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The effects of intermediate and post-annealing phenomena on the mechanical properties and microstructure of constrained groove pressed copper sheet

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ABSTRACT

Commercial purity copper sheets were subjected to a severe plastic deformation technique known as constrained groove pressing (CGP). The effect of pass number, intermediate and post-annealing on the yield strength, hardness and final microstructure of the copper specimens were investigated. The initial pass increases the strength much more than the subsequent passes. Intermediate and post-annealing up to 300 °C cannot change the mechanical properties significantly and even in some cases improve the strength and hardness while reduce the hardness inhomogeneity. Microstructure after post-annealing at elevated temperatures shows abnormal grain growth.

1. Introduction

Fabrication of ultra fine grained (UFG) materials is of great importance due to their superior mechanical properties, and during the last decade several methods have been introduced for this purpose [1,2]. UFG materials can be synthesized by either bottom-up or top-down approaches. In the bottom-up approach, the nanostructure is produced through atom-by-atom and layer-by-layer arrangement. This approach often leads to porous structure and is not applicable to industrial manufacturing [3]. In the top-down approach, the bulk material's microstructure changes to nanostructure by means of severe plastic deformation (SPD) [4–6]. The principle of SPD includes increase of dislocation density by heavily uniform deformation of materials, formation of dense dislocation walls, and transformation of dislocation walls into high-angle grain boundaries. Materials produced by SPD are of great importance because they have (1) non-porous structure, (2) great mechanical properties such as high strength and toughness and (3) proper dimension for mechanical and physical testing [7]. Different techniques have been used to impose large plastic strain on bulk metals, such as equal channel angular pressing (ECAP) [8], high pressure torsion (HPT) [9], accumulative roll bonding (ARB) [10,11], repetitive corrugation and straightening (RCS) [12], constrained groove rolling (CGR) [13], constrained groove pressing (CGP) [14], etc.

Of all the above techniques, only the last four techniques are applicable to sheet metals. But as ARB involves repetitive bonding between two rolled plates, if perfect bonding is not accomplished, the bonding interface may reduce the mechanical properties of the product. Thus, ARB is less considered feasible for severe plastic deformation of sheet metals. Also in CGR, probable bending through deformation leads to tensile stress and so reliable results may not be expected. Constrained groove pressing, originally proposed by Shin et al. [14], has the advantage of imposing more uniform severe plastic deformation on sheet metal. The principle of groove pressing, which can be easily scaled up, is that a material is subjected to repetitive plastic shear deformation with grooved and flat dies, by pressing alternatively. The groove pressing is performed such that the gap between the upper die and the lower die is the same as the sample thickness, resulting in pure shear deformation under plane strain condition at inclined regions of the sample, see Fig. 1. Recently, many researches have been carried out on mechanical properties and microstructure of sheet metals, processed by CGP indicating that this process can have great influence on mechanical properties of sheet metals such as increase of yield strength and mean hardness [15]. This process also has the ability of producing submicrometer polygonized grain structure with well-defined grain boundary as in the work of Shin et al. [14]. Still, the effect of annealing on the CGPed specimens has not been studied. In the current research, the effects of intermediate and post-annealing phenomena on the mechanical properties, hardness inhomogeneities and microstructures of commercial purity copper sheets under CGP process are investigated in order to study the thermal stability of microstructure and mechanical properties of

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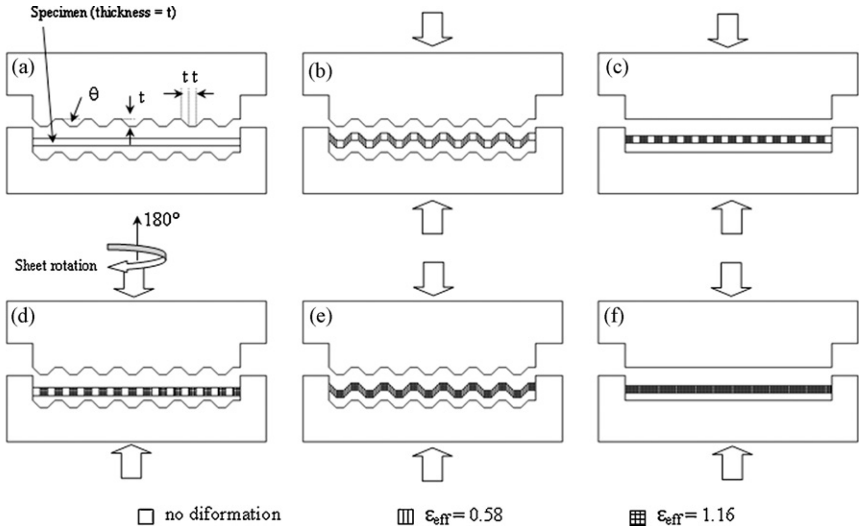

Fig. 1. Schematic of constrained groove pressing (CGP) stages.

CGPed sheets, and find the critical annealing temperature leading to sudden drop in mechanical properties due to abnormal grain growth.

2. Experimental procedure

In the present study, commercial purity (99.3%) copper sheets with dimension of $90 \times 60 \times 3\,\mathrm{mm}^3$ were used as work pieces. Before subjecting specimens to any deformation, the copper sheets were annealed at 750 °C for 3 h. The schematic design of the die is shown in Fig. 1(a).

Each time the work pieces are pressed between the grooved dies, under plain strain condition an effective strain of 0.58 is imposed on the inclined regions while the flat areas undergo no deformation (Fig. 1(b)). Then the work piece is removed from the dies and is flattened by means of a set of flat dies, resulting in another 0.58 effective strain in reverse direction which ends up in 1.16 strain for the deformed regions (Fig. 1(c)). After the straightening, the specimen is rotated 180° around the axis perpendicular to the plane of the sheet (Fig. 1(d)) which ensures that the subsequent groove pressing (Fig. 1(e)) and straightening (Fig. 1(f)) impose an overall strain of 1.16 on the undeformed regions. After this step, a uniform strain of 1.16 has been imparted throughout the specimen. In the present study, one pass is equivalent to two-groove pressing and two straightening stages which leads to a uniform strain on the sheet. All the passes were carried out using a 50 kN pressing machine with a ram speed of $0.15\,\mathrm{mm\,s^{-1}}$. The loads applied for pressing were in the range 30–45 kN. Fig. 2 shows the grooved dies used in laboratory and the experimentally processed sheet.

Annealing cycles were conducted in 200, 300 and 400 °C for 20 min. In this study, some specimens were annealed between passes (intermediate annealing) and some others were annealed after the final pass (post-annealing). In order to have better understanding of pressing and annealing combinations, a sample coding was used. In this coding, "P" represents pass and "A_2, A_3, A_4" stand for annealing the specimen for 20 min in 200, 300 and 400 °C, respectively. For example, sample PA_2P represents a specimen which has been pressed for one pass, intermediately annealed at 200 °C for 20 min, and then pressed for another pass. The annealed specimens were categorized into five groups:

PA_T: specimens with post-annealing after one pass.
PPA_T: specimens with post-annealing after two passes.
PA_TP: two-pass specimens with intermediate annealing.
PA_TPP: three-pass specimens with intermediate annealing after the first pass.
PPA_TP: three-pass specimens with intermediate annealing after the second pass.

After preparing the specimens, tensile test was carried out on specimens with gage length of 32 mm (ASTM E8M) using a 50 kN tensile testing machine at a cross head speed of 0.12 mm/s. Typical example of stress–strain curves are present in Fig. 3 which will be discussed later (Section 3.1.1). Vickers hardness was measured

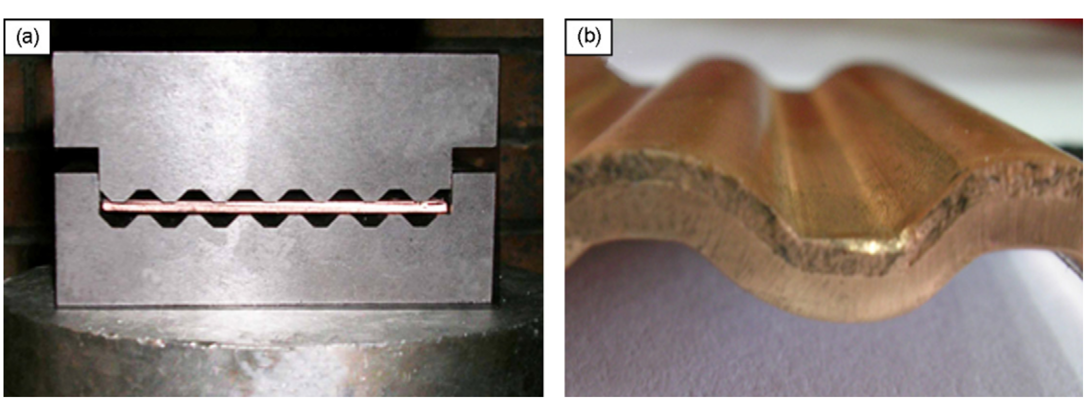

Fig. 2. (a) CGP die and the position of copper sheet; (b) copper sheet after the first stage of CGP ($\varepsilon_{\mathrm{eff}} = 0.58$).

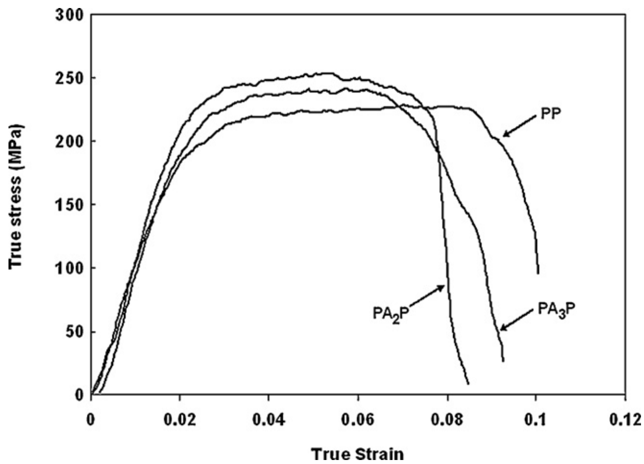

Fig. 3. True stress–strain curves of specimens PP, PA_2P, PA_3P.

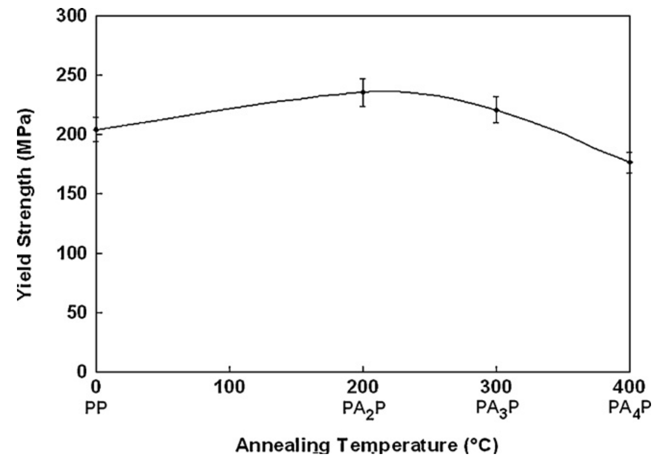

Fig. 5. Effect of intermediate annealing on tensile properties of PA_TP copper sheets.

along the longitudinal direction for minimum three measurements for each point in order to study its inhomogeneity throughout the specimens. Besides, the microstructure of post-annealed specimens was observed by an optical microscopy to investigate the effect of annealing on grain size distribution of CGPed sheets.

3. Results and discussion

3.1. Yield stress

Fig. 4 shows the yield stress of groove pressed copper sheets versus pass number at room temperature. The yield strength (YS) of initially annealed specimen is 72 MPa which after the first pass it is increased to 215 MPa. The subsequent passes result in lower tensile strengths indicating some degree of flow softening and disruption of dislocation configurations. As well Krishnaiah et al. described in his work that the loss of strength in subsequent passes may be due to flow softening associated with disruption of the initially developed dislocation configurations [16].

It should be noted that the strength dropping in ECAP and CGP is different. Two reasons can be presented: First, deformation paths in these processes are different. Second, for the sheet metals, free surface to volume ratio is larger than that of bulk materials and as claimed, free surface may act as dislocation annihilator and it is reasonable to observe higher dislocation annihilation in sheets [18].

3.1.1. The effect of intermediate annealing on yield strength

Fig. 5 shows the effect of intermediate annealing on yield stress of PA_TP specimen. Due to large amount of strain at the first pass, inter-granular dislocation cell structure is established. By annealing the specimen and supplying the required energy for restoration phenomena, the transformation of cell structure walls into grain boundaries and annihilation of inter-granular dislocations will occur simultaneously which results in much finer grain size. These two phenomena (increase of grain boundary density and decrease of dislocation density which happen during annealing) act against each other, causing the increase of the yield strength with lower temperatures of intermediate annealing (PA_2P) and the decrease of the yield strength with higher temperatures of annealing (PA_3P and PA_4P). It seems that the role of increase in grain boundary density is predominant at lower temperatures of annealing (up to 200 °C) while decrease of dislocation density and grain growth phenomenon are the main cause of lower tensile properties at elevated temperatures of annealing.

Fig. 6 shows the effect of intermediate annealing on the tensile properties of PA_TPP specimens. In this category, PA_2PP exhibited highest strength in comparison with other specimens due to finer grain size. It seems that new grain size distribution after annealing at 200 °C entangles the dislocations' movement severely during the subsequent passes leading to higher strength. At elevated annealing temperatures (such as 300 and 400 °C), as abnormal grain growth occurs during annealing, lower strength is obtained in subsequent passes (in comparison with PA_2PP) but still is overcomes specimen PPP. Fig. 7 demonstrates the effect of intermediate annealing on

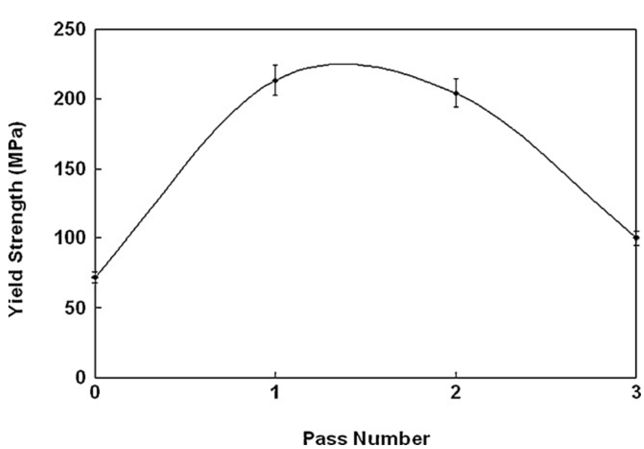

Fig. 4. Yield strength of copper sheet versus the number of passes.

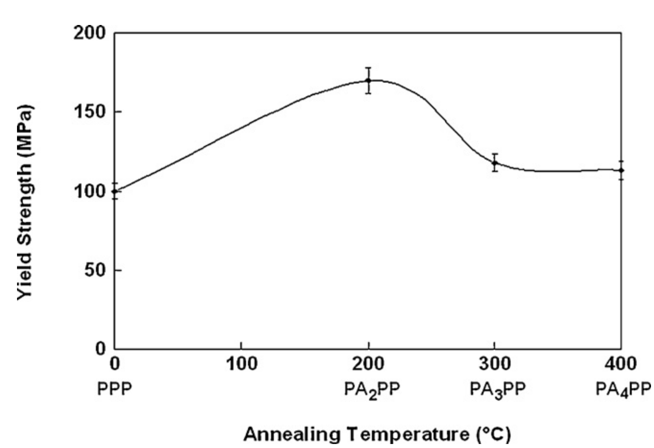

Fig. 6. The effect of intermediate annealing on yield strength of PA_TPP copper sheets.

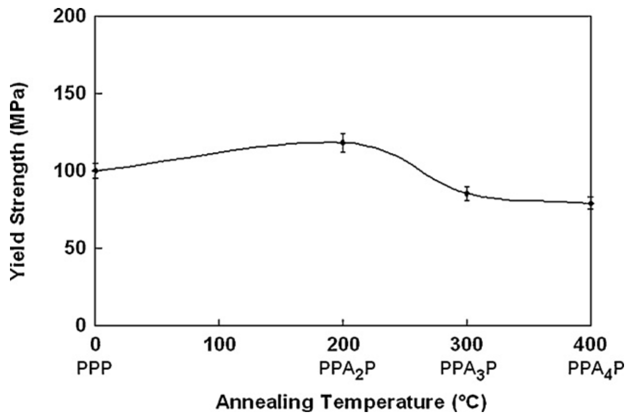

Fig. 7. The effect of intermediate annealing on yield strength of PPA_TP copper sheets.

the tensile properties of PPA_TP specimens. In this category, specimen PPA_2P also shows higher tensile properties among the other specimens. The trend of changes in strength of PPA_TP specimens is very similar to that of PA_TPP specimens. At lower temperatures of annealing, uniform and fine grain size distribution entangle the dislocations' movement resulting in higher strength. Due to non-uniform grain size distribution after annealing at higher temperatures, loss of strength occurs. Fig. 8 compares the effect of intermediate annealing on tensile properties of PA_TPP with that of PPA_TP. As it is shown, the yield strengths of all the specimens of PA_TPP are higher than that of PPA_TP. The reason is that since PPA_TP specimens experience larger initial strain before annealing, higher amounts of restoration will occur in these specimens, resulting in much lower strength than their counterparts in PA_TPP category.

3.1.2. The effect of post-annealing on yield strength

Fig. 9 shows the effect of post-annealing on yield strength of PPA_T specimens. The yield strength is increased with increasing annealing temperature up to 300 °C due to grain refinement resulted from recrystallization phenomenon. At this stage, increase of grain boundary density compensates the decrease of dislocation density and outweighs it to some extent. Thereafter, because of high temperature of annealing and large amount of initial strain ($\varepsilon = 2.32$), recrystallization and grain growth occur which lead to loss of some strength.

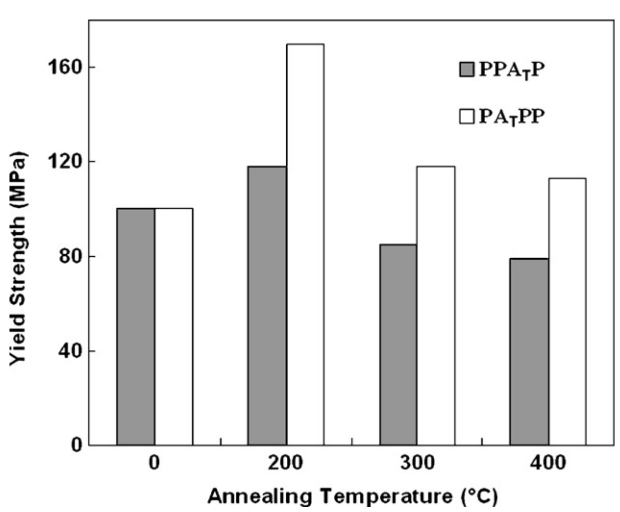

Fig. 8. The effect of intermediate annealing on yield strength of PA_TPP and PPA_TP categories.

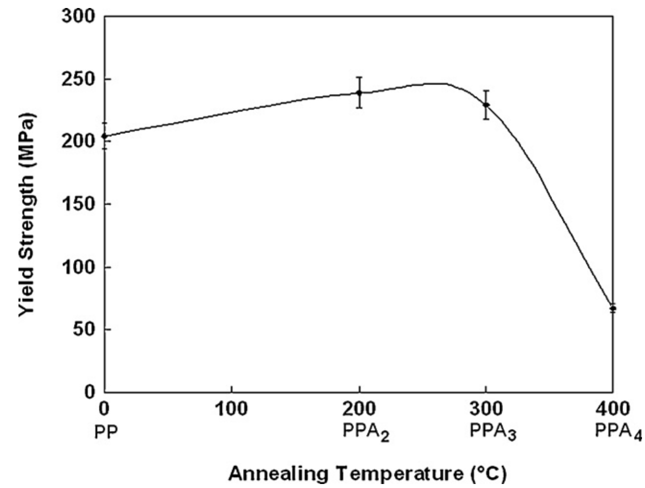

Fig. 9. The effect of post-annealing on tensile properties of PPA_T copper sheets.

3.2. Hardness

Fig. 10 illustrates the hardness distribution along the longitudinal direction of P, PP and PPP specimens. The maximum hardness can be obtained by imposing 2.32 strain; less or more deformations will cause the hardness value to descend which is related to the work hardening and flow softening due to larger strains.

3.2.1. The effect of intermediate annealing on hardness

Fig. 11 shows the effect of intermediate annealing on hardness properties of PA_TP specimens indicating that higher intermediate-annealing temperatures bring the loss of mechanical properties in PA_TP specimens.

Fig. 12 shows the effect of intermediate annealing on the hardness properties of PA_TPP specimens. Large amount of strain, imposed during the 2nd and 3rd pass, eliminates the effect of prior annealing and consequently the hardness values do not vary greatly in respect of changing the intermediate-annealing temperature in PA_TPP category.

Fig. 13 shows the effect of intermediate annealing on the hardness properties of PPA_TP category. In this category the first two passes of pressing cause specimens' dislocation density to increase greatly. Regarding the fact that higher dislocation density causes the higher restoration kinetics, the PPA_TP category is more liable to restoration than that of PA_TPP category (Fig. 13(a)). Also, as can be observed in Fig. 13(b), mean hardness value is decreased with increasing annealing temperature.

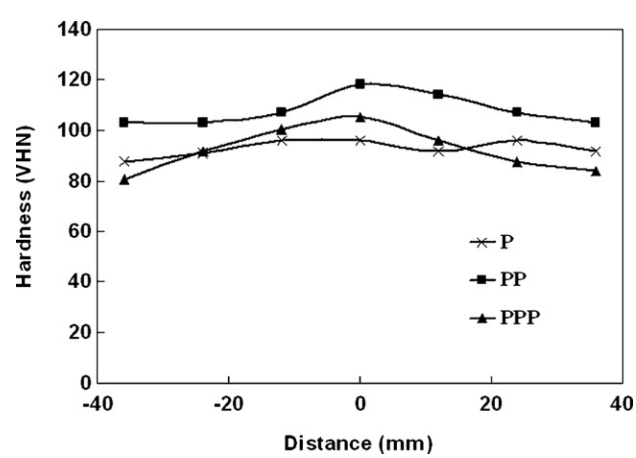

Fig. 10. Hardness distribution of CGPed specimens.

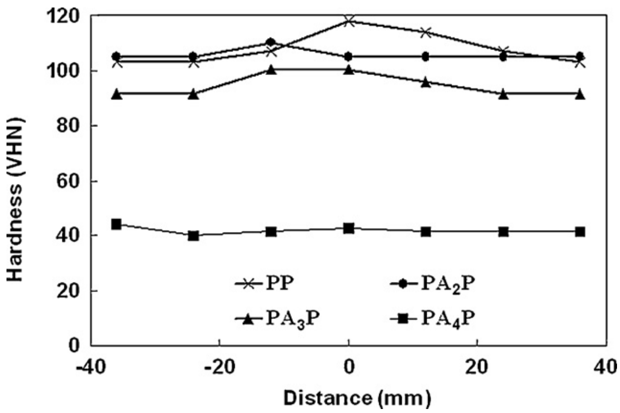

Fig. 11. Hardness distribution of PA_TP category.

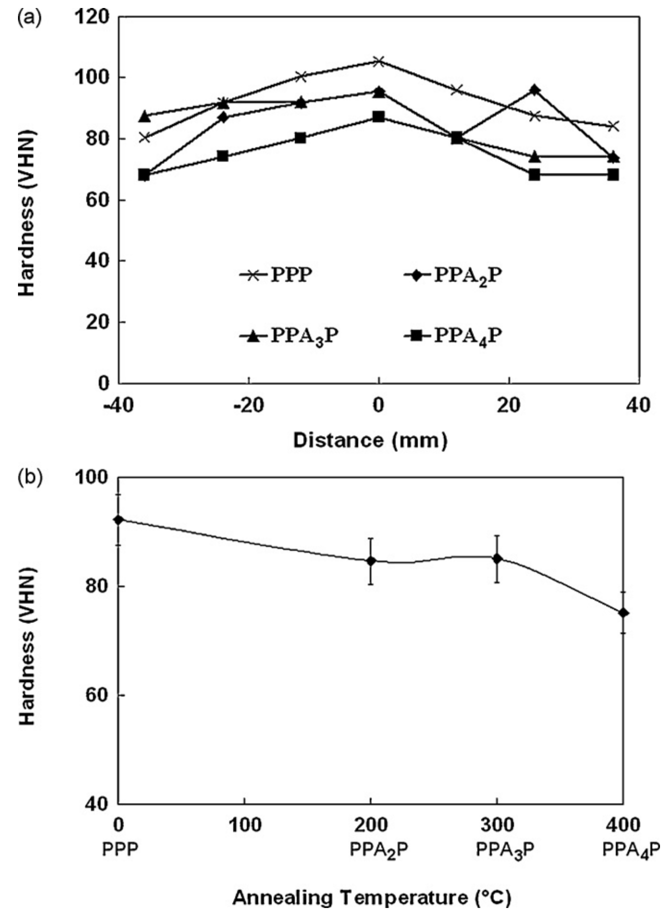

Fig. 13. (a) Hardness distribution of PPA_TP category; (b) mean hardness value of PPA_TP category.

3.2.2. The effect of post-annealing on hardness

Fig. 14 illustrates the hardness distribution along the longitudinal direction of PA_T category. Specimens with higher post-annealing temperatures show higher hardness values. In other words, the PA_3 is harder than PA_2 and PA_2 surpasses the P with a slightly higher hardness value. Finally, PA_4 shows a sharp decrease in hardness value and tensile strength due to rapid grain growth. Consequently, the temperatures above 300 °C are not appropriate for post-annealing the CGPed pure copper sheets.

Fig. 15 shows the hardness distribution along the longitudinal direction of PPA_T category. Specimens, which have been CGPed for two successive passes, have faced a great amount of strain, equal to 2.32. Thus, dislocation density in these samples is much higher than that of single-pass-CGPed specimens. This higher dislocation density causes the recovery and recrystallization to occur at lower temperatures somewhat about 200 °C. Consequently, it can be observed that higher post-annealing temperatures lead to slightly lower mean hardness values, comparing P & PA_2 & PA_3.

Again, PA_4 exhibits a sharp decrease of mechanical properties implying that the critical annealing temperature for these specimens is somewhat about 300 °C.

3.3. Inhomogeneity factor

In this study, inhomogeneity factor (I.F.) is used to evaluate the homogeneity of hardness through out the specimens. The inhomogeneity factor can be calculated by:

$$\text{I.F.} = \frac{\sqrt{\sum_{i=1}^{i=n}(H_i - \bar{H})^2/n-1}}{\bar{H}} \tag{1}$$

where n is the number of hardness measurements on each specimen, H_i is the hardness value of ith measurement, and $\bar{H}$ is the mean hardness value.

In general, less I.F. value indicates higher homogeneity of mechanical properties.

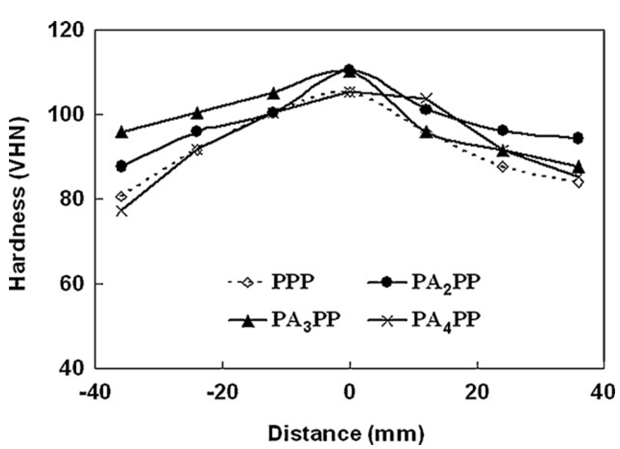

Fig. 12. Hardness distribution of PA_TPP category.

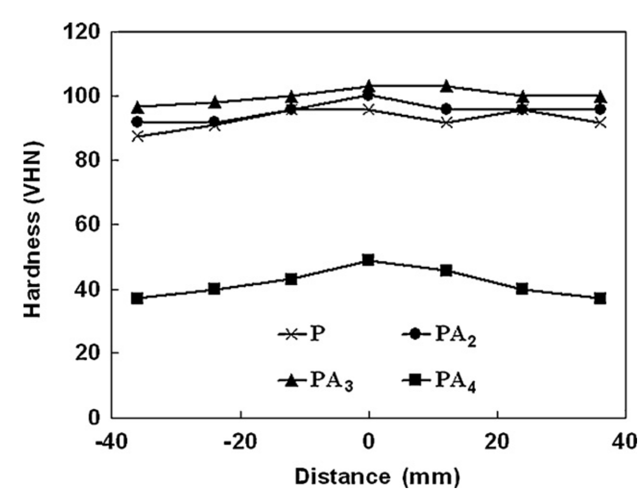

Fig. 14. Hardness distribution of PA_T category.

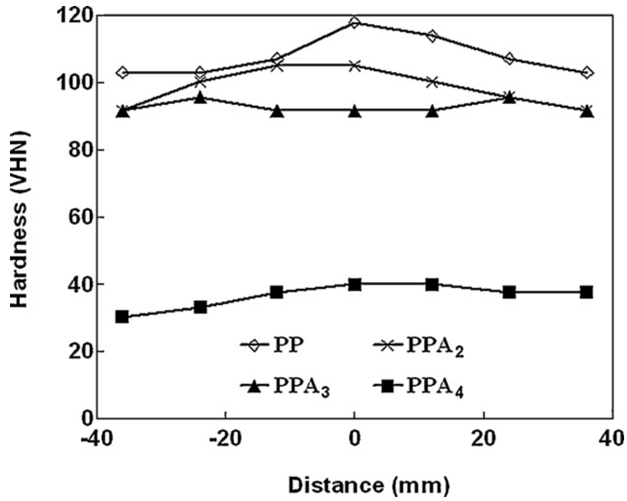

Fig. 15. Hardness distribution of PPA_T category.

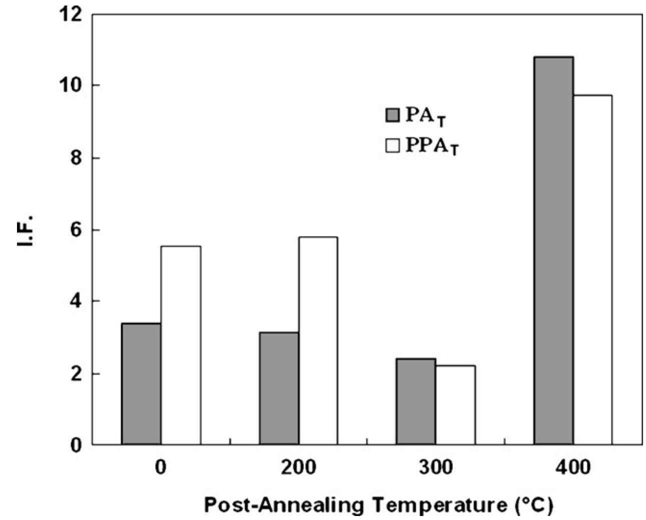

Fig. 16. The effect of post-annealing temperatures on I.F. for PA_T and PPA_T categories.

In conventional metal forming processes, as the annealing temperature is increased, the inhomogeneity factor declines [17]. In contrast to the expectation, the results of this study show that there is an optimum post-annealing temperature for acquiring the least I.F.

As it is shown in Fig. 16, the least I.F. can be obtained by post-annealing the P or PP specimens at 300 °C. The increase in I.F. after post-annealing at 400 °C can be interpreted by abnormal grain growth, resulted from high degree of deformation and high temperature. This is described in the next section.

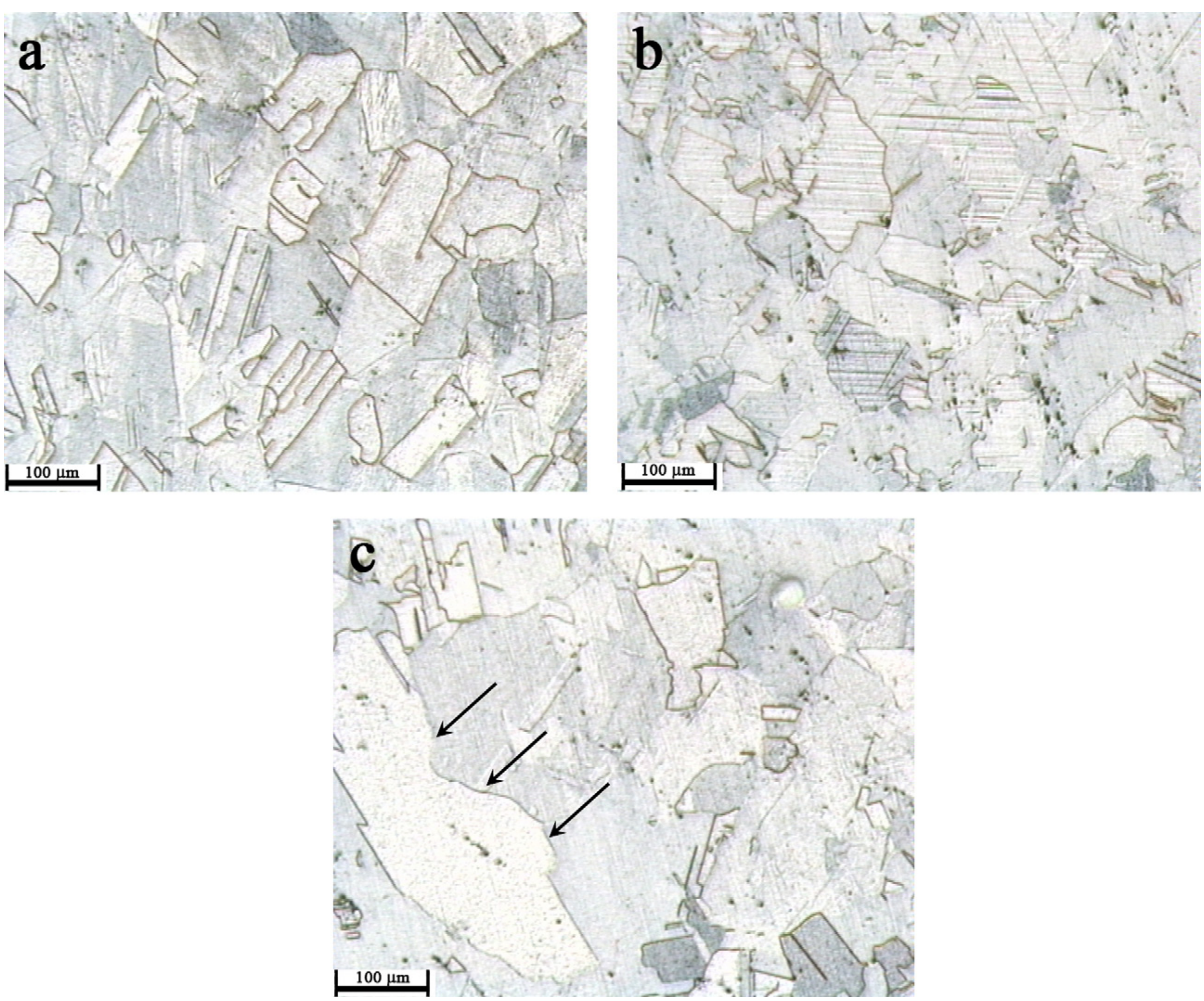

Fig. 17. Microstructure of specimens (a) PA_2; (b) PA_3; (c) PA_4.

3.4. Microstructural inspection

As previously shown, post-annealing the PA_T and PPA_T categories at 400 °C resulted in much higher inhomogeneity factors.

The microstructure of copper sheets after CGP process was studied in the work of Krishnaiah et al. [16] and it was revealed that submicrometer grain structure can be obtained. Here, the microstructure after post-annealing for a typical category of specimens (PA_T) is discussed and shown in Fig. 17. The microstructure of PA_2 (Fig. 17a) exhibits new grains, resulted from recrystallization. Fig. 17b shows the microstructure of PA_3 in which the grains with insignificant grain growth can be observed, yet no abnormal grain growth can be observed. By increasing the post-annealing temperature up to 400 °C (specimen PA_4), as it is indicated by arrows in Fig. 17c, abnormal grain growth can be observed. Regarding the presence of strain inhomogeneity in CGPed samples, driving force for restoration is not uniform, which leads to dissimilar grain size. Consequently, in the grain growth process (which occurs at elevated temperatures of post-annealing), the kinetic of growth for grains with dissimilar sizes is different resulting in abnormal grain growth.

4. Conclusions

Severe plastic deformation by constrained groove pressing technique and the effects of intermediate and post-annealing in this process have been studied. The critical findings of this study can be summarized as follows:

(1) CGP is an effective process for imposing large amounts of strain on sheet metals especially in one-pass specimens, two-pass specimens, and finally the specimens which have undergone an intermediate annealing.
(2) Intermediate annealing on PA_2P exhibited higher strength in comparison with PP.
(3) The mean hardness value of PA_2P is 105.84 VHN which is just 1.8% lower than the PP which possesses the highest hardness mean value.
(4) The I.F. of PA_2P is the lowest among all specimens. In other words, this specimen possesses the most homogenous microstructure.
(5) The microstructure produced by CGP showed relatively acceptable thermal stability up to 300 °C of annealing temperature.
(6) In three-pass specimens, the effect of intermediate annealing on increasing the strength after first pass is more than annealing after the second pass.
(7) At lower temperatures of annealing, increase of grain boundary density due to recrystallization, overcomes the dislocation density reduction and so the mechanical properties do not drop off sharply unless the temperatures above 300 °C are used for annealing the specimens.

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