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ScienceDirect
Scripta Materialia 63 (2010) 65–68
Scripta MATERIALIA
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Direct observations of microstructural evolution in a two-phase Cu–Ag alloy processed by high-pressure torsion

Y.Z. Tian,[a] X.H. An,[a] S.D. Wu,[a] Z.F. Zhang,[a,*] R.B. Figueiredo,[b] N. Gao[b] and T.G. Langdon[b,c]

[a] *Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, China*
[b] *Materials Research Group, School of Engineering Sciences, University of Southampton, Southampton SO17 1BJ, UK*
[c] *Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, CA 90089-1453, USA*

Received 9 January 2010; revised 27 February 2010; accepted 3 March 2010
Available online 6 March 2010

Disks of a coarse-grained Cu–28 wt.% Ag alloy were processed by high-pressure torsion, and the decorative eutectic regions were used to reveal the evolution of deformation. It is shown that the deformation starts at the peripheries of the disks in the form of local vortices and then spreads inwards with increasing the number of revolutions. The experimental evidence confirms that the center of the disk also experiences a strain after five revolutions.

Keywords: Cu–Ag alloy; Deformation processes; High-pressure torsion; Homogenization

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 with submicrometer or nanometer grain sizes [1,2]. Several SPD techniques are available [3] and significant interest has centered on high-pressure torsion (HPT) [4], equal-channel angular pressing (ECAP) [5,6], and dynamic plastic deformation (DPD) [7,8]. Of these various techniques, HPT is especially attractive because it is easy to conduct, it has the ability to impose exceptionally high strains, and it generally produces extremely small grain sizes.

There are several studies where microhardness measurements have been used to evaluate the extent of any microstructural evolution occurring in HPT [9–13]. Although these hardness measurements are generally taken after whole numbers of revolutions in torsional straining, the approach was recently extended to include the earliest stages of processing with fractional numbers of turns [14]. All these experiments are mutually consistent, and they show that there is a gradual evolution towards a homogeneous distribution of microhardness values with increasing pressure and/or increasing strain. Furthermore, since experiments on samples processed by ECAP have shown good correlation between microhardness measurements and microstructural observations undertaken using transmission electron microscopy [15], it is reasonable to anticipate the internal microstructure also evolves towards homogeneity when processing by HPT.

An evolution towards homogeneity in HPT has been predicted by making use of strain gradient plasticity modeling [16]. Nevertheless, this result is not consistent with the basic principles of HPT, where the shear strain γ is given by the relationship [17,18]:

$$\gamma = \frac{2\pi N r}{h} \tag{1}$$

where r and h are the radius and height (or thickness) of the disk, respectively, and N is the number of revolutions. It follows from Eq. (1) that the strain is zero at the center of the disk and, in principle at least, the microstructure across the disk should remain inhomogeneous. There is a suggestion that reports of microhardness homogeneity in HPT disks may arise because of a misalignment of the axes of the anvils or other deviations from the idealized HPT conditions [19].

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[*] Corresponding author. Tel.: +86 24 23971043; e-mail: zhfzhang@imr.ac.cn

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doi:10.1016/j.scriptamat.2010.03.014

This apparent dichotomy suggests that more information is critically needed concerning the nature of the flow process occurring in HPT. Two recent reports provide information on the shearing process in HPT. First, experiments on a two-phase duplex stainless steel revealed the development of swirls and vortices during HPT processing, where the appearance of these irregularities have similarities to the well-established Kelvin–Helmholtz instability observed in fluid flow when there are significant velocity variations within the liquid [20]. Second, the processing of a two-phase Zn–Al eutectoid alloy showed the occurrence of agglomeration and banding around the peripheries of the disks during the initial stages of HPT [21]. Accordingly, the present investigation was initiated to provide further information on microstructural evolution and the extent of any homogenizing process in HPT. A coarse-grained Cu–28 wt.% Ag alloy was selected for this study because this alloy contains two ductile phases and the morphological changes of the decorative eutectic region may be conveniently traced when processing by HPT.

The Cu–28 wt.% Ag binary alloy was fabricated from 99.999% purity Cu and 99.99% purity electrolytic Ag using the procedure described earlier [22]. The alloy is hypoeutectic and generally contains two components: eutectic component composed of Cu and Ag phases and proeutectic Cu dendrite embedded with Ag precipitates (see Fig. 2a). The Cu and Ag phases always have the cube-on-cube orientation in the proeutectic component, but they have the orientation relationship only in selective eutectic areas [23]. In addition, the dendrite and the eutectic component may also have the orientation relationship [22,24]. Detailed information on the microstructures and plastic deformation of this alloy and a Cu–16 wt.% Ag alloy is now available [22,24]. Disks were prepared with diameters of 10 mm and thickness of 0.75–0.80 mm, and they were processed by HPT at room temperature under quasi-constrained conditions [4] using the experimental facility and procedure described earlier [25], except that a lubricant was not placed around the peripheries of the depressions on the upper and lower anvils. The disks were processed through one, three, and five revolutions under an imposed pressure of 6.0 GPa. Following HPT, detailed microstructural scanning electron microscopy (SEM) was undertaken at the centers and at selected positions on the disks using a LEO SUPRA 35 instrument. Images were also recorded by SEM (quadrant backscattering detector, QBSD) to reveal the macroscopic deformation morphologies of the eutectic regions throughout the disks.

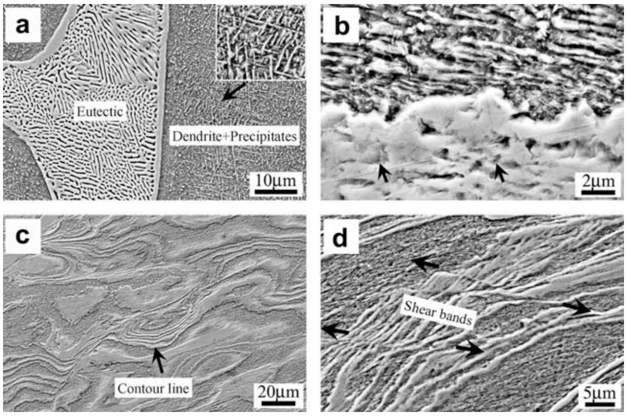

Figure 2. Microstructures in the disk at different radii after one revolution: (a) center, (b) 1.25 mm, and (c and d) 4.5 mm.

Figure 1a–c shows QBSD images of the surface deformation morphologies of disks strained through one, three and five revolutions, respectively. It is apparent from Figure 1a that the eutectic region retains a coarse morphology in the central area of the disk after one revolution, but there is some refining of the eutectic in the periphery regions. After three revolutions, the eutectic region is coarse only within a central region with a diameter of ∼2.5 mm, and much of the outer region of the disk contains a refined eutectic which lies in arcs delineating the torsional rotation. After five revolutions, the eutectic morphology in Figure 1c clearly divides into two well-defined areas with a central region of coarse eutectic with a diameter of ∼1 mm and a refined eutectic occupying the remainder of the disk. These macroscopic observations support the occurrence of a homogenizing process which is initiated at the outer edge of the disk and then proceeds inwards with increasing numbers of revolutions.

Figure 2 shows high magnification microstructures recorded by SEM at different positions in the disk after one revolution. In the central region, the Cu and Ag phases in the eutectic retain their as-cast morphologies, and the interface between the eutectic and the dendrite remains straight. In addition, it is apparent there is a zone of depleted Ag near the interface and, as shown by the inset in Figure 2a, the precipitates in the dendrite are distributed regularly. All these observations are similar to those reported earlier for the as-cast condition [24], and it is reasonable to infer that the central region has experienced essentially little or no plastic deformation during the first revolution. Since the shear strain

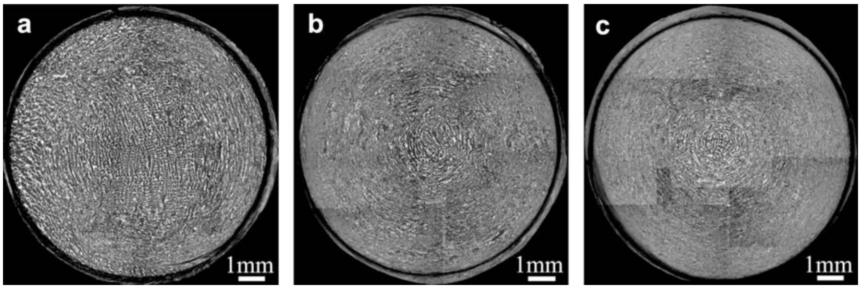

Figure 1. QBSD images showing the HPT disks after straining through (a) one revolution, (b) three revolutions, and (c) five revolutions.

varies linearly with the radius as shown in Eq. (1), it is reasonable to anticipate that there will be evidence for straining at positions removed from the center. This is confirmed in Figure 2b at a radius of ~1.25 mm where, although the eutectic region remains coarse, the Cu and Ag phases within the eutectic are severely distorted, and the interface between the eutectic and the dendrite has become significantly serrated. It is also evident that the zone of depleted Ag, visible in Figure 2a, is now absent. At a radius of ~4.5 mm, some of the eutectic region is refined into a fibrous morphology with evidence for local vortices delineated by contour lines as shown in Figure 2c. This is similar in appearance to the vortex areas reported in a duplex stainless steel processed by HPT, which was interpreted as incorporating larger shear strains than the surrounding areas [20]. There is also some evidence for the development of shear bands, as shown in Figure 2d, although much of the eutectic region continues to exhibit a coarse morphology after one revolution.

When the disk is further strained to three revolutions, the surface morphologies become different, as shown in Figure 3. Careful inspection near the center reveals evidence for some limited plastic deformation, as indicated by the arrows in Figure 3a. However, the vortices and contour lines appear more frequently, and they lie closer to the center of the disk by comparison with the disk processed through one revolution. For this disk, the fibrous eutectic regions and the vortices begin to appear at a radius of ~1 mm, they are well defined at a radius of ~2.5 mm, as shown in Figure 3b, and the fibrous region becomes finer and denser at a radius of ~4.5 mm, as shown in Figure 3c: this latter effect is reasonable because of the larger anticipated strain in the outer region of the disk. Figure 3d shows an enlarged image of the fibrous eutectic at a radius of ~4.5 mm, with a shear band passing through several eutectic regions. By contrast to the morphology after one revolution, the shear bands appear more frequently after three revolutions. In addition, the Cu phase within the eutectic is hardly distinguishable, as indicated by the upper arrow in Figure 3d.

After five revolutions, the microstructure becomes more homogenous throughout the disk, as shown in Figure 4. Inspection of Figure 4a shows that the Cu phase in the eutectic is severely distorted, as indicated by the two arrows on the left, the interface between the eutectic and the dendrite is serrated, as indicated by the arrow on the right, and the precipitates are no longer distributed regularly, thereby confirming that the center of the disk has experienced some plastic deformation. At a radius of ~0.5 mm in Figure 4b, the Cu and Ag phases within the eutectic are severely distorted: examples are indicated by the two arrows. There is also evidence at a radius of ~0.5 mm for the onset of some regions with vortices and contour lines. Figure 4c shows a clearly defined vortex and contour lines at a radius of ~1.25 mm where a major change is apparent from the image taken in the same position after one revolution in Figure 2b. Finally, the eutectic region is very significantly refined in the outer region of the disk at ~4.5 mm, as shown in Figure 4d, where most of the area is filled with fibrous eutectic, but some isolated areas of eutectic with a coarse morphology remain , as indicated and enclosed by the dotted lines. There is also significant evidence for the presence of shear bands in this condition.

The results from these observations of the morphologies of the decorative eutectic regions show that there is a gradual evolution with increasing numbers of revolutions in processing by HPT. Specifically, there is a localization of plastic deformation in the early stages of straining with vortices and contour lines formed at the periphery of the disk, and this straining spreads inwards with increasing numbers of revolutions, so that ultimately there is clear and direct evidence for the occurrence of plastic straining in the center of the disk. Based on these observations, it is reasonable to anticipate the development of an essentially homogeneous microstructure throughout the disk after a sufficient number of revolutions.

The preceding microstructural observations support the conclusions, based on microhardness measurements, that there is a gradual evolution in HPT towards a homogeneous microstructure. When the number of revolutions is low in the early stages of processing, the higher shear strain in the peripheral region leads to preferential deformation, and the development of vortices and contour lines associated with local variations in the shear strain: these vortices are similar in appearance

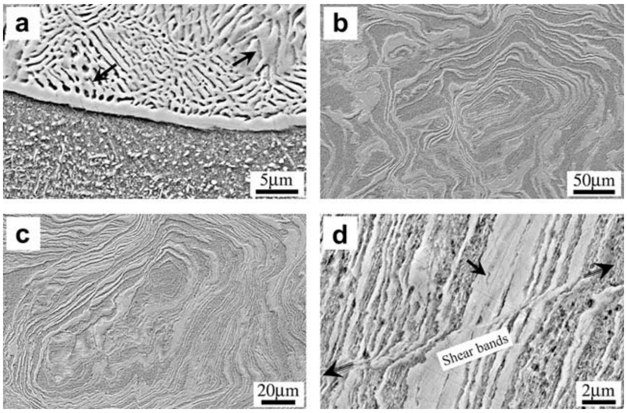

Figure 3. Microstructures in the disk at different radii after three revolutions: (a) center, (b) 2.5 mm, and (c and d) 4.5 mm.

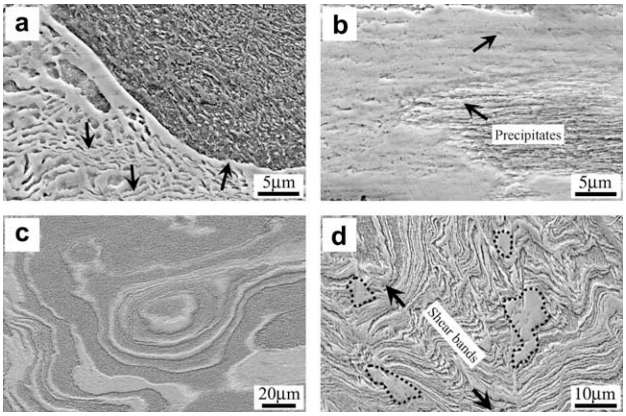

Figure 4. Microstructures in the disk at different radii after five revolutions: (a) center, (b) 0.5 mm, (c) 1.25 mm, and (d) 4.5 mm.

to those reported earlier for a duplex stainless steel processed by HPT [20]. Increasing the numbers of revolutions extends these vortices inwards towards the center of the disk, so that they are visible only outside an inner radius of ~1 mm after three revolutions and ~0.5 mm after five revolutions. In practice, plastic deformation will transfer with increasing strain from the region containing contour lines to an adjacent region which is less hardened, and this leads to new contour lines and the refining of the eutectic region to a fibrous condition. This evolutionary process is similar to a homogenizing mechanism proposed earlier based on conclusions derived only from microhardness measurements [9].

It should be noted that the conventional relationship given in Eq. (1) predicts an absence of any strain at the center of the HPT disk, and yet the present observations and Figure 4a demonstrate the occurrence of some limited plastic strain after rotation through five revolutions. Since the magnitude of the shear strain scales with the radial position, there will be a strain gradient throughout the disk, and geometrically necessary dislocations are therefore required to accommodate the inhomogeneous shear strain along the disk radius [26]. It has been suggested that the occurrence of hardening at the center of the disk may be associated with this strain gradient [27]. In addition, it will be assisted by the inhomogeneous nature of the deformation process.

In addition to work on the microstructural evolution, there is also a body of research showing the inter-diffusion of elements in two-phase materials during HPT processing, even if the phases are immiscible [28,29]. For Cu–Ag alloy, mechanical alloying has occurred during conventional cold drawing [30]. Meanwhile, Ohsaki et al. [31] had observed substantial mixing of Cu and Ag within the shear band. In the present study, it is proposed that mechanical alloying should also play an important role in the microstructural evolution of Cu–Ag alloy during HPT processing, since intense shear strain was applied.

In summary, experiments on a two-phase Cu–Ag alloy demonstrate an evolution in the microstructure during processing by HPT which matches that generally inferred indirectly from microhardness measurements. Straining occurs initially at the periphery of the disk and, due to local variations in the shear strain, this leads to the development of vortices and clearly defined contour lines. This deformation spreads inwards with increasing strain so that it becomes visible to within ~0.5 mm of the center of the disk after five revolutions of torsional straining. There is clear evidence for deformation in the center of the disk after five revolutions, and this is attributed to the presence of a strain gradient across the disk and the inhomogeneous nature of the deformation process.

The authors are grateful to Ms. W. Gao for her help with SEM observations. This work was supported by the National Natural Science Foundation of China (NSFC) under Grant Nos. 50890173 and 50931005, the National Outstanding Young Scientist Foundation of China under Grant No. 50625103 and the Royal Society of the UK under International Joint Project No. JP871294.

[1] Y.T. Zhu, T.C. Lowe, T.G. Langdon, Scripta Mater. 51 (2004) 825.
[2] M.A. Meyers, A. Mishra, D.J. Benson, Prog. Mater. Sci. 51 (2006) 427.
[3] R.Z. Valiev, Y. Estrin, Z. Horita, T.G. Langdon, M.J. Zehetbauer, Y.T. Zhu, JOM 58 (4) (2006) 33.
[4] A.P. Zhilyaev, T.G. Langdon, Prog. Mater. Sci. 53 (2008) 893.
[5] M. Furukawa, Z. Horita, M. Nemoto, T.G. Langdon, J. Mater. Sci. 36 (2001) 2835.
[6] R.Z. Valiev, T.G. Langdon, Prog. Mater. Sci. 51 (2006) 881.
[7] Y.S. Li, N.R. Tao, K. Lu, Acta Mater. 56 (2008) 230.
[8] Y. Zhang, N.R. Tao, K. Lu, Acta Mater. 56 (2008) 2429.
[9] A.P. Zhilyaev, G.V. Nurislamova, B.-K. Kim, M.D. Baró, J.A. Szpunar, T.G. Langdon, Acta Mater. 51 (2003) 753.
[10] C. Xu, Z. Horita, T.G. Langdon, Acta Mater. 55 (2007) 203.
[11] A.P. Zhilyaev, T.R. McNelley, T.G. Langdon, J. Mater. Sci. 42 (2007) 1517.
[12] C. Xu, Z. Horita, T.G. Langdon, Acta Mater. 56 (2008) 5168.
[13] C. Xu, T.G. Langdon, Mater. Sci. Eng. A 503 (2009) 71.
[14] C. Xu, Z. Horita, T.G. Langdon, Mater. Trans. 51 (2010) 2.
[15] C. Xu, M. Furukawa, Z. Horita, T.G. Langdon, Mater. Sci. Eng. A 398 (2005) 66.
[16] Y. Estrin, A. Molotnikov, C.H.J. Davies, R. Lapovok, J. Mech. Phys. Solids 56 (2008) 1186.
[17] R.Z. Valiev, Yu.V. Ivanisenko, E.F. Rauch, B. Baudelet, Acta Mater. 44 (1996) 4705.
[18] F. Wetscher, A. Vorhauer, R. Stock, R. Pippan, Mater. Sci. Eng. A 387–389 (2004) 809.
[19] A. Vorhauer, R. Pippan, Scripta Mater. 51 (2004) 921.
[20] Y. Cao, Y.B. Wang, S.N. Alhajeri, X.Z. Liao, W.L. Zheng, S.P. Ringer, T.G. Langdon, Y.T. Zhu, J. Mater. Sci. 45 (2010) 765.
[21] M. Kawasaki, B. Ahn, T.G. Langdon, Acta Mater. 58 (2010) 919.
[22] Y.Z. Tian, Z.F. Zhang, Z.G. Wang, Philos. Mag. 89 (2009) 1715.
[23] K. Han, A.A. Vasquez, Y. Xin, P.N. Kalu, Acta Mater. 51 (2003) 767.
[24] Y.Z. Tian, Z.F. Zhang, Mater. Sci. Eng. A 508 (2009) 209.
[25] M. Kawasaki, T.G. Langdon, Mater. Sci. Eng. A 498 (2008) 341.
[26] M.F. Ashby, Philos. Mag. 21 (1970) 399.
[27] Y. Todaka, M. Umemoto, J. Yin, Z. Liu, K. Tsuchiya, Mater. Sci. Eng. A 462 (2007) 264.
[28] X. Sauvage, P. Jessner, F. Vurpillot, R. Pippan, Scripta Mater. 58 (2008) 1125.
[29] X. Sauvage, R. Pippan, Mater. Sci. Eng. A 410–411 (2005) 345.
[30] D. Raabe, S. Ohsaki, K. Hono, Acta Mater. 57 (2009) 5254.
[31] S. Ohsaki, S. Kato, N. Tsuji, T. Ohkubo, K. Hono, Acta Mater. 55 (2007) 2885.