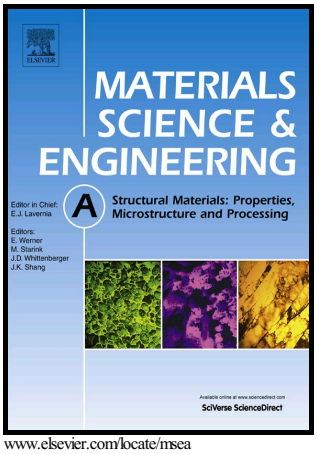

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Influence of Ag micro-alloying on the thermal stability and ageing characteristics of a Cu-14Fe in-situ composite

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Abstract

This paper studied the influence of Ag micro-alloying on the thermal stability and ageing characteristics of a deformation-processed Cu-14Fe in-situ composite prepared by thermo-mechanical processing. Heat treatment caused (i) edge recession, longitudinal splitting, cylinderization, break-up and spheroidisation of the Fe fibres in the Ag micro-alloyed Cu-14Fe in-situ composite, and (ii) recovery, recrystallisation and precipitation in the Cu matrix. Ag micro-alloying caused these processes to occur at lower temperatures. The index Z (a combination figure of merit that assesses the service performance) reached the peak value of 3.3×10^7 MPa^2·% IACS after isothermal heat treatment at 500 ℃ for 1 h, where IACS is the International Annealed Copper Standard, a measure of conductivity. The optimum combinations of tensile strength and conductivity were 1033 MPa and 56.6% IACS; 931 MPa and 58.9% IACS; or 851MPa and 60.6% IACS. The tensile strength and conductivity of Ag micro-alloyed Cu-14Fe in-situ composite at η = 7.8 after isochronal heat treatments were higher than those of the Cu-14Fe in-situ composite at each temperature.

Keywords: Cu-14Fe; Ag micro-alloying; microstructure; thermal stability; ageing characteristics

1. Introduction

In recent decades there has been much research on binary deformation-processed in-situ composite conductors based on a Cu matrix with bcc metal alloying, such as Cu-Nb, Cu-W, Cu-Ta, Cu-V, Cu-Cr and Cu-Fe [1-10]. These composites belong to the class of bulk nanostructured metallic materials, because of the small size of the structural nano-constituents produced via the in-situ deformation processing. The bcc metal fibres formed in the Cu matrix ensure high tensile strengths, and the limited solubility of bcc metals in the Cu matrix ensures the high electrical conductivity. Their high strength and high electrical conductivity allow their use as advanced materials for manufacturing components of electrical devices working under heavy mechanical loadings [11, 12].

The Cu-Fe system has attracted extensive attention because of potential applications in magnets, vacuum devices, contact bridging, next-generation storage and high-speed rail [9, 10, 12, 13]. However, Fe atoms dissolved in the Cu matrix severely decrease the conductivity of the Cu, due to the significantly harmful influence of Fe atoms in solid solution on conductivity [14, 15]. Further improvement of the combination of strength and conductivity of the Cu-Fe in-situ composites is necessary to extend their application. Recent research suggests that alloying and heat treatment are two principal approaches to increase the conductivity and strength of the in-situ composite [16, 17].

The addition of extra alloying elements allows many more possible kinetic paths to obtain the desired combination of strength and conductivity [18-20]. Wu et al. [21] studied the influence of rare earth elements on the microstructure and properties of a Cu-Fe in-situ composite. Rare earth elements refined the as-cast microstructure, promoted the precipitation of the secondary Fe particles from the Cu matrix, improved the recrystallisation resistance of the Cu matrix and increased the conductivity. Song et al. [20] investigated the effect of Cr and Ag on the microstructure and strength of a Cu-Fe in-situ composite. Ag strengthened the Cu matrix while Cr strengthened the Fe fibres. The refinement of fibres in the Cu-Fe-Cr in-situ composite was more difficult than in the Cu-Fe-Ag in-situ composite, and the tensile strength of the Cu-Fe-Ag in-situ composite was higher than that of the Cu-Fe-Cr in-situ composite due to the fact that the strength

of the in-situ composite is dependent on the fibre spacing in accordance with the Hall-Petch relationship.

Heat treatment with an appropriate temperature and an optimum duration allows high conductivities to be attained and good combinations of conductivity and strength to be produced [22-25]. Xie et al. [26] studied the influence of heat treatment on the microstructure and properties of two Cu-Fe-Ag in-situ composites. The prior homogenization treatment refined the primary Fe dendrites in the Cu matrix and promoted the precipitation of secondary Fe particles from the Cu matrix, which increased the strength and conductivity of the in-situ composites. Qu et al. [27] investigated the effect of the annealing at different temperatures for 1 h on the conductivity of a Cu-15Fe in-situ composite. The conductivity of the in-situ composite slightly increased with increasing annealing temperatures up to 500 ℃ due to the increasing fibre spacing and decreasing interface area, and the conductivity significantly decreased with increasing annealing temperatures above 500 ℃ because of the increasing Fe solubility in the Cu matrix.

The present work was undertaken to investigate the influence of Ag micro-alloying on the thermal stability and ageing characteristics of a deformation-processed Cu-14Fe in-situ composite produced by thermo-mechanical processing. The aims of this work were (i) to determine an appropriate heat treatment for the Ag micro-alloyed Cu-14Fe in-situ composite, and (ii) to produce optimum combinations of tensile strength and conductivity for the in-situ composite.

2. Experimental details

The Cu-14 wt.%Fe-0.1 wt.%Ag and Cu-14wt.%Fe (designated Cu-14Fe-0.1Ag and Cu-14Fe, respectively) ingots, 36 mm in diameter, were produced from electrolytic Cu, commercial Fe and Ag with at least 99.94 wt.% purity using a magnesia crucible in a vacuum induction furnace. The rod shaped ingots were hot rolled, heat treated and cold drawn to a cumulative cold deformation strain of η = 7, as described elsewhere [28]. The subsequent thermo-mechanical processing was similar to that used previously [28]: the in-situ composites were (I) isochronal heat treated at different temperatures from 200 to 700 ℃ for 1 h, and isothermal heat treated for different times from 1 to 8 h at 500 ℃, (II) cold drawn to η = 7.8, (III) aged at different temperatures from 200 to

600 ℃ for 1 h. All heating rates were 5 K/min and all heat treatment samples were furnace-cooled to room temperature. The cumulative cold deformation strain was evaluated as follows:

$$\eta = 1\mathrm{n}(A_0 / A_f \quad (1)$$

where A_0 is the original cross-sectional area after hot rolling, and A_f is the final cross-sectional area.

The longitudinal morphologies of the deformation-processed and heat-treated samples were examined using a JSM-6360LV scanning electronic microscope (SEM) and a JEM-2000EX transmission electronic microscope (TEM). The SEM samples were mechanically polished and etched using a solution of 120 ml H_2O, 20 ml HCl and 5 g $FeCl_3$. The TEM samples were mechanically thinned by grinding on grinding paper, the thickness was decreased using a dimple machine, and ion-milled using a Gatan model 600 ion beam thinner. The electrical resistivities of samples were measured at room temperature using a ZY9987 digital micro-ohmmeter with a precision of 1 μΩ, and the corresponding electrical conductivity was evaluated according to the definition of ICAS in which 1.7241μΩ·cm is defined as 100% IACS [28, 29]. Tensile tests were performed using an electronic tensile-testing machine equipped with custom designed wire grips [8]. The ultimate tensile stress (UTS) was used as a measure of tensile strength. At least four samples were tested for each electrical resistivity and UTS value.

3. Results and discussion

3.1. Thermal stability

Fig. 1 shows the longitudinal morphologies of the deformation-processed Cu-14Fe-0.1Ag in-situ composites at η = 7 after heat treatment at different temperatures in the range of 350 to 600 ℃ for 1 h. The uniformly distributed Fe fibres had formed in the in-situ composite at η = 7. Previous research [14, 30] suggested that a heavy deformation is necessary to produce a homogeneous size distribution of the second-phase fibres. The lighter phase corresponds to the Cu matrix and the darker phase corresponds to the Fe fibre, because the atomic number of Cu is larger than that of Fe. At low temperatures, there was no obvious change in the morphology of the Fe fibre. When the temperature was increased to 350 ℃, there was granular precipitation and edge recessions

occasionally on the fibres surface, as presented in Fig. 1(a). With increasing temperature, there were thermal grooves and pronounced unevenness on the fibres surface, and some fibres started to coarsen at the temperature of 450 ℃, as presented in Fig. 1(b). With a further increase of temperature, there were obvious longitudinal splitting and cylinderization in the fibres, and the break-up and spheroidisation of the fibres at the temperature of 550 ℃, as shown in Fig. 1(c). At the temperature of 600 ℃, the break-up and spheroidisation was essentially complete, and the fibres were changed into the chains of approximately oval Fe particles, as presented in Fig. 1(d). The morphologies of the fibres in the deformation-processed Cu-14Fe-0.1Ag in-situ composite experienced edge recession, longitudinal splitting, cylinderization, break-up and spheroidisation after exposed to elevated temperatures, which was similar to that in the Cu-14Fe in-situ composite [28]. Rabbe et al. [31] and Liu et al. [32] found that after heat treatments the second-phase fibres in the Cu matrix in-situ composites experienced coarsening, splitting, separating and breaking up into pieces. The observed break-up kinetics could be interpreted in terms of a capillary driven process which was principally controlled by interface diffusion [33]. But the temperature of the same morphology for the Cu-14Fe-0.1Ag in-situ composite was lower than that for the Cu-14Fe in-situ composite, which indicated that Ag micro-alloying decreased the heat stability of the Cu-14Fe in-situ composite.

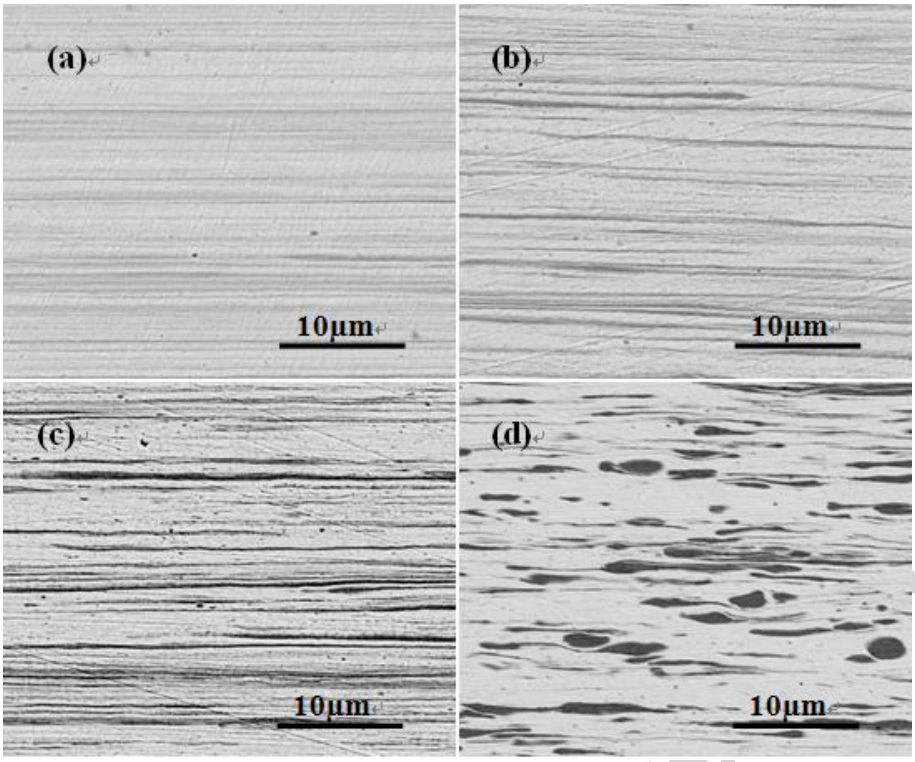

Fig. 1 Longitudinal morphologies of the Cu-14Fe-0.1Ag in-situ composite at η = 7 heat treated at different temperatures for 1 h: (a) 350 ℃, (b) 450 ℃, (c) 550 ℃, and (d) 600 ℃.

Fig. 2 shows the TEM images of the deformation-processed Cu-14Fe-0.1Ag in-situ composite at η = 7 after heat treatment at different temperatures in the range of 350 to 600 ℃ for 1 h. At the temperature of 350 ℃, there were granular precipitates and clear subgrain boundaries, and crystallisation nuclei in the junction of subgrains and in the interior of the Cu grains with high energy, as shown in Fig. 2(a). With increasing temperature, the grain crystallisation was essentially complete in the Cu matrix at the temperature of 450 ℃, as presented in Fig. 2(b). With a further increase of temperature, there were precipitates in the recrystallised grains at the temperature of 500 ℃, as illustrated in Fig. 2(c). However, even at the temperature of 600 ℃, there were subgrains in some grains, as presented in Fig. 2(d).This indicated that the migration of Cu grain boundaries was impeded and continuous recrystallisation accompanied by discontinuous recrystallisation during heat treatment. The Cu matrix in the deformation-processed Cu-14Fe-0.1Ag in-situ composite underwent recrystallisation and precipitation in the supersaturated Cu matrix after exposure to elevated temperatures, which was similar to that in the Cu-14Fe in-situ composite [28]. But the temperature of the same change for the Cu-14Fe-0.1Ag

in-situ composite was lower than that for the Cu-14Fe in-situ composite, which again indicated that Ag micro-alloying decreased the heat stability of the Cu-14Fe in-situ composite.

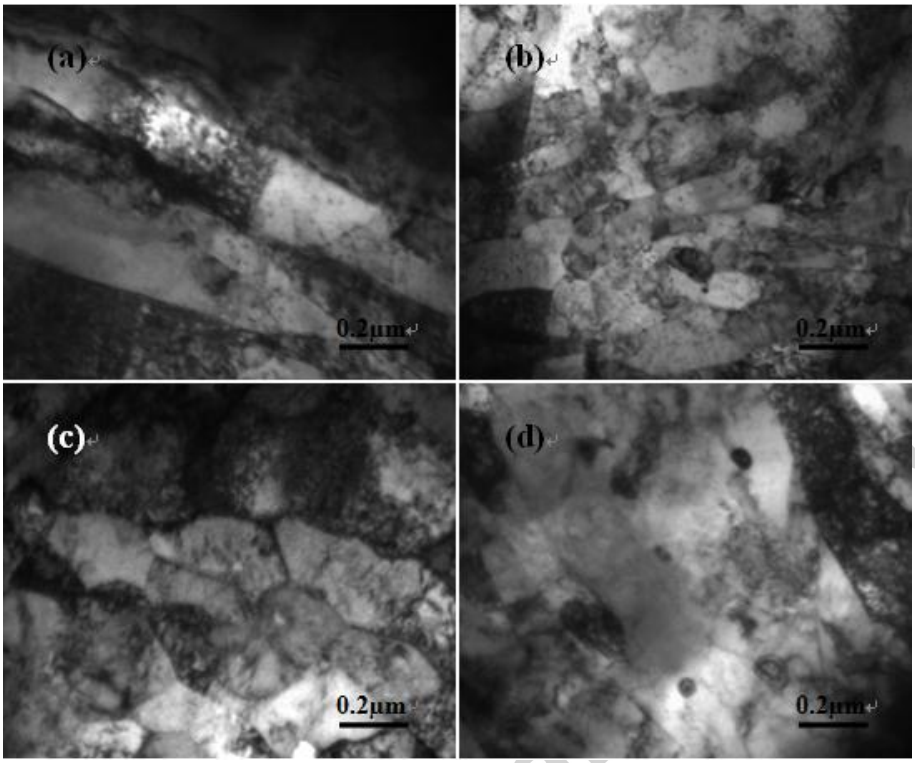

Fig. 2 TEM images of the Cu-14Fe-0.1Ag in-situ composite at η = 7 heat treated at different temperatures for 1 h: (a) 350 ℃, (b) 450 ℃, (c) 500 ℃, and (d) 600 ℃.

3.2. Determination of heat treatment process

Fig. 3 shows the tensile strength of the deformation-processed Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 versus the temperature after the isochronal heat treatment for 1 h. The tensile strength of the Cu-14Fe-0.1Ag in-situ composite was higher than that of the Cu-14Fe in-situ composite at the same heat treatment temperature, which is attributed to the fact that Ag micro-alloying refined the Fe phase. The details were explained in previous research [10, 14, 24]. The tensile strength of the Cu-14Fe-0.1Ag and Cu-14Fe in-situ composites first increased with increasing heat treatment temperature, reached peak values at 350 and 400 ℃, and subsequently decreased at higher temperatures, as presented in Fig. 3. For increasing temperatures less than the peak values, the increasing strength was attributed to fine Fe precipitates. There were no significant coarsening and pronounced unevenness of the Fe fibres. At higher temperatures, the

decrease in strength was due to the fact that the heat treatment promoted the overageing of the Fe precipitates, the recrystallisation of the Cu matrix, and the loss of fibre strengthening because of fibre coarsening and break-up [9, 24, 28]. The temperature corresponding to the peak strength for the Cu-14Fe-0.1Ag in-situ composite was lower than that for the Cu-14Fe in-situ composite, which is attributed to the decreased heat stability of the Cu-14Fe in-situ composite induced by Ag micro-alloying.

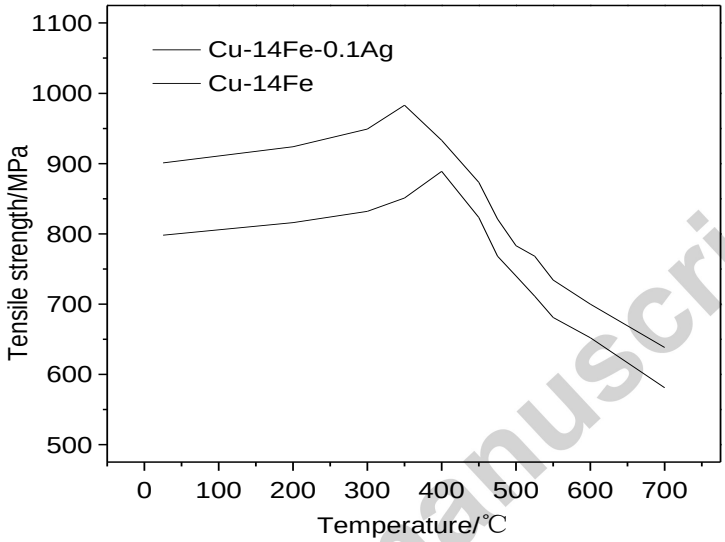

Fig. 3 Tensile strength curves of the Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 heat treated at different temperatures for 1 h.

Fig. 4 shows the electrical conductivity of the deformation-processed Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 versus the temperature for isochronal heat treatment for 1 h. The conductivity of the Cu-14Fe-0.1Ag in-situ composite was higher than that of the Cu-14Fe in-situ composite at the same heat treatment temperature, which is attributed to the decreased amount of dissolved Fe in the Cu matrix of the Cu-14Fe-0.1Ag in-situ composite caused by Ag micro-alloying. The detailed explanations are provided in previous research [10, 14, 24]. Similar to the effect of heat treatment temperature on the tensile strength, the electrical conductivity of the Cu-14Fe-0.1Ag and Cu-14Fe in-situ composites first increased with increasing heat treatment temperature, reached peak values at 500 and 525 ℃, and subsequently decreased at higher temperatures, as presented in Fig. 4. With increasing temperatures below the peak values, the increasing conductivity was caused by the decrease of Fe atoms in solid solution in the Cu

matrix. At higher temperatures, the decrease in conductivity was attributed to the fact that the heat treatment induced more cross sectional Cu/Fe phase interfaces due to fibre break-up [9, 24, 28]. The temperature corresponding to the peak conductivity for the Cu-14Fe-0.1Ag in-situ composite was lower than that for the Cu-14Fe in-situ composite, which is attributed to the fact that Ag micro-alloying decreased the heat stability of the Cu-14Fe in-situ composite.

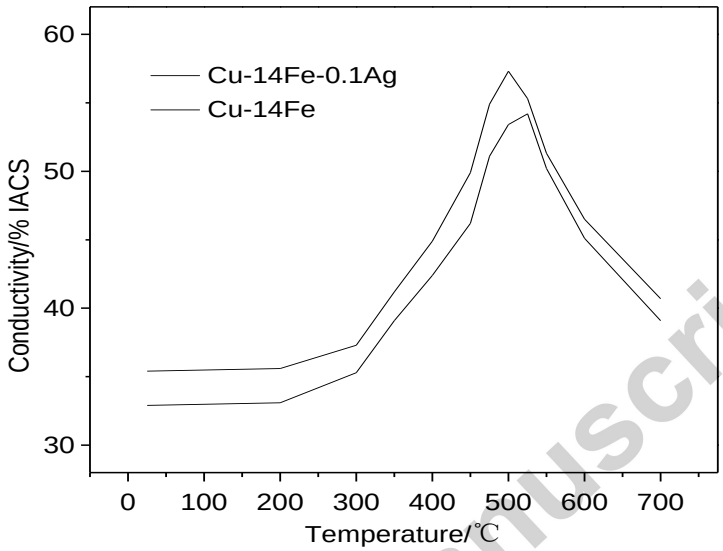

Fig. 4 Conductivity of the Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 heat treated at different temperatures for 1 h.

Fig. 5 shows the tensile strength of the deformation-processed Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 versus the time of isothermal heat treatment at 500 ℃. Fig. 3 and Fig. 4 indicated that the tensile strength and electrical conductivity of the Cu-14Fe-0.1Ag in-situ composite reached peak values at 350 and 500 ℃, and decreased with further increasing heat treatment temperature. Although the tensile strength and electrical conductivity peaks of the Cu-14Fe in-situ composite shifted to higher temperatures, the tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite were higher than that of the Cu-14Fe at each heat treatment temperature. For this reason, the proper heat treatment temperature should be lower than 500 ℃. Furthermore, the tensile strength could be easily increased by a subsequent cold drawing. Therefore, the isothermal heat treatment temperature was determined to be 500 ℃. As illustrated in Fig. 5, the tensile strength of the two in-situ composites decreased with increasing isothermal heat treatment time. The conglomeration of precipitated particles increased, the coarsening of

fibres intensified, and the recrystallisation of Cu matrix increased with increasing isothermal heat treatment time, which decreased the tensile strength of the two in-situ composites [24, 28, 29].

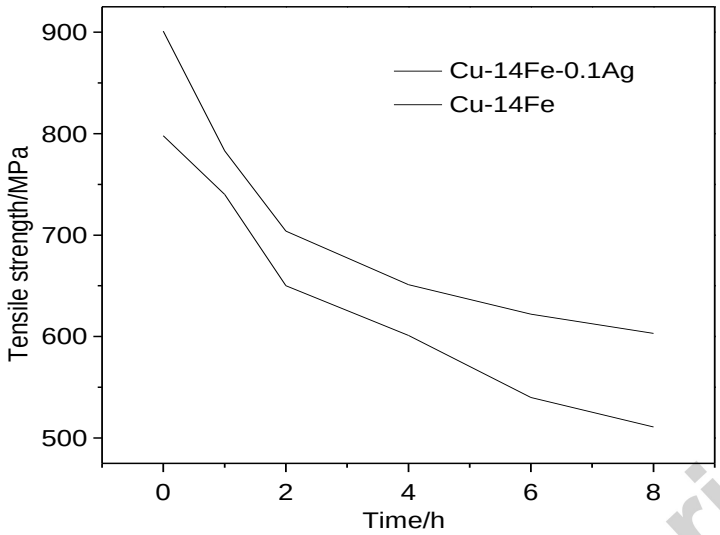

Fig. 5 Tensile strength of the Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 heat treated at 500 ℃ for different times.

Fig. 6 shows the electrical conductivity of the deformation-processed Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 versus the time of isothermal heat treatment at 500 ℃. The conductivity of the two in-situ composites increased with increasing isothermal heat treatment time, which is opposite to the change of the tensile strength. The increase in conductivity was attributed to the fact that the concentration Fe atoms in solid solution in the Cu matrix decreased with increasing isothermal heat treatment time, and the break-up and spheroidisation of the Fe fibres hardly occurred until after heat treatments at 500 ℃ for the times used in this investigation. The result is similar to that of previous studies [24, 28].

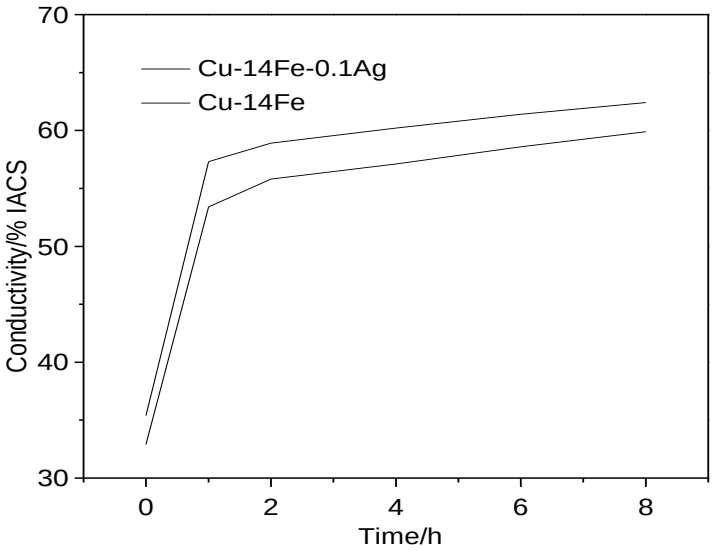

Fig. 6 Conductivity of the Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 heat treated at 500 ℃ for different times.

Fig. 5 and Fig. 6 show that the trends of the tensile strength and conductivity of the in-situ composites were opposite with increasing time of isothermal heat treatment, i.e., the tensile strength decreased while the conductivity increased. However, since both high strength and high conductivity are needed for service applications, a figure of merit, *Z*, has been proposed that combines the tensile strength and conductivity of the Cu matrix in-situ composites. The index is given by [28, 29, 34]:

$$Z = \sigma_b^{\ 2} \times \varphi \tag{2}$$

where σ_b is the tensile strength and φ is the conductivity.

Fig. 7 shows the index Z of the deformation-processed Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 versus the time of isothermal heat treatment at 500 ℃. The *Z* value of the Cu-14Fe-0.1Ag in-situ composite was higher than that of the Cu-14Fe at the same heat treatment time because of the higher tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite. The values of index *Z* of the two in-situ composites increased with increasing time of isothermal heat treatment and reached peak values at 1 h, and then progressively decreased. This result indicated that the optimum combination of properties of the in-situ composites was obtained as heat treated at 500 ℃ for 1 h. Accordingly 500 ℃ and 1 h were selected as the appropriate temperature and time for the heat treatment of the in-situ composites.

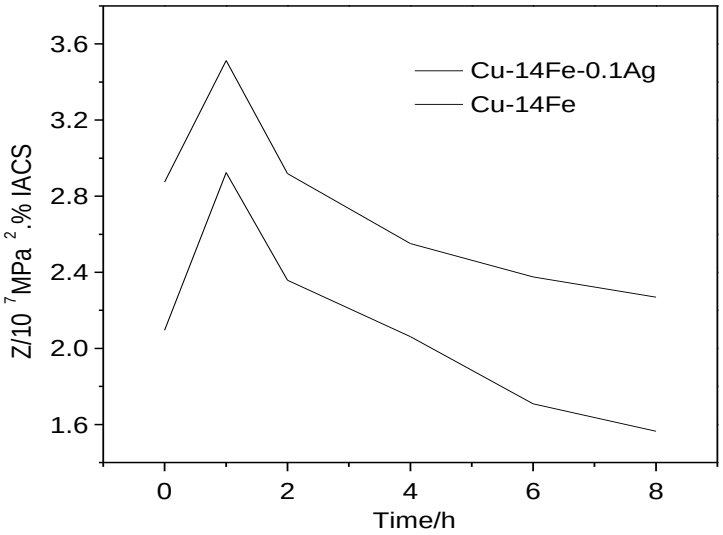

Fig. 7 Z value of the Cu-14Fe and Cu-14Fe-0.1Ag in-situ composites at η = 7 heat treated at 500 ℃ for different times.

3.3. Ageing characteristics

Fig. 8 shows the tensile strength and electrical conductivity of the deformation-processed Cu-14Fe-0.1Ag in-situ composites at η = 7 with heat treatment at 500 ℃ for 1 h, and η = 7.8. The conductivity of the in-situ composite slightly decreased with increasing cumulative cold deformation strain and the strength increased. These changes are attributed to the changes in the microstructure. The electrical resistivity of in-situ composites is principally determined by the resistivity contribution from four scattering mechanisms, i.e., phonon scattering ρ_{PHO}, impurity scattering ρ_{IMP}, dislocation scattering ρ_{DIS} and interface scattering ρ_{INT}. In-situ composites with the same composition and heat treatments have similar ρ_{PHO} and ρ_{IMP}. The cold deformation increased the dislocation density and interfacial area, which increased the electrical resistivity of the in-situ composite [10, 14]. Mattissen et al. [35] found that the electrical resistivity of the ternary Cu-Ag-Nb composite at large strains increased with increasing strain, and claimed that this was attributed to the size effect, i.e. to the inelastic scattering of the conduction electrons at the internal interfaces. In contrast, the tensile strength of the in-situ composite significantly increased with increasing cumulative cold deformation strain, which is attributed to the smaller fibre spacing and finer fibre size induced by the subsequent cold drawing [10, 14, 36, 37]. In addition, Rabbe et al.

[38, 39] studied the strengthening mechanisms of Cu-Nb in-situ composites and claimed that sometimes an enhanced solute content, individual confined phase strain hardening as well as even gradual amorphization was responsible for the increased strength.

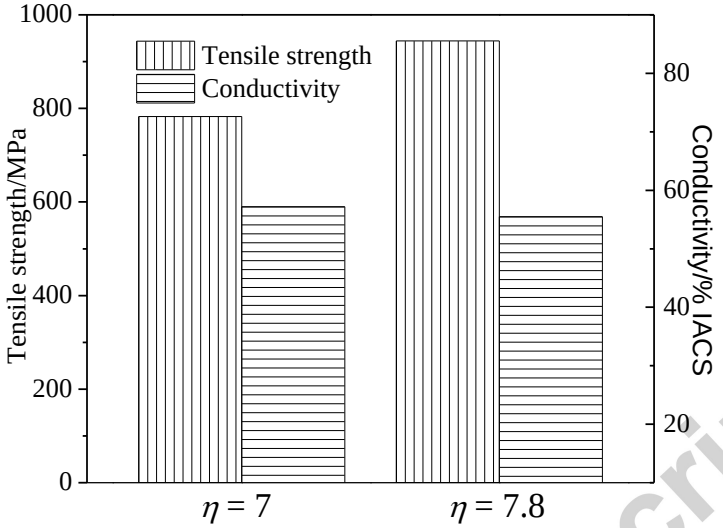

Fig. 8 Tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite at η = 7 heat treated at 500 ℃ for 1 h, and η = 7.8.

Fig. 9 shows the longitudinal morphologies of the deformation-processed Cu-14Fe-0.1Ag in-situ composites at η = 7 with heat treatment at 500 ℃ for 1 h, and η = 7.8. The external surfaces of the fibres were obviously uneven and the fibres had undergone significant coarsening in the in-situ composite at η = 7 with heat treatment at 500 ℃ for 1 h, as illustrated in Fig. 9(a). However, with further cold drawing, the uneven and coarsening fibres were changed into even and fine fibres in the situ composite at η = 7.8, as presented in Fig. 9(b). These results indicated that the changed morphology and size of the fibres caused by the heat treatment could be rapidly evened and refined by further cold drawing. The subsequent cold drawing promoted the thinning and homogenization of the changed fibres in the in-situ composite at η = 7 with heat treatment at 500 ℃ for 1 h, and decreased the fibre spacing and increased the interface area. After cold drawing to η = 7.8, the slight decrease in conductivity and the significant increase in tensile strength suggested that selecting the temperature corresponding to the peak conductivity as the appropriate heat treatment temperature was a rational decision.

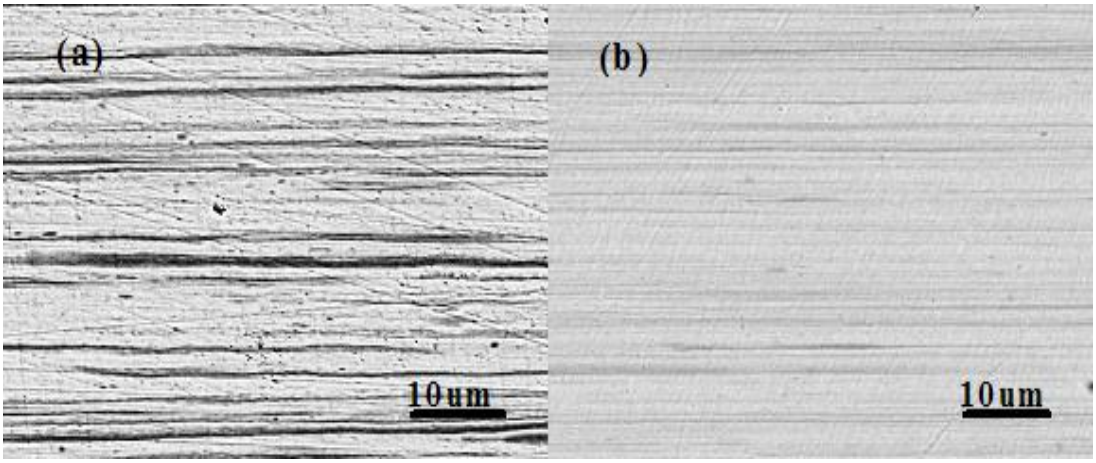

Fig. 9 Longitudinal morphologies of the Cu-14Fe-0.1Ag in-situ composite at: (a) η = 7 heat treated at 500 ℃for 1 h, and (b) η = 7.8.

Fig. 10 shows the tensile strength and electrical conductivity of the deformation-processed Cu-14Fe-0.1Ag in-situ composites at η = 7.8 versus the temperature for isochronal heat treatment for 1 h. Fig. 5 and Fig. 6 indicated that the tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite progressively increased with increasing isochronal heat treatment temperature and reached peak values at different temperatures, and then decreased at higher temperatures. Fig. 7 suggested that the index *Z* of the in-situ composite reached a peak value after isochronal heat treatment for 1 h. Accordingly, 1 h was selected as the duration time of isochronal ageing treatment to adjust and control the tensile strength and conductivity of the deformation-processed Cu-14Fe-0.1Ag in-situ composite at η = 7.8. Fig. 10 shows that the tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite at η = 7.8 first increased and then decreased with increasing ageing temperature, which is in agreement with the trends of the in-situ composite at η = 7. The temperatures corresponding to the tensile strength and conductivity peaks of the in-situ composite at η = 7.8 after isochronal heat treatment for 1 h were lower than those at η =7. This is due to the fact that the Fe fibres in the in-situ composite at η = 7.8 were finer than those in the in-situ composite at η = 7, which decreased the heat stability of the Cu-14Fe-0.1Ag in-situ composite [25, 28]. The tensile strength and conductivity of the deformation-processed Cu-14Fe-0.1Ag in-situ composite at η = 7.8 after isochronal heat treatment for 1 h reached 1033 MPa and 56.6% IACS; 931 MPa and 58.9% IACS; or 851MPa and 60.6% IACS, respectively. The tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite at η = 7.8 were higher

than those of the Cu-14Fe at each heat treatment temperature, although the tensile strength and electrical conductivity peaks of the Cu-14Fe-0.1Ag in-situ composite shifted to the lower temperatures [28].

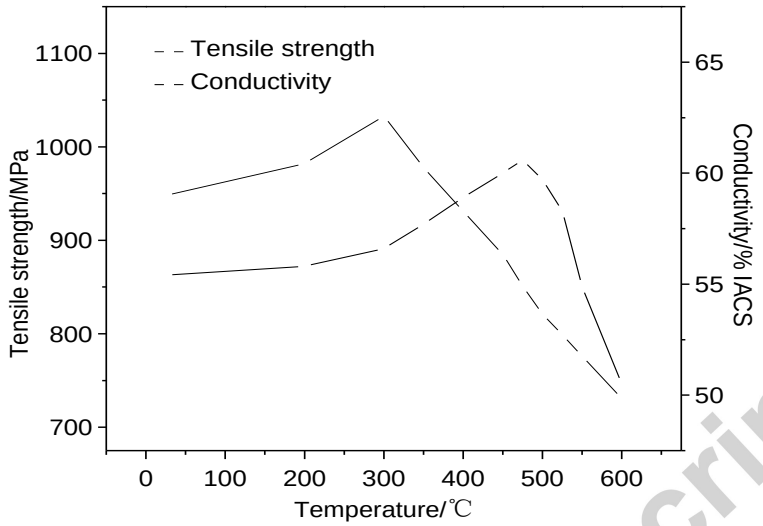

Fig. 10 Tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite at η = 7.8 heat treated at different temperatures for 1 h.

4. Conclusions

(1) Ag micro-alloying promoted edge recession, longitudinal splitting, cylinderization, break-up and spheroidisation of the Fe fibres in the Cu-14Fe-0.1Ag in-situ composite to occur at lower temperatures.

(2) The recovery, recrystallisation and precipitation phase transition of the Cu matrix of the Cu-14Fe-0.1Ag in-situ composite during heat treatment were shifted to lower temperatures by Ag micro-alloying.

(3) The index Z of the Cu-14Fe-0.1Ag in-situ composite reached the peak value of 3.3×10^7 $MPa^2 \cdot$% IACS after the isothermal heat treatment at 500 ℃ for 1 h.

(4) The tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite at η = 7.8 heat treated at 300, 400 and 475 ℃ for 1 h reached 1033 MPa and 56.6% IACS; 931 MPa and 58.9% IACS; and 851MPa and 60.6% IACS, respectively.

(5) The tensile strength and conductivity of the Cu-14Fe-0.1Ag in-situ composite at η = 7.8 were higher than those of the Cu-14Fe at each heat treatment temperature, although Ag

micro-alloying promoted the peaks of the Cu-14Fe-0.1Ag in-situ composite to occur at lower temperatures.

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