Use of nanoindentation to measure and map mechanical properties of SAC 305 solder

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The reliability of soldered connections in electronic packaging depends on mechanical integrity; mechanical failure can cause electrical failure. Mechanical integrity, in turn, depends on mechanical properties. Here, we focus on the SAC 305 solder alloy (96.5% tin, 3% silver, and 0.5% copper) due to its prevalent utilization in electronic packaging. First, we demonstrate the use of nanoindentation to measure the elastic and creep properties of SAC 305. Next, we utilize an advanced form of nanoindentation to quantitatively map mechanical properties of all the components of a realistic SAC 305 solder joint.

Experimental method

A Sn-3.0Ag-0.5Cu solder joint was prepared by melting Sn-3.0Ag-0.5Cu between two nickel/gold plated Cu platens. The solder paste was produced by Kester (Itasca, IL) and a heating plate was utilized to execute the reflow suggested by the manufacturer. To prepare the surface for nanoindentation, the sample was ground with silicon carbide paper and polished using alumina suspensions (1.0, 0.3, and 0.05 µm). After polishing, the sample was attached to a sample holder using a polymer repair putty with a rated temperature of 2,000°F.

An Agilent Nano Indenter G200 with an XP head fitted with a Berkovich indenter, was used for all testing. For the measurement of Young’s modulus and creep properties, the Continuous Stiffness Measurement (CSM) option was employed to measure elastic contact stiffness by oscillating the indenter. Sample heating was accomplished utilizing Agilent’s heating stage option (heater block, thermocouple, ceramic isolator, argon gas port, and coolant ports). The spatial map of hardness was accomplished using Agilent’s Express Test option because of its ability to perform each indentation in less than five seconds.

Measuring Young’s modulus

Nanoindentation is commonly used to measure the Young’s modulus of small volumes of material [1]. Figure 1 shows the Young’s modulus of SAC 305 as a function of temperature. Each bar represents the average of five indents, and the error bars represent the standard deviation. At room temperature, the Young’s modulus was 47.5 GPa, which agrees with measurements by tensile testing. Almit, a manufacturer of solder materials, cites 51 GPa for the Young’s modulus of their SAC 305 [2]. As expected, we see a slight decrease in Young’s modulus at higher temperatures, because heat tends to decrease inter-atomic forces [3].
Measuring creep properties

“A specimen undergoing continuous deformation under a constant load or stress is said to creep [4].” Nanoindentation allows characterization of the constitutive properties of creep, including stress exponent and activation energy.

The Dorn constitutive model for creep [5] takes the following form when rendered for nanoindentation, relating indentation strain rate ($\dot{\varepsilon}$) to hardness ($H$):

$$\dot{\varepsilon} = Be^{-Q/RT}H^n$$  \hspace{1cm} (1)

where $B$ is base strain rate (determined primarily by microstructure), $Q$ is activation energy, $R$ is universal gas constant (8.3145 J/mol/K), $T$ is absolute temperature, and $n$ is stress exponent.

The most intuitive definition of strain rate is

$$\dot{\varepsilon} \equiv \frac{\dot{h}}{h}$$  \hspace{1cm} (2)

where $h^*$ is displacement rate and $h$ is displacement. However, in 1999, Lucas and Oliver demonstrated that for force-controlled indentation systems, strain rate is better defined as [6]

$$\dot{\varepsilon} \equiv \frac{\dot{P}}{P}$$  \hspace{1cm} (3)

where $P^*$ is loading rate and $P$ is load. Taking the natural logarithm of both sides of Eq. 1 yields

$$\ln(\dot{\varepsilon}) = \ln(B) - \frac{Q}{RT} + n\ln(H)$$  \hspace{1cm} (4)

Thus, to measure the stress exponent, we perform indentations in which strain rate ($\dot{\varepsilon} = P^*/P$) is...
varied and temperature is held constant. The stress exponent, $n$, is the slope of $\ln(\tau)$ with respect to $\ln(H)$. The value for $n$ obtained in this way is the same as would be obtained from a tensile creep test [6]. Recent work in indentation creep has focused on reducing thermal drift when probing small strain rates [7, 8].

**Figure 2** shows the measured hardness of SAC 305 solder at a constant temperature for three different strain rates. As expected, SAC 305 has less resistance to plasticity (i.e., lower hardness) with decreasing strain rate. As shown in **Figure 3**, we use this data to calculate a stress exponent of $n = 5.0 \pm 1.0$, where the uncertainty is the standard error of the slope. This value of stress exponent is typical for metals which creep at room temperature.

![Hardness of SAC 305 solder at a constant temperature for three different strain rates](image_url)

**Figure 2** Hardness of SAC 305 solder at a constant temperature for three different strain rates
The strategy for measuring activation energy, $Q$, is revealed by rearranging Eq. 4 as

$$\ln(H) = \frac{Q}{nR} \frac{1}{T} + \frac{1}{n} \left[ \ln(\dot{\varepsilon}_i) - \ln(B) \right]$$

Thus, to measure the activation energy, we perform nanoindentations in which the temperature is varied and the strain rate is held constant. For such tests, the slope of $\ln(H)$ vs. $(1/T)$ is $Q/(nR)$.

Implementing indentation tests which can be used to derive $Q$ using the analysis suggested by Eq. 4 is the focus of an ongoing collaboration between Agilent and CALCE.

**Mapping hardness**

Solder joints have a complex microstructure that depends on many factors: the solder and plating materials, the size of the joint (which constrains grain size), and the exposure of the joint to stress and temperature over time. Some alloys favor the development of intermetallic compounds (IMCs) within a tin-rich matrix. Previous work on the present sample of SAC 305 has shown that the IMC AuSn$_4$ develops in the bulk of the joint and that the IMC (Cu, Au)$_6$Sn$_5$ develops at the interface with Sn metallization.

In order to quickly map the hardness of the entire joint, we used an advanced form of nanoindentation, called Express Test, which optimizes the indentation process and data handling for high-speed testing. With Express Test, the indenter tip hovers just over the surface and performs indents in rapid succession to form an array of discrete indentations [9].

Here, we used Express Test to perform two arrays of indents. The first array comprised 40×40
indents within a 100µm×100µm area and spanned the breadth of the solder joint. The second array was 40×20 indents within a 100µm×50µm area and spanned the interface between the solder and the copper platen with Sn metallization. All indentations were performed to a peak force of 2 mN.

Figures 4 and 5 show side-by-side images of the indentation arrays and the resulting hardness maps. The time required to make the large array (Figure 4) was 68 minutes. Without Express Test, the time required for a similar array of 1,600 indents would have been more than a day since traditional nanoindentation requires about one minute per indentation [10].

Figure 4 (a) The indentation array on SAC 305 solder joining two nickel/gold plated copper platens, and (b) resulting hardness map. Indentations within the top white box are averaged to determine the hardness of the AuSn₄; indentations within the bottom white box are averaged to determine the hardness of the tin-rich matrix.

Figure 5 (a) The indentation array on a SAC 305 solder and Sn-plated Cu platen, and (b) resulting hardness map. Indentations within the white box are averaged to determine the hardness of the Cu platen.

In Figures 4 and 5, the size of the residual impression indicates hardness, as all indentations were performed to the same force. In softer materials, this force leaves a large residual impression.
harder materials, the impressions are correspondingly smaller. At the interface between the solder and the Sn-plated copper, the residual impressions are not perfectly triangular. This is because the interface is so hard that the surrounding (i.e., softer) material is preferentially removed during preparation for nanoindentation, leaving a 'bump' at the interface. Because nanoindentation analysis assumes a test surface that is orthogonal to the direction of indentation, this bump compromises the nanoindentation results. An alternate method of preparation, such as focused-ion-beam milling, may be better for exposing soldered surfaces for nanoindentation.

The various materials in the joint are distinguished according to hardness. In order to report quantitative hardness values for each material, domains were selected that were clearly and entirely within one kind of material; white boxes on the hardness maps identify these domains. The bulk solder is the softest material, having a hardness of about 0.5 GPa. The hardness of the tin-rich matrix was about 0.51±0.07 GPa. It should be noted that this hardness is significantly greater than what we measured previously ($H = 0.22±0.03$ GPa at a strain rate of 0.05/s). The elevated hardness is simply due to the higher strain rates which are imposed for this rapid mapping. The hardness of the AuSn$_4$ was 2.12±0.18 GPa. The (Cu, Au)$_6$Sn$_5$ that develops at the Sn-plated copper platen has extraordinary hardness – greater than 8 GPa. This mismatch in hardness is a concern for reliability, because it means that plastic strains cause much higher stresses in the IMC than in the surrounding material. In other words, a strain large enough to cause plastic flow in the Sn or AuSn$_4$ may cause only elastic deformation in the (Cu, Au)$_6$Sn$_5$, thus causing a discontinuity in stress at the boundary. This discontinuity increases the local stress-intensity factor which drives fracture.

**Conclusion**

The reliability of a microelectronic device depends on the mechanical reliability of its many solder joints. Nanoindentation is a useful technique for the mechanical characterization of solder because it can be performed in situ [9], even on the circuit board itself. Such a localized technique can be employed under a wide variety of circumstances. For example, mechanical properties can be measured at elevated temperatures, or at room temperature following a certain number of thermal cycles, or even after failure.

Here, we demonstrated accurate measurements of the elastic and creep properties of SAC 305 solder. We also used an advanced form of nanoindentation to quantitatively map the hardness of a realistic SAC 305 solder joint in about an hour.

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**Also see:**

- What’s Next for Solder?
- Lead-free soldering more reliable after all?

**References**


