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Using an Al–Cu binary alloy to compare processing by multi-axial compression and high-pressure torsion

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ABSTRACT

An Al–4% Cu alloy was selected as a model material in order to compare two different procedures for imposing severe plastic deformation: multi-axial compression (MAC) and high-pressure torsion (HPT). In MAC a compressive strain is applied to prismatic samples in a sequential order along three orthogonal directions and the process is repeated to large numbers of passes in order to attain high strains. In HPT a thin disk is held between anvils and subjected to a high applied pressure and concurrent torsional straining. The results show that HPT is the optimum procedure for producing a homogeneous ultrafine-grained material. Specifically, HPT is preferable because it produces materials having a larger degree of homogeneity and the equilibrium grain sizes are smaller and the Vickers microhardness values are higher than when processing by MAC.

1. Introduction

The processing of metals through the application of severe plastic deformation (SPD) has attracted much attention over the last decade because it provides an opportunity for achieving ultrafine-grained (UFG) materials having grain sizes within the submicrometer (0.1–1.0 μm) or nanometer (< 100 nm) range [1,2]. A significant advantage of SPD processing is that it provides procedures in which bulk solids are subjected to very high strains without incurring any significant change in their overall dimensions [3]. Several different methods of SPD processing are now available but the primary methods are equal-channel angular pressing (ECAP) [4], high-pressure torsion (HPT) [5], accumulative roll bonding (ARB) [6] and multi-directional forging [7]. To date, most attention has focused on ECAP and HPT and it is now well established that processing by HPT produces materials having smaller grain sizes than ECAP [8–10].

Conversely, only limited attention has been devoted to the processing of metals by multi-directional forging although this appears to be a simple and cost-effective method for the production of bulk UFG solids for use in industry. In this process as originally formulated, the material is subjected to a forging operation in which loading is performed in compression, the axis of loading is changed periodically and there is no restraint on the outward flow of material. Specifically, the process involves applying straining to prismatic samples sequentially along three orthogonal directions in a procedure generally designated as *abc* deformation [11]. This process was used for detailed investigations of the properties induced in a Ti–6Al–4V alloy [11] and high purity oxygen-free Cu [12,13] but in practice the procedure has experimental limitations because it is necessary to machine or grind the samples after each straining in order to remove the barreling introduced by the compression.

Later, the general principles of this process were developed into the procedure of multi-axial compression (MAC) in which samples of Cu were contained within a channel die, there was a constraint on two of the four lateral sides of the sample and the processing operation was conducted without introducing any barreling on the faces used subsequently for the loading operation in the next pass so that it was not necessary to grind the faces between each separate straining [14]. The procedure of MAC has been used to investigate the properties of several materials including AA1100 [15], AA3104 [16] and AA6061 [17,18] aluminum alloys, Al–4.11% Cu [19], Al–5.5% Cu [20] and high purity Cu [19]. Nevertheless, only limited information is available at present comparing the properties and microstructures of samples processed by MAC and other SPD procedures. An early report attempted to compare MAC with ECAP and ARB by processing samples of an AA6061 alloy to similar strains using each procedure but the results were inconclusive and it was reported only that similar grain sizes of < 1.0 μm were

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achieved using each process with similar values of hardness and similar tensile properties after processing [17]. More recently, experiments were reported on an Al–4.11% Cu alloy using MAC and ECAP and it was reported that the hardness increased more rapidly with strain when using ECAP but there were no measurements of the grain sizes after processing [19]. Furthermore, there has been no attempt to date to compare MAC directly with HPT although it is known that HPT is especially effective in producing very small grain sizes.

Accordingly, the present investigation was initiated to provide a first detailed comparison between the microstructures produced by MAC and HPT with a special emphasis on the grain sizes and the levels of homogeneity achieved using these two techniques. The experiments were conducted using an Al–4% Cu alloy where this material was selected because earlier experiments demonstrated the successful processing of Al–Cu binary alloys when using MAC [19,20] and ECAP [19].

2. Experimental material and procedures

Selected quantities of an aluminum of commercial purity (99.9%) and an Al–52.3 wt% Cu alloy were melted in a graphite crucible in an electrical resistance furnace at 1073 K. The melt was maintained at 973 K for 10 min and then quickly and smoothly poured into a metal mold to give a cast Al–4 wt% Cu binary alloy. The ingot was homogenized at 758 K for 24 h in air and then cut into rectangular prisms with dimensions of $10.0 \times 10.0 \times 15.0\ mm^3$ for MAC processing or into rods with diameters of 10.0 mm and lengths of 8.0 cm for use in HPT processing. Following an earlier procedure [21], these samples were solution treated at 813 K for 2 h and then quenched in cold water and subsequently aged at 693 K for 2 h [19].

For processing by MAC, the rectangular prisms were initially compressed at room temperature (RT) through the various passes depicted schematically in Fig. 1 where this corresponds to the conventional *abc* deformation such that the shape of the prism after 3 passes is identical to the initial shape. Samples were processed up to a maximum number, N, of 15 passes. Subsequently, and in order to achieve high strains, MAC processing was conducted at a temperature of 373 K with intermediate annealing treatments for 10 min at 373 K up to a total of $N=35$ passes. For processing by HPT, the rods were sliced into disks with thicknesses of ~1 mm, the disks were polished to final thicknesses of ~0.85 mm and then processing was accomplished using an HPT facility operating under quasi-constrained conditions in which the disk is held under an applied pressure between two massive anvils and there is a limited outward flow of material around the periphery of the disk during the processing [22,23]. A detailed

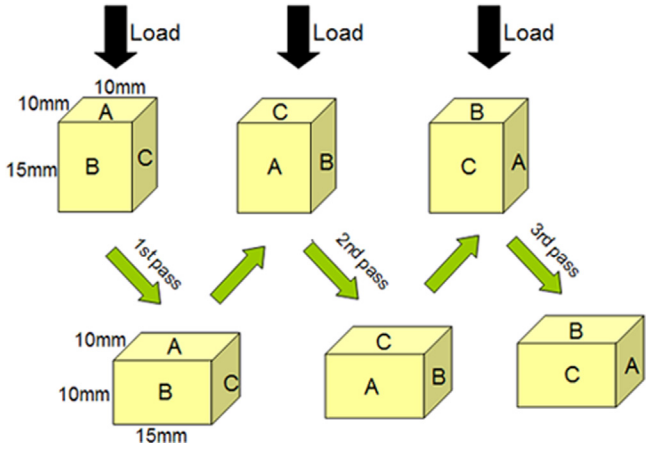

Fig. 1. Schematic illustration of the principles of MAC in *abc* deformation.

description of the HPT processing procedure was given earlier [24] except that in the present experiments a molybdenum disulfide lubricant was not placed around the disk on the upper and lower anvils. The HPT processing was conducted at RT under an applied pressure of 5.0 GPa using a rotational speed of 1 rpm for different numbers of turns, N, up to a maximum of 9 turns.

The samples processed by MAC and HPT were evaluated in different ways as illustrated schematically in Fig. 2. For the MAC samples, the rectangular prisms were cut horizontally perpendicular to the axis of the last compression and at positions close to the central planes. Hardness measurements were recorded on these planes at the positions shown in Fig. 2(a) and samples were prepared for transmission electron microscopy (TEM) at the positions indicated in the central and peripheral regions. For the HPT disk shown in Fig. 2(b), hardness measurements were recorded along diameters of the samples and small disks were prepared for TEM observations at both the centers and near the edges of the disks. Hardness measurements were undertaken at the points indicated in Fig. 2 by mounting the samples, carefully polishing to a mirror-like surface and then recording values for the Vickers microhardness, Hv, using a Micro-DUROMAT4000 facility with a load of 100 g and a dwell time of 15 s for each indentation. It is apparent from Fig. 2 that the incremental spacings between the microhardness indentations are generally 1.0 mm for both the MAC and HPT samples. For MAC, the hardness measurements represent the average of three separate indentations recorded at the same distance from the center of the plane of sectioning along the longitudinal and transverse directions, respectively. For the HPT samples, the values of Hv were determined by taking the average of four separate measurements recorded along four different radii at the same distance from the center of each disk.

The internal microstructures were observed by TEM using a TECNAI-G2 20ST instrument operating at 200 kV. The disks for TEM were mechanically ground below 90 μm and then electropolished with a solution of 30% nitric acid and 70% methanol at 10 V using an electrolyte maintained at a temperature below −25 °C. Selected area electron diffraction (SAED) patterns were recorded using an aperture size of 1.0 μm.

3. Experimental results

In order to evaluate the microstructural inhomogeneities in samples processed by SPD, the most convenient approach is to take measurements of the local microhardness and then correlate a selected set of these hardness values with microstructural observations undertaken using TEM [25,26]. Following this approach, the microstructural evolution occurring in the Al–Cu binary alloy is described in the following two sections when processing by MAC and HPT, respectively.

3.1. Hardness and microstructural evolution during MAC processing

The variation of the Vickers microhardness with the number of compression passes is shown in Fig. 3(a) for the central and edge regions of the specimens processed by MAC at room temperature where the values of hardness in the outer regions were determined by taking the averages of the longitudinal and transverse measurements at distances of 4 mm from the center. It is apparent that all of the microhardness values taken in the central regions of the rectangular prisms are higher than at the edge regions and for a condition of $N \approx 9$ passes both sets of measurements tend to reach saturation levels. The equivalent strain imposed in each pass in MAC is given by $\ln(h_o/h)$ where h_o and h are the initial height (15 mm) and final height (10 mm) of the samples, respectively [11]. This gives a strain of ~0.4 in every pass of MAC so that the