

# Reliability of Pb-free Soldered Joints for Automotive Applications

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## ABSTRACT

Tests were conducted to evaluate the grain growth rate of the structure and the intermetallic compound layer fracture rate. These tests included the physical properties, as well as peeling, which is a mechanical property of Pb free soldered joints. In addition, thermal shock tests (acceleration tests) were performed to evaluate the life span as related to thermal fatigue.

First, the grain growth rate and intermetallic compound layer fracture rate of the Pb-free soldered structure were confirmed to follow the rule of parabolic curves. Next, the life span of Pb-free soldered joints under market conditions was predicted using Miner's rule of reliability engineering based on the effects of heat stress obtained through acceleration tests and the heat stress frequency in an automotive application.

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## 1. Introduction

There are environmental concerns regarding the use of Sn-Pb solder. This has spurred much research toward a Pb-free solder. Although these global efforts have been led by America and Europe, such research has gained momentum in Japan, as well, with the recent passage of the Electrical Appliance Recycling Law (due to take effect in 2001). For instance, the Industrial Structure Council, of the Ministry of International Trade and Industry has also designated Pb reduction as a goal for automotive electronics and electrical equipment.

A Pb-free solder selected for automotive electronics and electrical equipment must maintain component functions in its respective utilization environment and for the duration of use. This, in turn, makes it necessary to fully understand the environment of the solder joints of automotive electronics and electrical equipment. The grain growth and intermetallic compound layer fractures that have been detected in the solder joints of automotive electronics and electrical equipment recovered after market use are said to have a significant impact on reliability, and so determination of mechanical strength alone (e.g., bulk material strength and joint tensile strength) is not sufficient for the reliability assessment of solder joints. There are few reports on reliability assurance against thermal fatigue for different Pb-free solder compositions from both the "micro" perspective of grain growth and intermetallic compound layer fracture as well as the "macro" perspective of mechanical properties.

The authors therefore studied the grain growth, intermetallic compound layer fractures, and mechanical strength of Pb-free solder, establishing testing conditions

that simulate the actual environments in which automotive electronic components are used, and using a failure model to calculate joint life.

## 2. Approach to the Reliability Assessment of Solder Joints

Automotive electronics and electrical equipment are used in extremely severe environments. The use of increasingly compact components has translated into narrower joint width. Automotive interior components, in particular, are simultaneously subjected to heat stress, vibration, humidity and other conditions. The most important aspect of solder joint reliability is thermal fatigue countermeasures. Solder has a low melting point, and it has a difference in coefficient of linear expansion between a substrate and a component that leads to considerable thermal stress, which, after repeated occurrence, can cause thermal fatigue failure. Pb-free solder is no exception even though it differs in composition from Sn-Pb solder and therefore also differs in melting point, grain growth rate, intermetallic compound layer fracture rate, and mechanical properties and similar, all of which have a large effect on solder joint life. The methods and standards for assessing components made with Pb-free solder are therefore particularly important. Assessment standards must focus on wear failure and degradation failure by providing testing conditions and testing times that simulate actual market environments, while the assessment method that must be constructed is one for indoor acceleration testing. As an example, the authors assess the reliability of bare chip die bonding.

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### 3. EXPERIMENT SAMPLES AND METHOD

#### 3.1. Experiment Samples

Elements having low toxicity that are already used in actual solder applications were selected for the solder used in this experimentation. With a Sn base, eutectic Pb, Bi, Ag, Sb, and Zn, as well as individual elements in combination, were used to create the following solders: Sn-37Pb, Sn-3.5Ag, Sn-3.5Ag-0.75Cu, Sn-58Bi, Sn-2Ag-0.5Cu-7.5Bi, Sn-5Sb, and Sn-9Zn. These are the solders from which the samples for each test were selected. Sn-9Zn was not subjected to intermetallic compound layer fracture rate testing or thermal shock testing because its poor wettability with Ni makes it difficult to establish sample conditions. Furthermore, Sn-5Sb was not subjected to grain growth testing because the similar atomic numbers of Sn and Sb make it difficult to distinguish their phases by microscope.

#### 3.2. Conditions for Intermetallic Compound Layer Fracture Rate Testing

Experimentation was carried out to determine how the fracture rate of the intermetallic compound layer fractures that form during solder melting and joining differs according to solder composition. Solder was exposed to a high temperature (120°C) to accelerate fracture growth rates. To identify the growth process, the solder was measured at the times shown in Table 1. Test pieces were made by soldering solder paste to Ni plates, after which the thickness of the intermetallic compound layer fracture formed on the joint interface was measured. As for the method of measurement, after high temperature exposure was applied for the predetermined period of time, a test piece was cut so that the cross-section of the solder would be perpendicular. The cross-section was then polished and thickness of the intermetallic compound layer fractures was measured by observing composition with element mapping and Backscattered Electron Image (BEI) made with an Energy Dispersive Spectrometer (EDS).

Table 1 Measurement time

| Time (hr) | 0 | 250 | 500 | 1000 |
|-----------|---|-----|-----|------|
|-----------|---|-----|-----|------|

#### 3.3. Conditions for Grain Growth Testing

Solder with JISA-grade impurity levels was used in all experimentation, the goal of which was to observe aging of the structure by accelerating the rate of aging by increasing temperature. Test pieces were made by dripping approximately 0.12 cc of flux (activated rosin) onto Ni-plated Cu sheets (17.5 × 17.5 × 1.75 t), then depositing 0.2 g of solder. The test piece was covered with paraffin paper and placed in a solder bath that was set to a temperature of 50°C over the solder's melting point and heated for approximately 30 seconds after the solder melted, thus completing the soldering process. After being air-cooled, the test pieces were washed with isopropyl alcohol to remove the remaining flux, followed by drying.

For testing, samples were placed in a thermostatic chamber maintained at 120°C and removed at different times

for observation. As Sn-58Bi has a low melting point (138°C) and its phase growth is faster than that of the other solders, samples were removed for microscopic observation (with a JEOL model JSM-5 310LV microscope) at ×1000 and ×3500 resolution at 0, 10, 20, 60, 120, 240, 1200, 3000, 12000, 24000, and 30000 minutes. Photographs taken during this observation were processed by linear analysis. An example is shown in Fig.1.

For instance, in the case of Sn-58Bi, a straight line of length L and parallel to one side of the observation results was drawn, and the size of the Bi phase was determined by dividing the ratio of Bi phases on that line by the number of Bi phases intersected.

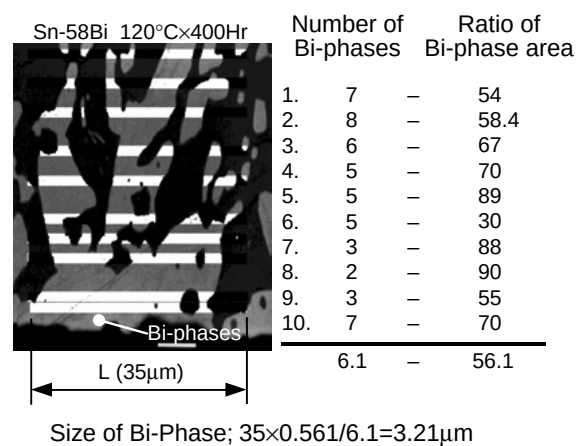


Fig.1 Linear analysis data

#### 3.4. Conditions for Peeling Testing

Experimentation was also carried out to determine how the heating of solder joints would have different effects on joint strength according to differences in solder composition. Test pieces were made by placing solder cut from solder tape into 5 × 15 × 0.1 mm strips between sheet copper (Fig.2) and placing it into a solder bath for reflow. Reflow time was set at 20 seconds after solder melting, and solder bath temperature was set according to each solder's melting point: Sn-58Bi = 200°C, Sn-5Sb = 270°C, other solders = 250°C. The heating temperature was 120°C, and six heat exposure times were used to simulate aging (Table 2).

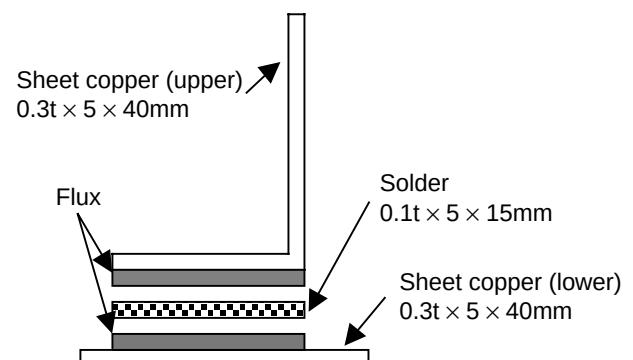


Fig.2 Test piece

Table 2 Heat exposure time

| Time (min) | 0 | 10 | 20 | 60 | 6000 | 12000 | 60000 |
|------------|---|----|----|----|------|-------|-------|
|------------|---|----|----|----|------|-------|-------|

For testing, a tensile tester (Shimadzu AG-2000B) was used to perform peeling testing by placing the bent portion of each test piece into the tester's gripping device to subject the test piece to a tensile load. The pull rate was 10 mm/min<sup>(1)</sup>. A constant load was used in this peeling testing (Fig.3). The point at which cracking occurred was defined as the end of life of the solder joint. Although peak load may seem more similar in actual conditions, a relatively stable constant load was used because of the effect on output of fillet shapes and other conditions.

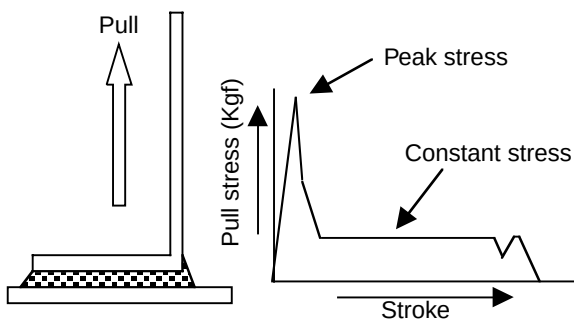


Fig.3 Pull test method

### 3.5. Conditions of Thermal Shock Testing

Experimentation was carried out to determine how the effect of thermal shock on solder joints differs according to solder composition. Bare chip die bonded test pieces were selected and an ultrasonic survey instrument was used to confirm sample quality (Fig.4). The three testing conditions applied simulated the conditions under which various automotive electronics and electrical equipment are actually used (Fig.5). In thermal shock testing, a thermal shock tester was used to determine and compare life of the joint. The ultrasonic survey instrument was used to observe the solder joints and determine the state of peeling at different times (Table 3).

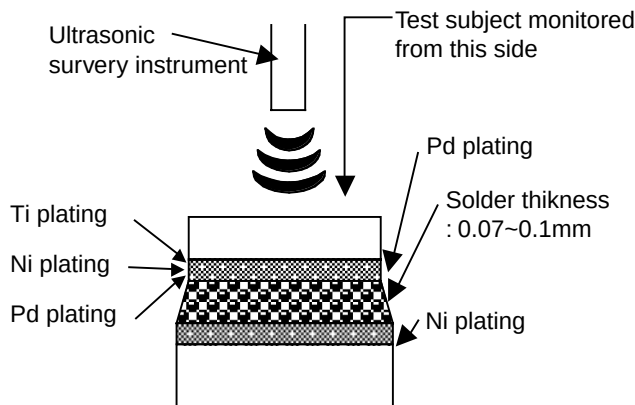


Fig.4 Specification of test subject

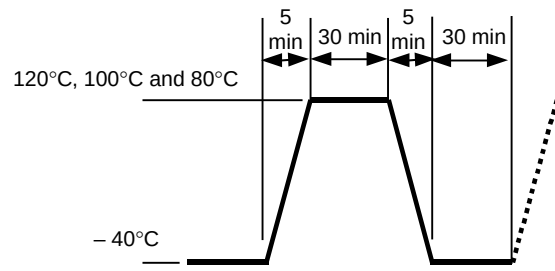


Fig.5 Conditions of thermal shock test

Table 3 Measurement time

| Temperature                    | Time (hour)                              |
|--------------------------------|------------------------------------------|
| $\Delta T 120^{\circ}\text{C}$ | 0, 505, 1000, 1500, 2000, 3770, 5000     |
| $\Delta T 140^{\circ}\text{C}$ | 0, 406, 1000, 1732, 3000, 4000, 5000     |
| $\Delta T 160^{\circ}\text{C}$ | 0, 100, 240, 537, 1060, 1533, 2000, 3000 |

## 4. Results of Intermetallic Compound Layer Fracture Rate Testing

Intermetallic compound layer fracturing occurs because of the formation of intermetallic compounds by Sn and other solder components due to mutual diffusion between these metals. This intermetallic compound layer fracturing occurs both during solder melting and joining and during post-joining high temperature exposure, making the joint brittle and creating Kirkendall void and cracks. Automotive electronics and electrical equipment, in particular, are subjected to repeated heating/cooling cycles, and require adequate consideration for the lowered reliability that can result when the thermal stress resulting from the materials' different coefficients of expansion causes joint cracks, thus having a major negative impact on mechanical strength. Fig.6 is an example of intermetallic compound layer fracturing.

All of the solder used in this testing underwent Sn-Ni intermetallic compound layer fracturing, the thickness of which varied depending on the type of solder. Fig.7 also suggests the occurrence of Sn-Cu intermetallic compound layer fracturing in Sn-3.5Ag-0.75Cu and Sn-2Ag-0.5Cu-7.5Bi. The rate of Sn-Ni intermetallic compound layer fracture is shown in Fig.8. This shows that in Pb-free solders, as in Sn-Pb solders, the intermetallic compound layer fracture rate is proportional to the square root of time, thus obeying the parabolic law<sup>(2)</sup>.

$$X = k\sqrt{D \cdot t}$$

where layer thickness = X, holding time = t, constant = k, diffusion constant = D.

The compositions exhibiting the slowest intermetallic compound layer fracture rate were Sn-3.5Ag-0.75Cu and Sn-2Ag-0.5Cu-7.5Bi, in both of which, as Fig.7 also shows, Cu is concentrated at the interface, suggesting that the addition of Cu tends to retard the growth of Sn-Ni intermetallic compounds<sup>(3)</sup>.

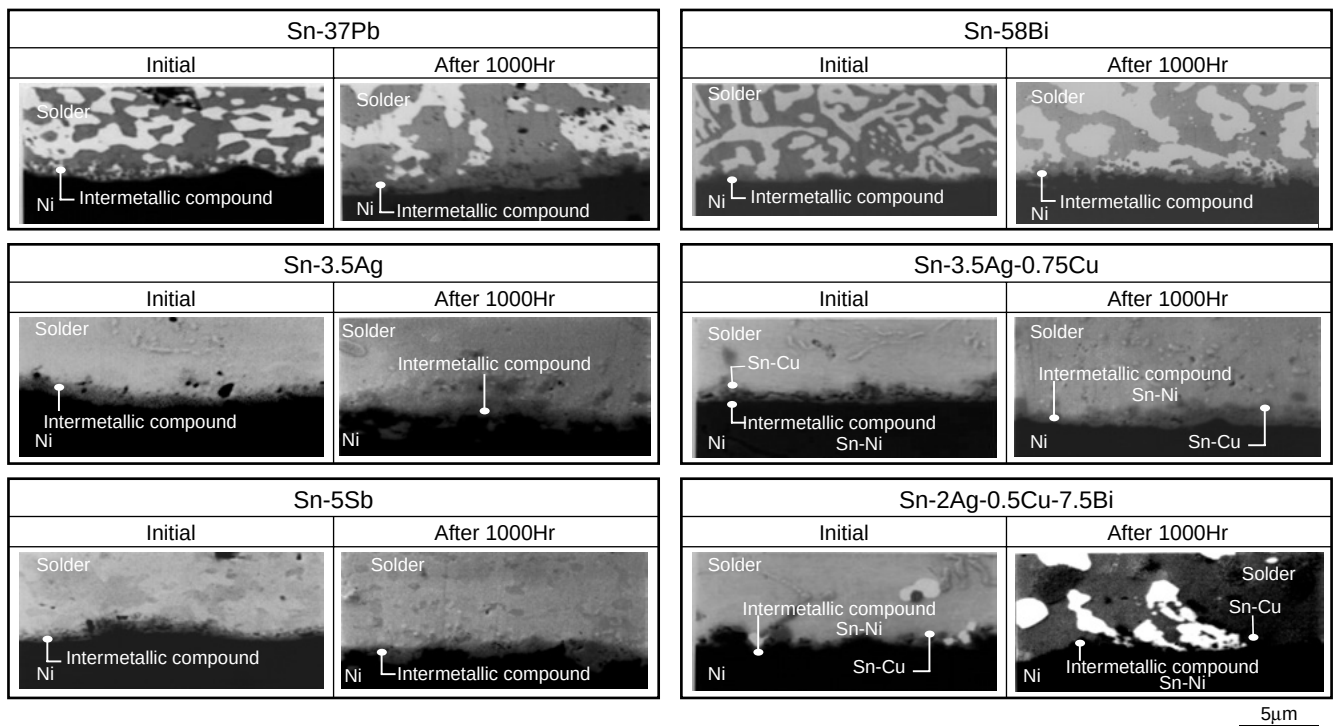


Fig.6 SEM image of intermetallic compound layer

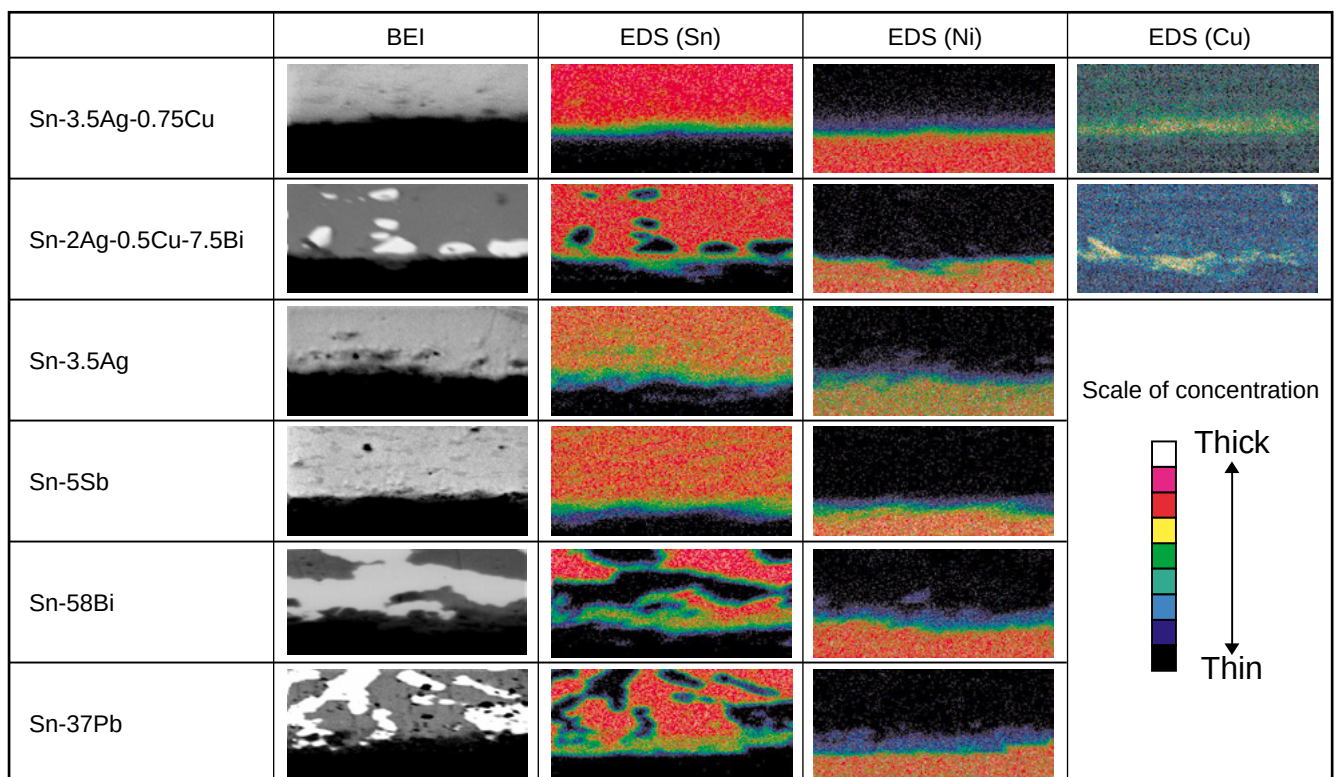


Fig.7 Distribution maps of the intermetallic compound layer of each element

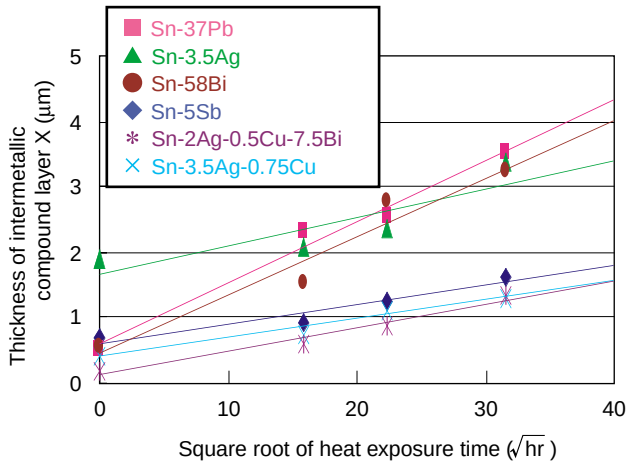


Fig.8 Growth pattern of the intermetallic compound layer

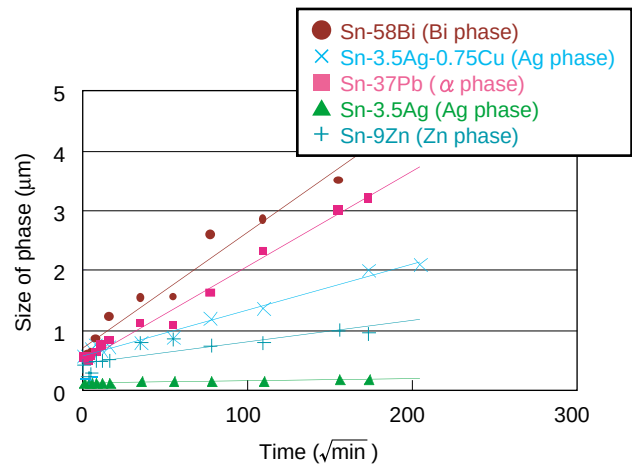


Fig.9 Change in phase size due to aging of each solder

## 5. Results of Grain Growth Testing

As in Sn-Pb solder, grain growth in Pb-free solder is due to a change in each phase's solid solubility limit density and surface energy, and has been shown to progress so as to decrease the free energy of phase equilibrium<sup>(4)</sup> at a rate that obeys the parabolic law (Fig.9). The fastest grain growth rate was exhibited by a Sn-Bi solder, Sn-58Bi; the slowest, by a Sn-Ag solder, Sn-3.5Ag. As in the phase diagrams for these Pb-free solders, a greater change in solid solubility

limit density at a given temperature tends to be accompanied by a faster rate of grain growth. In addition, Sn-2Ag-0.5Cu-7.5Bi exhibited peculiar structural change (Fig.10). After 10 minutes of high temperature exposure at 120°C, the Bi diffused throughout the Sn matrix, resulting in voids where the Bi had been. Further high temperature exposure was shown to enlarge or eliminate existing voids and create new voids. Sn-2Ag-0.5Cu-0.5Bi was omitted from the plot in Fig.9 as its Bi phase has been eliminated.

As for the effects of grain growth on solder joints, because

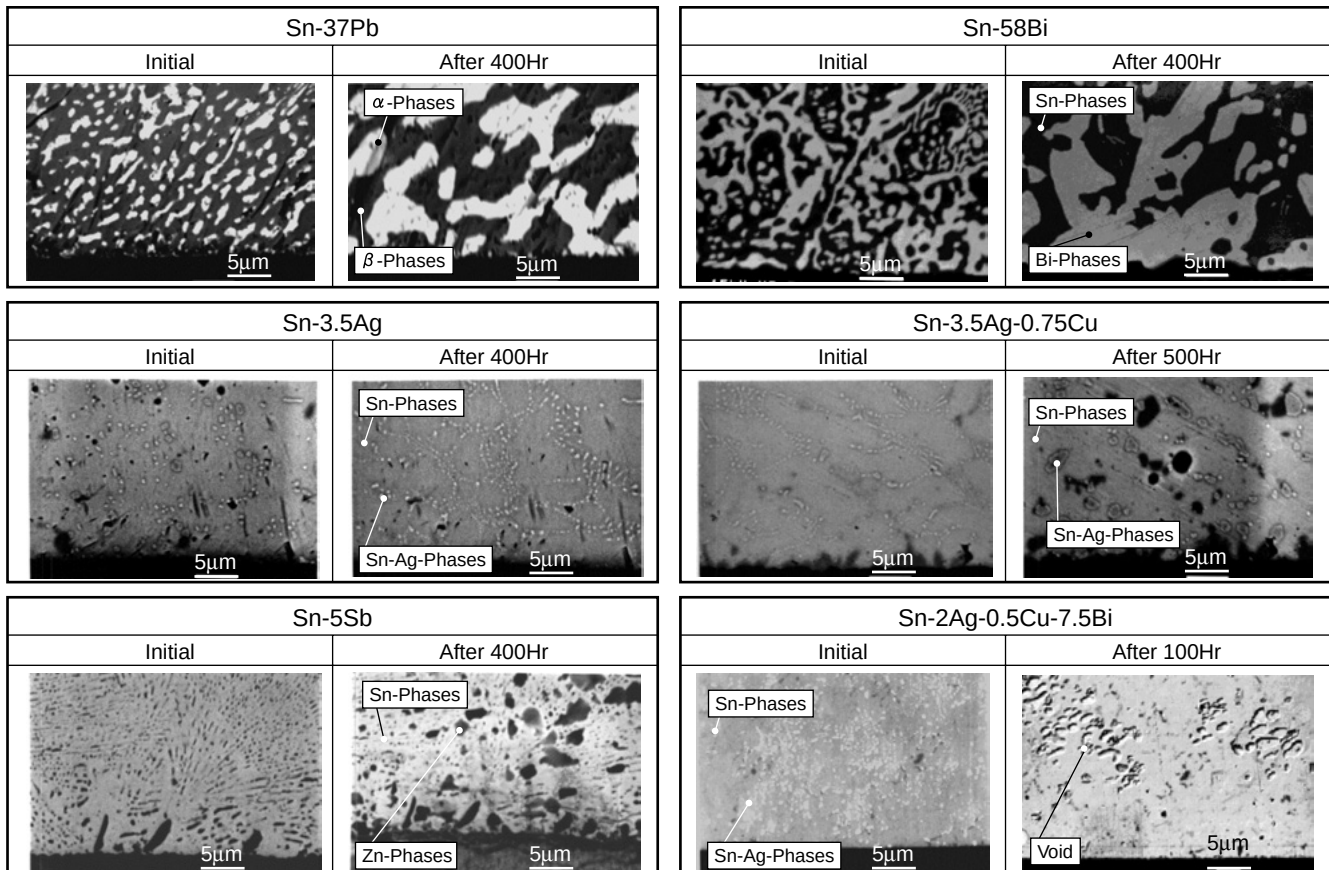


Fig.10 Aging of Pb-free soldered area relative to temperature

solder joint peeling due to thermal shock is the result of the formation and progression of microcracks caused by local plastic deformation (i.e., sliding deformation of dislocation), which in turn is caused by repeated stress at the site where maximum stress is concentrated, peeling in the solder interior is also most likely due to grain growth. Degradation of the solder joint strength due to grain growth is generally thought to obey the law of Hall-Petch<sup>(5)</sup>.

The relationship between phase (i.e., crystal particle) size and strength is expressed in terms of strength  $\rho$  thus, where  $D$  is average phase diameter:

$$\rho = k \sqrt{D}$$

This shows how greater phase growth through high temperature exposure decreases strength and makes peeling more likely.

## 6. Results of Peeling Testing

Fig.11 shows that high temperature exposure of 500 hours at 120°C caused Sn-9Zn, a Sn-Bi solder, to undergo a marked drop in solder load as determined by measurement of peeling load. As for the drop in load of Sn-2Ag-0.5Cu-7.5Bi, the high temperature exposure caused the Sn-Cu intermetallic compound layer to increase in thickness, causing Bi in the solder to remain on the surface between the intermetallic compound and the solder. Eventually, Bi density on the surface increases to the point where brittle peeling occurs. One factor in the decreased load of Sn-58Bi is believed to be the marked effect of Bi cleavage, a result of grain growth. The sharp decline in the peeling load of Sn-9Zn is believed to be due to the formation of Kirkendall void because of the high diffusion rates of Cu and Zn<sup>(3)</sup>. In contrast, Sn-Ag solder remained stable, showing little change in load. This is believed to be because of the slow growth of the Sn-Cu intermetallic compound, and the resultant few factors degrading the intermetallic compound/solder interface.

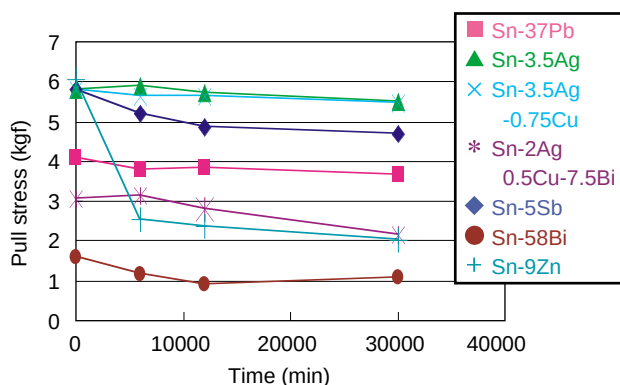


Fig.11 Pull strength of connection point after high temperature exposure

## 7. Results of Peeling Testing

The results of thermal shock testing (Fig.12) and peeling testing (Fig.13) verified that peeling starts with intermetallic compounds in the case of Sn-58Bi and Sn-2Ag-0.5Cu-7.5Bi, which are Bi solders, but in the solder interior in the case of all other solders. This is believed to be because of the extreme hardness and brittleness of Sn-58Bi and Sn-2Ag-0.5Cu-7.5Bi, which does not elongate and which, consequently, is

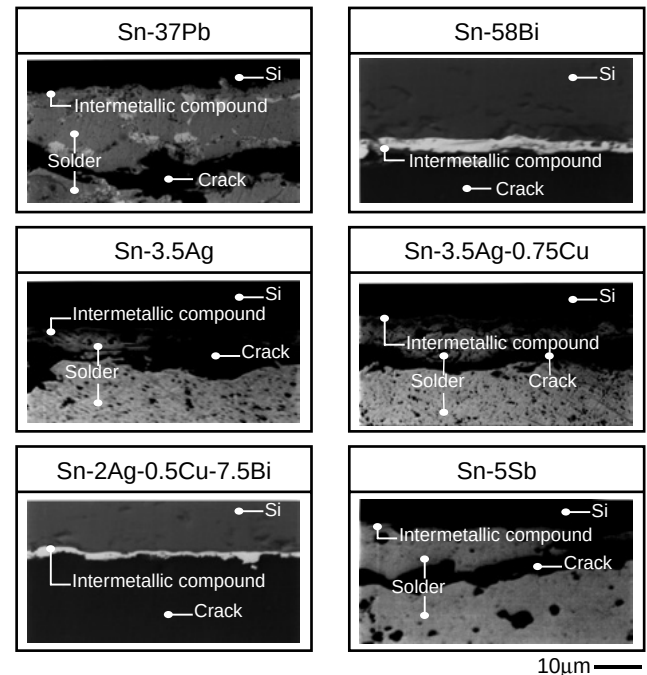


Fig.12 SEM image of cracks after thermal shock (under Si chip)

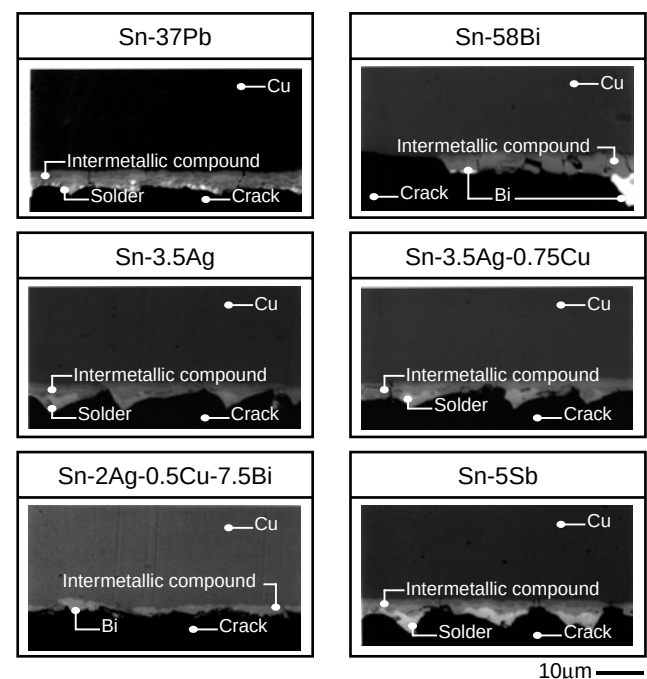


Fig.13 Peel strength of soldered area due to aging

unable to withstand distortion at the intermetallic compound/solder surface during thermal expansion, and so begins to peel at the surface where strength is lowest. In addition, intermetallic compound growth rates (i.e., change rates) are high in the case of both solders. Therefore peeling most likely began at the intermetallic compound surface. Other solders, in contrast, have a low intermetallic compound growth rate (i.e., rate of change) and are softer than Sn-Bi solders with respect to elongation. Thus no peeling occurs at the intermetallic compound/solder surface. Instead, it begins in the solder interior, where strength is lowest.

## 8. Prediction of Solder Joint Lifespan

### 8.1. Heat History in Automotive Applications

Thermal fatigue is the most important issue in the reliability of the solder joints of automotive electronics and electrical equipment. This is because these solder joints can ultimately undergo thermal fatigue failure due to the thermal stress and thermal distortion caused by repeated self-heat generation by the elements themselves, cooling, the manner and frequency of use, and seasonal changes in environmental temperature.

### 8.2. Thermal Fatigue Life Diagram

The thermal fatigue failure of a solder is believed to be compound fatigue/creep failure in which fatigue failure, the primary condition, causes creep to accelerate. Generally, the relationship expression for the thermal fatigue life ( $N_f$ ) of solder joints is expressed with the Coffin-Manson empirical equation:

$$N_f \propto C \cdot f^m \cdot (\Delta \epsilon)^{-n} \cdot \exp(Q / k \cdot T)$$

where  $C$  = material constant;  $m$ ,  $n$  = exponents,  $f$  = cycle frequency,  $\Delta \epsilon$  = plastic deformation,  $Q$  = activation energy,  $k$  = Boltzmann constant.

If  $C$  and  $f$  are constant and the creep term is coefficient  $K$ , then

$$N_f = a \cdot K \cdot (\Delta \epsilon)^{-n}$$

Taking the logarithm of both sides results in the following equation

$$\begin{aligned} \log N_f &= -n \log \Delta \epsilon + \log a \cdot K \\ y &= -n \cdot X + a \cdot K \end{aligned}$$

which results in a straight line on the log-log graph.

### 8.3. Calculation of Expected Life

In the mechanical design of a solder joint, not only the material strength but also the S-N curve, which indicates the relationship between stress and the number of cycles, is also important. A typical method for predicting joint life with an S-N curve is Miner's rule, which states that when cyclic stress  $S_n$  (a varying stress) is applied  $n_t$  times (i.e.,  $S_1 = 1$  time,  $S_2 = 2$  times,...), and  $N_t$  is the life cycle when stress  $S_t$  is applied, then

$$\sum_{t=1}^k n_t / N_t = 1$$

is the cumulative fatigue resulting from this varying stress<sup>(6)</sup>.

Thermal shock testing with different values of  $\Delta T$  was carried out to determine  $N_t$ , the life being defined as the cycle number at which peeling equal to a certain surface area or beyond occurred. Fig.15 is the life diagram for different values of  $\Delta T$ . A typical example of the thermal shock testing results for  $\Delta T = 140^\circ\text{C}$  is given in Fig.14. The stress frequency to which components are subjected in market conditions is also listed in Fig.15 to account for utilization frequency and season changes and such<sup>(7)</sup>. Table 4 gives the results of marketed Pb-free solder joint life predictions made with these results and by applying Miner's law.

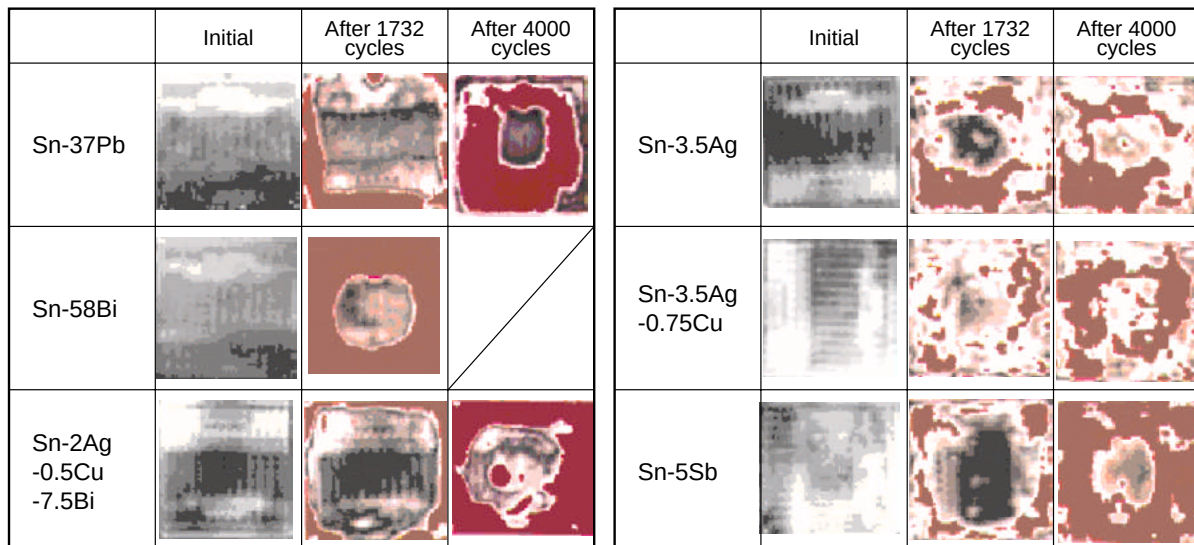


Fig.14 Cracks in soldered area due to heat impact (Red areas represent cracks)

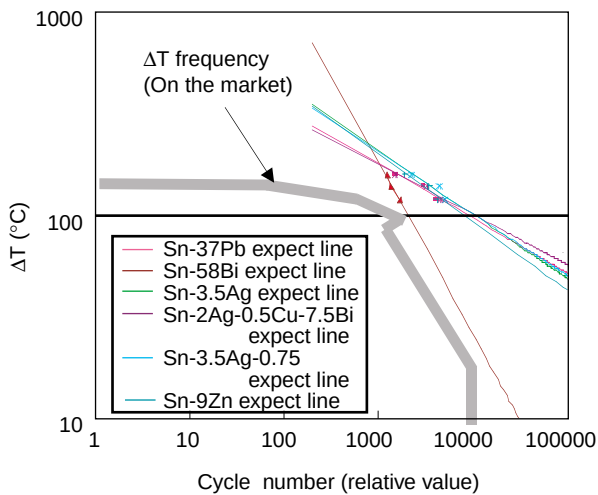


Fig.15 Requirement line for thermal fatigue

Table 4 Predicted life of solder joint

|                     | Sn-37Pb | Sn-58Bi | Sn-3.5Ag | Sn-2Ag-0.5<br>Cu-7.5Bi | Sn-3.5Ag<br>-0.75Cu | Sn-5Sb |
|---------------------|---------|---------|----------|------------------------|---------------------|--------|
| Time to failure (N) | 0.93    | 4.57    | 0.79     | 0.95                   | 0.85                | 0.90   |

The results show all solders other than Sn-58Bi to have a life equal to or greater than that of Sn-37Pb.

## 9. Conclusion

The findings of this research are as follows:

- (1) The intermetallic compound layer fracture rate and grain growth rate of Pb-free solder was shown to obey the parabolic law, being proportional to the square root of time.
- (2) The results of the thermal fatigue life prediction of Pb-free solder joints in automotive applications showed all solders other than Sn-58Bi to have a life comparable to that of Sn-37Pb.
- (3) Miner's law, which is concerned with reliability engineering, can be used in the life prediction of Pb-free solder joints.

The authors plan to perform similar life prediction for different solder compositions, different methods of soldering, and different environments (e.g., heat, thermal shock, and vibration) as a next activity.

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