

Article

Study on Microstructure Evolution and Influencing Factors of Pure Copper Wire After Directional Heat Treatment

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Abstract

The Ohon Continuous Casting is the main method for preparing single crystal copper wire, and it is also the research hotspot at present, but it is difficult to directly cast ultrafine single crystal copper wire (diameter < 0.05 mm). The copper wire obtained by continuous casting must be drawn and deformed before it can be used in practice, but this will bring a series of problems such as single crystal structure destruction and conductivity deterioration. Directional heat treatment technology can control the direction of heat flow at a low temperature, realize the directional migration of grain boundaries in the recrystallization process, and form columnar crystals or single crystals, which is of great significance for improving electrical conductivity. In this paper, the directional heat treatment method was used to investigate the microstructure evolution and influencing factors of pure copper wire, the process parameters were optimized, and the conductivity of pure copper wire was measured. It was found that the conductivity of pure copper wire increased by 5% when the heating temperature was 750 °C and the withdrawing velocity was 15 μm/s, which laid a foundation for the improvement of conductivity of pure copper wire.

Keywords: pure copper wire; directional heat treatment; columnar crystal; withdrawing velocity; conductivity



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1. Introduction

Information warfare has become the fourth battlefield other than land, sea and air in modern warfare. The importance of fighting for information superiority is self-evident. With the continuous development of electrical equipment and electronic devices, higher requirements are put forward for copper coils, copper wires, and copper bonding wires [1]. Therefore, it is increasingly important to improve the conductivity of pure copper wires.

Changing the internal structure was a breakthrough for the improvement of the conductivity of metal materials. In the 1980s, people realized the influence of grain boundaries on the conductivity of conductors, and began to improve the conductivity from the perspective of metal physics, that is, eliminating grain boundaries to prepare single crystal wires [2].

Grain boundaries have multiple effects on the electrical properties of pure copper wires. The first is resistivity, scattering is the root cause of metal resistance. Resistivity is caused by lattice thermal vibrations as well as lattice physical defects, and the more crystal defects, the greater the resistivity of the metal. Grain boundaries destroy the periodicity of the crystal structure and thus act as scattering sources for conduction electrons, increasing the

resistivity of the conductor. Mao et al. conducted high-temperature short-time annealing on as-drawn T2 pure copper wires at 850 °C for 40 s. After this treatment, both the tensile strength and elongation after fracture of the wires increased, and the electrical conductivity was approximately 5.2% higher than that of the as-drawn state. Both the comprehensive performance and annealing efficiency were better than those of the low-temperature long-time annealing process at 400 °C for 60 min [3]. Fan et al. studied the relationship between resistivity and grain boundary number in pure aluminum and found that the resistivity of polycrystalline aluminum increased with the increase in transverse grain boundary number [4]. The average resistivity of continuous casting single crystal industrial pure aluminum was $2.6455 \times 10^{-8} \Omega\text{m}$, which was 11.5% lower than that of metallic cast aluminum. Lu et al. studied that after pure copper wires are drawn and deformed to $\Phi 1.8$ mm, the number of grain boundaries increases, the tensile strength increases, the elongation after fracture decreases, and the resistivity increases from 0.0169 to 0.0172 $\Omega\cdot\text{mm}^2/\text{m}$. The simultaneous improvement of its strength and conductivity can be attributed to the fibrous structure formed during the drawing process [5]. Wu et al. increased the drawing speed to the range of 60–300 m/min, the surface deformation of pure copper wires was intensified, the grain size was refined, and the <101> texture was enhanced. This led to a slight increase in tensile strength and electrical resistivity, along with an improvement in surface quality. However, the strain and damage in the core decreased accordingly [6].

The second is the capacitive effect, grain boundaries not only increased the resistivity of the wire but also attenuated the transmission signal. Japanese scholar Kentada Chosei once used liquid metal mercury and copper wire to transmit audio signals [7]. The resistivity of liquid mercury was much higher than that of copper wire, but the transmission effect of liquid mercury was far higher than that of copper wire. Because the resistivity at the grain boundary was higher than that on both sides, additional capacitance was formed [8]. Chen et al. measured the relationship between the number of grain boundaries per unit length of the longitudinal section of copper wire and the capacitance value of the wire [9], and believed that the greater the grain boundary resistivity, the greater the additional capacitance value generated. The more grain boundaries there are, the stronger the capacitive effect and the greater the influence on the transmission signal.

Then there is grain boundary distribution, the longitudinal distribution of grain boundary was equivalent to the parallel capacitance, and the lateral distribution of grain boundary was equivalent to the series capacitance. Compared with the grain boundaries distributed longitudinally, the grain boundaries distributed laterally had a greater influence on the additional capacitance value of the wire, so they had a greater influence on the transmission signal [10].

Finally, signal distortion, Chen et al. prepared copper single crystal, twin crystal and polycrystalline samples to study the effect of grain boundaries on transmission signal distortion [11], and found that transmission signal distortion was the smallest in single crystal samples, the largest in polycrystalline samples, and the distortion was between the two in twin crystal samples.

In order to eliminate transverse grain boundaries and prepare ultrafine single crystal wires, people combine single crystal continuous casting technology with drawing technology [12–18], but it is easy to cause grain splitting and performance deterioration [19–22].

In response to the above situation, directional heat treatment is expected to be a new method for preparing single crystal copper wires. The earliest application of directional heat treatment technology is to prepare single crystal tungsten wire [23], and later people gradually found that many metal materials can be obtained by directional heat treatment to obtain a columnar crystal or a single crystal structure [24]. Professor Baker of Dartmouth University in the United States conducted directional heat treatment research on cold-rolled

copper single crystals [25], designed a set of experimental equipment by himself, and found that the temperature gradient before the hot zone, the moving speed of the hot zone, and the temperature of the hot zone have a very important effect on the growth of columnar crystals. Li et al. studied the directional heat treatment process of cold-rolled polycrystalline nickel and found that the columnar crystal growth of cold-rolled polycrystalline nickel during directional heat treatment was a secondary recrystallization process, the average grain size changed from the original 0.5 mm to 15 μm [26,27]. Zhu et al. through annealing treatment at 550 $^{\circ}\text{C}$ for 15 min, increased the proportion of $\Sigma 3$ grain boundaries in industrial pure copper wires to 49.80%, and the proportion of special grain boundaries reached 57.59%. The electrical conductivity significantly increased to 59.29% IACS (2.56 times that before annealing) [28]. Zhang et al. used directional heat treatment technology to study the microstructure evolution of industrial pure iron with different cold rolling deformation during directional heat treatment [29], and successfully prepared columnar crystals with a large aspect ratio, the length of the columnar crystal was approximately 12.2 mm, and the width was approximately 320 μm . Liu et al. obtained the best comprehensive performance when the deformation amount was 60% after multiple passes of drawing and annealing treatment on pure copper wires: tensile strength of 244.9 MPa, elongation of 51%, and conductivity of 99.4% IACS [30]. J.M. Vallejos et al. designed a set of directional heat treatment equipment [31], and successfully obtained low carbon steel with bamboo-like directional structure by directional heat treatment technology. Sahbaz et al. employed the severe plastic deformation technique of multi-directional forging (MDF) on AA6013 aluminum alloy under room temperature and low strain conditions. Although the deformation rate during the multi-directional forging process was low, cold deformation still achieved significant refinement of the microstructure, thereby improving the mechanical properties [32].

To sum up, it is of great significance to adopt directional heat treatment to eliminate transverse grain boundaries and prepare columnar or even single crystal copper wires with a large aspect ratio.

In this paper, the directional heat treatment method was used to study the microstructure evolution and influencing factors of pure copper wire, the process parameters were optimized, and the conductivity of pure copper wire was tested, which laid a foundation for improving the conductivity of pure copper wire.

2. Materials and Methods

The raw material is T1M20 copper billet with purity of 99.99%. The impurity composition is shown in Table 1. A FR-16 horizontal hydraulic drawing machine was used (Dongguan Fangrong Metallurgical Equipment Co., Ltd, Dongguan China), and the entire drawing process was carried out at room temperature with a drawing speed of 5–15 m/min, a pass processing rate ranging from 8% to 12%, and an original diameter of 8 mm. Copper wires with diameters of 1 mm, 0.85 mm and 0.7 mm were obtained by drawing, and the deformation was 87% and 89%, respectively.

Table 1. Impurity Composition of Raw Materials (wt.%).

Element	Selenium Tellurium Bismuth	Chromium Manganese Antimony Cadmium Arsenic Phosphorus	Lead	Sulfur	Tin Nickel Iron	Silver	Total Content
content	<0.00018	<0.00064	<0.0001	<0.0004	<0.00121	0.0015	<0.00403

The isothermal heat treatment was carried out in a commercial KSL-1100X-J small chamber furnace (Hefei Kejing Materials Technology Co., Ltd., Hefei, China). The operation method was to raise the box furnace to the set temperature, then put the experimental materials into the heat preservation for a period of time and then take them out for cooling.

Directional heat treatment adopts a high frequency induction directional solidification furnace (Zhengzhou Hengtong Furnace Industry Co., Ltd., Zhengzhou, China). The cavity was connected with a mechanical pump and a molecular pump for vacuumizing to 10^{-3} Pa. A high-frequency induction heating power supply was adopted, and the heating temperature could be controlled by adjusting the current. A K-type thermocouple (Shanghai Wolan Instruments Co., Ltd., Shanghai, China) was used for temperature measurement. A cooling crystallizer containing Ga-In-Sn alloy cooling liquid was arranged in the cavity, the temperature gradient before the hot zone could be controlled by adjusting the liquid level height, and the sample was controlled by PLC to move upward at a constant speed along with the pull-out rod.

In order to study the microstructure, the samples were polished with 240#, 400#, 600#, 800# and 1200# abrasive paper in turn, then polished with $0.5\ \mu\text{m}$ and $0.05\ \mu\text{m}$ Al_2O_3 polis solution in turn, and then etched with the etching solution of $10\ \text{g FeCl}_3 + 100\ \text{mL HCl} + 200\ \text{mL H}_2\text{O}$ for 3~5 s. Using an optical microscope (DM6M model, Leica Company, Wetzlar, Germany) and a scanning electron microscope (Auriga FIB/SEM double beam system, Zeiss Company, Oberkochen, Germany), as well as an EBSD (Auriga FIB/SEM double beam system, Zeiss Company, Oberkochen, Germany), experiments were conducted at 70° tilt angle, 20 kV acceleration voltage, 18 mm working distance, and with Channel 5 software (version 4.4.) package produced by HKL Company.

This experiment used the PPMS-9 equipment (Quantum Design Company, San Diego, CA, USA) to test the resistivity of pure copper samples. First, the resistance value R of pure copper wire was tested, each sample was tested 50 times, and the average value was taken. Then, the diameter d and length l of the wire was measured, the cross-sectional area S of the wire was calculated, and the following Formula (1) was substituted to calculate the resistivity ρ of the pure copper wire. Conductivity and resistivity are reciprocal to each other, from which conductivity can be calculated.

$$\rho = R \frac{S}{l} \quad (1)$$

3. Results and Discussion

3.1. Effect of Cold Rolling Deformation on Microstructure of Pure Copper Wire After Directional Heat Treatment

As shown in Figure 1, the initial microstructure after drawing deformation was fine equiaxed crystal. In this experiment, the intercept method was used to measure the average size of grains in the microstructure. Specifically, the number of grains along a straight line of a certain length was counted, and then the length of this straight line was divided by the number of grains to obtain the average diameter of each grain. According to statistics, the average crystal grain size was $2\ \mu\text{m}$, $3\ \mu\text{m}$ and $5\ \mu\text{m}$, respectively.

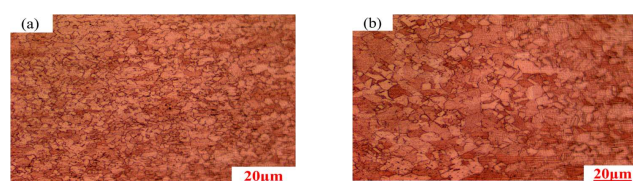


Figure 1. Initial microstructure of pure copper wire with different deformation: (a) 89%; (b) 87%.

For pure copper wires with a deformation of 87%, directional heat treatment was carried out under the conditions of a heat treatment temperature of 600 °C and withdrawing velocities of 3 $\mu\text{m/s}$, 8 $\mu\text{m/s}$ and 20 $\mu\text{m/s}$, respectively, with the drawing direction from left to right. When the withdrawing velocity was 3 $\mu\text{m/s}$, the directional heat treatment structure was as is shown in Figure 2a, large grains and small grains were mixed and distributed, the grain size was relatively uniform, and the maximum grain size was about 200 μm ; when the withdrawing velocity increased to 8 $\mu\text{m/s}$, the directional heat treatment structure was as is shown in Figure 2b, compared with the withdrawing velocity of 3 $\mu\text{m/s}$, the large grain size decreased, the maximum grain size was about 100 μm , and the number of small grains increased. When the withdrawing velocity was further increased to 20 $\mu\text{m/s}$, the grain size became smaller, as shown in Figure 2c, because the grain boundary migration rate of pure copper wire was slow, the average grain size was smaller than that when the withdrawing velocity was slow, and the grain heating time was shortened due to the increase in the withdrawing velocity, so that the grain could not grow sufficiently.

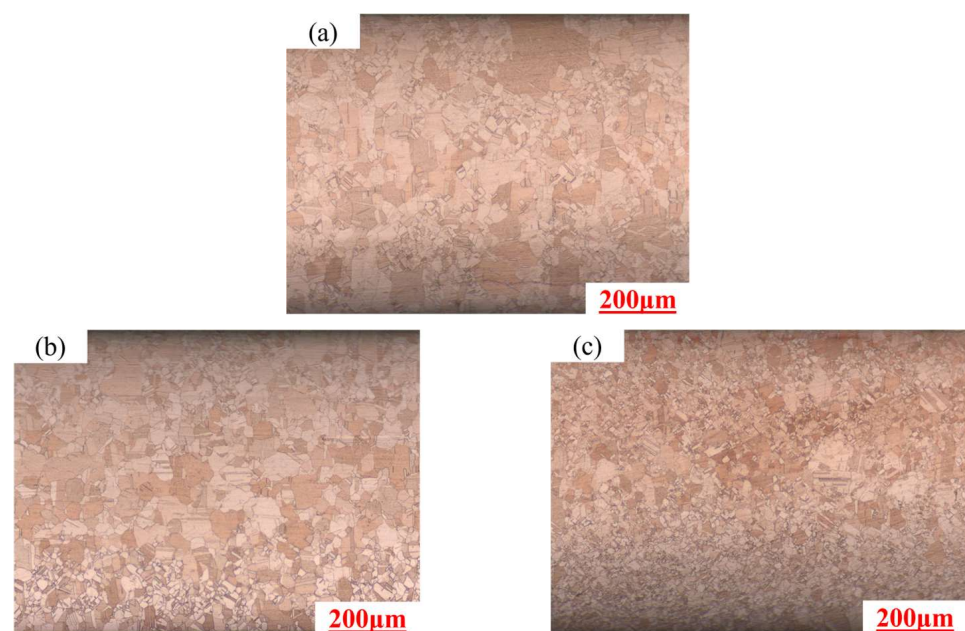


Figure 2. Directional heat treatment microstructure of 87% pure copper wire at 600 °C and different withdrawing velocities: (a) 3 $\mu\text{m/s}$; (b) 8 $\mu\text{m/s}$; (c) 20 $\mu\text{m/s}$.

For a pure copper wire with a deformation of 89%, when the withdrawing velocity was 3 $\mu\text{m/s}$, the directional heat treatment structure was as is shown in Figure 3a. Due to the slow withdrawing velocity, most of the grains had already occurred before entering the hot zone. Secondary recrystallization had occurred, and the structure after heat treatment was coarse equiaxed grains, with the largest grain size of about 380 μm , and some grains were shown to have a tendency to grow axially. When the withdrawing velocity increased to 8 $\mu\text{m/s}$, the microstructure after directional heat treatment was as is shown in Figure 3b, columnar crystals that grew along the axial direction can be clearly seen and the maximum columnar crystal length exceeded 2 mm. When the withdrawing velocity was further increased to 20 $\mu\text{m/s}$, the microstructure after directional heat treatment was as is shown in Figure 3c, because the withdrawing velocity was larger, the grain heating time became shorter and the growth was limited, the columnar crystal characteristics became weaker, the grains tended to form equiaxed crystals, and some small size grains were also mixed in the microstructure.



Figure 3. Directional heat treatment microstructure of 89% pure copper wire at 600 °C and different withdrawing velocities: (a) 3 µm/s; (b) 8 µm/s; (c) 20 µm/s.

The results show that no columnar crystals appeared in the pure copper wire with 87% deformation after directional heat treatment at 600 °C, and the columnar crystals could be obtained in pure copper wire with 89% deformation after directional heat treatment with the change in withdrawing velocity. The reason for this phenomenon lies in the difference in initial crystallographic texture of pure copper wires with two deformation amounts.

3.2. Effect of Heat Treatment Parameters on Microstructure of Pure Copper Wire After Directional Heat Treatment

In addition to deformation, the heating temperature, withdrawing velocity, and other heat treatment parameters were also important factors affecting the directional heat treatment microstructure of the pure copper wire.

Grain boundary mobility and temperature satisfied the Arrhenius equation as follows:

$$M = M_0 \exp\left(-\frac{Q}{RT}\right) \quad (2)$$

where M is the grain boundary mobility, T is the temperature, R is the gas constant, Q is the activation energy of grain boundary migration, and M_0 is the preceding factor.

According to Formula (2), the higher the temperature, the more favorable the grain boundary migration. Considering the advantages of directional heat treatment technology, the heating temperature should not be too high. When the heating temperature was 850 °C, the temperature at the front end of the hot zone far exceeded the critical temperature of secondary recrystallization, which had a weak inhibition effect on most grain growth. A total of 89% pure copper wire was used in this experiment. The heating temperature was set at 450 °C, 550 °C, 650 °C and 750 °C. The withdrawing velocity ranged from 1 to 100 µm/s.

Figure 4 shows the directional heat treatment microstructure at 450 °C with different withdrawing velocities. The drawing direction changes from left to right, and the withdrawing velocity changes from slow to fast. When the withdrawing velocity was 3 µm/s, the oriented heat treatment structure was a larger equiaxed crystal with an average grain size of about 240 µm, and when the withdrawing velocity was 5 µm/s, a more obvious oriented structure appeared, as shown in Figure 4b, several longer columnar crystals were distributed among them; the length was about 1.1 mm and the width was about 0.2 mm. When the withdrawing velocity increased to 10 µm/s, the orientation effect became worse, the columnar crystal character was weak, the grains showed equiaxed

tendency, the columnar crystal length was about 0.7 mm, and the width was about 0.3 mm. When the withdrawing velocity increased to 15 $\mu\text{m/s}$, the columnar crystal disappeared, the heat treatment microstructure was all equiaxed crystal, and the average grain size was small.

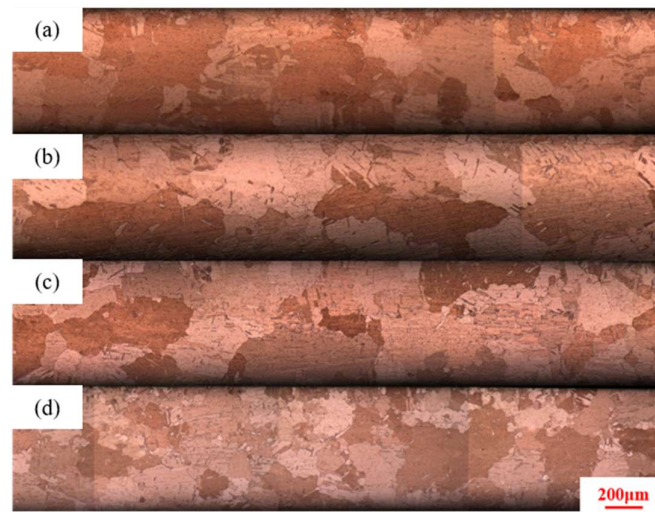


Figure 4. Directional heat treatment microstructure at 450 °C and different withdrawing velocities: (a) 3 $\mu\text{m/s}$; (b) 5 $\mu\text{m/s}$; (c) 10 $\mu\text{m/s}$; (d) 15 $\mu\text{m/s}$.

Figure 5 shows the directional heat treatment structure at different withdrawing velocities at heating temperature of 550 °C. The withdrawing velocities corresponding to Figure 5a–e are 3 $\mu\text{m/s}$, 5 $\mu\text{m/s}$, 10 $\mu\text{m/s}$, 15 $\mu\text{m/s}$ and 20 $\mu\text{m/s}$, respectively, and the drawing direction is from left to right. When the withdrawing velocity was 3 $\mu\text{m/s}$, the microstructure was coarse equiaxed crystal with an average grain size of about 260 μm . When the withdrawing velocity was increased to 5 $\mu\text{m/s}$, columnar crystal with a length of about 1.5 mm and a width of about 0.4 mm was obtained. When the withdrawing velocity was 10 $\mu\text{m/s}$, the aspect ratio of columnar crystal was larger, with a length of about 2 mm and a width of about 0.4 mm. When the withdrawing velocity increased to 15 $\mu\text{m/s}$, the columnar crystals were about 1.2 mm in length and 0.3 mm in width, and when the withdrawing velocity increased to 20 $\mu\text{m/s}$, the columnar crystals were almost equiaxed.

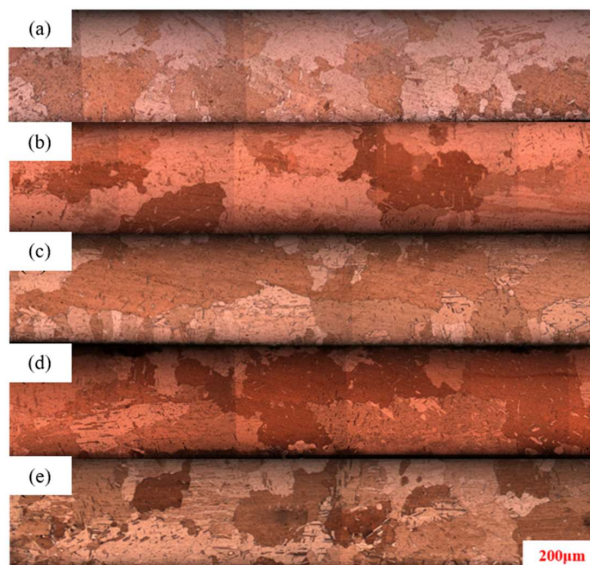


Figure 5. Directional heat treatment microstructure at 550 °C and different withdrawing velocities: (a) 3 $\mu\text{m/s}$; (b) 5 $\mu\text{m/s}$; (c) 10 $\mu\text{m/s}$; (d) 15 $\mu\text{m/s}$; (e) 20 $\mu\text{m/s}$.

When the heating temperature is 650 °C, the directional heat treatment structure is shown in Figure 6a–e, the corresponding withdrawing velocities are 3 $\mu\text{m/s}$, 5 $\mu\text{m/s}$, 12 $\mu\text{m/s}$, 18 $\mu\text{m/s}$ and 25 $\mu\text{m/s}$, respectively, and the drawing direction is from left to right. A withdrawing velocity of 3 $\mu\text{m/s}$ is relatively slow, the microstructure of oriented heat treatment is coarse equiaxed crystal with an average size of about 250 μm . With the increase in withdrawing velocity, the microstructure was gradually oriented, and when the withdrawing velocity increased to 5 $\mu\text{m/s}$, the crystal grew along the drawing direction into columnar crystal with a length of about 1.3 mm and a width of about 0.3 mm. When the withdrawing velocity was 12 $\mu\text{m/s}$, more obvious columnar crystal with a length of 2.6 mm and a width of about 0.4 mm appeared. When the withdrawing velocity was increased to 18 $\mu\text{m/s}$, the columnar crystal length decreased to 1.6 mm and width was about 0.3 mm.

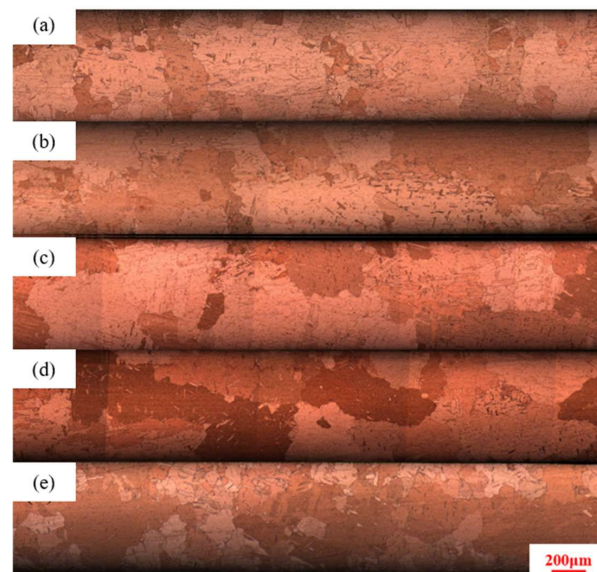


Figure 6. Directional heat treatment microstructure at 650 °C and different withdrawing velocities: (a) 3 $\mu\text{m/s}$; (b) 5 $\mu\text{m/s}$; (c) 12 $\mu\text{m/s}$; (d) 18 $\mu\text{m/s}$; (e) 25 $\mu\text{m/s}$.

When the heating temperature was 750 °C, the directional heat treatment structure can be seen in Figure 7a–f, and the corresponding withdrawing velocities were 3 $\mu\text{m/s}$, 5 $\mu\text{m/s}$, 15 $\mu\text{m/s}$, 25 $\mu\text{m/s}$, 35 $\mu\text{m/s}$, and 45 $\mu\text{m/s}$, respectively, and the drawing direction was from left to right. Because the withdrawing velocity of 3 $\mu\text{m/s}$ is slow, the average size of coarse equiaxed crystals exceeds 300 μm , as shown in Figure 7a. When the withdrawing velocity was increased to 5 $\mu\text{m/s}$, coarse columnar crystals began to form, with a length of about 2.2 mm and a width of about 0.5 mm. When the withdrawing velocity was increased to 15 $\mu\text{m/s}$, the columnar crystals in the oriented heat treatment structure had the longest length, exceeding 3.5 mm and a width of about 0.5 mm. The length and width of columnar crystals decreased with the increase in withdrawing velocity. When the withdrawing velocity was 25 $\mu\text{m/s}$, the length and width of columnar crystals were about 1.8 mm and 0.4 mm, respectively. When the withdrawing velocity was 35 $\mu\text{m/s}$, the length and width of columnar crystals were about 0.8 mm and 0.3 mm, respectively. When the withdrawing velocity was 45 $\mu\text{m/s}$, some small equiaxed crystals were mixed in the heat-treated microstructure due to the fast withdrawing velocity.

Figure 8 is the statistical diagram of microstructure characteristics after directional heat treatment at different heating temperatures and withdrawing velocities. It can be seen from Figure 8a that the grain length increased first and then decreased with the increase in withdrawing velocity at the same heating temperature; the withdrawing velocity

corresponding to the maximum grain length is considered as the optimal withdrawing velocity. The higher the heating temperature, the greater the optimal withdrawing velocity, and the longer the grains obtained after directional heat treatment. The aspect ratio is the ratio of the length to the width of columnar crystals. The aspect ratio of equiaxed crystals is 1. As shown in Figure 8b, columnar crystal structures could be obtained only within a certain range of withdrawing velocities. If the withdrawing velocity was too fast, the grain heating time was short, this meant that the grain boundary migration rate was far behind the sample movement rate, the advantage of preferential migration of grain boundaries could not be reflected, and therefore only equiaxed crystals could be obtained. If the withdrawing velocity was too slow, the grain heating time was long, this meant that the number of secondary recrystallization grains increased and the advantage of grain growth was relatively weakened, which is unfavorable for the development of columnar crystals, and therefore larger equiaxed crystals were formed.

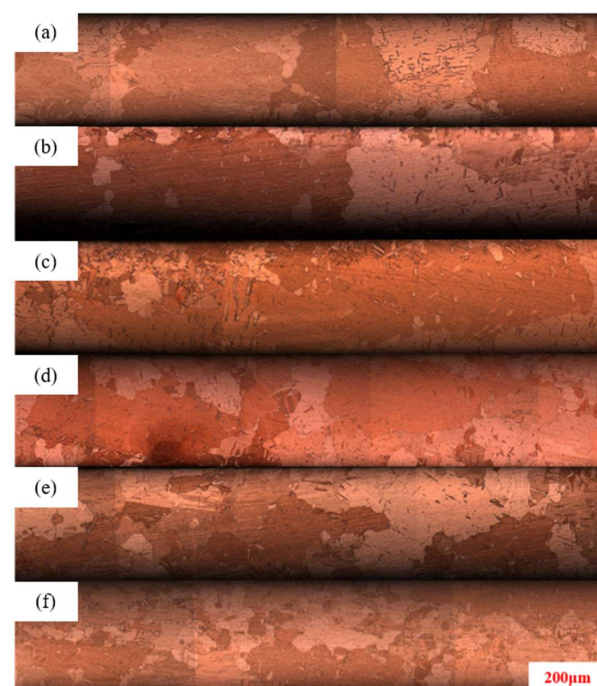


Figure 7. Directional heat treatment microstructure at different withdrawing velocities at 750 °C: (a) 3 $\mu\text{m/s}$; (b) 5 $\mu\text{m/s}$; (c) 15 $\mu\text{m/s}$; (d) 25 $\mu\text{m/s}$; (e) 35 $\mu\text{m/s}$; (f) 45 $\mu\text{m/s}$.

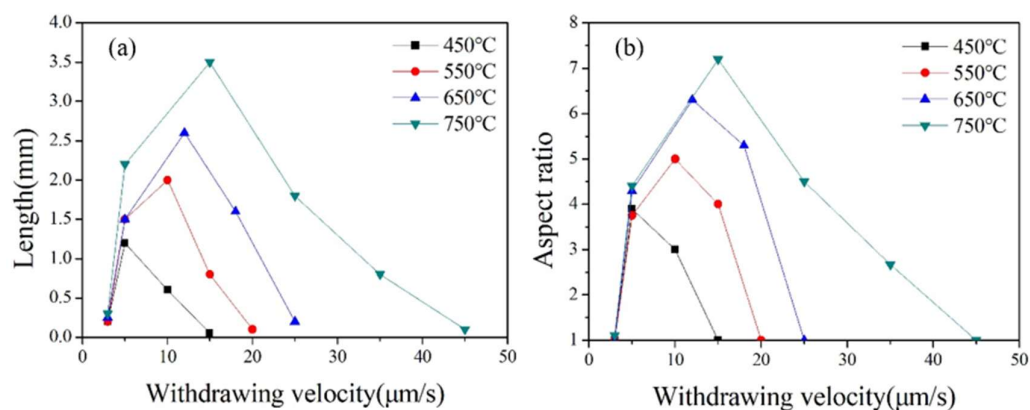


Figure 8. Statistical diagram of microstructure characteristics of directional heat treatment at different heating temperatures and withdrawing velocities: (a) maximum length of columnar crystals; (b) maximum aspect ratio of columnar crystals.

The microstructure after heat treatment is almost that of equiaxed crystal when the withdrawing velocity was low and the grains developed into columnar crystal, gradually, with the increase in withdrawing velocity. Different heat treatment temperatures correspond to an optimal withdrawing velocity, and the aspect ratio of columnar crystals obtained after heat treatment was the largest under this condition. When the withdrawing velocity exceeded the optimum withdrawing velocity, the aspect ratio of columnar crystals decreased with the increase in withdrawing velocity. The higher the heat treatment temperature, the larger the range of withdrawing velocity, the larger the optimal withdrawing velocity, and the larger the aspect ratio of columnar crystal.

In conclusion, the grain growth along the drawing direction was more and more obvious when the withdrawing velocity increased to the optimum withdrawing velocity, so the columnar crystal length can be lengthened by increasing the withdrawing velocity. When the withdrawing velocity reached the optimum value, the advantage of grain growth along the drawing direction reached the maximum, and the aspect ratio of columnar crystal reached the maximum under this condition. Further increases in withdrawing velocity will shorten the heating time of grains, restrain the growth of grains, weaken the tendency of preferential growth of grains, and decrease the aspect ratio of columnar grains. There is an average grain boundary migration rate at a certain heat treatment temperature, and the average grain boundary migration rate approaches the withdrawing velocity at the optimum withdrawing velocity, so the aspect ratio of columnar crystals obtained will be the largest.

When the actual withdrawing velocity is higher than the optimum withdrawing velocity, the average migration rate of grain boundary is lower than the withdrawing velocity during directional heat treatment, and the growth of columnar crystal is restricted. When the actual withdrawing velocity is less than the optimum withdrawing velocity, the average grain boundary migration rate is higher than the withdrawing velocity during directional heat treatment, most grains undergo secondary recrystallization, and the growth of columnar grains is limited, so the withdrawing velocity is the main limiting factor. The relationship between grain boundary migration rate and temperature satisfies Arrhenius equation. The higher the temperature, the greater the grain boundary migration rate, the greater the optimal withdrawing velocity matching it, and the greater the maximum aspect ratio of columnar crystals obtained after directional heat treatment.

3.3. Effect of Directional Heat Treatment Process on Electrical Conductivity of Pure Copper Wire

Electron scattering is the basic reason for metal resistance. Grain boundary is the accumulation place of vacancy, dislocation, and impurity, which often becomes the electron scattering center, making the resistivity of conductor increase and the conductivity decrease. Research shows that the grain boundary perpendicular to the electron movement direction, that is, transverse grain boundary, plays a leading role in the electron scattering process.

In this paper, columnar crystal structure is obtained by directional heat treatment of pure copper wire with different processes, and transverse grain boundary is eliminated. The resistance of pure copper wire was measured by PPMS equipment (physical property comprehensive measurement system). After taking the average value and substituting it into Formula (1), the average resistivity was calculated, and then the reciprocal was taken to obtain the average conductivity.

First of all, the effect of withdrawing velocity on conductivity was tested. The directional heat treatment process parameters of samples to be tested were heating temperature 750 °C, withdrawing velocity 0 $\mu\text{m/s}$, 3 $\mu\text{m/s}$, 5 $\mu\text{m/s}$, 15 $\mu\text{m/s}$, 25 $\mu\text{m/s}$, 35 $\mu\text{m/s}$, and 45 $\mu\text{m/s}$. When the withdrawing velocity was 0 $\mu\text{m/s}$, it means the heat treatment was 30

min. The test results are shown in Table 2. The change in conductivity and resistivity with withdrawing velocity is shown in Figure 9.

Table 2. Test results of pure copper wire after directional heat treatment at 750 °C.

Withdrawing Velocity/($\mu\text{m/s}$)	0	3	5	15	25	35	45
average resistance/ Ω	0.00357	0.00283	0.00272	0.00344	0.00258	0.00270	0.00352
cross-sectional area/ cm^2	0.00567	0.00567	0.00567	0.00567	0.00567	0.00567	0.00567
length/cm	11.497	9.231	8.992	11.647	8.568	8.913	11.378
average resistivity/($10^{-8} \Omega\text{m}$)	1.76037 ± 0.01760	1.73733 ± 0.01564	1.71228 ± 0.01884	1.67653 ± 0.02012	1.71049 ± 0.00855	1.71957 ± 0.00688	1.75315 ± 0.01753
average conductivity/(10^7 s/m)	5.68062 ± 0.05681	5.75596 ± 0.05180	5.84017 ± 0.06424	5.96470 ± 0.07158	5.84628 ± 0.02923	5.81541 ± 0.02326	5.70402 ± 0.05704

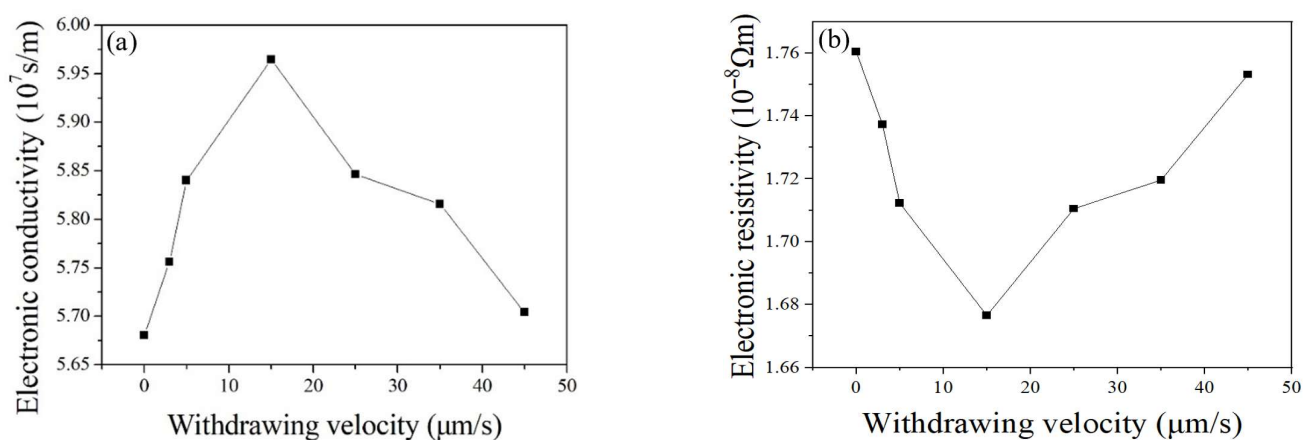


Figure 9. Electrical conductivity of pure copper wire after directional heat treatment at 750 °C: (a) average conductivity; (b) average resistivity.

The conductivity at a pull rate of 15 $\mu\text{m/s}$ is $5.9647 \times 10^7 \text{ s/m}$, was the highest of all test results. When the withdrawing velocity was lower than 15 $\mu\text{m/s}$, the conductivity increased with the increase in withdrawing velocity, and when the withdrawing velocity was higher than 15 $\mu\text{m/s}$, the conductivity decreased with the increase in withdrawing velocity. The overall variation trend was similar to the effect of withdrawing velocity on oriented heat treatment microstructure. Compared with the conductivity at 750 °C with a withdrawing velocity of 0 during directional heat treatment of $5.68062 \times 10^7 \text{ s/m}$, the maximum conductivity of pure copper wire after directional heat treatment increases by 5%.

Secondly, the effect of heating temperature on electrical conductivity was tested. The directional heat treatment process parameters were a withdrawing velocity of 10 $\mu\text{m/s}$ and heating temperatures of 25 °C, 450 °C, 550 °C, 650 °C, and 750 °C. A temperature of 25 °C corresponds to the conductivity at room temperature, the test results are shown in Table 3, and the change in conductivity and resistivity with heating temperature is shown in Figure 10.

It can be seen that the conductivity of pure copper wire after directional heat treatment is higher than that of the initial material, and the conductivity increases with the increase in heating temperature. This is because the temperature gradient at the front end of the hot zone increases with the increase in heating temperature; 10 $\mu\text{m/s}$ approaches the optimal withdrawing velocity, resulting in the increase in the aspect ratio of columnar crystals, which, in turn, results in the continuous increase in the conductivity of the wire. There-

fore, the conductivity of pure copper wire can be improved by adopting an appropriate directional heat treatment process.

Table 3. Test results of pure copper wire after directional heat treatment at different heating temperatures with withdrawing velocity of 10 $\mu\text{m/s}$.

Heating Temperatures/ $^{\circ}\text{C}$	25	450	550	650	750
average resistance/ Ω	0.00158	0.00155	0.00349	0.00268	0.00348
cross-sectional area/ cm^2	0.00567	0.00567	0.00567	0.00567	0.00567
length/cm	5.094	5.036	11.513	8.922	11.621
average resistivity /($10^{-8} \Omega\text{m}$)	1.76288 ± 0.00881	1.74715 ± 0.01223	1.71977 ± 0.01376	1.70366 ± 0.00341	1.69950 ± 0.00339
average conductivity /(10^7 s/m)	5.67254 ± 0.02836	5.72361 ± 0.04007	5.81473 ± 0.04652	5.86972 ± 0.01174	5.88408 ± 0.01177

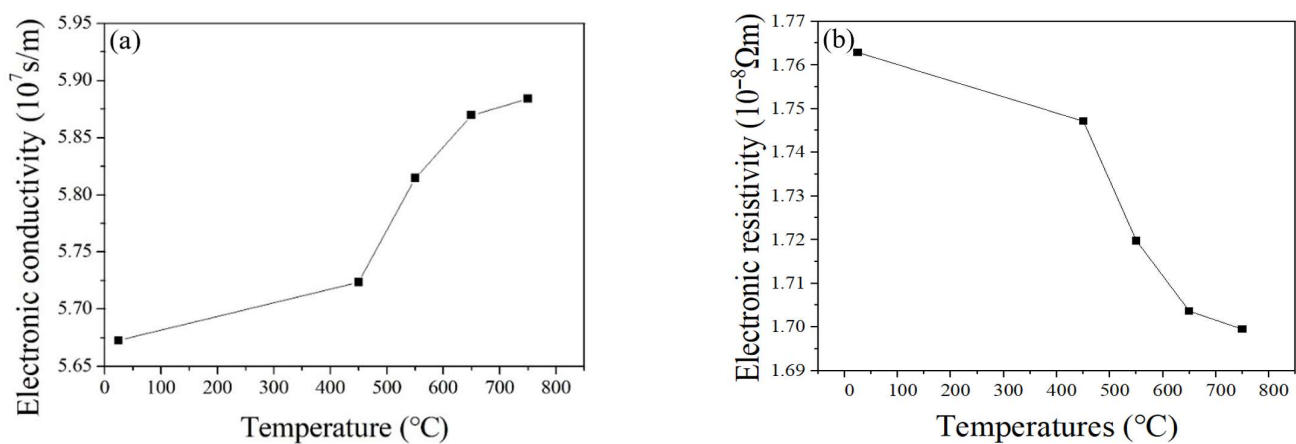


Figure 10. Electrical conductivity of pure copper wire after directional heat treatment at withdrawing velocity of 10 $\mu\text{m/s}$: (a) average conductivity; (b) average resistivity.

4. Conclusions

This paper studies the microstructure evolution and influencing factors of pure copper wires during directional heat treatment, optimizes process parameters such as heating temperature and withdrawing velocity, increases the electrical conductivity of pure copper wires by 5%, and draws the following main conclusions:

1. After directional heat treatment at 600 $^{\circ}\text{C}$, for the pure copper wire with a deformation amount of 87%, no columnar crystals appear regardless of the withdrawing velocity. For the pure copper wire with a deformation amount of 89%, after directional heat treatment at 600 $^{\circ}\text{C}$, when the withdrawing velocity increases to 8 $\mu\text{m/s}$, columnar crystals growing along the axial direction can be clearly observed.
2. The heating temperature and withdrawing velocity have significant effects on the directional grain growth of cold-drawn pure copper wire. Each heating temperature corresponds to an optimum withdrawing velocity, and the aspect ratio of columnar crystal is maximum. When the withdrawing velocity is less than the optimum withdrawing velocity, the aspect ratio of columnar crystal increases with the increase in withdrawing velocity, and when the withdrawing velocity is greater than the optimum withdrawing velocity, the aspect ratio of columnar crystal decreases with the increase in withdrawing velocity. When the heating temperature is 750 $^{\circ}\text{C}$ and the withdrawing velocity is 15 $\mu\text{m/s}$, the maximum aspect ratio of columnar crystal is seven.

3. The heating temperature and withdrawing velocity affect the conductivity of pure copper wire by affecting the columnar crystal structure. The larger the aspect ratio of columnar crystal, the higher the conductivity of pure copper wire. When the heating temperature is 750 °C and the withdrawing velocity is 15 µm/s, the conductivity of pure copper wire increases by 5%.

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