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The evolution of homogeneity and grain refinement during equal-channel angular pressing: A model for grain refinement in ECAP

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Abstract

Samples of pure aluminum and an Al-6061 alloy were subjected to equal-channel angular pressing (ECAP) for up to eight passes at room temperature. The Vickers microhardness was recorded on the polished cross-sections of each as-pressed billet and the results are plotted in the form of color-coded maps to provide a pictorial depiction of the hardness distributions throughout the cross-sections. The results show the hardness increases by a factor of ∼2 in the first pass through the die but thereafter there is only a minor increase. The microhardness distribution is homogeneous in the unpressed condition but becomes inhomogeneous after a single pass and then reasonably homogeneous in subsequent passes. The results reveal some differences between the behavior of pure Al and the Al-6061 alloy. These results and microscopic observations are used to develop a model for the process of grain refinement during ECAP.

Keywords: Equal-channel angular pressing; Grain refinement; Hardness; Homogeneity; Severe plastic deformation

1. Introduction

The processing of metals through the application of severe plastic deformation (SPD) has become attractive in recent years, because it provides the capability of achieving remarkable grain refinement in polycrystalline materials, typically to the submicrometer or even the nanometer level [1]. There are two primary advantages of using SPD processing. First, it has a potential for producing large bulk samples without the introduction of any porosity or the contaminants that are often inherent in alternative techniques such as inert gas condensation or ball milling. Second, conventional SPD processing can be applied relatively easily to a wide range of metallic alloys without making any significant changes in the processing procedure.

The most attractive, and potentially the most useful, SPD technique appears to be equal-channel angular pressing (ECAP) in which a metallic rod or bar is pressed through a die constrained within a channel which is bent through an abrupt angle that is often equal to, or very close to, 90° [2–4]. Processing by ECAP leads to significant strengthening of the material at ambient temperatures [5,6] and, provided the ultrafine grains have a reasonable thermal stability, to the occurrence of superplastic ductilities at very rapid strain rates at elevated temperatures [7,8].

Despite the apparent success in producing both high strength and high strain rate superplasticity in materials processed by ECAP, it is important to recognize that the tensile specimens used to establish these mechanical properties are generally cut from the as-pressed billets with their gauge lengths lying parallel to the longitudinal or pressing axes. This means, in effect, that the measured mechanical properties represent the characteristics associated with the as-pressed microstructure existing within the central core regions of the as-pressed billets and the mechanical testing provides no information on the general degree of microstructural homogeneity occurring in the cross-sections of the

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billets perpendicular to the pressing axis. An earlier study demonstrated the feasibility of effectively scaling the ECAP process to include billets having diameters from 6 to 40 mm [9] and in these experiments some tensile specimens, having gauge lengths of 5 mm, were cut from the 40 mm billet with their gauge lengths oriented in two mutually orthogonal directions perpendicular to the pressing direction. Although the subsequent curves of stress versus strain were essentially identical for all of these tensile specimens and for other specimens cut parallel to the pressing direction, thereby suggesting a reasonably homogeneous microstructure, the results are insufficient to provide detailed information on either the degree of homogeneity within the cross-sections or the changes in any inhomogeneities with increasing numbers of passes through the die. This information is necessary because the microstructure may change in the vicinity of the die walls if, for example, frictional effects become important.

The present investigation was initiated with three objectives. First, to use hardness measurements to evaluate the homogeneity of the microstructures on sections cut through the as-pressed billets perpendicular to the pressing direction. Second, to evaluate the significance of any evolution in the microstructural homogeneity as a function of the number of passes in ECAP and hence of the imposed strain. Third, to make use of these observations, together with earlier reports of microstructural development in ECAP, in order to construct a model to account for the evolution of an ultrafine-grained structure when processing by ECAP.

The experiments were conducted using two different materials: high-purity aluminum and an Al-6061 alloy. These materials were selected because there are several earlier studies documenting the basic characteristics of high-purity aluminum [10–15] and the Al-6061 alloy [16] when processing using ECAP. As will be demonstrated, there are microstructural inhomogeneities in both of these materials after ECAP through a single pass but the microstructures evolve with additional passes and, except only for very small regions in the vicinity of the bottom surfaces of the billets, they become essentially homogeneous in both materials.

2. Experimental materials and procedures

The experiments were conducted using pure aluminum of 99.99% purity and a commercial Al-6061 alloy containing, in wt.%, 1.01% Mg, 0.59% Si, 0.37% Fe, 0.29% Cu, 0.23% Cr and 0.20% Zn.

The aluminum was prepared by cold-rolling from an ingot into a plate with a thickness of 25 mm, cutting into a block with cross-sectional dimensions of 25 mm × 25 mm and swaging into cylindrical rods having diameters of 10 mm. For processing by ECAP, individual billets were cut from these rods with lengths of ∼70 mm. These billets were annealed at 773 K for 1 h prior to ECAP to give an initial unpressed grain size of ∼1 mm.

The Al-6061 alloy was received in the form of a plate, 10 mm in thickness, after a T651 heat treatment involving a solution treatment at 823 K, tensile straining to ∼2% at room temperature and aging at 433 K for 18 h. Samples with diameters of 10 mm and lengths of 70 mm were cut from the plate and annealed at 693 K for 4 h with a heating and cooling rate of 10 K h^{-1}. The initial grain size of the alloy in the unpressed condition was ∼50 μm.

The ECAP processing for both materials was performed at room temperature (298 K) using a hydraulic press of 150 tonnes capacity operating at a ram speed of ∼7 mm s^{-1}. The pressings were conducted using a solid die having an L-shaped channel bent through an angle of $\Phi = 90^\circ$ near the center of the die and with an additional angle of $\Psi = 20^\circ$ representing the outer arc of curvature where the two parts of the channel intersect. These values of Φ and Ψ lead to an imposed strain of ∼1 on each passage of the sample through the die [17]. An earlier investigation, also conducted using pure Al, showed that the use of a solid die with an arc of curvature of $\Psi = 20^\circ$ has no significant influence on measurements of homogeneity in the as-pressed samples by comparison with a conventional split die where the channel is bent through 90° and there is no arc of curvature so that $\Psi = 0^\circ$ [18]. Samples were processed by ECAP through selected numbers of passes using route B_C in which the samples are rotated by 90° in the same sense between each pass [19]. This processing route was selected because earlier experiments on high-purity Al showed it leads most expeditiously to an array of equiaxed grains separated by boundaries having high angles of misorientation [12].

Following ECAP, the billets were sectioned perpendicular to their longitudinal axes and then mounted and carefully polished to a mirror-like finish. Microhardness measurements were taken using an FM-1e microhardness tester equipped with a Vickers indenter. A series of individual measurements was recorded on each polished section whereby the Vickers indenter was moved over the surface and measurements of the Vickers microhardness, Hv, were recorded in a regular grid pattern with spacings between each separate measurement of ∼0.5 mm. This procedure gave a total of ∼220 individual values of Hv on each polished section. These individual values of Hv were then plotted in the form of color-coded contour maps depicting the variation of the local microhardness over the cross-sectional plane for each sample.

The internal microstructures of pure Al were examined using an Hitachi H-8100 transmission electron microscope (TEM) operating at 200 kV. Specimens were prepared for TEM in the form of disks, 3 mm in diameter, oriented in the plane of sectioning perpendicular to the longitudinal axes of the billets. These disks were ground mechanically to thicknesses of ∼150 μm and then thinned to perforation using a twin-jet electropolishing facility with a solution of 10% $HClO_4$, 20% $C_3H_8O_3$ and 70% C_2H_5OH with the solution maintained at a temperature of 278 K. Observations were recorded by TEM both in the central regions of the billets