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## Micro hot embossing of micro-array channels in ultrafine-grained pure aluminum using a silicon die



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#### ABSTRACT

An ultrafine-grained (UFG) Al with an average grain size of 1.0  $\mu$ m was produced by equal-channel angular pressing (ECAP) at room temperature. Micro hot embossing was performed on an UFG pure aluminum plate using a silicon die at a temperature of 523 K and under a force of 4 kN. Micro-array channels were embossed with feather widths in the range from 5  $\mu$ m to 100  $\mu$ m. The behavior of UFG pure aluminum during micro hot embossing was analyzed, and the results indicate that the channel dimension compared to the grain size is the main factor influencing the filling quality of micro-embossing. Micro hot embossing of UFG pure aluminum is characterized by the channel sidewall, surface quality, and fully transferred patterns, which shows potential for use in the fabrication of micro-electro-mechanical system (MEMS) components in mass production.

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#### 1. Introduction

Micro-array channels have potential application in the fields of micro-fluidics (e.g., components for micro-reactors or micro heat exchangers) or micro-optics (e.g., optical grating) and are crucial components of micro-electro-mechanical systems (MEMS) (Geiger et al., 2001). To improve the heat transfer efficiency, some metals with a high conductivity and low cost, such as aluminum, copper, and magnesium, have been used extensively to fabricate micro-array channels. Micro-forming becomes an attractive option in the manufacturing of these products because of its advantageous characteristics for mass production with controlled quality and low cost (Qin, 2006). The micro-forming review by Engel and Eckstein (2002) highlighted key technologies, and the recent review on the state-of-the-art micro-forming technologies by Fu and Chan (2013) demonstrated the size-related issues considered in the design and development of a micro-forming system. Neugebauer et al. (1999) developed a micro-embossing technology to fabricate metallic microstructures using a silicon die. Jiang et al. (2008) studied direct micro-embossing to fabricate microarray channels in conventional coarse-grained (CG) pure Al at room

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http://dx.doi.org/10.1016/j.jmatprotec.2015.06.025 0924-0136/© 2015 Elsevier B.V. All rights reserved. temperature. Obvious silicon die breakages were found that were caused by bending stresses induced by insert misalignment during embossing or uneven shear tractions during demolding. Otto et al. (2000) investigated high precision cold embossing in CG pure Al for the fabrication of a micro-optical grating using a silicon die. The embossed grating micro-array channels exhibited nonflat planks with ridges. Böhm et al. (2001) further investigated straight micro-channels and micro complex structures fabricated by cold and superplastic micro-embossing using a silicon die. As in conventional superplasticity, the widely accepted deformation mechanism in the micro-forming of polycrystalline metals proposed by Saotome et al. (1999) is grain boundary sliding and grain rotation rather than intragranular deformation, which results in the lack of ability to accurately form microstructures with sharp edges using CG metals due to the size effect (Vollertsen et al., 2009). Wang et al. (2007) investigated the effect of the microstructure on the formability of CG pure Al and found a minimum in the flow behavior during micro-embossing with a grain size close to two times the width of the forming die. Wang et al. (2012) validated the experimental results using a numerical simulation based on the multi-region model of the inner polycrystal region, the grain interior of the surface region and the grain boundary layer. Therefore, grain size appears to be the dominant factor that determines the limiting size of the geometrical features fabricated by microforming, this is, very small grain sizes, and especially materials

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having ultrafine grain sizes, are attractive for application in microforming operations

Among the processing techniques that may be used for grain refinement, the imposition of severe plastic deformation (SPD) has been well established as an effective process for producing significant grain refinement in polycrystalline metals (Valiev et al., 2006). At the present time, the most attractive procedures for processing by SPD are equal-channel angular pressing (ECAP) (Valiev and Langdon, 2006) and high-pressure torsion (HPT) (Zhilyaev and Langdon, 2008); however, ECAP is an especially attractive method from the research conducted by Stolyarov et al. (2001) because it is easy to perform in a simple laboratory operation and produce UFG materials with submicrometer or even nano-scale grain sizes. Yeh et al. (2006) investigated superplastic micro-formation using a fine grained Zn-22Al and found that this material exhibits very high ductility and good formability over an appropriate range of temperatures and strain rates. Estrin et al. (2007) investigated UFG net-shaped MEMS parts using ECAP and found that the UFG materials have the potential in micro-forming to fulfill an important condition: the average grain size is generally smaller than the smallest dimension of the component, which guarantees reproducible properties. Recent research performed by Xu et al. (2013) also demonstrated the potential for using UFG AZ31 alloy for applications in micro-forming at elevated temperatures, and some of these UFG materials have been used in the fabrication of MEMS components (Langdon, 2013). For example, Kim and Sa (2006) investigated micro-extrusion using an ECAP processed magnesium alloy to fabricate micro-gears with high strength. Qiao et al. (2010) investigated the fabrication of MEMS components using an UFG aluminum allov.

However, there is still limited research on micro-embossing with UFG materials to fabricate micro-array channels using a silicon die because the materials after ECAP processing are much harder than polymers and conventional CG metals, which can cause easy failure of the silicon die. The current study was initiated to produce micro-array channels with feathers that were  $5-100 \,\mu\text{m}$  in size by micro hot embossing in UFG pure Al that was processed by ECAP using a special tool that was designed with self-adaptive adjustment and a vacuum mounting system to protect the silicon die from damage. As will be demonstrated, the experimental results confirm that there is potential for using UFG pure Al for applications in the mass production of micro-array channels for MEMS components.

#### 2. Material and experimental procedures

#### 2.1. Preparation and characterization of UFG pure Al

The experiments were conducted using pure Al with a high purity of 99.999% supplied in the form of drawn rods having diameters of 10 mm and lengths of ~70 mm. ECAP processing was used to achieve a UFG pure Al 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, as shown in Fig. 1. Before ECAP, an annealing treatment was performed at a temperature of 773 K for 1 h to produce CG pure Al. All of the billets were sprayed with a MoS<sub>2</sub> lubricant and then processed by ECAP through 1, 2, 4, and 8 passes using processing route Bc, in which the billets are rotated by 90° in the same direction between each pass.

The samples for microstructural examination were prepared by slicing the as-pressed billets perpendicular to the pressing direction to yield disks with thicknesses of  $\sim 3$  mm. One side of the disk was ground on SiC papers and then mechanically polished with a 0.5  $\mu$ m diamond paste. Finally, the specimens were electropolished to mirror-like surfaces using a solution of 10% HClO<sub>4</sub> and 90% C<sub>2</sub>H<sub>5</sub>OH with a DC voltage of 35 V under a temperature of 253 K.



Fig. 1. Schematic illustration of the ECAP processing facility with an internal angle of  $90^{\circ}$ .

EBSD measurements were performed using an FEI Quanta 200FEG field emission SEM, and the data were analyzed using a TSL orientation imaging microscopy (OIM) system. For misorientation angle distribution statistics, the lowest cut-off angle was set at  $2^{\circ}$ , and a critical angle of  $15^{\circ}$  was used to differentiate the low-angle grain boundaries (LAGBs) (2–15°) from the high-angle grain boundaries (HAGBs) (>15°). In the EBSD maps, the HAGBs and LAGBs were depicted as black lines and red lines, respectively.

#### 2.2. Fabrication of the micro silicon die

Silicon polished wafers with a diameter of 100 mm and thickness of 1.5 mm were used as the micro-embossing die material. The micro silicon die was fabricated from the Harbin Research Institute of Transducer Technology using the standard induction coupled plasma (ICP) etching process, as shown in Fig. 2. First, AZ1500 photoresist was spin-coated onto the silicon wafer surface using a spin coater EVG101C. The photomask used for exposure was a positive resist formed from a Cr film with a thickness of 145 nm on a glass substrate from the Institute of Microelectronics of Chinese Academy of Science. The etched patterns, which are squares



Fig. 2. Silicon die fabrication using the ICP method.

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