Comparison of microstructures and mechanical properties of a Cu–Ag alloy processed using different severe plastic deformation modes


A Cu–8 wt.% Ag alloy was processed by equal-channel angular pressing (ECAP), dynamic plastic deformation (DPD) and high-pressure torsion (HPT) at room temperature, and the microstructures and mechanical properties were investigated. It is shown that the microstructures of the Cu–Ag alloy can be refined to different levels by these severe plastic deformation (SPD) modes with the smallest grains produced by HPT. The results provide a clear demonstration that the yield strength and microhardness are inter-related over a wide range of values.

1. Introduction

High-strength, high-conductivity materials are needed for the construction of high-field magnets [1]. Over the last 40 years, several Cu–X (Ag, Nb, Cr, Fe) in situ composites, having fibrous microstructures reduced to the nanoscale, have been developed by conventional cold rolling or cold drawing, and the strength of these materials often exceeds that estimated on the basis of the rule of mixtures [2–5]. Of these various two-phase alloys, the Cu–Ag alloy has attracted considerable attention due to its superior strength and good conductivity [1,6,7].

In addition to conventional cold-working techniques, the application of severe plastic deformation (SPD) to coarse-grained metals has now become accepted as a valuable procedure for the fabrication of bulk solids having submicrometer or nanometer grain sizes [8,9]. Several SPD techniques are available [10] and significant interest has centered on high-pressure torsion (HPT) [11] and equal-channel angular pressing (ECAP) [12,13]. In addition, a dynamic plastic deformation (DPD) technique was developed recently and this procedure has been applied to various materials [14,15]. Although Cu–X (Cr, Ag) alloys have been processed by ECAP and HPT, there are to date only limited reports of the mechanical properties of these materials [16,17]. A very recent investigation of the microstructure and mechanical properties of a Cu–8 wt.% Ag alloy showed that the ECAP route critically influences the tensile strength [18]; however, the tensile strength is much lower than those processed by conventional cold drawing techniques [7]. In an investigation of the microstructure and mechanical properties of iron produced by various deformation modes (ball milling, HPT, accumulative roll bonding and ECAP), Todaka et al. [19] proposed that the strain gradient plays an important role in strengthening and refining the material. However, it is anticipated that the strengthening mechanism for the two-phase Cu–Ag alloys may be different from those of single-phase alloys because the eutectic component contributes significantly to the strengthening [6]. Accordingly, the present study was undertaken with a Cu–8 wt.% Ag alloy to compare the three SPD modes of ECAP, DPD and HPT by evaluating the microstructural refinement and the consequent mechanical properties so that an optimum deformation mode can be obtained.

2. Experimental material and procedures

The Cu–8 wt.% Ag binary alloy was cast, hot forged at 450 °C and then annealed at 450 °C for 10 h to release the internal stresses...
and to facilitate processing by ECAP, DPD and HPT. This alloy is hypoeutectic and generally contains two components: a eutectic component of Cu and Ag phases and a proeutectic Cu dendrite embedded with Ag precipitates (see Fig. 1a). Detailed information on the microstructures and plastic deformation of Cu–Ag alloys is available [20–22].

Billets having diameters of 20 mm and lengths of 100 mm were processed by ECAP for 1–8 passes via route A where this is most effective in strengthening the alloy [18]. Cylinders 17.1 mm in diameter and 21.6 mm in thickness were machined for DPD processing and multiple impacts were applied to deform the sample to a strain of $\varepsilon = 1.8$. Detailed information on DPD processing is given elsewhere [14]. Disks with diameters of 10 mm and thicknesses of 0.75–0.80 mm were processed by HPT under quasi-constrained conditions [11] using the experimental facility and procedure described earlier [23]. The disks were processed through 5 revolutions under an imposed pressure of 6.0 GPa. All processing was conducted at room temperature to exclude any thermal effects. Following ECAP, DPD and HPT, detailed microstructural observations were undertaken using a LEO SUPRA 35 scanning electron microscope (SEM) and an FEI Tecnai F20 transmission electron microscope (TEM). The microstructure was investigated on the $Y$ plane of the ECAP specimen and the transverse section of the DPD and HPT specimens. The mechanical properties were determined using tensile specimens with nominal gauge dimensions of 0.8 mm $\times$ 1 mm $\times$ 2 mm and an Instron 8871 testing machine. Since there is a shear strain gradient within the disks processed by HPT, the Vickers microhardness and tensile properties were investigated at a distance of about 2.5 mm from the center.

3. Experimental results

Fig. 1a shows the microstructure of the as-received alloy. The white region is the eutectic component and the dark region is the dendritic Cu component embedded with Ag precipitates as indicated by the arrows. Most of the eutectic components are elongated due to the hot-forging treatment. When the alloy was processed by ECAP for 8 passes, a fibrous eutectic was found on the $Y$ plane of

Fig. 1. SEM micrographs taken from (a) the transverse section of an as-received specimen, (b) the $Y$ plane of an ECAP sample processed through 8 passes, (c) the transverse section of a DPD specimen and (d) the transverse section of an HPT specimen at a distance of 2.5 mm from the center after processing through 5 revolutions.

Fig. 2. Eutectic spacing and the transverse size of the eutectic component corresponding to the microstructures shown in Fig. 1(a)–(d): the error bars denote the calculated standard deviations.
Fig. 3. TEM micrographs and statistical grain size distributions taken from (a and b) Y plane of the ECAP sample (8 passes); (c and d) the transverse section of the DPD specimen (ε = 1.8); (e and f) the transverse section of the HPT specimen at a distance of ~3.5 mm from the center (5 revolutions): note that $d_w$ is the mean width of the banded grains.

the specimens as shown in Fig. 1b. Similarly, a fibrous eutectic was observed on the transverse sections of specimens processed by DPD as shown in Fig. 1c. By contrast, observations on the transverse sections of HPT disks revealed a much finer fibrous eutectic as shown in Fig. 1d: the latter result is consistent with an earlier report on HPT [24].

For Cu–Ag alloys produced by cold drawing, it is known that the strength increases with decreasing eutectic spacing (transverse size of the Cu phase) [7]. Accordingly, the eutectic spacing and the transverse size of the eutectic components were measured and the results are depicted in Fig. 2 corresponding to the different processing conditions. By comparison with the as-received condition, the eutectic spacing decreased to about 9 µm after ECAP and about 5 µm after DPD where Fig. 1b and c shows the similar microstructures by the two SPD modes. After HPT the eutectic spacing decreases to about 650 nm and the transverse size of the eutectic component decreases to about 85 nm. This is much smaller than those processed by ECAP and DPD.

Fig. 3 shows TEM micrographs and the statistical grain sizes of the specimens processed by these different SPD modes. It is found that both the ECAP and DPD samples consist of banded structures as shown in Fig. 3a and c. The statistical transverse sizes of the banded grains were measured as ~109 and ~114 nm, respectively, as shown in Fig. 3b and d. In addition, high-density dislocations were observed within the grains and the ECAP samples contained deformation twins as reported earlier for pure Cu [25]. Deformation twins were also present in the deformed microstructures after DPD but with a very low volume fraction: this is consistent with pure Cu processed by DPD at room temperature [14]. For the sample processed by HPT, both banded structure and equiaxed grains were found with an average size of about 40 nm as shown in Fig. 3e.
These observations demonstrate that the microstructures of the Cu–Ag alloy are refined to different levels depending upon the processing operation. Since the tensile strengths of the Cu–Ag alloys increase with decreasing eutectic spacing [7], it is important to evaluate the differences in the mechanical properties arising from these different SPD modes. Fig. 4a shows the tensile engineering stress–strain curves of the as-received material and specimens processed through the different SPD modes. For the as-received Cu–Ag alloy after the heat treatment, the tensile strength is about 330 MPa which is a consequence of the low-temperature heat treatment so that the material does not recrystallize. For the ECAP samples, the tensile strength increases with the number of passes and approaches about 720 MPa after 8 passes. In order to produce Cu–Ag alloys with high strength, it appears that ECAP followed by drawing may be better than ECAP or drawing only [26]. By contrast, the tensile strength of the sample deformed to a strain of 1.8 by DPD is slightly lower. For the alloy processed by HPT, the tensile strength increases to about 1.1 GPa where this high value is due to the much smaller grain size achieved by HPT processing. It should be noted that, because of the limitation of the gauge lengths, all tensile engineering strains were obtained by measuring the displacements of the crosshead of the testing machine so that there are higher elastic strains and the individual elongations are not directly comparable.

Generally, the yield strength and the hardness are related through the expression [27]:

\[ \sigma_y = \frac{H v}{3} \]  

(1)

where \( H v \) is the hardness and \( \sigma_y \) is the yield strength, so that the yield strengths may be converted directly from the hardness values [28–30]. In this study, the yield strength and Vickers hardness are plotted in Fig. 4b for the Cu–Ag alloy processed by the different SPD modes and the broken line corresponds to Eq. (1). It is apparent that all datum points fall very close to the line, thereby confirming that the theoretical relationship in Eq. (1) applies to the Cu–Ag alloy under all processing conditions.

4. Discussion

During conventional cold drawing, Hong and Hill [6] proposed a modified rule of mixtures to predict the strength of Cu–Ag alloys where the first term incorporated the contribution of the eutectic component and the second term for the dendritic component contained the strengthening effect due to alloying, dislocations and grain size refinement. In the present study, it is necessary to consider the two components of the Cu matrix and the eutectic so that the strength of the Cu–Ag alloy is calculated from

\[ \sigma_{\text{Cu–Ag}} = (1 - f_E) \sigma_M + f_E \sigma_E \]  

(2)

where \( f_E \) is the volume fraction of the eutectic component and \( \sigma_M \) and \( \sigma_E \) are the strengths of the eutectic and the Cu matrix, respectively. When the shear strain is small, the microstructure is coarse and the strengths of both components can be calculated from Hall–Petch relationships of the form

\[ \sigma_M = \sigma_M^0 + k_1 r_{\text{Cu}}^{-1/2} \]  

(3)

and

\[ \sigma_E = \sigma_E^0 + k_2 r_e^{-1/2} \]  

(4)

where \( \sigma_M^0 \) and \( \sigma_E^0 \) are the intrinsic friction stresses of the eutectic and the Cu matrix, respectively. \( r_{\text{Cu}} \) is the band width of the Cu matrix, \( r_e \) is the spacing between the Cu or Ag phases inside the eutectic, and \( k_1 \) and \( k_2 \) are two strengthening coefficients for the Cu–Ag alloy. It follows from Eqs. (3) and (4) that any deformation which induces a refinement of the eutectic and the matrix will thereby enhance the strength of the two-phase alloy.

The observations of the microstructures in Figs. 1–3 and the corresponding mechanical properties in Fig. 4 show that there are significant differences when the alloy is subjected to different SPD modes. During ECAP the deformation route is very important in strengthening the two-phase Cu–Ag alloy [18]. Route BC, which is generally the most effective in refining metals [31], produces a Cu–8 wt.% Ag alloy with a saturation strength after ECAP for 3 passes whereas there is no evidence for a saturation strength when processing up to 4 passes by route A [18]. In the present study, there was no saturation in tensile strength and the eutectic spacing remained relatively large even for the alloy processed through 8 passes. It should be noted that a Cu–7.5 wt.% Ag alloy processed by conventional cold-drawing had a tensile strength approaching 1 GPa and a eutectic spacing in the submicrometer range [7] which is smaller than in the sample produced by ECAP.

During DPD, the microstructure of the Cu–Ag alloy is also dominated by dislocation activity which is consistent with results for pure Cu at room temperature [14]. It was reported that the microstructure and mechanical properties of pure Cu are greatly affected by the processing temperature [14] such that when Cu is processed by DPD at room temperature (RT-DPD) the tensile strength is comparable to processing by ECAP (~450 MPa) whereas for DPD at liquid nitrogen temperature (LNT-DPD) the tensile strength is much higher (~630 MPa). This difference originates primarily from the high volume fraction of deformation twin bundles and the small grain size. In the present study, the deformation strain was only 1.8 so that the tensile strength was relatively low. It is rea-
reasonable to anticipate that the tensile strength will be improved if the Cu–Ag alloy is processed by DPD at liquid nitrogen temperature.

During HPT processing, an extremely high shear strain is imposed and the eutectic component of the Cu–Ag alloy was also elongated to become fibrous. Fig. 5a shows high-magnification images taken from a transverse section of the HPT sample. It is found that the eutectic component, composed of Cu and Ag phases, is severely refined to the nanometer scale. A banded structure was also observed in the Cu matrix, as indicated by the arrows, with the band width ranging from ∼20 to ∼50nm where this is significantly smaller than in pure Cu. In pure Cu the recovery and recrystallization processes lead to a constant grain size and saturation of strength. However, in the composites the continual deformation–recovery–recrystallization cycle permits further microstructural refinement in the Cu matrix [3]. Small domains of several nanometers were found inside the eutectic, as shown in Fig. 5b where it was impossible to distinguish the Cu and Ag phases. Accordingly, it is reasonable to explain the high tensile strength of the HPT-processed alloy because it follows directly from Eqs. (2) to (4). Detailed information on the microstructural evolution process of a two-phase Cu–28 wt.% Ag alloy processed by HPT is given elsewhere [32].

It should be noted that, although some deformation twins appeared, the refinement of the microstructure in HPT was dominated by dislocation activities. Furthermore, the band width, the eutectic spacing and the transverse size of the eutectic were all much smaller than when processing by ECAP and DPD. Although it was proposed that strain gradients may play an important role in strengthening and refining single-phase materials [19], the two-phase Cu–Ag alloy in the present study is different and instead it is necessary to consider the strengthening effects of both the Cu matrix and the eutectic component via the different SPD processing modes.

5. Conclusions

In summary, experiments on a two-phase Cu–Ag alloy demonstrate the effect of different SPD modes on the microstructure and mechanical properties. Large billets 20mm in diameter were processed by ECAP for 8 passes giving banded grains and a tensile strength of 720 MPa. Processing by DPD at room temperature (RT-DPD) led to a microstructure and mechanical properties similar to those processing by ECAP for 8 passes. Deforming the alloy to a higher strain by DPD at liquid nitrogen temperature (LNT-DPD) may lead to a significantly higher tensile strength. When the Cu–Ag alloy is processed by HPT for 5 revolutions, a banded structure is observed on the transverse section and the tensile strength increases to about 1.1 GPa. Among these various SPD modes, any difference in the tensile strength is dominated primarily by the internal band width of the Cu matrix and the refinement of the eutectic. It is shown that the theoretical relation between yield strength and hardness applies to all of the samples of the Cu–Ag alloy although they were processed using different SPD modes.

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