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Erosion Behavior and Interfacial Reaction of Stainless Steels in Molten Lead-free Solder[†]

NISHIKAWA Hiroshi*, KANG Songai** and TAKEMOTO Tadashi***

Abstract

The dissolution of iron and stainless steel during soldering presents a serious issue for manufacturing equipment such as wave soldering baths and soldering-iron tips. Severe erosion damage of stainless steel wave solder equipment has been encountered in operation. So, it is necessary to study the erosion behavior of stainless steel by molten lead-free solders. In this study, to investigate the erosion behavior of stainless steel in molten lead-free solder, an immersion test on 304 and 316 stainless steels was performed. It was found that the maximum erosion depth of the stainless steel was affected by the immersion conditions. A reaction phase was formed at the interface between the Sn-3.0Ag-0.5Cu solder and stainless steel during the immersion test.

KEY WORDS: (Lead-free solder), (Stainless steel), (Erosion), (Micro-focus X-ray system)

1. Introduction

The use of lead-free solder is expanding widely in the world. Among various lead-free solders, Sn-Ag(-Cu) system solders are currently considered the most promising for wave and reflow soldering processes. To develop lead-free soldering technology, considerable research into the characteristics of lead-free solder is in progress¹⁻⁷⁾. However, there are still some problems to be solved for the technology. In lead-free soldering, the operation temperature is higher than in conventional Sn-Pb soldering processes due to the high melting temperatures of Sn-Ag(-Cu) system solders. The high reaction rate of metals in molten lead-free solders, that is, the high dissolution rate of metals, has recently been pointed out as the basic characteristic of a lead-free solder. For example, Takemoto *et al.*⁸⁾ showed that iron does dissolve in a molten solder, and the dissolution rate of iron in the lead-free solder is greater than that in the conventional Sn-Pb eutectic solder.

The dissolution of iron and stainless steel during soldering presents a serious issue for manufacturing equipment such as wave soldering baths and soldering-iron tips. Concerning the soldering-iron tip, Nishikawa *et al.*⁹⁾ investigated the dissolution of plated iron in a molten solder to reveal the reaction between the lead-free solder and plated iron on soldering-iron tips. As a result, it has been made clear that the dissolution of plated iron into a molten lead-free solder is largely

attributable to the grain size of the plated surface, and the rate decreased with increasing grain size.

Meanwhile, severe erosion damage of stainless steel wave solder equipment has been encountered in operation. The resulting higher maintenance frequency and reduced life time of wave solder machine components are serious issues in a manufacturing processes. Therefore, it is necessary to study the erosion mechanism of stainless steel by molten lead-free solders.

In the previous work¹⁰⁾, a method for evaluating the erosion depth of stainless steel by molten lead-free solder was investigated using micro-focus X-ray systems for fluoroscopic and computed tomography (CT) to establish a method for measuring the maximum erosion depth. It was found that fluoroscopic images could truly reconstruct the cross-sectional shape of a stainless steel sample after an immersion test without destruction and the maximum erosion depth of the sample could be determined with high accuracy using this method.

In this study, the effect of immersion test parameters, which were the solder bath temperature, and the rotation rate, and the immersion time, on the erosion behavior of 304 and 316 stainless steels into a molten solder were investigated and the interface between the lead-free solder and stainless steel was studied.

2. Experimental procedure

To examine the erosion behavior of stainless steel

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into a molten lead-free solder, an immersion test on stainless steel was performed. **Figure 1** shows the experimental apparatus used for the immersion test. The test samples were vertically immersed and rotated at a fixed rate in a molten solder bath maintained at a fixed temperature. The solder bath temperature, the rotation rate of the test sample and the immersion time were the test parameters. For the experiments, the solder bath temperature was set to 673, 623, 573, and 523 K, the rotation rate was set to 150, 100, 50, 0 rpm, and the immersion time was set to 864, 600, 384, and 216 h, respectively. **Tables 1 and 2** show the chemical compositions of the lead-free solder and stainless steel specimens used for the immersion test.

After the immersion test, the erosion depth was measured by using a micro-focus X-ray system. **Figure 2** shows the shape and size of test samples fabricated in this study. The measurement position, location 1, was as noted in this figure. Test samples were made of 304 stainless steel and 316 stainless steel. **Figure 3** shows the fluoroscopic image of a test sample after the immersion test, which correspond to location 1 in Fig. 2. A horizontal part, curved part, and vertical part were defined, as shown in Fig. 3. The maximum erosion depth was respectively measured for each part. Using a micro-focus X-ray system for fluoroscopic and computed tomography (CT), it is possible to find the maximum erosion point and measure the maximum erosion depth without destructive cutting¹⁰⁾. The maximum erosion depth is expressed by the following equation:

$$\Delta d = h_0 - h_l \quad (1)$$

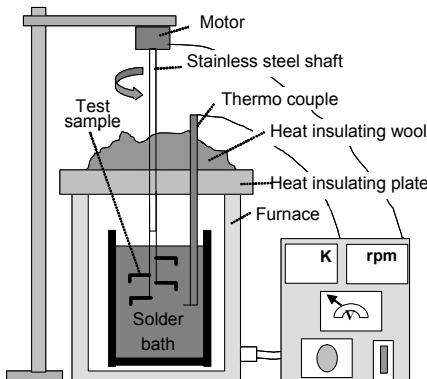


Fig. 1 Schematic diagram of experimental apparatus for immersion test

where Δd is the maximum erosion depth, h_0 the thickness of the test sample before the immersion test, and h_1 the minimum thickness of the test sample after the immersion test. The erosion depth refers to the loss of thickness of the test sample.

Scanning electron microscopy (SEM) was used to observe the interface between the solder and stainless steel, and electron probe microanalysis (EPMA) was used to determine the compositions of the intermetallic compound layer at the interface.

3. Results and Discussion

To clarify the effect of test parameters, which were the solder bath temperature, the rotation rate, and the immersion time, on the erosion behavior of 304 and 316 stainless steels into a molten solder, the immersion test was performed. **Figure 4** shows the effect of solder bath temperature on the maximum erosion depth of the 304 and 316 stainless steel samples immersed in a molten solder for 384 h. The rotation rate of the sample was fixed at 0 rpm. The maximum erosion depth tended to increase with increasing solder bath temperature regardless of the part of the sample and the stainless steel composition. The higher solder bath temperature accelerates the erosion rate of the stainless steel, and, in other words, the reaction between the molten lead-free solder and stainless steel is enhanced by the solder temperature. Then, the erosion depth of 304 stainless steel, shown in Fig. 4(a), was larger than that of 316

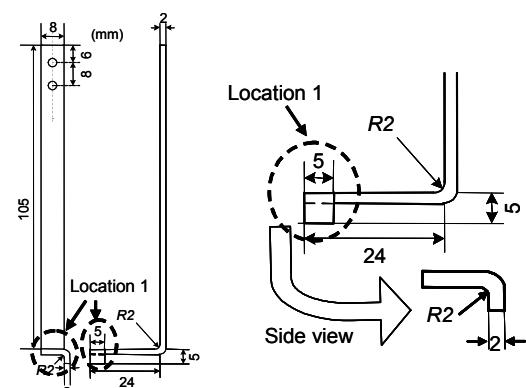


Fig. 2 Shape and size of test sample (a) and enlarged expression fro location 1 (b)

Table 1 Chemical compositions of lead-free solder used in this study

Sn- 3.0Ag- 0.5Cu	Elements (mass%)										
	Pb	Ag	Sb	Cu	Bi	Zn	Fe	Al	As	Cd	Sn
	0.027	3.000	0.008	0.500	0.003	0.000	0.002	0.000	0.000	0.000	Bal.

Table 2 Chemical compositions of stainless steel specimens used in this study.

Specimen	Elements (mass%)								
	C	Si	Mn	P	S	Ni	Cr	Mo	Fe
SUS 304	0.05	0.52	0.91	0.032	0.004	8.05	18.11	-	Bal.
SUS 316	0.03	0.62	0.91	0.034	0.004	10.16	16.78	2.06	Bal.

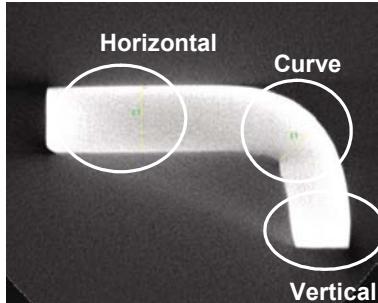


Fig. 3 Fluoroscopic images of the sample location 1, which correspond to the position in Fig. 1, after the immersion test.

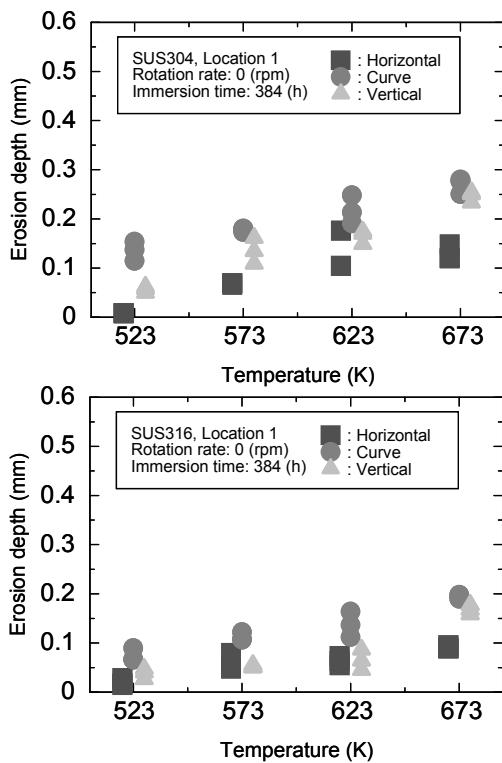


Fig. 4 Effect of solder bath temperature on maximum erosion depth, which corresponds to stainless steel: (a) SUS 304, (b) SUS 316. (0 rpm, 384 h)

stainless steel, shown in Fig. 4(b), regardless of solder bath temperature. The difference between 304 and 316 stainless steels is obvious at higher temperature range. 316 stainless steel has higher erosion resistance than 304 stainless steel.

Figure 5 shows the effect of rotation rate on the maximum erosion depth of the 304 and 316 stainless steel samples immersed in a molten solder for 384 h. The solder bath temperature was fixed at 623 K. The maximum erosion depth tended to increase with increasing rotation rate regardless of the part of the sample and the stainless steel composition. In this case, the erosion depth of 304 stainless steel, shown in Fig. 5(a), was also larger than that of 316 stainless steel, shown in Fig. 5(b), regardless of rotation rate of the sample. So, the flow rate of a molten solder in the solder bath

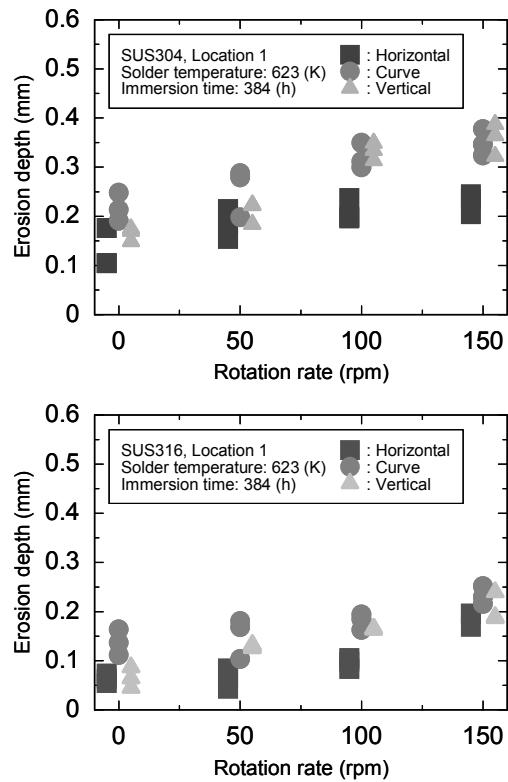


Fig. 5 Effect of rotation rate on maximum erosion depth, which corresponds to stainless steel: (a) SUS 304, (b) SUS 316. (623 K, 384 h)

strongly affect on the erosion behavior of the stainless steel.

Figure 6 shows the maximum erosion depth of the 304 and 316 stainless steel samples immersed in a molten solder at 623 K as a function of immersion time. The rotation rate was fixed at 0 rpm. For both cases, the maximum erosion depth increased with increasing immersion time and the erosion depth is proportional to the square root of the immersion time. Accordingly, the erosion behavior of the stainless steel into a molten solder can be considered as a diffusion control process.

The erosion behavior clearly showed that the maximum erosion depth of the stainless steel was affected by conditions such as the solder bath temperature, flow rate of the solder and immersion time regardless of the part and the stainless steel composition.

Figure 7 shows the SEM image and EPMA mapping analysis profile of the solder and stainless steel interface. The solder bath temperature was 623 K, the immersion time was 384 h, and the rotation rate was 100 rpm. In the SEM image, the upper, bottom and middle materials are respectively solder, 304 stainless steel, and reaction phase. Only one reaction phase can be confirmed at the interface. The EPMA mapping profiles clearly show that the reaction phase at the interface is composed of Sn, Cr and Fe. On the other hand, the element Ni doesn't exist in the IMC layer. **Table 3** shows the result of the quantitative EPMA analysis of point A, shown in Fig. 6. The reaction phase for Sn-3.0Ag-0.5Cu solder / 304 stainless steel was

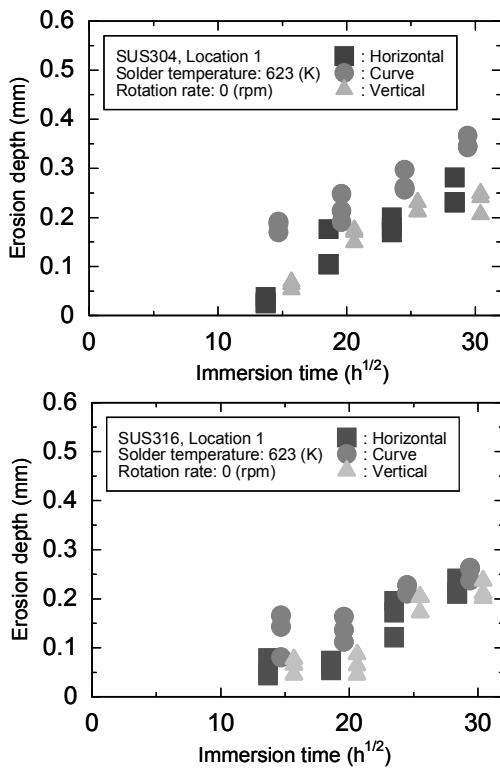


Fig. 6 Effect of immersion time on maximum erosion depth, which corresponds to stainless steel: (a) SUS 304, (b) SUS 316. (623 K, 0 rpm)

likely to be expressed as $(\text{Fe}, \text{Cr})\text{Sn}_2$, which was the intermetallic compound, because the compositional ratio between $\text{Fe} + \text{Cr}$ and Sn is so similar with 1:2 as FeSn_2 . In the case of 316 stainless steel, $(\text{Fe}, \text{Cr})\text{Sn}_2$ IMC layer was also formed at the interface.

4. Conclusions

The immersion test of 304 and 316 stainless steels into a molten solder was performed in order to clarify the effect of test parameters and the composition of stainless steel on the erosion behavior. The main results obtained in this study are summarized as follows.

- (1) The maximum erosion depth of stainless steels by a molten lead-free solder tended to increase with increasing solder bath temperature, sample rotation rate, and immersion time regardless of the part of the sample and the stainless steel composition.
- (2) The erosion depth of 304 stainless steel was larger than that of 316 stainless steel, regardless of test conditions. 316 stainless steel has higher erosion resistance than 304 stainless steel.

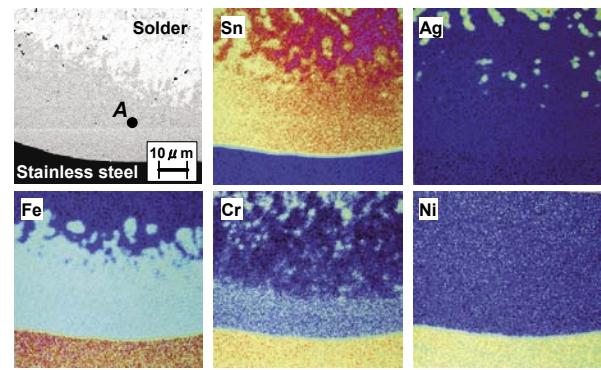


Fig. 7 SEM image and EPMA mapping analysis profile of the solder and 304 stainless steel interface. (Solder bath temperature: 623 K, immersion time: 384 h, rotation rate: 100 rpm)

Table 3 Composition of interfacial layer determined by EPMA.

Site	Composition (at. %)				Identified phase
	Fe	Cr	Ni	Sn	
Position A	26.58	7.76	0.47	65.19	$(\text{Fe}, \text{Cr})\text{Sn}_2$

- (3) The reaction phase for Sn-3.0Ag-0.5Cu solder / 304 stainless steel at the interface was likely to be expressed as $(\text{Fe}, \text{Cr})\text{Sn}_2$.

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