Ductile-to-brittle transition induced by increasing strain rate in Sn–3Cu/Cu joints

H.F. Zou and Z.F. Zhang[a)]
Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Science, Shenyang 110016, People's Republic of China

The current study revealed the effects of strain rate on tensile strength and ductile-to-brittle transition of Sn–3Cu/Cu joints in the strain rate range of 4.2×10^{-5} to 2.4×10^{-1} s^{-1}. Experimental results indicate that these joints broke in a ductile manner at low strain rates with a rapid increase in the tensile strength but displayed a brittle manner at higher strain rates with a slow increase in the tensile strength, indicating a typical ductile-to-brittle transition feature. A method was proposed to estimate the interfacial strength between the solder and the intermetallic compounds.

I. INTRODUCTION

Due to the toxicity of Pb or its alloys to the environment, more and more researchers have dedicated efforts to finding new lead-free alloys to replace Sn–Pb alloys in the electronic packaging field.[1,2] Currently, some lead-free solders, such as Sn–Ag and Sn–Ag–Cu solders and the eutectic or nearly eutectic Sn–Cu solders, have been developed. Compared with a series of Sn–Ag solders, Sn–Cu has many advantages, such as low cost, prohibiting dissolution of Cu substrate and good availability.[1] It is well known that soldering materials can not only provide the electronic connection, but also ensure the mechanical reliability of solder joints under service conditions.[3] For Sn–Pb solder joints and some lead-free solder joints, much mechanical property data have been obtained by most researchers. They found that some mechanical properties of lead-free solders are better than those of Sn–Pb alloys.[1,2] According to previous results, it is known that the strength of solders or solder joints has an approximately linear relationship with the effective logarithmic shear strain rate. Therefore, the effect of strain rate on the strength of solders and their joints must be considered because electronic products often undergo complicated loading conditions, leading to damage or failure of the solder joints at high strain rate. However, there are only a few data available about the mechanical properties of the solder joints at different strain rates, especially for the lead-free solder joints.[4–7]

Meanwhile, in a solder joint, there is a ductile solder alloy and at least one brittle intermetallic compound (IMC) formed at the interface.[8] As a result, the deformation and fracture behavior of the solder joint between the brittle IMC and the ductile solder is rather complicated under various strain rates. In particular, it is very difficult to estimate the true interfacial strength of the solder joint because fracture does not necessarily occur along the very interface between the solder and the IMC even at low strain rates.[8–12] So it is important to further investigate the fracture mode, interfacial strength, and ductile-to-brittle transition of the solder joints at different strain rates. In this letter, we use the Sn–3Cu/Cu joints as an example to reveal the effects of strain rate on the fracture behavior, interfacial strength, and ductile-to-brittle transition.

II. EXPERIMENTAL PROCEDURE

In this study, single-crystal Cu was used as a substrate and Sn–3wt%Cu alloy was used as a solder. First, a single-crystal Cu plate with the dimensions of $40 \times 150 \times 10$ mm^3 was grown from Cu with a purity of 99.999% by the Bridgman method in a horizontal furnace. Second, Sn–3Cu was prepared by melting high-purity (4N) Sn and Cu in vacuum ($<10^{-1}$ Pa) at 800 °C for 0.5 h. The single-crystal Cu and Sn–3Cu solder were ground with 800, 1000, 2000 grit SiC paper and then carefully polished with the 2.5, 1.5, and 0.5 μm polish pastes. These prepared samples were ultrasonically cleaned in ethanol for 10 min after polishing. Some rosin mildly activated (RMA) flux was used on the surface of Sn–3Cu and Cu substrates. Finally, the prepared samples (as illustrated in Fig. 1) were bonded in an oven with a constant temperature of 260 °C for 15 min. The as-reflowed samples were isothermally aged at 160 °C for 5 days to form an IMC layer with a thickness of 16 μm. Then, all the samples
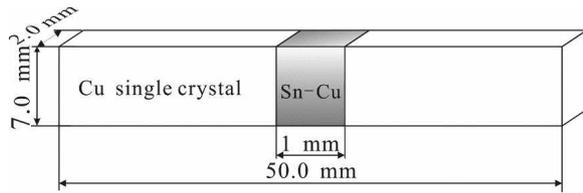
FIG. 1. Schematic diagram of as-prepared Sn–3Cu/Cu single-crystal couple.

were carefully polished with polishing pastes. To investigate the effects of strain rate on fracture behavior and tensile strength of the solder joints, tensile tests were carried out on the aged samples using an Instron (UK) 8871 testing machine at room temperature in air. In this study, seven tensile strain rates (in the range of 4.2×10^{-5} to 2.4×10^{-1} s^{-1}) were selected, and three samples were used at each strain rate. After fracture, all the fracture cross sections and fracture surfaces were observed with a LEO (Germany) Supra 35 scanning electron microscope (SEM).

III. EXPERIMENTAL RESULTS AND DISCUSSION

Figure 2 shows the tensile stress–strain curves of Sn–3Cu/Cu joint samples under various strain rates. It is apparent that the tensile strength has an increasing tendency with increasing strain rate. The experimental results show that the tensile fracture strength increases from about 38.0 MPa at the strain rate of 4.2×10^{-5} s^{-1} to 72.1 MPa at the strain rate of 2.4×10^{-1} s^{-1}. At the same time, it is also found that the samples can exhibit a higher tensile strain at higher strain rates. Such phenomenon was also observed in other metallic materials.[13,14] In our work, this phenomenon can be explained as follows. When the strain rate increases, work-hardening effect will make the solder have higher fracture strength. In turn, the higher stress applied on copper causes a higher
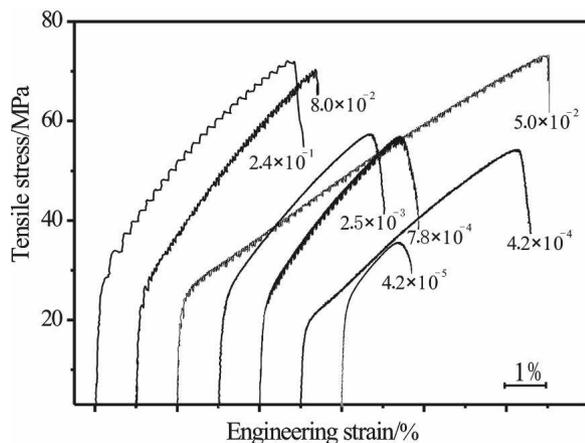
plastic deformation during tension process at higher strain rate.

Figure 3 shows the cross-sectional view and the top view of fracture for the joint samples deformed at low and high strain rates. It is clearly seen that there is much residual solder clinging to Cu substrate on the cross section of the joint sample deformed at low strain rate, as shown in Fig. 3(a). On the top view of fracture, bulk solder was detected on the interface, as shown in Fig. 3(b), which indicates a macroscopically ductile fracture behavior of the solder joint. Figure 3(c) shows the local fracture feature of fracture surface, and many dimples were detected. Within most dimples, IMC particles were clearly observed in the bottom, as indicated by the arrows. Occasionally, only a few IMCs can be detected to expose at the local smooth region. This provides clear evidence that those joint samples broke in a ductile manner under low strain rate condition, which is consistent with many observations on solder joints that failed under conventional shear or tensile conditions.[8,11]

Compared with the failure at low strain rate, there is little residual solder in the cross section of the joint samples deformed at high strain rate, as demonstrated in Fig. 3(d). The fracture surface is rather flat, as illustrated in Fig. 3(e). It is interesting to find that, in addition to a few solder residues, IMC layer is almost fully exposed, as shown in Figs. 3(e) and 3(f). Many broken IMC particles and many parallel cracks were detected on the fracture surface, as indicted by the circles in Fig. 3(f). It is indicated that the fracture should mainly occur within the IMC layer when the failure occurred at high strain rates. Sometimes, a little Cu$_3$Sn IMC can be detected near those parallel cracks, as indicated by the arrows in Fig. 3(f). The local parallel cracks should result from the impingement of the slip bands in copper single crystal to the IMC layer because the high tensile stress induced by high strain rate is easier to produce strong slip bands in Cu single-crystal substrate. In fact, fracture with a flat top surface has been observed previously in the joint samples aged at high temperature for long time.[15–17] Therefore, based on the observations in Figs. 3(d)–3(f), it can be concluded that those joint samples actually broke in a brittle manner mainly in the IMC layer under high strain rate condition, which is quite different from the failure mode under low strain rate condition. In other words, a ductile-to-brittle transition does occur in the joint samples due to the obvious increase in the strain rate.

In our study, seven strain rates were performed on the joint samples. It was found that the joint samples broke in a ductile manner when the strain rates are equal to 4.2×10^{-5} s^{-1}, 7.8×10^{-4} s^{-1}, 4.2×10^{-4} s^{-1}, and 2.5×10^{-3} s^{-1}, as demonstrated in Figs. 3(a)–3(c). In contrast, the samples failed in a brittle manner when the strain rates are equal to 5.0×10^{-2} s^{-1}, 8.0×10^{-2} s^{-1}, and

FIG. 2. Tensile stress–strain curves of Sn–3Cu/Cu solder joints in the strain rate range of 4.2×10^{-5} to 2.4×10^{-1} s^{-1}.
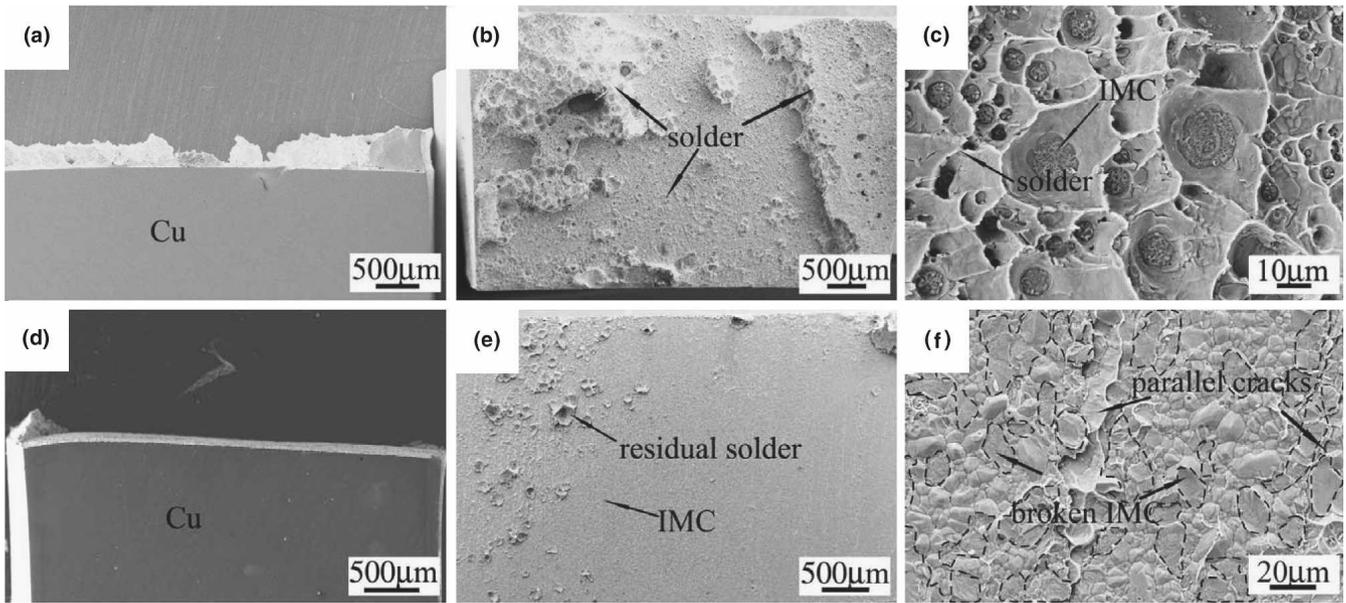
FIG. 3. Fracture morphologies of the solder joints subjected to tensile loading at low and high strain rates. (a) Macroscale fracture morphology with a ductile fracture feature. (b) Top view of the fracture surfaces. (c) Top view of local dimples, at a low strain rate of 4.2×10^{-4} s^{-1}. (d) Macroscale fracture morphology with a brittle fracture feature. (e) Top view of fracture surface. (f) Top view of local brittle IMC fracture, at a high strain rate of 5.0×10^{-2} s^{-1}.

2.4×10^{-1} s^{-1}, as indicated in Figs. 3(d)–3(f). As mentioned previously, the brittle failure manner mainly occurred in the IMC to form the flat fracture surface, while the ductile failure manner normally took place in the solder near the interface, characterized by much residual solder on the fracture surface, as shown in Figs. 3(a)–3(c). The ductile-to-brittle transition of the solder joints can be further explained as below.

Some researchers found that the relationship between ultimate tensile strength, σ_s, of solder and strain rate, $\dot{\epsilon}$, can be expressed by the following equation[5,6]:

$$\sigma_s = C_1 \dot{\epsilon}^{m1} \quad , \qquad (1)$$

where C_1 is a constant and m_1 is the strain rate sensitivity index of the solder. Figure 4 shows the plot of the $\dot{\epsilon}$ for the Sn–3Cu/Cu joint samples aged at 160 °C for 5 days based on Fig. 2. It can be obviously seen that those data dots do not form a straight line in the total strain rate range. According to the observations in Figs. 3(a)–3(f), it is known that the tensile fracture manner has experienced a great change from ductile fracture of the solder in low strain rate range to brittle fracture of the IMC layer in high strain rate range. Therefore, it is reasonable to consider that the $\dot{\epsilon}$ curve in Fig. 4 can be separated into two parts in the total strain rate range due to the change in the failure manner. In the low strain rate range, the $\dot{\epsilon}$ curve should represent the effect of strain rate on tensile strength of the solder itself, which follows the Eq. (1). However, since the failure of the joint samples mainly occurred in the IMC layer in the high strain rate range, it is natural to assume that the tensile strength, σ_i, of the
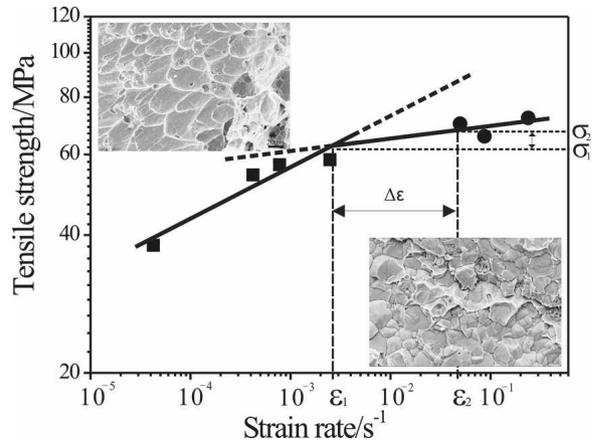
FIG. 4. Plot of the lg σ_s – lg $\dot{\epsilon}$ curve of Sn–3Cu/Cu joint samples aging at 160 °C for 5 days.

IMC layer obeys a similar equation with different constants C_2 and m_2 as below:

$$\sigma_i = C_2 \dot{\epsilon}^{m2} \quad , \qquad (2)$$

where, C_2 is a constant and m_2 is the strain rate sensitivity index of the IMC layer. According to Eqs. (1) and (2), it is easy to understand why the lg σ_s–lg $\dot{\epsilon}$ curve in Fig. 4 has two different slopes in the total strain rate range because there are two different failure mechanisms, i.e., ductile fracture of solder and brittle fracture of IMC layer. Furthermore, the two values of m_1 and m_2 were calculated from the two slopes of the lg σ_s–lg $\dot{\epsilon}$ curves. It is apparent that m_1 (0.112) of the Sn–3Cu solder is approximately equal to that (0.080) of Sn–3.5Ag

solder,[5,6] but is obviously higher than m_2 (0.026) of the IMC layer. This indicates that the tensile strength of the solder is more sensitive to the strain rate than that of the IMC layer.

On the other hand, it is well known that the solder has a lower tensile strength than the IMC layer at low strain rate range.[18] Therefore, the lg σ_s–lg $\dot{\epsilon}$ curve of the solder must intersect with that of IMC layer at certain strain rate, as demonstrated in Fig. 4. The region near the intersection point $\dot{\epsilon}_1 \rightarrow \dot{\epsilon}_2$ was defined as ductile-to-brittle transition region. When the strain rate is smaller than $\dot{\epsilon}_1$, the joint samples would break in a ductile manner within the solder because the tensile strength of the solder is smaller than that of the IMC layer. The picture inserted in the upper-left corner in Fig. 4 illustrates a clear ductile fracture feature (elongated dimples) for the joint samples deformed in the low strain rate range. When the strain rate increases to $\dot{\epsilon}_2$, the joint samples would break in a brittle manner mainly within the IMC layer because the strength of the IMC layer is lower than that of the solder, as illustrated in Fig. 4. The picture inserted at the lower-right corner in Fig. 4 displays a typical brittle fracture feature for the joint samples subjected to tension in the high strain rate range. When the strain rate range is between $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$, the samples would break approximately along the interface rather than within the IMC layer. Therefore, the interfacial strength of the Cu/Sn–3Cu joint samples can be approximately estimated in the range of σ_1 (60 MPa) $\leqslant \sigma \leqslant \sigma_2$ (68 MPa) for the current joint sample subjected to tension in the strain rate range of $\dot{\epsilon}_1$ to $\dot{\epsilon}_2$ (see Fig. 4). Furthermore, to evaluate the interfacial properties of a solder joint, it is necessary to analyze its lg σ–lg $\dot{\epsilon}$ curve, via which the interfacial strength can be deduced from its ductile-to-brittle transition region.

IV. CONCLUSIONS

In summary, strain rate plays an obvious role in the tensile strength and fracture mode of the solder joints. The current experimental results reveal that tensile strength of Sn–3Cu/Cu joints would increase with increasing strain rate. However, the ductile-to-brittle transition will occur with gradually increasing the strain rate. Based on the lg σ–lg $\dot{\epsilon}$ curve, it is possible to estimate the interfacial strength of the Cu/Sn–3Cu joint samples to be about 60 to 68 MPa.

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