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A new designed incremental high pressure torsion process for producing long nanostructured rod samples

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Abstract

High pressure torsion (HPT) is one of the most important and effective severe plastic deformation (SPD) processes for producing nanostructured (NS) and ultrafine grained (UFG) metals. Whereas HPT presents excellent mechanical properties, its applications are limited to small disk-shaped samples. In this study a new design of incremental HPT (IHPT) process entitled SIHPT is developed which is much convenient for the production of large NS and UFG metallic rods. In this new design, some steppers along the length of the rod-shaped sample are used while applying an axial load from two ends of it. Step twisting of stepper parts with simultaneous axial loads extend the deformed region to the whole length of the sample. The five turn IHPT process was applied to a 50 mm length and 10 mm diameter pure copper sample and microstructure and mechanical properties were evaluated. The microstructural study of SIHPT processed samples using TEM and EBSD micrographs clearly reflected the NS sample having average grain size of less than 120 nm. Also,

microhardness measurements showed that the sample has fairly good homogeneity through both axial and radial directions. Besides, tensile test measurements indicate that there is about four times improvement in yield strength of nanostructured sample compared to unprocessed metal which is accompanied with satisfactory ductility as a result of high hydrostatic compressive stresses.

Keywords: High pressure torsion; Severe plastic deformation; Nanostructured; SIHPT.

1. Introduction

High pressure torsion (HPT) is an important severe plastic deformation (SPD) method for producing nanostructured disk-shaped samples. In this process, the specimen with normally in millimetre order thickness and diameter experiences extraordinary strains. While the metals normally fail in strains lower than 0.6, in HPT process (also other SPD) the specimen withstands equivalent strains much higher than ten thanks to the very high hydrostatic compressive stresses which increase the workability of the metal. This phenomenon that constitutes the fundamental of HPT process was first reported by Bridgman in 1943 [1]. Despite the successful experiments on the application of HPT process on different materials [2, 3], known failure criteria such as Tresca and Von-Mises cannot explain the effect of hydrostatic pressure in increasing material strength. However, it is possible to study some aspects of the HPT process using numerical methods [4, 5]. The first step in HPT process is the application of a very high hydrostatic pressure on the material. The results of the finite element (FE) analysis showed that this stage plays a major role in grain refinement process at the following stages [6, 7]. The second step which is considered as a main grain refinement stage is the application of high shear plastic strain on the sample by twisting [2]. Limitation of sample thickness is one of greatest hindrances which limits the application of HPT process to thin disks. Applying HPT process to samples with arbitrary length is a

challenging task that attracted the interest of several researchers. Sakai et al. [8] showed that when increasing sample thickness, obtaining a sample with homogeneous microstructure using conventional HPT method is not possible. Edalati et al. [9] presented a special die design of HPT process that applies to wire and sheet parts, but cannot be utilized for bulk rod-shaped long samples. Recently, Ivanisenko et al. [10] have developed a new modification of the conventional high pressure torsion technique named as High Pressure Torsion Extrusion (HPTE) which is capable to process rods or billets of metals. The key highlight of this method includes the possibility to accumulate large shear strain during one pass of the process. But the main shortcoming is the limitation in the magnitude of the applied pressure. In HPTE system the application of high pressures which is required during the processing of hard materials is challenging. Another valuable technique in this regard offered by Hohenwarter [11] as an Incremental HPT (IHPT) process. Hohenwarter has successfully applied IHPT to a technically pure copper sample with 70 mm length and 50 mm diameter. Despite the admirable results from IHPT, it has few shortcomings as well. The most important disadvantages of the IHPT process is that it takes too time to process the material and needs to disassemble the die in each shifting step. In this paper, a new technique is offered to do IHPT process continuously. The new technique, which afterwards will be addressed as single-task IHPT or SIHPT, is faster, more efficient and is much suitable for industrialization. In this study, SIHPT method is developed and the working principle of that has been provided. This method has not been reported hitherto. A copper rod is processed in order to reveal the capability of this new presented approach. Mechanical and microstructural properties of the processed sample were examined. Results showed that mechanical properties including microhardness and tensile strength have significantly improved in the nanostructured material using the presented method.

2. Principles of SIHPT Process

Figure 1 schematically shows the different steps of SIHPT method. The key innovation in the design of SIHPT die is using multi-piece die instead of the one-piece die in IHPT process. Each piece of the die is called “stepper” which has about 4 mm thickness. Steppers have a 10 mm hole in their centres to locate the specimen. Because the sample length is high, then it cannot be processed at one stage. Hence, different sections of the sample are processing step by step using four millimetre thick squared steppers.

Fig. 1. Schematic of different stages of SIHPT process

The processing of the sample starts from the bottom and ends at the top of the sample. At the starting, all steppers are fixed, and only one of them at the lower part of the specimen is rotating (Fig. 1(a)). This causes that a small region of the sample to be plastically sheared (Fig. 1(a)). In next step, all steppers are fixed except two of them from the bottom of the sample (Fig. 1(B)). Then, two bottom steppers are rotating simultaneously. This causes a small length of the sample at the vicinity of the temporary rotational interface to be processed. This algorithm is repeated until the entire of the sample is plastically sheared (Fig. 1c and d).

The application of high contact pressure helps to stick the sample to the internal wall of steppers and prevents the sliding of the specimen during the rotation. In experiment, the specimen length is about 10 mm longer than the total thickness of all steppers from both ends as shown in Fig. 2a. This extra length compensates any metal loss as a result of any clearance between the sample and steppers for easily locating at the start of the process. It also increases frictional forces which are needed to prevent the slippage.

3. Experimental Material and Procedure

The SIHPT process is applied to a commercially pure copper sample which was annealed at 700°C for one hour. The sample length and diameter were respectively 50 mm and 10 mm. Fig. 2b shows different parts of SIHPT die manufactured and used in this study. After locating SIHPT rig on a hydraulic press machine at the beginning of the process, for the production of about 2 GPa hydrostatic pressures on the specimen, 16 tons force is applied to the sample. It was seen that the length of the specimen was reduced about 8 mm under 2GPa pressure. That was because of the material flow into the clearance gap between the specimen

and the steppers. The rotation of the steppers is performed via two 1.5 m levers located at the four lateral holes of the die. The rotational speed was about 0.05 rotations per minute to avoid any heat productions and temperature rise effects. In the current experiment, each stepper is rotated to five complete revolutions. Steppers have square shapes, and their thickness was 4 mm (Fig. 2a). This amount is a suitable value for the thickness of steppers because according to previous studies [11] about 8 mm of the specimen at the vicinity of the rotational interface is affecting by torsional strains after the rotation. The length of this strain affected zone mainly depends on the specimen diameter as well as steppers internal corner radius. However as a conservative decision, it was chosen 4 mm for the thickness of the steppers. Following microhardness tests confirmed that 4mm for the steppers thickness is desirable. Steppers also have small fillets in their corner which helps to the concentration of the torsional strain in a small region of the sample. To study the mechanical properties of the processed sample, the specimen is split from the middle off and then polished and its hardness is measured in points located at the axial and radial directions using microhardness tester (Fig. 3a). The Vickers microhardness tests were performed at an applied load of 50gr and dwelling time of 15sec. The microstructure of the samples at both radial and axial positions was studied using SEM, TEM and EBSD techniques. Using micrographs of processed and unprocessed samples, the grain size and its distribution were analysed.

Five samples for SEM examination were cut perpendicular to axes of the cylindrical specimen in the axial direction. After mechanical polishing, the samples were etched within 20-40 sec using a solution of 20ml NH_4OH , 10ml H_2O and 10ml H_2O_2 . For TEM characterization, samples have been lifted using Focused Ion Beam (FIB) from XY-plane (Fig. 3(a)) in to the depth of the sample which is referred to as axial direction of the specimen. Thin lamella TEM samples with the sizes of $20 \times 8 \mu\text{m}$ were prepared from the processed specimen using in-situ lift-out procedure on a dual beam system (FEI Nova Nanolab 600).

After the final thinning procedure, regions with thickness less than 150nm were produced for the following TEM characterisation.

Fig. 2. (a) Sample inside of the steppers (b) different parts of SIHPT rig

(a)

(b)

Fig. 3. (a) Schematic of the microstructure and microhardness test positions, (b) tensile test sample dimensions.

In addition, a field emission gun scanning electron microscope (JEOL 7100F FEGSEM) equipped with an EBSD camera (EDAX TSL) was operated at 20 kV and ~ 26nA is used for EBSD data collection. Data was collected and then analysed using TSL OIM Data Collection and Analysis software. For EBSD analysis, samples were mounted in un-conductive resin, a thin layer of carbon coating was applied using sputter machine to allow examination under electron microscope. Prior any data analysis, a clean-up procedure was carried out on the data set to remove all data with confidence index less than 0.1. Inverse Pole Figure (IPF) maps with rotation angle between 15° and 65° are highlighted in black lines.

Besides a microstructural study, a tensile test is performed according to ASTM E8 standard for both unprocessed and processed samples (Fig. 3b).

4. Results and Discussion

Figure 4 indicates the unprocessed and SIHPT processed specimens. As it is evident, the final surface of SIHPT processed sample is not smooth enough and needs serious surface preparation before the application. By the current arrangement of SIHPT die, processed specimens are moving out of the steppers by the force of hydraulic ram. Then, an external appearance of the sample is extremely affected during the process of removing specimen from the die. Figures 5(a&b) show EBSD and related colour code presentation of the unprocessed sample prior to SIHPT process. In addition, SEM micrographs from positions located at the near edge and mid radius of the SIHPT processed sample are presented in Fig. 6. Although it is not possible to precisely measure the size of the nanostructured grains using SEM photos; but these photos give good qualitative approximation about the amount of grain refinement in different locations of the processed sample. SEM investigation is much easier and faster than the TEM or EBSD and therefore, in this study, several SEM photos from

different locations of the processed sample were used to study the microstructural homogeneity across the sample.

Fig. 4. Unprocessed (a) and 5-turn SIHPT processed (b) samples.

Fig. 5. EBSD illustration of as annealed sample (a), EBSD colour code (b)

Fig. 6. SEM microstructure of SIHPT processed sample at mid-radius (left), at near the surface (right).

For further detailed investigation, TEM micrographs from strategically selected locations of the same sample were taken in order to confirm SEM finding about the homogeneity of the sample. Figure 7 indicates one of TEM samples of this study which had been prepared using

FIB from near edge of the sample. Figure 8 shows the corresponding microstructure with larger magnification. According to Fig. 8 the microstructure composed of grains with different sizes, but their average size is under 120nm which is calculated using linear intercept method. This is substantially lower than the average grain size of the unprocessed annealed sample (40 μm).

Fig. 7. TEM lamella prepared through axial direction of the sample (depth of the cross section) at the edge

The obtained average grain size also is relatively lower than that of the copper sample processed with similar methods such as equal channel angular pressing (ECAP) Process (150-300 nm) [12]. In Fig. 8 there are many neighbouring grains which their features are different. Furthermore, inhomogeneity in deformation can be widely observed within individual grains. While the area of a particular grain is completely dislocation-free (arrows A, Fig. 8), its neighbouring grains are severely deformed (arrows B, Fig. 8). This inhomogeneity in

deformation could be related to misorientation within a grain [13]. It means that grains with full dislocation density break down to smaller ones, but grains which still have not been saturated with dislocation remain less affected. That is why the final HPT processed microstructure always has inhomogeneous grain sizes.

Fig. 8: TEM micrographs of SIHPT sample at a position near the edge after five revolutions

Fig. 9: TEM micrographs of SIHPT sample at the inner position after five revolutions

Figure 9 also illustrates TEM micrograph of the same sample in another location through the length of the specimen and at the inner position of the rod sample. As it is obvious from Fig. 9, there are many curved grain boundaries at the microstructure of the processed sample (Arrows 'a' in Fig. 9) which are evidence of non-equilibrium grain boundaries. These non-equilibrium curved boundaries appear less at the micrographs of the sample's edge. It could be reason that the grain refinement process has not been completed at the inner part of the sample but it reached its saturation at the edge of the sample. Another feature of the microstructure shown in Fig. 9 is that grains which their orientation is perpendicular to axial Z direction (Arrow B in Fig. 9) have not been considerably affected by torsion stress and then are not fully refined. However, grains with orientation toward the length of the sample (Arrow C in Fig. 9) are refined in each twisting step and heavily affected by the torsion stress.

As it can be calculated using Eq. 5 like the conventional HPT process, during the twisting of the sample in SIHPT process no considerable strains occur at the centre of the sample. As a result it is expected that the microstructure at the central region of the sample to remain less

Fig. 10: EBSD illustration of the microstructural evolution at the centre of SIHPT processed sample

affected by the SIHPT process. In order to examine this issue a relatively low magnification EBSD map was collected from the centre region of the SIHPT processed sample. According to Fig. 10 the microstructure at the centre has been refined a little; but the original coarse grains are still visible and the density of low-angle boundaries (LAGBs) is very high. Furthermore, sub-grain structures can be observed in the EBSD illustration which is indicated by different colours, reflecting different orientations within a same grain. As it is obvious from Fig. 10, there are great fluctuations in the orientations within the grains and there are still larger changes in the orientations adjacent to the grain boundaries and near the triple junctions. These sub-grains and LAGBs are the evidence of on the eve of refinement process which would happen by more twisting. These results for the microstructural evolution at the centre are in good agreement with the finding of An et al. [14] using conventional HPT and for copper sample.

Figures 11a and 11b show the result of Vickers microhardness values for both SIHPT processed and unprocessed samples along the radial and axial directions, respectively. As it is clear from Figs. 11a and 11b, the hardness of the SIHPT processed sample improved substantially (more than 50%) in comparison with that of the unprocessed sample. As it can be seen, there is a little inhomogeneity in the hardness measurement values of the sample along the axial direction. In addition, the hardness along the radius of the sample was increasing by increasing distance from the centre of sample, which confirms that the strain saturation has not been occurred along the radius. This is in good agreement with the microstructural studies discussed before.

Figure 12 shows the stress-strain curve of the pure copper sample before and after SIHPT process. Figure 12 also compares the tensile strength of SIHPT processed sample with corresponding four pass ECAP and five turn conventional HPT process. As it can be seen, imposing severe plastic deformation via SIHPT process improved yield and tensile strengths

of the copper sample about 4 and 1.5 times, respectively. These improvements have been achieved to the worth of about 33% reduction in elongation. Furthermore, as shown in Fig. 12, the yield point of the processed sample consists of about 90% of the ultimate strength,

(a)

(b)

Fig. 11. Vickers microhardness variation along the (a) radial and (b) axial directions.

however, at the stress-strain curve of annealed sample a yield point consists only about 55% of ultimate strength. This means that there is a sizable difference in the initial part of the

stress-strain curves of as annealed and SIHPT processed samples; indicating very high yield strength in SIHPT processed sample and very low one for as annealed sample. Comparing the stress-strain curve of the SIHPT processed sample with four-pass ECAP process indicates that there is no substantial improvement at the ultimate strength of the copper sample by using SIHPT, however, as it is clear from Fig. 12, SIHPT sample showed considerable improvement in elongation comparing with four-pass ECAP sample.

According to Fig. 12, both elongation and ultimate strength properties of the SIHPT processed sample have been improved sizeably; however elongation and ultimate strength of the SIHPT processed sample is not as good as conventional HPT. It should be noted that available data for the tensile curve of the conventional HPT was measured using micro-tensile test method and there is possibility for scale errors.

Fig. 12. The stress-strain curve of the pure copper sample before and after SIHPT process, after four-pass ECAP process [15] and after five turn conventional HPT process [16].

To avoid any slippage during SIHPT process, it is crucial to ensure that frictional forces between the specimen and the internal wall of the steppers are enough to overcome the flow stress of the copper specimen during the rotation stage. Edalati et al. [17] studied the significance of slippage in processing by high-pressure torsion process. According to their studies, the extent of any slippage at the HPT and IHPT processes depends critically upon the material. Where, there is slightly more slippage in Cu than Al. Actually, with increasing the pressure, the frictional shear stress exceeds the yield stress of the material and by increasing in torsional stress, a plastic flow occurs at the material. It means that by rising in hydrostatic pressure a material sticks to the die wall and the slippage do not occur [18]. According to Amonton's friction model, the relation between the hydrostatic pressure and the frictional shear strength is as following:

$$\tau_f = \mu P_a \quad (1)$$

where, P_a denotes hydrostatic pressure, τ_f is the frictional shear strength of the interference and μ is friction coefficient. It is known that the friction coefficient between material pairs changes drastically by changing in normal pressure but remains approximately unchanged after reaches to its minimum value in higher pressures [19]. In this study, the coefficient of friction between the copper specimen and steel die was measured experimentally. The results revealed that the friction coefficient for steel-copper pair system is about 0.22 for pressures higher than 0.8 GPa. By neglecting the friction between the sample tips with a die, the friction force between die and specimen (F_f) can be calculated as Eq. (2):

$$F_f = \tau_f \times \pi dL \quad (2)$$

In this equation d is specimen diameter and L is the length of specimen inside of the fixed steppers. On the other hand, the required force (F_r) to overcome the shear yield strength (τ_{sy}) of the nanostructured specimen is as Eq. (3):

$$F_r = \tau_{sy} \times \frac{\pi d^2}{4} \quad (3)$$

No slippage state will happen if the condition $F_r < F_f$ satisfies. For the experiment of the current study it is satisfied as following:

$$\begin{cases} F_f = \mu P_a \times \pi d L = (0.25 \times 2 \text{ GPa}) \times \pi (10 \text{ mm}) L = 15.7 L \\ F_r = \tau_{sy} \times \frac{\pi d^2}{4} = (230 \text{ MPa}) \times (78.5 \text{ mm}^2) = 18 \text{ N} \\ 18 < 15.7 L \Rightarrow \underline{L > 1.15 \text{ mm}} \end{cases} \quad (4)$$

As it is obvious from Eq. 4, considering 2 GPa hydrostatic pressure and 10mm specimen diameter, $L > 1.15 \text{ mm}$ is theoretically enough to stick the material to steppers wall during the rotation stage. Because the thickness of the steppers is 4mm, then one stepper is sufficient as a fixed side to avoid any slippage. As it is evident from Fig. 13, after SIHPT process, small thickness of specimen remains on a stepper wall, which confirms that full stick occurred during the process and there is no considerable slippage during the rotational period. It should be noted that for minimum frictional forces at the other rotating members of the die, it was used graphite powder and graphite foil as a lubricant whenever required. This issue helped a

Fig. 13. A thin layer of the copper sample on a stepper hole after the test which implies a perfect stick between the sample and the stepper.

required torque to rotate the die to remain minimal. Equivalent plastic strain of SIHPT process, ϵ_{eq} is given by the relationship of the form of Eq. 5:

$$\epsilon_{eq} = \frac{2\pi Nr}{h\sqrt{3}} \quad (5)$$

Where N is the number of SIHPT turns, r denotes the specimen diameter and h is the thickness of the steppers. It is reported that the strain saturation of pure metals occurs at strains between 16-22 [20]. For 4mm stepper thickness and $N = 5$, the equivalent strain in different radius of the sample will differ from zero at the centre to 21.75 at the edge of the sample as per Eq. 5. This is in agreement with the microhardness results (Fig. 11) that implies the saturation had not occurred entire the thickness of the sample in radial direction.

5- Conclusion

In this study, SIHPT is introduced as a new generation of HPT process. Different features of the process were discussed, and the application of the process to pure copper sample was demonstrated. The evaluations based on Microhardness tests and SEM images besides the TEM and EBSD analysis showed that the SIHPT processed samples have good homogeneity through their length and radius and according to microhardness measurements inhomogeneity along the length is only about 12%. TEM micrographs from different locations of the processed sample showed that after five full revolutions, a nanostructured sample with average grain size of about 120 nm can be achieved. According to the results, four times improvements in yield strength and 1.5 times improvement in Vickers microhardness can be obtained to the worth of 33% reduction in elongation. The new design keeps the fundamental of conventional HPT and IHPT designs with the advantages of overcoming thickness limitation in conventional HPT. In this new design, the multi-step stage of shifting along the length of the sample during IHPT process has been changed to a one-step task. Thanks to the

multi-piece die design, SIHPT is capable of processing a larger range of lengths and diameters.

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