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The Effect of Sr composition on the microstructure and mechanical properties of Al-Si-Zn filler for the brazing of AA6061

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Abstract
The microstructure and properties of Al-Si-Zn-Sr filler metals and brazed 6061-T6 aluminum alloy after heat treatment were studied, and the adsorption energy of Sr element on Si (111) surface was calculated. After Sr was added to Al-6Si-40Zn filler metal, its melting range remained unchanged, and the spreading wettability was improved. The microstructure mainly includes $\alpha$-Al solid solution, fibrous Si, and $\eta$-Zn solid solution. Generally speaking, increasing Sr content could improve the average microhardness of brazed joints. The shear strength of the brazed joint containing Al-6Si-40Zn-0.8Sr filler metal was 134.23 MPa, which was 39.3% higher than that of the brazed joint without Sr. The calculation results of adsorption energy showed that the surface of Si (111) mainly adsorbed Sr at the vacancy, which changed the growth mode of Si.

1. Introduction
Aluminum alloy is widely used in plenty of industries such as automotive, aerospace and construction due to its high strength-to-weight ratio, stiffness, formability, excellent fatigue and corrosion resistance. In terms of application, brazing has always been considered as one of the most important connection methods in aluminum component manufacturing because of its advantages of small deformation, high dimensional accuracy as well as high production efficiency [1–3]. Al-Si system has special fluidity and thermal tear resistance due to the action of Si. The brittle hard eutectic Si particles can strengthen the strong ductile Al matrix. The shape, size, and distribution of the Si phase affect thermal machinability [4] and fatigue performance [5]. Si in Al-Si system is layered or needle-like structure, which may lead to the premature failure of castings as these acicular Si particles serve as the stress concentration sites when the castings are loaded [6]. In addition, when brazing alloy with Al-Si solder, the melting point range is 559–591 °C. This is suitable for brazing at 590–605 °C and is close to or even higher than the melting point of many base metals. Therefore, it is of great significance to develop new filler metals with lower melting point, lower cost and high quality.

To solve the problem of excessively high brazing temperature, in recent years, many researchers based on eutectic composition of binary alloys such as Al−Si, Al−Zn, etc, added some new alloying elements by alloying method, and studied the corresponding properties [7–9]. The addition of alloying elements will significantly change the mechanical and chemical properties of aluminum alloy [10, 11], and the existence of the second phase and precipitated phase in the alloy is the main reason for such problems [12]. It is reported that the melting point of Al−Si 20Cu filler metal is about 520 °C [13], but its formability is severely deteriorated because of the existence of a brittle Al$_3$Cu phase, which makes it difficult to process them into wires. S. Y. Chang [14] studied the Al-9.6Si-10Cu-10Zn-0.1Re filler metal used for brazing 6061 aluminum alloy at 530 °C, and found that there were more Al$_3$Cu brittle phases in the brazed joints, leading to a lower shear strength of 72.9 MPa. Moreover, the high-cost Al−Si−Cu−Zn−Re filler metal can’t be used as a commercial brazing alloy. M Kagayama et al [15] studied that the addition of Zn can reduce the melting point of the Al−Si−Zn filler metal as well as improve the spreading performance of the filler metal and the filler metal with good thermof ormability. However, the disadvantage lay in the existence of coarse primary Si in Al−Si−Zn filler metal, which could reduce the strength of...
brazed joint, whereas, the structure of Si and the mechanical properties of the system can be improved by introducing some elements into the filler metal, such as Sr, so that Si could be refined [16, 17]. Dai et al [18] studied the Al-6.5Si-42Zn-0.07Sr filler metals, and found that the addition of Sr did not change the melting point, which was around 520 °C. When brazing 6061 aluminum alloy with this solder, the joint tensile-strength was 14% higher than that of Al-Si-Zn solder. Niu et al [19] reported that the addition of Sr could improve the spreading areas of Al–Si–Ge–Zn filler metal, which was first increased and then decreased. Additionally, the shear strength of the filler metal containing 0.7% Sr is as high as 153.7 MPa, but Ge is so costly that Al-Si-Ge filler metal is unsuitable for industrial applications. Nevertheless, Sr has been widely studied for its good modification effect and long duration.

At present, most studies show that the addition of Sr element is still trace (〈0.1wt%). Therefore, on the basis of Al-Si-Zn filler metal, a new type of Al-Si-Zn-Sr multi-element Al-based solder with low melting point was designed by increasing the Sr content. The adsorption energy of Sr on Si(111) surface was calculated. The microstructure and brazing properties of the filler metals with different Sr contents were tested.

2. Experiments

The base material used in this experiment was 6061-T6 aluminum alloy, and its melting temperature was approximately 592–652 °C. The compositions of the filler metals are listed in table 1. Firstly, the Al-Si-Sr master alloy was prepared by using Al (≥99.99%), Al-20Si (≥99.99%) and Al-10Sr (≥99.99%) as raw materials, which were preheated in a dry crucible. Then the crucible was put into a electrical resistance furnace at 750 °C. After the master alloy melted, the alloy was fully mixed to minimize the specific gravity segregation of Si in the alloy solution. After keeping the temperature for 20–30 min, the refining agent was added. When the refining agent was removed from the surface, it was taken out for air cooling. Secondly, the corresponding Al-Si-Sr master alloy (prepared in Step 1) and Zn (≥99.99%) were calculated and weighed, and the Al-Si-Zn-Sr filler metals with different compositions were prepared by the same smelting method as in Step 1. The alloys need to be remelted at least 3 times to obtain a homogeneous composition.

The melting temperature of these filler metals was measured by Japanese Seiko nano-Tg/DTA7300 at a heating rate of 5 °C min⁻¹ under an argon atmosphere, which was heated from room temperature to 600 °C.

| Alloy   | Chemical compositions (wt%) | Al | Si | Zn | Sr |
|--------|----------------------------|----|----|----|----|
| Filler1 | Bal.                       | 6  | 40 | —  | —  |
| Filler2 | Bal.                       | 6  | 40 | 0.2| —  |
| Filler3 | Bal.                       | 6  | 40 | 0.4| —  |
| Filler4 | Bal.                       | 6  | 40 | 0.6| —  |
The wetting test and the brazing experiment were completed in a box-type resistance furnace, in which the QJ201 flux was used. 0.1 g of filler metals placed in the center of the base material 6061 aluminum alloy (40 mm × 40 mm × 2 mm) (figure 1(a)) and wetted at the temperature of 575 °C for 2 min. The brazing experiment was also carried out at 575 °C for 8 min, and the filler metals (20 mm × 1 mm × 0.5 mm) were used. In the tensile test according to the National Standard of China GB/T 11363–2008 [20], 6061-T6 (50 mm × 20 mm × 2 mm) was used, and the lap length of the joint was 2 mm ± 0.3 mm, and the gap was 0.2 ± 0.05 mm, as shown in figure 1(b). The filler metal foil was placed at the edge of the weld. When the welding temperature was reached, the filler metal foil melted and flowed into the weld. Immediately after brazing, all the samples were taken out of the box-type resistance furnace, air-cooled to room temperature, and cleaned with hot water repeatedly (50 ~ 60 °C) to remove the flux residue. To ensure the accuracy of the results, three specimens were tested with each filler metal under the same conditions, and the average values of the test results were calculated. To improve the mechanical properties of the brazed parts, the heat treatment of the brazed parts was carried out at 500 °C for 1 h and cooled by water, and then the brazed parts were immediately subjected to aging at 190 °C for 4 h and cooled by water.

After brazing, the shear strength of the brazed joint was measured on an Instron 8501 universal testing machine at a tensile speed of 0.5 mm min⁻¹. The microhardness of brazed joints was measured by an MHV-2.0 automatic microhardness tester at three points in each area, and the average value of the results was taken. The microstructures of the filler metals were analyzed by an x-ray diffractometer (XRD, Cu Kα, X’PertProMPD, Nalytical, Holland). The scanning angle was 10° to 90°, the scanning rate was 0.188°/s, the accelerating voltage was 45 kV, and the working current was 40 mA. Scanning electron microscopy (SEM. S-4800, Hitachi, Japan) was coupled with energy dispersive spectrometry (EDS) and was used to observe the microstructure of the filler metals and the fracture morphology of brazed joint. A flowchart of the methodology is shown in figure 2.
3. Computational methods

The calculations were performed in the Materials Studio on the basis of the density functional theory. The Perdew–Burke–Ernzerhof (PBE) pseudopotential [21] with the generalized gradient approximation (GGA) was adopted in the calculations. The surface of Si(111) was modeled by a supercell containing a slab of 4 layers of silicon atoms, and the vacuum area between adjacent layers is 2 nm. To prevent the transfer of charge between surfaces, one hydrogen atom saturates each Si atom on the bottom surface. Sr atoms are placed on the top of Si(111) surface to simulate the adsorption systems. The Monkhorst-Pack scheme [22] was used for Brillouin zone integration, with a grid of 6 × 6 × 4 k points, and a plane wave energy cutoff set at 300 eV. Choosing a Fermi broadening of 0.05 eV to smear the occupation of the bands around EF by a finite-temperature Fermi function and extrapolating to T = 0 K. The adsorption energy barriers of Sr at different sites on Si(111) were calculated by the nudged elastic band method [23]. Basically, Sr atoms can be absorbed at the four most probable adsorption sites on the surface of Si(111), as shown in figure 3.

4. Results and discussion

4.1. The calculation of adsorption energy of Sr element on Si (111)

Figure 4 shows the model of Sr adsorption at different positions on the surface of Si (111). The coverage was calculated as 1 ML at the system energy and adsorption energy of the stable structure after the adsorption of Sr at the top (T), vacancies (V1 and V2) and bridge (B) on the surface of Si (111) are shown in table 2.

The adsorption energy of Sr on the surface of Si(111) can be defined as

\[ E_{ad} = [E_{Sr/Si(111)} - (E_{Si(111)} + nE_Sr)] \]  

(1)
Where $E_{ad}$ is the adsorption energy, $E_{Sr}/Si(111)$ is the system energy to stabilize the structure of Sr adsorbed on the Si (111) surface, $E_{Sr}$ is the energy of a single Sr atom, and $E_{Si(111)}$ is the total energy of the Si (111) surface. The calculated value of adsorption energy is negative, indicating that the adsorption process is exothermic reaction. The smaller the value, the easier the adsorption, and the stronger the interaction force between surfaces of Sr and Si. It can be seen from table 2 that the adsorption energy of the top site (T) is 4.0566 eV, the adsorption energy of the two bridge sites is equal and the value is $-3.8642$ eV, and the adsorption energy of the vacancy is $5.0888$ eV, indicating that vacancy has stronger adsorption ability. Due to the adsorption energy of bridge sites and top sites is greater than zero, Sr atoms cannot be adsorbed. The calculation results show that the adsorption of vacancy sites is stable, indicating that the vacancy sites are the stable adsorption sites on the surface of Si (111).

The stable structure and adsorption characteristics of Sr adsorption on the surface of Si (111) were calculated, which provided basic data for further study on the effect of Sr adsorption on the stacking and growth process of Si atoms on the surface of Si (111). In Al-Si alloy, the preferred growth direction of primary Si is $<111>$ crystal direction. The preferred growth direction of Si phase in eutectic Si is related to solidification rate. At lower solidification rate, the preferred growth direction of Si phase is $\langle 111 \rangle$ crystal direction. At higher solidification rate, the preferred growth direction of Si phase is $\langle 100 \rangle$ crystal direction. After Sr modification, the preferred growth direction of eutectic Si changed to $\langle 2\bar{1}1 \rangle$ crystal direction. Therefore, when Sr atoms are adsorbed on the surface of Si (111), Sr atoms are easily adsorbed on the vacancy sites, and the growth is inhibited, so that the growth direction of Si is changed.
4.2. The melting range of Al-Si-Zn-Sr filler metal

Figure 5 shows the melting point of Al-Si-Zn-Sr filler metals with different Sr contents. The results show that the melting point of Al-Si-Zn filler metal without Sr doping is 537.62 °C. In order to achieve the brazing conditions, the brazing temperature should be about 20 °C higher than the liquidus temperature. Therefore, considering the melting point of Al-Si-Zn filler metal, the brazing temperature of 6061 brazing should reach around 575 °C. After the addition of Sr, the melting point of the Al–Si–Zn-Sr system is hardly changed significantly. The liquidus temperatures of Al-Si-Zn-0.2Sr, Al-Si-Zn-0.4Sr, Al-Si-Zn-0.6Sr and Al-Si-Zn-0.8Sr are 536.6 °C, 537.3 °C, 536.1 °C and 537.6 °C, respectively. From the melting range of the filler metal, the small melting range is helpful to improve the spreading ability of the filler metal on the aluminum alloy plate. The difference of melting point is the slight difference of Al, Si and Zn contents in the casting filler metal.

4.3. The microstructure of Al-Si-Zn-Sr filler metals

Figure 6 shows the XRD pattern of Al-6Si-40Zn- (0.2, 0.4, 0.6, 0.8)Sr-filled metal. It can be seen that Al-6Si-Zn-Sr filler metal is mainly composed of α-Al phase, Si phase and η-Zn phase. Due to the little difference exits in the composition content of the five filler metals, the presence of Sr element cannot be observed in XRD pattern. Many researchers [18, 19] have shown that the amount of Sr in the filler metal was too small in volume as compared to the bulk phases to be detected by XRD.

Figure 7 shows the microstructure of Al-Si-Zn filler metals with different Sr contents. As shown in figure 7(a), the microstructure of the undoped Al-Si-Zn filler metal contains dispersed, well-developed dendrites, acicular and massive eutectic Si phases. As can be seen from figure 7(b), the fibrous tissue changed significantly when 0.2wt%Sr was added. The coarse acicular eutectic Si phase becomes fibrous, and aggregates around the massive Si particles, showing a chrysanthemum shape. With the further increase of Sr content, the block Si is divided into two parts, the slices become smaller, and some of them contain voids. The bulk Si crystals are considered as primary Si phase, and their formation is mainly due to the segregation of components in the microstructure during melting and the slow cooling rate during solidification. When the addition amount of Sr reaches 0.8wt%, the microstructure of the alloy becomes finer, the massive Si particles completely disappear, the boundary between the α-Al zone and the η-Zn becomes clearer, the distribution of Si phase becomes more dispersed, and the size of the dendrite and eutectic zone decreases.

In the unmodified alloy, Si is the dominant phase. After adding Sr, the coupling growth of α-Al solid solution and Si phase formed Al-Si eutectic. With the increase of Sr content, the primary Si particles gradually disappear, and the Al-Si eutectic phase changes from layered to fibrous structure [24]. According to the theory of “impurity-induced twins” [25], Sr can be used as an adsorption element to affect the Growth method of Si phase. When the amount of eutectic undercooling is quite small, the Si grows and branches along the plane morphology, but
Figure 8. EDS element mappings of microstructures of (a) Al-6Si-40Zn, (b) Al-6Si-40Zn-0.4Sr filler metals.

Figure 9. The spreading wettability of Al-Si-Zn-Sr filler metals on AA6061.
when the amount of undercooling is significant, the Si grows uniformly along the non-plane plane. Therefore, the modification to reduce the nucleation temperature of Si phase will lead to the change of morphology [17].

In order to further analyze the element distribution of filler metals and the influence of Sr on the microstructure of the Al-Si-Zn filler metal, an EDS element mapping was carried out on the microstructure of Al-6Si-40Zn and Al-6Si-40Zn-0.8Sr filler metal respectively. The mapping of EDS elements is shown in figure 8, and the results of EDS are shown in table 3. According to EDS results, B and E are mainly Si phases, while A and D are mainly α-Al phases, and eutectoid region appears. The elemental distribution shows that part of Si phase is embedded in α-Al phase, and the addition of Sr makes Si become fibrous. At the same time, the Si fiber makes the α-Al phase boundary clearer.

![Figure 10. Microstructure of (a) Al-6Si-40Zn and (b) Al-6Si-40Zn-0.8Sr filler metals spreading interface; (c) Line-EDS analysis of the spreading interface in (b).](image)

### Table 3. EDS results of points marked in figure 8 (a) and (b).

| Testing points | Al   | Si   | Zn   | Sr |
|----------------|------|------|------|----|
| A              | 75.90| 1.24 | 22.86| /  |
| B              | 1.11 | 96.19| 2.70 | /  |
| C              | 19.60| 1.98 | 78.43| /  |
| D              | 69.24| 0.96 | 29.27| 0.33|
| E              | 17.24| 76.06| 3.79 | 2.81|

4.4. Wettability of Al-Si-Zn-Sr filler metals on substrates

The fluidity of filler metal is vital to the quality of brazing joint. The spreading area of filler metal on 6061 aluminum alloy can reflect the wetting flow filling capacity of filler metal. Figure 9 shows the spreading area of Al-Si-Zn-Sr filler metal per unit mass. The spreading area of Al-Si-Zn filler metals without Sr element doping is only 187 mm². When Sr element is added, the wettability is significantly improved. The spreading area of Al-Si-Zn-0.2Sr filler metal is 246 mm². The wettability of the filler metals is much higher than that of filler metals without Sr element, and the spreadability of the filler metal increases with the addition of Sr element until 0.4% Sr element is added.

Figure 10 shows the microstructures of Al-6Si-40Zn matrix material and Al-6Si-40Zn-0.8Sr / matrix material, respectively. According to Fick’s law of diffusion, and the line scanning results in figure 10(c), there is a large concentration difference between the filler metal and the base metal. Therefore, in the process of diffusion, Zn will enter the base metal along the grain boundaries and react with Al in the base material to form α-Al solid solutions. The base materials interlaced with each other at the spreading interface, forming a diffusion layer between the filler metal and the base material. However, Mg in the base metal will move into the filler metal and form Mg2Si intermetallic compounds with Si in the diffusion layer. According to the results in figure 9 and figure 10, the addition of Sr not only promotes the interdiffusion of elements between the filler metal and the base metal, but also separates the solid solutions of α-Al and η-Zn on the coarse interface, resulting in finer microstructure.
4.5. Brazeability of 6061 Aluminum Alloy with Al-Si-Zn-Sr Filler Metal

Figure 11 shows the microstructure of aluminum alloy brazing joint after heat treatment. It can be seen that the joint microstructure is relatively dense, showing a good metallurgical bonding. Table 4 shows the results of energy spectrum analysis at three positions in figure 11(a), from which it can be concluded that the microstructure of the brazing joint of Al-6Si-40Zn filler metal is mainly composed of $\alpha$-Al phase, $\eta$-Zn phase and Si phase. There are a small amount of Mg$_2$Si intermetallic compounds at the weld interface, and part of Si particles are embedded in the matrix \[8\]. Figure 11(b–d) show the microstructure diagrams of Al-6Si-40Cu-Sr brazed joints. In the center of brazing joint, the degree of Si segregation is small due to the addition of Sr element, which is beneficial to reduce the influence of $\alpha$-Al and Si phase on the brittleness of joints. Figure 11(f) shows the element line scanning distribution in figure 11(a). Zn and Si are enriched at the interface between the weld and the base metal during the brazing process, and the post-weld heat treatment promotes the interdiffusion between atoms, so Al and Mg content at the interface of the weld is significantly reduced \[7, 8\]. Combined with the results of EDS analysis in table 4, it can be seen that the contents of Si and Zn at point A of the joint interface are higher than that of the matrix material, indicating that the elements in the brazing process between the liquid filler metal and the base metal are mutually diffused, forming a diffusion layer. During the post-weld heat process, Mg and Si diffuse to the interface, resulting in a reduction in the content of Al and Mg at the interface. This reduces the formation of brittle phase Mg$_2$Si, thereby improving the joint strength and reducing the weld brittleness.

Table 4. Chemical compositions (wt%) of the points in figure 11(a).

| Point | Al   | Si   | Zn   | Mg  |
|-------|------|------|------|-----|
| A     | 47.10| 4.36 | 48.02| 0.52|
| B     | 78.39| 1.59 | 19.39| 0.30|
| C     | 12.85| 83.16| 3.99 | /   |

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treatment of the joint, the element diffusion is enhanced. The metallurgical bonding reaction between filler metal and base metal is effectively promoted [6].

Figure 12(a) shows that the shear strength of the Al-6Si-40Zn brazed joint was approximately 96.32 MPa. With the increase of Sr content, the shear strength of Sr brazed joints gradually increased to 120.59 MPa, 123.22 MPa and 134.23 MPa. Compared with the the shear strength (72.9 MPa) of joints obtained by brazing 6061 aluminum alloy with Al-9.6Si-10Zn-10Cu-0.1Re solder studied by Chang et al [14], the shear strength (134.23 MPa) of the joint brazed with the Al-6Si-40Zn-0.8Sr filler metal developed in this experiment increased by 84.12%. Figure 12(b) shows the microhardness curve of the brazed joints. There were $\alpha$-Al solid solution, $\eta$-Zn solid solution and primary Si in the brazed seam center area of the weld, and the hardness of this zone was 155 ~ 246 HV. During the brazing process, the elements between the filler metal and the base metal diffused, forming a diffusion zone with hardness was slightly higher than that of the base metal. The hardness of the interface area of the brazed seam was 72 ~ 160 HV, and the hardness of the base metal was 58 ~ 67 HV. Therefore, the microhardness of the brazing seam appears to decrease from the central area of the brazed joint to the base material.

Figure 13 shows the shear fracture morphology of 6061 aluminum alloy joints brazed with Al-6Si-40Zn and Al-6Si-40Zn-(0.2, 0.4, 0.6, 0.8) Sr filler metals. A few dimples and brittle fractures appeared in the fracture surface of brazing joint before adding Sr element. The fracture morphology of the brazed joints indicates the characteristics of brittle fracture and ductile rupture. The $\alpha$-Al phase and $\eta$-Zn phase in the weld zone of brazing joint are the main causes of dimples. However, the existence of needle-like elemental Si phase in the weld zone deteriorates the microstructure of the brazing joint, resulting in the fracture of the brazing joint around the Si phase, and there are a few brittle fracture steps in the fracture of the brazing joint. With the addition of Sr element, the dimples and brittle fracture steps on the surface of brazing joint increased. The dimples became smaller and smaller with the increase of Sr element. The existence of tearing edges was also observed, indicating that the addition of Sr element promoted the diffusion of elements, improved the bonding between filler metal and brazing joint, as well as improved the mechanical properties.
By analyzing the microstructure, shear strength, and shear fracture morphology of the joint, it is found that when Al-6Si-40Zn brazed the 6061 aluminum alloy, there was a large Si brittle phase in the brazing seam, but the deformation compatibility between the large Si phase and the matrix was poor. The strain was concentrated at the phase interface, the crack initiation near the Si phase was accelerated. The fracture of the welded joint was accelerated, so the shear fracture of the brazed joint was a smooth and typical brittle fracture. When 6061 aluminum alloy was brazed with the Al-6Si-40Zn-Sr filler metal, the existence of Sr not only made the brittle phase of the Si finer more dispersed, but also improved the deformation coordination between Si phase and matrix and the shear strength of the brazed joint, reduced the strain concentration and presented a mixed fracture mode.

5. Conclusions

In summary, an Al-Si-Zn-Sr filler metal was designed and studied. From the above study, the conclusions can be drawn as follow:

(1) Sr was adsorbed on the Si (111) surface mainly at the vacancy points V₁ and V₂, which changed the growth mode of Si and provided a theoretical basis for the refinement of Si phase.

(2) Al-Si-Zn filler metal was mainly composed of α-Al solid solution, η-Zn solid solution and acicular primary Si. The melting range of Al-6Si-40Zn filler metal was 514.9 °C~537.4 °C, and the spreading area of 0.1g filler metal was 187 mm².

(3) The Al-Si-Zn-Sr filler metal was mainly composed of α-Al solid solution, η-Zn solid solution and fibrous Si. The addition of Sr can not only change the block or acicular Si into fibrous, but also make the melting temperature almost unchanged. With the increase of Sr content, the spreading ability of Al-Si-Zn-Sr filler metal decreases. The melting intervals of Al-Si-Zn-Sr filler metals are 20.8, 21.7, 21 and 21.4, respectively. The spreading areas of 0.1g solder are 246mm², 335mm², 332mm² and 349mm² respectively.

(4) When brazing 6061 aluminum alloy with Al-6Si-40Zn and Al-6Si-40Zn-(0, 0.2, 0.4, 0.6, 0.8) Sr brazing filler metals, the hardness in this area is 155 ~ 246 HV, and the shear strength is 96.32Mpa, 117.68 MPa and 120.50 respectively. Therefore, the addition of Sr can effectively increase the shear strength of the brazed joints.

Figure 13. Shear fracture morphology of the 6061 aluminum alloy joints brazed with (a) Al-6Si-40Zn, (b) Al-6Si-40Zn-0.2Sr, (c) Al-6Si-40Zn-0.4Sr, (d) Al-6Si-40Zn-0.6Sr, (e) Al-6Si-40Zn-0.8Sr, filler metals.
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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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