JOURNAL OF MATERIALS SCIENCE 36 (2001) 5881 – 5884

# Comparison of microstructure and strength in wire-drawn and rolled Cu-9 Fe-1.2 Ag filamentary microcomposite

J. S. SONG, J. H. AHN, H. S. KIM, S. I. HONG
*Department of Metallurgical Engineering, Chungnam National University, Taedok Science Town, Taejon 305-764, Korea*
*E-mail: sihong@hanbat.chungnam.ackr*

Comparison of microstructure and strength of Cu-9 Fe-1.2 Ag microcomposite wires and sheets obtained by cold drawing or cold rolling combined with intermediate heat treatments has been made. The primary and secondary dendrite arms are aligned along the drawing or rolling direction and elongated into filaments after cold working. The microstructural scale of wire-drawn microcomposites was found to be finer than that of rolled microcomposites at the same drawing strain. The more effective microstructural refinement induced by unidirectional metallic flow and co-deformation of filament and Cu matrix resulted in finer microstructure in microcomposite wires. The ultimate tensile strength and the conductivity of wire-drawin Cu-Fe-Ag microcomposite were higher than those of rolled Cu-Fe-Ag microcomposites. The strength of Cu-Fe-Ag microcomposites is dependent on the spacing of the Fe filaments in accord with a Hall-Petch relationship. The good mechanical and electrical properties of wires may be associated with the more uniform distribution of fine filaments. The fracture surfaces of Cu-Fe-Ag wires and sheets showed ductile-type fracture and iron filaments were occasionally observed on the fracture surfaces. The fracture surface of Cu-Fe-Ag wires showed generally finer microstructural morphology than that of Cu-Fe-Ag sheets, consistent with the finer microstructural scale in Cu-Fe-Ag wires. © *2001 Kluwer Academic Publishers*

## 1. Introduction

Microcomposites fabricated by mechanical working of ductile two phase mixtures prepared by various techniques have been the subject of considerable research [1–6]. Tensile strengths greater than those predicted by a rule of mixtures are observed in heavily cold worked microcomposites. The tensile strengths of deformation processed composites have been shown to correlate with filament spacings, leading to a Hall-Petch type relationship [3]. Spitzig *et al*. [3] showed that the strength of heavily cold-worked Cu-Nb microcomposite wires increased as the spacing between filaments decreased. The particularly attractive feature of these microcomposites is the combination of high strength plus high electrical and thermal conductivity. Since the solid solubility of alloying element in Cu matrix for these microcomposites is quite small, the conductivity of the copper is not substantially reduced by the addition of alloying elements. It has been demonstrated that the similar mechanical properties were observed in other deformation processed microcomposites [7–9] such as Cu-Cr, Cu-Ta and Cu-Fe. The Cu-Fe system is of particular interest because of the relatively low cost of iron compared to the other possible insoluble b.c.c. elements [7–9]. In this study, the evolution of microstructures and structure-property relationships in wire-drawn and rolled Cu-Fe-Ag microcomposites were examined.

## 2. Experimental procedure

Billets of Cu-9 wt% Fe-1.2 wt% Ag were prepared by induction melting. Cylindrical billets were about 60 mm in diameter and about 112 mm in length. Extrusion of cylindrical billets was carried out at 500°C, reducing the billets from 60 mm diameter to 24 mm diameter. The extruded rods were then rod rolled to 6 mm in a series of steps and subsequently drawn into wires using successively smaller dies, to a minimum diameter of 2 mm with three intermediate heat treatments at 450°C. The cold drawing strain $\eta$ is 4.8 where $\eta = \ln\,(Ao/A)$ and $Ao$ and $A$ are original and final cross-sectional areas. Square billets were cold rolled to 0.5 mm thickness with three intermediate heat treatments. The cold deformation strain $\eta$ during rolling is 4.6 where $\eta = \ln\,(t_o/t)$ and $t_o$ and $t$ are original and final thickness. In rolling of thin sheets in which the spreading perpendicular to the rolling direction is inhibited by the friction between the rolls and the sheet, the effective strain is given as $\eta_e = (2/\sqrt{3})\eta$. In this study, however, rolling in the initial stage resulted in the spreading of the workpiece perpendicular to the rolling direction because of the relatively free lateral expansion during rolling due to the similar length of the workpiece in height and width. Indeed, the width of the rolled sheets increased by 40–50% and the effective rolling strain is reduced if the lateral spread of the sheets is considered. Therefore, the regular rolling strain $\eta$ can be directly compared with the $\eta$ for wire-drawing. The evaluation of mechanical properties of wires was carried out by tensile testing using a United machine equipped with an extensometer for accurate strain measurements. All tensile tests were performed at room temperature using a strain rate of $5.5 \times 10^{-4}$ $S^{-1}$. The evolution of the microstructure was examined by optical microscope and SEM (scanning electron microscope). The inter-filamentary widths were determined using an image analyzer (Leica Q5001W with Leica Qwin software). Fracture surfaces of the tensile specimens were examined in a SEM (JSM 5410) to characterize fracture behavior. Electrical resistivity measurements were made using a standard four-probe technique.

## 3. Results and discussion

Fig. 1 shows optical images of the morphology of the Fe dendrites in the as cast ingot. Fig. 2 shows the transverse (2a) and longitudinal (2b) sections of drawn Cu-Fe-Ag wires. The ribbon-like morphology of Fe filaments is clearly evident on the transverse sections (Fig. 2a). On the longitudinal sections (Fig. 2b), the alignment of Fe filaments with wire axis is readily apparent. The cross-sectional shape of Fe filaments becomes ribbon-like because b.c.c. metals develop a ⟨110⟩ fiber texture during axisymmetric deformation which promotes plane strain deformation of Fe filaments [3]. Fig. 3 shows the microstructure of transverse (3a) and longitudinal (3b) sections of the Cu-Fe-Ag sheets after cold-rolling. During cold rolling the primary and secondary Fe dendrite arms are aligned along the rolling direction and elongated into filaments. Fe appears as flattened plates in rolled sheets. Ag was found to have no solubility in Fe [10]. Indeed, it was found by EDS analysis that Ag atoms were mostly observed in Cu matrix and Fe filament was almost free of Ag atoms [11]. Filaments were found to be much finer and more well-developed in the wires than in the sheets, which shows the relatively thick filaments. The spacings between filaments was 1.79 $\mu$m in Cu-Fe-Ag wires and 2.45 $\mu$m in Cu-Fe-Ag sheets.

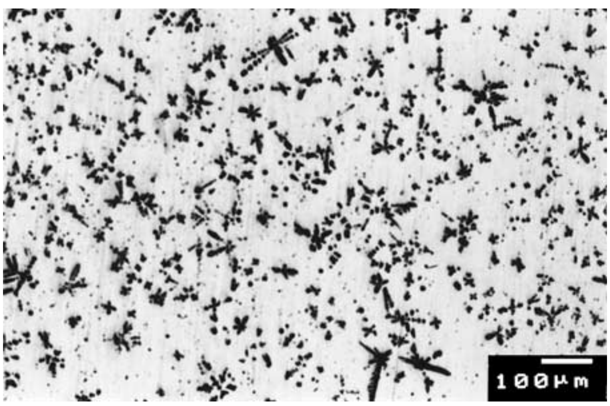


*Figure 1* Cast structures of Cu-9 Fe-1.2 Ag microcomposites.

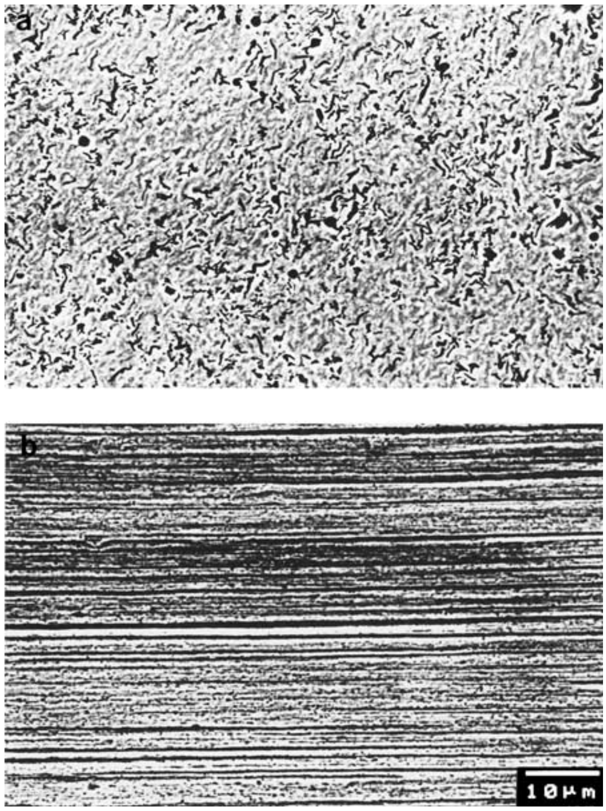


*Figure 2* Microstructures of transverse (a) and longitudinal (b) sections of Cu-9 Fe-1.2 Ag microcomposite wires.

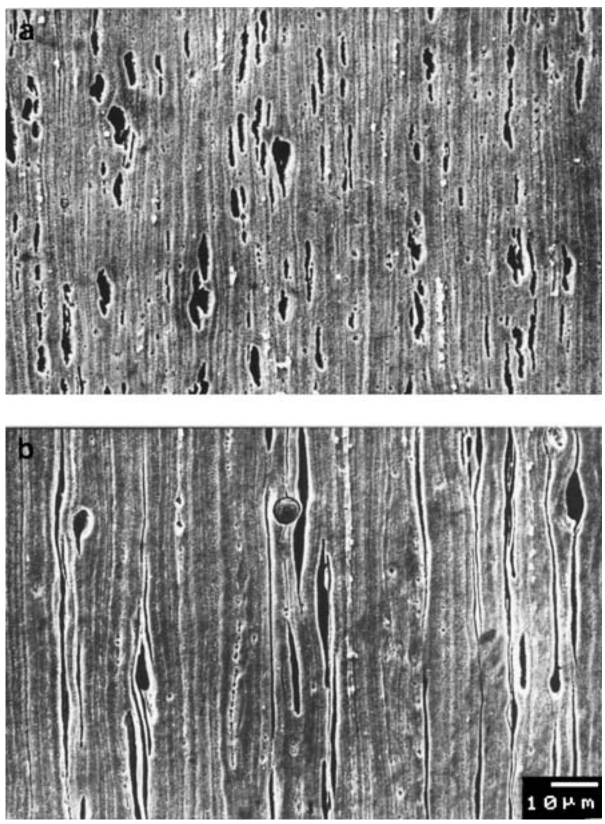


*Figure 3* Microstructures of transverse (a) and longitudinal (b) sections of Cu-9 Fe-1.2 Ag microcomposite sheets.

The reason for the finer filaments in the wires than in the sheets can be explained using the difference of the strees state between the two processes. The deformation of the microcomposite during the rolling is similar to uniaxial compression at initial stage because of the similar length of the workpiece in height and width, although it the plane strain condition is fulfilled at the later stage, corresponding to the geometric condition of the rolling process. Indeed, the width of the rolled sheets increased by 40–50% during the initial stage of rolling. Since both the uniaxial compression and the plane strain rolling constrain the workpiece less effectively than the axisymmetric drawing because of the free side surfaces of the workpiece parallel to the rolling direction, the filaments in the rolled sheet experience less deformation that those in the drawn wire. Metallic flow of the matrix occurs relatively easily in two directions (lateral and forward directions) when the workpiece is relatively thick during rolling compared with the unidirectional metallic flow during drawing and the refinement induced by co-deformation of filament and Cu matrix is more effective during drawing.

The ultimate tensile stresses of Cu-Fe-Ag wires and sheets at the final deformation ratio were observed to be 916 MPa and 800 MPa respectively. The stengths of the wires were found to be greater than those of the sheets, which also can be associated with the presence of finer filaments in the wires. The strengthening in these microcompsoites results from the presence of the aligned filaments and the higher strength of Cu-Fe-Ag microcomposites correlates with the fineness of the microstructural scale. Therefore, the strength of Cu-Fe-X microcomposites can be described by the following Hall-Petch equation:

$$\sigma = \sigma_0 + k\lambda^{-1/2} \quad (1)$$

where $\sigma_0$ is the friction stress, $k$ the Hall-Petch coefficient and λ the spacing between filaments. The friction stress may include the lattice friction and precipitation strengthening. The coefficient $k$ was reported to be 1.0 $MN/m^{3/2}$ [3]. In Cu-Fe-Ag microcomposites, the friction stress is associated with the lattice friction stress including solution and precipitation hardening. Since the Cu matrix is strengthened by Ag precipitates, the friction stress in Cu-Fe-Ag may be mostly due to precipitation of Ag precipitates. Hong and Hill [5] suggested that the strengthening component due to alloying in Cu-8 wt% Ag alloy was 300 MPa. If the strengthening component due to silver precipitation is proportional to $C^{1/2}$, where C is the silver content, the friction stress due to silver precipitation in Cu-9Fe-1.2Ag is calculated to be 116 MPa. In Table I, the Hall-Petch strengthening component, the friction stress component, the predicted strength and the experimental strength for Cu-Fe-Ag microcomposites are summarized. The experimental strength is in good agreement with the prediction of the present study.

TABLE I The predicted and experimental strength

| | Cu-9Fe-1.2Ag wire | Cu-9Fe-1.2Ag plate |
|---|---|---|
| Interfilamentary spacing | 1.79 $\mu$m | 2.45 $\mu$m |
| Friction stress, $\sigma$ o | 116 MPa | 116 MPa |
| Hall Petch Strengthening | 747 MPa | 639 MPa |
| Predicted strength | 863 MPa | 755 MPa |
| Experimental strength | 916 Mpa | 800 Mpa |

The electrical conductivities of Cu-9 Fe-1.2 Ag wires and sheets were 56.2% IACS and 53% IACS respectively. It is interesting to note that the electrical conductivities of wires are generally higher than those of sheets despite the higher strength of wires. The good mechanical and electrical properties of wires may be associated with the more uniform distribution of the finer filaments in the wires. Verhoeven *et al.* [7] observed that the conductivity of sheet materials is much lower than that of wire at the same strength level. In the sheet materials it is found that the filaments subdivide the Cu matrix into planar sheets, whereas in the wire the Cu matrix is subdivided into cylinders having irregular, but essentially equiaxed cross section. The difference of the conductivity between wires and sheets may be associated the geometrical difference of the Cu matrix. It was suggested by Verhoeven *et al.* [7] that the planar geometry of Cu matrix is not effective in increasing the mobility of electrons. The interesting result on the geometry of Cu matrix on the electrical conductivity remains to be understood.

The fracture morphologies of Cu-9 Fe-1.2 Ag microcomposite wires and sheets are shown in Fig. 4. Both Cu-9 Fe-1.2 Ag microcomposite wires and sheets exhibited highly ductile fractures. The surfaces of the Cu-9 Fe-1.2 Ag microcomposites show many fine dimples and relatively coarse dimples. The coarse dimples in Cu-Fe-Ag microcomposite sheets appear quite

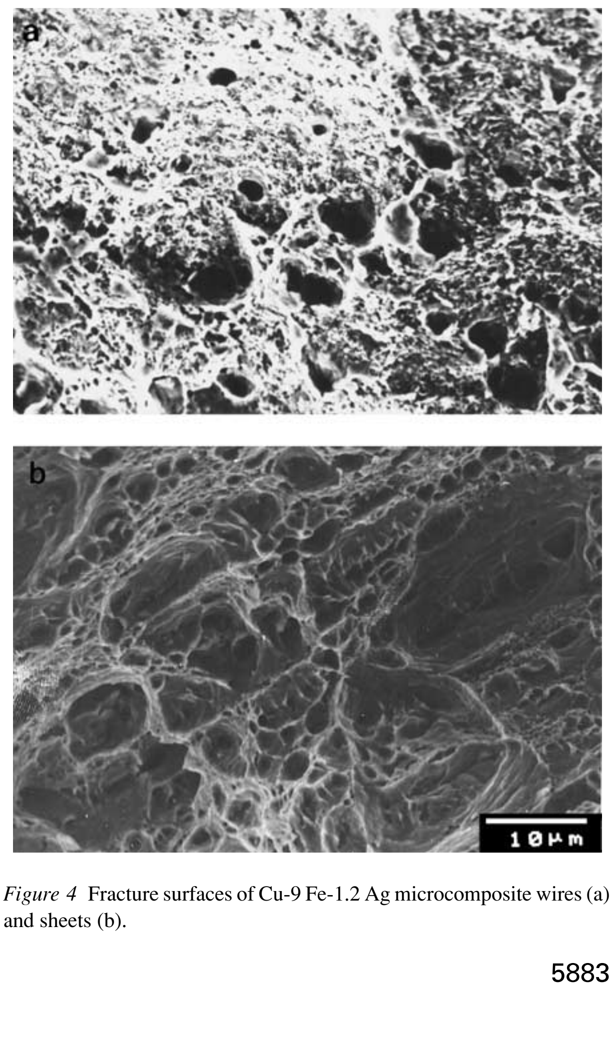


*Figure 4* Fracture surfaces of Cu-9 Fe-1.2 Ag microcomposite wires (a) and sheets (b).

elongated parallel to the sheet surface, compatible with the planar geometry of Fe filaments. Examination of the coarse dimples on the fracture surfaces revealed the fractured Fe filaments in the center. The fracture surfaces of the Cu-9 Fe-1.2 Ag microcomposite wires showed generally finer fracture surfaces than those of Cu-9 Fe-1.2 Ag microcomposite sheets, consistent with the finer microstructure in Cu-9 Fe-1.2 Ag microcomposite wires.

## 4. Conclusions

Based upon a study of the microstructure, strength and conductivity in wire-drawn and rolled Cu-Fe-Ag microcomposites, the following conclusions can be obtained.

1. The strengths of the wires were found to be greater than those of the sheets, which also can be associated with the presence of finer filaments in the wires. The strengthening in these microcomposites results from the presence of the aligned filaments and the higher strength of Cu-Fe-Ag microcomposites correlates with the fineness of the microstructural scale.
2. Since metallic flow of the matrix occurs relatively easily in two directions (lateral and forward directions) when the workpiece is relatively thick during rolling compared with the unidirectional metallic flow during drawing, the refinement induced by co-deformation of filament and Cu matrix is more effective during drawing, resulting in the finer microstructure in Cu-Fe-Ag microcomposite wires.
3. The electrical conductivities of wires are generally higher than those of sheets despite the higher strength of wires. The good mechanical and electrical properties of wires may be associated with the more uniform distribution of the finer filaments in the wires.


## Acknowledgments

The authors acknowledge the support from Korea Research Foundation (KRF-2000-042-E00095).


## References


1. C. BISELLI and D. G. MORRIS, *Acta Mater.* **44** (1996) 493.
2. P. D. FUNKENBUSCH and T. H. COURTNEY, *Acta Metall.* **33** (1985) 913.
3. W. A. SPITZIG, A. R. PELTON and F. C. LAABS, *ibid.* **35** (1987) 2427.
4. J. D. VERHOEVEN, L. S. CHUMBLEY, F. C. LAABS and W. A. SPITZIG, **39** (1991) 2825.
5. S. I. HONG and M. A. HILL, *Acta Mater.* **46** (1998) 4111.
6. S. I. HONG, *Scripta Mater.* **39** (1998) 1685.
7. J. D. VERHOEVEN, W. A. SPITZIG, L. L. JONES, H. L. DOWNING, C. L. TRYBUS, E. D. GIBSON, L. S. CHUMBLY, L. S. FRITZEMEIER and G. D. SCHNITTGRUND, *J. Mater. Eng.* **12** (1990) 127.
8. Y. S. GO and W. A. SPITZIG, *J. Mater. Sci.* **26** (1991) 163.
9. J. D. VERHOEVEN, S. C. CHUEH and E. D. GIBSON, *ibid.* **24** (1989) 1748.
10. L. J. SWARTZENDRUBER, in "Binary Alloy Phase Diagrams," edited by T. B. Massalski, H. Okamoto, R. R. Subramanian and L. Kacprzak, 2nd ed. (ASM International, Materials Park, Ohio, 1990) p. 35.
11. S. I. HONG and J. S. SONG, Chungnam National University, unpublished work.



*Received 1 August 2000*
*and accepted 8 August 2001*