MICROSTRUCTURAL EVOLUTION OF CU-AG IN-SITU COMPOSITES PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING (ECAP)

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Keywords: ECAP, Cu-Ag, Microstructure, mechanical properties

1 Introduction
Equal-channel angular pressing (ECAP) technique has been proven to be very useful in improving strength of ingot-processed metallic alloys and composites through grain refinement to, typically, the submicrometer level [1-5]. Although ECAP processing has been employed extensively to many metals and alloys, application of ECAP to in situ composites is quite limited. Recently, Cho and Hong [6] and Tian et al. [7] studied the effects of processing routes on the microstructure and mechanical properties during ECAP of Cu-Ag in-situ composites. Since the microstructure Cu-Ag in-situ composites consist of two phases, the microstructural refining by severe plastic deformation is more effective than pure metals because the dislocation annihilation is limited by the presence of second phase. Furthermore, the second phase in Cu-Ag in-situ composite is ductile and deformed to accommodate the imposed strain during deformation processing, the redistribution of Ag phase renders the higher strength to Cu-Ag in-situ composites.

The microstructural development and strengthening mechanism of deformation processed Cu in-situ composites have been the subjects of extensive studies [1-8]. Deformation processing such as drawing or rolling has been employed to refine the microstructure, which leads a fine two-phase microstructure with strong crystallographic textures [1-8]. The strength of severely deformed Cu base composites exceeds that predicted by the rule of mixtures (ROM), and a fundamental understanding of the strengthening mechanisms has been the subject of much discussion [1–8]. One advantage of ECAP processing compared to drawing is that the size of the heavily processed composite is not reduced by ECAP whereas it decreases rapidly with drawing. Recently, Cho and Hong suggested that the strengthening mechanism of ECAPed Cu-Ag in-situ composites is dependent on the processing routes. In order to develop the strengthening model of Cu-Ag in-situ composites, the microstructural evolution during ECAP as functions of processing route and number of pressing should be understood. The objective of this study is to investigate the effect of the number of pressing and feeding methods on the microstructural evolution and stress-strain responses of Cu-Ag in-situ composites during ECAP.

2 Experimental
Billets of Cu–15 wt. % Ag were prepared by induction melting in vacuum. ECAP was carried out using a solid die made of SKD 61 with an internal angle of 90° between the vertical and horizontal channels. Repetitive pressing of the same rod was attempted by using A, C or Bc routes at room temperature. The sample was pressed without rotation between each pass in route A, rotated by 90 degree after each pass in route Bc, rotated by 180 degree after each pass in route C. Optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to examine the microstructural evolution as a function of the number of pass. Hardness was measured on the y plane with a load of 300 g using a Vickers microhardness tester. Tensile specimens with the stress axis parallel to the ECAP axis was machined and mechanical testing was performed in a tensile testing machine.

3. Results and Discussion
In Fig. 1, the microstructures of as-cast (a) and ECAPed Cu-Ag in-situ composite for 1 pass are shown. It is well established that as-cast structure of Cu–Ag in-situ composites consists of primary copper-rich α phase and eutectic lamella structure.
The as-cast structure of Cu-Ag in-situ composites can be modified greatly by the feeding methods of samples during ECAP because the morphology and the distribution of Ag lamella can be changed by the application of shear strain on Ag lamella and Cu matrix. After 1 pressing, Ag lamellae were shown to be elongated by shear strain (Fig. 1(b)).

Fig. 1. Microstructures of as-cast (a) and ECAPed Cu-Ag in-situ composite for 1 pass.

Fig. 2 displays the microstructural evolution of Cu-Ag composites ECAPed for 2(a), 3(b), 4 (c) and 8 (d) passes using route A. In Cu-Ag ECAPed by route A, deformation bands and elongated Ag lamellae developed with increasing number of passes and the angle of elongated lamellae on the y plane (with respect to the horizontal line) decreased and approached the horizontal line in route A as shown in Fig. 1 and 2, suggesting the accumulation of the shear strain toward the pressing direction. The Ag lamellae became more elongated and thinner and the spacing between Ag lamellae decreased as the number of passes increased in route A because of the accumulation of the shear strain along the same direction. Since the sample was pressed without rotation between each pass, the shear strain was accumulated along the same direction, resulting in the elongated filamentary structure. The second phase lamellae developed into elongated filaments in route A (Fig. 2(b)).

The microstructural evolution by the application of route C with Cu-Ag specimen ECAPed repetitively with a 180 degree rotation between each pass was found to be different from that of route A and no elongated Ag lamellae and filaments were observed. The Ag lamella network structure sheared and elongated after 1 pass (Fig. 1(b)) can be easily recovered in the next pressing although Ag lamella networks were severed appreciably by shearing in route C. Fig. 3 demonstrated that the network structure elongated after odd-numbered passes (1 and 3 passes) appeared to be recovered in the even-numbered passes (2, 4 and 8 passes). Since the slip by shearing is not completely reversible due to the statistically trapped dislocations and geometrically introduced dislocations in the presence of the second phase, Ag lamellae appeared to be gradually elongated and sheared. Nevertheless, the modification of the Ag lamellae structure by ECAP pressing in route C is least effective because of the reversible nature of the shearing in each pass.

Fig. 2. Microstructural evolution of Cu-Ag composites ECAPed for 2(a), 3(b), 4 (c) and 8 (d) passes using route A.

Fig. 3. Microstructural evolution of Cu-Ag composites ECAPed for 2(a), 3(b), 4 (c) and 8 (d) passes using route C.

Fig. 4 displays the microstructural evolution of Cu-Ag composites ECAPed (repetitively with a 90 degree rotation) for 2(a), 3(b), 4 (c) and 8 (d) passes using route Bc. In route Bc, the sheared plane in the first pass is sheared along the another plane which intersects the first sheared plane at 120° in the second pass. In the third pass, the first sheared plane in the first pass is recovered by shearing in the opposite direction to the first shear along the first
sheared planes. Likewise, the second sheared plane in the second pass is recovered by shearing in the opposite direction to the fourth shear along the second sheared plane. The same pattern continued every four passes. However, the recovery of the shear every other pass became more irreversible because of the shearing along the intersecting plane between two combinational set of passes with the parallel shearing planes. Therefore, the Ag lamellae network structure was broken into segmented and sheared Ag lamella and smaller particles by shearing along two intersecting planes with increasing number of pass. The microstructural evolution by the application of route C was found to be less effective in refining the original structure and no elongated filaments were observed. The shape of Ag network structure was maintained with a minor modification even after 8 passes since the shear strain after one pass can be easily recovered in the next pressing although dendritic networks were severed appreciably by shearing in route C.

The observation of the surface morphology using SEM revealed the more detailed microstructure in Cu-Ag composites. Fig. 5(a)-5(d) displayed the phase distribution in as cast Cu-Ag composites (a) and ECAPed Cu-Ag for 8 passes using A (b), C(c) and Bc (d) routes. In the as-cast microstructure (a), it is shown that small Ag particles are present in the primary copper-rich α-phase. It is interesting to note the Ag particles in copper-rich α-phase and Ag depleted zone [7] along the interface between copper-rich α-phase and the eutectic lamella, suggesting that the copper-rich α-phase is oversaturated with silver. In route A (b), Ag particles as well as eutectic lamella were observed to be elongated, in compatible with the accumulation of the shear strain toward the pressing direction as shown in Fig. 2. In route C (c) the modification of eutectic lamella structure was found to be less effective. The Ag-depleted zone was found to exist along the interface of eutectic lamella as indicated by arrows. In route Bc (d), the eutectic lamella was observed to be thinner than that of the as-cast structure by shearing and segmentation. The population of Ag particles was shown to be clearly increased by ECAP processing using route Bc. It is also interesting to note the disappearance of Ag depleted zone along the Ag lamella. The absence of Ag depleted zone and the increase of Ag particle population are thought to be caused by the frequent shearing and segmentation of eutectic lamella in route Bc. The comparison of the particle population after ECAP processing in route Bc (d) and C (c) clearly demonstrated that Ag particles were effectively generated by shearing and segmentation of Ag lamella in route Bc.

Fig. 6 displays the TEM structure of Cu-Ag in-situ composites ECAPed by A (a), Bc (b) and C (c) routes for 8 passes. Examination of TEM microstructure revealed that the dislocation tangles, walls and subgrain bands formed after 1 pass and the band structure became refined with increasing number of passes. The Ag filaments and subgrains in Cu matrix were found to be more elongated and the boundary became clearer with less dislocations between filament and/or subgrain walls in route A (a) possibly due to the increasing misorientations of
boundaries [9]. Ag filaments indicated by arrows in Fig. 6(a) was found to have the thickness of 3–5 nm. The width of Cu channel between Ag filaments and/or subgrains in Cu matrix (45 nm) is much smaller than the width of elongated dislocation cells and/or subgrain boundaries (500 nm) in heavily deformed pure Al or Cu [9,10], suggesting the equilibrium distance between dislocation walls and subgrains became much smaller in Cu-Ag because the recovery of heavily deformed dislocation structure was hindered in the presence of Ag filaments, Ag particles and Ag atoms. In route C, we observed the microstructure consists of elongated subgrains and Ag filaments (indicated by arrows), which appears similar to that of route A, but the width of Cu matrix between Ag filaments and/or subgrains in Cu matrix (70 nm) in route C (Fig. 6(b)) was larger than that (45 nm) obtained in route A (Fig. 6(a)). It is interesting to note that elongated subgrains between Ag filaments in route C are not well developed as in route A. This observation is in agreement with that of Yoon et al. [11] that the subgrain boundaries were not well developed and less clear in Cu-Zn ECAPed by route C. The less-well defined subgrain boundaries between Ag filaments in route C is thought to result from the activation of the primary dislocations and the lack of the secondary dislocations because same slip plane is activated in each pass [11, 12]. In route Bc, more equiaxed subgrains were observed as shown in Fig. 6(c). It appears that a rotation of 90 degree between pressings has led to the development of two intersecting subgrain bands [9,10,13]. In Cu-Ag ECAPed by route Bc, equi-axed subgrains and Ag particles (indicated by double arrows) as well as short Ag filaments and lamellae (indicated by an arrow) were observed. It seems that more Ag lamellae were fragmented by shearing than elongated along the shear direction because of the rotation of the sample by 90° between each pass. Fig. 7 exhibits the variations of the hardness against the number of passes for Cu-Ag in-situ composites. A steady increase of the hardness was observed up to 8 passes in route Bc whereas the hardness saturated after 4 passes in route C. The hardness of Cu-Ag processed by route A and route Bc are far greater than that of route C after 8 passes because of the finer elongated filamentary structure in route A and finer segmented structure in route Bc. The relatively low hardness in route C is also supported by no pronounced change of Ag lamellar network and less-well developed elongated boundaries after ECAP.

Fig.6. TEM microstructures of Cu-Ag composites ECAPed by A (a), C(b) and Bc (c) routes for 8 passes.

Fig.7. Variation of the hardness against the number of passes for Cu-Ag composites in routes A, C and Bc.

The electrical conductivity decreased with number of pressing, reflecting the refining of deformed structure. The electrical conductivity decreased more drastically in route Bc and route A than in route C. The lower conductivity in route A compared to that in route C can be
attributed to the narrower width between Cu filaments and/or sub-grain boundary width in route A. In route A, interface scattering is likely to increase with the number of ECAP passes because of the decreasing spacing between Ag filaments. The more drastic decrease of electrical conductivity in route Bc is thought to result from more effective deformation-induced dissolution and re-precipitation due to the frequent fragmentation of lamellae by severing and shearing of Ag lamellae in route Bc.

Fig. 8. Variation of the conductivity against the number of passes for Cu-Ag composites in routes A, Bc and C.

4. Conclusions

Equal channel angular pressing was carried out on Cu-15 wt. % Ag in-situ composites at room temperature and the effects of the number of passes and feeding methods on the microstructural evolution were studied. Following conclusions were made as a result of this study.

1. In Cu-Ag ECAPed by route A, the Ag lamellae became more elongated and thinner and the spacing between Ag lamellae decreased as the number of passes increased in route A because of the accumulation of the shear strain along the same direction. The second phase lamella structure developed into elongated filamentary structure.

2. In route C, Ag lamella network structure elongated after add-numbered passes (1 and 3 passes) was recovered in the even-numbered passes (2, 4 and 8 passes). The modification of the Ag lamellae structure by ECAP pressing in route is least effective because of the reversible nature of the shearing in each pass.

3. In route Bc, the sheared plane in the first pass is sheared along the another plane which intersects the first sheared plane at 120° in the second pass. The recovery of the shear every other pass became more irreversible because of the shearing along the intersecting plane between two combinational set of passes with the parallel shearing planes. Therefore, the Ag lamellae network structure was broken into segmented and sheared Ag lamella and smaller particles by shearing along two intersecting planes with increasing number of pass.

Acknowledgement

This work was supported by a grant from the Fundamental R & D Program for Core Technology for Materials (2010) funded by the Ministry of Knowledge Economy, Korea.

References


