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Study on microstructure regulation and strengthening mechanism of high performance copper cable

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Abstract: The objective of this study is to determine the correlation between the mechanical and electrical characteristics of copper wire subjected to diverse annealing parameters and to characterize its microstructure. The findings reveal that annealing treatment exerts a considerable influence on the properties of copper wire. The strength of copper wire drops significantly upon annealing, while the conductivity rises. After annealing, the grains within the copper wire enlarge and the grain boundary transitions from a non-equilibrium state to an equilibrium one. Additionally, through the quantitative analysis of the relationship among microstructure, strength, and conductivity, it is discovered that the increase in grain size will diminish the strength of copper wire but enhance the conductivity. Overall, the copper wire possesses favorable strength and conductivity after annealing at 180°C for 30 minutes.

1. Introduction

With the increasing scarcity of global natural resources and the increasing severity of environmental problems, countries continue to advocate energy conservation and emission reduction to combat climate change and promote green development, leading to the rapid development of new energy industry (such as new energy vehicles)[1,2]. However, charging is still one of the main bottlenecks in the development of new energy vehicles at present[3]. The mechanical and electrical properties of charging cable material directly affect the charging efficiency and energy transmission stability of new energy vehicles, so it is essential for charging cable material to have good mechanical performances and power transmission stability [4-6].

Currently, the cable material for charging is mainly made of pure copper material with good electrical conductivity[7]. Due to the low strength of pure copper, plastic deformation (such as cold drawing) is usually used in the penultimate step of the copper cable forming process to improve its strength[8-10]. A large number of studies have shown that plastic deformation can cause a large number of dislocation in the grains and the grain refinement for copper wire, and the increase of the proportion for strong grain orientation in the copper cable, resulting in the increase of the strength for the copper cable[11-14]. However, the plastic deformation of copper also causes electron scattering, resulting in a decrease of conductivity[15-18]. Therefore, heat treatment processes (such as recrystallization annealing) are often followed by plastic deformation to improve the conductivity of the copper cable.[19]. Generally speaking, there is a restrictive relationship between the strength of copper cables and electrical conductivity. How to make the strength and conductivity of copper cable reach the ideal state by adjusting the annealing temperature and holding time is an important research topic in the new energy industry[20-23].

Many researchers are constantly trying to adjust the annealing temperature and holding time, so that the strength and conductivity of copper materials to achieve a synergistic effect. For example, Sun Pengfei et al.[24] studied the effects of different annealing temperatures and holding time on the mechanical properties of cold drawn oxygen-free copper wire with a diameter of 2.92mm. The results show that when annealing at low temperature (80℃-150℃) and holding time exceeds 1 h, the yield strength of copper wire decreases from 450MPa to 420MPa, and the conductivity increases from 96%IACS to 96.2%IACS. When the holding time of medium temperature annealing (210℃-300℃) exceeds 1h, the yield strength of copper wire decreases from 450MPa in cold drawing to about 96MPa, and the conductivity increases from 96%IACS in cold drawing to more than 97.9%IACS, up to 100%IACS. Chen Yanxu et al.[25] studied the mechanical and electrical properties of a copper wire with a diameter of 2mm after annealing at 400℃ for 1h. The results show that the tensile strength of annealed copper wire decreases from 406.13MPa in tensile state to 229.85MPa, and the electrical conductivity increases from 98.93%IACS in tensile state to 101.5%IACS. Wu Heng et al.[26] studied the effect of high temperature and short time annealing (860℃) on the mechanical properties of 2mm diameter copper wire. When the holding time is 15s, the tensile strength of copper wire decreases from 406.13MPa during cold drawing to 235.15MPa, and the conductivity increases from 98.93% to 102.4%. When the holding time is 30s, the tensile strength of the copper wire is reduced to 230MPa, and the conductivity is increased to 103.29%IACS. All in all, there is no uniform annealing condition to optimize the mechanical and electrical properties of copper wires with different diameters. In this paper, the mechanical and electrical properties of copper wire materials for high-performance ultra-flexible charging cables under different annealing temperatures and holding times are systematically studied. The microstructure of copper conductors is observed and characterized by various testing methods, and the influencing mechanism of copper wire conductivity and strength is studied.

2. Methods

2.1 Sample preparation

The effect of recrystallization annealing process on mechanical and electrical properties of copper wire was studied. In order to achieve this research purpose, we choose a high-purity copper wire with a diameter of 0.5 mm as the experimental material. The EDS analysis results of the copper wire are shown in Figure 1. The proportion of pure copper in the conductor material of the copper wire is 100%, and there are no other elements or impurities. The copper wire after drawing is selected as the blank, and recrystallization annealing is carried out to optimize the structure and properties of the copper wire by adjusting the annealing temperature and holding time. In this study, KSL-1200 heat treatment furnace was used for recrystallization annealing. The specific recrystallization annealing process parameters were shown in Table 1, and different recrystallization annealed copper wire cable conductor materials were obtained.

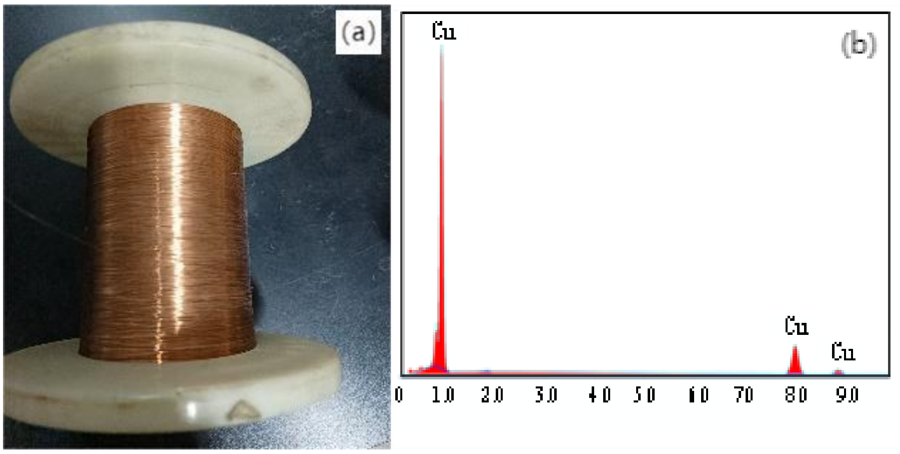


Figure 1: (a) Φ0.5mm copper wire (b) EDS of copper wire

2.2 Experimental method

2.2.1 Microstructure observation

The microstructure and microstructural characteristics of copper conductor materials in both the as-received and annealed states were observed. Due to the small diameter of the copper wire, direct observation and analysis are challenging, requiring mounting, polishing, cleaning, and etching. The specific preparation process for the test sample is as follows: a 26mm length of copper wire is cut and placed at the bottom of a metallographic sample mounting machine (model: XQ-2Bφ30). Hot mounting powder (DJM-B black) is added, with the temperature set at 150 ℃ for 600s. After mounting, a cylindrical specimen with a diameter of Φ30mm is produced. The specimen is then polished using water sandpaper with specifications of 800 mesh, 1200 mesh, and 2000 mesh respectively on a polishing machine (Model: MP-2), along with polishing cloth and W1.5 diamond grinding paste. Subsequently, ultrasonic cleaning (model: JP-031S) in anhydrous ethanol for 10min followed by drying with a hair dryer completes sample preparation. The surface of the copper wire is then etched to observe its internal grain using an etch solution consisting of orthophosphoric acid (50ml), glacial acetic acid (28ml), nitric acid (22ml), hydrochloric acid (10ml), ferric chloride (8g), and alcohol (50ml). The corrosion process involves applying drops of etching solution to the surface for about 5 seconds each time before rinsing with water and air drying; this step is repeated until thoroughly washed away residue remains before ultrasonic cleaning.In order to study the evolution mechanism of strength and conductivity of copper wire, the microstructure of copper wire was analyzed by X-ray diffraction (XRD). The XRD step is set to 0.02°, and the signal acquisition range is 2θ: 30~80°.

Table 1: Copper wire annealing parameters

Annealing temperature	180℃	190℃	200℃	210℃	220℃	230℃	550℃	800℃
Holding time	30min	30min	30min	30min	30min	30min	5s,15s,18s , 20s	5s,8s
Cooling mode	Furnace cooling	Furnace cooling	Furnace cooling	Furnace cooling	Furnace cooling	Furnace cooling	Air cooling	Air cooling

2.2.2 Mechanical property analysis

Tensile test refer to GB/T4909.3-2009 "Bare wire test method Part III: Tensile test", the MTS810 universal testing machine is used to measure the mechanical properties of copper wire, the measurement length is 250mm, the standard distance is 200mm, the speed is 50mm/min. The tensile strength, yield strength and elongation of copper wire can be measured by tensile test.

The bend test is in accordance with CQC1103-2015 "Technical Specification for Electric Vehicle Conductive Charging System Cables". The specific testing process is as follows: Firstly, a cable sample of not less than 600mm in length is cut from the finished cable. Then, the sample is installed on the experimental device, with one end fixed on the bending fixture and external force applied to the other end. Subsequently, a monitoring current of 0.1A is applied to the conductor during the test, and the sample is bent at a fixed speed (90° per side as one cycle). Finally, when the copper wire breaks, counting stops and the bending performance of the copper wire is judged by its number of bends. The bending testing machine model used here is WSWP-180, with a weight of 50g and a bending rate of 50rad/min at a bending radius of 10mm.

2.2.3 Electrical performance test

The methods for testing metal resistance include the bridge method, direct reading measurement method, pulse heating method, and induction method. Bridge measurement resistance can be categorized into single bridge and double bridge. Single bridge is generally suitable for high resistance measurement due to the inability to eliminate lead resistance and contact resistance. Metal materials typically have a resistance value lower than 1Ω, falling under low resistance measurement where double bridges are commonly used with a measurement range between 10-6 and 100Ω, achieving an accuracy of 0.02%. In this study, the conductivity of pure copper wire is measured using the double bridge method for practical and economic reasons.

In addition, electrical performance testing was executed as per the provisions of GB/T 3048.2-2007 "Test Methods for Electrical Performance of Wire and Cable, Part 2: Test Method for Conductivity of Metal Materials". A QJ57 double-arm DC bridge was used to conduct a conductivity test on the copper wire. The test temperature was maintained at 25 °C, the measurement length was 1000mm, and the aim was to discern how the microstructural variations of the copper wire influences its electrical performance characteristics.

$$\omega = \frac{L}{R \cdot S \times 5.8 \times 10^7} \times 100\% \tag{1}$$

$$\rho = \frac{R \cdot S}{L} \tag{2}$$

Where is the conductivity of the wire, ρ is the resistivity of the measured copper wire.L is the length of the tested segment when measuring the resistance, typically at a length of 1000mm. R is the resistance of the sample being measured, and S is the cross-sectional area of the oxygen-free copper wire being measured.

3. Results and Analysis

3.1 Metallographic structure observation

3.1.1 The metallographic of the blank state

The copper wire used in this research work is obtained after drawing process, and its diameter is 0.5mm. EDS point scanning analysis showed that the copper content in the copper wire was 100%, and no other elements existed. The microstructure of copper wire in blank state was observed, as shown in Figure 2. It can be clearly seen from Figure 2(a) that after drawing, the grains in the copper wire extend along the drawing direction and show a linear shape, most of the crystals are fibrous, and there is no obvious grain boundary. SEM analysis further found that the initial microstructure of copper wire in the blank state has the characteristics of grain extension, vacancy and defect at the grain boundary.

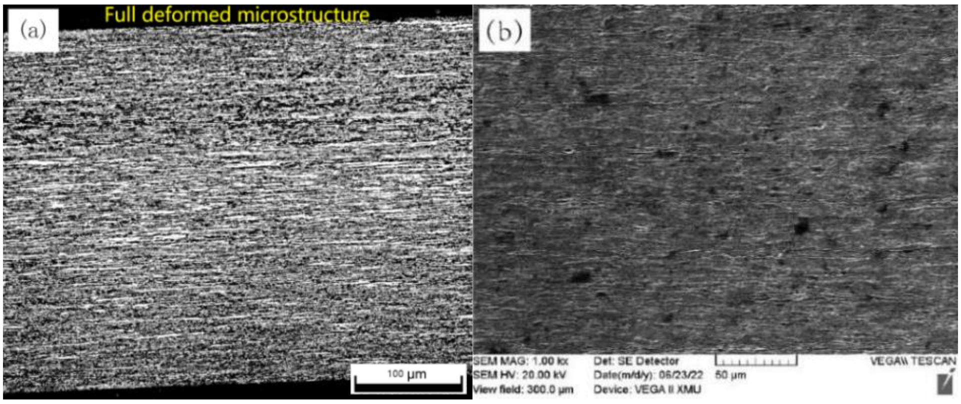
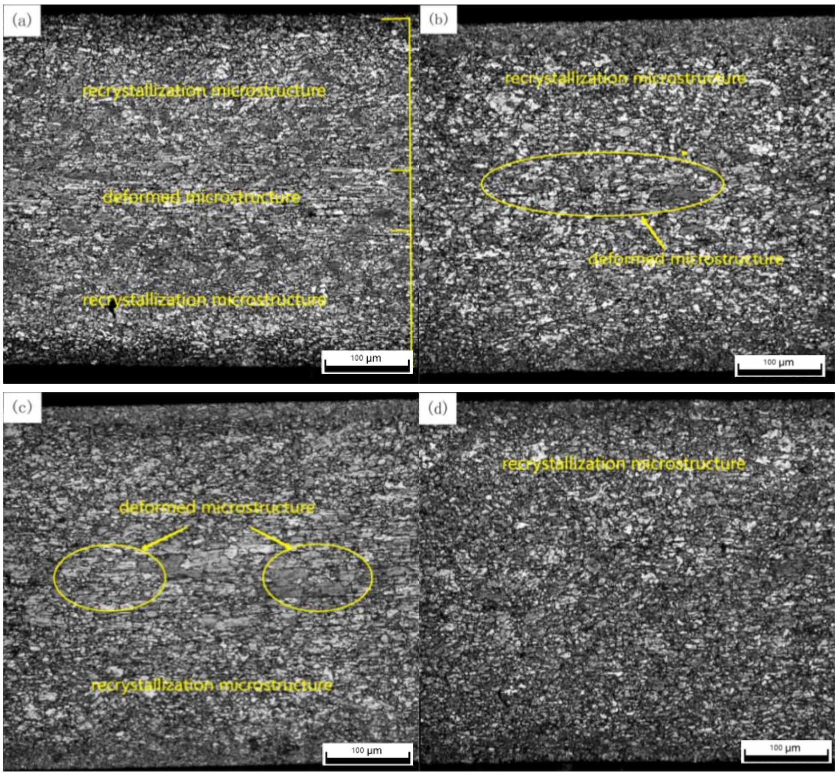


Figure 2: Metallographic observation of copper wire in initial state a) ultra depth of field microscope; b) SEM diagram

3.1.2 The microstructure after annealing

Figure 3 shows the grain evolution process after low-temperature annealing of the blank copper wire. When annealing at 180 ℃ for 30 minutes, recrystallization occurred in some areas, and the internal grains showed obvious stratification characteristics. The equiaxed grains have appeared near the surface, while the central ones are mainly long strips, and the proportion of recrystallized grains is about 80%. After annealing at 190 ℃ and 200 ℃ for 30 minutes, there are still 5%-10% elongated grains, and recrystallization has occurred in most areas. When the temperature rises to 210 ℃, the grain structure inside the copper wire tends to be stable, and the proportion of elongated grains is less than 1% (as shown in Figure 3d). At 220 ℃ and 230 ℃, the interior of the copper wire is completely recrystallized, and its grain morphology is 100% uniform recrystallized grains. After annealing at low temperature for a long time, the internal grains of the copper wire gradually evolved from the blank state of fiber (as shown in Figure 2a) to the long strip (as shown in Figure 3b, 3c), and finally obtained a uniform equiaemic crystal (as shown in Figure 3d).

In order to enhance production efficiency and reduce manufacturing time, a comprehensive investigation was conducted on the grain size and characteristics of copper wire under medium and high temperatures. Subsequently, heat treatment was performed on two different diameter copper wires at 550 ℃ and 800 ℃, enabling observation of grain transformations and measurement of various properties. These results were then compared with those obtained from low temperature annealing.



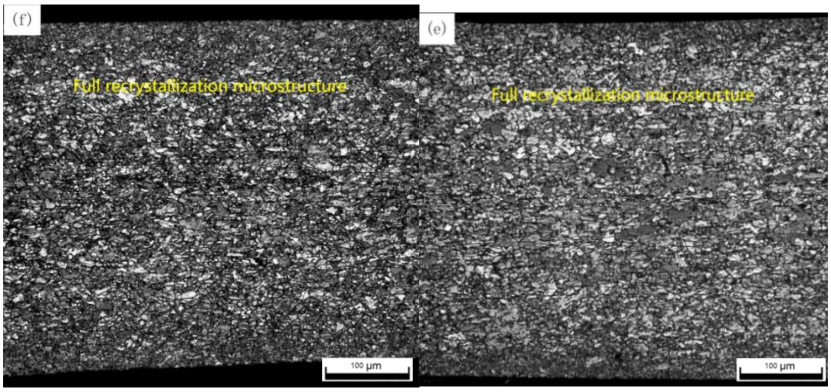


Figure 3: Metallographic observation of annealed copper wire; a) 180℃, b)190℃, c)200℃, d)210℃, e)220℃, f)230℃

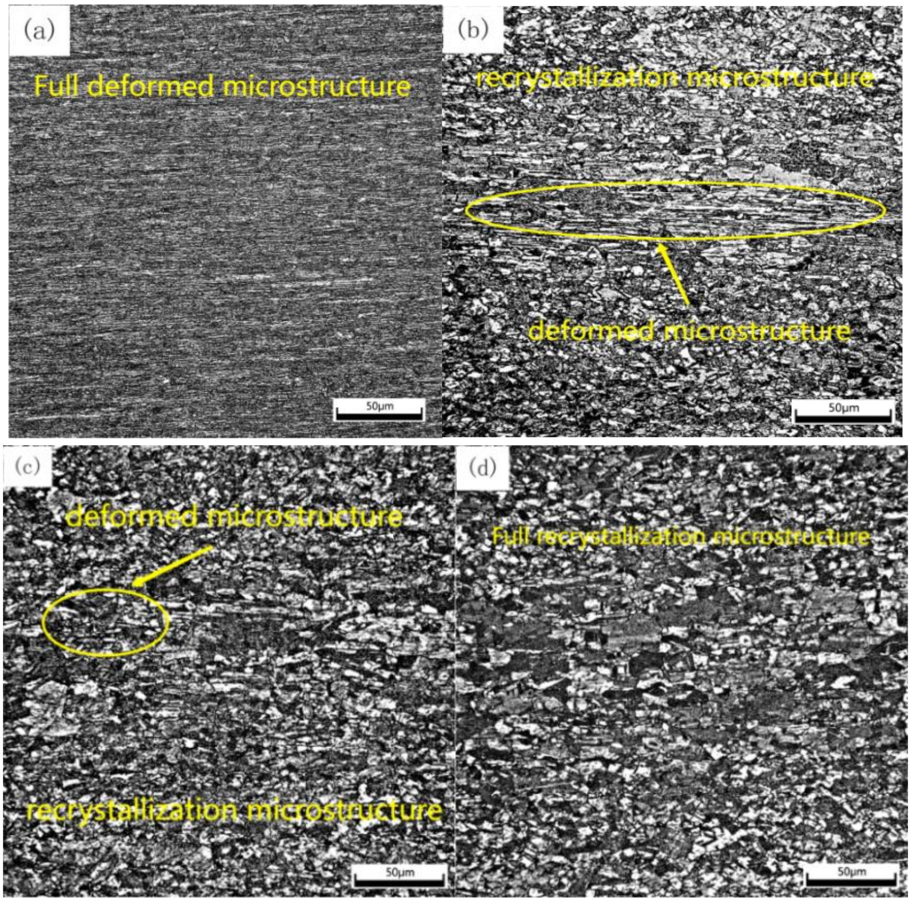


Figure 4: Metallographic observation of copper wire after annealing at 550℃ a) 5s, b)15s, c)18s, d)20s

The blank copper wire was annealed at medium and high temperature for a short time. Figure 4 shows the metallographic structure inside the copper wire after annealing at medium temperature of 550 ℃. As shown in Figure 4 (a), after annealing the copper wire at 550 ℃×5s, the internal grain is still fibrous, and the shape changes little compared with the blank state. As shown in Figure 4 (b), after annealing at 550℃×15s, recrystallization of the internal grains in the surface part of the copper wire has occurred, of which more than 85% of the grains have been transformed into uniform equiaaxial grains, and only a small amount of fibrous deformation structure (accounting for about 15%) has been distributed in the center. As shown in Figure 4 (c), after annealing for 18s, most of the internal grains of the copper wire have completed the recrystallization transformation, and the deformation structure accounts for less than 5% of the total. As shown in Figure 4 (d), after 20s annealing, there is no fibrous deformation structure inside the copper wire.

The blank copper wire was annealed at high temperature for a short time, as shown in Figure 5 (a). After 5s annealing at 800 ℃, more than 90% of the grains inside the copper wire were fibrous, and only a small part of the grains were recrystallized. As shown in Figure 5 (b), after 8s of heat treatment, less than 1% of the slender crystals exist in the interior, and most of them have been recrystallized.

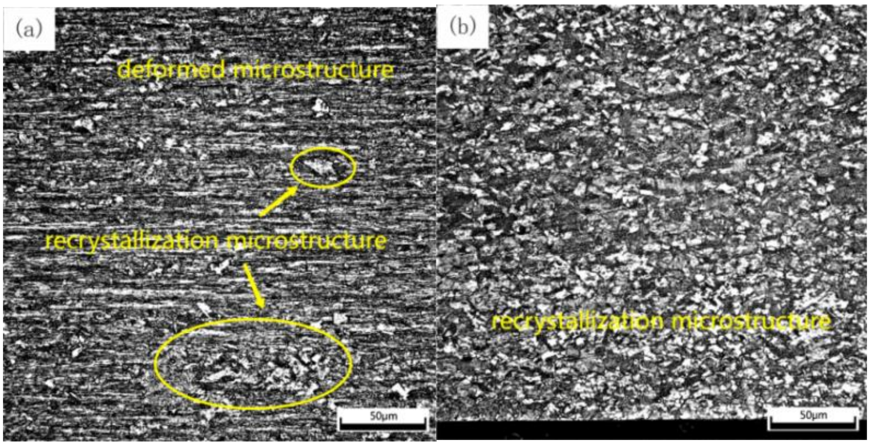


Figure 5: Metallographic observation of copper wire after annealing at 800℃ a) 5s, b) 8s

This section describes in detail the change of metallographic structure of copper wire after long annealing at low temperature and short annealing at medium high temperature. When annealing at low temperature for a long time, the recrystallization transformation of Φ0.5mm copper wire began after annealing at 180℃×30min, and equiaxed grains appeared near the surface, while the central position was dominated by long strips. Recrystallization was completed after annealing at 210℃×30min, and all of them became uniform equiaxed crystals. After annealing at 550℃×5s, the internal grain of copper wire is still fibrous deformed grain, recrystallization transformation occurs after annealing at 550℃×15s, and recrystallization transformation is completed after annealing at 550℃×20s. The recrystallization of copper wire began after annealing at 800℃×5s, and most of the grains completed recrystallization after annealing at 800℃×8s.

3.2 XRD analysis

The axial cross-section of the polished copper wire was characterized by XRD, and the analysis results are shown in Figure 6. During the drawing process, the (111), (200) and (220) diffraction peaks shift significantly to the left and widen, which is caused by the grain fibrosis caused by the increase of dislocation density. At the same time, the distortion of copper lattice under tensile stress will also cause the peak to move to a low Angle direction. After annealing at 180℃×30min, the diffraction peak and the standard peak basically coincide. When the temperature increased to 200℃, most of the elongated grains in the copper wire were recrystallized, the grain size decreased and the peak position shifted to the right. After annealing at 550℃×5s, the internal grain composition is similar to that at 200℃×30min, and the peak position shifts to the right. In general, the diffraction peak position after annealing almost coincides with the standard position and decreases its width. This indicates a decrease in dislocation density, recovery of lattice distortion, and a change in grain size.

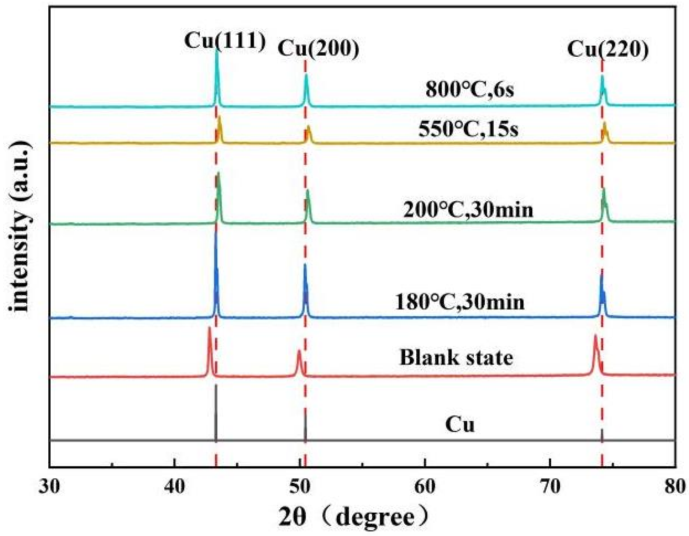


Figure 6: XRD characterization of copper wire under different annealing conditions

3.3 Evolution of mechanical properties

Related studies have shown that for the heat-treated copper wire, when the internal uniform equiaxed crystals and deformed grains coexist, the point with the best mechanical properties can be optimized in this state. It can be observed from the above metallography after annealing at low temperature that the coexistence of fibrous grains and recrystallized grains is present in the copper wire after annealing at 180 ℃ for 30 minutes, and the internal grain structure is in a stepped distribution. After annealing at 200 ℃ for 30 minutes, there are only a few fibrous grains in the copper wire. After annealing at 220 ℃ for 30 minutes, the grains inside the copper wire were completely recrystallized. Under annealing conditions of 550 ℃, 15 seconds and 800 ℃, 5 seconds, long grains appeared in the copper wire concentrated in the central region and coexisted relatively evenly with recrystallized and fibrous grains. Therefore, copper wire samples annealed at 180 ℃, 200 ℃, 220 ℃ for 30 minutes, 550 ℃ for 15 seconds and 800 ℃ for 6 seconds were selected for performance tests and their mechanical properties were analyzed.

Figure 7 (a) shows the bending properties and elongation of the annealed copper wire. The bending property of the blank copper wire is 478 times. After annealing, the bending property of the copper wire is obviously improved. After annealing at 180℃×30min, 200℃×30min, 220℃×30min, 550℃×15s and 800℃×6s, the bending performance of Φ0.5mm copper wire is increased to 606, 592, 582, 536 and 532 times, respectively. The elongation of the blank copper wire after breaking is only 0.43%. After annealing at 180℃×30min, 200℃×30min, 220℃×30min, 550℃×15s, 800℃×5s. The elongation after fracture was significantly increased to 34.17%, 30.15%, 29.55%, 18.65%, 29.86%.

When the copper wire is annealed at 550℃ for 15 seconds or 800℃ for 6 seconds, the bending properties and elongation are better than the initial state, but the improvement is not obvious compared with the long-term exposure to low temperature conditions. In summary, the bending properties and extension characteristics of copper wire can be enhanced by annealing at 180℃ for 30 minutes.

Figure 7 (b) shows the strength of the copper wire after annealing. The results show that the tensile strength and yield strength of the copper wire in the initial state are at a high level, and the strength decreases significantly after annealing. However, under different annealing temperatures and holding times, the tensile strength and yield strength of the copper wire are basically at the same level, and the tensile strength of the copper wire in the blank state is 385MPa. After annealing at 180°C×30min, 200°C×30min, 220°C×30min, 550°C×15s and 800°C×5s, the tensile strength decreased significantly to 262MPa, 260MPa, 255MPa, 258MPa and 256MPa. The yield strength of copper wire in blank state is 378MPa. After annealing at 180°C×30min, 200°C×30min, 220°C×30min, 550°C×15s and 800°C×5s, the yield strength of the alloy was significantly reduced to 137MPa, 140MPa, 126MPa, 135MPa and 132MPa. After annealing at 200℃ and 220℃, the tensile strength of copper wire is slightly lower than that after annealing at 180℃, but the reduction range is small and basically at the same level, indicating that when the holding time is 30min, the influence of the increase of annealing temperature on the tensile strength of copper wire is reduced after reaching the recrystallization temperature. The tensile strength of the copper wire after holding at 800℃ for 6s is significantly higher than that after annealing at low temperature, indicating that there are more dislocations inside the copper wire after annealing at 800℃. The results show that the strength of copper wire is influenced by the recrystallization process, and the microstructure change is an important factor affecting the strength of copper wire.

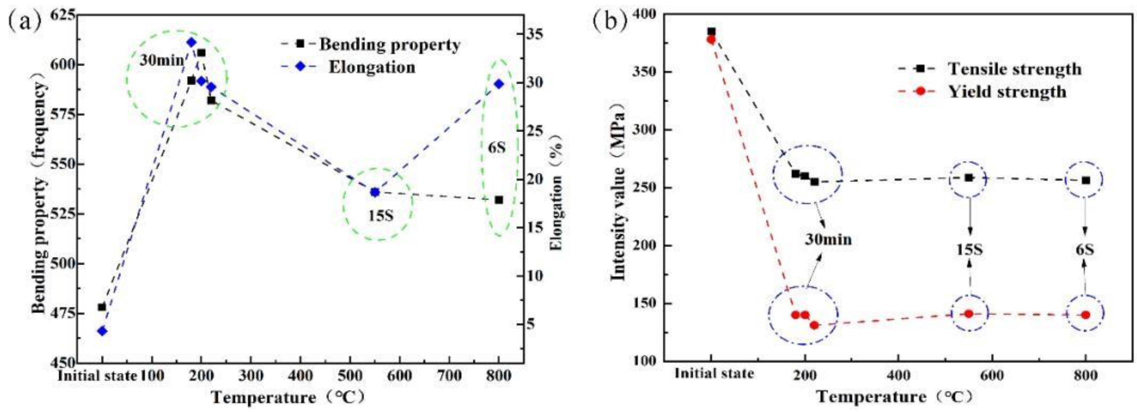


Figure 7: Comparison of properties of copper wire after annealing a) Bending properties and elongation changes; b) Variation diagram of tensile strength and yield strength

Figure 8 shows the electrical conductivity and resistivity of the annealed copper wire. It can be observed from the figure that conductivity is relative to resistivity, and good conductivity means low resistivity. The conductivity of the blank Φ0.5mm copper wire is 96.43%IACS, and the conductivity of Φ0.8mm copper wire is 99.44%IACS. After annealing Φ0.5mm copper wire at 180°C×30min, 200°C×30min, 220°C×30min, 550°C×15s and 800°C×5s, The conductivity was increased to

99.72%IACS, 100.08%IACS, 100.43%IACS, 97.72%IACS and 97.17%IACS, respectively. In particular, after 180 ℃×30min, the copper wire reached a peak and increased by 4.3% compared to the initial state. As the temperature rises to 200 ℃, 220 ℃ and 550 ℃ for annealing, although the conductivity gradually decreases compared to 180 ℃, it is still improved from the initial state. After annealing at 800 ℃, the electrical conductivity of the copper wire is not much different from the initial state. These results show that the internal grain structure changes have a significant effect on the electrical conductivity of copper wires.

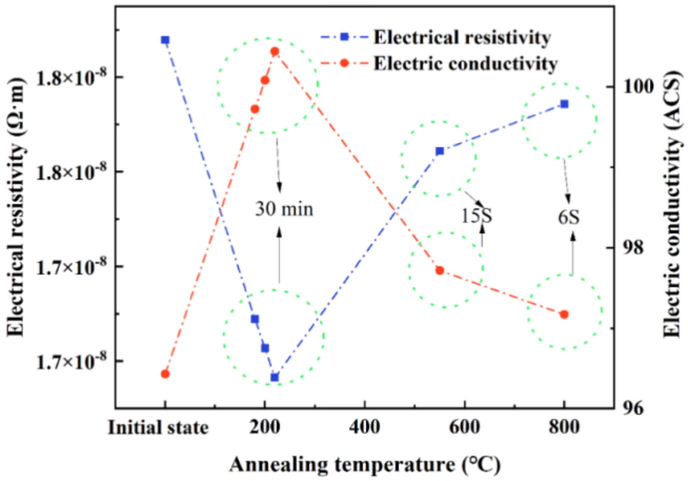


Figure 8: Changes of conductivity and resistivity of copper wire after annealing

4. Conclusions

In this study, a series of annealing experiments were conducted to regulate the microstructure of copper wires and measure the strength and conductivity using instruments. The microstructure of the copper wire was characterized, and we have reached the following conclusions:

(1)The copper wire exhibited optimal comprehensive properties after annealing at 180°C for 30 min. Its electrical conductivity increased by 4.3% to 100.68% IACS, and its bending performance increased by 23.85% to 592 times compared to its initial state. The tensile strength increased to the highest value of 262 MPa, and elongation increased 34.17% compared to its initial state.

(2)The copper wire had a high strength in its initial state, with a yield strength of 378 MPa. After annealing, the strength decreased, and the yield strength dropped to 140 MPa. When uniform equiaxed grains and deformed grains coexist in copper wire, the optimum point of mechanical properties can be optimized in this state.

(3)The copper wire had low conductivity in its initial state with a value of 96.42% IACS. After annealing, the wire's conductivity increased to 100.68% IACS. These findings highlight the substantial influence of grain structure on both electrical conductivity and resistivity within copper wires.

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