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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.

Keywords: Microforming; Subgrain; Microstructure; Size effect; Dislocation Cells.

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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 future (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).

$$\sigma = \sigma_0 + \frac{k}{\sqrt{d}} \quad (1)$$

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 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 hour. 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 hour as the recovery annealing (RA) cycle for the copper alloy (Van Drunen and Saimoto, 1971). This was done in the Lenton vacuum tube furnace under vacuum of 10^{-5} mbar using a ramp of 10°C/min, and then cooled in the air. The recovery-annealed micro-pins were named as RA micro-pins in this study.

Consequently, there were two sets of micro-pins for investigation with similar dimensions and similar grain size, but different subgrain size: as-formed micro-pins vs. RA micro-pins.

2.4. Characterization methods

A programmable servo press (SCHMIDT ServoPress 420) was used for the microforming process and also mechanical testing. The load-displacement behavior of the punch was monitored precisely, with a maximum of 1% error. The maximum forming force was recorded with a resolution of 0.01 kN.

To compare the pre-production mechanical properties of the initial strips, compression test was done on cylindrical specimens of 1.67 mm in diameter and 2.5 mm in height (aspect ratio of 1.5), machined in the normal direction of the strips. Two layers of Teflon of thickness 0.1 mm, were used as a lubricant on the top and bottom surface of the test-specimens. To further eliminate the friction effects on the compression results, the tests were stopped at steps of 10% reduction to renew the lubricant layer. The punch speed in

compressions tests was set to 0.1 mm/s. An average of the three upsetting test results was reported as the mean stress-strain behavior of the copper strips annealed at different temperatures.

An Olympus light microscope was used for microstructural observations. The specimens were mounted, ground, polished and subsequently etched in an Ammonia Persulfate solution.

Field Emission Scanning Electron Microscope (FESEM, JEOL JSM-7600F) was used for Electron Backscatter Diffraction (EBSD) analysis. The FESEM was conducted with an Oxford Instruments HKL EBSD system working at 20 kV with a working distance of 28 mm and a 70° tilt angle. The CHANNEL 5 suites of programs developed by Oxford Instruments was adopted to manipulate, analyze and display EBSD data in the current study.

For the microstructure determination, individual specimen locations were investigated using step size of 0.2 μm to be able to detect small subgrain/dislocation cell size with the least error. Lower values of step size had no significant effects on the EBSD results (Ghassemali et al., 2013d). The rate of successful indexing for EBSD data collection was higher than 70% in all cases, which is reliable for determining the crystallographic texture (Wu and Juul Jensen, 2008).

In the high-resolution EBSD, high angle grain boundaries (HAGBs, misorientation $>15^\circ$) were depicted as black lines. In order to avoid spurious boundaries, misorientations below 2° were not taken into account. It is worth noting that although most of researchers consider the upper limit of 15° as for the low angle grain boundaries (LAGBs), but some do the 10° as a threshold misorientation (Humphreys, 2001). Therefore, to be in a safe zone, misorientations between 2° to 10° were considered as LAGBs. Misorientations between 10° to 15° were also depicted for reference. Grain size measurement was carried out in the EBSD software by determining the grain areas exclusive of border grains. The mean values of the subgrain/grain size of the microstructures were obtained from the

statistical evaluation tool embedded in the EBSD software. The same route has been used before (Bate et al., 2005; Cao et al., 2003; Salimyanfard et al., 2011).

Due to the symmetry of the micro-pin, merely half-cross-section of the pins was investigated using EBSD technique, as shown in Fig. 1. To reduce the EBSD processing time, only a portion of the pins' microstructure was analyzed.

Fig. 1 Schematic of the half-cross-section of the micro-pin, showing the location for EBSD mapping

2.5. Micro-compression

Due to very small dimensions, fabrication of the tensile test specimens from the micro-pins was difficult. As an alternative to obtain the mechanical response of the micro-pins or micro-pillars, the micro-compression test was proposed. The main challenge in micro-compression tests is holding the micro-cylinder during the test. Zhang et al. (2006) used finite-element simulation to propose the usage of a base attached to the pins for stabilizing the pins under the indenter. The reported feature is similar to the final product in our progressive microforming process (Ghassemali et al., 2013c). Therefore, the manufactured micro-pins can be directly used for studying the post-production size effects via micro-compression test (Fig. 2).

Fig. 2 Schematic of the micro-compression test

Zhang et al. (2006) also studied the effects of the pin aspect ratio (pin length to its diameter) on the deformation behavior in micro-compression. To be in a safe region in case of buckling, a constant aspect ratio of 2.5 was chosen for our micro-pins. As previously investigated (Ghassemali et al., 2013b), the aspect ratio of the micro-pins can be easily controlled in the progressive microforming process by selecting a proper strip thickness and punch diameter. The micro-compression result was obtained as an average of at least three micro-compression tests.

To ensure the accuracy of the results, a visual inspection was done in each sample after the test. As shown in Fig. 3, buckling/tilting can occur due to inaccurate positioning of the pins under the punch. The center of the pin must be exactly matched with the center of the punch, which was done empirically. Zhang et al. (2006) also mentioned that no lubricant must be applied on the pin tip to prevent a significant buckling or tilting. A 0.1 mm thick Teflon layer was, used under the base (head part) to eliminate the extra friction effects on the mechanical response.

2.6. Finite Element (FE) simulation

DEFORM-2D™ (Version 10.1.2) was used for the micro-compression simulation. Due to the symmetry of the process, only the half-cross-section of the material was simulated using cylindrical coordinated in axisymmetric condition (indicated X for radial direction and Y for height).

The micro-specimen mechanical behavior was considered as rigid-plastic. The four-node quad element was used to mesh the strip. By running the simulation, the top-die moves down in the $-Y$ direction and deforms the strip into the die orifice. The mesh size of $0.05\ \mu\text{m}$ was used.

After setting the conditions, re-meshing sensitivity test was done for various re-meshing criteria values, and based on that, the re-meshing criteria were set to either every $0.5\ \text{s}$ or every $0.05\ \text{mm}$ punch displacement. Decreasing the re-meshing criteria under the mentioned values increased the computing time, without any significant effect on the accuracy of the results. More details of the FE simulation can be found in (Ghassemali et al., 2014)

3. Results and discussion

3.1. Test procedure, validation and quality assertion

It is important noting that due to the geometry of the specimens, it is impossible to avoid buckling at the bottom half of the pin near to the base. This fact is depicted in Fig. 3-a as a barreling shape on the bottom-half of the micro-pin under compression, which could cause inhomogeneous stress states.

Fig. 3 Compressed micro-pin: a) without buckling-uniformly deformed; b) with buckling-faulty

Regarding the inhomogeneous deformation in the micro-compression test, two separate parameters were investigated: (i) friction on the pin tip, and (ii) the head part (Base) effect.

3.1.1. Friction effects on micro-compression results

Two different values of shear friction coefficients of 0.1 and 1 were selected, to represent the low (Teflon) and high (Dry) friction conditions, respectively (Ghassemali et al., 2014). FE simulation showed that using lubrication on the pin top surface has no specific effect on the stress distribution (Fig. 4). Nevertheless, a so-called dead-metal-zone (DMZ) was detected at the center of the pin top-surface in the case of high friction (without lubrication, Fig. 4b), which is common in compression tests (Ghassemali et al., 2013a).

Fig. 4 FE simulation of the half-cross-section of the micro-compression showing effective-stress distribution in the micro-parts with two different friction coefficient values of: a) 0.1, and b) 1

The specimens' mechanical response proved such a conclusion, as the flow stress of the two specimen showed below 10% difference as the stress error (Fig. 5).

Fig. 5 Effect of friction on the mechanical response of the micro-specimens obtained from the FE simulation. “m” values represent the friction coefficient values. Stress error represents the difference between the two curves in %

3.1.2. Head (Base) effects on micro-compression results

The effect of the head (Base) material on the overall mechanical response of the micro-parts was also investigated using FE simulation. Results showed maximum 6% difference in flow stress, illustrated as the stress error curves in Fig. 6. In this regard, the flow stress of the specimens with Base showed lower values, which is due to the compensation of the plastic deformation by the Base material.

Fig. 6 Effect of the Base material (head part) on the mechanical response of the micro-specimens obtained from the FE simulation ($m=1$). Stress error represents the difference between the two curves in %

This result is in accordance to the report by Fei et al. (2012). He showed that the share of the base deformation on the overall flow stress could be negligible, since the main deformation (more than 90%) occurs at the pin rather than the base. Nevertheless, these factors could induce a geometrical error in measurements (less than 10% in strain/stress measurements). However, the magnitude of the geometrical error is rather the same for all the micro-compression test samples, due to similarity in shape and dimensions. On the other hand, since size effect investigation is a comparative study and not an absolute-value study, the comparisons and related discussions would be still valid.

3.2. Initial material properties

Figure 7 shows the microstructure of the initial strips annealed at different temperatures for 1 hr (Pre-production annealing). As can be seen, there is a reasonable range of grain sizes (from about 14 μm to around 110 μm) for investigation. To normalize the results, the t/d ratio of the strips was calculated, as presented in Table 1.

Fig. 7 Optical micrograph, grain size distribution, and mean grain size of the C11000 copper alloy annealed at different temperatures for 1 hr: a) As-received, b) 400°C, c) 800°C

Table 1. Mean values of t/d for the initial strips annealed at different temperatures for 1 hr

Annealing temp.(°C)	As-received	400	600	800
Number of grains over strip thickness	180	45	26	23

Figure 8 illustrates the effects of the pre-production annealing temperature on the mechanical properties of the C11000 copper strips. From the slope of the flow curves of the annealed specimens, it can be concluded that annealing above 400°C has a negligible effect on the strain hardening of the strips.

Fig. 8 Room temperature flow stress curves of the CC11000 copper alloy achieved by upsetting test after annealing at different temperatures for 1 hr

3.3. In-production grain size effect

The pin height and the maximum forming load were recorded. As can be seen in Fig. 9, there is a negligible change in the average pin height from 2.5 to 2.6mm by increasing the initial grain size. This marginal increase can be attributed to the annealing treatment that softened the material, leading to a slight ease of material flow into the die orifice at the initial stages of the process. However, it is difficult to make a specific conclusion from this figure as the results overlap on their standard deviation.

As mentioned in a previous study (Ghassemali et al., 2013c), the micro-part final dimension in this type of open-die process is determined by the location of the neutral plane, which mainly depends on the relative dimensions of the process (Avitzur, 1968).

Indeed, the location of the neutral plane determines the amount of material flowing towards the die orifice, which forms the final micro-part. Therefore, the initial properties of the forming metal, theoretically, have no specific effect on the final part dimensions under these conditions. It is worth noting that a pin aspect ratio (Pin height/Pin diameter) of more than 8.3 was achieved by this process, in dry conditions (Fig. 9).

Fig. 9 The effect of grain size (annealing temperature in 1 hr) on the final part dimensions in the open-die micro-forming process. (For the pin diameter of 0.3 mm manufactured from the 2.5 mm strip thickness)

The required forming load during the process is shown in Fig. 10 for specimens with different grain size. As reported previously (Ghassemali et al., 2013c), the load-displacement curve consisted of three stages, including indentation at the beginning, the middle stage of upsetting with lower slope, and the final extrusion stage with a relatively sharper slope. As can be seen in Fig. 10, increasing the grain size leads to lowering the forming load during the first two stages. Indeed, at the first stages of the process, by increasing the grain size, there is less force required for metal flow. However, at the final stage (extrusion), where the metal mainly flows towards the die orifice, there is no significant difference in forming load for various specimens (Fig. 10). This is so because by a relative increase of the grain size, it is more difficult for the metal to flow inside the die orifice, as reported by Wang et al. (2007). Therefore, by increasing the grain size, although the metal gets

softer, more redundant work must be done to develop a network of dislocation cells inside the grains and shear the grains to flow inside the die orifice. The balance between softening and the redundant work makes the total force relatively closer to that of the as-received specimen at this stage. This again implies no specific “in-process” grain size effect, since the t/d ratio of the initial strip was above its critical value.

Fig. 10 Load-displacement curve for pin forming using samples with different initial grain sizes (For the pin diameter of 0.3 mm manufactured from the 2.5 mm strip thickness)

3.4. Post-production grain size effect

3.4.1. Hall-Petch breakdown

The micro-compression test revealed no significant specimen-grain size effects in the mechanical response of the as-formed micro-pins (Without RA) (Fig. 11). For these type of samples, there was a little difference between the flow stress of the as-received and 800°C, which is logical due to the well-known Hall-Petch equation.

Fig. 11 Effects of initial grain size and recovery annealing (RA) on the micro-compression of the CC11000 copper alloy

Mechanical response of the RA micro-pins, however, showed a different behavior: compare to the as-formed micro-pins, a decline in the flow stress for each RA micro-pin was noticed. It is important noting that the difference between the two curves in this condition is higher than the stress error calculated in Fig. 6, which validates the discussion here.

Furthermore, since the heights of the compressing micro-pins were relatively small (around 0.75 mm), there were only a few numbers of data points obtained from the micro-compression test, as plotted in Fig. 11.

▶ The consistency of the results with the Hall-Petch equation for the as-formed micro-pins could be derived from Fig. 12-a, with a linear relationship (with R^2 value of nearly equal to unity) between the flow stress and the inverse square root of the grain size ($\frac{1}{\sqrt{d}}$). In comparison, for the RA micro-pins, size effect could be seen as a deviation of the results from the Hall-Petch linear relationship (Fig. 12-b). This is shown quantitatively with the R^2 values, which was relatively lower

than that of Fig. 12-a. Nevertheless, to clearly validate the above-mentioned hypothesis, more experimental results seem necessary.

Fig. 12 Flow stress vs. grain size for distinct strains obtained from the micro-compression test of micro-pins with different grain sizes, under different post-production conditions of: a) as-formed, without RA; b) after RA

Using Fig. 12 and Eq. (1), the change of σ_0 and k in the Hall-Petch relationship with strain is depicted in Fig. 13. As shown in Fig. 13-a, the σ_0 is smaller for the RA micro-pins, for the whole

range of strain. In fact, due to recovery annealing, the density of dislocation forests is decreased, and therefore, less stress is required for dislocations' mobility inside the grains. This behavior also implied that a lower dislocation density was required in the grain interior of the RA micro-pins to accomplish deformation, as reported by Chan and Fu (2012). The strengthening coefficient of k , in general, explains the significance of the grain size effect on the overall mechanical properties. Figure 13-b illustrates a relative increase in the k value with recovery annealing, for the whole strain range. As a result, recovery annealing on the highly deformed micro-pins could significantly increase the grain size effects (see Fig. 11).

Fig. 13 Changes of: a) σ_0 ; and b) k ; with strain for the RA and as-formed micro-pins

3.4.2. Microstructural studies

To justify the above-mentioned behavior, the substructure of the micro-pins was investigated using EBSD mapping, which showed a weak preferred $\langle 001 \rangle$ orientation (Fig. 14).

As can be seen, considering the LAGBs misorientations as higher than 15° instead of 10° had no significant effects on the subgrain size reported here. Besides, it can be concluded from Fig. 14 that the volume fraction of the twin boundaries in the microstructure is almost the same for all the micro-pins, as presented in Table 2.

Table 2. Fractional volume of the $\Sigma 3$ boundaries (twin boundaries) obtained from EBSD analysis on micro-pins having different grain sizes

Annealing temperature of the initial strip, $^\circ\text{C}$	As-received	400	800
Volume fraction of $\Sigma 3$ boundaries, %	4.7	5.3	5.4

At the first glance, it is qualitatively seen that RA had negligible effects on the grain size (e.g. compare Fig. 14-a and Fig. 14-b). Although the recovery annealing temperature was controlled precisely, a little recrystallization in the microstructure was inevitable. However, this had no specific effect on the mean grain size. A decrease in the density of the LAGBs was seen after RA (i.e. the amount of silver-colored lines in Fig. 14-a vs. Fig. 14-b).

The interactive effects of pre-production annealing and post-production annealing on the grain size and subgrain sizes can be seen quantitatively in Fig. 15. A general increase on the mean grain size by raising the pre-production annealing temperature is observed for all samples, regardless of the post-production (recovery) annealing (Fig. 15-a).

Fig. 14 Inverse Pole Figure (IPF) EBSD maps obtained from half cross section of the as-formed 0.3 mm micro-pins manufactured from strips annealed at: a) as-received initial strip, c) 400°C, e) 800°C; and that of the RA 0.3 micro-pins manufactured from the strips annealed at: b) as-received initial strip, d) 400°C, f) 800°C. The grain boundaries with different misorientation angles are shown in a random area of each sample in the upper-right corner of each image (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article)

A different behavior is, however, observed for the mean subgrain size (Fig. 15-b): a very slight change in the subgrain size by increasing the initial grain size. This is because the size of the subgrains developed during forming is directly related to the pre-forming (initial) grain size (Keller et al., 2010; Staker and Holt, 1972). The same trend was seen for the RA micro-pins, with more magnitudes. In fact, the recovery annealing coarsened the substructure size, up to a size comparable to the mean grain size. It occurs via dislocation annihilation, and subgrain coalescence and growth (Humphreys and Hatherly, 2004b, c).

Fig. 15 Effects of recovery annealing (RA) on the: a) Mean grain size; and b) Mean subgrain size for the 0.3 mm micro-pins manufactured from CC11000 copper strips, initially annealed at different temperatures

As mentioned by Keller et al. (2011), the size effect could be only obvious if the t/d value of the metal goes below the critical number. This value has been reported between 5 to 8 as the critical t/d

ratio for copper, depending on the plastic strain path and sample thickness (Dubos et al., 2013; Hug et al., 2013; Liu et al., 2013; Molotnikov et al., 2012; Weiss et al., 2002). To have a quantitative comparison, the extracted data from Fig. 15 is presented in Table 3.

Table 3. Mean grain size, mean subgrain size, and number of grains and subgrains across the diameter of the 0.3 mm micropin in different pre- and post-production annealing conditions

Pre-production annealing condition	Mean grain size (μm)		Mean subgrain size (μm)		N_G		N_{SG}	
	With RA	Without RA	With RA	Without RA	With RA	Without RA	With RA	Without RA
As-Received	5.4 ± 1.3	7.2 ± 2.1	4.3 ± 1.0	0.8 ± 0.2	56	42	70	375
400°C	13.0 ± 4.1	14.5 ± 3.4	11.3 ± 2.1	0.9 ± 0.3	23	21	27	333
800°C	47.0 ± 7.3	44.3 ± 6.7	38.2 ± 5.0	1.5 ± 0.4	6	7	8	200

* RA: Post-production recovery annealing at 230°C for annealing.

** N_G and N_{SG} are the number of grains and number of subgrains over the micro-pin's diameter, respectively.

As can be seen in Table 3, especially for the coarse-grained micro-pin, the number of grains over the diameter of the micro-pin (N_G) was decreased down to around the critical value (the upper bound value) for the copper in both cases of the RA and as-formed micro-pins. However, in as-formed micro-pins (without RA), since the number of subgrains over the diameter (N_{SG}) was relatively higher than the critical value, no size effect was seen. In comparison, in the RA micro-pins, since the number of subgrains across the diameter was around the critical value, size effect on the mechanical behavior was more evident (Fig. 12-b).

It has been proven that at the early stages of deformation, grain boundaries are the main obstacles for dislocation mobility (William D. Callister and Rethwisch, 2010). In higher strains (or in a previously deformed metal), however, subgrain structure could also slow down the dislocation motion. Indeed, subgrain structures are considered as arrays of dislocations, which could be an

obstacle for dislocations' mobility, or at least delay the escape of dislocations through the specimen surface, especially when forming is done at low temperatures (Hallberg et al., 2010). Thus, the smaller the number of subgrains over the cross-section (N_{SG}), the fewer will be the amount of obstacles for dislocations to escape out from the specimen surface, which in return, decreases the flow stress.

This implies the importance of the number of subgrains rather than grains over the cross-section of the component for determining the size effect on mechanical behavior. To the best knowledge of authors, this fact has not been addressed in literature by now. It can be also a reason for contradicting reports on size effects in the literature (Vollertsen, 2008).

4. Conclusion

The size effect was experimentally analyzed via examining the substructure of micro-pins manufactured by an open-die micro-forging/extrusion (subgrain size effects). The proposed open-die forging/extrusion process was introduced as a microforming process which was not sensitive to the “in-process” size effect. This fact made the process more suited to be used for mass production of axisymmetric micro-parts. It was experimentally shown that decreasing the number of grains over the thickness of the specimen (t/d) will not always cause a size effect on the mechanical behavior. Instead, the initial substructural dimensions must be considered. In this case, recovery annealing in coarse-grained micro-parts caused a deviation from the Hall-Petch equation in studying the mechanical properties of the final product, which was due to the coarsening of subgrain dimensions. In fact, recovery annealing leads to annihilation of the dislocation structure and subgrain growth, especially for coarse-grained specimens. This increases the mean free path of the dislocations through the grains. Consequently, the flow stress decreases for the recovery-annealed (RA) samples. It is suggested to analytically validate the interactive effects of the specimen-grain-

subgrain dimensions on the mechanical behavior, and according to that, modify the Hall-Petch equation.

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Figure Captions

Fig. 1 Schematic of the half-cross-section of the micro-pin, showing the location for EBSD mapping

Fig. 2 Schematic of the micro-compression test

Fig. 3 Compressed micro-pin: a) without buckling-uniformly deformed; b) with buckling-faulty

Fig. 4 FE simulation of the half-cross-section of the micro-compression showing effective-stress distribution in the micro-parts with two different friction coefficient values of: a) 0.1, and b) 1

Fig. 5 Effect of friction on the mechanical response of the micro-specimens obtained from the FE simulation. “m” values represent the friction coefficient values. Stress error represents the difference between the two curves in %

Fig. 6 Effect of the Base material (head part) on the mechanical response of the micro-specimens obtained from the FE simulation (m=1). Stress error represents the difference between the two curves in %

Fig. 7 Optical micrograph, grain size distribution, and mean grain size of the C11000 copper alloy annealed at different temperatures for 1 hr: a) As-received, b) 400°C, c) 800°C

Fig. 8 Room temperature flow stress curves of the CC11000 copper alloy achieved by upsetting test after annealing at different temperatures for 1 hr

Fig. 9 The effect of grain size (annealing temperature in 1 hr) on the final part dimensions in the open-die micro-forming process. (For the pin diameter of 0.3 mm manufactured from the 2.5 mm strip thickness)

Fig. 10 Load-displacement curve for pin forming using samples with different initial grain sizes (For the pin diameter of 0.3 mm manufactured from the 2.5 mm strip thickness)

Fig. 11 Effects of initial grain size and recovery annealing (RA) on the micro-compression of the CC11000 copper alloy

Fig. 12 Flow stress vs. grain size for distinct strains obtained from the micro-compression test of micro-pins with different grain sizes, under different post-production conditions of: a) as-formed, without RA; b) after RA

Fig. 13 Changes of: a) σ_0 ; and b) k ; with strain for the RA and as-formed micro-pins

Fig. 14 Inverse Pole Figure (IPF) EBSD maps obtained from half cross section of the as-formed 0.3 mm micro-pins manufactured from strips annealed at: a) as-received initial strip, c) 400°C, e) 800°C; and that of the RA 0.3 micro-pins manufactured from the strips annealed at: b) as-received initial strip, d) 400°C, f) 800°C. The grain boundaries with different misorientation angles are shown in a random area of each sample in the upper-right corner of each image (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article)

Fig. 15 Effects of recovery annealing (RA) on the: a) Mean grain size; and b) Mean subgrain size for the 0.3 mm micro-pins manufactured from CC11000 copper strips, initially annealed at different temperatures

Table Captions

Table 1. Mean values of t/d for the initial strips annealed at different temperatures for 1 hr

Table 2. Mean grain size, mean subgrain size, and number of grains and subgrains across the diameter of the 0.3 mm micropin in different pre- and post-production annealing conditions

Table 3. Mean grain size, mean subgrain size, and number of grains and subgrains across the diameter of the 0.3 mm micropin in different pre- and post-production annealing conditions

Research Highlights

The paper demonstrates the following research highlights

- The interactive grain/subgrain size effects in micro-parts' mechanical properties.
- A low value of t/d will not always cause size effect.
- Substructural dimensions must be considered in for Hall-Petch modifications.