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Effect of grain size and specimen dimensions on micro-forming of high purity aluminum

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ABSTRACT

Micro-compression testing was conducted using high purity Al processed by equal-channel angular pressing (ECAP) with both coarse-grained (CG) and ultrafine-grained (UFG) samples. The effects on the flow stresses of the initial grain size and the specimen size were investigated and the results show the initial grain sizes and the specimen dimensions affect the flow stresses during micro-compression for both CG and UFG specimens. There is a transition from strain hardening to strain softening with decreasing grain size during micro-compression but the transition grain size is dependent upon the size of the specimen. These results are interpreted using a model based on the separate influences of dislocation annihilation and dislocation accumulation in the UFG and CG materials, respectively. The results demonstrate that the occurrence of surface roughening is improved when using UFG pure Al and this shows there is a significant potential for using UFG pure Al in micro-forming operations.

1. Introduction

Over the last 20 years, the fundamental requirements for the fabrication of micro-parts have increased significantly due primarily to the rapid development of micro-electro-mechanical systems (MEMS). Micro-manufacturing is now becoming an important technology due to the increasing trend for miniaturization where micro-forming is defined as the production of parts or structures having at least two dimensions in the sub-millimeter range. In practice, micro-forming is rapidly becoming one of the promising micro-manufacturing techniques due to the potentials for high productivity, low cost and good mechanical properties [1–3].

Nevertheless, although the knowledge of tool design and fabrication methods are now well established for conventional macro-forming, there is evidence that the presence of size effects may lead to a breakdown in these basic manufacturing characteristics when the specimen dimensions are scaled down to the micro-scale [4,5]. To date, significant attention has been directed to examining the geometry size effects in micro-deformation [6]. For example, it was shown that the flow stress increases with decreasing specimen thickness when the ratio of specimen size to grain size is reduced below a critical value [7]. Similar results were also reported for several different materials [8–10]. In an investigation of the influence on mechanical properties and strain hardening of the material thickness and grain size, and specifically on the thickness to grain size ratio, it was shown that three deformation domains may be identified [11]: these domains were specifically designated as polycrystalline, multicrystalline and quasi-single crystalline, where the designations depend upon the thickness and the numbers of grains across the sample thickness. A study of size effects in miniaturized polycrystalline samples indicated that grain size, grain orientation and the nature of the grain boundaries all play important roles in delineating the intrinsic competition between the strengthening and weakening contributions [12].

As a consequence of the ongoing and increasing need for miniaturization, the grain size of conventional coarse-grained (CG) materials, which is typically a few to several tens of microns, becomes reduced to the same scale and with the characteristic dimensions of the micro-parts. In practice, if only a few grains are present in the micro-component then the response to the applied forces will show significant variations such that the reproducibility of the mechanical properties will become a serious problem in any micro-forming operation [13]. It follows, therefore, that these size

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effects are critical and must be considered in micro-forming in order to fabricate high quality micro-components.

By comparison with CG materials, ultrafine-grained (UFG) materials have the potential for simultaneously fulfilling two important conditions [14]. First, the average grain size is generally smaller than the smallest dimension of the component and this guarantees reproducible properties. Second, the grain size is sufficiently fine to ensure a high strength and fracture toughness. In addition, it is anticipated that materials with ultrafine grains will exhibit good formability over appropriate ranges of temperature and strain rate [15]. Therefore, it is reasonable to conclude that the grain size is the dominant factor determining the limiting size of the geometrical features that may be conveniently fabricated by micro-forming and this means in practice that very small grain sizes, and especially materials having ultrafine submicrometer grain sizes, are attractive for use in micro-forming operations.

It is now well established that UFG materials are most easily produced through the application of severe plastic deformation (SPD) to large bulk samples [16] using processes such as equal-channel angular pressing (ECAP) [17] and high-pressure torsion (HPT) [18]. Recent research has demonstrated the potential for using UFG materials for applications in micro-forming at elevated and ambient temperatures [19,20]. Nevertheless, only very limited information is at present available on the micro-forming deformation behavior when the material grain size is reduced to the sub-micrometer level although these problems are beginning to attract attention within the materials science community [21–24].

The present research was initiated specifically to examine the micro-compression behavior of high purity aluminum having grain sizes from the ultrafine scale to the coarse scale achieved by ECAP processing and annealing treatments. The surface roughness and microstructural evolution after micro-compression were investigated for both UFG and CG pure Al and the effects on the deformation mechanism of the initial grain size and specimen dimensions were critically analyzed.

2. Experimental material and procedures

Pure aluminum of very high purity (99.999%) was used in this investigation. The high purity aluminum was supplied in the form of drawn rods having diameters of 10 mm and lengths of ~70 mm. These rods were annealed in air for 1 h at a temperature of 773 K to obtain an initial grain size of ~300 μm. The experimental investigation followed four separate steps as illustrated schematically in Fig. 1.

In the first step, processing by ECAP was conducted at room temperature using a die with an internal angle of 90° between the two parts of the channel and an outer arc of curvature of 20° at the point of intersection [25]. This geometry leads to an imposed strain of ~1.0 on each separate pass through the die [26]. The billets were sprayed with an MoS_2 lubricant and processed repetitively by ECAP through 8 passes corresponding to a maximum imposed strain of ~8. All samples were processed using route B_C in which the sample is rotated by 90° in the same direction between each pass [27] where this processing route was selected because it leads most expeditiously to an array of equiaxed grains separated by a high fraction of high-angle grain boundaries [28].

In the second step following ECAP, billets were machined along the extrusion direction (ED) by electric discharge machining (EDM) to give small cylinders having dimensions of 4 (diameter) × 6 (height), 2 × 3 and 1 × 1.5 mm × mm. The transverse direction (TD) was located randomly for this machining and this is reasonable based on the lack of any orientation dependence of hardness measurements after processing by ECAP [29]. The specimens were then prepared by micro-forward extrusion at room temperature using three different dimensional tools with diameters of 2, 1 and 0.5 mm. These extruded billets were cut and ground to form micro-compression specimens having good dimensional accuracy with specimen diameter, D, x specimen height, H_0, of 2 × 3, 1 × 1.5 and 0.5 mm × 0.75 mm, respectively.

Before micro-compression testing, the micro-compression specimens were annealed for 1 hour at temperatures of 423, 573, 673 or 773 K, respectively, and the microstructures were then observed using an FEI Quanta 200FEG field emission scanning electron microscope (FESEM) with the data analyzed using a TSL OIM system. Samples were prepared for the microstructural observations by slicing the as-pressed billets perpendicular to the pressing direction to give disks having thicknesses of ~3 mm. One side of each disk was ground on SiC papers and mechanically polished with 0.5 μm diamond paste and the disks were then electro-polished to mirror-like surfaces using a solution of 10% $HClO_4$ and 90% C_2H_5OH with a DC voltage of 35 V under a temperature of 253 K. This procedure is summarized as the third step in Fig. 1.

In order to remove the scratches formed by grinding, all samples were electro-polished to smooth surfaces using the same solution and conditions in the fourth step in Fig. 1. Ultimately, micro-compression testing was conducted with engineering strains, ε, of 30% or 50% using an Instron machine at room temperature (~298 K) with initial strain rates in the range from 3.3×10^{-4} to 0.1 s^{-1}. No lubricants were used during these tests in order to avoid any frictional effects on the flow stresses [30] and finally the surface morphologies were examined using FESEM.

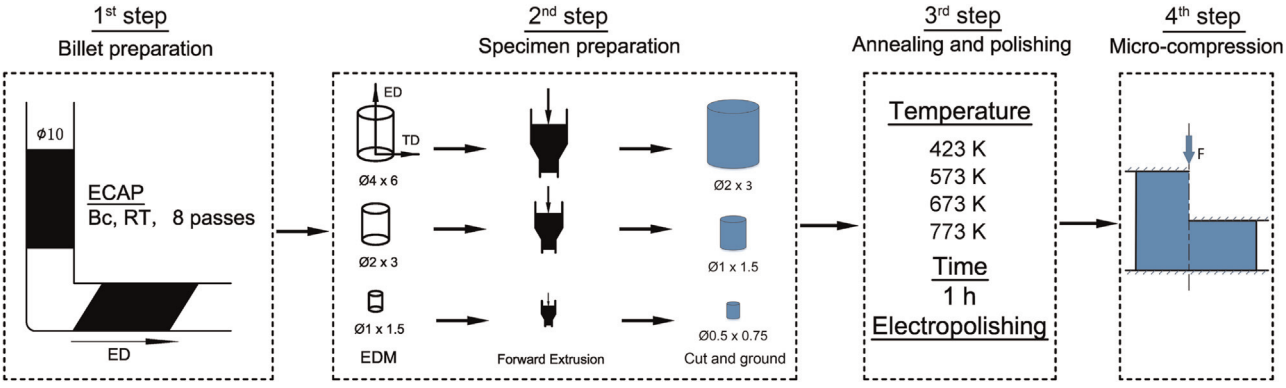

Fig. 1. Schematic illustration of the four stages of specimen preparation for micro-compression testing (unit: mm).