Effect of revolutions number on mechanical properties of HPT processed copper

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Abstract. This paper studies the effect of high-pressure torsion (HPT) method at ambient temperature on mechanical properties of material. The aim is to compare copper subjected to HPT for 2, 10 and 30-revolutions with coarse grain structured copper in a cold-rolled state. Miniaturized tensile tests were performed to evaluate anisotropy of mechanical properties within a disc product of HPT process. Also the strain rate sensitivity was examined. The results of mechanical tests demonstrate that increasing shear strain leads to ultra-fine grain structure (UFG) which resulted in increasing of material strength. The plasticity of material decreases correspondingly. With respect to character of HPT process, discs are known as non-homogenous products, where shear strain effect increases in radial direction from the centre section to the edge. Results show that with different number of HPT revolutions the anisotropy of mechanical behaviour is changing. With increasing number of revolutions the anisotropy within a single disc is increasing.

1 Introduction
Mechanical properties of materials are a key factor for their successful application. Achieving the optimal combination of strength and ductility is the main challenge in material processing. One of the way how to enhance mechanical properties of material is to use high pressure torsion (HPT) which belongs to severe plastic deformation (SPD) techniques in order to achieve suitable combination of high strength and ductility. SPD processes create ultra-fine grain (UFG) structure with high angle grain boundaries (HAGB) via imposing severe shear strains [1 – 2]. UFG materials may have strength exceeding that of coarse grained (CG) and even alloyed metals [3]. Grain size distribution is in the sub-micrometer or nanometer scale [4]. However, SPD techniques have their limitations. With increasing number of HPT revolutions the strength of material increases but regarding to that the ductility of material decreases [5]. This results in the application limitation of material. For reliable application the material must be characterized by a good combination of strength and ductility. It means rather limitation of number of revolutions processing via HPT, or application of post-processing. Volume of material, which can be HPT processed is insufficient to produce standard specimens. Thus, only small specimen test techniques are possible to use.

In this study, cylindrical rods of pure copper in an as cold-rolled state were subjected to HPT processing at 2, 10 a 30-revolutions. Set of miniaturized tensile tests were performed at room
temperature. The effect of number of revolutions on mechanical properties was discussed as well as dependency of material anisotropy.

2 Experimental material and procedure

Resulting properties after HPT are dependent on the initial material state. The base pure copper (99.9%) of M1 grade with coarse-grained structure was delivered in the form of cold-rolled rods with 20 mm in diameter. This material state is referred as the Coarse Grained (CG). Chemical composition is presented in Table 1.

| As   | Bi  | Fe  | Ni  | O₂  | Pb  | S   | Sb  | Sn  | Zn  | Cu+Ag |
|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-------|
| <0.001 | <0.001 | <0.001 | 0.04 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | base  |

The rod of CG material of diameter 20 mm and 2.5 mm thickness were cut perpendicular to the rolling direction. The UFG structure of the material was produced by SPD using the HPT method [6]. HPT was conducted on Walter-Klement GmbH HPT-07 press at room temperature, presented on Figure 1a). Schema of material processing also is presented, see Figure 1b).

The HPT processing is defined by applied force F and total strain ε expressed by equation (1) [7], wherein N is revolution number, t is the specimen thickness and r is the distance from the centre of the disc to the edge. The equation clearly shows that with increasing distance from the centre and increasing number of revolutions, imposed strain increases.

\[ \varepsilon = \frac{2\pi rN}{t\sqrt{3}} \]  

The billets were compressed under required pressure (6 GPa), then the torsion with a given number of revolutions (2, 10 and 30) was conducted under conditions of hydrostatic pressure and the speed of 1 rev/min. The absence of slippage was checked by the coincidence of the drawn strips on the sample surfaces after torsion. As a result, the load capacity and anvil dimensions allow to produce the discs with diameter of 20 mm and thickness of ~1.9 mm shown on Figure 1c). Two discs of UFG copper after 2 revolutions, 10 revolutions and one disc after 30 revolutions were produced in total. Equivalent strain in the middle of the radius was 4.2, 5.9 and 7 for 2 revolutions, 10 revolutions and 30 revolutions, respectively.

![Fig. 1. a) Walter-Klement GmbH HPT-07 press, b) Schema of HPT process, c) CG and HPT processed copper.](image)

Evolution of ultrafine-grained structure for different revolution number was described in many studies [8-10]. It was shown, that for smaller number of revolutions the differences in grain size from the center of disc to the edge are smaller, since the recrystallization and limited grain growth
occurred. These effects act against refining process and lead to creation of coarse grains. For higher number of revolutions no significant recrystallization areas were observed in the edge area. Nevertheless, in the center area of the disc, equivalent strain for certain revolution number may exceed value, when recrystallization may appear.

3 Miniaturized tensile test (MTT)

MTT is widely used testing technique on different fields of mechanical science nowadays, [11-13]. To examine mechanical properties of HPT discs processed by different number of revolutions, sets of miniaturized tensile tests (MTT) were performed. Examination of mechanical properties of UFG material via MTT was successfully used in different studies [14-17]. Three testing specimens with known location were extracted from each produced disc. MTT specimens in longitudinal (Y) and perpendicular (X) orientation were also produced from CG copper. Specimens were extracted using a wire electro-discharge grinding machine. To keep the same dimensions, specimens extracted from discs were polished to final thickness $t = 0.5$ mm. Extracted specimens and their geometry are presented on figure 2 a) – b).

Figure 2. a) MTT specimens extracted from disc, b) MTT specimens geometry.

Effect of absolute size or state of material microstructure can significantly influence reliability of measured results. State of microstructure, especially grain size has major effect on plastic deformation and Yield strength (YS). With changing of grain size, the number of grains in the specimen cross section is also changing. The main mechanism of plastic deformation for steels is slip of dislocations. With increasing number of grains, the number of dislocations and number of its interaction with grain boundaries increases too. A phenomenon of dependency of YS on number of grains is well known and described by Hall-Petch equation (2) [18]. With respect to miniaturized specimen the predominant dimension is thickness of specimen. The ratio $t/d$, where $t$ is specimen thickness and $d$ is grain diameter, is the decisive parameter, whether the microstructure affects the tensile results or not. Nevertheless, in case of UFG materials, the number of grains located in the cross-section of a specimen is large due to their small size. Therefore, tensile test results are more comparable to standard specimens.

$$YS = \sigma_0 + \frac{k_Y}{\sqrt{d}}$$

MTT were carried out on testing machine with linear drive with the loading capacity of 5kN. Deformation was measured using optical extensometer of Mercury RT system. All the tests were performed at ambient temperature. Cross-head speeds of 0.25 mm/min and 10 mm/min were set up to examine the strain rate effect.

4 Tensile tests results

For all test specimens tensile curves were evaluated. Stress – strain curves of tested MTT specimens are presented on Figure 3. For better clarity and readability, only average curve of each tested batch is
shown on graph. In case of specimens subjected to 30 revolutions, all results are presented. Table 2 present average results and standard deviation of mechanical parameters for each tested group.

![Graph showing engineering stress-strain curves](image)

**Figure 3.** Average engineering stress-strain curves of tested MTT in CG and UFG state.

| Specimen  | Value       | R_p0.2  | R_m    | A_g    | A_s    | Z     | Cross-head | copper state |
|-----------|-------------|---------|--------|--------|--------|-------|------------|--------------|
|           |             | MPa     | MPa    | %      | %      | %     | mm/min     |              |
| CG_X_0.25 | Average     | 99.2    | 207.7  | 35.5   | 47.0   | 74.2  | 0.25       | Coarse Grain |
|           | St. Dev.    | 6.7     | 2.0    | 0.8    | 2.2    | 5.7   |            |              |
|           | Average     | 91.8    | 218.4  | 39.1   | 53.1   | 72.6  | 0.25       | HPT Processing 2 rev. |
|           | St. Dev.    | 5.6     | 0.5    | 0.7    | 2.4    | 0.2   |            |              |
|           | Average     | 104.1   | 220.4  | 33.2   | 45.3   | 69.2  | 0.25       | HPT Processing 10 rev. |
|           | St. Dev.    | 4.1     | 2.7    | 1.0    | 1.2    | 0.2   |            |              |
|           | Average     | 106.2   | 235.5  | 31.6   | 46.4   | 74.3  | 0.25       | HPT Processing 30 rev. |
|           | St. Dev.    | 1.0     | 3.0    | 0.2    | 0.2    | 6.7   |            |              |
| CG_Y_0.25 | Average     | 353.8   | 444.2  | 1.6    | 11.1   | 72.2  | 0.25       | HPT Processing 2 rev. |
|           | St. Dev.    | 18.1    | 6.2    | 0.4    | 0.8    | 3.0   |            |              |
|           | Average     | 401.7   | 475.8  | 1.7    | 11.8   | 72.7  | 0.25       | HPT Processing 10 rev. |
|           | St. Dev.    | 7.7     | 6.1    | 0.2    | 0.4    | 9.1   |            |              |
|           | Average     | 334.2   | 427.1  | 2.0    | 15.2   | 80.5  | 0.25       | HPT Processing 30 rev. |
|           | St. Dev.    | 15.9    | 10.4   | 0.0    | 1.7    | 3.6   |            |              |
|           | Average     | 390.3   | 446.7  | 1.5    | 10.2   | 75.1  | 0.25       |              |
|           | St. Dev.    | 23.0    | 15.1   | 0.2    | 1.0    | 3.9   |            |              |
|           | Average     | 367.3   | 452.1  | 1.9    | 12.4   | 75.7  | 0.25       |              |
|           | St. Dev.    | 20.9    | 21.3   | 0.2    | 0.8    | 1.6   |            |              |

### 4.1 Tensile parameters comparison

As can be seen from presented graph, CG copper structure was characterized by ultimate tensile strength (UTS) about 210 MPa, YS = 100 MPa and elongation of 50 %, thanks to as-rolled state with no heat treatment processing slight anisotropy of base material was present. Considerable difference of tensile properties is visible for HPT processed copper with UFG structure. Results for copper subjected to 2 revolutions show more than 3-times and 2-times higher value of YS and UTS, respectively. Correspondingly to UTS and YS increase, there is significant decrease of material ductility, when uniform elongation changes from 40 % to 2 %. This difference has significant
influence on applicability of UFG material. For copper processed by 10 revolutions, UTS and YS values are slightly lower compared to 2-revolutions specimens.

4.2 Strain rate sensitivity
Effect of strain rate to UFG behavior was examined on CG, 2 and 10 revolutions specimens. Strain rates $\dot{\varepsilon} = 0.0009$ and $0.03 \text{ s}^{-1}$ were applied. For all three material states the increase of UTS and YS is visible. Nevertheless, changes are more noticeable for HPT processed copper after 2 and 10 revolutions. This can be effect of history of deformation, since for increasing number of revolutions the strain sensitivity slightly increases, too.

4.3 Anisotropy of mechanical properties
The last stage of experimental program was to produce UFG copper after 30 revolutions. Three MTT specimens were extracted and tested with applied strain rate of $0.0009 \text{ s}^{-1}$. The results show, that for higher number of revolutions, the changes of tensile properties are almost negligible. Nevertheless, with increasing number of revolutions there is more significant anisotropy of mechanical properties within the produced disc. For discs produced by 2 and 10 revolutions, good repeatability of tests is clearly visible, regardless to the area of extraction. For each tested group of three specimens the results are in good agreement with relatively small standard deviation. On the other hand, in case of specimens subjected to 30 revolutions, lower values of tensile parameters are noticeable for specimens extracted from the center of the disc, compare to specimens from sides of disc. This clearly demonstrates, that although increasing number of revolutions doesn’t affect the tensile properties, continuous shear deformation affects the anisotropy of discs. Described behavior can be a result of recrystallization of structure in the center of disc, whereas on the edge of the disc, imposed strain causes structure’s homogeneity. Comparison of average values and standard deviation of YS and UTS for HPT processed copper is presented of Figure 4 a) and b).

![Figure 4](image)

**Figure 4.** Average and standard deviation comparison of a) Yield stress, b) Ultimate tensile stress.

5 Conclusion
In the presented work, the effect of different revolutions on creation of ultrafine-grained structure was examined. Copper in 4 different structure states was investigated. Base coarse grain material in the cold-rolled state and HPT processed copper after 2, 10 and 30 revolutions were produced. Series of miniaturized tensile tests were performed on extracted specimens to determine differences of processed material. There were found following differences of material behavior.

- With increasing number of revolutions, the tensile parameters, such as YS and UTS increased till saturated state was achieved.
- UFG structures show higher strain rate sensitivity in comparison to CG structure.
• For smaller number of revolutions (2 – 10) anisotropy of mechanical properties within one disc was not observed.
• For 30 revolutions, there is noticeable dependency of mechanical parameters on location of specimen extraction.

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