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Research Note

Nano-grain refinement and strengthening of copper under room temperature RUE process

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Abstract. In this investigation, the Repetitive Upsetting-Extrusion (RUE) process was used to investigate effect of severe plastic deformation on the microstructural changes and flow behavior of commercial pure copper. Initial materials together with two, four, and eight passes of RUE under annealing and non-annealing conditions were studied. Results show that grain refinement, on the scale of nanometer, has been mostly achieved only after two passes of RUE, which is essentially a combination of one upsetting and one extrusion path. Increasing the number of passes, following four RUE passes, did not have discernible effect on the grain refinement. Such behavior is explained through saturation of dislocations and the formation of high angle grain boundaries after only two passes of RUE. The grains even became slightly larger after eight passes of RUE process as compared to the two and four passes of RUE; such a result corresponds to restoration phenomena occurring during a high number of passes of RUE process. Flow strength of the material after different passes substantially increased, though the rate at which the flow stress increased declined by increasing the number of passes. ETMB model was used to explain the deformation behavior of the RUE samples.

1. Introduction

Ultrafine-grained (UFG) metallic materials containing grain sizes in the range of nanometer were extensively studied in the past two decades, owing to their somehow exceptional mechanical properties [1]. Various Severe Plastic Deformation (SPD) methods were employed to achieve a range of nano-grain sizes. Some of the usual SPD methods include Accumulative Roll Bonding (ARB) [2], Equal Channel Angular Pressure (ECAP) [3], Hot Pressure Torsion (HPT) [4], twist extrusion [5], and Accumulative Back Extrusion (ABE) [6]. Among them, Repetitive Upsetting-Extrusion (RUE) is a relatively new process among severe deformation processes [7,8]. Aizawa and Tokimutu first introduced the process for mechanical alloying of powders [9]. One of the characteristics of RUE process is the relative ease of the instrumentation to be used and compared with other SPD methods. Apart from this, other advantages of RUE process include higher strain in each cycle, higher number of shear planes, and, consequently, more effective grain refinement, no need for extra machining, etc.

Excessive strain in the structure results in grain refinement in which, based on Hall-Petch relationship, mechanical properties, particularly of the strength are developed [10]. This is principally the case when the grain size reaches below one micron, where a good combination of strength and ductility is often achieved. In the past few years, attempts were made to modify the die design so as to use the RUE process for bulk ma-

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terials such as copper and aluminum alloys [11,12]. In addition, numerical analysis, mostly by finite-element analysis, was employed to understand the flow patterns during successive upsetting and extrusion in the RUE process [13].

Despite the efforts made to establish the process for different materials, there is still much work to do to analyze experimentally the flow pattern and microstructural changes in different materials, especially of copper alloy. Though some efforts were made on the pure copper in the past, the aim of this study was to explore some parameters of the RUE process such as the number of passes, annealing on the evolution of microstructure, and changes in mechanical properties in a copper alloy.

2. Materials and experimental procedure

2.1. Materials

Commercial pure copper was used for RUE tests. Table 1 shows the chemical composition of the materials used for such tests. Cylindrical samples with the diameter of 30 mm and the height of 45 mm were cut from the initial sample. These samples were then used for RUE as well as annealing processes.

2.2. RUE process

Generally, a RUE process includes successive application of upsetting and extrusion. Figure 1 shows a schematic of the principle of the RUE process. In this process, first, the cylindrical sample undergoes an upsetting process (Figure 1(a)). In this process, the flow of the material is perpendicular to the punch direction. Therefore, an elongated grain structure, with its axis being horizontal, is observed after the upsetting process. Subsequent to the upsetting process, an extrusion is applied where the length of the specimen increases at the expense of the cross-sectional area. In addition, it is well known that, in the extrusion process, grains are elongated in the direction of the extrusion process in line with the flow of the material [14]. Such upsetting and extrusion processes are repeated several times, depending on how much strain is intended to be applied. It is important to note that changing the directions of the grains in the RUE process gives a very high compressive strain and homogenous small-sized grains.

An oil hydraulic press with the capacity of 250 Tons was used to apply the deformation. A modified die design proposed by Balasundar et al. was used to conduct the RUE process. Additionally, appropriate die, punches and holder were designed to conduct RUE tests. Punches used include the main punch, upsetting punch, and extrusion punch (Figure 2). The die and all the punches were made from hot work tool steel of grade 2344 [15].

The die and punches were first pre-heated at 200°C to reduce porosity prior to the RUE processes. As the RUE process starts with the upsetting, the upsetting punch was first inserted into the press. To reduce the friction, a graphite lubricant was used on the surfaces of the die and the punches. The press moved up after the upsetting stage to open up the punch and replace it with the extrusion punch. Sample was then taken out of the die, and after reversing the direction, it was again put into the die for the next straining stage, i.e., extrusion. The extrusion process was performed similar to the upsetting one. Such a process is considered as two passes of RUE. Four and

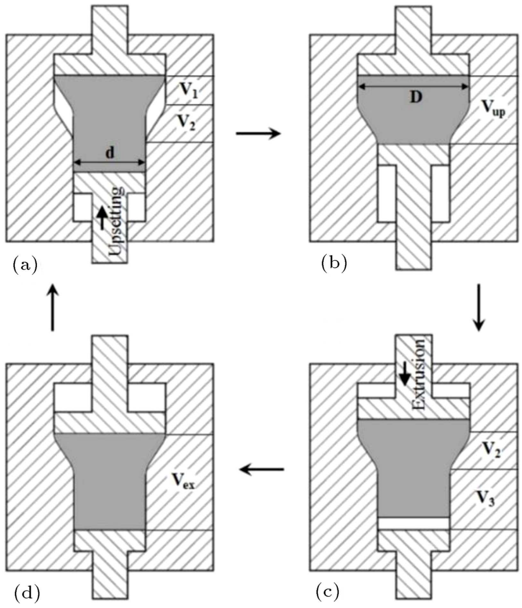


Figure 1. Schematic illustration of the RUE process: (a) Upsetting, (b) finishing of upsetting, (c) extrusion, and (d) finishing of extrusion.

Figure 2. Die and punches used for the RUE process.

Table 1. Chemical composition of the materials used for RUE tests.

Element	Cu	Sb	Ca	Sn	S	Mg
Weight %	99.44%	0.007	0.02	0.006	0.025	0.47

Figure 3. Specimen before and after two passes of RUE.

eight passes of RUE also were performed similar to the method explained above. As an example, Figure 3 shows the sample before and after two passes of RUE.

2.3. Annealing process

Annealing was performed on the RUE processed samples. Given that the recrystallization temperature for pure copper is about 250°C and providing that relatively high amount of strain is stored in the system after successive deformation, the recrystallization temperature for such a copper alloy would reduce. Therefore, 200°C was selected as the annealing temperature of the RUE processed materials. All the samples were annealed at this temperature for 20 min.

2.4. Compression test

The RUE samples were first cut in half (Figure 4). One half was then used for microstructural analysis and another half for performing compression tests. Compression samples from the lower section of RUE processed specimens, where the highest deformation is applied [16], were taken after two, four, and eight passes (Figure 5). Cylinders with the diameter of 4 mm and the height of 10 mm were prepared for the tests. Compression samples from the initial material were also prepared. Room temperature compression tests with the ram speed of 1 mm/s were conducted by a testing machine (Zwick/Roell 250) on the annealed and non-annealed specimens. All tests were conducted up to the strain of 0.8. True stress-strain curves were then drawn for each case. To remove the effect of friction, the modified extrapolation of Cook and Larke was used to correct true stress-strain curves [17].

Figure 4. Half of the RUE samples cut after different passes of RUE.

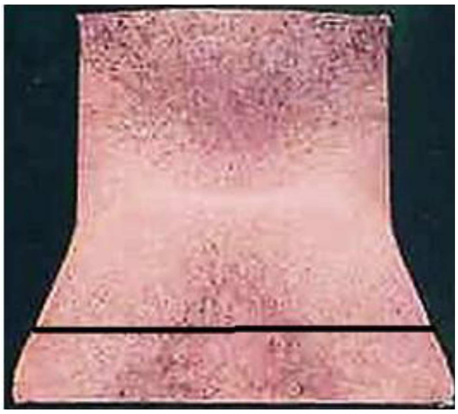


Figure 5. Location where compression samples were taken.

2.5. Microstructural analysis

For microstructural analysis, samples from the lower part of the annealed cases were first polished and mounted by a standard method. An MI solution with the composition of 100-120 ml Ethanol, 20-50 HCl, and 5-10 $FeCl_3$ was used to etch the samples. The samples were then analyzed by an optical microscope at different magnifications and from different locations. In addition, an SEM microscope model VEGA-TESCAN-LMU was used for electron microscopy purposes.

3. Results and discussion

3.1. Compression stress strain curves

As explained above, compression tests were conducted to understand the effect of the number of RUE passes on the flow strength and defining the flow pattern in different sections of the specimen. In that sense, compression stress-strain curves for annealed and non-annealed cases were derived. Figure 6 shows the true

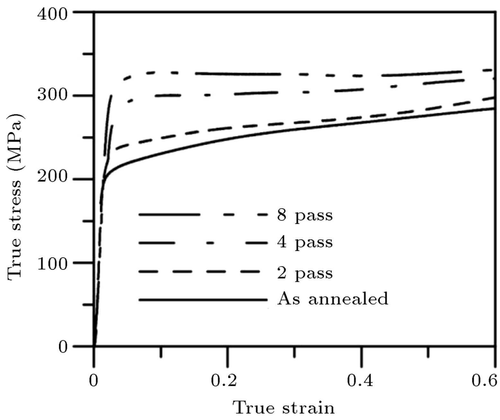


Figure 6. True stress-strain curves of the non-annealed samples.

stress versus strain curves for non-annealed samples. This figure shows the curve for the initial specimen together with the curves for two, four, and eight passes of RUE. The figure shows that the RUE process increases compression yield strength due mainly to grain refinement and dislocation density increase. Such an increase is more pronounced in the initial stages of RUE up to four passes. By applying only two passes of RUE, the 0.05 strain flow strength has increased from 216 MPa to 241 MPa. This situation is more noticeable in four passes of RUE where the flow strength increases to 293 MPa. At eight passes of RUE, the flow strength reaches 322 MPa. Saturation of dislocation in a higher number of RUE passes is the main reason for the decline in a work-hardening rate process that can be observed in eight passes of RUE. This is of crucial importance as in other SPD processes, such as ECAP and ARB, where a high number of cycles are required to achieve the highest yield strength level. For example, the maximum yield strength is achieved after five cycles in ECAP of copper [18].

Generally, two mechanisms increase the strength and hardness of materials in SPD processes. The first mechanism is the grain refinement, based on Hall-Petch relationship. Formation of low angle grain boundaries is often the main mechanism in that sense [19]. The second strengthening mechanism is the increase in dislocation density based on Taylor relationship. With the formation of small-sized grains up to four passes of RUE, the effect of dislocation density on the strengthening reduces with the higher number of passes. The formation of small-sized grains with high angle grain boundaries prevents the movement of dislocations and locks them up. Increasing the passes after this stage results in the reduction of dislocation density. Such a reduction in dislocation density causes a softening, more probably dynamic in nature, in eight passes of RUE. A similar trend of softening in higher passes was reported in aluminum [8] and AZ61 [20]. For example, in the aluminum alloy, the yield strength increased from 60 MPa to 170 MPa after two passes of RUE, which is proportionally similar to the copper alloy of this research. The strength did not increase at higher number of passes in aluminum alloy. In magnesium alloy AZ61, the highest strength was achieved after four passes of RUE and did not change in six and eight passes [20]. Reaching a critical average grain size at low number of passes in all these cases is the main reason for the declined work-hardening rate observed after four passes of RUE. Apparently, the grain size does not change at higher number of RUE passes.

To understand the effect of annealing, stress-strain curves of compression samples annealed at 200°C were drawn, too. Figure 7 shows the results of the compression tests on the annealed specimens. As observed in this figure, the yield strength of the initial specimen

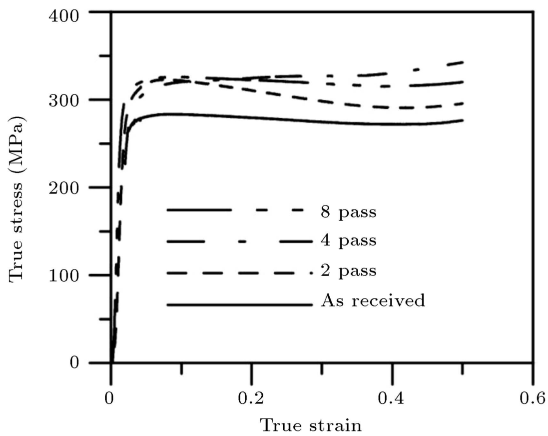


Figure 7. True stress-strain curves for samples annealed at 200°C.

increases after the annealing process. However, such an increase is quite insignificant for the two passes of the RUE sample. The yield strength of annealed samples after four and eight passes remained unchanged. The main reason is probably because annealing in these cases only slightly changes the grain configuration from elongated configuration to the rounded one and does not change the grain size itself.

3.2. Microstructural analyses

3.2.1. RUE samples

Figure 8 shows the microstructure of the initial commercial copper used in this research. As observed in this figure, the initial structure contains large and coarse grains. It is also a very inhomogeneous structure with a large-sized grain distribution.

Figure 9 shows microstructures at different locations of sample after two passes of the RUE process. Since the deformation pattern is different in various areas, a non-uniform structure is observed in this case. It can be seen from the figure that two passes of RUE result in the elongated grains. These elongated grains are observed more in the lower half (Figure 10(e)) of the specimen, indicating that this region has gone through

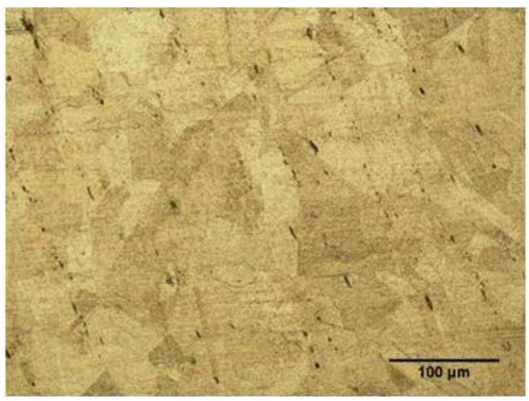


Figure 8. Microstructure of the initial material without annealing.

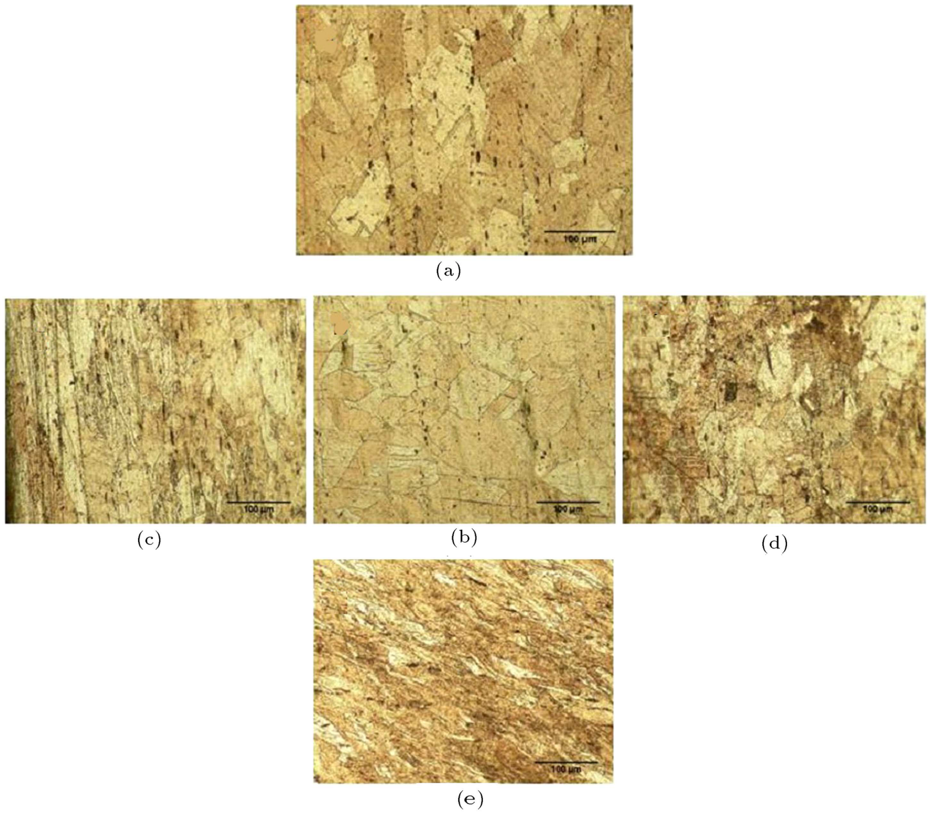


Figure 9. Microstructures of the specimen after two passes of RUE: (a) Top section, (b) middle section, (c) left side, (d) right side, and (e) bottom of the sample.

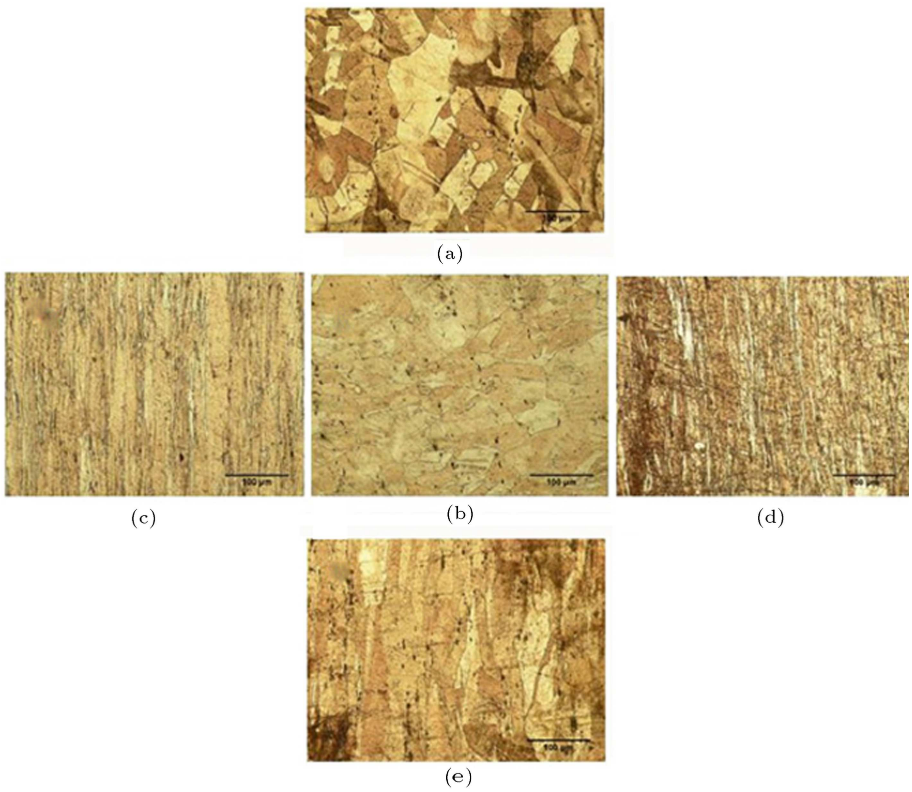


Figure 10. Microstructures of the specimen after four passes of RUE: (a) Top section, (b) middle section, (c) left side, (d) right side, and (e) bottom of the sample.

both upsetting and extrusion processes sequentially. Although some elongated grains, particularly in the lower half of the specimen, are observed in this case, the structure still contains some coarse and large grains. The structure is also partly inhomogeneous.

Figure 10 shows the structure in different sections of the specimen for samples that have gone through four passes of RUE. As can be seen in this figure, the left and right sides of the sample (Figure 10(c) and (d)) show elongated vertical grains. The deformation is also more severe on the sides of the sample. The main reason for this phenomenon is that, in the extrusion sequence, the grains are elongated in the extrusion direction (sample axis). It is also known that, in the upsetting process, the elongation of the grains is perpendicular to the sample axis. Moreover, the figure shows that the grains are more refined compared to the two passes of RUE. In the upper section and middle of the sample, grains are broken and converted to smaller ones. It is important to note that there are still some large and coarse grains in the structure. In the upper section, grains are elongated both in the vertical and horizontal directions, indicating that this area has been influenced by both upsetting and extrusion processes, although the grains are larger in this section which shows that less deformation was applied to the upper section of the specimen.

Figure 11 shows the grain structure after eight passes of RUE. As observed in this figure, grains are more elongated in this case. In addition, in the side sections of the sample, grains are highly elongated and vertically positioned, indicating that they have gone through the extrusion process. Compared with the four passes case, grains in the middle of the sample are refined and elongated at the same time. The grains in the middle of the sample are elongated horizontally which indicates that this section has been affected by the upsetting process. Moreover, in the areas where grains were coarse after four passes, elongated and refined grains can now be observed after eight passes of RUE. Additionally, the grains are elongated in the lower section, indicating that extrusion was at play in this area. In this section, small grains are embedded inside the large ones. On the other hand, the upper section of the sample still shows some equiaxed grains, indicating that, even after eight passes, straining in this section is not significant.

SEM analysis of the initial sample was performed after two, four, and eight passes of RUE. Figure 12 shows SEM micrographs for these cases. The images show that, in line with the analyses elaborated in earlier sections of this article, grains get smaller and less elongated with the increasing number of passes. In fact, after eight passes of RUE, equiaxed grains were developed. A cluster of very small grains is also observable in this case. The grain size after four passes was in the

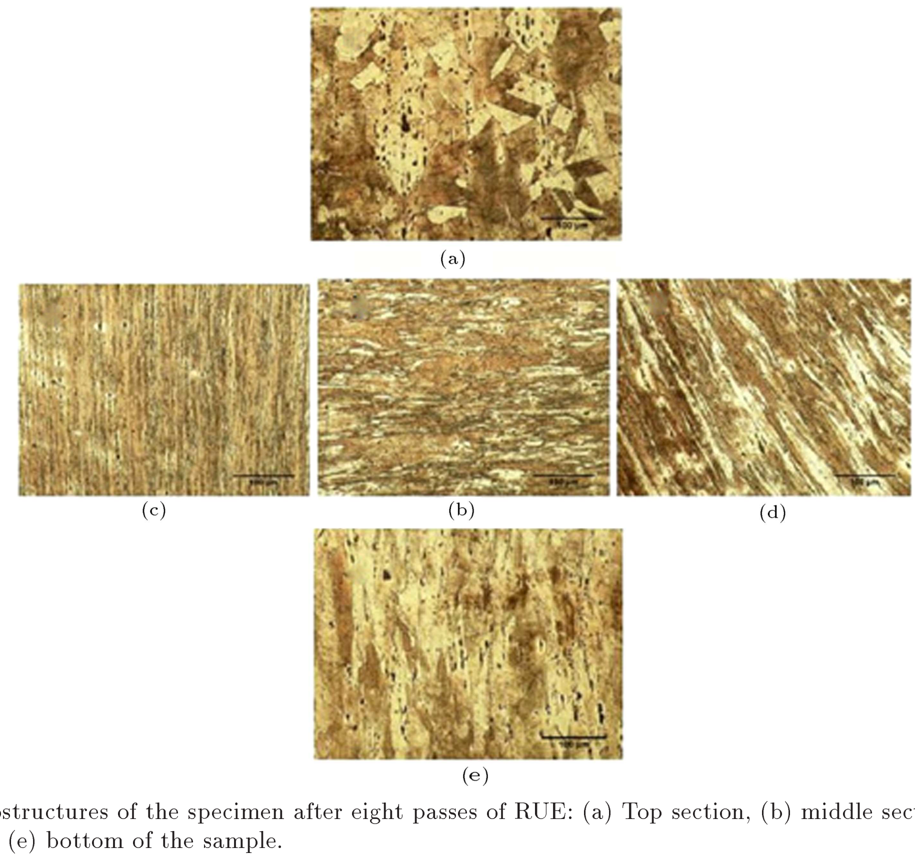
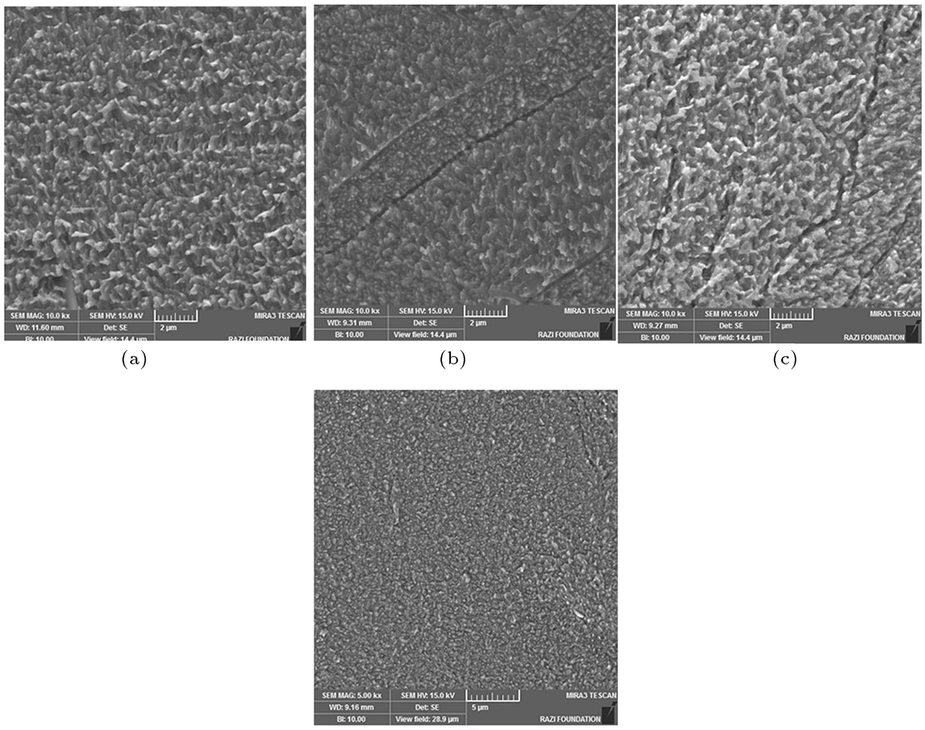


Figure 11. Microstructures of the specimen after eight passes of RUE: (a) Top section, (b) middle section, (c) left side, (d) right side, and (e) bottom of the sample.



(a) (b) (c) (d)

Figure 12. SEM micrographs in different stages: (a) Initial specimen, (b) after two passes of RUE, (c) after four passes of RUE, and (d) after eight passes of RUE.

range of 190-215 nm and after eight passes in the range of 190-210 nm. The results show that grain size was in the range of 200 nm after these many passes. The grain sizes of OFHC cooper reduced to 0.05 through 2 μm after 10 passes of RUE [11], and an equiaxed ultrafine grain size of about 200 nm was introduced into an aluminum alloy LY12 by 10 passes of RUE [21].

By comparing these micrographs with eight cycles of ECAP process performed on Cu sample [22], it is clear that, after annealing of ECAPed sample, no changes in the size of the grains or their directions can be observed. Habibi and Ketabchi reported that, annealing of ECAPed Cu sample, only changes the grains from elongated configuration to an equiaxed configuration, and no changes of the grain size can be achieved by such an annealing process [22]. Apparently, grain refinement and the elongation of the grains are more evident in RUE sample than in the ECAP one.

After two passes of RUE, small grains start to form inhomogenously in different sections of the sample. As the number of passes increases, more of the large grains break and elongated grains break, too. This is particularly the case for a very high number of RUE passes, i.e., eight passes. Such small grains are also an indication of dynamic recrystallization during the high number of passes of RUE [11]. Such dynamically recrystallized grains were reported during RUE processing of other materials in the past. Specifically, they were observed after three passes RUE of alloy AZ61 [20]. Increasing the number of passes of RUE in that case did not significantly change the grain size. These dynamically recrystallized grains were observed in RUE of AZ80 [23]. It was reported that the rate of dynamic recrystallization is high during the initial passes of RUE. The rate of dynamic recrystallization reduces as the number of passes increases.

The main mechanism of grain refinement is the formation of Low Angle Grain Boundaries (LAGB) during initial stages of straining. As the straining increases, these LAGBs transform to High Angle Grain Boundaries (HAGB), and new grains are revealed in such a situation. During the RUE process, small grains are formed in limited areas and, as the amount of deformation increases, the volume fractions of these grains increase as such. The main mechanism in this case is a shear band formation [24], formed in the RUE processed samples, as shown in Figure 12. In addition, the formation of shear bands, which are perpendicular to the deformation direction, is the main characteristics of RUE process [11]. The formation of these perpendicular shear bands is due to the inverse flow directions of materials during upsetting and extrusion sequences of RUE. The patterns in each case of upsetting and extrusion are different. The strain in each zone is proportional to the inversion of shear band positions. Therefore, the rate of grain formation in each zone is dependent on the amount of strain and varies from one zone to another. Analyzing the grains of different passes of RUE reveals that only large grains can be transformed. In fact, after the formation of small grains due to dynamic recrystallization, more deformation only changes large grains. The small grains in such a situation do not change by increasing the amount of strain. Only when all grains are in the same size, small grains start to deform, mostly through slip or rotation mechanisms. The dislocations formed during the early stages of RUE passes can be canceled out at higher amount of straining. Refining the grains makes a uniform strength in different sections of the specimen after a high number of passes of RUE. Therefore, once the grain size distribution reaches a certain level, increasing the straining does not increase the strength of the specimen.

3.2.2. ETMB model

In a metal forming process, when the strain path is reversed, dislocations with the opposite effect are generated. This is specially the case for the RUE process as the successive upsetting and extrusion where the material flows in an orthogonal direction during upsetting and extrusion. In this situation, there is a great chance that dislocations may cancel out at higher amount of strain and during high number of passes, which leads to the reduction of dislocation density and the hardness of the specimen at high number of passes, i.e., eight passes of RUE. This refinement of the structure during severe plastic deformation has been explained by ETMB model. The main components of the ETMB model were the evolution equations for the dislocation densities in the cell walls and cell interior, determining the deformation process. This model considers the refined grains as a form of inside cells and the boundaries of the cells. Enikeev modified the ETMB model by assuming that dislocation generation starts in the cell boundaries, and their annihilation occurs in both inside the cells and the boundaries [25]. The modified ETMB, unlike the ETMB model that predicts the saturation of the dislocations, predicts that the dislocation density initially increases, reaches a maximum, and, finally, reduces rapidly to a constant level [11].

According to the FEM results, the maximum effective strains were 1.78, 3.16, 4.73, and 6.28 for the two-, four-, and eight-pass RUE processed samples, respectively [7]. Variation of the flow stress at 0.05 and 0.3 strains with the RUE imposed strain is shown in Figure 13. It can be observed that, in contrast to the result of Balasundar et al. [11], the variation of the flow strength with strain in the RUE specimens is matched with the ETMB model.

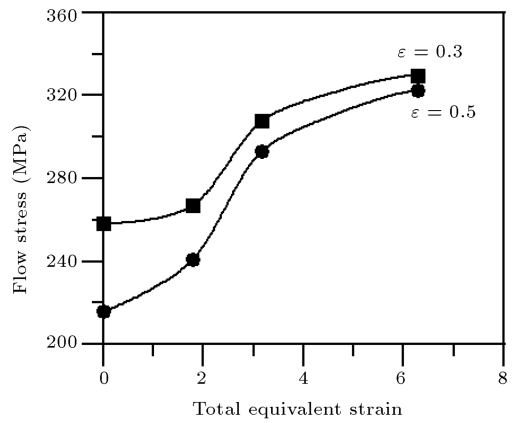


Figure 13. Variation of the flow stress at 0.05 and 0.3 strains with the RUE imposed strain.

3.2.3. Microstructure of the annealed specimens at 200° C

To see the effect of annealing on the microstructure, optical micrographs of the samples were observed after annealing at 200°C. Figure 14 shows the microstructure of different locations of the RUE samples after annealing at 200°C. As seen in this figure, the grains are less elongated after the annealing process. Figure 14(d) shows that, after eight passes of RUE and annealing, small grains are formed inside the coarse large grains. In fact, small grains are formed due to recrystallization process after eight passes of RUE, and a more homogenous structure is obtained.

4. Conclusions

In this investigation, the effects of different amounts of deformation, in the form of different RUE passes, on mechanical properties and microstructural evolution were analyzed in a commercial pure copper. Two, four, and eight passes of RUE were applied, and the mechanical properties were measured by compression testing. The structure before and after annealing was analyzed, too. The following conclusions can be drawn by conducting this research:

- Flow strength increases by applying subsequent processes of upsetting and extrusion in the RUE process. Substantially, high strength was achieved only after four passes of RUE. Due to dislocation density saturation, the work hardening rate after four passes was reduced;
- A nano-sized grain structure with the size around 200 nm was achieved after eight passes of RUE. A refined and recrystallized grain structure was obtained after annealing at 200°C, too;
- Dynamic recrystallization is quite plausible at room temperature through RUE processes as indicated by the microstructural studies and the flow strength behavior;
- Similar to other severe plastic deformation, the flow strength increases rapidly in the initial stages of deformation, and the increase rate slows down at

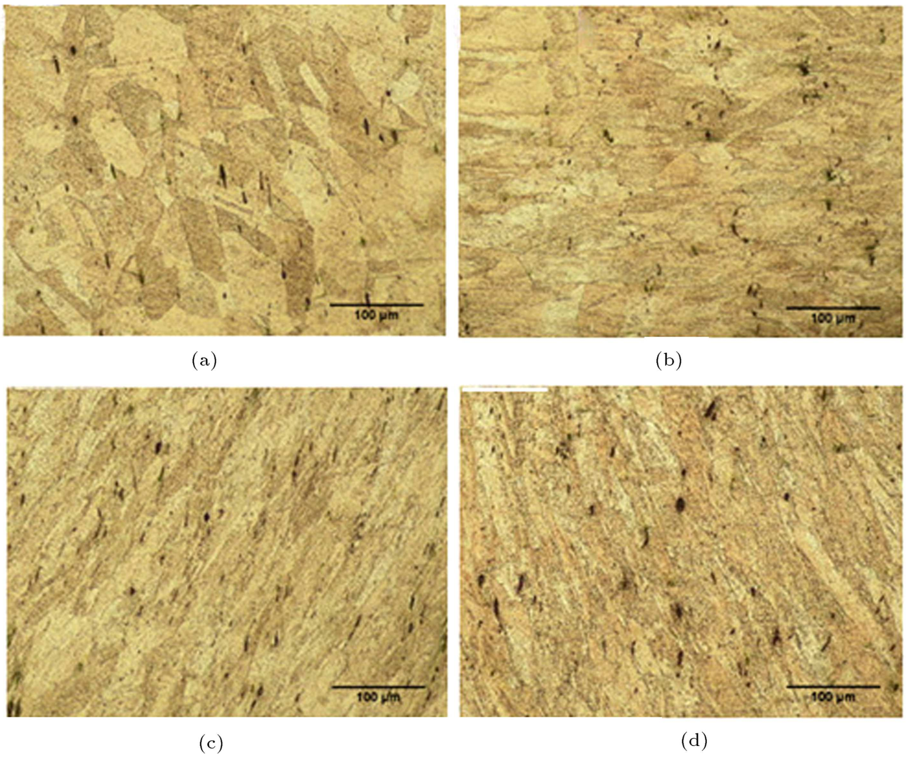


Figure 14. Microstructures of the samples after annealing at 200°C for 20 min: (a) Initial specimen, (b) after two passes of RUE, (c) after four passes of RUE, and (d) after eight passes of RUE.

higher amount of deformation applied in the form of a high number of passes;

- It has been found that the change of flow stress with the effective strain imposed on the RUE specimen is matched with the ETMB model.

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