Phase composition and microstructure formation mechanism of in-situ Cu-Fe micro-composites

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Abstract. The phase composition and microstructure formation mechanism of in-situ Cu-Fe micro-composites were investigated. The microstructures of longitudinal and transverse sections were analyzed by light microscopy and scanning electron microscopy. The phase analysis was executed by X-ray diffraction. The common microstructure characteristic of Cu-XFe (X = 11, 14 and 17) alloys was that the second phase α-Fe dendrites were uniformly distributed in the Cu matrix. The disorderly distributed Fe dendrites of Cu-14Fe alloy underwent initial inhomogeneous deformation and then were gradually changed into the directionally arranged Fe fibers of in-situ Cu-14Fe micro-composite in the longitudinal section, and were gradually transformed into the irregular V-shaped Fe fibers in the transverse section. The initial inhomogeneous deformation and the irregular V-shaped Fe fibers in the transverse section are closely related to the formation of <110> texture.

1. Introduction
In-situ Cu-based micro-composites have been developed gradually since the late 1970s. It has been found by Harvard University scholar Bevk et al [1] that as-cast Cu-20% Nb alloys after severe plastic deformation can form composites with Nb fibers distributed on the copper matrix, and their tensile strength and conductivity can reach about 2000 MPa and 70% IACS, respectively. This kind of composites with in-situ formation of fiber in the process of cold deformation is called in-situ micro-composites. The excellent comprehensive properties matching ultra-high strength and good conductivity are the remarkable characteristics of this kind of materials.

A large number of studies have shown that these alloys consisting of Cu with face-centered cubic (fcc) lattice and transitional metals W, V, Mo, Cr and Fe with body-centered cubic (bcc) lattice or Ag, have similar microstructural characteristics, but due to the different kinds of alloying elements and preparation methods, there are some differences in mechanical properties and conductivity of the materials [2-5]. In recent years, a series of progress has been made in high strength and high conductivity Cu-based conductive materials. Among them, binary in-situ Cu-based micro-composites such as Cu-Nb, Cu-Ta, Cu-Ag, Cu-Fe and Cu-Cr have good strength and conductivity [6-11].

Over the past two decades, in-situ Cu-Fe micro-composites have become a hot spot for researchers
all over the world because of the relatively low cost of Fe [12-15]. In this work, the phase composition of Cu-Fe alloys and the microstructure evolution of in-situ Cu-Fe micro-composites were investigated. The microstructure formation mechanism during cold deformation was analyzed.

2. Experimental details
Ingots of Cu-XFe (X = 11, 14 and 17 wt.%) alloys were produced by a vacuum induction furnace. The casting temperature ranged from 1300 to 1400℃ and the ingots were cooled by furnace-cooling. The raw materials were commercial Fe with 99.94 wt.% purity and electrolytic Cu with 99.96 wt.% purity. The diameter of cylindrical ingots was about 36 mm, and the length was about 380 mm. The ingots of Cu-XFe (X = 11, 14 and 17 wt.%) alloys were hot rolled, cold drawn, and heat treated to in-situ Cu-XFe micro-composites with different cold deformation strains. The detailed preparation process was described elsewhere [16]. The cold deformation strain $\eta$ was obtained by cold drawing and calculated by $\eta = \ln(A_0/A_f)$ due to the large cumulative cold deformation strain, where $A_0$ and $A_f$ are the transverse sectional area before and after cold deformation, respectively [2,7].

The specimens were sectioned from the Cu-XFe ingots and in-situ micro-composites with different cold deformation strains, mounted, grinded, polished and then etched in an etching solution of 20 ml HCl, 5 g FeCl$_3$ and 120 ml H$_2$O. The microstructures of longitudinal and transverse section were investigated by light microscopy (LM) and scanning electron microscopy (SEM). The phase composition was analysed by X-ray diffraction.

3. Results and discussion

3.1. Microstructure and phase composition

![Figure 1](image-url)

**Figure 1.** LM microstructures of (a) Cu-11Fe alloy, (b) Cu-14Fe alloy and (c) Cu-17Fe alloy; (d) X-ray diffraction curves of Cu-11Fe, Cu-14Fe and Cu-17Fe alloys.

Figures 1(a), 1(b) and 1(c) show the LM microstructures of as-cast Cu-11Fe, Cu-14Fe and Cu-17Fe alloys, respectively. It can be seen that the microstructures of the three alloys are basically similar, mainly consisting of the matrix and the second phase dendrites distributed uniformly in the matrix. The Cu matrix is lighter than the Fe dendrites because the atomic number of Cu is bigger than that of Fe.
Figure 1(d) shows the X-ray diffraction curves of the three alloys. The XRD phase analysis presents that the three alloys are all composed of copper matrix and α-Fe. Obviously, the second phase dendrites in the as-cast microstructure are α-Fe and the matrix is Cu-based solid solution. That is to say, the common phase composition characteristic of the three as-cast alloys is that the second phase α-Fe is relatively uniformly distributed in the Cu matrix.

3.2. Longitudinal section microstructure

According to the phase diagram of binary Cu-Fe alloys, the Fe-rich solid solution of Cu-Fe alloys with 10-20% Fe is first precipitated in liquid Cu, and the solubility of Cu in γ-Fe decreases with the decrease of temperature. The eutectoid transformation of γ-Fe occurs at 850°C to form α-Fe. When the temperature further drops below 600°C, the two solvents are almost insoluble with each other. Figure 2 shows the microstructures of longitudinal sections of Cu-14Fe alloy after different cold deformation strains. The bright area is the Cu matrix and the dark area is the Fe phase. Figure 2(a) is the SEM microstructure of as-cast Cu-14Fe alloy after hot rolling at 850°C before cold deformation. The distribution of Fe dendrites has not changed significantly. The secondary dendrite arm elongates slightly along the drawing direction, but the size change is not obvious. Figures 2(b)-2(d) presents the formation of Fe fibers in the drawing direction of in-situ Cu-14Fe micro-composite during cold deformation. It can be seen that the disorderly distribution of Fe dendrites on the longitudinal section of as-cast Cu-14Fe alloy is gradually elongated into fibers arranged along the drawing direction with the increase of cold deformation strain. At η=1, the Fe dendrite has been broken and elongated partly in the drawing direction, but the deformation of the Fe dendrite is not uniform, some fine particles are elongated, some particles are elongated only at one end, while the other end remains unchanged, and some coarse particles remain unchanged, as shown in figure 2(b). At η=4, the directional arrangement of elongated Fe grains is gradually formed, but there are still uneven tadpole-like Cr grains, as shown in figure 2(c). With the further increase of cold deformation strain, the Fe fibers are basically formed, and the average size and spacing of the fibers decrease gradually, as shown in figure 2(d).

![Figure 2. SEM microstructures of longitudinal section of Cu-14Fe alloy after different cold deformation strains: (a) η=0; (b) η=1; (c) η=4; (d) η=7.8.](image)
3.3. Transverse section microstructure

Figure 3 shows the SEM microstructures of transverse section of Cu-14Fe alloy after different cold deformation strains. The bright area is the Cu matrix and the dark area is the Fe phase. Figure 3(a) is the microstructure of as-cast Cu-14Fe alloy after hot rolling at 850°C before cold deformation. The as-cast Fe dendrites were broken mainly by fracturing the primary dendrite arm. Figures 3(b)-3(d) presents the microstructure evolution of Fe grains in the transverse section of in-situ Cu-14Fe micro-composites during cold drawing. At $\eta=1$, the width of most Fe grains did not change obviously, but the thickness decreased, as shown in figure 3(b). With the increase of cold deformation strain, the thickness change of Fe grains was gradually discernable, and the end of the grains began to bend, as shown in figure 3(c). With the further increase of cold deformation strain, at $\eta=7.8$, the twist and bending of the grains were more obvious, and the deformation and distribution were basically uniform, as shown in figure 3(d). The result suggests that the width and thickness of the hot-rolled dendrites decrease with the increase of cold deformation strain, but the speed of reduction is quite different. The thickness deceleration is obviously faster, which increases the width-thickness ratio. Accordingly, the cold-drawn second phase gradually bended and broke, and the transverse section of the grains gradually transformed irregular V-shaped morphology.

![Figure 3. SEM microstructures of transverse section of Cu-14Fe alloy after different cold deformation strains: (a) $\eta=0$; (b) $\eta=1$; (c) $\eta=4$; (d) $\eta=7.8$.](image)

3.4. Microstructure formation mechanism

The non-uniform deformation of in-situ Cu-Fe micro-composites at the initial stage of deformation is related to the non-uniform distribution of radial shear strain during cold drawing and the rotation of the second phase along the drawing direction during deformation. In the initial stage of deformation, the critical shear stress changes under a given stress state, and the partially oriented grains begin to deform, while the partially oriented grains rotate and reorient. When the plastic deformation reaches a certain degree, the bcc Fe phase orientation tends to be the same, and the $<110>$ texture is formed along the principal strain direction [17].

The irregular morphology of Fe fibers in transverse section of in-situ Cu-14Fe micro-composites is also closely related to the formation of $<110>$ texture [17]. The deformation process of bcc Fe phase can be analyzed by figure 4 [18]. The slip plane and orientation of bcc Fe phase are $\{110\}$ and $<111>$.
respectively. The deformation direction in figure 4 is parallel to $[\overline{101}]$. There are four possible $<111>$ slip orientations of Fe phase $[\overline{111}]$, $[\overline{111}]$, $[111]$ and $[\overline{111}]$. The $[\overline{111}]$ and $[\overline{111}]$ orientations are within the slip plane $(110)$, so they can produce slip during the whole deformation process. Strains perpendicular to the direction of deformation can be produced in the orientations of $[111]$ and $[\overline{111}]$. If these two directions are not slippable, they will be plane strain deformation. The slip systems of the Cu matrix of fcc are $\{111\} <110>$ or $\{100\} <110>$. In order to coordinate the deformation with the axial uniform flow of the matrix, the $(110) [111]$ and $(110) [\overline{111}]$ slip systems of the Fe phase of bcc must be movable $[18,19]$. As a result, the Fe phase is forced to bend or break around the tensile axis, forming the irregular V-shaped morphology in the transverse section.

![Figure 4. Analysis of bcc phase deformation.](image)

4. Conclusions

- The common phase composition characteristic of Cu-XFe ($X = 11, 14$ and $17$) alloys is that the second phase $\alpha$-Fe is relatively uniformly distributed in the Cu matrix.
- The disorderly distributed Fe dendrites of Cu-14Fe alloy underwent initial inhomogeneous deformation and then were gradually changed into the directionally arranged Fe fibers of in-situ Cu-14Fe micro-composite in the longitudinal section.
- The randomly distributed Fe dendrites underwent initial inhomogeneous deformation and then were gradually transformed into the irregular V-shaped Fe fibers in the transverse section.
- The initial inhomogeneous deformation and the irregular V-shaped Fe fibers in the transverse section of in-situ Cu-14Fe micro-composite are closely related to the formation of $<110>$ texture.

Acknowledgments

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