Grain refinement and mechanical properties of metals processed by constrained groove pressing

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Abstract. Constrained groove pressing (CGP), as a new severe plastic deformation technique, can be used for fabricating high performance sheet materials. Because of its potential for large-scale industrial production, CGP is prospective for application in aviation, aerospace, transportation and many other fields. In this work, Cu-Zr and titanium (CP-Ti) sheets were processed by CGP. Microstructures of these two kinds of sheets before and after CGP were analyzed and the grain sizes were moderately refined after two-pass CGP processing. Mechanical testing results show that Vickers hardness, yield stress and ultimate strength of Cu-Zr alloy after two-pass CGP increased by 70%, 273% and 64% respectively, while those of CP-Ti increased by 26%, 66% and 48% respectively comparing with the unprocessed material. These two kinds of materials exhibit pronounced positive strain rate sensitivity (SRS) and the SRS of Cu-Zr alloy increases with increasing pass number of CGP, while the SRS of CP-Ti is not so sensitive. The strengthening mechanism analysis indicates that the stress elevation after CGP is mainly due to dislocation accumulation and interaction, while grain boundary strengthening is not significant. The grain refinement efficiency of CGP is comparatively studied with respect to other SPD methods.

1. Introduction

Constrained groove pressing (CGP) is a newly developed severe plastic deformation (SPD) method with a simple process, which can be used for fabricating fine grain (FG)/ultrafine grain (UFG) sheet materials and improve the mechanical performance of the materials with its unique advantages [1-6]. Because of its potential for large-scale industrial production, CGP is prospective for application in aviation, aerospace, transportation and many other engineering fields. To the best of the author’s knowledge, Shin et al [1] proposed this method firstly in 2002, and successfully refined the grain size of annealed pure aluminum from 1.2 μm to 0.5 μm. In recent years, materials with good deformation ability such as aluminum [1,2], copper [3, 5] and nickel [4] have been extensively studied by CGP method. Moreover, the effects of CGP process on mechanical properties and biological properties of pure titanium were investigated by Thirugnanam et al [6].

A schematic illustration of a CGP process is presented in Figure 1. CGP mould is composed of a set of flat dies and asymmetric groove dies. During the first route of pressing, sheet specimen is extruded by a pair of asymmetric groove dies with a groove angle θ and width T. The inclined region
of the material is subjected to pure shear deformation under plane strain condition, while no deformation is introduced in the flat region. Following pressing, also referred as the first flat pressing, is performed with a pair of flat dies. The previously deformed region experience reverse shear deformation, while the previous undeformed region remains undeformed. Ideally, the plate would be flat again after the first pressing. After that, the specimen is rotated by 180° around the Y-axis, this ensures the undeformed region to be deformed by the second groove pressing and the second flat pressing due to the asymmetry of the groove dies. Generally, a total of two groove pressings and two flat pressings is defined as one CGP pass, would result in large homogeneous effective strain throughout the specimen. When the groove angle \( \theta \) is 45°, one CGP pass induces a total effective strain of 1.16 throughout the specimen.

Cu-Zr alloy as a face-centered cubic (FCC) metal material and Commercial pure titanium (CP-Ti) as hexagonal close-packed (HCP) lattice metal, both have been widely used in aerospace, transportation, and other fields, due to their excellent performance. In this paper, Cu-Zr alloy and CP-Ti sheet will be processed by CGP technique, the effect of CGP on the microstructure and mechanical properties of the two materials will be comparatively investigated by experiments, and the deformation mechanism will also be discussed.

![Figure 1. Schematic of constrained groove pressing (CGP).](image1)

![Figure 2. Two sets of dies and its processing sheet: (a) Cu-Zr alloy, (b) CP-Ti.](image2)

2. Experimental procedure

Cu-Zr alloy sheets with dimensions of 100×100×2 mm\(^3\) and CP-Ti sheets of 48×48×4 mm\(^3\) were used in this study. The Zr element content of Cu-Zr alloy is 0.87%wt, and the chemical composition of CP-Ti sheets is \( \geq 99.285\% \) Ti, \( \leq 0.30\% \) Fe, \( \leq 0.10\% \) C, \( \leq 0.05\% \) N, \( \leq 0.015 \) H and O balance. In order to improve the homogeneity of microstructure and to eliminate residual stress, the Cu-Zr alloy sheets were fully annealed at 873 K for 30 min and the CP-Ti sheets were annealed at 773 K for 30 min prior to CGP. Pressings were conducted up to two passes on a 2000 KN computer-controlled electro-hydraulic servo testing machine operating at a constant press speed of 2 mm/min at room temperature. In order to reduce the frictional effects, MoS\(_2\) was used as lubricant. Since each pass yields an effective strain of 1.16 throughout the specimen, two passes are expected to accumulate an effective strain of 2.32. Two sets of dies and its processing sheet as shown in Figure 2.

In order to examine the homogeneity of the processed sheets, Vickers Microhardness tests were conducted by HVS-1000 digital micro hardness tester. A 1kg load for 10 s was applied across the transverse section of the plate for all the measurements. The microstructure of these two kinds of materials was examined by utilizing a scanning electron microscopy (SEM). The Chemical etchants of
Cu-Zr alloy and CP-Ti are Ferric chloride hydrochloric acid aqueous solution (1g FeCl$_3$ +2ml HCl +20ml H$_2$O) and Kroll reagent (1ml HF +2ml HNO$_3$ +50ml H$_2$O), respectively.

Tensile tests were carried out for the CGP processed metals. The tensile specimens of Cu-Zr alloy with gage dimension of 8×3.5×1 mm$^3$ for quasi-static test and 8×2.2×1 mm$^3$ for dynamic test were machined. All the CP-Ti specimens hold the same gauge dimension of 6×1.6×1.6 mm$^3$. Room temperature quasi-static tensile testing was carried out on a 2 KN Instron machine operating at constant engineering strain rate of 5×10$^{-4}$ s$^{-1}$. Dynamic tensile testing was carried out by using a split Hopkinson tensile bar (SHTB) operating at strain rates of 1×10$^3$ s$^{-1}$ (for Cu-Zr alloy specimen), 3×10$^3$ s$^{-1}$ and 4×10$^3$ s$^{-1}$ (for CP-Ti specimen). In order to ensure the accuracy of the measurements, at least three sets of parallel tests were conducted under each condition.

3. Experimental results

3.1. Microstructure observations

Microstructure of the annealed and CGP processed Cu-Zr alloy and CP-Ti are shown in Figure 3 and Figure 4, respectively. Clearly, uniform equiaxed grains are present in both the as-annealed and the CGP processed materials. The grain sizes of these two kinds of sheets were determined by intersection point method. The grains of Cu-Zr alloy sheets are moderately refined from 81 μm for as-annealed alloy to 64 μm for that after one CGP process, and 62 μm for that after two CGP process. The grain refining effect is apparent after the first pass, while the grain size is only slightly reduced after the second pass. Compared with Cu-Zr alloy, the grain size refinement of CP-Ti is much more significant, from 61 μm of the as-annealed material to 7 μm of that after two passes.

![Figure 3. SEM image of Cu-Zr alloy: (a) Annealed, (b) after one-pass CGP, (c) after two pass CGP.](image)

![Figure 4. SEM image of CP-Ti: (a) Annealed, (b) after two-pass CGP.](image)

3.2. Hardness

The Vickers hardness of Cu-Zr alloy and CP-Ti as a function of the indentation location is shown in Figure 5. Slight fluctuation in Vickers hardness along the transverse section was found for sheets after each CGP passes. Inhomogeneity factor $I.F. = \{[\sum_{i=1}^{n}(H_i - \bar{H})^2/(n - 1)]^{1/2}/\bar{H}\} \times 100$, is introduced here to quantitatively characterize the hardness uniformity of the material. Where $n$ is the number of tests for each sheet, $H_i$ is the magnitude of hardness for $i$th test, and $\bar{H}$ is the average value of all measurements. Smaller I.F. value indicates better homogeneity.
The average Vickers hardness and I.F. value of Cu-Zr alloy and CP-Ti sheets with respect to CGP passes are shown in Figure 6. The average Vickers hardness of Cu-Zr alloy sheet increases significantly with increment of pressing passes, from 104 HV for the annealed condition to 177 HV for two CGP passes, with an enhancement of 70%. The I.F. value of the hardness for Cu-Zr alloy sheet after one pass is larger than that of the annealed one, indicating that the uniformity of the material becomes worse. This is due to the interface region (as shown in Figure 1) that was less affected by shear deformation at CGP process and its hardness was smaller than the rest of the area. As the pressing goes on, the I.F. value of hardness decreases and the material gets more and more homogenous. Similar with the Cu-Zr alloy, the micro-hardness of CP-Ti increases from 173 HV at annealed condition to 218 HV after two CGP passes, with an increment of 26%.

3.3. Tensile properties
Tensile properties of these two groups of materials are characterized under quasi-static and dynamic loading. The stress-strain curves of Cu-Zr alloy under quasi-static tension are shown in Figure 7(a). It is observed that the yield stress (YS) and ultimate tensile strength (UTS) of specimens processed by CGP are significantly improved compared with those of the as-annealed material. The YS increased from 104 MPa for the as-annealed material to 388 MPa for that after two CGP passes and an increment of 270% was achieved. The UTS of Cu-Zr alloy also shows a considerable increase by 60% from 279 MPa to 457 MPa. It should be noted that the first CGP pass brings in the major part of the enhancement of mechanical properties, where the YS and UTS increased by 203% and 37% respectively. The stress-strain curves of CP-Ti specimens before and after CGP processing is shown in Figure 8. Under quasi-static conditions, the YS of CP-Ti specimens increased from 189 MPa to 314 MPa after the second pass, with an increment of 66%, while the UTS increased from 241 MPa to 357 MPa, with an increment of 48%.

![Figure 5. Hardness distribution of Cu-Zr alloy and CP-Ti sheets, indentation force 1kg, holding time 10s.](image1.png)

![Figure 6. Average Vickers hardness and I.F. as a function of CGP passes of Cu-Zr alloy and CP-Ti Specimens.](image2.png)

![Figure 7. Engineering stress-strain curves at (a) quasi-static tension (5×10^4 s^-1) and (b) dynamic tension (1×10^3 s^-1) of Cu-Zr alloy specimens at various passes.](image3.png)
The engineering stress-strain curves of Cu-Zr alloy under dynamic tension are shown in Figure 7(b). Comparing with Figure 7(a), the flow stress increases obviously with strain rate, which indicating positive strain rate sensitivity of this material. Both of Cu-Zr alloy and pure Ti show apparent strain rate dependence, while the depending degree differs a lot with different CGP passes, prefiguring that strain rate sensitivity of the two materials may rely on their microstructures.

4. Discussion

4.1. Effects of strain rate

Strain rate sensitivity (SRS) factor $m$, is usually expressed as $m = \frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}}$, where $\sigma$ and $\dot{\varepsilon}$ are the flow stress and the strain rate respectively. However, it is unrealistic (and usually unnecessary) to present the SRS in every strain levels. Instead, SRS is usually given at a certain strains in practice. By definition, the SRS value can be determined approximately by deriving the slope of a linear regression fit in the logarithmic stress-strain rate curve at a certain strain.

With the CGP pass number increases from 0 to 2, the SRS of Cu-Zr alloy increases sharply from 0.0018 to 0.0124, while the SRS of CP-Ti decreases moderately from 0.0378 to 0.0289. This distinct difference indicates that the strain rate strengthening mechanism of these two materials differs a lot. In fact, some recent papers have reported changes in SRS in UFG metals, including copper (FCC lattice) and titanium (HCP lattice). Summarizing data from those literatures [7-20], the grain size effect on SRS can be further explored, those result are shown in Figure 9. It can be seen that with the decrease of grain size, the SRS of flow stress of copper metal increases monotonically, and the SRS of copper alloy measured in the present work is closely rounded with the results in the literature. However, the dependence of SRS with grain size is much stronger in this work than those in the literatures.

The theoretical models for grain size effect of SRS of copper (FCC lattice) was derived by Wei [21] and Zehetbauer [14]: When the dislocation density is increased in an FCC metal with a given grain size after the large plastic deformation, the SRS would increase [14]. On the other hand, when the grain size is refined into the UFG/NC regime, the SRS should increase with decreasing grain size [21]. The curve in Figure 9(a) expresses the relationship between SRS exponent and grain size, while the effect of dislocation density is not considered. After two CGP passes, the material undergoes large plastic deformation and its dislocation density increases significantly, which leads to the increase of SRS exponent. In the present work, the grain size changes little, the variation of $m$ with CGP pass is mainly due to the increment of dislocation density. Special attentions should be paid regarding the data of grain size effects on SRS in literature, because dislocation density and processing state, such as annealing, quenching, large deformation and so on, would also affects SRS a lot. There is no mature model or theory for grain size effect on SRS of titanium (HCP lattice) by far, due to limited experimental results. A collection of the available data of titanium is shown in Figure 9(b). The SRS of titanium (HCP) does not vary so much with grain size, which is different from FCC or BCC metals [15]. Actually, HCP metals are usually regarded as materials that behave as those between FCC and BCC metals. For example, Zerilli and Armstrong [22-23] combined the constitutive expressions of
FCC and BCC materials to describe HCP metals in their well-known Z-A model. However, the intrinsic strain rate hardening mechanism of HCP materials needs further investigation.

Figure 9. Variation curves of strain rate sensitivity of copper (a) and titanium (b) with grain size.

4.2. Analysis of strengthening mechanisms

In order to interpret the flow stresses behavior, the specific contributions of various strengthening mechanisms should be quantified. Some researchers [12, 24] attempt to introduce a functional form that can be used to describe an experimental stress-strain curve by separating the thermal contribution $\sigma_T$ and athermal contribution $\sigma_A$ to the flow stress. The athermal part reflects the long-range elastic interactions of mobile dislocations with far field dislocation forests, grain boundary strengthening (GBs), impurities, etc [25]. It can be listed as [24, 26]:

$$\sigma_A = \sigma_{A0} (\mu, C_i) + f(d)$$ (1)

Where $\sigma_{A0}$ is the athermal friction stress and is mainly determined by the solid-solute strengthening which in turn depends on the content of interstitial solutes. $f(d)$ represents GB strengthening mechanism, which is usually expressed by the well-known Hall-Petch relation $f(d) = \beta d^{-1/2}$. The coefficient $\beta$ for pure titanium and copper is estimated to be 0.28 MPa·m$^{-1/2}$ [25] and 0.16 MPa·m$^{-1/2}$ [27] respectively, and is relatively independent of the interstitial impurity.

The thermal part of the plastic flow stress mainly originates from the obstacles to dislocation motion due to interstitial impurities or existing dislocations [8], and could be depicted by the following equation:

$$\sigma_T = \sigma_{TC} (T, C_i) + \sigma_{T\rho} (T, \dot{\varepsilon}, \rho)$$ (2)

Where $\sigma_{T\rho}$ is proportional to the square root of interstitial concentration and is dependent on temperature [25]. $\sigma_{T\rho}$ reflects the effect of dislocation density on the flow stress, which follows the pattern of Hirsch-Baily equation [28]:

$$\sigma_{T\rho} = \alpha(T) \left[ 1 + m \cdot \ln \left( \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \right) \right] M \mu b \rho^{1/2}$$ (3)

Where $b$ is the magnitude of Burger’s vector, $M$ denotes the Taylor factor (take 2.5 for titanium [24] and 3.06 for copper [29] in the present study), $\alpha(T)$ is a coefficient depending on temperature (take 0.3 for titanium and 0.25 for copper in the present study) [30], $m$ is the strain rate sensitivity and $\dot{\varepsilon}_0$ is the reference strain rate (usually taken as the lowest experimental strain rate). It is not easy to measure the dislocation density directly, especially when the material has gone through severe plastic deformation. Nevertheless, some researchers examined the dislocation density of CP-Ti and copper by X-ray technique and found that it was as high as $6 \times 10^{14}$/m$^2$ for CP-Ti and $1.04 \times 10^{15}$/m$^2$ for copper at the effective strain of 2.32 to 3, as compared to $10^{14}$/m$^2$ and $2.9 \times 10^{13}$/m$^2$ in the as-received condition, respectively [28-29].
The contributions of stress from different strengthening mechanisms are summarized in Table 1. Comparing these stress components helps us understand the weight of different contributions. The $\sigma_{\text{GB}}$ and $\sigma_{\text{DIC}}$, which are independent of grain size and dislocation density, were not considered in the present work. It is shown from Table 1 that the bulk contribution of the yield stress is dislocation interaction, especially after CGP processing. Apparently, the main reason for increment of the flow stress after CGP processing for the two kinds of materials is not grain refinement, but dislocation accumulation. Compared with the as-annealed samples, the large plastic deformation formed in the material processed by CGP leads to the increase of dislocation density and the same time, decrease of the distance between dislocations. Dislocation moment becomes more difficult due to the interaction between dislocations and it requires larger stress to generate plastic deformation, which results in the increase of ultimate strength and yield stress.

### Table 1. Contributions of strengthening mechanisms for Cu-Zr and CP-Ti at room temperature.

| Sample            | Grain Boundary Strengthening/MPa | Dislocation Interaction/MPa | Measured Yield Stress/MPa |
|-------------------|---------------------------------|----------------------------|---------------------------|
| As-annealed, Cu-Zr| 19                              | 54                         | 104                       |
| CGP, Cu-Zr        | 20                              | 325                        | 388                       |
| As-annealed, CP-Ti| 36                              | 95                         | 189                       |
| CGP2, CP-Ti       | 105                             | 233                        | 314                       |

### 4.3. Grain refinement and strengthening effects of various SPD methods

The ability of grain refinement is an important parameter for SPD technique. However, it should be noted that the grain refining ability of CGP method is not so effect as other SPD methods, such as HPT and ECAP. A summary of features of commonly-used SPD techniques are listed in Table 2 [3, 6, 8, 16, 17, 19, 20, 24, 31, 32]. For the convenience of comparison, we define two indexes, i.e., grain refinement efficiency (GRE) and strengthening factor (SF). GRE is defined as the ratio of original grain size to processed grain size per effective strain and SF is define as the ratio of processed yield stress to original yield stress. From Table 2, the average GRE for copper alloy and titanium after CGP, ECAP and HPT processes are 0.52, 14.21, 35.77, and 5.47, 21.64, 103.02, respectively. As we can see, the grain refinement ability of CGP is remarkably lower than the other two processes. Peng, et al [31] conducted in-depth study on this issue and contributed the low refining ability to two reasons. The first reason is that the statistically stored dislocations (SSDs) generated under CGP process is fewer than other SPD methods. Generation of SSDs requires multiple shear planes that helps activate multiple sliding systems. However, there is only one maximum shear-stress plane in CGP (parallel with the Y-Z plane as shown in Figure 1), which is less than other SPD processes. The second reason is that the shear strain applied in two consecutive pressing of CGP is in the opposite direction. Some dislocations generated in the groove pressing may recover during the following flat pressing, if they are not locked by certain obstacles. These two reasons leads to lower GRE for CGP compared to ECAP and HPT. However, the strengthening ability of CGP is comparable with other SPD methods. The average SF after CGP, ECAP and HPT are 2.91, 3.23 and 3.28 for copper, and 1.37, 1.73 and 1.91 for titanium respectively. This good strengthening ability, combined with its potential of producing large sheet materials, is very attractive in industrial applications.

One may note that the GRE of copper alloy is much lower than of titanium for all the SPD methods. This is related to the intrinsic characteristics of the two kinds of materials during deformation. CP-Ti has HCP lattice structure, which has less slip systems. Thus, it is difficult for dislocation sliding and more grain boundaries are needed to form to accommodate the large plastic deformation during CGP process. While for FCC metals like copper, there are abundant slip systems to adapt the large plastic deformation and the new grain boundaries are mainly formed due to dislocation accumulation and tangling. Dislocation density increases as CGP processing, while dynamic recovery of dislocation accelerates with the increment of dislocation density. Dynamic recovery leads to dislocation
annihilation and limits the formation of new grain boundaries. Therefore, the grain refinement efficiency is not as significant for materials with good deformation ability, such as copper.

**Table 2.** A comparison between grain size and accumulated effective strain obtained after various SPD processes for Cu alloy and CP-Ti.

| Process   | As-annealed | Processed | Grain Refinement Efficiency | Strengthening Factor | Reference |
|-----------|-------------|-----------|----------------------------|---------------------|-----------|
|           | Grain Size /μm | Yield Stress /MPa | Accumulated Effective Strain | Yield Stress /MPa |             |           |
| ECAP, Cu  | ~60         | 144       | ~0.3                       | 6.4                | 322       | 31.25     | 2.24       | [17]      |
| ECAP, Cu  | ~11         | 103       | ~0.47                      | 8.0                | 347       | 2.93      | 3.37       | [19]      |
| ECAP, Cu  | ~28         | 99        | ~0.24                      | 13.8               | 404       | 8.45      | 4.08       | [20]      |
| HPT, Cu   | ~13         | 159       | ~0.16                      | 5.7                | 584       | 14.71     | 3.67       | [16]      |
| HPT, Cu   | ~60         | 144       | ~0.2                       | 5.28               | 414       | 56.82     | 2.88       | [17]      |
| CGP, Cu   | ~78         | 70        | ~41                        | 3.48               | 220       | 0.55      | 3.14       | [3]        |
| CGP, Cu-Zn| ~23         | 244       | ~11                        | 4.64               | 455       | 0.45      | 1.86       | [3]        |
| CGP, Cu-Zr| ~81         | 104       | ~62                        | 2.32               | 388       | 0.56      | 3.73       | This work  |
| ECAP, CP-Ti| ~30        | 300       | ~0.83                      | 2.80               | 750       | 12.91     | 2.50       | [8]        |
| ECAP, CP-Ti| ~20        | 740       | ~0.2                       | 4.20               | 990       | 23.8      | 1.34       | [24]       |
| ECAP, CP-Ti| ~13        | 680       | ~0.2                       | 6.40               | 1038      | 10.16     | 1.53       | [33]       |
| ECAP, CP-Ti| ~25        | 625       | ~0.15                      | 4.20               | 973       | 39.68     | 1.56       | [34]       |
| HPT, CP-Ti| ~65        | 380       | ~0.07                      | 5.97               | 800       | 155.54    | 2.11       | [35]       |
| HPT, CP-Ti| ~45        | 200       | ~0.15                      | 5.94               | 340       | 50.50     | 1.7        | [32]       |
| CGP, CP-Ti| ~50        | 400       | ~3                        | 2.32               | 432       | 7.18      | 1.08       | [6]        |
| CGP, CP-Ti| ~61        | 189       | ~7                        | 2.32               | 314       | 3.75      | 1.66       | This work  |

5. Conclusions
The fabrication of high performance metal sheets was carried out in this work. Cu-Zr alloy and CP-Ti processed by CGP method were comparatively studied. The effect of CGP on the microstructure and mechanical properties of the two materials was investigated by experiment, and the deformation mechanism was discussed. The main findings of this paper are concluded as follows:

(1) High-performance metal sheets were successfully fabricated by CGP technique. The average grain size of Cu-Zr alloy sheet was refined from 81 μm to 62 μm after two CGP passes. The efficiency of grain refinement on CP-Ti sheet was more remarkable than that of Cu-Zr alloy and its average grain size was refined from 61 μm to 7 μm.

(2) CGP process significantly improved the strength of two kinds of materials. After two CGP passes, the average hardness of Cu-Zr alloy and CP-Ti sheet increased by 70% and 26% respectively. Under quasi-static tension, the yield and ultimate strength of Cu-Zr alloy increased by 273% and 64% respectively after the second pass of CGP, while those of the CP-Ti were improved by 66% and 48% respectively.

(3) Mechanical properties of both of the two types of metals exhibited apparent strain-rate dependency. The SRS of Cu-Zr alloy increases with the increase of CGP pass number, which is mainly due to the increment of dislocation density. The SRS of CP-Ti does not show notable change during CGP processing.
(4) The strengthening mechanisms of copper and titanium after CGP processing were discussed. The analysis indicated that dislocation interaction accounted for the majority of strength elevation, while grain refinement only gave rise to insignificant strengthening effects.

(5) The ability of grain refinement and strength enhancement for various SPD techniques were comparatively studied. Results showed that CGP methods displayed much lower grain refinement efficiency but comparable strengthening effects compared with ECAP and HPT.

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