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Effect of cold-work on the Hall–Petch breakdown in copper based micro-components

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ABSTRACT

Effects of substructural dimensions on the mechanical properties of micro-pins produced by an open-die micro-extrusion/forging process were studied. Micro-pins of diameter 0.3 mm were manufactured from copper strips, having different initial grain sizes. Micro-compression tests on the micro-pins revealed no significant size effect, even if the number of grains over the diameter of the micro-pins falls below its critical value. However, relaxation of the as-formed substructure using recovery annealing led to a surprising drop in the flow stress of the micro-pins. This was explained and attributed to the number of subgrains over the diameter of the micro-pins, showing the important role of subgrains rather than grains in determining the mechanical properties.

1. Introduction

Nowadays, there is an increasing demand for production of miniaturized metallic parts, especially in electronic and medical industries. Microforming processes, due to their well-known advantages such as high production rate, high material yield, and low costs, hold a promise for mass-manufacturing in a near feature (Geiger et al., 2001). However, well-established conventional rules of mechanics cannot simply be transferred and used in micro-scaled forming processes without considering possible size effects (Arzt, 1998; Miyazaki et al., 1979a).

Generally, the size effect is defined as a challenge that becomes significant as the dimension of grains becomes comparable to the dimension of the cross-section of the workpiece (Armstrong, 1961; Chan et al., 2010; Hutchinson, 2000). It causes unexpected and sometimes contradicting material behavior (Chan et al., 2011; Messner et al., 1994; Parasiz, 2008; Thompson et al., 1973; Yun et al., 2010).

Specifically, mechanical behavior of a polycrystalline metal mainly depends on the microstructural features such as grain size, as modeled by the Hall–Petch relationship in Eq. (1) (so-called grain size effect) (Arzt, 1998).

$$\sigma = \sigma_0 + \frac{k}{\sqrt{d}} \tag{1}$$

where σ_0 represents the grain interior resistant to deformation, and k is the strengthening coefficient. Both these parameters are constant at a given strain, based on Hall–Petch explanation (Hall, 1951; Petch, 1953).

Nevertheless, it has been reported that the thickness of the workpiece must be considered together with the grain size to determine the mechanical properties of a metal (Keller et al., 2011). In fact, depending on the stacking fault energy of the metal, there is a critical number of grains over the thickness of the workpiece (t/d), below which

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the mechanical behavior of metal does not follow the Hall–Petch relationship (Grain-Specimen Size Effect) (Daw-Kwei, 2009; Kim et al., 2007; Miyazaki et al., 1979b; Peng et al., 2007; Yeh et al., 2008).

As reported by Chan and Fu (2012), at a constant grain size, decreasing the specimen dimensions reduces the fraction of grain boundaries. This affects the grain boundary strengthening coefficient. In addition, it was stated that to accomplish deformation, smaller specimens required a lower dislocation density inside the grains. Therefore, both grain size and specimen dimensions contribute to the grain strengthening and overall material behavior during the forming process. The same conclusion was made by Ran et al. (2013). Based on this fact, a modified Hall–Petch equation was reported considering the interaction effects of specimen and grain size for fully annealed metals (Chan and Fu, 2012).

Keller et al. (2011) studied the microstructural aspects of the grain and specimen size effects as two separate parameters. It was reported that decreasing the t/d ratio below a critical value leads to a reduction in flow stress, and strain hardening rate. The interactive effects of grain- and specimen size on the dimension of dislocation cells during sheet forming was also evaluated.

In another report, Keller et al. (2010) stated that there must be a delay in cross slip mechanism by increasing the t/d value. Thus, decreasing the t/d ratio leads to formation of smaller dislocation cells (subgrains) during forming. Following that, Hug and Keller (2010) defined a relationship between the dislocation cell size and the strain hardening during forming processes. Furthermore, Gracio et al. (1989) had reported before that especially at large strains, the substructural properties have a significant effect on the mechanical properties of copper.

The material used in all the above-mentioned reports was fully annealed before mechanical characterization. Consequently, the initial dislocation structure of all the previous studies was rather the same, with almost no initial dislocation cells or subgrain developed in the microstructure before mechanical testing. This indicates a lack of detailed investigation in subgrain size effects.

The aim of this study is to further understanding of the interactive effects of grain/subgrain size and specimen dimensions on the mechanical behavior. Micro-pins having various grain sizes, and different initial dislocation states were manufactured for micro-compression test. Electron Backscattered Diffraction (EBSD) was used to study the substructure. The Hall–Petch relationship was utilized to evaluate the effects of microstructure on the mechanical behavior.

2. Materials and methods

2.1. The microforming process

A previously developed open-die progressive microforming process (Ghassemali et al., 2013c) was used to manufacture micro-pins. The process consisted of two stages: (I) pin forming by forward extrusion, and (II) blanking. In the first stage, which was the main stage, a strip was deformed by a punch of a defined diameter, and specified displacement. As a result, a portion of the material was forward extruded into the die orifice. In the second stage, the as-formed pin was blanked out from the strip material. More details of the process can be found in Ghassemali et al. (2013c).

All the experiments were done under dry non-lubricated condition. The punch speed was 0.1 mm/s. The strip was punched until 0.2 mm of the remaining thickness to ensure that the amount of plastic deformation had applied to the workpiece was the same for all the micro-pins (the thickness of the head part was 0.2 mm at the end).

2.2. Material

Electrical Tough Pitch (ETP) C11000 copper (99.94%) strips, in the as-received cold-rolled condition were used as the initial feed for the process. In a previous study (Ghassemali et al., 2013a), it was shown that under certain geometrical conditions, a dead metal zone (DMZ) appeared at the pin surface. According to those findings, to eliminate the effects of the DMZ on the pin's behavior, a punch of diameter 2.0 mm was used for production of the 0.3 mm pin from a 2.5 mm thick strip.

2.3. Grain/subgrain size variation

The initial grain size of the Copper strip was 14 ± 4 μm, excluding twin boundaries. To change the number of grains over the thickness (t/d), the initial strips were annealed at 400 °C and 800 °C in a Lenton vacuum tube furnace under vacuum of 10^{-5} mbar using a ramp of 3 °C/min for 1 h. Longer dwelling time for annealing had no significant effects on the mean grain size.

After pin forming, as the micro-pins were work-hardened, they all contained a relatively dense dislocation structure inside the grains. To have a comparison, one batch of the micro-pins was annealed below the recrystallization temperature to recover the dislocation substructure without changing the mean grain size.

Recovery is an annealing phenomenon occurring prior to recrystallization, to annihilate the substructure without altering the grain size. The percentage of the substructural relaxation depends on the stacking fault energy of the metal (Humphreys and Hatherly, 2004b). Since Copper has a relatively medium range stacking fault energy, under precise temperature control, recovery occurs in the microstructure before recrystallization (Luton et al., 1980). The main phenomena occurring in the microstructure during recovery includes, dislocation annihilation within dislocation cells and at grain boundaries, coalescence, and growth of subgrains. Subgrain growth rate depends on the mobility of subgrain boundaries and their misorientation angles (Humphreys and Hatherly, 2004a). In fact, recovery annealing stabilizes the subgrain structure without having any significant effects on the mean grain size (Van Drunen and Saimoto, 1971).

Although it is difficult to define and control the recovery process without having any recrystallization in medium or high stacking fault energy metals, a temperature of 230 °C was selected for 1 h as the recovery annealing (RA) cycle