



Mechanical properties, electrical conductivity and microstructure of CuCrZr alloys treated with thermal stretch process

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Abstract: CuCrZr alloys were treated with the thermal stretch process at various temperatures from 100 to 300 °C. The results reveal that the thermal stretch process is successfully developed to manufacture the precipitation hardening CuCrZr alloys with a good combination of microhardness and electrical conductivity. By increasing the tensile elongations at each temperature from 100 to 300 °C, the microhardness increases whereas the electrical conductivity decreases slightly. Cr-containing precipitate phases with a Nishiyama–Wasserman orientation relationship to the copper matrix were observed by TEM. The achievement of high micro-hardness and acceptable electrical conductivity in the thermal stretch treated alloys is ascribed to the interactions of the heteroatom solution, dislocation increment, grain refinement and dispersive precipitation effect.

Key words: CuCrZr alloys; thermal stretch treatment; microhardness; electrical conductivity

1 Introduction

Due to high electrical conductivity and high strength, CuCrZr alloys have been considered to be promising materials in a wide range of industrial applications, for example, in the pantograph-OCS system where they play a dual role of conducting electricity and contacting slip orbit [1–6]. However, the developments of many modern engineering applications require such electrical contact materials to have a rather sophisticated combination of electrical and mechanical properties [7]. To satisfy the requirements, intensive researches have been undertaken on new material manufacturing processes, such as mechanical alloying [8], aging treatment [9] and equal-channel angular pressing [10]. In particular, the solid solution treatment with a subsequent aging process demonstrates tremendous potential in large-scale industrial production because it can significantly enhance the mechanical properties of the alloys with smaller electrical conductivity sacrifice. However, in this case, the ultimate material property is sensitively affected by the complicated intermediate treatment steps, which finally results in the difficulty in controlling the material quality. Moreover, the rather long aging time would lower the manufacturing

productivity.

With respect to the control techniques of the combination properties of CuCrZr alloy, much research effort has revealed that the key issue lies in the composition, morphology and distribution of the Cr-rich precipitates, which progressively dissolve out from the supersaturated Cu matrix during aging [11–13]. However, there has been no unanimous agreement on the precipitate phases and their effects on the final mechanical and electrical properties. EDWARDS et al [14] reported that the microstructures of the precipitates under the prime aging condition were small Guinier–Preston (G–P) zones. A high temperature annealing would result in the replacement of the G–P zones with other precipitates thought to be predominately incoherent Cr-rich particles. HUANG et al [15] observed a new $\text{Cu}_{51}\text{Zr}_{14}$ phase in the matrix of CuZrCr alloy while ZENG et al [16] declared the existence of three phases of Cr, Cu_5Zr and Cu in the CuCrZr system. In a similar CuZrCr(Mg) system, SU et al [6] found that, after solid solution treating at 920 °C for 1 h and aging at 470 °C for 4 h, three types of precipitates of CrCu_2 , Cu_4Zr and Cr occurred, which took the alloy hardness and electrical conductivity to HV 109 and 80% IACS (International Annealed Copper Standards), respectively. In comparison with this, by 60% rolling and aging at

470 °C for 1 h, the alloy hardness could even reach up to HV 165 and the electrical conductivity of 79.2% IACS can be maintained [6].

In this work, a combination of the aging treatments and the simultaneous mechanical deformation (thermal stretch) was carried out with an expectation of accelerating the CuCrZr aging process. The CuCrZr alloys were thermally stretched by heating up to various temperatures from 100 to 300 °C after solution annealing. At each heating temperature, different tensile elongations were fixed. The effects of thermal stretch treatments on the mechanical properties, electrical conductivity and microstructure of CuCrZr alloys were discussed in detail.

2 Experimental

The as-cast CuCrZr alloys had nominal compositions of 0.6% Cr (mass fraction) and 0.13% Zr (mass fraction) evaluated by a direct-reading spectrometer. They were first solution-annealed up to 920 °C for 1 h followed by water-quenching treatment. Afterwards, thermal stretch was carried out on an INSTRON universal testing machine equipped with a thermal cycling chamber for accurate temperature control. The round rod samples with a gauge length of 40 mm were stretched at 100, 200 and 300 °C, respectively. A heating time of 30 min was fixed to guarantee a homogenous temperature in the thermal cycling chamber before tensile deformation. The tensile deformation rate was 2×10^{-3} mm/min. To investigate the effects of different tensile elongations on the precipitate phases, the mechanical properties of alloy as well as electrical properties, progressive advanced tensile elongations were introduced at each temperature. As the setting tensile elongation reached, a thermal insulation process of 1 h was applied before the unloading.

The phase compositions of the thermal stretch treated samples were investigated by X-ray diffraction (XRD) with Cu K_{α} radiation ($\lambda=0.15406$ nm). The microstructure was observed using a Leica microscope and a scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) unit. The electric conductivity was evaluated from the sample electrical resistance by a QJ36S-type standard direct-current four-probe system. The Vickers microhardness was measured on a HVS-1000-type hardness tester with a load of 0.98 N for 15 s.

In order to identify the precipitate phases, the transmission electron microscopy (TEM) observation was conducted on a JEOL 2000 FX transmission electron microscope. For TEM sample preparation, the samples were cut from the gauge sections perpendicular to the tensile axis and thinned mechanically to a thickness of about 0.1 mm with a diameter of 3 mm. These discs were

then twin-jet electro-polished before loading into TEM vacuum chamber.

3 Results

3.1 Mechanical and electrical properties

For the aging process of the precipitation hardening alloy, the aging time and temperature have a significant effect on the alloy properties. Aging at low temperature only results in the formation of G–P zones. On the other hand, when the aging treatment is conducted at high temperature, over aging often occurs and thereby the precipitates tend to coarsen and lose coherency with the matrix [17]. In the present work, the thermal stretch temperature was fixed in the range of 100 to 300 °C in order to avoid either only the formation of the G–P zones or the overage behaviors, since the overage temperature of the CuCrZr alloy has been reported to be above 450 °C [18].

After the thermal stretch treatment, the hardness and electrical conductivity tests were first carried out to assess the combined mechanical and electrical properties. It should be noted that the maximum alloy elongations were carefully controlled to avoid the necking phenomenon.

Figure 1 shows the Vickers microhardness and electrical conductivity of the thermal stretch treated alloys as a function of the tensile elongation. The solid solution treated sample was taken as a reference, which possesses a microhardness of HV 78.4 and an electrical conductivity of 93.6% IACS. In contrast to these, the improvement of microhardness and the slight decrease of electrical conductivity for the thermal stretch treated samples are evident, indicating that the thermal stretch process can regulate the combination properties of CuCrZr alloys. By increasing the tensile elongation at each temperature, the microhardness increases. On the other hand, the electrical conductivity demonstrates a reverse trend as the tensile elongation increases. However, in spite of the decrease of electrical conductivity, the value maintained is still greater than 80% IACS, which is acceptable for most applications, for example, the application of railway contact wire [19]. The maximum microhardness of HV 106.2 is achieved at 300 °C with a tensile elongation of 22%, which is HV 27.8 larger than that of the solution treated sample and HV 23 higher than the obtained minimum microhardness (HV 83.2) after thermal stretch treatment. The combined mechanical and electrical properties for the sample treated at 300 °C with a tensile elongation of 22% are analogous to the normal aging treated CuZrCr(Mg) alloy at 470 °C for 4 h [6]. However, the process in this work demonstrates a lower treating temperature, less time as well as less alloy elements,

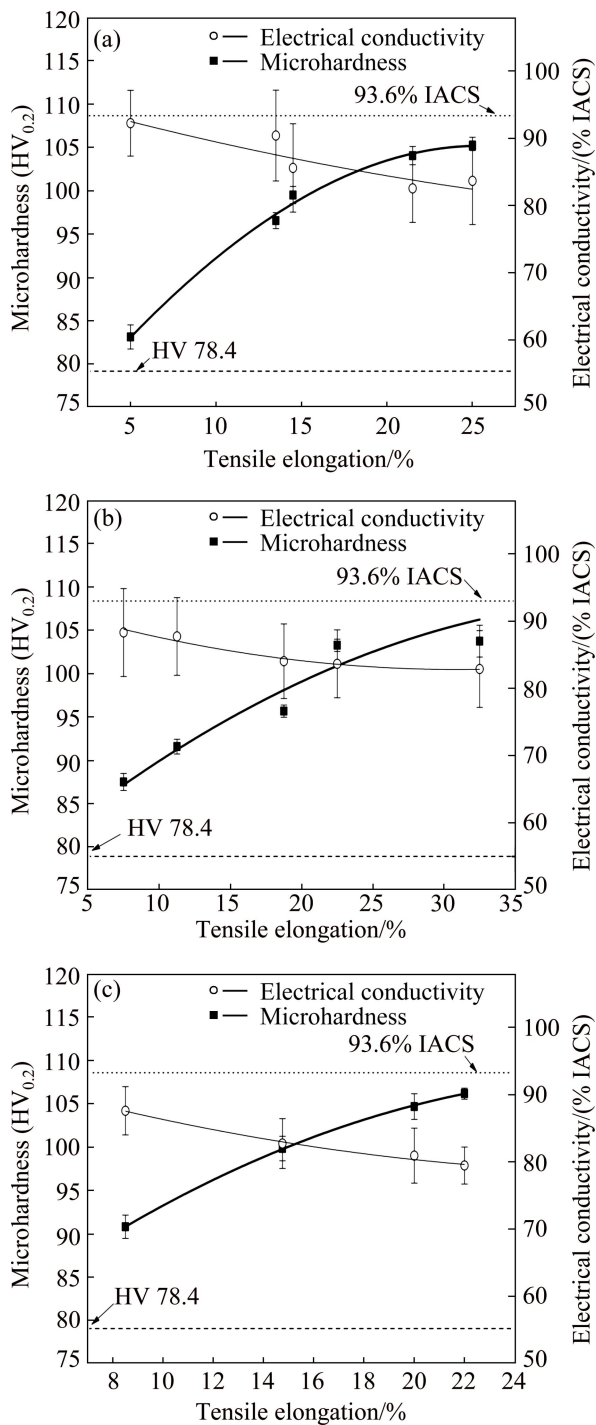


Fig. 1 Evolution of electrical conductivity and microhardness of CuCrZr alloys with tensile elongation at different temperatures: (a) 100 °C; (b) 200 °C; (c) 300 °C

which would improve the manufacturing efficiency.

To demonstrate the effect of the stretch temperature on the combination properties of CuCrZr alloy, the microhardness and electrical conductivity at a low temperature of 100 °C and a high temperature of 300 °C are illustrated in Fig. 2. It is found that the hardening response at high temperature is larger than that at low temperature when the samples are exerted with the same

tensile elongation. Conversely, the electrical conductivity at low temperature is a little higher than that at high temperature.

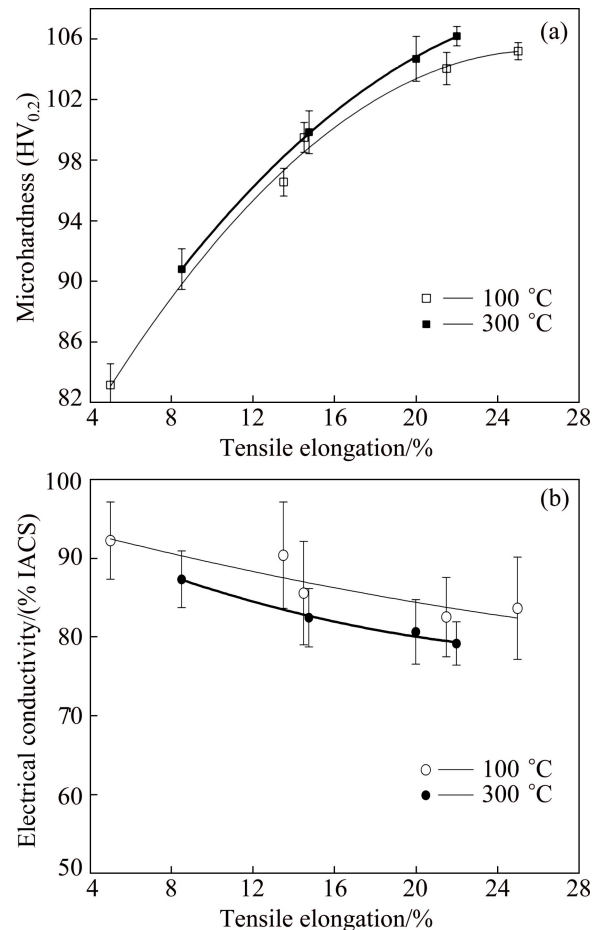


Fig. 2 Comparison of microhardness (a) and electrical conductivity (b) evolution with tensile elongation at low temperature (100 °C) and high temperature (300 °C)

3.2 Microstructure

To correlate the microstructure and combination properties of CuCrZr alloy, X-ray diffraction for the thermal-stretch treated samples was conducted and the typical XRD patterns for different tensile elongations at 300 °C are shown in Fig. 3. It is found that only the pure Cu diffraction peaks can be identified in spite of the variation of tensile elongations. The as-expected precipitate phases showed no traces in the XRD profiles. These results suggest that the precipitate phases are too tiny or their amounts are quite small. Compared with the pure Cu diffraction peak positions, the corresponding diffraction peaks in all CuCrZr alloys shift to high angles, indicating the solution of Cr or Zr in the Cu matrix and resulting in Cu lattice shrinkage. It is noted that, in contrast to the solution treated sample, the Cu(111) diffraction peak in the thermal stretch treated samples shows little shift, whereas Cu(200) and Cu(220) peaks demonstrate slight shift to low angles, which suggests

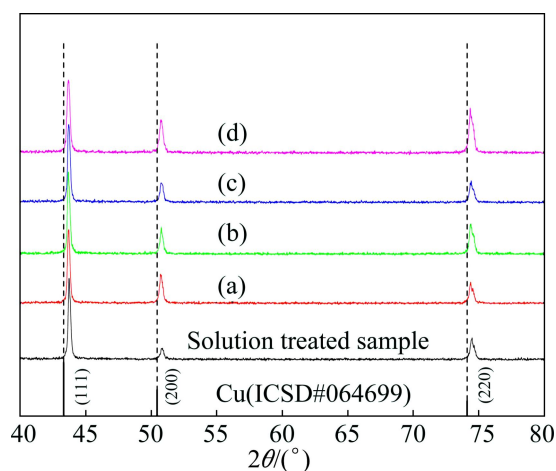


Fig. 3 XRD patterns of CuCrZr alloys with different tensile elongations at 300 °C: (a) 8.5% elongation; (b) 14.8% elongation; (c) 20% elongation; (d) 22% elongation (XRD patterns of pure Cu and solid solution treated sample are taken as references)

that the solution atoms precipitate along with certain crystal planes of the matrix. In addition, the vibrated shift of Cu(200) and Cu(220) peaks with increasing the tensile elongations implies that the precipitate crystal planes would change with the increment of deformation.

Figure 4 shows the optical images of CuCrZr alloy after solid solution treatment and thermal stretch treatment at 300 °C with an elongation of 22%. Figures 4(a) and (b) are the cross-sectional images of the solid solution treated sample in the longitudinal (along the tensile axis) and transverse (perpendicular to the tensile axis) directions, respectively. It is seen that the sample demonstrates a typical recrystallized structure, which mainly consists of equiaxed grains of 200–450 μm in size and some annealing twins. After thermal stretch, the coarse equiaxed grains are broken and become refined. They are elongated in the thermal stretch directions as shown in Fig. 4(c). The grain distortion can also be observed from the transverse direction images, as

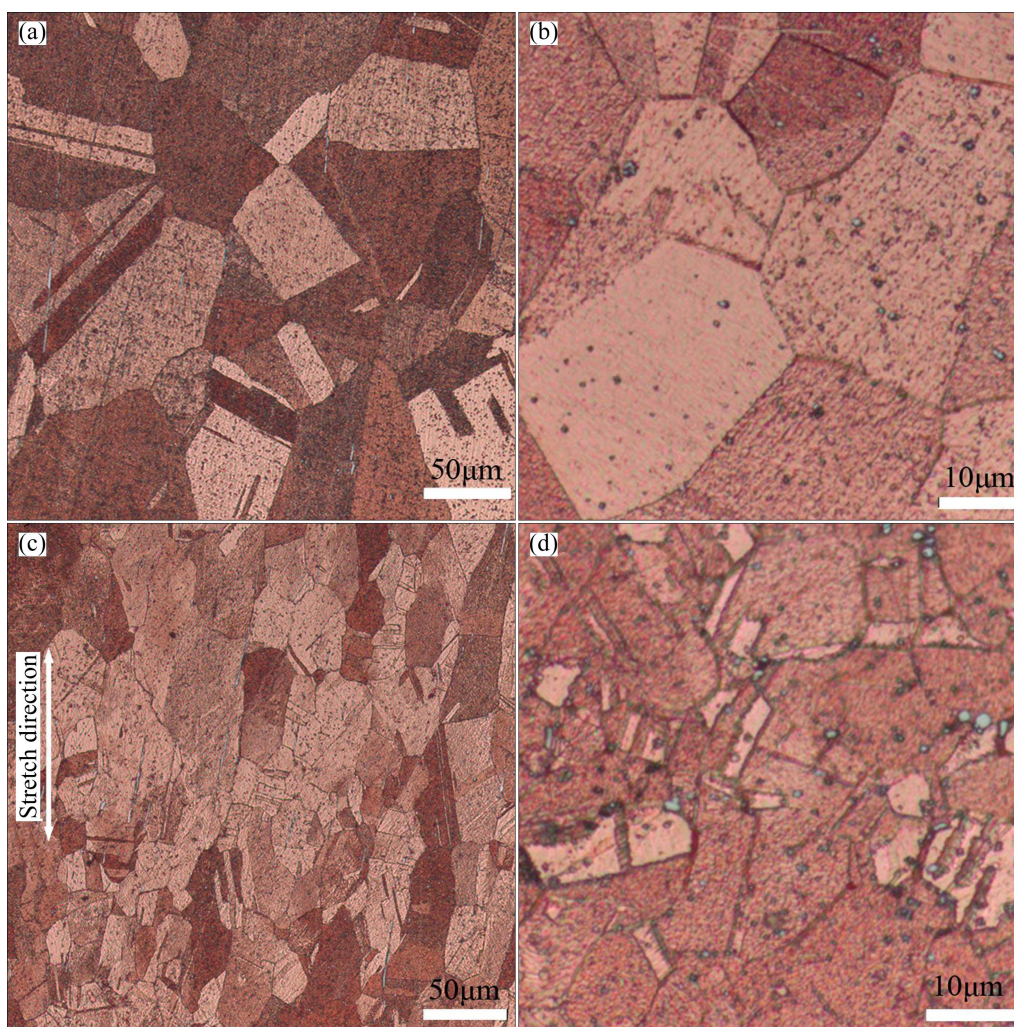


Fig. 4 Optical micrographs of solution treated sample in longitudinal (along tensile axis) direction (a) and transverse (perpendicular to tensile axis) direction (b), and longitudinal direction morphology (c) and transverse direction morphology (d) of samples treated at 300 °C with elongation of 22%

shown in Fig. 4(d).

To further confirm the element composition and distribution, the EDX element mapping image in the SEM in the transverse direction of the sample treated at 300 °C with an elongation of 22% was acquired. As shown in Fig. 5, both Cr and Zr element signals can be clearly detected. The quality analysis reveals element compositions of 0.63% Cr (mass fraction) and 0.14% Zr (mass fraction), which confirms the composition results obtained from the direct-reading spectrometer analysis.

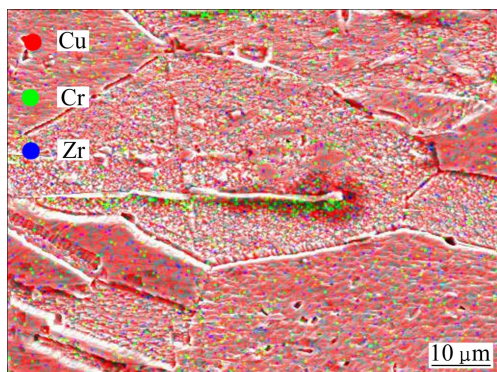


Fig. 5 Elemental mapping image of sample treated at 300 °C with elongation of 22%

3.3 Analysis of precipitate phases

The solid solution and thermal stretch treated CuCrZr alloy samples were examined by TEM in the transverse direction. Figure 6(a) shows the bright field (BF) TEM image of the solid solution treated sample. The precipitates demonstrate a low density with the dimension of several nanometers. They are distributed randomly inside the Cu matrix. The corresponding composition was analyzed using an EDX spectrum on TEM. As shown in Fig. 6(b), in addition to the significantly strong Cu signal, only the Cr signal can be identified. The Zr signal is probably too weak and submerges into the background signals due to the small content in the observed zone.

Figure 7 shows the TEM morphologies in the transverse direction and the corresponding electron patterns of the CuCrZr alloys treated at 300 °C with elongations of 5% and 22%, respectively. The precipitated particles under the small deformation condition demonstrate sphere-shaped morphology on the matrix, as shown in Fig. 7(a). The corresponding electron diffraction pattern shows extra reflection spots other than those of the matrix. It is supposed that the precipitated particles would be predominately incoherent Cr-rich particles, for the reason that they are commonly observed in such an alloy [6,14–16]. The further indexes of these Cr-rich phase reflections reveal that its electron diffraction evidence is consistent with the crystal structure of the phase belonging to the intermetallic

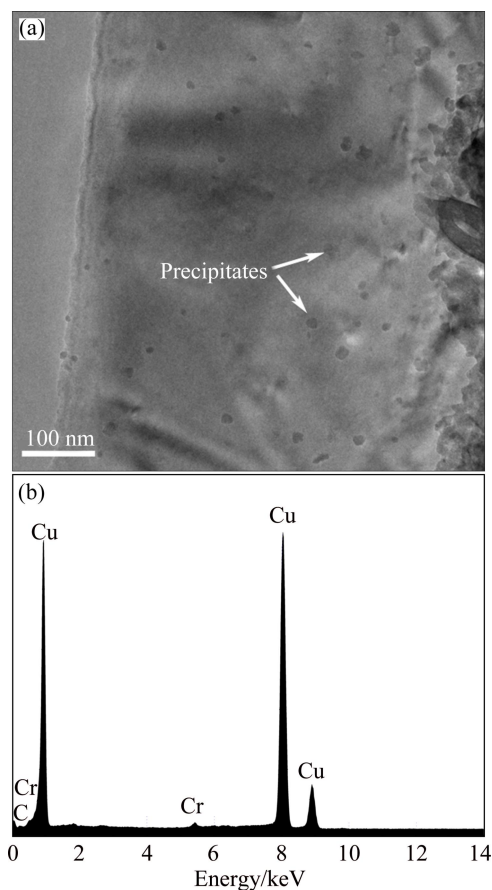


Fig. 6 TEM bright field image (a) and EDX spectrum (b) of solid solution treated sample

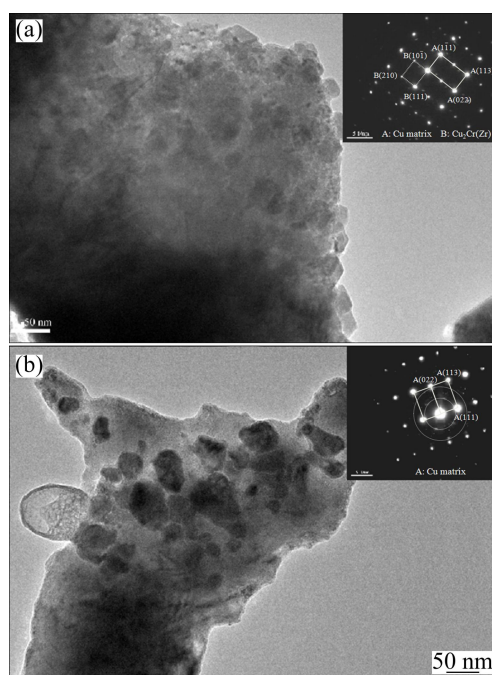


Fig. 7 TEM morphologies in transverse direction and corresponding electron patterns of CuCrZr alloy treated at 300 °C with elongations of 5% (a) and 22% (b)

compounds, known as the Heschel phase. For the CuCrZr alloy in this work, the composition of the precipitate is likely to be CrCu_2 . However, it should be postulated that Cr and Zr could be enriched together, thereby easily forming the intermetallic phase of Cr and Zr. In spite of no Zr signal trace in the TEM–EDX examination, the SEM–EDS and the direct-reading spectrograph analyses have clearly reveal the existence of Zr. Therefore, the precipitate phase could be attributed to $\text{CrCu}_2(\text{Zr})$, whose orientation relationship is expected to obey the Nishiyama–Wasserman (N–W) relationship with the face centered cubic (FCC) matrix [6,17,20,21].

In contrast to the morphology of the sample treated at 300 °C with the elongation of 5%, the sample treated at the same temperature with larger elongation of 22% demonstrates a larger particle size of more than 50 nm and a higher precipitated particle density. Its electron diffraction pattern is similar to that of the sample with a small elongation. However, in addition to the bright scattered reflection spots from the matrix, the extra reflection spots demonstrate a diffusion appearance, suggesting an ultrafine grain structure. Moreover, the extra reflection spots appear more complicated, suggesting that they may consist of several precipitate phases. Similar results have been reported in the CuCrZrMg alloy [6].

Figure 8 shows the EDS linear scanning spectrum crossing the two precipitated particles. The results reveal that the precipitated particles were composed of Cr-containing phase.

4 Discussion

Cu–Cr system alloy doped, with a small amount of other elements, usually demonstrates typical precipitation-hardened characteristics. The precipitation-hardening effect depends on the composition, morphology and distribution of Cr-rich precipitates

dissolved from the supersaturated solid solution [11,12]. In order to increase the nucleation sites and diffusion paths for dissolved solute atoms during the aging process, cold deformation is often carried out prior to aging in the conventional treatments for precipitation hardening alloys. However, these separated processes would result in discontinuous manufacture and thus increase the manufacturing cost. Moreover, the rather long aging time hinders the increment of manufacturing productivity. In this work, the thermal stretch treatment presents the potential to combine the deformation and aging processes simultaneously. The investigated results show that the thermal stretch treated CuCrZr alloys have large hardening responses with an acceptable electrical conductivity reduction. Good combinations of high strength and high conductivity are achieved after proper treatment. Therefore, this process is effective and practical for the manufacture of precipitation-hardened alloys. Refined grains are found after the treatment, which may provide good microstructure preparation for further treatments.

Alloy strengthening is usually associated with the heteroatom solution, dislocation increment, grain refinement and dispersive precipitation effect. The Cu lattice shrinkage inferred from the XRD diffraction patterns indicates that the Cr (or Zr) atoms precipitated from the matrix after the thermal treatment, which would result in the reduction in solution strength effects and thereby the reduction in microhardness. Due to the decrease of the scattering conducting electron of the heteroatom, the precipitation of Cr (or Zr) from the matrix would contribute to the increase of electrical conductivity [22].

Figure 9 shows the typical average grain size via the tensile elongation at 300 °C under thermal stretch condition. The average grain size was evaluated from the metallographic observations. From Fig. 9, the average grain size decreases with increasing the tensile

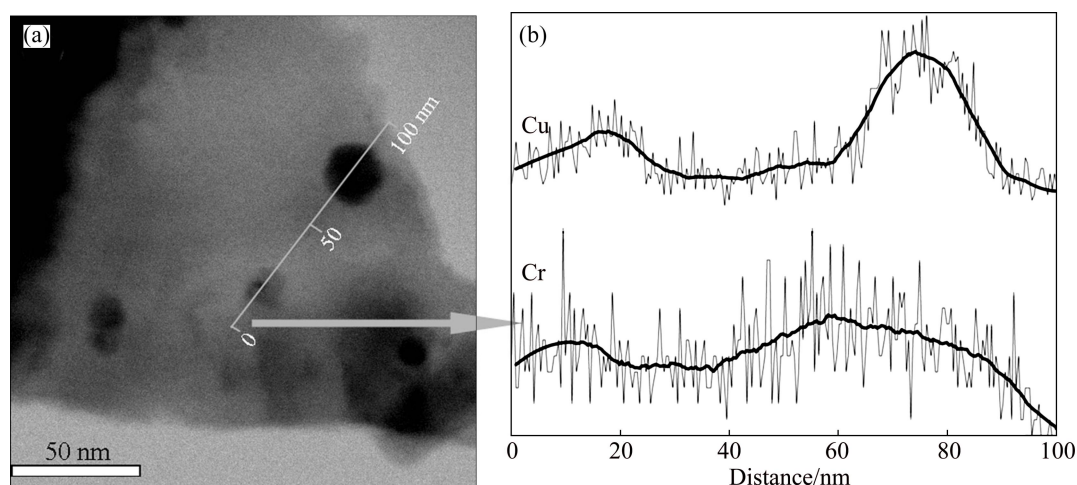


Fig. 8 TEM image (a) and EDS linear profile (b) near precipitates of CuCrZr sample treated at 300 °C with elongation of 22%

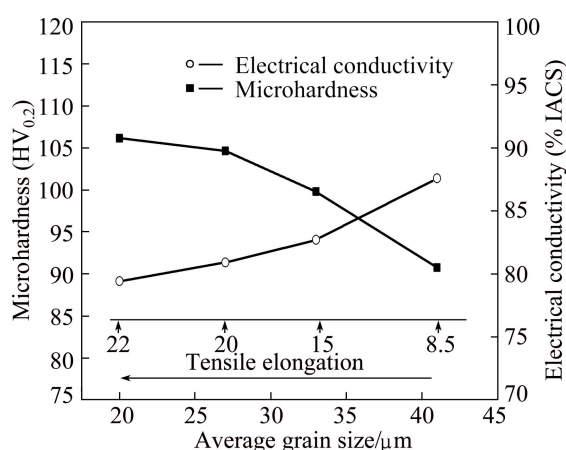


Fig. 9 Relation of microhardness and electrical conductivity versus average grain size for CuCrZr alloy treated at 300 °C

elongation, which would contribute the increase of the microhardness to the grain refinement effect. Correspondingly, the grain refinement effect would result in a slight decrease of electrical conductivity because of the increase of scattering surface for the conducting electron [23].

With regard to the variation in dislocation density, the cold deformation could introduce a high density of defects such as dislocations and vacancies. However, since the deformation was performed at a relatively high temperature, dynamic recovery or even recrystallization may appear, which would limit the strengthening effect and result in a slight increase of electrical conductivity.

Precipitation strengthening would play an important role in the evolution of microhardness and electrical conductivity via tensile elongations. The TEM observations reveal that the density and size of the precipitated particles increase with increasing the tensile elongations. The large deformation is caused by the thermal stretch treatment, the high density of produced defects, which would provide more diffusion paths for solute atoms, and the nucleation size of the precipitate during the aging treatment and thereby results in the strong precipitation-hardening effect. Moreover, the main precipitate phase of $\text{Cu}_2\text{Cr}(\text{Zr})$, which has been reported to have a high hardness, could strongly inhibit the movement of dislocation and enhance the micro-hardness [6]. Due to the scattering of the conducting electrons by the dissolved solute atoms, the precipitation strengthening could cause a little decrease of conductivity.

In summary, the combination effect of the heteroatom solid solution, dislocation increment, grain refinement and dispersive precipitation effect, results in an increase of microhardness and a slight decrease of electrical conductivity with the increase of tensile elongations.

5 Conclusions

1) The thermal stretch process has been successfully developed to manufacture the precipitation hardening CuCrZr alloys. The process is effective and practical in manufacturing the alloys with good combination of strength and conductivity.

2) By increasing the tensile elongations of CuCrZr alloy at each temperature from 100 to 300 °C, the microhardness increases, whereas the electrical conductivity decreases slightly.

3) After thermal stretch treatment, $\text{CrCu}_2(\text{Zr})$ phase precipitates from the supersaturated solid solution, which has a Nishiyama–Wasserman orientation relationship to the matrix.

4) The achievement of high strength and high electrical conductivity in the thermal stretch treated alloys is ascribed to the interactions of the heteroatom solution, dislocation increment, grain refinement and dispersive precipitation.

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热拉伸处理 CuCrZr 合金的 力学性能、导电性能和显微组织

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摘 要: 采用热拉伸方法在 100 至 300 °C 温度范围内对 CuCrZr 合金进行处理, 实验结果表明: 热拉伸处理工艺能成功地制备具有高硬度和一定导电能力的 CuCrZr 合金。在不同的处理温度下, 随着延伸率的增加, 所得材料的显微硬度均有所增加, 而导电性均有一定程度的降低。通过 TEM 观察到大量的含 Cr 沉淀相, 且这些沉淀相与 Cu 基体之间存在 Nishiyama–Wasserman 位向关系。热拉伸处理后的 CuCrZr 合金的高显微硬度和可接受的导电性归因于固溶原子析出、位错增殖、晶粒细化以及沉淀强化的共同作用。

关键词: CuCrZr 合金; 热拉伸处理; 显微硬度; 导电性能

(Edited by Mu-lan QIN)