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Microstructure evolution of Cu/Ag interface in the Cu–6 wt.% Ag filamentary nanocomposite

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Abstract

A Cu–6 wt.% Ag filamentary nanocomposite was prepared by melting, aging and cold drawing. The changes in phase morphology and interface behavior in the preparation process were investigated. The Ag precipitates evolve into filaments during drawing deformation. The interface changes into coherence from semi-coherence if the filament diameter decreases to ~2.0 nm during drawing deformation. The evolution of semi-coherent interface into coherent interface is analyzed on the basis of the minimum energy principle.

Keywords: Deep drawing; Copper alloys; Deformation structure; Interface structure

1. Introduction

The conductor materials for the manufacture of excitation windings in pulsed high-field magnets must have high mechanical strength and electrical conductivity in order to minimize Joule heating and endure Lorentz force caused by large electrical currents during operation. Promising candidates for conductor materials used in the pulsed high-field magnets are Cu–steel macrocomposites, and Cu–Nb and Cu–Ag nanocomposites [1–5]. Strong drawing deformation is an effective approach to producing the double-phase filamentary structure in Cu–Nb and Cu–Ag alloys. The filamentary nanocomposite structure can show a strength level far higher than predicted from the strength summation of phase components and still maintain a relatively high electrical conductivity [6–9].

Compared to Cu–Nb nanocomposites, Cu–Ag nanocomposites could obtain high strength at relatively small drawing strain. The properties of Cu–Ag nanocomposites are mainly dependent on the drawing strain, Ag content and heat treatment. In special, the microstructure evolution during cold drawing can play an important role in the change of the strength and conductivity with the drawing strain. The original microstructure of Cu–Ag alloys with an Ag content higher than 6 wt.% generally contained primary Cu dendrites, eutectic colonies and precipitated particles [8,10,11]. These structure components were elongated in the drawing direction and refined in the radial direction while the morphology of each structure component in the transverse section of the drawn wires remained unchanged during cold drawing. The dislocation density increased in initial drawing strain and decreased once the drawing strain had exceeded a certain limit or the filament diameter decreased to nanoscale. The mechanism responsible for the decrease in dislocation density at high drawing strains was considered to be the stagnation of dislocation reproduction from Frank–Read sources in the fibers of several nanometers and the annihilation of dislocations due to dynamic recrystallization and interface traps [12,13]. Many defects were found to locate at the interface between Cu and Ag phases [8,14]. Further investigation revealed that there were misfit dislocations arranged periodically at the interface at a certain drawing strain [15]. However, no

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detailed information on the misfit dislocation evolution during the cold drawing was given.

The presence of Ag precipitates and eutectic Ag laminas can form phase interfaces in Cu–Ag alloys. The interface characteristics should be different due to the different formation conditions of both Ag components. For example, a cube-on-cube orientation was maintained at all interfaces between the Ag precipitate and Cu matrix but only at some interfaces between the eutectic Ag and Cu matrix [14,16]. It was thought that the structural strengthening depended mainly on the interface between the Ag precipitate and Cu matrix and the electronic scattering depended mainly on the interface between the eutectic Ag and Cu matrix [14,15,17,18].

In this study, the interface between the Ag precipitate and Cu matrix was investigated for Cu–6 wt.% Ag at various drawing strains. The interface characterization and structure evolution during cold drawing were analyzed.

2. Experimental procedures

Cu–6 wt.% Ag was melted in a vacuum induction furnace. The Cu and Ag both had an initial purity of at least 99.95%. Cylindrical ingots of 23 mm diameter were cast under an Ar shielded atmosphere at a pressure of 2.7×10^4 Pa. The ingots were solution treated at 780 °C for 4 h followed by water quenching and then aged at 450 °C for 32 h. The cold deformation was performed by drawing. Drawing reduction was presented in terms of the logarithmic strain and referred as drawing strain:

$$\eta = \ln(A_0/A) \tag{1}$$

where A_0 and A are the cross-section areas of the original and drawn specimens, respectively.

The microstructure of the specimens at various drawing strains was observed by optical microscopy, transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HRTEM). The metallographic samples were ground, polished and subsequently etched in a solution of 100 ml C_2H_5OH, 5 ml HCl and 2 g $FeCl_3$. TEM and HRTEM samples were prepared by grinding the slices cut from the wire specimens to 75–100 μm thickness and punching into discs of 3 mm diameter. When the diameter of the drawn wires was smaller than 3 mm, the slices were stuck to the Cu ring plates of 3 mm outer diameter and 1 mm inner diameter. The discs were cooled to ~−110 °C by liquid nitrogen and then thinned by ion-beam at 3 kV with an incidence angle of 12°.

3. Results

3.1. Non-drawn microstructure

The as-cast Cu–6 wt.% Ag is composed of primary Cu dendrites and small eutectic colonies dispersed between the dendritic arms (Fig. 1a). The dendritic arm interval is ~9.1 μm and the diameter of the eutectic colonies ~1.7 μm. Since the Ag content in Cu–6 wt.% Ag is lower than the solid solubility limit (7.9 wt.% Ag) of Ag solute in Cu phase at eutectic temperature [19], those eutectic colonies in the as-cast Cu–6 wt.% Ag should be formed from the non-equilibrium freezing process. The Cu dendrites evolve into equiaxed grains and the non-equilibrium eutectic colonies disappear or most of the eutectic Ag phase dissolves into the Cu matrix in solution treatment (Fig. 1b).

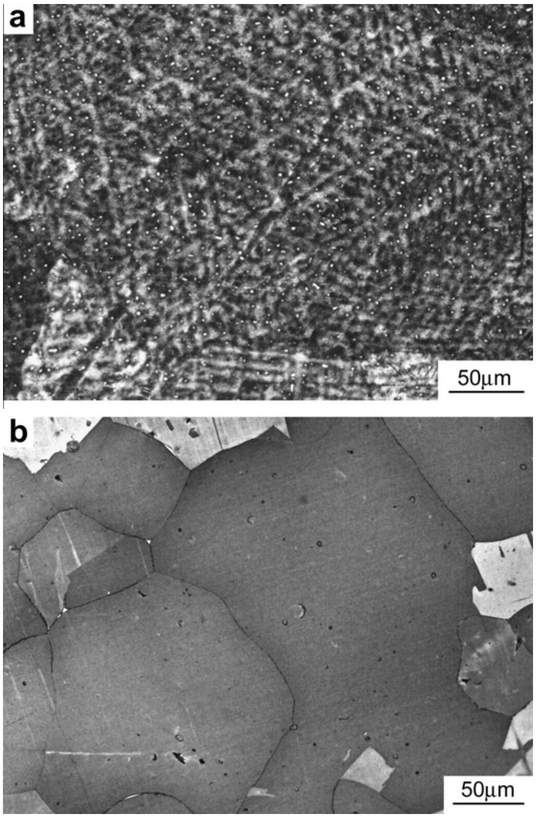

Fig. 1. Microstructures of the (a) as-cast and (b) quenched Cu–6 wt.% Ag.

There is a large amount of rod-like precipitates in the aged Cu–6 wt.% Ag (Fig. 2a and b). The average diameter of the precipitates is ~24 nm. The selected area electron diffraction (SAED) patterns (Fig. 2c and d) indicate that there should be a cube-on-cube orientation relationship, $\langle 0\,1\,1\rangle_{Ag}//\langle 0\,1\,1\rangle_{Cu}$ and $\{1\,1\,1\}_{Ag}//\{1\,1\,1\}_{Cu}$, between the Ag precipitate and Cu matrix. The Ag precipitates are determined to have a growth direction $\langle 0\,1\,1\rangle$ and a habit plane $\{1\,1\,1\}$, which is similar to the case in a Cu–5.7 wt.% Ag single crystal [20]. The parallel orientations, such as $(1\,1\,1)_{Cu}//(1\,1\,1)_{Ag}$ and $(\bar{1}\,1\,1)_{Cu}\,//\,(\bar{1}\,1\,1)_{Ag}$, further confirm the cube-on-cube orientation relationship between both phases [14,16,21]. The interface is planar and parallel to $(\bar{1}\,1\,1)_{Cu}$ or $(\bar{1}\,1\,1)_{Ag}$ (Fig. 2e). The image from the inverse fast Fourier transformation (IFFT) shows some misfit dislocations at the interface (Fig. 2f). Similar misfit dislocations were also observed at the eutectic Cu/Ag interface in Cu–71.8 wt.% Ag and at the Cu/Nb interface in Cu–Nb nanocomposites after heavy drawing deformation [22–26].