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Understanding the effect of aging treatment on the electrical properties of Al-4Cu (wt.%) alloy

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Abstract
Binary Al–Cu alloy has always been used to illustrate the phenomenon of precipitation hardening. Meanwhile, Al–Cu alloy has good heat resistance and is a potential heat-resistant conductor material, but there is not much research work on its electrical properties. In this work, the residual resistivity variation of Al-4Cu alloy at different aging stages was investigated. At the aging temperature of 160 °C, the residual resistivity of the Al-4Cu alloy decreases with the longer aging time, and it decreases rapidly in the early stage and slowly in the later stage, mainly due to the precipitation of solid solution Cu atoms and the interface electron scattering effect of different precipitates. In the early stage, massive precipitation of Cu atoms from the matrix can effectively reduce the crystal lattice distortion of Al matrix and significantly decrease the residual resistivity. Specifically, the residual resistivity of Al-4Cu alloy that aged at 160 °C for 13 h is 3.353 μΩ·cm, which is only 82.6% of that in the supersaturated solid solution state. In later stage of aging, the main change is the evolution of the precipitated phase, i.e., the gradual disruption of the coherent relationship between the second phase and the Al matrix. And the coherency-strain fields decrease gradually, which is also beneficial in decreasing the residual resistivity. The results show that Al–Cu alloys can also be a potential heat-resistant conductor material for the power transmission industry.

1. Introduction

As a heat-treatable alloy, Al–Cu alloy has been wildly used in automobiles, aerospace, and even the military industries due to the amazing combination of low density, high specific strength, and good machinability [1–4]. Binary Al–Cu alloys have always been a textbook alloy system to illustrate the phenomenon of precipitation hardening, including the precipitation sequence and kinetics [5–8] and the interface energy between the Al matrix and the second phase [3, 9–13]. Most of the research works related to Al–Cu alloys have focused on microstructural changes and the improvement of mechanical properties based on microalloying, processing technology, and different heat treatments. For instance, the addition of trace alloying elements, such as Cd, Sn, In, Ag, or Au can effectively obtain higher strength because these trace elements have an essential effect on the precipitation process of Al–Cu alloys, i.e., inhibiting the formation of Guinier-Preston (GP) zones and promoting a more refined and uniform distribution of θ′ phases [3, 9, 12–15]. Severe plastic deformation techniques develop rapidly to obtain ultrafine-grained alloys, which possess high strength and elongation [16–18]. Multi-stage aging and post-deformation heat treatments are also being used frequently to improve the mechanical properties of Al–Cu alloys [19–21].

Meanwhile, with the development of the power transmission industry, the demand for extreme use environments such as high temperatures are gradually increasing, which poses new challenges to traditional conductive materials. The current research on heat-resistant conductive aluminum alloys is mainly focused on microalloying with the addition of Ce, Sc, Zr and other elements, but the high price of these elements limits their use [22–25]. The Al–Cu alloy itself has good heat resistance properties and can maintain excellent mechanical properties below the service temperature range of 200 °C [26–29]. The room-temperature and high-
temperature mechanical properties of Al–Cu alloys have been well studied [1, 30], but not much research work has been done on their electrical properties. In this research work, we have taken Al–Cu alloy as an example and studied the variation of its residual resistivity during the isothermal aging process systematically. The results show that with proper adjustment of the aging process, Al–Cu alloy can have good electrical properties while ensuring certain mechanical properties, and is a potential heat-resistant and conductive material [31, 32].

In the process of aging treatment, the solid solution degree of Cu atoms and the nature of the matrix/precipitate interface (coherent, semi-coherent, or incoherent) are the main influence factors of the change in the residual resistivity of Al–Cu alloy. Revealing the change law and influence mechanism of electrical properties for Al–Cu alloy during aging treatment is of importance to the design of new heat-resistant conductor material that based on Al–Cu alloys.

2. Materials and methods

99.7% pure aluminum and Al–50Cu master alloy were added in a graphite crucible to fabricate Al–4Cu alloy, and the melting temperature of the electrical furnace was maintained at 740 ± 2 °C. After the alloys were totally dissolved, 0.5wt% C2Cl6 was added to the melt to minimize the hydrogen absorption. Then the molten alloy was subjected to ultrasonic treatment for 20 min. Finally, after slag removal treatment, the molten alloy was cast into a steel mold with a plate-like cavity pre-heated to 200 °C, subjected to ultrasonic treatment for 20 min. Finally, after slag removal treatment, the molten alloy was cast into a steel mold with a plate-like cavity pre-heated to 200 °C. It is important to note that the entire melting process is under a high purity argon gas atmosphere to avoid the gas contamination of O2, N2, and H2O from the air.

Solution heat treatment of the casting alloy plates (12 h at 540 °C) was performed in a muffle furnace and water quenched. Finally, the samples were subjected to different heat treatments, such as natural aging (NA, 168 h at 25 °C), artificial aging (AA), and high-temperature artificial aging treatment (20 h at 280 °C). The parameters for artificial aging treatment are at 160 °C for various aging times (3, 5, 10, 13, 25, 30, 72, and 120 h, respectively).

All specimens used for characterization and measurement were air-cooled to room temperature following the aging treatment. The samples used for metallographic observations were etched at room temperature for 10 s in Keller’s reagent (a mixture containing 1 ml of HF, 1.5 ml of HCl, 2.5 ml of HNO3, and 95 ml of H2O), and the corresponding grain size distribution data were obtained using Image-pro Plus 6.0 software with reference to the GB T 6394–2017 standard. The chemical compositions of the studied alloy were measured by spark direct-reading spectrometer (CX-9800) under adequate argon gas pressure. The temperature of the measurement chamber is maintained at 34 ± 0.2 °C, and its vacuum is maintained at 7.5–17 Pa using a vacuum pump to guarantee the normal excitation of high-frequency electric sparks. The Vickers hardness values of the as-aged and the as-cast alloys were measured at room temperature with a load of 0.3 kg and a dwell time of 10 s. And the Vickers hardness values of at least five different locations are averaged as the hardness value of the sample to increase data reliability. The residual resistivity of the alloy (in a 20°*20°*10 mm plate) was measured by an eddy current conductivity meter (PZ.60A). Microstructures were characterized using a Phenom Pharo Desktop scanning electron microscopy (SEM) in backscatter electron (BSE) mode, an accelerating voltage of 15 kV and effective working distance of 13 mm were used. Precipitates inside the grain were further analyzed using transmission electron microscopy (TEM, FEI Talos F200X) with an accelerating voltage of 200 kV, and the image analysis was performed using Gatan DigitalMicrograph software. Meanwhile, differential scanning calorimetry (DSC) was performed on DSC 200PC with a heating rate of 10 °C min−1, and the test temperature range was set to 25 ~ 500 °C. The phase identification was performed using Shimazu XRD-6100 diffractometer with Cu-Kα radiation (λ = 1.5418 Å). The range of 2θ was from 10° to 95° and the scan speed was controlled at 2° min−1.

3. Results and discussion

The main chemical compositions of the studied alloy are listed in Table 1. It is close to the nominal design composition of Al–4Cu (wt.%) alloy.

Figure 1 shows the metallographic photo of the as-cast Al–4Cu alloy. Relatively uniform equiaxed grains are obtained. The illustration in the upper right corner shows its average grain size is about 125 microns. The reason may be that the ultrasonic treatment was used during alloy smelting. When the alloy melt is subjected to high-power ultrasonic treatment, a large number of microcavities will be produced, namely the cavitation effect. After

| Alloy | Cu | Si | Fe | Al |
|-------|----|----|----|----|
| %     | 4.01 | 0.09 | 0.10 | Balance |
the formation of microcavities, it will expand rapidly, at the same time, the temperature on the surface of the microcavity decreases rapidly, providing undercooling for melt nucleation [33]. Therefore, the cavitation effect caused by ultrasonic vibration makes a large number of nucleation cores appear in the melt, which promotes the formation of equiaxed crystals in the alloy.

For Al–Cu alloy, GP zones are the first precipitate to nucleate when the alloy is aged at room temperature or a temperature below the GP zones solvus. GP zones with a disc-shape are fully coherent with the matrix, and these zones are usually about 2 atomic layers thick and 10 nm in diameter. Another two transition phases are $\theta'$ and $\theta''$, respectively, where plate-like $\theta'$ precipitates are fully coherent with the matrix and are about 10 nm thick and 100 nm in diameter. The thick $\theta'$ plates are coherent with the Al matrix only in its broad face, their thickness and diameter can reach 15 nm and 1 $\mu$m, respectively. As for the equilibrium $\theta$ phase, it has a complex body-centered tetragonal (BCT) structure and remains incoherent with the matrix, and these large size precipitates possess a coarse distribution in the matrix [12, 18].

The types of metastable precipitated phases after aging treatment in this investigation are determined by thermal analysis [34] and microstructure identification.

Figure 2(a) shows the Al-rich corner of the equilibrium Al–Cu phase diagram and presents the metastable solvus boundaries of GP zones, $\theta'$, $\theta''$ and the equilibrium $\theta$ phase. For Al-4Cu alloy, the dissolution temperature
for the metastable GP zone, $\theta'$, $\theta'$ and $\theta$ is around 160 °C, 250 °C, 350 °C, and 450 °C, respectively [35]. Figure 2(b) shows the DSC curve of solution treated Al–4%Cu alloy in a supersaturated solid solution (SSSS) state, there find eight peaks, i.e., four exothermic peaks (A, B, C, D) and four endothermic peaks (E, F, G, H). The four exothermic peaks are ascribed to the formation of the GP zones (A), $\theta'$ (B), $\theta'$ (C) and $\theta$ phase (D), and the four endothermic peaks are related with the dissolution of GP zones (E), $\theta'$ (F), $\theta'$ (G), and $\theta$ phase (H). The above result means solution treated Al–4%Cu alloy has undergone a complete precipitation sequence during the heating process, namely SSSS $\rightarrow$ GP zones $\rightarrow$ $\theta'$ $\rightarrow$ $\theta'$ $\rightarrow$ $\theta$. If aging is carried out at a temperature above the GP zones solvus but below the $\theta'$ solvus, the first precipitate will be $\theta'$. If aging is carried out at higher temperature for enough time, the only precipitate existed is $\theta$. Conversely, if an alloy containing GP zones is heated to above the GP zone solvus the zones will dissolve and transform to $\theta'$, $\theta'$, and eventually to $\theta$ if the heating temperature is high enough or after enough aging time.

Compared with the solution treated alloy (figure 2(b)), peak A was not found in the DSC curve of the naturally aged specimen (figure 2(c)), in which the first peak observed (E) corresponded to the dissolution of the GP zones, implying these precipitates have already existed [36]. Similarly, the first obvious peak observed in figures 2(d) and (e) is ascribed to the dissolution of $\theta'$ (F) and $\theta'$ (G), respectively, indicating the main precipitation phases in Al–4%Cu alloy under the artificial aging at 160 °C for 13h and 72 h are $\theta'$ and $\theta'$ phases, respectively. The microstructure features of the Al–4%Cu alloy at different aging stages are marked in the curves of microhardness and residual resistivity in figure 3, and the main precipitation phases at different aging stages are summarized in table 2.

The evolution of Vickers hardness value and the residual resistivity of Al–4%Cu alloy as a function of the artificial aging time are shown in figure 3. The Vickers hardness value increases rapidly with the increase of aging time in the initial stage. The maximum Vickers hardness value (about 105 HV0.3) was obtained in the peak aging stage, corresponding to the aging time of about 13 h. Afterwards, the Vickers hardness value abruptly decreases with aging time increasing up to 30 h and slightly drops with the further extension of the aging time. The last corresponds to the over-aging stage of Al–4%Cu alloy. The above is the representative age-hardening response of the Al–Cu alloys. Different from the change of microhardness value, after longer aging times, the residual

**Table 2. Summary of the data of microhardness and residual resistivity, as well as the main precipitation phases of Al–4%Cu alloy at different aging stages.**

| Aging time/h | Corresponding alloy state           | Increase of microhardness/HV0.3 | Decrease of residual resistivity/μΩ·cm | Main precipitation phases |
|--------------|------------------------------------|---------------------------------|----------------------------------------|--------------------------|
| 0            | SSSS                               | —                              | —                                      | —                        |
| 1–12         | Under-aging                        | —                              | —                                      | GP zones $+\theta'$      |
| 13           | Peak-aging                         | 37.32                          | 0.704                                  | $\theta'$                |
| 14–30        | Over-aging                         | 17.38                          | 0.871                                  | $\theta'$ $+\theta'$    |
| 30–120       | Over-aging                         | 3.8                            | 0.966                                  | $\theta'$                |
| —            | Natural aging                      | 23.38                          | 0.277                                  | GP zones                 |
| —            | High-temperature aging             | —1.72                          | 0.978                                  | $\theta$                |

Figure 3. Evolution of the microhardness and the residual resistivity of the Al–4%Cu alloy as a function of aging time at 160 °C (artificial aging). Error bars stand for the standard deviation corresponding to at least 5 tests.

![Graph showing Vickers hardness and residual resistivity with aging time.](Image)
The electrical resistivity of the Al-4Cu alloy firstly decreases rapidly, and then the decreasing rate slows down. Finally, at the aging time of 120 h, the minimum residual resistivity value of the Al-4Cu alloy about 3.091 $\mu\Omega\cdot\text{cm}$ can be obtained. Besides, in order to clarify the influence of the precipitate phase type on the residual resistivity of Al-Cu alloys, natural aging (168 h at 25 °C) and high-temperature artificial aging (20 h at 280 °C) were conducted to get the expected GP zones and $\theta$ phases, respectively. The corresponding microhardness and residual resistivity values are $90.7 \pm 1.63$ HV0.3, $3.78 \pm 0.064 \mu\Omega\cdot\text{cm}$ for the natural aging sample, and $60.20 \pm 0.353$ HV0.3, $3.079 \pm 0.052 \mu\Omega\cdot\text{cm}$ for the high-temperature artificial aging sample. The residual resistivity of the latter is nearly the same as that of AA at 160 °C for 120 h.

The changes in microhardness and the residual resistivity (figure 3) during isothermal aging treatment are summarized in table 2. The aging time 0 h corresponds to the SSSS state of the Al-4Cu alloy, the aging time 13 h corresponds to the peak aging state of the Al-4Cu alloy, the under-aging state of the alloy is from 0 h to 13 h, and the over-aging state of the alloy is after 13 h. Compared to the solution treated Al-4Cu alloy, the microhardness of the alloy in the peak aging state increased by 37.32 HV0.3 to about 105 HV0.3, with a corresponding decrease in residual resistivity of 0.704 $\mu\Omega\cdot\text{cm}$. The aging time of 30 h and 120 h corresponded to different stages of the over-aging state, with an increase in microhardness of 17.38 HV0.3 and 3.8 HV0.3 and a corresponding decrease in residual resistivity of 0.871 $\mu\Omega\cdot\text{cm}$ and 0.966 $\mu\Omega\cdot\text{cm}$, respectively.

Electrical properties are sensitive to microstructure. The electrical properties of the alloy are affected by precipitates and the defects such as grain boundaries, dislocations, solid atoms. In addition, the average free path of electrons is larger parallel to the preferential orientation, making the corresponding resistivity of the alloy is smaller than that of other orientation. According to the well-known Matthiessen’s rule [18, 33], $\rho_{\text{total}} = \rho + \rho_a + \rho_p + \rho_b + \rho_{gb}$, where $\rho_{\text{total}}$ is the total electrical resistivity of alloy, $\rho$ is the electrical resistivity of the lattice, $\rho_a$ is the electrical resistivity added by solute atoms dissolved in the matrix, $\rho_p$ is the electrical resistivity caused by second-phase precipitates, $\rho_b$ is the electrical resistivity of the dislocations that existed in the microstructure, and $\rho_{gb}$ is the electrical resistivity of the grain boundaries. Considering that the alloys have similar grain size (figure 1) and dislocation density under different heat treatments in this study, it can be concluded that the difference in the electrical resistivity is mainly due to the solid solution degree of Cu atoms in the Al matrix and the existence of different types of precipitated phases. And among the defects, the solute atoms in the Al matrix are recognized as having the most harmful effect on electrical properties [37].

Figure 4 shows the HAADF-STEM imaging of Al-4Cu alloy artificially aged at 160 °C for 10 min. Because the aging time is very short, only a few precipitates are found inside grain with a size of about 20 nm. Only a few Cu atoms precipitate to form the second phase, and Cu atoms are mainly in solid solution since the alloy is in the early stage of artificial aging. Considering the size and shape of the precipitates in figure 4 (a) and (b), they can be inferred as G.P. zones, which share a perfect coherent interface with the matrix. At this time, the resistivity of the alloy (4.03 ± 0.057 $\mu\Omega\cdot\text{cm}$) is only slightly reduced compared with that of solution treated alloy.

As shown in figure 5, the $\theta'$ phases are also identified in Al-4Cu alloy aged at 160 °C for 30 h by the HAADF-STEM micrographs and corresponding selected area electron diffraction (SAED). After artificial aging at 160 °C for 30 h, the $\theta'$ phases are uniformly distributed inside the alloy (figure 5(a)). The length of some $\theta'$ phases exceeds 1 $\mu$m, which indicates that the alloy was already in the over-aging stage. The high-resolution TEM images of $\theta'$ phases were shown in figure 5(c), and the $\theta'$ phase is completely coherent with the Al matrix in the...
broad face. Both the SAED (figure 5(b)) and the FFT patterns (figure 5(d)) appear two sets of diffraction spots, where the red parallelogram represents the spot of the $\theta'$ phase, and the yellow Square represents the spot of Al matrix. Further analysis indicates the orientation relationship between the $\theta'$ and the Al matrix is $[001]_{\theta'} // [001]_{\alpha}$ and $[100]_{\theta'} // [100]_{\alpha}$. Compared with the microstructure of the alloy artificially aged for 10 min (figure 4), plenty of mutually perpendicular $\theta'$ phases precipitated inside the alloy after 30 h artificially aging. The $\theta'$ plates are obviously larger than G.P. zones and $\theta'$ in size being up to $\sim$10 nm thick and 1 $\mu$m in diameter, but only share a coherent interface with the matrix in its broad face. Therefore, the coherency-strain fields at the coherent interface are greatly reduced, corresponding to a smaller resistivity ($3.17 \pm 0.045 \mu \Omega \cdot \text{cm}$).

After aging at 280 °C for 20 h, the severely coarsened precipitates (figure 6) are visible by scanning electron microscopy (SEM), a large number of $\theta$ phases are found in the crystal and the grain boundary. In this case, the alloy is close to the annealed state.

Figure 7 shows the XRD curves and the corresponding lattice constants of Al-4Cu alloy under different heat treatments. According to the famous Bragg formula, namely $2d \sin \theta = n \lambda$, and the calculation formula of the interplanar spacing of the cubic crystal system ($d_{hkl} = a/\sqrt{h^2 + k^2 + l^2}$), the lattice parameters of the alloy can be obtained and is shown in figure 7(b).

The lattice constant of Al will be reduced when Cu atoms are dissolved into the Al matrix due to the smaller diameter of the Cu atom compared with that of the Al atom. Figure 7(b) shows that the lattice constant is the smallest ($a = 0.40364 \text{ nm}$) when the alloy is in the SSSS state because all Cu atoms are dissolved into the Al matrix. While, with the aging treatment, the dissolved Cu atoms will gradually precipitate from the Al matrix, so
the lattice constant will increase progressively. Artificial aging treatment will accelerate the precipitation rate of Cu atoms compared with natural aging treatment. Therefore, as figure 7(b) shows, AA samples show a larger lattice constant value. When the artificial aging time is greater than 13 h at 433K, it is noteworthy to note that the lattice constant is almost the same as that of pure aluminum, indicating that most of the Cu atoms in solid solution already precipitate out of the Al matrix after peak aging treatment, concentrating on the $\theta'$ phases rather than in the Al matrix [3, 12] (figure 7). With the longer aging time (greater than 13h), the major occurrences are the evolution and growth of precipitation phases, rather than Cu atoms precipitate from the Al matrix. Figure 8(a) high magnification micrographs of $\theta'$ in Al-4Cu samples aged at 160 °C for 30 h and its corresponding EDS mapping (c)–(d) [3, 12].

The essence of metal conduction is the directional movement of free electrons, so all factors that hinder the directional movement of free electrons will increase the resistivity of alloy. Defects, precipitates and strain fields all hinder the movement of free electrons, i.e., electron scattering effect.

In the initial aging stage (namely 0–13h, figure 3), Cu atoms change from solid solution state into precipitation state, leading to the precipitation of GP zones and $\theta'$ phases and reducing the lattice distortion of $\alpha$-Al matrix, the reduction of solid solution Cu atoms plays a dominant role in reducing the residual resistivity. The decrease of the residual resistivity caused by the precipitation of solid solution Cu atoms is much larger than the increase of the resistivity caused by the generation of the GP zones or $\theta'$ phase, so the overall residual resistivity of the alloy is still significantly reduced. After peak aging (13–120h, figure 3), most of the Cu atoms have already precipitated out of the Al matrix, and the main change in this period is the evolution of the precipitated phase, i.e., $\theta' \rightarrow \theta'$. $\theta'$ precipitates are fully coherent with the matrix and the thick $\theta'$ plates are only coherent with the matrix only in its broad face, the coherency-strain fields that greatly impede electron transmission appear since...

Figure 6. High magnification SEM micrographs of the as-aged Al-4Cu alloys after aging at 280 °C for 20 h.

Figure 7. XRD curves of alloys (a) and the corresponding lattice constants (b) under different heat treatment regimes. The dotting line in (b) represents the lattice constants of pure aluminum.
the coherency misfit between the precipitates ($\theta'$ or $\theta''$) and matrix. Free electrons tend to pass through Al matrix while some free electrons were scattered by precipitates and its surrounding strain fields during electron transport process. The transformation from $\theta'$ to $\theta''$ decreased the electron scattering probability because the coherent relationship is gradually broken, more free electrons can go through Al matrix and the corresponding electron flow passing rate increased. Therefore, the residual resistivity of the Al-4Cu alloy at the later stage of artificial aging decreases slightly.

Table 3 lists the results of recent studies on the electrical properties and strength (microhardness) of Al–Cu and Al–Si based alloys, from which it can be seen that the residual resistivity of deformed Al–Cu based alloys is much lower than that of undeformed Al–Cu alloys for a small difference in microhardness. As for the common Al–Si based conductive aluminum alloy, although its residual resistivity is at a low level, its absolute strength is insufficient,
and the processing method is complicated. As for the Al–4Cu alloy in this study, by controlling its aging process reasonably, its residual resistivity is also at a low level under the condition of having a certain strength, which reconciles the inverse relationship between electrical properties and strength relatively well. This indicates that the Al–Cu alloy is also a potentially excellent conductive material under reasonably controlled heat treatment.

4. Conclusions

The residual resistivity of the Al–4Cu alloy decreases with longer artificial aging time, and it decreases rapidly before peak aging stage and slowly in the later stage. In the initial artificial aging stage, massive precipitation of Cu atoms from the aluminum matrix can effectively reduce the lattice distortion of the Al matrix, which can effectively lessen the electron scattering and significantly decrease the residual resistivity. After peak aging, most of the solid-solution Cu atoms have already precipitated out of the Al matrix. The change of residual resistivity is mainly determined by the precipitation phase, after longer aging time, the coherent relationship between the precipitated phase and the Al matrix is gradually broken, decreasing the amount of the coherency-strain fields. And the number of precipitated phases is gradually reduced, consequently, the residual resistivity of the Al–4Cu alloy at the later stage decreases slightly.

Understanding the changes in residual resistivity of the Al–Cu alloys during aging treatment and their intrinsic mechanisms is of great significance for the design of heat-resistant conductive aluminum alloys that based on heat-treatable strengthened alloys.

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Declaration of competing interest

The authors formally announced that they have no known correlative financial interests or personal relationships that could influence this research work.

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