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An examination of the saturation microstructures achieved in ultrafine-grained metals processed by high-pressure torsion☆

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ABSTRACT

Experiments were conducted on two commercial alloys, a Cu–0.1%Zr alloy and an Al-7075 aluminum alloy, to investigate the significance of the saturation microstructure which is achieved after processing by high-pressure torsion (HPT). Samples were processed by HPT and also by a combination of equal-channel angular pressing (ECAP) followed by HPT. The results show that the saturation conditions are dependent upon the grain size in the material immediately prior to the HPT processing. Additional grain refinement may be achieved in HPT by initially processing the material to produce an ultrafine-grain size before conducting the processing by HPT.

1. Introduction

It is now well established that it is often advantageous to produce metals having very small grain sizes. This is because if the grain size is small the metal is generally fairly strong because the yield stress at low temperatures varies with the reciprocal of the square root of the grain size through the Hall–Petch relationship [1,2]. Furthermore, at elevated temperatures small grain sizes provide a potential for achieving a superplastic forming capability [3]. The interest in attaining very small grain sizes led to the development of extensive

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thermo-mechanical processing operations, which were generally effective in producing grain sizes as small as ∼3–5 μm. Nevertheless, this type of processing was not effective in fabricating metals having grain sizes in the submicrometer range and more recently attention has focused instead on the processing of metals through the application of severe plastic deformation (SPD), where it is possible to achieve grain refinement to the submicrometer and even the nanometer range [4].

Ultrafine-grained materials (UFG) are defined as polycrystals having average grain sizes less than 1 μm [5]. Thus, UFG structures include both the submicrometer (100 nm to 1 μm) and the nanometer (<100 nm) ranges. These materials may be produced using SPD techniques [6] and accordingly they have attracted much attention over the last two decades. Several different SPD processing methods are now available but the two techniques receiving the most attention are equal-channel angular pressing (ECAP) [7] and high-pressure torsion (HPT) [8]. In ECAP the sample is in the form of a rod or bar and it is pressed through a die constrained within a channel that is bent internally through an abrupt angle. In HPT the sample is generally in the form of a thin disk which is held between massive anvils and then subjected to an applied pressure and concurrent torsional straining. Experiments show that processing by HPT produces both smaller grain sizes and a higher fraction of grain boundaries having high-angles of misorientation than when processing using ECAP [9–11].

The higher strength introduced by SPD processing is demonstrated most effectively by taking individual readings of the Vickers microhardness, Hv, at selected positions on the polished surfaces of samples processed by either ECAP or HPT. There are now numerous reports describing these measurements recorded on the cross-sectional planes [12–14] and the longitudinal planes [14–16] of ECAP samples and on the surfaces of disks processed by HPT [17–19]. There is a significant problem in using this approach to evaluate homogeneity in HPT because the strain varies across the disk. Specifically, the equivalent von Mises strain, ε_{eq}, is given by a relationship of the form [20,21]

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

where N is the number of revolutions in HPT processing and r and h are the radius and height (or thickness) of the disk, respectively. It follows from Eq. (1) that ε_{eq} is a maximum at the edge of the disk and decreases to zero at the center of the disk where $r = 0$. There are numerous experiments [10,18] showing a gradual evolution during HPT processing so that the microhardness values become essentially constant over the disk surfaces after a reasonably large number of revolutions. Furthermore, this evolution is also consistent with a theoretical model for hardness evolution in HPT [22].

Nevertheless, questions remain concerning the limits of grain refinement that may be attained through processing by ECAP or HPT [23,24] and at present these questions remain unresolved. Accordingly, this paper examines whether there is a true limitation on the refining of grains when processing by HPT and experiments are described which are designed to provide a critical examination of the possible occurrence of a true refining limit.

2. Experimental materials and procedures

The experiments were conducted by processing in HPT using two different face-centered cubic materials. First, tests were conducted on a commercial Cu-151 alloy with a composition of Cu–0.1 wt% Zr. This alloy was obtained from Olin Brass (East Alton, OH) as a rolled strip having dimensions of $760 \times 500\,mm^2$ and a thickness of 1.5 mm. Disks were machined from the strip with diameters of 10 mm and these disks were then polished to thicknesses of ∼0.83 mm. In the initial unprocessed condition, the measured grain size was ∼20 μm. Earlier reports described the SPD processing of this Cu–Zr alloy [11,14,25–28]. Second, experiments were conducted on a commercial Al-7075 alloy containing (in wt.%) 5.6% Zn, 2.5% Mg, 1.6% Cu with the balance as Al. This alloy was received in the form of extruded rods with diameters of 10.0 mm and these rods were annealed in air for 1 h at 753 K and then cooled in air to room temperature over a period of ∼5 min. In the annealed and unprocessed condition, the grains were elongated with lengths up to ∼450 μm and widths of ∼8 μm. This alloy was processed by HPT and also by ECAP and a combination of ECAP and HPT. Earlier reports described the processing of the Al-7075 alloy by SPD techniques [29–31].

For ECAP, the processing was performed at 473 K using a solid die with a channel having an internal angle of 110° and an outer arc of curvature of 20°. These angles lead to an imposed strain of ∼0.8 on each separate passage through the die [32]. The billets were processed using processing route B_C in which the rods are rotated by 90° in the same sense between each pass [33]. Following ECAP through either 4 or 8 passes, disks were cut from some of the billets and then used for processing by HPT: these samples are henceforth designated the ECAP + HPT samples. All HPT processing used disks with diameters of 10.0 mm and thicknesses of ∼0.83 mm. The disks were polished prior to HPT and then processed at room temperature (RT) for selected numbers of revolutions, N, under an applied pressure of 6.0 GPa and with a rotational rate of 1 rpm. The HPT processing was conducted under quasi-constrained conditions in which there is a small outflow of material around the periphery of the disk during the processing operation [34,35]. Full details of the HPT processing were given earlier except that a lubricant was not placed on the upper and lower anvils prior to processing [36].

The major emphasis in this investigation was to evaluate the limits of grain refinement under different HPT processing conditions. In practice, however, it is experimentally easier to compare the values of the saturation hardness which are attained by processing since these values relate directly to the microstructures in the materials. Two separate procedures were developed earlier for achieving rigorous measurements of the microhardness values [36] and the same procedures were followed in the present investigation. The principles of these two procedures are illustrated schematically in Fig. 1 [36]. All values of the Vickers microhardness, Hv, were recorded using a microhardness tester equipped with a Vickers indenter using a load of 100 gf and a dwell time of 15 s