Precipitation Behavior of Cu-1.9Be-0.3Ni-0.15Co Alloy During Aging

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Abstract In this paper, the evolutions of microstructure and mechanical properties of Cu-1.9Be-0.3Ni-0.15Co alloy were studied. The alloys in the condition of the solution treated (soft state) and 37% cold rolled (hard state) were aged at 320 °C for different time, respectively. The mechanical properties, electrical conductivity and microstructure of the alloy aged for different time were analyzed. Additionally, the precipitation kinetics of Cu-1.9Be-0.3Ni-0.15Co alloys was investigated. X-ray diffraction and transmission electron microscopy results reveal that both continuous precipitation and discontinuous precipitation existed in the hard-state Cu-1.9Be-0.3Ni-0.15Co alloy during the whole aging process; the sequence of continuous precipitation is G.P. zone $\rightarrow \gamma'' \rightarrow \gamma' \rightarrow \gamma$. Furthermore, the precipitation transformation mechanism of soft-state alloy is homogeneous nucleation, while hard-state alloy shows the heterogeneous nucleation (interface nucleation) with the nucleation rate of both states decaying rapidly to zero during aging at 320 °C.

KEY WORDS: Cu-1.9Be-0.3Ni-0.15Co alloy; Aging; Microstructure; Mechanical property; Electrical property; Precipitation kinetics

1 Introduction

Beryllium copper is a typical precipitation hardened alloy which has received great attention due to the high strength and high elasticity but little elastic hysteresis after deformation aging treatment. Furthermore, it also possesses the unique properties of high corrosion resistance, high fatigue resistance, non-magnetic and no sparks when bearing collision, etc. [1–3]. Because of these attractive advantages, beryllium copper has been widely applied in production of

significant elasticity and wearable parts, such as electronic components of automobile engine control system, spring, contactors and switches in computers, etc. [4–6].

Over last decades, substantial works have theoretically investigated the precipitation behavior of beryllium copper during aging process. Watanabe et al. [7] indicated that the precipitation sequence of Cu-1.9Be-0.3Ni alloy is G.P. zone $\rightarrow \gamma'' \rightarrow \gamma' \rightarrow \gamma$. According to the report of Monzen et al. [8], the precipitation sequence of Cu-1.2Be-0.1Co alloy during 320 °C aging is G.P. zone $\rightarrow \gamma'' \rightarrow \gamma' \rightarrow \gamma$, and the γ' phase heterogeneous nucleates on γ'' phase. The orientation relationships between γ'' phase and Cu matrix are $\{001\}_{\alpha} // \{001\}_{\gamma''}$ and $\langle 110 \rangle_{\alpha} // \langle 100 \rangle_{\gamma''}$. The orientation relationships between γ' phase and Cu matrix are $\{112\}_{\alpha} // \{012\}_{\gamma'}$, $\{113\}_{\alpha} // \{013\}_{\gamma'}$ and $\langle 110 \rangle_{\alpha} // \langle 100 \rangle_{\gamma'}$. Monzen et al. [9] have also studied the precipitation processes of Cu-0.9Be single crystal and found that the precipitation sequence of it is G.P. zone $\rightarrow \gamma'' \rightarrow \gamma_1' \rightarrow \gamma_1 + \gamma' \rightarrow \gamma$. The γ_1' phase is body-centered monoclinic structure with $a = b = 0.254$ nm and $c = 0.324$ nm and $\alpha = 86.8^\circ$.

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In applications, cold rolling prior to aging treatment is usually necessary for beryllium copper, but most of those researches mentioned above concentrate on the aging process of undeformed beryllium copper after solution treatment. This study mainly focuses on the precipitation process of cold rolled Cu-1.9Be-0.3Ni-0.15Co alloy during aging at 320 °C.

2 Experimental

The beryllium copper alloy used in the experiment was Cu-1.9Be-0.3Ni-0.15Co, which was hot rolled into plates after melting, casting and homogenizing. The plates were quenched online immediately after hot rolling, followed by annealing, cold rolling and solution treatment. The specimens showed two kinds of states after solution treatment: (1) soft-state specimens with the thickness of 0.27 mm (without cold rolling) and (2) hard-state specimens with the thickness of 0.17 mm (with cold rolling reduction of 37%). The rectangular specimens were cut from the plates which had been solution-treated and were put into tubular resistance furnace to carry out aging treatment. Argon gas was used as protective gas during aging at (320 ± 5) °C for 0.5–480 min.

The tensile properties and Vickers microhardness were tested by Germany FPZ100 universal material testing machine and 430SVD Vickers hardness tester, respectively. Resistance measurement was taken by ZY9987 digital micro-ohmmeter at room temperature, and then, the result was converted into conductivity. The solution-treated and aged specimens were analyzed by X-ray diffraction (XRD) on TTRIII multifunctional X-ray diffractometer with the scan angle range of 20°–120°, and the scanning rate was 10°/min. The microstructure of Cu-1.9Be-0.3Ni-0.15Co alloy was observed by the Cambridge S-360 scanning electron microscope (SEM) and the JEM-2010 high-resolution transmission electron microscope (TEM). TEM specimens were prepared by using a twin-jet polishing method with a solution of 70 vol% of methanol and 30 vol% of nitric acid at –20 to –25 °C; the employed voltage and current are 9.5 V and 20 mA, respectively.

3 Results

3.1 XRD Analysis

Figure 1 shows the XRD patterns of the alloy aged for different time. Compared to the pattern of solid-solution-treated alloy, new peaks representing precipitates were observed at 33.4° and 34.9° after aging treatment. Furthermore, the peak intensity and peak area of precipitation

increased with aging time. The mass fraction of precipitates in the alloy after aging for different time can be calculated by heat insulation method [10, 11]. If there are n phases in a system, the mass fraction of the i -th phase (W_i) is

$$W_i = \frac{I_i}{K_A^i \sum_{j=1}^n \frac{I_j}{K_A^j}} \quad (1)$$

where A is any selected phase in the alloy; i and j represent the i -th and j -th phases; I_i and I_j are the strongest peak area of the i -th and j -th phases; $K_A^i = R_i/R_A$, R_i and R_A represent the reflective intensity ratio (RIR) of the i -th phase and A phase, respectively.

The qualitative analyses of XRD patterns show that the main precipitates are CuBe, NiBe and CoBe, while the main precipitates should be CuBe precipitates according to chemical composition of the specimens. Thereby, the entire system can be regarded as containing Cu and CuBe only. With the RIR value and the strongest peak area of various phases after aging for different time, the mass fractions of precipitates in soft and hard-state alloys can be obtained, as shown in Fig. 2. For soft-state alloy, few precipitates were detected after aging for 30 min. The mass fraction of precipitates in soft-state alloy rose up to a considerable level after aging for 60 min, and the precipitation rate maintained a uniform level. The condition of the hard-state alloy was totally different, while numerous precipitates were detected after aging for only 30 min and the precipitation rate rose rapidly at the beginning then became slow gradually. A large amount of defects were induced by cold rolling which might work as the nuclei of precipitates and promoted the precipitation rate. Compared to hard-state alloy, there were much fewer defects in soft-state alloy, so the precipitation process depended greatly on homogeneous nucleation. As a result, the precipitation rate was much slower.

3.2 Microstructure Observation

The TEM images of hard-state specimens aged at 320 °C for 10 min are shown in Fig. 3. A striated microstructure presenting a sharp contrast between alternating light and dark can be observed. The striated microstructure is basically parallel to the $(\bar{1}\bar{1}\bar{1})_\alpha$ and coherent with the parent phase, according to selected area electron diffraction (SAED) results. In addition, the striated microstructure forms rapidly at the early stage of precipitation transformation and distributes uniformly. Therefore, it can be referred that the striated microstructure is G.P. zone formed at the early stage of precipitation transformation [12]. From Fig. 4, subtle granular precipitates which are coherent with the parent phase can be observed, presenting a strict

Fig. 1 XRD patterns of Cu-1.9Be-0.3Ni-0.15Co specimens aged for different time: **a** soft-state specimens; **b** hard-state specimens

Fig. 2 Fraction of precipitates in Cu-1.9Be-0.3Ni-0.15Co alloy aged for different time

orientation relationship with parent phase. SAED results show that the habit plane of precipitates is $(\bar{1}\bar{1}\bar{1})_{\alpha}$, indicating that the precipitates heterogeneously nucleate on G.P.

zone. Based on above results, it can be deduced that the subtle granular precipitates are γ'' phase [13]. The γ'' phase has a body-centered tetragonal (BCT) lattice with $a = b = 0.253$ nm and $c = 0.29$ nm and $\alpha = \beta = \gamma = 90^\circ$ [8]. Figure 5 depicts the TEM microstructure of hard-state specimens aged at 320°C for 120 min. The precipitates appear to be slender strips, with a clear direction of parallel to the $(21\bar{1})_{\alpha}$ and $(31\bar{1})_{\alpha}$, with the size of 5–15 nm and semi-coherent with the parent phase. On the basis of the above results, it can be judged that the precipitates are γ' phase [14]. According to Monzen *et al.* [9], γ' phase has a body-centered tetragonal (BCT) lattice with $a = b = 0.254$ nm and $c = 0.279$ nm. As shown in Fig. 6, a part of γ' phase transform to γ phase (equilibrium phase) and grow up in the way of spheroidization and aggregation, which is the symbol of over-aging phenomenon. The average size of γ phase in Fig. 6 is 230 nm, which is much larger than the metastable phase γ' . These results showed that the precipitation sequence of hard-state Cu-1.9Be-0.3Ni-0.15Co alloy was G.P. zone $\rightarrow \gamma''$ phase $\rightarrow \gamma'$ phase $\rightarrow \gamma$ phase and the precipitates coarsened and gathered with aging time.

Fig. 3 TEM image **a** and the selected area electron diffraction (SAED) pattern **b** of hard-state Cu-1.9Be-0.3Ni-0.15Co alloy aged at 320°C for 10 min

Fig. 4 TEM image **a** and the selected area electron diffraction (SAED) pattern **b** of hard-state Cu-1.9Be-0.3Ni-0.15Co alloy aged at 320 °C for 30 min

Fig. 5 TEM image **a** and the selected area electron diffraction (SAED) pattern **b** of hard-state Cu-1.9Be-0.3Ni-0.15Co alloy aged at 320 °C for 120 min

During aging process of beryllium copper, the precipitates are always prone to precipitate on the grain boundary of α phase [15], which will result in inhomogeneity of microstructure and properties, as shown in Fig. 7. The flake precipitates were similar to pearlite when observed by SEM. The secondary electron morphology and backscattered

Fig. 6 TEM image of hard-state Cu-1.9Be-0.3Ni-0.15Co alloy aged at 320 °C for 360 min

electron image of grain boundary precipitates are shown in Fig. 7. Backscattered electrons are sensitive to atomic number, and region with higher atomic number will present brighter contrast of backscattered electron image. Energy dispersive spectroscopy (EDS) results of grain boundary precipitates show that the grain boundary precipitates are compounds formed by Ni, Co and Cu, with the mass fraction of 13.7%, 6.2% and 80.1%, respectively. While the atomic number of both two other elements are lower than Cu, the grain boundary precipitates show dark black in the back-scattered electron image, which are distinguished from the gray matrix apparently. The volume fraction of grain boundary precipitates in Fig. 7c is 24.5%.

The discontinuous precipitation on grain boundary found in SEM microstructure of Cu-1.9Be-0.3Ni-0.15Co alloy could also be observed in TEM microstructure. Actually, this phenomenon can be explained as cellular reaction: This precipitation is a two-phase precipitation ($\alpha \rightarrow \alpha' + \gamma$), where α is supersaturated solid solution, γ is equilibrium precipitation phase, α' phase has the same crystal structure with α phase but the components of α' and α phase are different (α' phase is solute depleted) and they

Fig. 7 Grain boundary precipitates during aging process: **a** SE-SEM of grain boundary precipitates; **b** morphology of grain boundary precipitates with magnification of $\times 10,000$; **c** BE-SEM image of grain boundary precipitates

are separated by phase interface. During the discontinuous precipitation, the redistribution of solute is independent on volume diffusion of matrix but dependent on diffusion of interface [16].

The discontinuous precipitation (DP) cell on the grain boundary is shown in Fig. 8a. As shown in Fig. 8b, γ phase formed by discontinuous precipitation gradually gathers and grows up on the grain boundary with aging time. Then, DP cell appears inside grain, as shown in Fig. 8c. Coarse equilibrium precipitation phase will be obtained by discontinuous precipitation, and the metastable phase formed by the precious precipitation will dissolve accompanied by the promotion of cellular products, which will result in deterioration of mechanical properties.

3.3 Mechanical Properties

The tensile properties of the alloy after aging for different time are shown in Fig. 9. As for hard-state alloy, the yield and tensile strength increased with aging time and reached the peak value after aging for 120 min. The peak yield strength and tensile strength were 1,251 and 1,480 MPa, respectively. The alloy was over-aging as the aging treatment continued, resulting in the decrease in strength. For soft-state alloy, the yield strength and tensile strength are near the peak value after aging for 180 min then the strength increased slightly with aging time. The peak yield strength and tensile strength are 1,093 and 1,283 MPa,

respectively. The elongation of both state alloys decreased with aging time.

When the alloy is aged at the same temperature, the time required to reach peak aging is reduced and the peak tensile strength of the alloy is increased by cold rolling prior to aging treatment [14]. The time required to reach peak aging of hard-state alloy was shortened by 60 min, and the peak tensile strength showed increases of 200 MPa compared with the soft-state alloy.

As for hard-state alloy, the hardness increases with aging time and it reaches the peak value after aging for 120 min, then it begins to decrease, as shown in Fig. 10. For soft-state alloy aging at 320 °C, the hardness of specimens increases with time. The hardness reaches peak value after aging for 180 min and then increases slowly.

3.4 Electric Conductivity and Precipitation Kinetics

Compared with pure metal, the conductivity of solid-solution-state alloy will reduce owing to the distortion of the lattice of solvent and the destruction of the periodic of lattice potential field, which were caused by the dissolving of the solute atoms into the lattice of solvent. Thereby, the probability of electron scattering increases, resulting in the decrease in conductivity. Cold working will also make the conductivity of the copper alloy decrease because cold rolling will cause the distortion of lattice and increase the probability of electron scattering. Therefore, the

Fig. 8 Discontinuous precipitation of Cu-1.9Be-0.3Ni-0.15Co alloy aged for different time: **a** 15 min; **b** 120 min; **c** 360 min

Fig. 9 Tensile properties of Cu-1.9Be-0.3Ni-0.15Co alloy aged at 320 °C for different time: **a** hard state with thickness of 0.17 mm; **b** soft state with thickness of 0.27 mm

Fig. 10 Vickers hardness of Cu-1.9Be-0.3Ni-0.15Co alloy aged for different time

concentration of the solute atoms in copper matrix is the main factor which influences the conductivity of alloy. The increment in the concentration of solute atoms will cause stronger effect of electron scattering, leading to the decrease in conductivity. During the aging process, the solute atoms precipitated from matrix, resulting in the increase in conductivity. So the changes in conductivity could be used to study the precipitation regularity of alloy during aging. The conductivity of Cu-1.9Be-0.3Ni-0.15Co alloy increases with aging time during the aging process, as shown in Fig. 11. For soft-state alloy, conductivity rose slowly with time and the increasing rate was uniform; while the conductivity of hard-state alloy increased rapidly in the early stage of aging, then the increasing rate slowed down gradually.

During the aging process of Cu-1.9Be-0.3Ni-0.15Co alloy, solute atoms precipitate from the matrix continuously and the transformation ratio of precipitates (X) can be defined as:

$$X = V/V_c \quad (2)$$

where V is the relative volume of a new phase in the unit volume of a system at a certain moment; V_c is the relative volume of a new phase in the unit volume of a system at the end of the phase transformation.

According to Matheson's law, the resistivity of copper alloys can be expressed as:

$$\rho = \rho_0 + a\zeta \quad (3)$$

where ρ is the resistivity of copper alloys; ρ_0 is the resistivity of pure copper and it is only the function of temperature; a is the concentration of solute elements in copper alloys; ζ is the residual resistance of copper alloys caused by unit solute elements.

Therefore, the resistivity of the copper alloy has a linear positive correlation with solute content. It can be assumed that the conductivity of the copper alloy (C) and X follow the linear relationship:

$$C = C_0 + \beta X \quad (4)$$

Fig. 11 Conductivity of Cu-1.9Be-0.3Ni-0.15Co alloy aged for different time

where C is the conductivity of alloy; C_0 is the conductivity of solution-state alloy; β is the increase in conductivity caused by unit solute element precipitation; X is the relative volume fraction of precipitates.

According to Eq. (4), the transformation ratio of precipitates is:

$$X = (C - C_0)/\beta \quad (5)$$

According to the results of XRD analysis, the mass fraction of precipitates in soft-state alloy after aging at 320 °C for 360 min was 7.9%, while the mass fraction of precipitates was 13.6% when they completely precipitated on the basis of the chemical composition of the specimens. So the transformation ratio of precipitates in the alloy was 58.1% after aging for 360 min. As a consequence, the transformation ratio of precipitates in the alloy was 2.0% after aging for 30 min. It could be concluded that the conductivity increased with the transformation ratio of precipitates during aging process.

For the purpose of depicting the relationship between the transformation ratio of precipitates and transformation time, the empirical equation [17] was used.

$$X = 1 - \exp(-Bt^n) \quad (6)$$

where B and n are corresponding coefficients, which mainly depend on the value of phase transformation temperature, phase transformation free energy, interfacial energy and other parameters. The exponent n mainly depends on the type of phase transformation, especially the microscopic mechanism of nucleation and growth.

By taking the natural logarithm and common logarithm on both sides of Eq. (6), the following linear equation can be obtained. It can be utilized to describe the relationship between transformation ratio and transformation time of precipitates:

$$\log \ln \left(\frac{1}{1-X} \right) = \log B + n \log t \quad (7)$$

With the precipitates transformation ratio of the soft-state alloy after aging for 30 and 360 min, $n = 1.5142$ and $B = 2.3786 \times 10^{-7}$ can be obtained.

Figure 12 shows the relationship between $\log(\ln(1/(1-X)))$ and $\log t$ of the soft-state Cu-1.9Be-0.3Ni-0.15Co alloy. All the experimental data fall on a single straight line, which means the precipitation process is reflected precisely by the calculated results. Therefore, the phase transformation kinetics of the soft-state Cu-1.9Be-0.3Ni-0.15Co alloy during aging at 320 °C can be expressed as:

$$X = 1 - \exp(-2.3786 \times 10^{-7} t^{1.5142}) \quad (8)$$

According to the results of XRD analysis, the mass fraction of precipitates in hard-state specimens after aging

Fig. 12 Relationship between $\log(\ln(1/(1-X)))$ and $\log(t)$ of the soft-state Cu-1.9Be-0.3Ni-0.15Co alloy

at 320 °C for 360 min was 8.7%, while the mass fraction of precipitates was 13.6% when they completely precipitated on the basis of the chemical composition of the specimens. So the transformation ratio of precipitates in the specimens was 64.0% after aging for 360 min.

By bringing the conductivity data of hard-state Cu-1.9Be-0.3Ni-0.15Co alloy into Eqs. (5) and (7), the result of $n = 0.4284$ and $B = 0.0142$ could be obtained. The relation of $\log(\ln(1/(1-X)))$ and $\log t$ of the hard-state Cu-1.9Be-0.3Ni-0.15Co alloy can be drawn by above results, as shown in Fig. 13. The experimental data points presented a remarkable linear relationship, which indicates a credible prediction of the precipitation process. As a result, the phase transformation kinetics of the hard-state Cu-1.9Be-0.3Ni-0.15Co alloy during aging at 320 °C can be expressed as:

$$X = 1 - \exp(-0.0142 t^{0.4284}) \quad (9)$$

Figure 14 shows the relationship between transformation ratio of precipitates and transformation time. Define 5% transformation ratio ($X = 0.05$) as the beginning of transformation and 95% transformation ratio ($X = 0.95$) as the end of transformation. As for aging process of soft-state alloy, the exponent n of phase transformation kinetics equation was 1.5142. As shown in Fig. 14, the magnitude of transformation time is 1.175. Therefore, it could be inferred that the aging process of soft-state alloy was homogeneous nucleation and the nucleation rate rapidly decayed to zero [17]. For aging process of hard-state alloy, the exponent n of phase transformation kinetics equation was 0.4284. The magnitude of transformation time was 4.25, so the aging process was interface nucleation and the nucleation rate rapidly decayed to zero [17].

Fig. 13 Relationship between $\log(\ln(1/(1-X)))$ and $\log(t)$ of the hard-state Cu-1.9Be-0.3Ni-0.15Co alloy

Fig. 14 Relationship between transformation ratio of precipitates and aging time

4 Discussion

As shown in the results of tensile and microhardness tests, the strength and hardness of hard-state alloy increased rapidly at the beginning stage of aging and reached the peak value after aging for 120 min; however, afterward they started to fall down. This may be attributed to the high supersaturation of matrix and driving force of precipitation during the early stage of aging. Large amounts of fine and dispersed precipitates form consequently and lead to significant precipitation harden, manifested as the rapidly increase in strength and hardness. As it could be observed in TEM microstructure, the size of precipitation particles coarsened with aging time and the precipitates lost the coherent relationship with parent phase after aging for 360 min. As a result, the precipitation hardening effect is weakened, leading to the decrease in strength and hardness.

However, both the strength and hardness of soft-state alloy were near the peak value after aging for 180 min and increased slightly afterward. It is probably because that the number of crystal defects such as vacancy, dislocation, etc. is much lower than that in hard-state alloy. The nuclei which can be used for heterogeneous nucleation of precipitates are much fewer consequently. Therefore, the aging process is much slower than that in hard alloys, and the soft-state alloy is also difficult to reach over-aging stage. So the hardness increases slowly during aging process, and over-aging phenomenon of decreasing in strength and hardness doesn't occur. The elongation of Cu-1.9Be-0.3Ni-0.15Co alloy decreased with aging time. As shown in Fig. 14, the volume fraction of precipitates increases with the aging time. Meanwhile, the precipitates coarsened and gathered gradually. Owing to the strain incoordination between matrix and precipitates during plastic deformation, the microvoids will initiate and grow up at the interfaces of matrix and precipitates. Then, the microvoids coalesce into microcracks which will cause a significant reduction in elongation of the alloy.

The increasing rate of conductivity in hard-state alloy was much faster than that in soft-state alloy at the beginning of aging process, and then the increasing rate of conductivity in hard-state alloy slowed down. For soft-state alloy, the conductivity rose slowly and the increasing rate was uniform. As shown in Fig. 14, the phase transformation process of soft-state alloy was homogeneous nucleation while it of hard-state alloy was interface nucleation. Since the nucleation rate of homogeneous nucleation is much slower than that of interface nucleation, the precipitation process of soft-state alloy is much gentler, resulting in slower increasing rate of conductivity. However, crystal defects such as vacancy, dislocation, etc. induced by cold rolling can provide nuclei for the heterogeneous nucleation and growth during aging process of hard-state alloy. Once the nuclei provided by defects are consumed, the nucleation rate rapidly decays to zero, which leads to slowdown of precipitation rate. Furthermore, the concentration of solute elements in the solid solution decreases with aging time, causing the reduction in nuclear driving force. As a result, the increasing rate of conductivity declined.

5 Conclusions

1. The precipitation transformation mechanisms of Cu-1.9Be-0.3Ni-0.15Co alloy in soft and hard-state are different. The former is homogeneous nucleation, while the latter is interface nucleation, but the nucleation rates in both states rapidly decay to zero. Heterogeneous nucleation of hard-state alloy leads to rapid precipitation transformation. The transformation ratio

of precipitates in the hard-state alloy reaches a considerable level (21.4%) after only 5 min. However, homogenous nucleation needs a much longer incubation period. The transformation ratio of precipitates in the soft-state alloy achieves 6.4% until aging for 60 min.

2. According to the observation of TEM microstructure, the precipitation sequence of hard-state Cu-1.9Be-0.3Ni-0.15Co alloy during 320 °C aging is G.P. zone $\rightarrow \gamma''$ phase $\rightarrow \gamma'$ phase $\rightarrow \gamma$ phase. The size of precipitation particles coarsen with aging time and the precipitates lose the coherent relationship with parent phase after aging for 360 min.
3. The differences of precipitation transformation mechanisms between soft- and hard-state alloys cause the distinctions between their mechanical properties and electric conductivity. The strength and hardness of hard-state alloy increase rapidly at the beginning of aging and then start to fall down after reaching the peak value with the aging time of 120 min. However, the strength and hardness of soft-state alloy are near the peak value after aging for 180 min and increase slightly afterward. The increasing rate of conductivity in hard-state alloy is much faster than that of soft-state alloy at the beginning of aging process and it slows down subsequently. For soft-state alloy, the conductivity rises slowly and the increasing rate is uniform during aging process.

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