Effect of Er content on the interfacial microstructure, shear properties and creep properties of Sn58Bi joints

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Abstract. The influences of Er content on the interfacial microstructure shear properties and creep properties of Sn58Bi joints were investigated in this study. The intermetallic compound composition of Sn58Bi-xEr/Cu was Cu6Sn5 compound. The addition of Er suppressed the activity of Sn element, decreased the driving force for the growth of Cu6Sn5 intermetallic compound and decreased the thickness of Cu6Sn5 intermetallic compound layer. The shear properties and creep durability of Sn58Bi-xEr/Cu welded joints were improved to a certain extent. At the Er content of 0.1%, the shear strength and creep durability properties of the solder alloy are relatively optimal. When wt%Er was more than 0.1%, with the increasing Er content of rare earth elements, the internal organization of the joint interface is coarsened, and the flatness of the IMC layer at the interface is reduced, which leads to the decrease of the creep performance of the final joint.

Keywords: Sn58Bi; Interfacial microstructure; Shear properties; Creep properties.

1. Introduction
Sn-58Bi solder has attracted the attention of researchers in the field of low-temperature lead-free solder due to its low melting point, good wettability, low thermal expansion coefficient, good tensile strength and creep resistance[1,2]. But Bi is a brittle element and crystallizes into a coarse and irregular shape in the alloy, the Sn-Bi solder is brittle and has low ductility, which seriously affects the performance of the welded joint[3]. During the high-temperature aging process of Sn-58Bi/Cu solder joints, with the extension of aging time, the Bi-rich phase coarsens and the intermetallic compounds at the interface thicken[4]. Bi particles are precipitated at the interface junction, and voids are formed around the interface. Greatly reduces the reliability of solder joints.

In order to improve the overall performance of Sn-Bi solder, many scholars added alloy elements and nanoparticles to improve the structure of the solder, in order to obtain the ideal Sn-Bi solder with excellent comprehensive performance[5,6]. Mokhtari and Nishikawa[6] studied the effects of different contents of In and Ni in Sn-58Bi based solder. It was found that compared with Sn-58Bi solder, the In-containing solder exhibits a higher elongation because it promotes the formation of a large number of
primary Sn dendrites with good toughness in the solder matrix. Wu et al. [7] Studied the effect of the addition of rare earth element Er on the structure and properties of Sn-58Bi solder alloy, and found that the addition of Er element can improve the tensile strength of the solder alloy. The Er-containing solder exhibits a higher elongation. The rare earth Er can remarkably refine the eutectic structure of Sn-58Bi solder alloy.

Existing literature has not systematically reported the effect of Er content on the interface microstructure of Sn-58Bi solder and the mechanical properties of joints, so it has the value of further research. In this paper, Sn-58Bi series low-temperature solder is used as a matrix. By adding different amounts of rare earth element Er (0.05~1.0wt%), wetting of Sn-58Bi solder on Cu substrate by different mass fractions of Er is studied. Performance, and the mechanical properties (shear and creep endurance properties) of Sn58Bi-xEr/Cu welded joints were explored.

2. Experimental

2.1. Alloy design and preparation
Sn, Bi pure metal and Sn-10Er master alloy with a purity of 99.9wt% are used as raw materials. The composition of the solder alloy in this paper is shown in Table 1. According to the designed composition ratio, the experimental elements Sn, Bi and Sn-10Er are weighed and configured on the BSA224S-CW electronic balance, and then on the VIF-10 with argon protection Melting and casting in a vacuum melting furnace. The alloy was kept at 250℃~300℃ with argon protection and kept for 0.5 hour. The molten metal is cast in a stainless steel mold, cooled and solidified in the air, and cast into a cylindrical solder.

| SOLDER ALLOYS   | 1 | 2 | 3 | 4 | 5 | 6 |
|-----------------|---|---|---|---|---|---|
| Er(wt%)         |   | 0 | 0.05 | 0.1 | 0.25 | 0.5 | 1.0 |
| Base mental     | Sn-58Bi |

2.2. Wetting and spreading experiment
This paper adopts the JIS-Z-3198 standard for the wet spreading experiment. The Cu substrate used in the spreading rate experiment is a 99% oxygen-free copper sheet specified in JIS-H-3100[5]. Use BSA224S-CW electronic balance for accurate weighing, accurate to 0.001g. Record the data to calculate the density of the solder alloy, and take 0.35g of the solder alloy for wetting and spreading experiments. The flux (25Wt%Grade 2 Colophony +75wt% C12H26O3) is obtained as a soldering process.

![Figure 1. Schematic diagram of wetting performance during soldering](image-url)
As shown in Fig. 1, prepare 0.35 g solder alloy in the center of the treated Cu substrate. Use a syringe to drop 0.02 ml of flux onto the solder alloy to cover the solder alloy surface. Put it into ZB2015HL precision lead-free reflow soldering furnace for wet spreading. The size and morphology of the reflow soldering furnace are shown in Fig. 1. The heating temperature curve is shown in Fig. 2. The flux residue on the surface of the Cu substrate is cleaned with a circuit board cleaner. Use a vernier caliper to measure the height of the solder alloy spread, and use the spread rate $S_R$ (%) to characterize the wettability of the solder alloy[5]:

$$S_R = \frac{D-H}{D} \times 100\%$$

(1)

In the formula: $S_R$—spread rate, %; H—the height of solder after spreading, mm; D—Diameter when the solder is spread as a spherical shape, mm, $D=1.24 \times V^{1/3}$; V—The mass/density of the solder in the experiment. The thickness H measured after the experiment is substituted into the formula (1) to obtain the spread ratio.

2.3. Shear performance

This article uses JIS-Z-3198 standards to test the shear performance of welded joints. The Cu substrate used in the experiment is a 99% oxygen-free copper sheet specified in JIS-H-3100. The dimensions of the Cu substrate are shown in Fig. 3. Remove the oxide scale on the surface of the Cu substrate to be welded, polish it with a series of sandpaper to keep it smooth, and clean it with running water and alcohol in sequence. Use wire cutting to prepare a disc-shaped solder alloy with a size of $\Phi 5 \text{mm} \times 0.45 \text{mm}$, wash away the oil stain with acetone and polish the surface with 800 # and 1000 # sandpaper. At the soldering point, use a syringe to drop 0.02 ml of flux onto the solder alloy to cover the solder alloy surface with a layer of flux. Put the sample to be welded into ZB2015HL precision lead-free reflow furnace for welding. Use the heating temperature curve shown in Fig. 2.

![Figure 2. The reflow profile of solder](image)

![Figure 3. Schematic illusion of shear strength tests and dimension for solder joints](image)
A CMT 4204 electronic universal testing machine was used to perform a shear test on the welded joint sample. The fixture used in the experiment is shown in Fig.2. The shear strength of the welded joint was measured at room temperature at a shear rate of 1 mm / min. Five shear samples were prepared for each component of the solder alloy, and the arithmetic average of the 5 sets of data was taken as the test result.

2.4. Creep performance
This article uses the GB/T 2039-2012 standard to test the creep performance of welded joints. The Cu substrate used in the experiment is a 99% oxygen-free copper sheet specified in JIS-H-3100. The dimensions of the Cu substrate are shown in Fig.4. Remove the oxide scale on the surface of the Cu substrate to be welded, polish it with a series of sandpaper to keep it smooth, and clean it with running water and alcohol in sequence. Use wire cutting to prepare cuboid sheet solder alloy with dimensions of 5mm×4mm×0.35mm, wash away the oil stain with acetone and polish the surface with 800 # and 1000 # sandpaper.

![Figure 4. Dimensions of creep specimen](image)

3. Results and discussion

3.1. Wetting performance of Sn58Bi solder and joint interface morphology
An energy spectrum analyzer is used to scan the intermetallic compounds in the joint interface structure, and the composition is determined according to the ratio of various element compositions obtained. Fig.5 shows the EDS spectrum of Sn58Bi-1Er/Cu weld joint interface compound. Table 2 shows the results of EDS analysis of intermetallic compounds at the interface of Sn58Bi-1Er / Cu welded joints. As it can be seen from the table, the atomic mass ratio of Cu element and Sn element is 57.03: 42.41. Therefore, it can be judged that the intermetallic compound in the joint interface structure is $\text{Cu}_6\text{Sn}_5$.

![Figure 5. Sn58Bi-0.1Er/Cu solder joints and EDS spectrum](image)
Table 2. EDS analysis results of the intermetallic compounds in Sn58Bi-1Er/Cu solder joint

| Area | Elements | Atomic% | Weight% |
|------|----------|----------|---------|
| 1    | Cu       | 57.03    | 41.34   |
|      | Sn       | 42.41    | 57.41   |
|      | Er       | 0.18     | 0.34    |
|      | Bi       | 0.38     | 0.91    |

The interface structure of Sn58Bi-\(x\)Er/Cu solder joints after reflow soldering is shown in Fig. 6. It can be observed that Sn-58Bi reacts with the Cu matrix to form a fan-shaped \(\text{Cu}_6\text{Sn}_5\) intermetallic compound layer at the interface, and a large amount of Bi-rich phase is enriched here. After the addition of rare earth element Er, as the rare earth element Er increases, the thickness of the \(\text{Cu}_6\text{Sn}_5\) intermetallic compound layer tends to decrease first and then increase. It can be seen from Fig.6(b)~(c) that by adding rare earth element Er, the \(\text{Cu}_6\text{Sn}_5\) intermetallic compound layer formed at the interface of the Sn58Bi-xEr/Cu welded joint can be refined to promote the shape of the intermetallic compound layer flat. As can be seen from Fig. 6(d)~(f), when the addition of the rare earth element Er continues, a zigzag \(\text{Cu}_6\text{Sn}_5\) intermetallic compound layer will appear. This is because the \(\text{Cu}_6\text{Sn}_5\) intermetallic compound layer at the welded joint interface is mainly grown by a diffusion mechanism. Among them, the activity of Sn element affects the growth driving force of \(\text{Cu}_6\text{Sn}_5\) intermetallic compound. The lower the activity of Sn element, the smaller the driving force of \(\text{Cu}_6\text{Sn}_5\) intermetallic compound growth. The addition of the rare earth element Er suppresses the activity of the Sn element, reduces the driving force for the growth of the \(\text{Cu}_6\text{Sn}_5\) intermetallic compound, and reduces the thickness of the \(\text{Cu}_6\text{Sn}_5\) intermetallic compound layer. However, when the amount of Er exceeds the appropriate range, it will increase the driving force for growth, making the thickness of the \(\text{Cu}_6\text{Sn}_5\) intermetallic compound layer increase[8]. At the same time, the intermetallic compound layer has a part where rare earth Er and Bi elements will be dispersed and a part where no rare earth element Er adsorption effect occurs. Among them, the diffused distribution of the rare earth elements Er and Bi elements inhibited the reaction between the Sn element and the Cu element, resulting in a decrease in the growth rate of the intermetallic compound layer. In the part where the Er adsorption effect of the rare earth element does not appear, the growth speed of the intermetallic compound layer at the interface is relatively fast, which eventually causes the intermetallic compound layer to appear jagged.

Figure 6. Interfacial microstructures of Sn58Bi-xEr/Cu solder joints (a)\(x=0\); (b)\(x=0.05\); (c)\(x=0.1\); (d)\(x=0.25\); (e)\(x=0.5\); (f)\(x=1\)
3.2. Effect of Er content on shear properties of Sn-58Bi solder after welding

The experimental results of the shear force of Sn58Bi-xEr/Cu welded joints are shown in Fig. 7. It can be seen from the figure that after the addition of Er element, with the increase of the content of Er element, the shear force tends to increase first and then decrease. When 0.1wt% Er is added, the shear force of the welded joint reaches a maximum of 190.16N, which is an increase of 78.10N and an increase of 69.69% compared to the Sn-58Bi welded joint without Er element, which is 112.06N. When 0.25wt% Er is added, the shear force of the welded joint is 181.23N, which is still higher than that without the addition of Er element. However, when the content of Er is more than 0.25wt% Er, due to the presence of too much brittle and hard Cu6Sn5 intermetallic compound, the deformation process will affect the local stress concentration of the brazing filler metal and intermetallic compound layer, resulting in the welding joint shear force drops significantly. In summary, when the amount of Er added is within a certain range, the shear force of the welded joint can be effectively increased. The Er element can refine the Cu6Sn5 phase intermetallic compound formed at the interface of the Sn58Bi-xEr/Cu welded joint, reduce the thickness of the intermetallic compound layer, and make the intermetallic compound layer tend to be flat. Thereby improving the shear performance of Sn58Bi-xEr/Cu welded joints. From the perspective of inter-compounds, since the rare earth Er is almost insoluble in the Sn matrix, but is enriched at the Sn matrix phase interface or grain boundaries, during the solidification process, the proper amount of rare earth Er is beneficial to hinder the Cu6Sn5 brittle IMC (metal Intermetallic compounds), and refine these IMC particles, thereby improving the tensile strength and shear strength of the solder alloy. However, when the content of rare earth Er exceeds a certain critical value, it will cause the grain boundary of the solder alloy to be widened, the distribution of precipitates in the rare earth-rich Er-rich phase within the crystal is uneven, and the amount of rare earth Er-rich IMC increases, eventually makes the shear strength of solder alloy joints show a downward trend[9].

![Figure 7. Effect of Er contents on shear strength of Sn58Bi-xEr/Cu solder joints](image)

3.3. Effect of Er content on creep resistance of Sn-58Bi solder joint

The creep test results of Sn58Bi-xEr/Cu welded joints are shown in Fig. 8. It can be observed from Fig. 8 that as the content of Er in the solder increases, the creep fracture life of Sn58Bi-xEr/Cu welded joints tends to increase first and then decrease. The creep fracture life of Sn-58Bi/Cu welded joint is 6490.8s. When adding 0.05wt% Er, the joint creep rupture life began to be improved to 8064s. When 0.1wt% Er is added, the creep rupture life of the joint reaches a maximum of 10620s, which is an increase of 63.62% compared with no Er element. When the Er element continues to be added, the creep creep life of the joint gradually decreases. The creep fracture life of Sn58Bi-xEr/Cu(0.25/0.5/1.0%) welded joints was 9432s, 9018s and 7441.2s respectively, but still higher than that of Sn-58Bi/Cu welded joints. Therefore, in summary, the optimal addition amount of Er element is 0.1 wt%. Combined with the morphology and cross-sectional structure of Sn58Bi-0.1Er solder, it can be seen that an appropriate amount of Er can refine the structure in the solder, reduce the size of the IMC phase in the solder matrix, and then reduce
the strain and interface energy at the grain boundary. It hinders the grain slip and dislocation movement, reduces the loss of the welded joint during creep fatigue and prevents the spread of micro-cracks during creep[10]. Finally, the creep resistance of the welded joint is improved. However, when rare earth Er was more than 0.1%, with the increasing content of the rare earth element Er, the internal structure of the joint interface is roughened, and the IMC layer at the interface becomes uneven, resulting in the final reduce of joint creep performance.

![Figure 8. Effect of Er contents on creep rupture life of Sn58Bi-xEr/Cu solder joints](image)

4. Conclusions
The properties of Sn58Bi-xEr alloys were systematically investigated in this research. The conclusions can be drawn as follows:

1. With the addition of rare earth element Er, the thickness of the serrated Cu₆Sn₅ intermetallic compound layer Sn58Bi-xEr/Cu the interface of the welded joint decreases and becomes flatter. Er elements can reduce Sn activity and reduce the driving force for the growth of Cu₆Sn₅ intermetallic compounds. Meanwhile, the thickness of Cu₆Sn₅ intermetallic compound layer decreases, which makes the shear force of Sn58Bi-xEr/Cu welded joints increase. The maximum shear force is 190.16 N.

2. The creep fracture life of Sn58Bi-xEr/Cu welded joints increased by adding proper amount of rare earth elements Er. When 0.1 wt%Er is added, the creep fracture life time of the Sn58Bi-0.1Er/Cu welded joint is 10620 s, which improves 63.62% compared with the unadded Er elements, and the creep resistance is the best.

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