Local Creep in SnAg3.8Cu0.7 Lead-Free Solder

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In this study, local creep of lead-free solder SnAg3.8Cu0.7 has been investigated for the first time by using surface markers prepared by focused ion beam (FIB). The test setup was optimized with respect to the applied shear stresses. Two different microstructures have been investigated at 30°C, 50°C, and 80°C, respectively. A high sensitivity of the steady-state creep rate to the microstructure has been demonstrated. The shear deformation was observed to be inhomogeneous and concentrated in specific areas. Local dislocation bands dominate the solder deformation.

Key words: Lead-free solder, creep, shear test, microstructure, focused ion beam (FIB)

INTRODUCTION

With the European directive on the restriction of the use of certain hazardous substances in electrical and electronic equipment (RoHS)¹ being effective, lead is banned from electronic products which are put on the European market after July 1, 2006. This requires the replacement of classical SnPb solder by another suitable material, of which several are under investigation. From the many, proposed lead-free solders, SnAgCu alloys are preferred.²

A common failure mechanism of electronic packaging and assemblies is the cracking of solder joints. To estimate reliability and lifetime based on accelerated testing, degradation and deformation mechanisms of solder material have to be understood. A large difference of these parameters for SnPb and SnAgCu solder is expected due to their different microstructure. In SnPb, the tin- and lead-rich phases have similar dimensions with the Pb-phases being somewhat smaller. The SnAgCu solder has an extended β -Sn phase and finely dispersed Ag₃Sn and Cu₆Sn₅ particles. The intermetallics have dimensions that are usually smaller than 400 nm. In addition, the creep rate is influenced by the different melting points of 183°C and 217°C for SnPb and SnAgCu, respectively. Therefore, these alloys differ in homologous temperature at the same absolute

testing temperature. The microstructural and physical differences of these two materials lead to significant deviations in their creep rate and therefore to different acceleration factors in thermal cycling tests. The standard test procedures for accelerated aging established for PbSn solder have to be adapted to the lead-free solder.

The existing data of mechanical and fatigue behavior of lead-free solders are limited and hardly can be analyzed consistently due to different testing procedures.^{3,4} They range from ball shear and large bulk material tensile tests to highly sophisticated four-point bending tests; the size of specimen varies from centimeters to the dimension of a solder joint of a single ball grid array (BGA) and the testing frequency ranges from several Hertz to 0.001 Hertz or lower for low cycle analysis.^{5–10} The samples used in this study exhibit a well-defined geometry with the thickness being adapted to industrial produced solder joints.

EXPERIMENTAL PROCEDURE

A sample holder was designed to allow the use of replaceable inserts. The inserts with a front area of 10 × 10 mm² consisted of two cylindrical bolts for a precise and reproducible fit in the holder. The setup was optimized for shear testing without any tensile and compression forces (Fig. 1). The solder gap between the two bolts was set to 100 μ m to cover a broad range of solder applications in electronic

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Fig. 1. Test setup for shear creep experiments with replaceable cylindrical sample inserts.

production. In this way, a homogeneous and well-defined amount of solder could be shaped. The large area and the simple geometry of the solder joint reduce the influence of boundary effects on the measurement results.

The bolts were made of bronze (92wt.%Cu and 8wt.%Sn; UNS C52100). The contact area was ground with a 1200 SiC grindpaper, cleaned with ethanol in an ultrasonic bath, and finally dried with nitrogen gas. Areas which should not be wetted were covered with a solder stop finish. For the solder process the gap was adjusted to 100 μm and the bronze parts were fixed on a gauge made of bronze, too. The gauge and the fixed bolts were preheated to 100°C. Then, the solder paste was added manually to the specimen and the sample was soldered with a peak temperature of 250°C in a conventional oven. The cooling of the samples was done stepwise, first in a second oven in air of 100°C and then on aluminum block at room temperature.

The soldering temperature profile was adapted to a standard industrial reflow profile (Fig. 2). A commercial SnAg3.8Cu0.7 solder paste was used. Following the soldering procedure, the specimens were ground with a 1200 SiC abrasive paper by applying ethanol as a lubricant. Then, an optical inspection was carried out to ensure that no visible defects were present and to determine the exact sample width.

The metallographic microstructure was investigated in microsections. The preparation routine was as follows: grinding with SiC papers 320 and 500 with water as a lubricant, followed by 1200 SiC paper grinding with ethanol and 5 vol.% isopropanol as lubricant to reduce corrosion. The following polishing with diamond paste of grain sizes 6 μm , 3 μm , and finally 1 μm was carried out on a soft tissue using ethanol with 5% isopropanol as a lubricant. The load on the samples was <5 N to prevent wash-out effects. After each grinding/polishing step the samples were cleaned in an ultrasonic bath of ethanol containing 5% isopropanol for 2 minutes and then rinsed with ethanol and dried with nitrogen gas.

The creep experiments were carried out on a universal testing machine (Zwick UPM Z020), which

Fig. 2. Temperature profiles of sample cooling for shear sample preparation. The cooling rate of the slowly solidified samples is close to the common reflow production process cooling rate.

was equipped with a high-resolution extensometer directly fixed to the specimen holder. The tests were done in dead load weight mode, where the force is controlled and kept constant during the entire test, and inside a climatic chamber to keep the sample and the strain sensor at a constant temperature. Test series were carried out at 30°C, 50°C, and 80°C in air. For small loads and slow creep rates, the samples were tested to a predefined deformation, and not to rupture, in order to limit the duration of the experiment, which otherwise would have taken several weeks.

After sample failure, both fracture surfaces were investigated optically. Microsections of one sample half were taken vertical to the shear plane. These were investigated by optical and electron microscopy as well as by energy-dispersive x-ray (EDX). Some samples were inspected with a focused ion beam (FIB) microscope (FEI Strata 235 dual beam). With the FIB, micro cross sections were prepared and surface etching was applied to reveal more details of the microstructure.

To track local deformation in the sample, some specimens were mechanically polished and markers were applied to the solder at several locations by milling lines or depositing platinum patterns on the surface with FIB. Deposited platinum lines with a width of 1 μm have been shown not to be suitable as deformation markers due to their insufficient deformation capability and surface connection. Deposited pearl-necklace-like platinum dots and milled holes with diameters of 0.5 μm to 1 μm could be used, but are not suitable to uncover sharp steps in the deformed sample. The different patterns milled or deposited with FIB were investigated to exclude that they induced failures themselves. There was no evidence that the markers caused any damage to the sample, which can be explained by the large dimensions of the sample compared with the marker size. The best results were obtained by the milled lines (Fig. 3). It was found that a line width of 500 nm with a depth of around 300 nm to 500 nm is the best indicator for local deformation.

Fig. 3. (a) Top view of ion-beam secondary electron detection (SED) image of 500-nm wide and 300-nm deep lines milled by FIB. (b) Tilted view (52°) of E-beam SED image of FIB etched hole with a radius of 250 nm and a depth of 800 nm.

The depth of the lines depends strongly on the local material composition and crystal orientation. The etching rate of the ion beam is highly dependant on the composition and the orientation of the etched material. For this reason, the depth of the lines is not well defined and cannot be adjusted to a specific value.

RESULTS

Microstructure of Samples Solidified According to Standard Temperature Profile

The slowly solidified samples showed a relatively homogenous microstructure with a uniform intermetallic interface between the bronze and the

solder. The average thickness of this interface was $2.4 \mu\text{m} \pm 0.25 \mu\text{m}$, with a typical scalloplike shape. This alloying zone was representative of an industrial process solder joint. The β -Sn dendrites seemed to be oriented slightly in the interface direction. Intermetallic particles such as Cu_6Sn_5 and Ag_3Sn were small and evenly dispersed in the eutectic. Several Ag_3Sn plates were uniformly distributed in the sample, but were always connected to the interface between the solder and the substrate. These plates are shown to be not crack actuating and thus did not pose reliability problems. Sometimes they were observed to stop cracks.¹¹

Measurements with an energy-dispersive x-ray/scanning electron microscope (EDX/SEM) indicated that the microstructure and the elemental distribution of the solder were identical to those of an industrially-produced solder joint. The EDX mapping shown in Fig. 4 was taken from an undeformed, slowly solidified sample.

Microstructure of Water-Quenched Samples

The water-quenched samples solidify in less than 5 sec. Therefore, their microstructure was much finer and rather globular than dendritic (Fig. 5). The mean diameter of the β -Sn dendrites was about $2 \mu\text{m}$. The β -Sn phases were evenly surrounded with the ternary eutectic. There was no discernable global orientation. The intermetallic interface was less homogeneous and less dense than in the slowly solidified samples. The average thickness of this interface was $1.8 \mu\text{m} \pm 0.35 \mu\text{m}$. In contrast to the slowly solidified specimens, no intermetallic platelets were found.

Global Creep

The creep experiments were carried out at three different temperatures and exhibited a strong temperature dependence of the creep rate. The time dependence of the creep rate had a normal shape with a short period of primary creep followed by a long constant secondary creep, and finally a ternary period ending with the rupture of the sample (Fig. 6). These observations proved that the external conditions during the test did not change and that there was no effect that changed the properties of the solder drastically during secondary creep. In the limited tested temperature range during each experiment, no change in the creep mechanism was observed.

Creep can be described by the Norton law:¹²

$$\dot{\gamma} \propto \tau^n \quad (1)$$

where $\dot{\gamma}$ is the steady-state creep rate, n a material constant, and τ the applied stress.

This phenomenological model describes quite accurately to global creep deformation of any metal alloy. The temperature dependence is accounted for by an Arrhenius term and the stress is normalized with the temperature-dependant shear modulus G . This leads to the equation of Bird et al.¹³

Fig. 4. (a) SE image of a slowly solidified sample. The EDX mappings of (b) Sn, (c) Ag, and (d) Cu show the extended β -Sn dendrites and the large Ag_3Sn plates.

Fig. 5. Top view of ion-beam SED images of water-quenched solder sample showing the fine microstructure caused by the rapid solidification. The Sn dendrites are evenly surrounded by the eutectic and no large intermetallic particles are shown.

Fig. 6. Normalized creep strain rate curves of slowly solidified samples tested at 80°C. The curves show a long and quite stable steady-state creep after the primary creep.

$$\dot{\gamma} = A \cdot \left(\frac{\tau}{G} \right)^n \cdot \exp \left[-\frac{Q}{RT} \right] \quad (2)$$

by adding a proportionality parameter A .

The mechanical behavior of the SnAg3.8Cu0.7 lead-free solder cannot be described by a simple power law. Since two deformation mechanisms are expected, an extended Norton law expressed by the sum of two different power laws was required to parameterize the data of the tested temperature and stress range.

$$\dot{\gamma} = A \cdot \left(\frac{\tau}{G} \right)^n \cdot \exp \left[-\frac{Q_{\text{gbs}}}{RT} \right] + B \cdot \left(\frac{\tau}{G} \right)^m \cdot \exp \left[-\frac{Q_{\text{dc}}}{RT} \right] \quad (3)$$

A constitutive equation of the form of Eq. 3, where Q_{gbs} and Q_{dc} are the activation energies of grain boundary sliding (GBS) and dislocation climbing (DC), respectively, has been used before.¹⁴ The steady-state creep rates are plotted as a function of the reciprocal temperature in natural logarithmic versus linear coordinates to receive the activation energies. The value of Q_{gbs} was found to be 48 kJ/mol and Q_{dc} 105 kJ/mol with stress exponents n equal to 2 and m equal to 9.

The water-quenched samples exhibited much higher creep rate and temperature sensitivity in the steady-state creep period than the slowly cooled samples (Fig. 7b). This can be explained by the finer microstructure with an average diameter of the β -Sn dendrites of about 2 μm instead of about 8 μm of the slowly cooled specimen.

The creep data had a relatively broad distribution. Large deviations in the stationary creep rates are attributed to local phenomena.

Local Creep

Samples with and without marker lines to observe local deformations were tested under the same conditions. After a predefined deformation step, the specimens were dismantled and investigated. It was observed that deformation was concentrated in regions of variable size, with deformation zones being mostly parallel to the shearing direction. The deformation zones exhibited a width of

Fig. 7. (a) Shear creep data of slowly solidified SnAg3.8Cu0.7, compared with extended Norton law (solid lines). The two different mechanisms GBS and DC can be seen. (b) Shear creep data of water-quenched SnAg3.8Cu0.7, compared with extended Norton law (solid lines). The high-temperature sensitivity of the water-quenched samples in the high-stress area is shown.

about 5 μm to 20 μm and a length of about 20 μm to several 100 μm . The images in Fig. 8 show a sample with a total shear deformation of 25% at 80°C and 10 MPa, but on different locations of the solder slit. The deformation is inhomogeneous with respect to the sample length and the width of the solder slit. The observed intermetallic Ag_3Sn plates did not break. Solder glides along these plates and deformation zones are mostly stopped by this hard phase (Fig. 8b).

Higher global deformations lead to increasing local strain concentrations. As revealed by FIB micro cross sections, the cracklike lines indicated by arrows (Fig. 8a–d) were not open defects, but grain boundaries. Micro cross sections from heavily deformed zones showed no visible evidence of voids or cracks (Fig. 9). Recrystallization occurred in the highly deformed areas of the sample, and was accelerated at elevated temperature as reported before.¹⁵ The grain size in the strongly deformed areas was reduced to a few microns due to recrystallization.

The localized fine grains allowed high deformations without generating voids and cracks in the material. The shear deformation in the center of

Fig. 8. Top view of SEM SED images of a shear sample with 25% shear deformation at 80°C and 10 MPa. (a) Deformation concentration in the center. (b) Homogeneous deformation with solder gliding along large intermetallic particles. (c) Steps and kinks in the solder due to deformation. (d) Inhomogeneous deformation without steps.

these zones was often up to 10 gammas. Figure 10 shows an image of such a local zone, marked with lines etched by FIB. A few sporadic open cracks could be observed at the surface. They did not extend deeply into the solder, but can be explained by different rotation of grains at the surface. The deformation concentration was also observed in the water-quenched samples. Ternary creep to rupture occurred only in these strongly deformed zones.

Fracture Morphology

The fracture surface showed elongated dimples from high deformation prior to sample rupture. Analysis of the fracture surface demonstrated that failure begun at microcracks (Fig. 11). These local defects form cavities which propagate with increas-

ing deformation reducing the sample cross section. This increases the stress in the remaining solder cross section, leading finally to sample rupture. The fracture is mostly intergranular and only partially transgranular.

The fracture of the samples was observed neither at the interface with the bronze nor at the interface between alloying zone and bulk solder, but was located in the solder material itself. No cracks starting at a large intermetallic particle were observed.

DISCUSSION

Microstructure

Investigations showed the large influence of the cooling rate on the microstructure and the mechani-

Fig. 9. Tilted view (52°) of SEM SED images. (a) Section of a highly deformed area; the line marks the location of the micro cross section. (b) Micro cross section of a highly deformed local zone with no cracks.

cal properties of lead-free solder. The thinner and less homogeneous intermetallic interface had no major influence on the creep behavior, because the fracture was not observed at the interface between the solder and the intermetallics, neither in the slowly solidified nor in the water-quenched samples.

As known from literature,¹⁶ samples with smaller phases exhibit a higher creep rate. The water-quenched samples were investigated to prove that a finer microstructure exhibits a faster steady-state creep rate. With the results from the quenched samples, it could be verified that the recrystallization to small grains in highly deformed zones leads to a locally faster creep. The fact that two different microstructures exhibited strongly different creep

Fig. 10. SEM SED image of a highly deformed area where grain boundary sliding and grain plasticity are observable; the lines indicate the FIB-etched markers.

properties is important for specimen preparation and test design. Therefore, special attention should be given to sample preparation for reliability investigations of solder joints.

Global Creep

The global creep properties strongly depend on temperature and on microstructure. Temperature dependence is based on accelerated mechanisms such as GBS and DC, as well as on the recrystallization of the solder alloy. The data can well be described with a dual mode parametric Norton power law representing two different deformation mechanisms. For GBS, the stress exponent n was found to be 2, which is in agreement with literature.^{17,18} The activation energy was found to be 48 kJ/mol, which is higher than expected from self-diffusion in pure Sn of 40 kJ/mol.⁷ For DC, the derived stress exponent was found to be 9 and the apparent activation energy was 105 kJ/mol, which is consistent with published data of 92.7 kJ/mol to 106 kJ/mol.^{19,20}

Microstructural differences caused by different temperature profiles during solidification lead to large deviations of creep behavior. This could explain the inconsistencies of data found in the literature. The relatively broad distribution of data from this study can be attributed to local deformation observed in the marked samples. The fact that different sample processing caused different material behavior has important consequences for the design of reliability tests.

Local Creep

The well-defined and very fine surface patterning made by FIB revealed the local phenomena precisely.

Fig. 11. SEM SED images of fracture surface with vertical shear direction. (a) Typical morphology of fracture with high deformation showing dimples. (b) Microcracks in the ternary creep; the vertical lines are scratches caused by sample rupture.

The details of the deformation could be investigated much better than in previous work.^{21,22} No influence of the method developed in this study on the creep and fracturing behavior could be observed.

Deformation was found to be localized. The deformation mechanisms in these highly deformed zones are dominated by GBS and DC. The β -Sn crystals have only six primary gliding planes.²³ This could be one reason for their inhomogeneous and local creep behavior in the Sn-rich phases. At present, it is not possible to predict zones where concentrated deformation will take place. Boundaries between two dendritic trees with different orientation and the accumulation of ternary eutectic phases could be indicators. In Reference 21, inhomogeneity of creep in the center of the sample was explained by the solidification history. If both sides of the specimen cool at the same rate, then the solidification ends in the middle of the sample, which could lead to voids and dross at that location. However, the samples investigated in this study showed no solidification porosity and the zones of higher deformation were spread over the entire sample and not just in the middle.

A major limitation for predicting local deformation zones is the restriction of initial inspection to the surface. Three-dimensional microstructural data would provide more data for credible prediction of local deformation.

Fracture Morphology

The morphology of the fracture surfaces is rich in deformation, but lacks any evidence of brittle fracture. Failure is generated by merging microcracks during ternary creep and, therefore, by an increase in the shear stress induced by constant load. The lack of evidence of failure caused by large inter-

metallic plates indicates that these plates are not a severe reliability problem of the solder joint.

CONCLUSIONS

The influence of microstructure is one of the main factors in creep behavior of lead-free solder. Different sample preparation procedures generate different microstructures. Surface patterning by FIB is a useful method for investigating the local deformation of a shear sample with small dimensions. The aggravating factors are the sample dimensions and the aspect ratio of 1:100 of width to length. At the testing temperatures, a slowly solidified sample has a significantly lower steady-state creep rate than a water-quenched sample. This is caused by the very different microstructure of the water-quenched samples. Local creep is observed to dominate over global creep deformation. Areas with a high γ of up to 10 without crack formation were observed. The surfaces of fractured samples show no evidence of brittle fracture and do not indicate failure induced by large intermetallic particles.

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