

# Keysight Technologies

## In Situ Young's Modulus and Strain-Rate Sensitivity of Lead-Free SAC 105 Solder

### Application Note

#### Abstract

Motivated by our desire to understand and improve the mechanical reliability of solder joints in integrated circuits, we used instrumented indentation to measure the Young's modulus ( $E$ ) and strain-rate sensitivity ( $m$ ) of a common lead-free solder alloy, SAC 105 (98.5% Sn, 1% Ag, 0.5% Cu). Measured values ( $E = 49.1 \pm 1.6 \text{ GPa}$ ,  $m = 0.184 \pm 0.013$ ) were remarkably close to what others have measured for unalloyed Sn by means of uniaxial tension and compression experiments, thus lending credibility to the indentation method.

#### Introduction

The reliability of a microelectronic device depends on the mechanical reliability of its many solder joints. Instrumented indentation (also known as nanoindentation) is a useful technique for the mechanical characterization of solder, because it can be performed *in situ*, 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. The purpose of this work is to demonstrate the use of instrumented

indentation to measure the *in situ* Young's modulus and strain-rate sensitivity of a common lead-free solder alloy known as SAC 105, which by weight is predominantly tin (Sn) alloyed with 1% silver (Ag) and 0.5% copper (Cu).

Since SAC 105 is predominantly (98.5%) tin, it is appropriate and helpful to compare our results to those measured by others for unalloyed tin. The nominal Young's modulus of tin is 50 GPa<sup>1</sup>. Recently, Burek et al. measured the strain-rate sensitivity of tin by compressing nano-scale pillars fabricated by electron-beam lithography. Although the yield stress depended strongly on the pillar diameter, strain-rate sensitivity did not. For pillars having a diameter of 920 and 560 nm, they reported a strain rate sensitivity of 0.181<sup>2</sup>.

Instrumented indentation testing is a technique for measuring the mechanical properties of materials. It is a development of traditional hardness tests such as Brinell, Rockwell, Vickers, and Knoop. Instrumented indentation testing is similar to traditional hardness testing in that a hard indenter, usually diamond, is pressed into contact with the test material. However, traditional hardness testing yields only one measure of deformation at one applied force, whereas during an instrumented indentation test, force and penetration are measured for the entire

time that the indenter is in contact with the material. Nearly all of the advantages of instrumented indentation derive from this continuous measurement of force and displacement. In 1992, Oliver and Pharr proposed an analytic method by which contact area could be calculated from the force-displacement data, thus eliminating the need to image the residual impression when determining hardness ( $H$ ) as the indentation force divided by the contact area<sup>3</sup>. Also, the displacement recovered as the indenter is withdrawn manifests elastic recovery and thus can be used to obtain Young's modulus ( $E$ )<sup>3,4</sup>. Superimposing a small oscillation on the indentation force allows the separation of elastic and plastic components of deformation, and the practical benefits of this separation are myriad<sup>3,5</sup>. Instrumented indentation is particularly well suited for testing small volumes of material such as thin films, particles, or other small features. Even for larger volumes of material which could be tested in a tensile configuration, instrumented indentation is often preferred for its speed and simplicity; sample preparation is relatively easy, and many tests can be performed on a single sample.

In addition to hardness and Young's modulus, instrumented indentation can be used to characterize creep in metals<sup>6</sup>. This is because hardness is a manifestation of the yield stress ( $\sigma$ ) of the metal.

Under conditions of creep, the yield stress depends on temperature and strain rate. As a manifestation of yield stress, hardness is not a constant, but instead depends on temperature and strain rate just as yield stress does.

The phenomenon of creep in metals is governed by diffusion: vacancies diffuse into the material and enable dislocations to move more freely and overcome obstacles to motion. Thus, the relationship between stress ( $\sigma$ ) and strain rate ( $\dot{\epsilon}$ ) can be captured with an Arrhenius term multiplied by stress raised to an exponent:

$$\dot{\epsilon} = A e^{-Q/RT} \sigma^n \quad (1)$$

where  $A$  is the base strain rate (determined primarily by the microstructure),  $Q$  is the activation energy,  $R$  is the universal gas constant,  $T$  is the absolute temperature, and  $n$  is the stress exponent for creep. In the literature of creep, Eq. 1 is called the “Dorn” model, after John E. Dorn, who proposed and developed the model in his foundational work on creep in the 1960’s<sup>7,8</sup>. Thus, the values of three constants— $A$ ,  $Q$ , and  $n$ —must be determined for a particular material in order to fully describe its creep behavior with the Dorn model.

In the configuration of an indentation test, a form of the Dorn model can be used to relate indentation strain rate ( $\dot{\epsilon}_i$ ) with hardness ( $H$ )<sup>6</sup>:

$$\dot{\epsilon}_i = B e^{-Q/RT} H^n. \quad (2)$$

If the indenter is a pyramid or a cone, then the indentation strain rate ( $\dot{\epsilon}_i$ ) is the displacement rate divided by the displacement ( $\dot{h}/h$ )<sup>9</sup>. However, Lucas and Oliver demonstrated the practical equivalence of defining the indentation strain rate as the force-application rate divided by the force ( $\dot{P}/P$ )<sup>6</sup>. Because the Keysight Technologies, Inc. G200 NanoIndenter is a force-controlled instrument, it is logistically easier to control  $\dot{P}/P$  than  $\dot{h}/h$ . Thus, in this work,

$$\dot{\epsilon}_i = \dot{P}/P. \quad (3)$$

In Eq. 2,  $Q$  and  $n$  have exactly the same theoretical meaning and value as in Eq. 1, thus allowing indentation to be used to determine these two constants in the Dorn model. However, the leading coefficient,  $B$ , in Eq. 2 does not have the same meaning as its analogue  $A$  in Eq. 1. Presently, there is no established

technique for using instrumented indentation to determine the base strain rate  $A$  in Eq. 1. Taking the natural logarithm of both sides of Eq. 2 yields:

$$\ln(\dot{\epsilon}_i) = \ln(B) - \frac{Q}{RT} + n \ln(H). \quad (4)$$

Thus, if temperature is held constant and strain rate is varied, then the stress exponent,  $n$ , can be determined as the slope of  $\ln(\dot{\epsilon}_i)$  with respect to  $\ln(H)$ . Likewise, if hardness is invariant, then the slope of  $\ln(\dot{\epsilon}_i)$  vs.  $(1/T)$  is equal to  $-Q/R$  which leads directly to a value for the activation energy,  $Q$ . Obviously, the determination of  $n$  is far simpler, because it can be accomplished at room temperature.

In the literature of creep, the strain-rate sensitivity ( $m$ ) is the inverse of the stress exponent ( $m = 1/n$ ). Assuming a constant temperature, Eq. 2 simplifies to

$$\dot{\epsilon}_i = C H^m, \quad (5)$$

where  $C$  is a constant that incorporates both the base strain rate and the Arrhenius term. Raising both sides of Eq. 5 to the power of  $m = 1/n$  and rearranging to solve for hardness yields

$$H = D \dot{\epsilon}_i^m \quad (6)$$

where  $D$  is a constant. Eq. 6 reveals that if  $m = 0$ , then the right hand side of Eq. 6 is simply the constant  $D$ , which means that the hardness of the material does not depend at all on how fast the material is deformed. (Sapphire has a strain-rate sensitivity which is very near zero.) However, as  $m$  increases, the hardness of the material depends increasingly on the rate at which it is deformed. Metals typically have strain-rate sensitivity of  $0.10 < m < 0.25$ . Working from Eq. 6, the strain rate sensitivity,  $m$ , is the slope of  $\ln(H)$  with respect to  $\ln(\dot{\epsilon}_i)$ :

$$\ln(H) = m \ln(\dot{\epsilon}_i) + \ln(D). \quad (7)$$

## Experimental Procedure

### Sample

We tested a sectioned solder joint formed between a printed wiring board (PWB) and 2512 chip resistor held in an epoxy potting compound (Figure 1). The underlying metallization was copper and the board was finished with Organic Solderability Preservative (OSP). The solder paste was

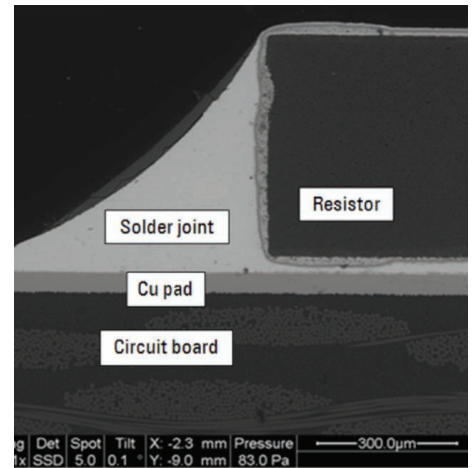


Figure 1. On-board SAC 105 solder joint.

SAC 105 (98.5%wt Sn, 1%wt Ag, 0.5%wt Cu). The reflow was conducted in air. The reflow temperature was 244°C, and the time above liquidus was 50–74 seconds. The ramp-up rate was 0.48°C/sec, and the ramp-down rate was 3.41°C/sec. Following reflow, the board was aged at 150°C for 24 hours.

### Equipment

A Keysight G200 NanoIndenter (XP head, Berkovich indenter) was used for all testing. The Continuous Stiffness Measurement (CSM) option was employed in order to measure the elastic contact stiffness by oscillating the indenter.

### NanoSuite Test Method

The test method “G-Series XP CSM Thin Film SRS” was used for all testing. This test method imposes a user-defined strain rate and returns the hardness for that rate. This method is ideally suited for testing at slow strain rates, because it is insensitive to thermal drift. The insensitivity to drift is achieved by calculating displacement and contact area, not from the gross motion of the indenter, but from the elastic contact stiffness as measured by the CSM option. This is legitimate so long as the Young’s modulus of the material is independent of strain rate, which is a sound assumption for metals. The maximum indentation depth was 1200nm. All properties were determined at an indentation depth of 1000nm (1 μm).

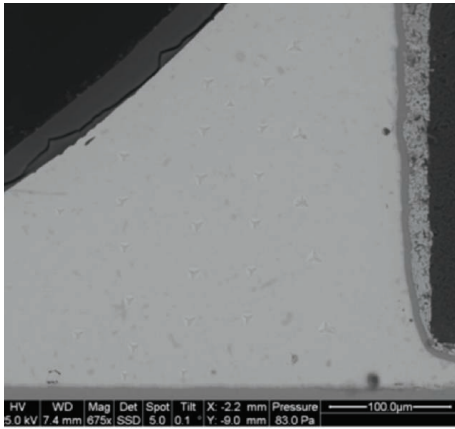


Figure 2. Residual impressions from indentation testing on SAC 105 solder joint.

### Testing

Six indentations were performed at each of three different strain rates: 0.05/sec, 0.01/sec, and 0.002/sec. The CSM option was used to cause the indenter to oscillate at 45Hz with an amplitude of 2nm. All tests were conducted at a temperature of 29.15±0.10°C.

### Results and Discussion

Figure 2 shows the residual indentation impressions on the SAC 105 solder joint. (Impressions from preliminary tests are also visible.) Table 1 summarizes the results for all successful tests. Two tests at the highest strain rate failed due to an

unexpected degree of surface roughness. (When surface roughness is expected, it is easily accommodated by using a larger approach distance.) Figure 3 shows the Young's modulus as a function of penetration depth for tests at the highest strain rate of 0.05/sec. Anomalous values at small displacements are likely due to the presence of an oxide layer which fractured as a result of indentation. Nevertheless, Young's modulus is about 50GPa once the indenter displacement exceeds 200nm, and the cited value (49.9±1.6GPa) is obtained from the displacement range identified in Figure 3 (950-1050nm). This measured value compares remarkably well with the nominal Young's modulus for Sn, which is 50GPa. Thus, we conclude that for SAC 105, the alloying elements of Ag and Cu do not strongly influence the Young's modulus.

Figure 4 shows the linear fit to the  $\ln(H)$  vs.  $\ln(\dot{\epsilon})$  results for all 16 successful indentations. The slope of this line is 0.184 and the standard error of the slope<sup>1</sup> is 0.013, giving a strain rate sensitivity of  $m = 0.184 \pm 0.013$ . The corresponding stress-exponent for creep is  $n = 5.43$ . These values compare remarkably well with those measured by Burek et al. for bulk tin (c.f.  $m = 0.181$ ,  $n = 5.54$ )<sup>2</sup>. Thus, it seems that for SAC 105, the alloying elements do not strongly influence strain-rate sensitivity either.

Indent	Strain Rate 1/s	E GPa	H GPa
1	0.002		0.1058
2	0.002		0.1105
3	0.002		0.1357
4	0.002		0.1915
5	0.002		0.1444
6	0.002		0.1049
7	0.01		0.1871
8	0.01		0.1770
9	0.01		0.1744
10	0.01		0.2026
11	0.01		0.2041
12	0.01		0.1929
13	0.05	50.81	0.2491
14	0.05	48.20	0.2292
15	0.05	51.70	0.2339
16	0.05	49.01	0.2089

Table 1. Summary of indentation results for SAC 105 at various strain rates.

The CSM option, which returns the instantaneous elastic stiffness of the contact, is an essential tool for accurately measuring the Young's modulus of soft metals. This kind of dynamic measurement completely deconvolutes the elastic and plastic deformation, thus allowing the accurate characterization of elasticity.

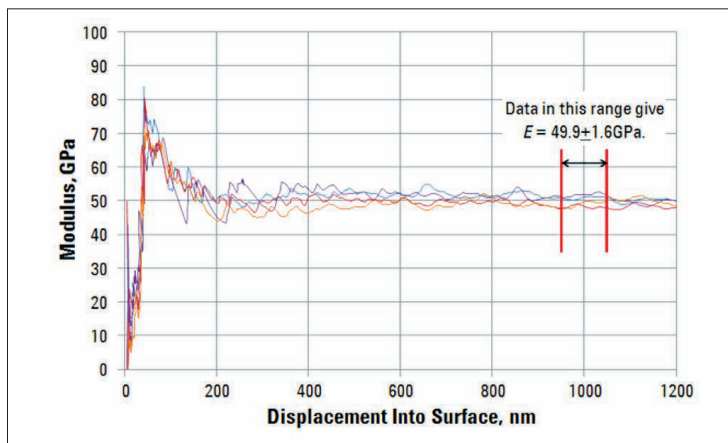


Figure 3. Young's modulus as a function of indenter displacement for indentation tests at the highest strain rate (0.05/sec). Anomalous values at small displacements are due to an oxide layer. Measured Young's modulus compares well with the nominal value for Sn (50GPa).

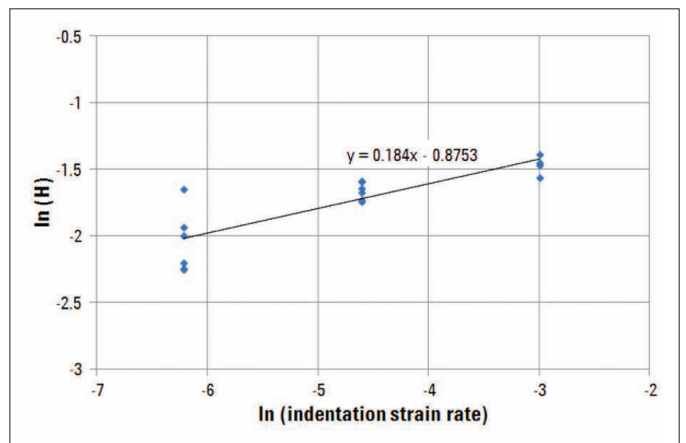


Figure 4. Natural logarithm of hardness vs. natural logarithm of strain rate; the slope of these data give a strain-rate sensitivity of  $m = 0.184 \pm 0.013$ .

1. Calculated using the array form of Microsoft Excel's LINEST function.

Furthermore, with a continuous measure of Young's modulus, we can discern various phenomena such as surface anomalies and substrate influence. Armed with such information, we can design our experiment and analysis so as to avoid or minimize the influence of such phenomena. For example, in this work, we only include measurements of Young's modulus which are relatively deep (950-1050nm), because we see that shallower measurements, being affected by a surface layer, are not truly representative of the solder material (Figure 3).

These results demonstrate the benefit of using the CSM option (rather than the gross position of the indenter) to infer the penetration of the indenter into the sample. Each indentation test at the slowest strain rate (0.002/sec) takes about 10 minutes, yet the hardness results are remarkably consistent.

## References

1. Anonymous. Tin. 2013 February 3, 2013 [cited 2013 February 5]; Available from: <http://en.wikipedia.org/wiki/Tin>.
2. Burek, M.J., Budiman, A.S., Jahed, Z., Tamura, N., Kunz, M., Jin, S., Han, S.M.J., Lee, G., Zamecnik, C., and Tsui, T.Y., "Fabrication, Microstructure, and Mechanical Properties of Tin Nanostructures," *Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing* 528(18), 5822–5832, 2011.
3. Oliver, W.C. and Pharr, G.M., "An Improved Technique for Determining Hardness and Elastic-Modulus Using Load and Displacement Sensing Indentation Experiments," *Journal of Materials Research* 7(6), 1564–1583, 1992.
4. Doerner, M.F. and Nix, W.D., "A Method for Interpreting the Data from Depth-Sensing Indentation Instruments," *Journal of Materials Research* 1(4), 601–609, 1986.
5. Hay, J.L., Agee, P., and Herbert, E.G., "Continuous Stiffness Measurement during Instrumented Indentation Testing," *Experimental Techniques* 34(3), 86–94, 2010.
6. Lucas, B.N. and Oliver, W.C., "Indentation Power-Law Creep of High-Purity Indium," *Metallurgical and Materials Transactions A-Physical Metallurgy and Materials Science* 30(3), 601–610, 1999.
7. Dorn, J.E. and Shanley, F.R., *Mechanical Behavior of Materials at Elevated Temperatures*. 1st ed, p. 1961, New York: McGraw-Hill.
8. Amin, K.E., Mukherjee, A.K., and Dorn, J.E., "A Universal Law for High-Temperature Diffusion Controlled Transient Creep," *Journal of the Mechanics and Physics of Solids* 18(6), 413–426, 1970.
9. Pollock, H.M., Maugis, D., and Barquins, M., *Characterization of Submicrometre Surface Layers by Indentation, in Microindentation Techniques in Materials Science and Engineering*, P.J. Blau and B.R. Lawn, Editors. 1986, ASTM: Philadelphia. p. 47–71.

Thus, the CSM option opens the door to the evaluation of creep at very small strain rates.

## Conclusions

We used instrumented indentation to measure the Young's modulus and strain-rate sensitivity of the solder alloy SAC 105. At room temperature, the Young's modulus was  $E = 49.1 \pm 1.6$  GPa and the strain-rate sensitivity was  $m = 0.184 \pm 0.013$ . These values are remarkably close to what others have measured for unalloyed Sn by means of uniaxial tension and compression experiments, thus lending credibility to the indentation method. Even at the smallest strain rate, hardness values were repeatable, because indenter penetration was inferred from the dynamic stiffness measurement rather than from the gross motion of the indenter.

For more information on Keysight Technologies' products, applications or services, please contact your local Keysight office. The complete list is available at: [www.keysight.com/find/contactus](http://www.keysight.com/find/contactus)

### Americas

Canada	(877) 894 4414
Brazil	55 11 3351 7010
Mexico	001 800 254 2440
United States	(800) 829 4444

### Asia Pacific

Australia	1 800 629 485
China	800 810 0189
Hong Kong	800 938 693
India	1 800 112 929
Japan	0120 (421) 345
Korea	080 769 0800
Malaysia	1 800 888 848
Singapore	1 800 375 8100
Taiwan	0800 047 866
Other AP Countries	(65) 6375 8100

### Europe & Middle East

Austria	0800 001122
Belgium	0800 58580
Finland	0800 523252
France	0805 980333
Germany	0800 6270999
Ireland	1800 832700
Israel	1 809 343051
Italy	800 599100
Luxembourg	+32 800 58580
Netherlands	0800 0233200
Russia	8800 5009286
Spain	0800 000154
Sweden	0200 882255
Switzerland	0800 805353
	Opt. 1 (DE)
	Opt. 2 (FR)
	Opt. 3 (IT)
United Kingdom	0800 0260637

For other unlisted countries:  
[www.keysight.com/find/contactus](http://www.keysight.com/find/contactus)  
(BP-07-10-14)

[www.keysight.com/find/nano](http://www.keysight.com/find/nano)