Effect of doping with Zr on the properties of the deformation-processed Cu-Fe in-situ composites

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Effect of doping with alloying element Zr on the structure, the electrical resistivity and the strength of deformation-processed Cu-Fe in-situ composites were studied respectively by scanning electron microscope (SEM), transmission electronic microscope (TEM), material test system (MTS) and resistance measuring apparatus. The experimental results show that the ultimate tensile strength (UTS) and the conductivity of Cu-11.5% Fe-Zr wire cold drawn to the drawing strain $\eta = 7.57$ with intermediate heat treatments were observed to be 824 MPa and 61.4% IACS respectively, and those of Cu-11.5% Fe were 752 MPa and 64.6% IACS. Doping Zr can improve the thermal stability of Cu-Fe composites. The strength of Cu-Fe-Zr wire does not drop more rapidly at higher annealing temperatures (above 300°C) than that of Cu-Fe wire.

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1. Introduction

The term in-situ composite is used in the present context to describe materials where oriented fibrous microstructure is created by very heavy mechanical working as the mechanical strength increases. Among the alloys showing such behaviour it is possible to distinguish those typically containing a dendritic body centred cubic (b.c.c.) phase dispersed in a face centred cubic (f.c.c) matrix, such as Cu with Nb, Ag, Ta, Cr or Fe. The literature indicates that high strength and high conductivity Cu-X in-situ composites are widely used for applications such as lead frames and electrical connector. The Cu-Fe system is particular interest because of the relatively low cost of iron compared to the other possible components. But the relatively high solubility of iron in copper at high temperature, coupled with the slow kinetics of iron precipitation at low temperatures makes it difficult to achieve good electrical conductivity. The study on the high strength potential in drawn Cu-Fe alloys had begun several decades ago. It is reported that Hodge and his team had carried out a research programme to develop high strength and high conductivity wires for the US Army in the late 1940s [1]. They had invented a new wire with high strength and high conductivity. The alloy wire containing Cu-15%Fe-0.1%Mg, had tensile strength of 1080 MPa with conductivity of 56% IACS. The particularly attractive feature of these alloys is the combination of high strength plus high electrical conductivity. For this reason studies have employed difference mechanical and thermal treatments, for evaluating the effectiveness of various treatments at improving the strength and electrical conductivity of Cu-Fe alloys. Previous studies have been mostly directly to binary Cu-Fe alloys, with the reduction of Fe content, the conductivity is expected to increase at the expense of the strength. Verhoeven introduced proper intermediate heat treatment to the process of the deformation to promote precipitation of Fe from Cu-matrix, his experiment results shown that the electrical conductivity of alloy wires is improved [2]. Meanwhile, several investigators analyzed the strengthening mechanism [3, 4] and the relationship between the strength and the microstructure of Cu-Fe in-situ composite [5, 6]. In order to achieve higher strength and better resistance to recrystallization of Cu phases during aging treatment, small amount of third element has been added into the Cu-X alloys, such as Zr has been added into Cu-Cr alloy. It was found that the addition of the alloying element could dramatically reduces the thickness of the primary second phase at the same drawing strain, retard the dynamic recovery and recrystallization of Cu phase and lead to the banded structure in Cu. In this study the structure-property relationship in thermo- mechanically processed Cu-Fe based in-situ composites alloyed with the third alloying element Zr was examined. The purpose of this study is to investigate the effect of the third alloying element on the microstructure and physical properties of deformation processed Cu-Fe-Zr in-situ composite wires.

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2. Experimental

Two compositions were chosen for investigation, namely, Cu-11.5\%Fe and Cu-11.5\%Fe-0.2\%Zr. The melts were prepared using electrolytic Cu with at least 99.99 wt\% purity, ingot iron with at least 99.9 wt\% purity and commercial Zr. The alloys were separately melted in a vacuum induction furnace using a magnesia crucible. The alloys were cast into a Cu-mould at a temperature about 1300°C. Table I shows the final compositions of the cast alloys.

Its diameter of cast samples was about 25 mm. Cylindrical ingots of 22 mm diameter were cut from the as cast samples in order to remove the oxidation layer and surface defects. The samples were produced by rotary swaging to 16 mm diameter and subsequent by rolling to 8.5 mm diameter. Then the samples were cold drawn to various strains through hard metal drawing bench dies with three intermediate heat treatments. The strains were defined by \( \eta = \ln(A_0/A_f) \), where \( A_0 \) is the initial section and \( A_f \) is the final section. At last some samples were heat treatment in a vacuum furnace. The experimental processes are shown in Fig. 1.

The microstructure of samples were investigated by transmission electron microscopy (TEM) TecnaiG^2^20 with an accelerating voltage of 200 kV. The tensile test were carried out using material test system (MTS) at strain rate of \( 10^{-2} \) mm/s. UTS was calculated as an average for five samples with error of \( \pm 5 \) MPa. The fractures of the tensile specimens were examined in scanning electron microscopy (SEM) JSM-6360 with an accelerating voltage of 20 kV. The electrical resistivity was measured at room temperature with the conventional four-point method at constant current of 100 mA.

3. Results and analysis

3.1. Microstructure

Details of microstructural evolution of the as cast Cu-Fe alloys have been given elsewhere [7]. The dendrites of Fe were homogeneously distributed in the Cu-matrix. Fig. 2

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<table>
<thead>
<tr>
<th>Alloys</th>
<th>Cu</th>
<th>Fe</th>
<th>Zr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-11.5%Fe</td>
<td>Bal.</td>
<td>11.5</td>
<td>–</td>
</tr>
<tr>
<td>Cu-11.5%Fe-Zr</td>
<td>Bal.</td>
<td>11.5</td>
<td>0.200</td>
</tr>
</tbody>
</table>

Table I Material composition (in wt.\%)

Figure 1 Processing route of Cu-Fe wires manufacture. (HT1, HT2, HT3–intermediate heat treatment, \( \Delta \eta = 0.02 \sim 0.1 \)).

Figure 2 Longitudinal (a) and transverse section (b) TEM micrographs of the Cu-Fe in-situ composite (\( \eta = 6.63 \)).