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MICROSTRUCTURE EVOLUTION AND PROPERTY CHANGES OF Al-Si-Cu ALLOY BY CONTROLLING DIRECTIONAL SOLIDIFICATION PARAMETERS

RAZVOJ MIKROSTRUKTURE IN SPREMEMBA LASTNOSTI ZLITINE Al-Si-Cu S KONTROLIRANJEM PARAMETROV DIREKTNEGA STRJEVANJA

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As the properties of the Al-Si-Cu alloy depend on the microstructure, it is necessary to study the effects of processing parameters on the microstructure to control the properties of the Al-Si-Cu alloy. A novel Al-Si-Cu alloy rod was prepared using directional solidification (DS) of liquid metal cooling and the Al-Si-Cu alloy rod was investigated by using a scanning electron microscope (SEM), a JVJ–50s test machine, a MHVD–1000IS microhardness tester and a FT300 resistivity tester. The SEM was used to capture micrographs. The JVJ–50s test machine was employed to measure stress-strain relationships. The MHVD–1000IS microhardness tester was used to measure the microhardness of the specimens. The FT300 resistivity tester was used to evaluate the electrical conductivity. Experimental results indicate that the dendrite structure of the microstructure can be refined by increasing the melting temperatures during DS, but the exorbitant temperature would make the precipitated phase coarsen. It was found that when the melting temperature was 750 °C and the pulling rate was 300 $\mu m\cdot s^{-1}$, the maximum tension strength and microhardness were 232 MPa and 102 HV, respectively. Moreover, when the vacuum was 0.4×10^{-3} MPa, the electrical properties of the alloy are the best, i.e., the minimum resistivity was 17.6 $\mu\Omega\cdot mm$. Therefore, a set of optimum processing parameters, i.e., the melting temperature of 750 °C, the pulling rate of 300 $\mu m\cdot s^{-1}$ and the vacuum of 0.4×10^{-3} MPa, can be selected to improve the microstructure and properties of the Al-Si-Cu alloy.

Keywords: directional solidification, Al-Si-Cu alloy, microstructure, mechanical properties, electrical resistivity

Lastnosti Al-Si-Cu zlitin so močno odvisne od mikrostrukture. Zato je za nadzor njihovih lastnosti potrebno raziskati učinek procesnih parametrov na formiranje njihove mikrostrukture. Avtorji so sintetizirali novo zlitino na osnovi Al-Si-Cu s postopkom direktnega strjevanja (DS) raztaljene kovine. Ohlajene palice zlitine Al-Si-Cu izbrane kemijske sestave so preiskovali s pomočjo metalografskih metod z uporabo vrstičnega elektronskega mikroskopa (SEM), trgalnega stroja JVJ–50s, merilnika mikrotrdote MHVD–1000IS, in merilnika električne upornosti FT300. SEM so uporabili za izdelavo metalografskih posnetkov. S pomočjo JVJ–50s stroja so izdelali krivulje odvisnosti med napetostjo in deformacijo. Z merilnikom mikrotrdote MHVD–1000IS so določili mikrotrdoto vzorcev sintetizirane zlitine. Z merilnikom FT300 pa so določili njihovo električno upornost. Eksperimentalni rezultati preiskav so pokazali, da je dendritno mikrostrukturo možno rafinirati (udrobiti) z višanjem temperature pregretja nad tališče zlitine med DS. Toda pretirano povišvanje temperature pregretja lahko vodi do nastanka grobih izločkov sekundarne faze. Avtorji ugotavljajo, da so pri temperaturi pregretja 750 °C in hitrosti vlečenja strjene palice iz taline 300 $\mu m\cdot s^{-1}$, dosegli maksimalno natezno trdnost 232 MPa in mikrotrdoto zlitine 102 HV. Nadalje ugotavljajo, da so pri vakuumiranju na podtlak 0,4 $\times 10^{-3}$ MPa dosegli najboljše električne lastnosti zlitine z najmanjšo električno upornostjo 17,6 $\mu\Omega\cdot mm$. Tako so določili skupino optimalnih procesnih parametrov, in sicer temperaturo pregretja pri 750 °C, hitrosti vlečenja 300 $\mu m\cdot s^{-1}$ in vakuum 0,4 $\times 10^{-3}$ MPa. Na ta način so lahko izboljšali mikrostrukturo in lastnosti izbrane Al-Si-Cu zlitine.

Ključne besede: direktno strjevanje, zlitina Al-Si-Cu, mikrostruktura, mehanske lastnosti, električna upornost

1 INTRODUCTION

Aluminum alloys are widely used in the fields of aerospace, ships and automobiles.[1] The Al-Si-Cu alloy is one of the most popular cast aluminum alloys, which not only has good casting properties, but also has the advantages of low density, good corrosion resistance, small linear expansion coefficient, high strength and microhardness, good wear resistance and heat resistance, etc.[2,3] However, with the development of advanced manufacturing technology, the comprehensive mechanical properties of Al-Si-Cu alloys need to be further improved to meet the requirements of modern mechanical structures.

A lot of research has been done on improving the properties of Al-Si-Cu alloys. Similarly, some publications focused on the improvement of properties of Al-Si-Cu alloys by adding Mg,[4] rare-earth elements[5] and trace elements and changing the heat-treatment process and different casting forming processes includes squeeze casting, semi-solid forming,[6] lost foam casting[7] and vacuum die casting,[8] etc.

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The directional solidification (DS) technique used in this work is a new casting process that controls the unidirectional heat flow through specific temperature gradients to achieve the desired orientation of the tissue and high-performance materials.[9] Especially in the study of changing the performance of aluminum-based alloys,[10,11] it has been rapidly developed since its birth. Y. J. Sun et al.[12] studied the effect of directional solidification on the microstructure and properties of pure aluminum; the results show that the directional solidification can improve the electrical resistivity of the aluminum rods compared with the ordinary casting process. G. Wan et al.[13] studied the effects of current treatment on the DS of the Al-Si alloy, the results show that the directional solidification is the most obvious at the pulling rates of 3 mm/min. K. Q. Sun et al.[14] studied the effect of melt overheating on the microstructure and properties of the DS of the Al-Cu alloy. The results show that the strength of the alloy increases by nearly 60 % and the elongation nearly doubled after the melt overheat treatment. L. H. Cui et al.[15] studied the effects of Cu content on the DS of Al-Cu alloy; it was found that when the content of Cu increased from 1.77 % to 3.00 %, the spacing of primary dendrite increased gradually, but the increase of dendrite spacing was small. N. C. Si et al.[16] studied the effects of the temperature gradient on the primary dendrite spacing of the directionally solidified Al-4.5 % Cu alloy. The results show that when the other solidification parameters are unchanged, with the increase of temperature gradient, the primary dendrites become smaller and smaller, straight, basically parallel distribution. It can be seen that the research on the DS of Al-base alloys is mainly focused on the study of alloy properties by changing the chemical composition of the alloy, the processing method and changing a single processing parameter. However, few researches focus on the comprehensive effect of multiple processing parameters on the microstructure and properties of the DS Al-based alloys. Furthermore, the electrical resistivity of Al-based alloys has no related studies.

Therefore, the aim of this work was to investigate the effects of three processing parameters, i.e., the melting temperatures, the pulling rates and the vacuum, on the microstructure, mechanical properties and electrical resistivity of the Al-Si-Cu alloy under DS conditions. Based on the above objectives, we have done the following work: firstly, Al-Si-Cu alloy rods are prepared using DS under different process parameters. Secondly, a scanning electron microscope (SEM), JVJ–50s test machine, MHVD–1000IS microhardness tester and FT300 resistivity tester are used to measure the microstructure, mechanical properties and electrical resistivity. Finally, the correlation between the processing parameters and the microstructure and the properties are discussed to determine the optimum range of processing parameters.

2 MATERIALS AND METHODS

2.1 Specimens prepared by directional solidification

The DS experiment was carried out in high-vacuum DS equipment, as shown in **Figure 1a**. The schematic diagram of the directional solidification system is shown in **Figure 1b**. DS equipment mainly consists of two parts: the electric arc melting function and the directional solidification function. The raw materials chemical composition consists of 99.99 % Al, 99.95 % Si and 99.99 % Cu, etc., shown in **Table 1**. The raw material particles polished surface clean, put acetone in the surface with ultrasonic shocks out of the stain. Arc melting was carried out to ensure that the alloy was fully melted and mixed evenly. During the smelting process, electromagnetic stirring was applied and reversed repeatedly for more than five times to obtain the experimental speci-

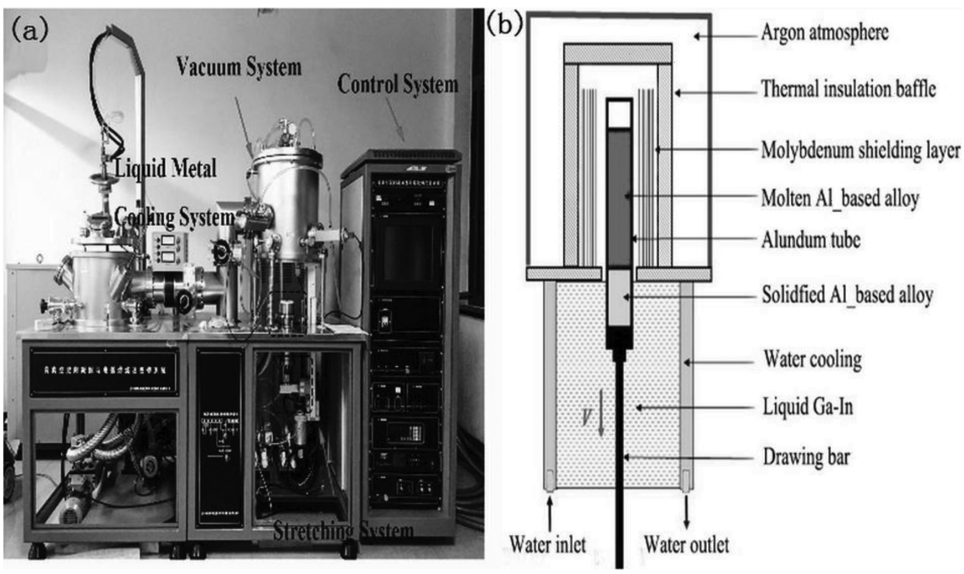

Figure 1: a) directional solidification equipment, b) schematic diagram of directional solidification system[18]

mens. Wire cutting equipment was used to take out the rod with a diameter of 10 mm × 150 mm as the DS specimen.

The DS experiment was carried out using three stages: the heating stage, the insulation stage and the pulling stage. 1) The heating stage: the alloy test rods were placed in a crucible with an inner diameter of 10 mm in the DS equipment. The crucible was an Al_2O_3 corundum tube with a purity of 99.9 %. 2) The furnace of the DS equipment was pumped to a high vaccum using a mechanical pump and a molecular pump. 3) The heating furnace was filled with high-purity argon gas. The alloy rod was heated and melted by resistance heating in a non-pollution environment. To prevent the consumption of alloy elements from evaporation, when the temperature of the test rod reaches the set temperature by the thermocouple, stop heating and begin to enter the insulation stage, which lasts for 5 min. 4) The smelten alloy rod was pulled into the Ga-In alloy solution with a certain pulling rate, when the alloy was cooled to ordinary temperature, the DS experiment was completed. The DS specimens were obtained, as shown in **Figure 2**. 5) The DS specimens were heat treated in a SX2-5-12N box resistance furnace following the T6 temper, which comprised solution heat treatment at 540 °C for 8 h, followed by quenching in 60 °C water. The specimens were then aged at room temperature for 24 h, followed by artificial aging at 155 °C for 5 h, then air cooled.[17]

Table 1: Al-Si-Cu alloy chemical composition (*w*/ %)

Al	Si	Cu	Fe	Mn	Zn
≥87.7	7	4	≤0.5	≤0.5	≤0.3

In DS experiment, different DS specimens were prepared by changing the processing parameters. The melting temperatures varied from 700 °C to 800 °C. The pulling rates varied from 50 $\mu m \cdot s^{-1}$ to 300 $\mu m \cdot s^{-1}$, and the vacuum degree of smelting changes in the range of 4×10^{-4} MPa to 1.2×10^{-3} MPa, shown in **Table 2**.

2.2 Mechanical properties test of specimens

Figure 3 shows the standard plate-shaped tension specimen. Tensile tests were conducted following the ASTM: E8M standard. The mechanical properties were measured using the JVJ–50s tension machine at room temperature. The tension speed of the specimens was 1 $mm \cdot min^{-1}$.

The microhardness of the transverse section was measured by the MHVD-1000IS microhardness tester. Specimens were polished with the abrasive paper of #800 to #2000, cleaned and blown dry with a hair dryer, then placed on a microhardness tester with a set load of 200 N, maintaining the load for 3 s. The microhardness of 7 points was measured, a point was measured 10 times and the average value being obtained.

Table 2: Processing parameters of directionally solidified Al-Si-Cu alloy

Experiment number	Smelting temperature (°C)	Pulling rates ($\mu m \cdot s^{-1}$)	Vacuum ($\times 10^{-3}$ MPa)
1	700	50	0.8
2	700	100	0.8
3	700	200	0.8
4	700	300	0.8
5	750	50	0.4
6	750	50	0.8
7	750	50	1.2
8	750	100	0.8
9	750	200	0.8
10	750	300	0.8
11	800	50	0.8
12	800	100	0.8
13	800	200	0.8
14	800	300	0.8

2.3 Electrical properties test of specimens

The specimen with a size of 10 mm × 15 mm was cut by the DK7632-type linear cutting machine. The surface of the specimen was polished by using water and alcohol to ensure that the surface was smooth and bright. The resistivity of the specimens was tested by using the four-terminal measuring method with an FT300 resistivity tester.

Figure 2: Directional solidification specimens

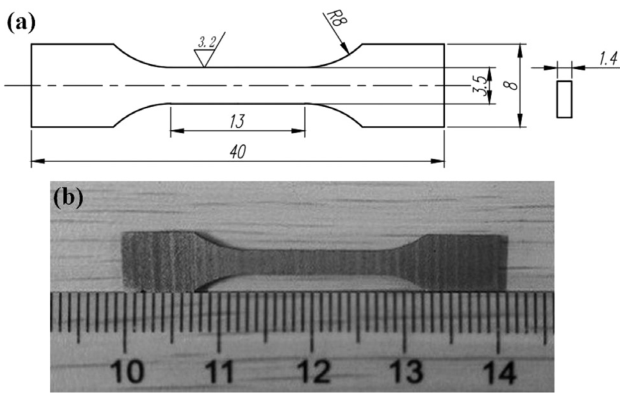

Figure 3: Tension specimens

2.4 Microstructure observation of specimens

Observation specimens were prepared with a size of 2 mm × 2 mm × 2 mm using a linear cutting machine. Grinding and polishing were performed using the machine. Specimens were etched within a solution of 0.5 % HF with 10 s, and then were washed with water and alcohol. Finally, blown dry with a blower. The microstructures of the specimens were captured by SEM.

3 RESULTS AND DISCUSSION

3.1 Microstructure analysis

Figure 4 shows the microstructure of the Al-Si-Cu alloy at different melting temperatures of (700, 750 and 800) °C under the pulling rates of 100 μm·s^{-1} at vacuum degree of 8 × 10^{-4} MPa. It can be seen from **Figure 4a** that the solid-solution dendrites are coarse at 700 °C, which are irregularly distributed in the α-Al matrix. And, there are many eutectics and some disk-shaped eutectic compounds in **Figure 4a**. The grain boundaries can be clearly seen from **Figure 4b**, and a large number of fine second-phase granular eutectics and strengthening phases are dispersed and precipitated in the vicinity of grain boundaries. The solid-solution dendrites are further refined compared with **Figure 4a**. At the same time, a small amount of disk eutectic compounds was distributed. It can be seen from **Figure 4c** that the melting grain boundary disappeared. A large number of short rod-shaped eutectics were found around the matrix,

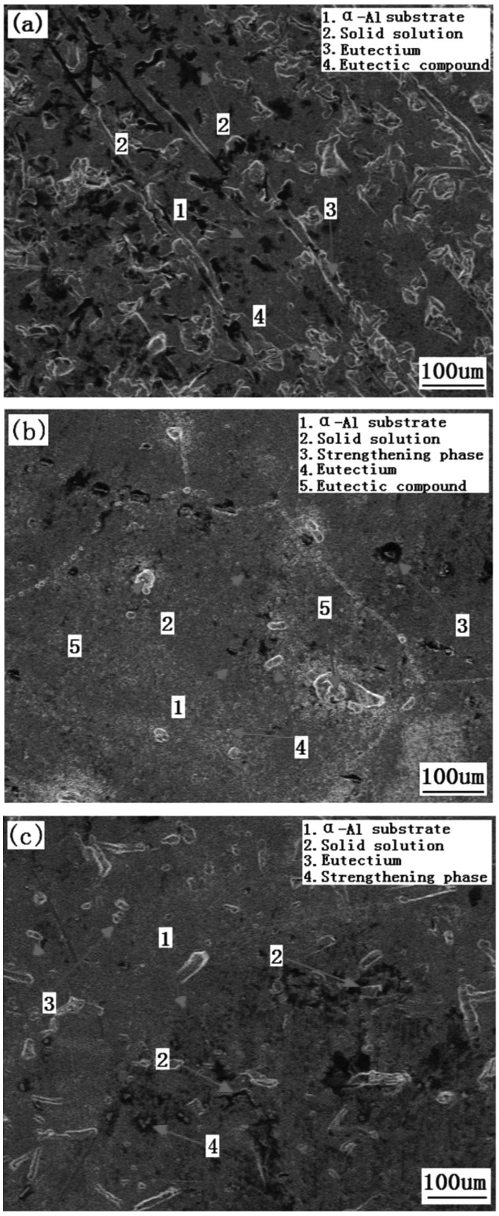

Figure 4: Microstructure of Al-Si-Cu alloy at different melting temperatures with: a) 700 °C, b) 750 °C and c) 800 °C

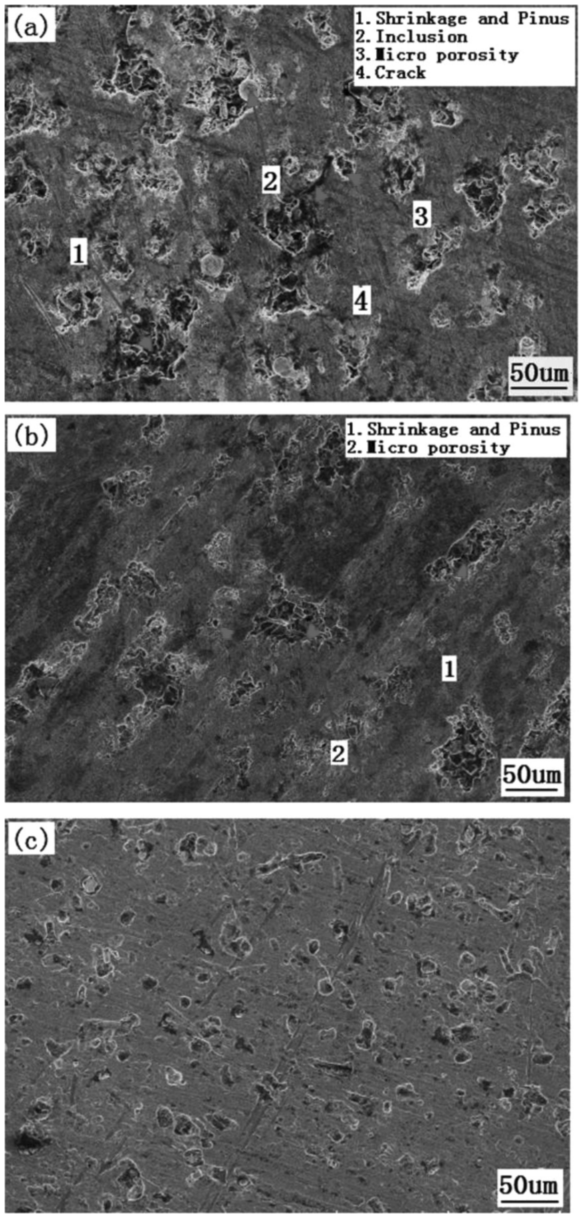

Figure 5: Microstructure of Al-Si-Cu alloy at different degree of vacuum with: a) 1.2 × 10^{-3} MPa, b) 8 × 10^{-4} MPa and c) 4 × 10^{-4} MPa

which are unevenly distributed and also produced a small amount of strip-shaped and plate-shaped eutectics.

Figure 5 shows the effect of the vacuum degree on the microstructure of the Al-Si-Cu alloy under a certain melting temperature of 750 °C and pulling rate of 50 $\mu m \cdot s^{-1}$. It can be seen from **Figure 5a** that there are more defects, shrinkage and pinus more defects when the vacuum degree is 8×10^{-4} MPa, the defects of alloy microstructure are obviously reduced, shown in **Figure 5b**.The size and number of shrinkage pinus are obviously reduced, and a few areas of micro porosity are produced. **Figure 5c** shows that, the alloy evenly organized and the densification effect of the alloy is remarkable, and no obvious defects of the microstructure are produced when the vacuum degree is 4×10^{-4} MPa.

3.2 Effect of the melting temperatures and pulling rates on the mechanical properties and electrical properties of Al-Si-Cu alloy

Figure 6 shows the effect of the melting temperatures and pulling rates on mechanical properties and electrical properties of Al-Si-Cu alloy. It can be seen from **Figure 6a** that the tension strength of the directional solidification alloy first increases and then decreases with the increase of the melting temperatures. When the melting temperature was 750 °C, the maximum tension strength was 232 MPa. Compared to sand mould casting Al-Si-Cu alloy,[19] the tension strength increased by about 18.97 %. In general, at 750 °C, the tension strength of the Al-Si-Cu alloys was the highest. Additionally, the tension strength of Al-Si-Cu alloy first decreases and then increases with the increase of the pulling rates at a given melting temperature. When the pulling rates increase from 100 $\mu m \cdot s^{-1}$ to 300 $\mu m \cdot s^{-1}$, the tension strength of the alloy increased about 23.6 %. So, the pulling rates have a significant influence on the tension strength. It can be seen from **Figure 6b** that the elongation at break increases with the increase of the pulling rates. When the pulling rate increases from 50 $\mu m \cdot s^{-1}$ to 300 $\mu m \cdot s^{-1}$ at a given melting temperature, the elongation at break of the Al-Si-Cu alloy increased about 33–50 %. With the increase of melting temperatures, the elongation at break of the alloy increased first and then decreased. **Figure 6c** shows the effect of different processing parameters on the microhardness of the Al-Si-Cu alloy, when the pulling rates increases from 50 $\mu m \cdot s^{-1}$ to 300 $\mu m \cdot s^{-1}$ at a given melting temperature, the microhardness of the specimen increases. When the melting temperature was 750 °C and the pulling rate was 300 $\mu m \cdot s^{-1}$, the maximum microhardness of the alloy was 102 HV.

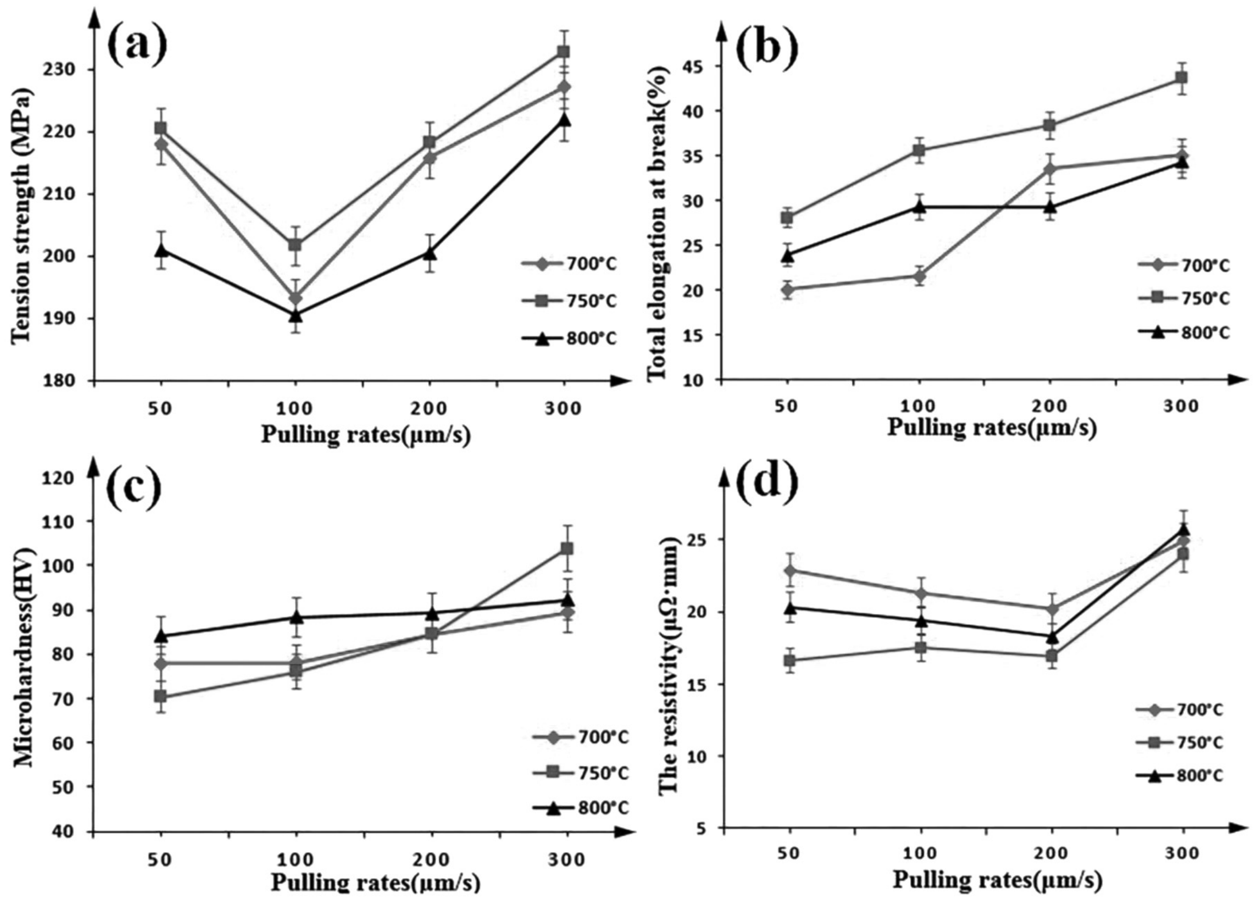

Figure 6: The effect of the melting temperatures and pulling rates on the mechanical properties and electrical properties of the Al-Si-Cu alloy

Mechanical properties variation trend depends on the microstructure evolution in **Figure 4**. In **Figure 4a**, the dendrite of solid solution on α-Al matrix is coarse and uneven. A coarse dendrite structure easily leads to stress concentration, which produces microcracks, and will accelerate the crack growth. Thus, the strength and microhardness of the alloy were reduced. In **Figure 4b**, with the improvement of melting temperatures, the dendritic structure is refined, similar to A356 alloy proposed by H. Liao.[20] Dispersion strengthened granular eutectic not only increases the strength and plasticity of the alloy but also enhances the toughness of the Al-Si-Cu alloy. So, the mechanical properties of the alloy are better. In **Figure 4c**, when the melting temperature continues to rise, the results in granular eutectic aggregation, coarsening phenomenon, so reduces the strength and plasticity of the alloy.

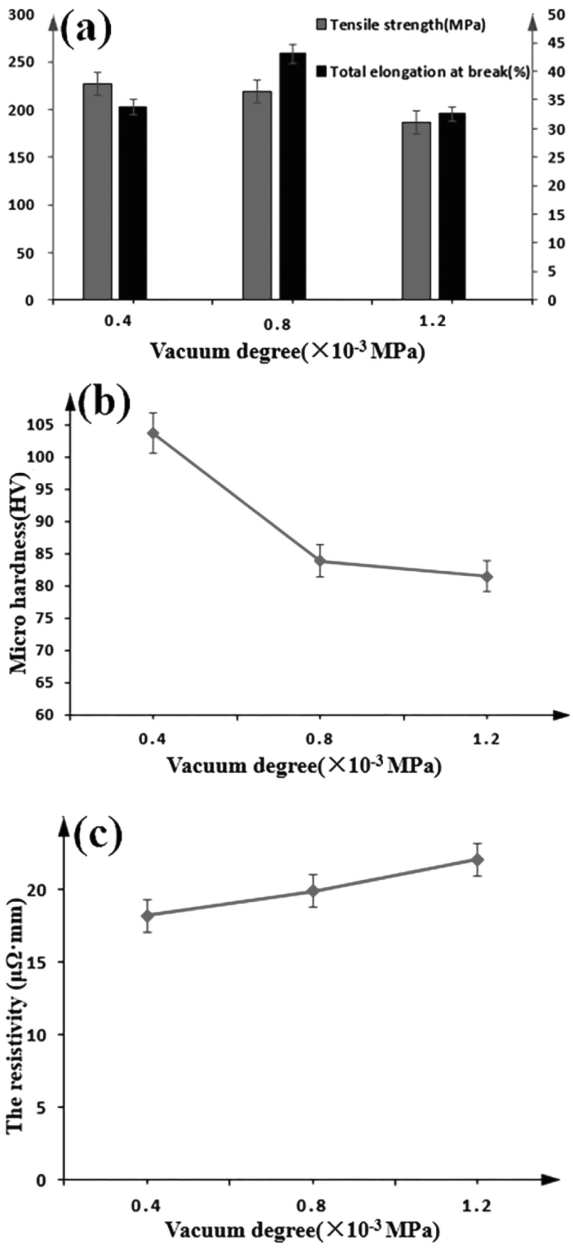

Figure 7: Effects of different vacuum degrees on the mechanical properties and electrical resistivity of the Al-Si-Cu alloy

Figure 6d shows the effects of melting temperatures and pulling rates on the electrical resistivity of the Al-Si-Cu alloy. At room temperature, pure aluminium had a resistivity of 2.94×10^2 μΩ·mm and the pure silicon a resistivity of 2.52×10^5 μΩ·mm. The resistivity of pure copper was 1.85×10^2 μΩ·mm.[21] As can be seen from **Figure 6d**, the resistivity of the novel Al-Si-Cu alloy was lower than that of any alloying element. When the pulling rate varies from 50 μm·s^{-1} to 200 μm·s^{-1}, the resistivity gradually decreases. When the pulling rate exceeds 200 μm·s^{-1}, the resistivity sharply increases, the resistivity increased about 41.9 %. When the melting temperature was 750 °C and the pulling rate was 200 μm·s^{-1}, the average resistivity was about 18.7 μΩ·mm. It indicates that the conductivity was better at the melting temperatures of 750 °C and the pulling rate of 200 μm·s^{-1}.

As shown in **Figure 4**, when the melting temperature was 700 °C, the solid solution on the α-Al matrix has coarse dendrites, and there are many eutectics in the form of strip and plate and some discoid eutectic compounds. Most of these eutectic structures composed of silicon.[22] Silicon is a semiconductor material, which has less conductivity than metal compounds at room temperature. Many primary silicon and eutectic silicon are disordered along with the growth direction, which against current delivery, so the resistivity is larger. When the melting temperature was 750 °C, a large number of fine secondary granular eutectic and strengthening phases are dispersed in the α-Al matrix, and the dendrite structure of the solid solution is further evenly refined, as shown in **Figure 4b**. The fine microstructure is beneficial to current delivery, so the resistivity is less. As the temperature of 800 °C, too high temperature results in a large number of second phase short rod eutectic and a small amount of stripe and lamellar eutectic in the α-Al matrix, as shown in **Figure 4c**. It also increases the current hindrance, which leads to an increase of the resistivity.

3.3 Effects of vacuum degrees on the mechanical properties and electrical resistivity of Al-Si-Cu alloy

Figure 7 shows the effects of different vacuum degrees on the mechanical properties and electrical resistivity of Al-Si-Cu alloy. It can be seen from **Figure 7a** that the tension strength decreases with the increase of the degree of vacuum. When vacuum degree was 1.2×10^{-3} MPa, the tension strength of the alloy was the lowest, i.e., 191 MPa. When the vacuum degree was 0.4×10^{-3} MPa, the tension strength of the alloy reaches 226 MPa, which is about 19 % higher than that of the alloy rod at the vacuum degree of 1.2×10^{-3} MPa. **Figure 7a** shows that the elongation at break of the alloy rod first increases and then decreases with the increase of the degree of vacuum, and the maximum elongation at break was 43 %. **Figure 7b** shows that when the degree of vacuum increased from 0.8×10^{-3} MPa to 1.2×10^{-3} MPa, the microhardness of the alloy decreased rarely. As the vacuum continues to decrease, the microhardness of the alloy rod increases rapidly, and the microhardness reaches 102 HV at 0.4×10^{-3} MPa. Compared with 1.2×10^{-3} MPa, the microhardness of the alloy rod increases by about 30.7 %.

C. Lee et al.[23] found that the tension properties of Al–Si alloys depend on the microstructural characteristics. It can be seen from **Figure 5** that the microstructure of the alloy rod was compact at 0.4×10^{-3} MPa, and no cracks, shrinkage porosity and other defects were observed. With the decrease of vacuum degree, the shrinkage pinus, micro porosity, crack and other defects in the test rods were obviously decreased and accompanied by inclusions. The shrinkage pinus is caused by liquid shrinkage and volume shrinkage between solid and liquid lines during solidification of the alloy. The vacuum degree is too low, the original air in the cavity invades the liquid metal.

Figure 7c shows the effect of different vacuum degrees on the electrical resistivity of the Al-Si-Cu alloy. It can be seen that the resistivity of the test rods gradually decreases with the increases of the degree of vacuum, and the conductive ability becomes better. When the degree of vacuum was 0.4×10^{-3} MPa, the lowest resistivity was 17.6 μΩ·mm. It can be seen from **Figure 5** that when the vacuum degree is lower, the microstructure defects are much more, so that the resistivity of the alloy is higher.

4 CONCLUSIONS

1) With the increase of melting temperatures, the eutectic on the α-Al matrix was refined from strip and plate to short rod and granular, and the number of second phases increased. When the melting temperature is too high, the second phase begins to coarsen and grow. At 750 °C, the microstructure of Al-Si-Cu is the best and more uniform. Furthermore, the higher the vacuum degree of the alloy in solidification process, the less defects within the microstructure of the alloy.

2) With the increase of the pulling rates, the tension strength first decreases and then increases, the elongation at break and microhardness increase all the time, the resistivity first decreases and then increases. At the melting temperature of 750 °C and the pulling rate of 300 $\mu m \cdot s^{-1}$, the mechanical properties of the alloy are the best, i.e., the tension strength is 232 MPa, and the microhardness is 102 HV, the maximum tension strength and microhardness increased about 21.4 % and 47.1 %, respectively. At the melting temperature of 750 °C and the pulling rate of 200 $\mu m \cdot s^{-1}$, the electrical properties of the Al-Si-Cu are the best, the minimum resistivity is 18.2 μΩ·mm.

3) With the increase of the vacuum degrees, the tension strength and microhardness of the alloy gradually increase, and the electrical resistivity gradually decreases. The maximum tension strength and microhardness of the Al-Si-Cu alloy is 226 MPa and 102 HV, respectively, at a vacuum degree of 0.4×10^{-3} MPa. When the vacuum degree is 0.4×10^{-3} MPa, the minimum electrical resistivity is the best, i.e., 17.6 μW·mm.

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