by controlling the temperature, the imposed pressure and the effective strain rate in the workpieces [7].

Ultrafine-grained (UFG) Al–Mg alloys exhibit improved mechanical strength by comparison with the material subjected to conventional metal forming [8–12]. The addition of magnesium in this material delays its recovery kinetics and promotes further grain refinement during processing by ECAP

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[8–10] and HPT [11,12]. Nevertheless, UFG Al–Mg alloys display significant grain coarsening after annealing at relatively low temperatures [13,14] and scandium is added to this material in order to enhance its microstructural stability and thereby preserve the benefits achieved during severe plastic deformation [15–18].

Excellent thermal stability and superplastic properties are documented for Al–Mg–Sc alloys processed by ECAP [19–21]. The Al–3Mg–0.2Sc alloy processed by ECAP at room temperature has an average grain size of ~0.2 μm immediately after processing and achieves a maximum elongation of ~2580% during tensile testing at 723 K at $3.3 \times 10^{-3}\,s^{-1}$ [19]. By contrast, the Al–5Mg–0.2Sc–0.08Zr alloy has a grain size of ~1 μm after ECAP processing at 598 K, however, this alloy exhibits superior superplastic properties with a record elongation of ~4100% attained at $5.6 \times 10^{-2}\,s^{-1}$ at 723 K [21]. This outstanding superplastic behaviour is attributed to a larger proportion of high-angle grain boundaries and to an improved microstructural stability in the material processed by ECAP at 598 K.

Recent investigations have demonstrated that HPT processing at elevated temperatures leads to slightly larger grain sizes and lower hardness values in Mg alloys [22,23], stainless steel [24,25] and pure Ni [26] by comparison with HPT at ambient temperature. Conversely, these materials usually have more stable grain structures when annealed after HPT [24–26] and display higher superplastic elongations, as reported for the AZ61 [22] and the Mg–9Al alloy [23]. Accordingly, processing by HPT at high temperatures has emerged as a promising strategy to delay the grain coarsening kinetics in Al–Mg–Sc alloys and retain submicrometre grains at temperatures suitable for superplastic forming.

Therefore, the present study was initiated to evaluate the thermal stability of an Al–3Mg–0.2Sc alloy subjected to 10 turns of HPT processing at 450 K by examining the mechanical properties and microstructural evolution in this UFG material after systematic annealing for 1 h at temperatures ranging from 423 to 773 K.

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, discs with a thickness of ~1 mm were cut from the solution treated material and then ground to a final thickness of ~0.8 mm.

The discs were processed by HPT processing under quasi-constrained conditions [27,28] at 450 ± 5 K. The elevated processing temperatures were achieved by incorporating small heating elements around the upper and lower 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 investigations [22,23,29]. Initially, in the compression stage of HPT processing [30], the discs were compressed within the shallow central depression of the anvils already heated at ~450 K under a nominal pressure of 6.0 GPa. Thereafter, the facility was held at the processing temperature for ~10 min and finally the lower anvil was rotated at a constant rate of 1 rpm for 10 turns imposing high torsional straining within the HPT sample.

Following HPT processing, discs were annealed at temperatures from 423 to 773 K for 1 h and cooled in air. Afterwards, both the HPT-processed material and the annealed samples were ground and polished to obtain mirror-like surfaces and hardness measurements were then taken at the middle-section of the samples using the same procedure as in other studies [31–33]. Hardness values were evaluated along the diameters of the discs at positions separated by 0.3 mm such that the microhardness in each position was calculated as the average of the measurements taken from four indentations separated by 0.15 mm. Furthermore, the area-weighted average microhardness of the Al–3Mg–0.2Sc alloy was estimated using the hardness measurements recorded along the diameters of the discs and noting that the hardness values near the edges of the sample are associated with larger surface areas. The microhardness values were recorded using an FM300 microhardness tester equipped with a Vickers indenter under a load of 200 gf and a dwell time of 15 s.

The grain structures of the UFG metal were analysed by scanning electron microscopy (SEM) and electron backscattered diffraction (EBSD). Discs were ground, polished using 1 μm diamond paste and 0.06 μm colloidal silica and thereafter etched using a solution of 5% HBF_4 dissolved in water. The microstructure of the Al alloy was examined using a JSM6500F thermal field emission scanning electron microscope and the average grain size, *d*, was estimated using the linear intercept method. EBSD patterns were collected for the materials annealed at 573, 623 and 773 K using step sizes as small as 0.06 μm. A cleaning procedure, including grain dilatation, was performed after data collection such that the total number of modified points was <20%. High-angle grain boundaries (HAGBs) were defined as having misorientation differences between adjacent points higher than 15° and low-angle grain boundaries (LAGBs) had misorientations within the range from 2° to 15°.

3. Experimental results

Fig. 1 shows the variation of the Vickers microhardness along the diameter of the Al–3Mg–0.2Sc discs processed through 10 turns of HPT at 450 K and further annealed at different temperatures. It is readily noted from these plots that there is a significant increase in the microhardness of the solution treated material after 10 turns of HPT at an elevated temperature. The Vickers microhardness of the unprocessed alloy is ~58 Hv, whereas hardness values as high as ~185 Hv were detected in the HPT-processed sample as noted in the hardness measurements denoted by open circles. Furthermore, although higher hardness values were attained in the same alloy after HPT at 300 K [32], the metal processed by HPT at 450 K displays a more uniform microhardness distribution with hardness values varying from ~160 to ~185 Hv at the centre and near to the periphery of the disc, respectively.

It follows from Fig. 1 that post-HPT annealing at 473 K leads to a slight decrease in the hardness recorded along the diameter of the Al–3Mg–0.2Sc sample, but nevertheless