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Structural impact on the Hall–Petch relationship in an Al–5Mg alloy processed by high-pressure torsion

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ABSTRACT

The evolution of microstructure and microhardness was studied in a commercial 5483 Al–5Mg alloy processed by high-pressure torsion (HPT) under a pressure of 6.0 GPa up to 10 turns. Significant grain size refinement was observed even after 1/4 turn, and additional processing led to a further grain size reduction and a shift in the distribution of grain boundary misorientation angles towards higher values. An essentially fully homogeneous microstructure was reached after 10 turns with a final grain size of ~70 nm, a saturation Vickers microhardness of Hv ≈ 240 which was attained at and above equivalent strains of ~150, a relatively narrow grain size distribution and a fraction of ~80% of high-angle grain boundaries. Analysis shows the Hall–Petch plot deviates from the conventional linear relationship for samples processed through small numbers of turns, but after 3 or more turns there is a direct correlation between the results obtained in HPT processing and coarse-grained samples.

1. Introduction

It is well known that grain refinement leads to a significant increase in strength in metals due to the Hall–Petch relationship [1,2] which correlates the mechanical properties with microstructural features such as the grain size. This relationship demonstrates the potential for improving the mechanical strength by reducing the grain size and this has been a major driving force in the development of ultrafine grained (UFG) and nanocrystalline (NC) materials. In practice, these materials are most readily produced in bulk form by subjecting materials to severe plastic deformation (SPD).

A number of techniques are now available for the SPD processing of metals [3–6] but the procedures receiving the most attention are equal-channel angular pressing (ECAP) [7] and high-pressure torsion (HPT) [8]. Both these methods impose high strains but in practice the largest strains are imposed by HPT and, by comparison with other available SPD procedures, HPT processing gives the smallest grains [9,10] and the highest fraction of high-angle grain boundaries [11].

During HPT processing, a thin disk is placed horizontally between two massive anvils, it is subjected to a high pressure and then strained torsionally through rotation of one of the anvils. In order to compare the shear strain in HPT with linear strain values for other SPD methods, the equivalent von Mises strain, ε_{eq}, is expressed for HPT as [12]

$$\varepsilon_{eq} = \frac{2\pi N r}{h\sqrt{3}} \tag{1}$$

where N is the number of turns, r is the distance from the centre of the disk and h is the sample thickness. It follows from Eq. (1) that the strain varies across the disk and reaches zero at the disk centre where $r=0$. This suggests that the microhardness distributions will be extremely inhomogeneous but numerous reports show there is usually a gradual evolution with increasing strain towards a reasonable level of structural homogeneity [13–19]. Furthermore, this evolution is consistent with theoretical modelling [20].

Only limited information is currently available on the microstructure and mechanical properties of 5XXX series aluminium alloys processed by HPT. There are also shortcomings in the data because the disks are often processed up to only 5 turns of HPT [21,22] which may be insufficient to attain a high level of homogeneity, and there has been no attempt to evaluate the significance of the grain boundary (GB) characteristics, and especially the boundary misorientations, in determining the properties of the alloy. In practice, the GB characteristics are important in metals processed by SPD because the misorientation angles change significantly during processing such that many of the low-angle grain boundaries (LAGBs) evolve into high-angle grain boundaries

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(HAGBs). There have been some discussions on the influence of the GB misorientation distributions on the mechanical properties [23,24] but there are only limited experimental results to address this problem [25–27]. Accordingly, the present research was initiated to evaluate the influence of HPT processing on the grain refinement, grain boundary characteristics and the homogeneity of a commercial AA5483 aluminium alloy processed by HPT.

2. Experimental material and procedures

The material investigated in this study was a hot-pressed commercial AA5483 aluminium alloy containing 5% Mg and 1% Mn with an average grain size of ~25 μm. The material was cut into disks with diameters of 10 mm and thicknesses of ~0.8 mm. These disks were polished and then processed at room temperature using quasi-constrained HPT [28,29] with a constant pressure of 6.0 GPa and a rotation speed of 1 rpm. The disks were torsionally strained through different numbers of revolutions, N, up to a maximum of 10.

After HPT processing, microhardness measurements were conducted on the polished surfaces of disks using an FM-300 microhardness tester. These measurements were taken under a load of 100 g and a dwell time of 10 s. The values of the Vickers microhardness, Hv, were recorded for each disk both along a diameter with a separation between points of 0.3 mm and over the total areas of selected disks following a rectilinear grid with a spacing of 0.3 mm between each consecutive point.

For inspection using transmission electron microscopy (TEM), small disks with a diameter of 3 mm were punched from the edge regions of each disk so that the areas of observation were ~3.5 mm from the central points. This is shown in Fig. 1 where the illustration on the right depicts three different orientations for the TEM disks: orientation 1 is parallel to the HPT disk plane and orientations 2 and 3 are on the cross-sectional planes and either parallel or perpendicular to the rotation direction. These disks were cut using a focus ion beam microscope Hitachi FB 2100 and then experimental data were collected using a Hitachi 5500 STEM operating at an accelerating voltage of 30 kV. The microstructures were evaluated quantitatively using a computer-aided image analyser. The grain sizes were described in terms of the equivalent grain diameter, d_{eq}, defined as the diameter of a circle with a surface area equal to the surface area of the grain. For the cross-sectional planes, the short and long axes of the elongated grains were also measured. Standard deviations (SDs) were estimated for all the grain populations.

The GB misorientations were determined for samples cut parallel to the disk plane in orientation 1 using TEM with the Kikuchi patterns obtained for adjacent grains using convergent beam electron diffraction (CBED) [30]. Diffraction images were taken from neighbouring grains with a high resolution scanning TEM (Hitachi HD 2700) and then processed using a KILIN programme to calculate the crystallographic orientations of individual grains and the misorientations across the GBs.

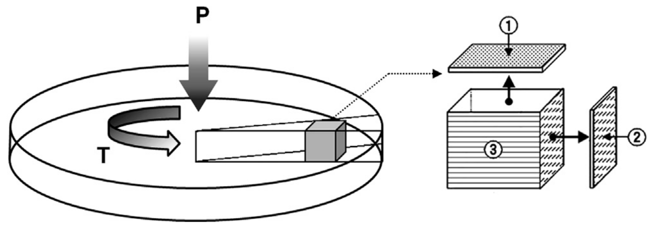

Fig. 1. Thin disk processed by HPT showing cutting for the TEM samples (on the left) and the three planes used for the TEM observations (on the right).

3. Experimental results

3.1. Evolution of microstructure during HPT

A series of representative TEM images is recorded in Fig. 2 for three disk planes after processing through (from the top row) 1/2, 1, 5 and 10 turns: the images in (a–d) show the structures visible on the disk plane (plane 1), (e–h) show the cross-sectional plane parallel to the rotation direction (plane 2) and (i–l) show the cross-sectional plane perpendicular to the rotation direction (plane 3). On plane 1, grain size quantification showed the average grain size was reduced to ~175 and ~155 nm after 1/4 and 1/2 turns, respectively. Further straining led to additional grain refinement and the detailed results and SD values are summarised in Table 1. After 10 revolutions the grain size was measured as ~70 nm, which is significantly smaller than the size of ~100 nm recorded for the same alloy after processing by ECAP followed by hydrostatic extrusion [27] or ~200 nm recorded for the same alloy after processing by hydrostatic extrusion [31].

Images taken from the two cross-sectional planes provide information on the three-dimensional size and shape of the grains formed during HPT processing. On plane 2 the grains are highly elongated with respect to the shearing direction and lie at angles between 45° and 55° to the disk plane. Measurements showed that the shortest axes decreased with increasing N from ~100 nm for 1/2 turn to ~40 nm after 10 turns. A similar tendency was found on plane 3 as summarised in Table 2 but for this plane the grains were elongated along the disk diameter and the elongation was parallel to the surface.

A careful examination showed there were also differences in the grain size distributions. Processing with only 1/2 turn of HPT produced a highly inhomogeneous structure (Fig. 2a) with relatively large grains having diameters of ~1 μm surrounded by smaller grains with diameters from 100 to 200 nm. With increasing N, it is apparent that the grain structure gradually becomes more homogenous.

The distributions of GB misorientation angles are shown in Fig. 3 for samples processed through 1/2, 1, 5 and 10 turns with at least 100 boundaries examined in each sample. In general, the number fractions of LAGBs decrease and the number fractions of HAGBs increase with increasing N. The shapes of these distribution diagrams are typical for materials processed by SPD and in the present investigation the fraction of HAGBs reached a maximum of 80% after 10 turns.

3.2. Microhardness evolution across the disk diameters after HPT

The microhardness distributions across the disk diameters are shown in Fig. 4 for samples processed by HPT through different numbers of revolutions from 1/4 to 10. The lower dashed line at $\text{Hv} \approx 92$ denotes the initial microhardness prior to processing. Inspection shows the microhardness increases significantly as a function of distance from the disk centre even after 1/4 turn. For this early condition, there is a clear linear dependence in the hardness values along the radius of the disk and this corresponds directly to the linear dependence of the applied strain in Eq. (1). After 1/4 and 1/2 turns, the areas around the centres of the disks exhibit very low microhardness values with $\text{Hv} \approx 96$ which is close to the value for the unprocessed sample whereas, by contrast, the hardness is $\text{Hv} \approx 160–180$ near the edges of each disk. Nevertheless, after larger numbers of turns the central region undergoes significant strengthening and finally, after 10 turns, there is a reasonable level of homogeneity throughout the disk with a hardness of $\text{Hv} \approx 240$.