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Abstract

A newly developed severe plastic deformation method, continuous high-pressure torsion (CHPT), was modified for continuous processing of metallic wires. In this study, using the CHPT, wires of high-purity aluminum (99.99%) and copper (99.999%) with diameters of 2 mm and total lengths of 100 mm were successfully processed by employing the same features as conventional high-pressure torsion (HPT) technique. The results of hardness measurements, 35 Hv for Al and 116 Hv for Cu, after CHPT at an imposed equivalent strain of ~ 13 were consistent with those of conventional HPT using disk and ring specimens, as well as with those of CHPT using sheet specimens. Transmission electron microscopy (TEM) demonstrated that the microstructural elements are elongated in the shear direction after CHPT. The average grain size reaches the steady-state level, $\sim 1.3\ \mu\text{m}$, in Al, but the microstructure is at the non-steady state in Cu with subgrain sizes in the range of $0.3\text{--}4\ \mu\text{m}$.

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Introduction

Ultrafine-grained (UFG) microstructures and resultant high strength and reasonable ductility are achieved by processing materials through the application of high-pressure torsion (HPT) [1-4]. In the HPT method, a thin disc or ring is held between two anvils under a high pressure and severe plastic deformation is imparted by rotating the two anvils with respect to each other [5-7]. The HPT method has two main merits when compared to other severe plastic deformation (SPD) techniques. The first merit of HPT is the continuous-straining feature of the method and thus it is effective to introduce giant shear strains up to a steady-state level [8-16]. On the contrary, other SPD methods, such as equal-channel angular pressing (ECAP), are noncontinuous in straining and the workpiece should be subjected to several repetitive passes to attain high strains [17-22]. As a second merit of HPT, since the process is conducted under high hydrostatic pressures, the fracture of workpiece is significantly suppressed and thus it is applicable to all kinds of metals and semi-metals [23] even including hard and less ductile intermetallics and ceramics such as Ni_3Al [24] and Al_2O_3 [25].

Despite the merits with HPT, the use of the technique in industrial applications is limited because of the small dimensions of samples although the sample size is currently extended to 40 mm in diameter for discs [26,27] and 100 mm in diameter for rings [28]. The HPT process has been used in a form of disc or ring but it is strongly desired to be performed in a form of sheet or wire. An alternative technique, which was called continuous high-pressure torsion (CHPT) [29], was recently developed that is applicable for the continuous processing of metallic sheets. The CHPT method should be effective in scaling up the sample size without sacrificing the pressure whereas the sample size is limited in conventional HPT because the pressure is reduced with any increase in the sample size. The method of CHPT was conducted using the sheets of Al, Cu and Fe with 0.6mm thicknesses, 3mm widths and 40-80mm lengths and it could provide a continuous process for reaching a steady level of hardness and a minimum grain size in these metals [29].

In this study, in order to process metallic wires, which are more appropriate than disc and ring shapes for industrial applications, the CHPT using wire specimens is introduced with a simple modification of the earlier procedure for the CHPT using sheet specimens. Thus taking advantage of the merits of CHPT, it is shown that wires of Al and Cu are processed in an expeditious way while maintaining the fundamental characteristics of HPT.

Principles and Facilities

The principles of CHPT using sheet specimens were introduced in Ref. [29]. The facility for the CHPT using wires, as schematically illustrated in Fig. 1, is similar to that for the CHPT using sheet specimens. The facility consists of two anvils: the lower anvil, which is rotated during process, has a roughened ring-shaped groove; and the upper anvil, which is fixed during process, has a half ring-shaped groove on the surface. The grooves have circular cross sections with 2mm diameter, 0.9mm depth and outer diameter (OD) of 40 mm. A wire with 2mm diameter is bent to a U shape, as shown in Fig. 1, and it is used as an initial sample. Each sample is placed on the lower anvil and the pressure is applied on the sample by raising the lower anvil up to a rigid contact with the upper anvil. The lower anvil is then rotated with respect to the upper anvil and shear strain is introduced in

the sample under a high pressure. Since the surface roughness of the groove on the upper anvil is intentionally reduced with respect to the surface roughness of the groove on the lower anvil, a continuous flow of the material occurs in the rotation direction because of the difference in slippage. The wire is also bent slightly upward and pulled out in tension manually during the process to keep the material flow continuous.

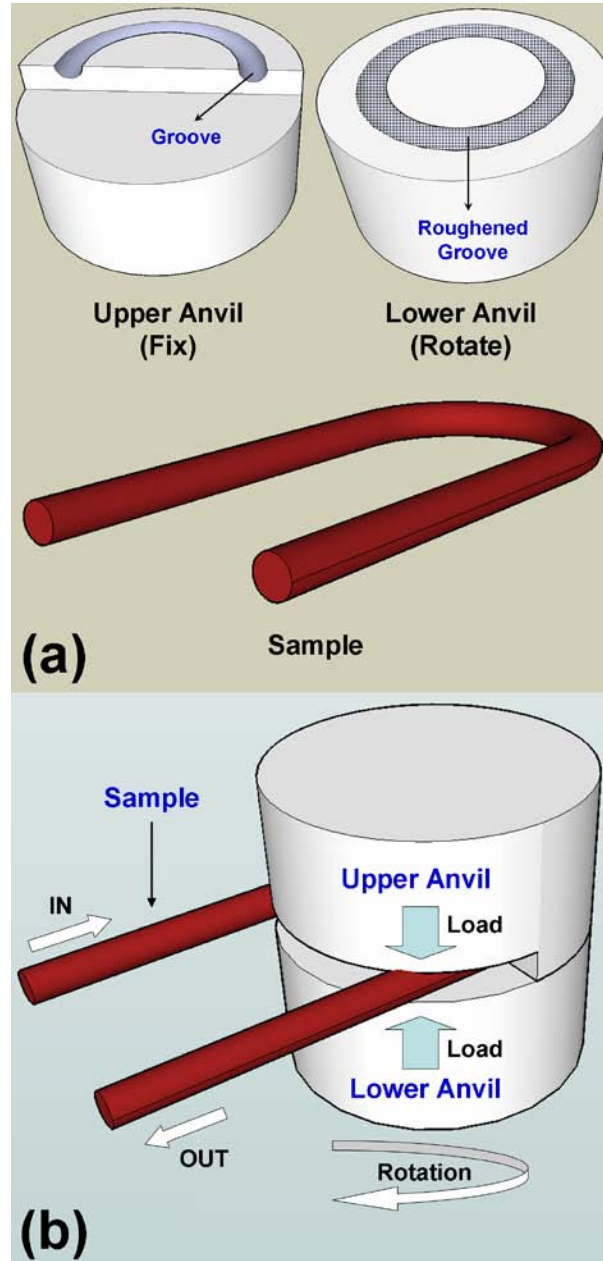


Figure 1. Schematic illustration of CHPT using wire samples. Groove width: 2 mm, groove depth: 0.9 mm, outer diameter of groove: 40 mm, wire diameter: 2 mm.

Fig. 2 shows the appearance of the Al and Cu wires before CHPT and after CHPT for 1 revolution. It is apparent that wires of Al and Cu with diameters of 2 mm and total lengths of 100 mm were successfully processed for the duration of 1 minute using the CHPT. It should be noted that the wire is not round especially in the lower portion on the cross section after processing, but it is almost uniform along the longitudinal wire direction. Moreover, the cross section of the

processed wire is decreased by ~30% when compared to that of the initial wire. Therefore, subsequent shaping processes such as wire drawing or round-concave rolling may be used to modify the cross sectional shape of the wire.

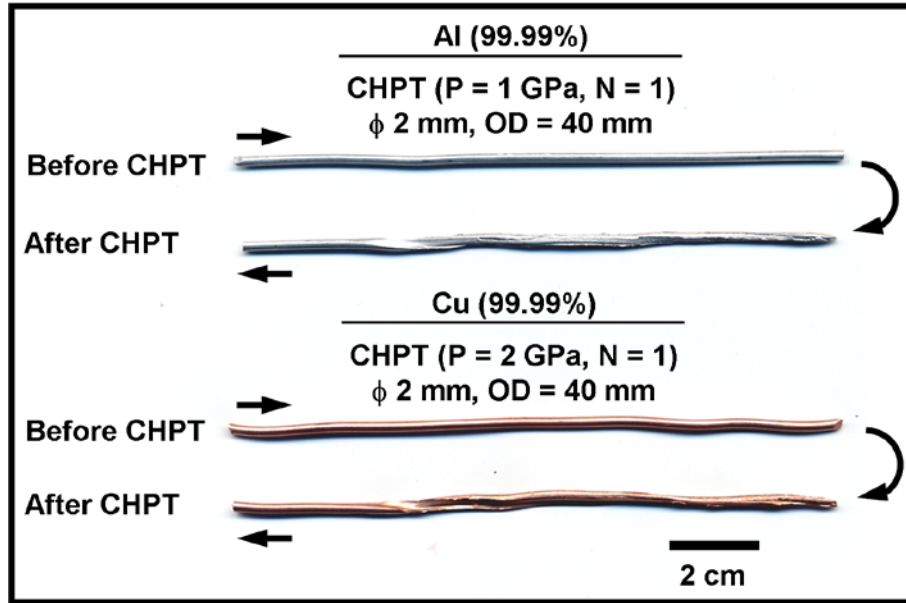


Figure 2. Appearance of Al and Cu wires with 2mm diameter before and after CHPT for 1 revolution.

The equivalent strain imposed by CHPT, ε , is given as [29]

$$\varepsilon = (1 - s) \frac{\pi R}{\sqrt{3} t} \quad (1)$$

where s is the fraction of sample slippage as described in an earlier report [30], R is the mean radius of the groove, and t is the sample thickness. For this study, R is 19.5 mm, t is 2 mm, and s is in the range of $0 < s < 1/2$ as estimated in Ref. [29]. It should be noted that $t = 2$ mm is the maximum value at the wire diameter but in practice, it is smaller as the position being away from the wire center. Since the real equivalent strain may scatter significantly from the nominal level because of the effect of slippage in proportion to $(1-s)$ [31], here, the equivalent strain was estimated under the condition of $s = 1/4 \pm 1/4$. The estimation leads to $\varepsilon = 13 \pm 4$ in this study.

Experimental Materials and Procedures

High purity Al (99.99%) and Cu (99.999%) were received in the form of drawn wires with diameters of 2 mm and lengths of 2 m for Al and 5 m for Cu. These materials were selected because they match the material processed in earlier reports with HPT using disc specimens [9,32-35], HPT using ring specimens [7,28,35], CHPT using sheet specimens [29] and ECAP using rod specimens [20-22], although the purity of Cu in this study is slightly higher than 99.99% used in Refs. [32-35] and higher than 99.96% used in Ref. [22]. The hardness of wires in the drawn direction was 30 Hv and 82 Hv for Al and Cu, respectively, of which values are similar to the hardness values of disc

samples after compression state of HPT [28,35]. The wires were bent to U-shaped specimens and each specimen was placed on the lower anvil. The pressures of 1 and 2 GPa were applied at room temperature on the Al and Cu specimens, respectively, by raising the lower anvil up to a rigid contact with the upper anvil. The lower anvil was then rotated with respect to the upper anvil with a rotation speed of 1.0 rpm until the rotation was terminated after 1 revolution. Following CHPT, the samples were evaluated in terms of Vickers microhardness and transmission electron microscopy (TEM).

For hardness measurement, discs with 2 mm in diameter and 1 mm in thickness, were cut in the direction perpendicular to the longitudinal axis of the wire at positions 5 mm away from the exit of the upper anvil. The disc samples were then polished to a mirror-like surface and the Vickers microhardness was measured along the radii from the disc center to periphery at 8 different radial directions with 0.4 mm increments. For each hardness measurement, loads of 50 g for Al and of 200 g for Cu were applied for 15 seconds. For TEM, rectangular strips with dimensions of $0.15 \times 2 \times 3 \text{ mm}^3$, having a plane parallel to the shear direction and parallel to the anvil surface, were prepared from the positions 5-8 mm away from the exit of the upper anvil. The strips were polished with an electro-chemical polisher using a solution of 10% HClO_4 , 20% $\text{C}_3\text{H}_8\text{O}_3$ and 70% $\text{C}_2\text{H}_5\text{OH}$ for Al, and 15% HNO_3 , 15% $\text{C}_3\text{H}_5(\text{OH})_3$ and 70 % CH_3OH for Cu. TEM was performed at an accelerating voltage of 200 kV for microstructural observation and for recording selected-area electron diffraction (SAED) patterns.

Results and Discussion

Figure 3 plots the hardness variation across the cross section at the wire center as well as at the distances of 0.4 and 0.8 mm from the wire center for (a) Al and (b) Cu. The microhardness values lie reasonably on a constant level for each metal, suggesting that the homogeneity is established throughout the wire samples after CHPT. The average of hardness across the cross section was taken for each metal and plotted in Fig. 4 together with earlier data of (a) Al and (b) Cu obtained by conventional HPT using disc and ring specimens [28,35]. In addition, the data obtained by CHPT using sheet specimens were taken from Ref. [29] and plotted in Figs. 4(a) and (b). The results of HPT and CHPT are consistent except somewhat higher value obtained for Al wire processed with CHPT; and somewhat lower value obtained for Cu wire processed by CHPT. It should be noted that the present data were plotted using $s = 1/4$ and this may result in overestimation of the equivalent strain ($\varepsilon = 13$ for wires). Inspection of Figs. 4(a) and (b) reveals that the imposed strain for CHPT-processed Al wires lie almost on the steady state of straining, whereas the imposed strain for Cu wire is not enough to reach the steady state. It is noted that the steady state begins at an equivalent strain of ~ 10 for Al [28] and at equivalent strains of ~ 15 for Cu [35] using conventional HPT. In order to reach the steady level of hardness for Cu, the wire should be processed for a second pass through the anvils or the ring diameter should be increased to 60-80 mm.

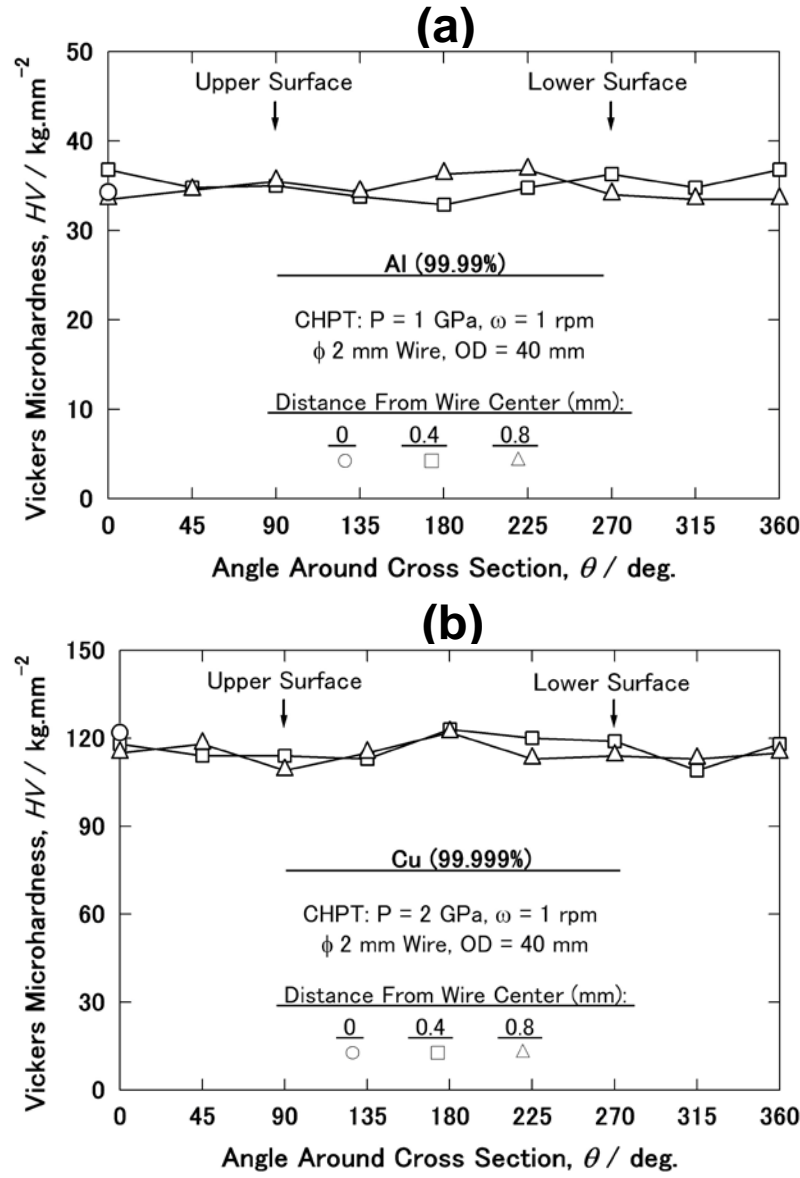


Figure 3. Hardness variation across cross section of (a) Al and (b) Cu wires after CHPT.

TEM microstructures are shown in Fig. 5 for the Al wire after CHPT processing, where a bright-field image and a dark-field image including the corresponding SAED pattern are shown in (a), (b) and (c), respectively. Note that the dark-field image was taken with the diffracted beam indicated by the arrow in the SAED pattern. It is apparent from Fig. 5 that few dislocations are visible in the grains and the grain boundaries are straight and well defined. These microstructural features, which are typical of grains after annealing, are consistent with the earlier observations using conventional HPT [7,9,35], CHPT with a sheet form of samples [29] and ECAP [20,21]. However, observation of Figs. 5(a) and (b) reveals that the grains are elongated in the rotation direction with an aspect ratio of 1.5 and with the average grain size of $\sim 1.3 \mu\text{m}$. These features of grains are different from the earlier observation using conventional HPT and CHPT with sheet samples where the elongated grains were rarely seen within the microstructure. Two reasons may be

considered for this difference: one is that the strain imparted in the sample was rather small as discussed in association with the higher hardness shown in Fig. 4(a), and the other may be due to the cross sectional shape which is round for the wire sample. For the latter, the introduction of shear strain and thus the evolution of microstructure may be different from the thin disc and ring samples with an equal thickness.

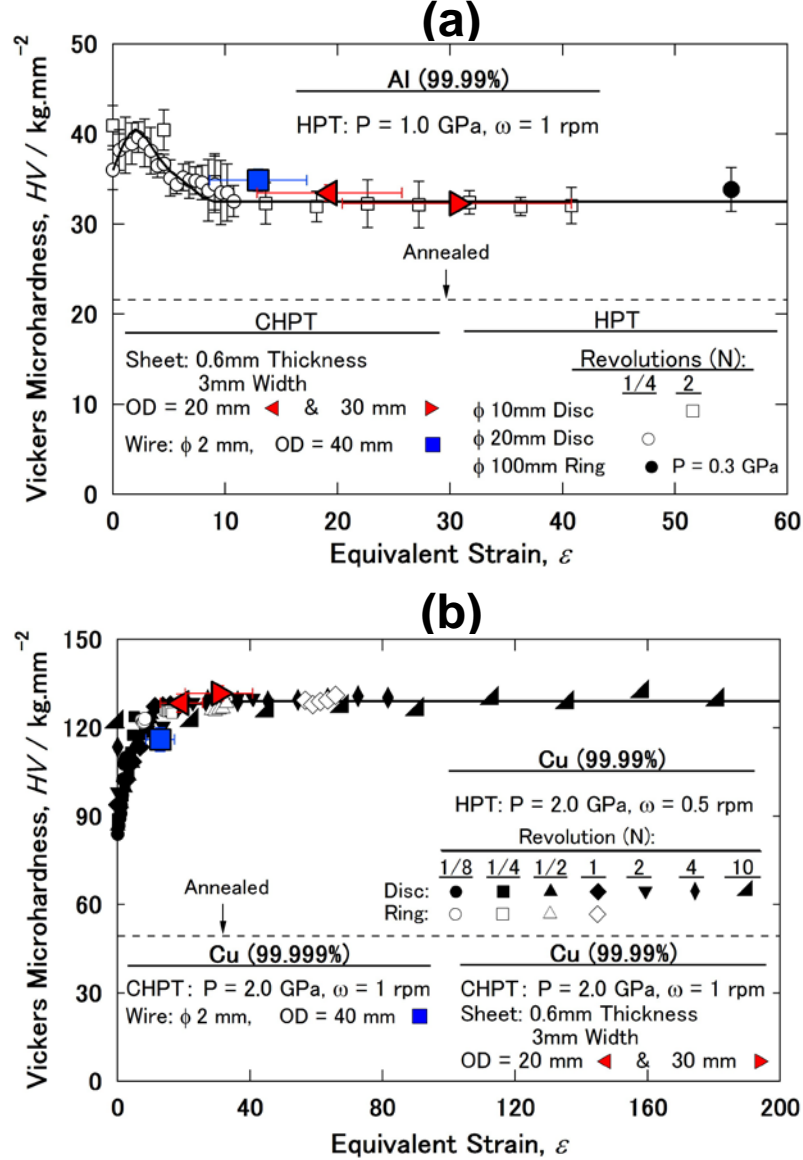


Figure 4. Plots of Vickers microhardness against equivalent strain obtained earlier using conventional HPT for disc and ring samples of (a) Al [28] and (b) Cu [35] including results of CHPT using sheet [29] and wire samples.

TEM observations are summarized in Fig. 6 for Cu after CHPT, where (a) is a bright-field image, (b) is a dark-field image of (a) taken with the diffracted beam indicated by the arrow in the SAED pattern shown in (c). Observation reveals that the microstructure consists of ill-defined subgrains and there are many dislocations within them. The average subgrain size appears to be in the range of 0.3-4 μm and the misorientation angle should not be developed high as evident from a

discrete appearance of the diffractions spots in the SAED pattern. The average subgrain size in Fig. 6 is larger than the average steady-state grain size of $\sim 0.2 \mu\text{m}$ and $\sim 0.27 \mu\text{m}$ reported for HPT- and ECAP-processed samples, respectively [22,35]. However, the microstructural features observed in Fig. 6 are typical of microstructures at an early stage of straining by HPT and ECAP [22,35]. As mentioned above, the imposed strain using the designed anvils is not high enough to reach the steady state in Cu.

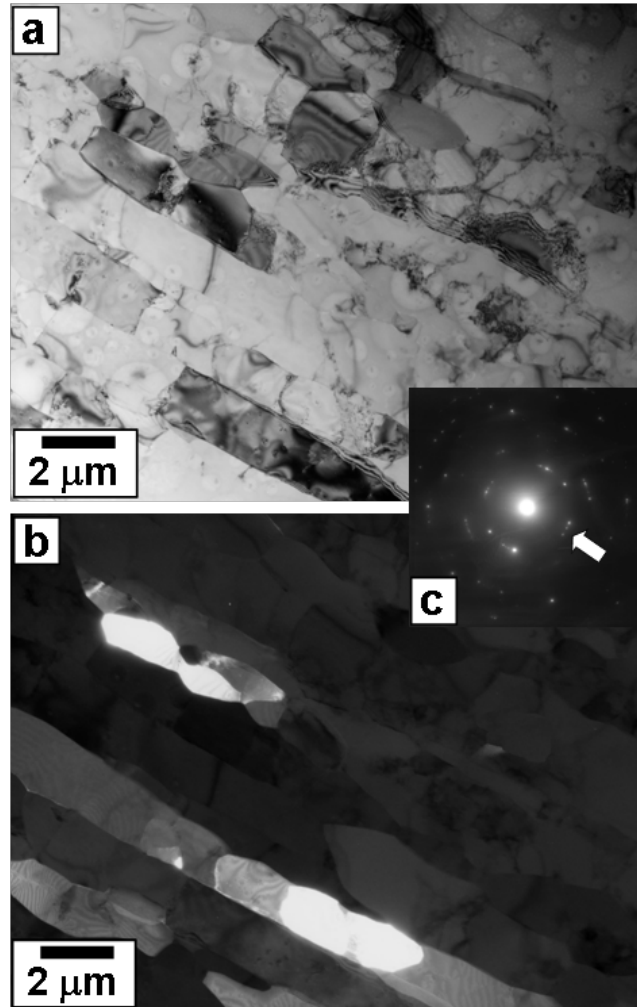


Figure 5. TEM (a) bright-field and (b) dark-field micrographs including (c) SAED pattern of Al after processing with CHPT.

In summary, the method of CHPT can process long sizes of the wires in short time (100 mm per 1 minute). Although there are over 20 continuous SPD techniques for producing wires [36], the CHPT is only the method that is feasible to reach a steady level of hardness and minimum grain size just after one pass through the anvils. However, the diameter of groove on the surface of anvils should be increased for processing wires of harder metals such as Cu. It is noted that both imposed strain and processing speed are increased with an increase in the ring diameter.

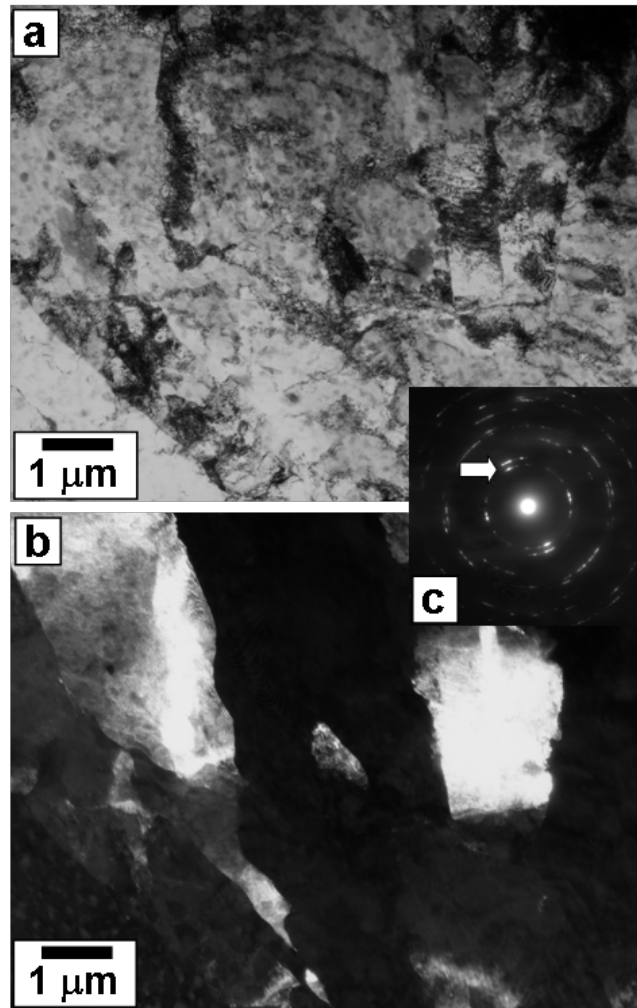


Figure 6. TEM (a) bright-field and (b) dark-field micrographs including (c) SAED pattern of Cu after processing with CHPT.

Summary and conclusions

Continuous high-pressure torsion (CHPT) using wires was demonstrated for microstructural refinement in metallic materials. Wires of high purity Al and Cu with 2mm diameter and 100mm length were successfully processed by continuous-torsional straining under high pressure using CHPT. The hardness and grain size reached a steady-state level for Al, but the imposed equivalent strain (13 ± 4) was not high enough to reach the steady state for Cu.

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