Materials Characterization 109 (2015) 181–188

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Effects of temperature, strain rate and specimen size on the deformation behaviors at micro/meso-scale in ultrafine-grained pure Al

Jie Xu [a,b,c,*], Jianwei Li [a,c], Lei Shi [a,c], Debin Shan [a,c], Bin Guo [a,c,*]

[a] Key Laboratory of Micro-systems and Micro-structures Manufacturing of Ministry of Education, Harbin Institute of Technology, Harbin 150080, China
[b] Academy of Fundamental and Interdisciplinary Sciences, Harbin Institute of Technology, Harbin 150080, China
[c] School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China

ARTICLE INFO

Article history:
Received 12 June 2015
Received in revised form 1 October 2015
Accepted 5 October 2015

Keywords:
Flow stress
Size effect
Micro/meso-scale
Ultrafine grain
Aluminum

ABSTRACT

An ultrafine-grained (UFG) high purity (99.999%) aluminum with the average grain size of ~1.0 μm was processed using equal-channel angular pressing (ECAP) through 8 passes. The UFG pure Al was annealed at different temperatures to obtain different grain sizes. Micro-compression testing was conducted with the specimen diameters of 2.0, 1.0, and 0.5 mm at elevated temperature and with the strain rate ranging from 3.3×10^{-4} to 0.1 s^{-1}. The results show that UFG pure Al has a strong temperature dependence and high strain rate sensitivity (SRS) compared with the coarse-grained (CG) pure Al. It is found the flow stress decreases with specimen size during micro-compression of both CG and UFG pure Al, which demonstrates that UFG pure Al still exhibits the conventional size effect of micro-forming. However, the significant improved surface roughening effect in micro-compression of the UFG pure Al demonstrates that UFG materials have a potential application in micro-forming to fabricate MEMS components.

1. Introduction

Micro-forming is becoming an important micro-manufacturing method with the potential to fabricate miniature/micro-parts for micro-electro-mechanical systems (MEMS) due to its advantageous characteristics for mass production with controlled quality and low cost [1–4]. Although the knowledge of tool design and fabrication methods are now well established for the conventional macro-forming, there is evidence that the presence of grain size effects may lead to some differences in these basic manufacturing characteristics when the feature size of specimen dimensions is scaled down to micro-scale [5,6]. Ultrafine-grained (UFG) materials have recently attracted considerable attentions in micro-forming because ultrafine grains can improve the formability, surface quality and high mechanical properties of MEMS components [7–9], which have a strong potential in making micro-parts by micro-forming processes [10–12]. At the present time, a number of severe plastic deformation (SPD) techniques such as equal-channel angular pressing (ECAP) [13] and high-pressure torsion (HPT) [14] are developed to produce UFG metallic materials. In practice, the ECAP processing is especially attractive as it is easy to perform with simple operation and this process can also produce bulk materials with resultant fine microstructures. It has been demonstrated that UFG materials including pure aluminum and alloys can be achieved by ECAP processing [15–18].

When the grain size of materials is decreased to submicron or nano-scale, micro-deformation behavior changes from dislocation dominated in large grains to grain boundary dominated in small grain regimes [19, 20]. To explore more on this, Yu et al. studied compressive behavior of UFG pure aluminum processed by ECAP and post-annealed specimens at room temperature and the results show that different work hardening behaviors were observed when the grain size was increased from 0.35 to 45 μm [21]. In addition, Sabirov et al. investigated the effect of strain rate on the micro-compression behavior of UFG pure aluminum and the results show that a decrease of strain rate results in the activation of micro shear banding [22]. May et al. investigated the strain rate sensitivity of UFG commercial purity aluminum using compressive tests and the results demonstrate that the pronounced strain rate sensitivity exists in UFG aluminum at elevated temperature [23]. Wang and Shan studied the effect of strain rate on tensile behavior of UFG pure aluminum and revealed that the deformation mechanism at lower strain rate in UFG pure aluminum may be related to grain boundary sliding [24]. Thus, it is believed that grain boundary activities, such as grain boundary sliding and grain rotation, are the main deformation mechanism in UFG materials [25]. However, there is still only limited information available in terms of micro-deformation behavior at

* Corresponding authors at: Key Laboratory of Micro-systems and Micro-structures Manufacturing of Ministry of Education, Harbin Institute of Technology, Harbin 150080, China.
E-mail addresses: xjhit@hit.edu.cn (J. Xu), bguo@hit.edu.cn (B. Guo).

http://dx.doi.org/10.1016/j.matchar.2015.10.003
1044-5803/

elevated temperature when the material grain size is reduced to sub-micrometer although these problems and limitations have attracted attention within the materials science community [26–28].

In this work, micro-deformation behavior in UFG pure Al processed by ECAP at elevated temperature and various strain rates at micro/meso-scale was investigated using micro-compression tests. The effects of temperature, strain rate and specimen size on flow stress during micro-compression were analyzed. Furthermore, the experimental results confirm that there is potential for using the UFG pure Al for applications in making micro-parts using micro-forming technology.

2. Experimental material and procedures

The experiments were conducted using a very high purity (99.999%) aluminum supplied in the form of drawn rods with the diameter of 10 mm and length of ~70 mm. An annealing treatment was performed at the temperature of 773 K for 1 h to give an initial grain size of ~300 μm. The ECAP processing was conducted at room temperature using a die with the internal angle of 90° between the two channels and an outer arc of curvature of 20° at the point of intersection. The materials were processed up to 8 passes by using route B_c. A UFG pure Al was obtained with the average grain size of ~1.0 μm and the fraction of high angle grain boundaries (HAGBs) (>15°) of ~71%, as shown in Fig. 1. The detailed information of ECAP processing of pure Al is described in the recent reports [29,30].

Following ECAP processing, the UFG pure Al rods were machined along the extrusion direction by electric discharge machining (EDM) to make the small cylinders with the dimensions of 4 × 6, 2 × 3 and 1 × 1.5 mm. These small cylinders were extruded with three different tools with the diameter of 2.0, 1.0, and 0.5 mm and at room temperature, as shown in Fig. 2. These extruded rods were cut into smaller cylinders with the diameter of 2.0, 1.0, 0.5 mm and the height-to-diameter ratio of 1.5, respectively, and then the end surfaces of the cylinders were ground to prepare micro-compression specimens with smooth surfaces.

Before micro-compression testing, these samples were annealed for 1 h at the temperature of 423, 573, 623, 673, 723 and 773 K, respectively. The samples for microstructural examination were prepared by grinding one end surface of the micro-compression sample on SiC papers and then mechanically polished with a 0.5 μm diamond paste. The samples were finally 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 at the temperature of 253 K. After electro-polishing, the microstructures of the annealed samples were observed by electron backscatter diffraction (EBSD) using a Quanta 200FEG field emission scanning electron microscope (FESEM) with the voltage of 30 kV and the data was analyzed using a TSL orientation imaging microscopy (OIM) system in the FESEM. To establish the settings of the OIM analysis, the scanning step size was pre-determined according to the measured area and the anticipated

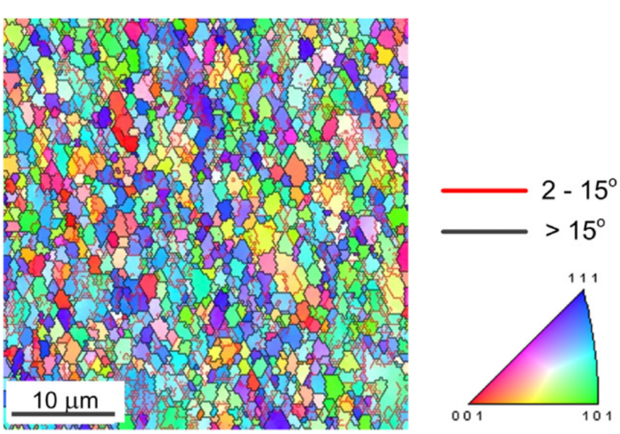

Fig. 1. Microstructure of UFG pure Al after ECAP processing through 8 passes.

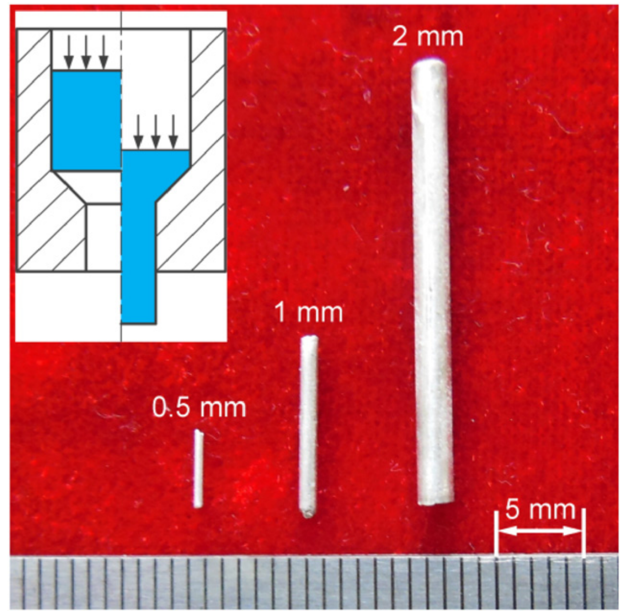

Fig. 2. Preparation of micro-compression samples with the diameter of 2, 1 and 0.5 mm using micro-extrusion.

grain size. Specifically, EBSD measurements were conducted over the area of 60 μm × 60 μm using a step size of 0.3 μm for the samples with ultrafine grain structures, the area of 120 μm × 120 μm using a step size of 0.7 μm for the samples with fine grain structures, and the area of 300 μm × 300 μm using a step size of 2 μm for the samples with coarse grain structures. According to Taylor's theory, the Taylor factor is a multiplication factor on the yield strength of an individual crystal. So Taylor factor can be used to analyze the influence of individual grain property on surface roughening during micro-compression. A graphical display of microstructure with Taylor factor distribution was generated by OIM analysis from each measurement area.

Micro-compression tests were conducted using the specimens with the diameter of 2.0, 1.0 and 0.5 mm and an Instron 5965 testing machine at elevated temperature from 298 to 523 K with initial strain rate ranging from 3.3×10^{-4} to 0.1 s^{-1}. The experimental parameters are shown in Table 1 in detail. The specimens were compressed by 50% of the original height for three repeated tests. In order to avoid the friction size effect in micro-compression, no lubricant was adopted in the experiments [31].

3. Experimental results

3.1. Microstructure of pure Al after annealing treatment

The microstructures with the distribution of Taylor factor for the annealed samples were analyzed and shown in Fig. 3. The average grain sizes were calculated from the OIM images using grain-to-grain measurement and these values were recorded in Table 2. The results show that ultrafine grains can be kept after annealing treatment at the temperature of 423 K, and then the grain size is increased from ~1.5 μm at 423 K to ~4.0 μm at 573 K, ~9.0 μm at 623 K, ~30 μm at

Table 1
Experimental parameters of micro-compression.

Experimental conditions	Values				
Temperature (K)	298	373	423	473	523
Strain rate $\dot{\varepsilon}$ (s^{-1})	1.0×10^{-1}	1.0×10^{-2}	1.0×10^{-3}	3.3×10^{-4}	–
Specimen dimension (mm)	2.0	1.0	0.5	–	–