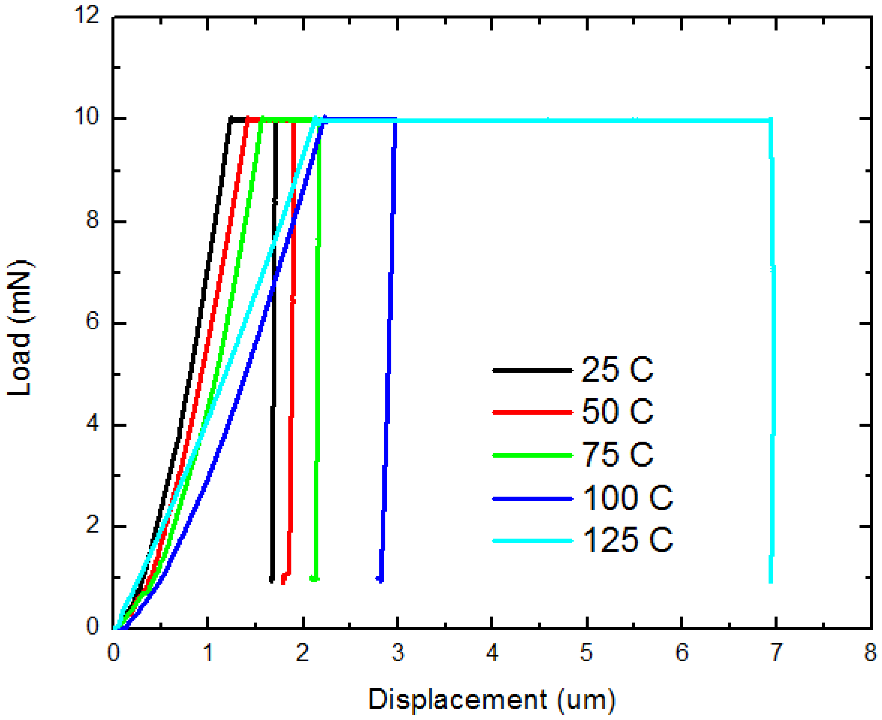
THERMOMECHANICAL ANALYSIS OF SOLDER USING NANOINDENTATION



Prepared by

Duanjie Li, PhD & Alexis Celestin

INTRODUCTION

The quality of soldering is determined by the bonding quality, the metallurgical interaction and formation of the solder bump. Properties of metals and alloys vary as the temperature elevates. Creep failure occurs when solder alloys is under a constant stress at elevated temperatures[i]. Therefore, the thermomechanical behavior of the solder joints is vital when the electronics components are exposed to a high temperature service environment.

IMPORTANCE OF HIGH TEMPERATURE NANOINDENTATION TEST FOR SOLDER

Solder joints are subjected to thermal and/or external stress when the temperature exceeds 0.6 T_m where T_m is the melting point of the material in Kelvin. The creep behavior of solders at elevated temperatures can directly influence the reliability of solder interconnections[ii]. As a result, a reliable and quantitative test of the thermomechanical behavior of the solder at different temperatures is in need. The Nano module of the Nanovea Mechanical Tester applies the load by a high-precision piezo and directly measures the evolution of force and displacement. The advanced heating oven provides a uniform temperature at the tip and sample surface, which ensures measuring accuracy and minimizes the influence of thermal drift.

MEASUREMENT OBJECTIVE

In this application, we showcased that the Nanovea Mechanical Tester in Nanoindentation mode is an ideal tool for studying the thermomechanical properties of a Sn60Pb40 solder sample at high temperatures.

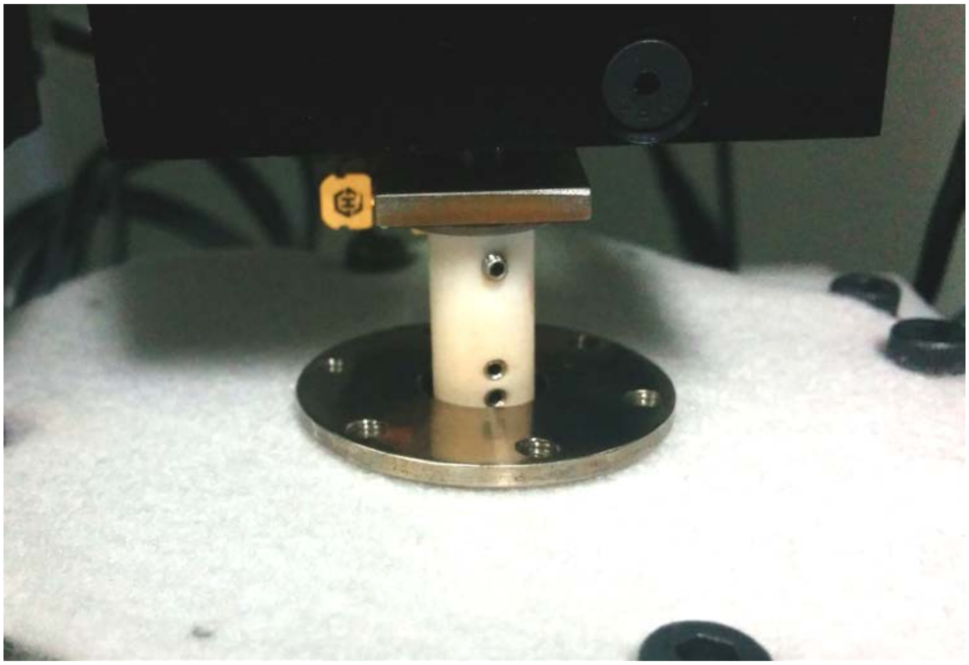


Fig. 1: Setup of the high temperature nanoindentation test.

TEST CONDITIONS

Sn60Pb40 solder is composed of 60% tin and 40% lead, and it is widely used for hand and automated soft soldering in electronics and semiconductor industries. In this study, a Sn60Pb40 solder sample is tested by nanoindentation at different temperatures ranging from the room temperature to 125 °C. The thermal drift is minimized by using an oven that fully encloses both the sample and indenter in an environment with uniform temperature. The creep is measured by the change of indentation depth at the maximum load of 10 mN for 300 s. A 60 s holding period was taken at 90% unloading load to perform further thermal drift correction in all experiments. The test conditions are summarized in Table 1.

Temperature (°C)	23, 50, 75, 100 and 125
Maximum load (mN)	10
Loading rate (mN/min)	20
Unloading rate (mN/min)	20
Creep time (s)	300
Indenter type	Berkovich Diamond

Table 1: Test conditions of the nanoindentation.

RESULTS AND DISCUSSION

The load vs. displacement plot of the nanoindenation tests at different temperatures is shown in Fig. 2 and the creep curves are compared in Fig. 3. The evolution of hardness and creep as a function of the temperature is summarized in Fig. 4. As an example in Fig. 2, the AB, BC and CD portions of the load-displacement curve for the measurement at the temperature of 125 °C represent the loading, creep and unloading processes, respectively.

As the sample temperature increases from room temperature to 125 °C, the load-displacement curve shifts towards higher penetration depth, resulting in progressively decreased hardness from ~0.28 to ~0.09 GPa. At the same time, the creep gradually increases from 477 to 751 nm when the sample is heated up to 100 °C. However, it exhibits a significant jump to a value of 4813 nm as the temperature continues to rise to 125 °C. The increased creep behavior at higher temperature is attributed to the reduced work hardening effect by a faster recovery rate [iii].

The thermomechanical properties of the solder joints at elevated temperature are critical when the electronics devices are used for high temperature industrial applications, such as in the aerospace and automotive industries. By precisely measuring the hardness and creep of the solder at different elevated temperatures, a more complete picture of the reliability of solder interconnections in different environment can be obtained. The change of displacement at the maximum load under temperatures of 23, 50, 75, 100 and 125 °C provides a quantitative and reliable measurement on the thermomechanical behavior of the solder sample.

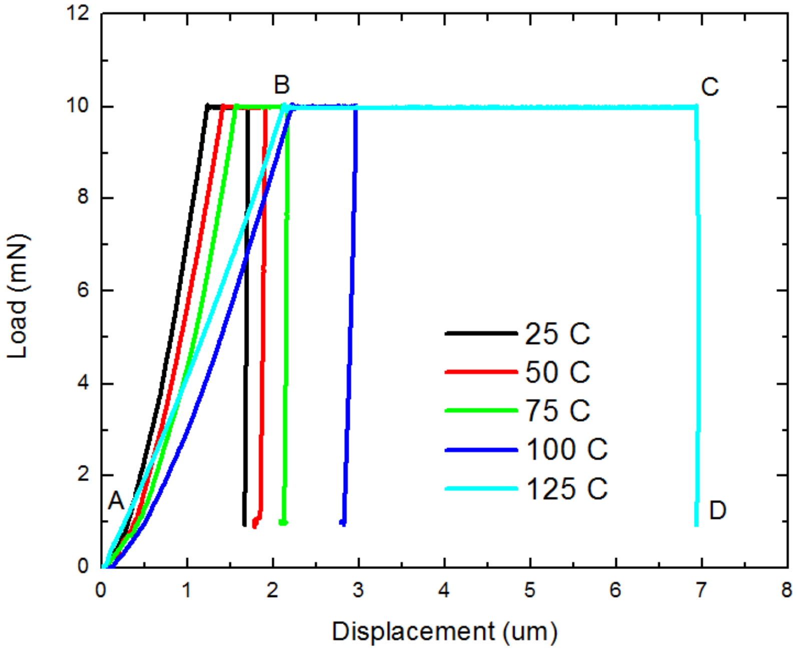


Fig. 2: The load vs. displacement plots at various temperatures.

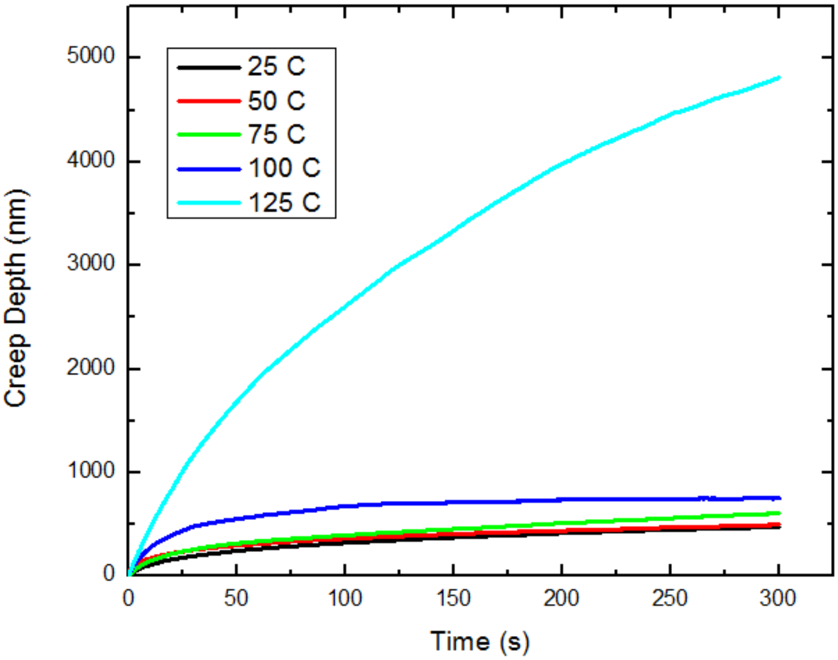


Fig. 3: Creeping at a maximum load of 10 mN for 300 s at various temperatures.

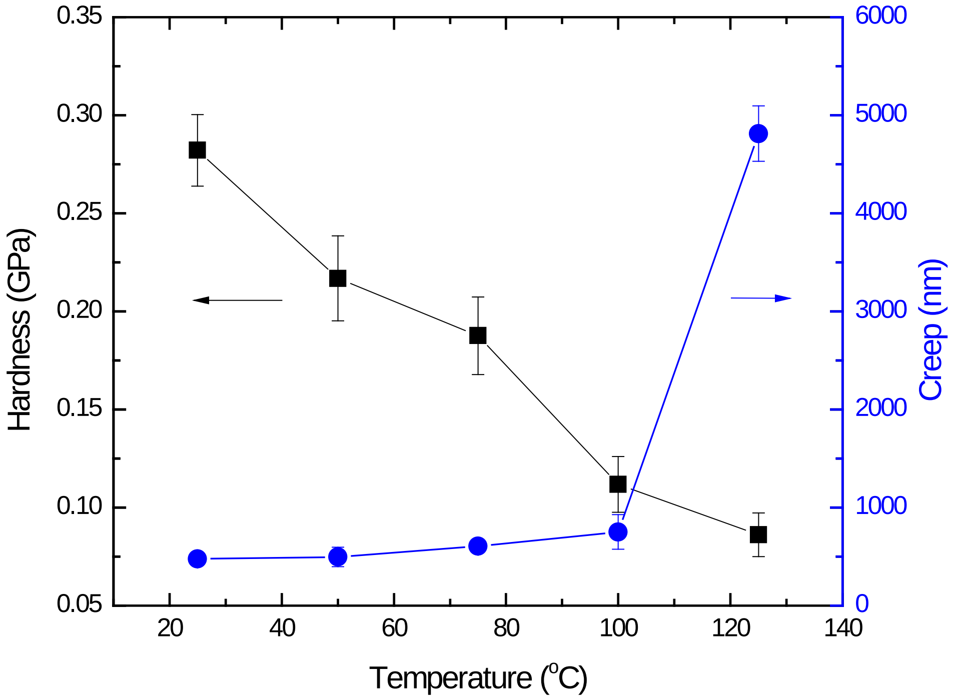


Fig. 4: The change of hardness and creep depth at different temperatures.

CONCLUSION

In this study, we showcased the capacity of the Nanovea Mechanical Tester in performing the nanoindentation test on a solder sample at elevated temperatures. This test measures the thermomechanical properties, including hardness and creep, of the Sn60Pb40 solder sample at different temperatures, which is essential in selecting the proper solder material for high temperature applications. The solder shows lower hardness and increased creep behavior as the temperature elevates. The amount of the creep contributes to significant plastic deformation during the indentation at high temperatures, leading to lower measured hardness.

The advanced oven of the Nanovea Mechanical Tester fully encloses both the sample and indenter in an environment with uniform temperature to minimize the effect of thermal drift. Moreover, it ensures that the contact of the sample surface and the indenter reaches and maintains the desired temperature during the test process to ensure measurement accuracy.

The Nanovea Mechanical Testers provide unmatched multi-function Nano and Micro/Macro modules on a single platform. Both the Nano and Micro/Macro modules include scratch tester, hardness tester and wear tester modes, providing the widest and most user friendly range of testing available on a single module.

To learn more about Nanovea Mechanical Tester or Lab Services.

MEASUREMENT PRINCIPLE

Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until complete relaxation occurs. The load is applied by a piezo actuator and the load is measured in a controlled loop with a high sensitivity load cell. During the experiment the position of the indenter relative to the sample surface is precisely monitored with high precision capacitive sensor.

The resulting load/displacement curves provide data specific to the mechanical nature of the material under examination. Established models are used to calculate quantitative hardness and modulus values for such data. Nanoindentation is especially suited to load and penetration depth measurements at nanometer scales and has the following specifications:

Maximum displacement (Dual Range) : 50 μm or 250 μm
Depth Resolution (Theoretical) : 0.003 nm
Depth Resolution (Noise Level) : 0.15 nm
Maximum force : 400 mN
Load Resolution (Theoretical) : 0.03 μN
Load Resolution (Noise Floor) : 0.3 μN

Analysis of Indentation Curve

Following the ASTM E2546 (ISO 14577), hardness and elastic modulus are determined through load/displacement curve as for the example below.

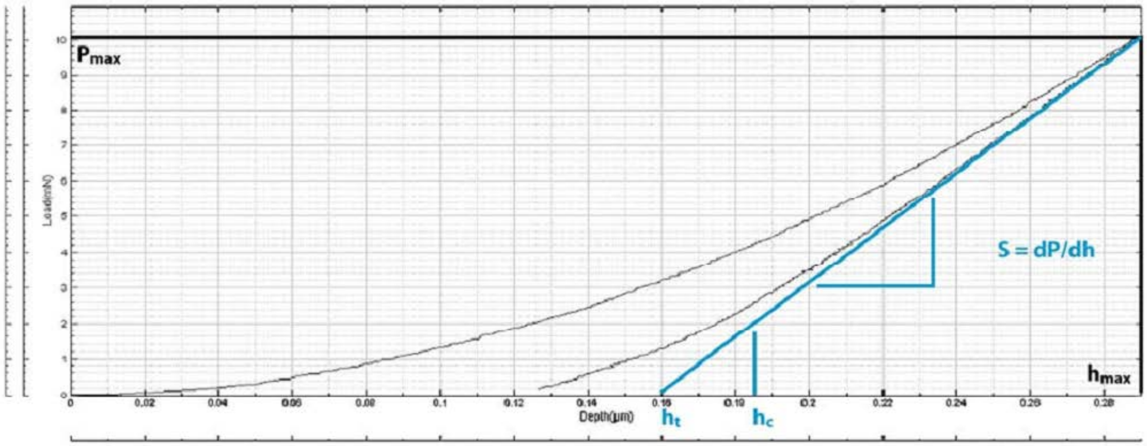


Fig. 5: Load-displacement curve of nanoindentation.

Hardness

The hardness is determined from the maximum load, $P_{\max}$, divided by the projected contact area, A_c:

$$H = \frac{P_{\max}}{A_c}$$

Young's Modulus

The reduced modulus, E_r, is given by:

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}}$$

Which can be calculated having derived S and A_C from the indentation curve using the area function, A_C being the projected contact area. The Young's modulus, E, can then be obtained from:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$$

Where E_i and ν_i are the Young's modulus and Poisson's ratio of the indenter and ν the Poisson's ratio of the tested sample.

How are these calculated?

A power-law fit through the upper 1/3 to1/2 of the unloading data intersects the depth axis at h_t. The stiffness, S, is given by the slope of this line. The contact depth, h_c, is then calculated as:

$$h_c = h_{\max} - {3P_{\max}}/{4S}$$

The contact Area A_c is calculated by evaluating the indenter area function. This function will depend on the diamond geometry and at low loads by an area correction.

For a perfect Berkovich and Vickers indenters, the area function is $A_c=24.5h_c^2$. For Cube Corner indenter, the area function is $A_c=2.60h_c^2$. For Spherical indenter, the area function is $A_c=2\pi Rh_c$, where R is the radius of the indenter. The elastic components, as previously mentioned, can be modeled as springs of elastic constant E, given the formula: $\sigma = E\varepsilon$ where σ is the stress, E is the elastic modulus of the material, and ε is the strain that occurs under the given stress, similar to Hooke's Law. The viscous components can be modeled as dashpots such that the stress-strain rate relationship can be given as $\sigma = \eta\frac{d\varepsilon}{dt}$, where σ is the stress, η is the viscosity of the material, and $d\varepsilon/dt$ is the time derivative of strain.

Since the analysis is very dependent on the model that is chosen, Nanovea provides the tool to gather the data of displacement versus depth during the creep time. The maximum creep displacement versus the maximum depth of indent and the average speed of creep in nm/s is given by the software. Creep may be best studied when loading is quicker. Spherical tip is a better choice.

Other possible measurements by Nanovea Mechanical Tester:
Stress-Strain & Yield Stress, Fracture Toughness, Compression strength, Fatigue testing and many others.

DMA SINUS MODE PRINCIPLE

Sinus Mode (Ranging from 0.1 Hz to 100 Hz): A sinusoidal stress is applied and the strain in the material is measured. This allows plotting hardness and elastic modulus versus depth and can be used to study viscoelastic materials such as polymers, varnishes, plastics.

Storage modulus *E'* characterizes the elastic behavior.
Loss Modulus *E''* characterizes the viscous behavior (loss of energy due to internal friction).

$$E^* = E' + iE'', \qquad E' = \frac{\sqrt{\pi}}{2\sqrt{A_{co}}}\frac{\Delta P}{\Delta h} Cos\phi \ (1-\nu^2), \qquad E'' = \frac{\sqrt{\pi}}{2\sqrt{A_{co}}}\frac{\Delta P_o}{\Delta h_o} Sin\phi \ (1-\nu^2)$$

Where ϕ, the phase shift between depth and load curves, $\frac{\Delta P_o}{\Delta h_o}$, the variation of load and depth respectively for one oscillation. A_{co}, the projected contact area for the oscillation. The viscosity factor λ can be calculated from $\lambda = \frac{1}{2\pi f}\frac{\Delta P_o}{\Delta h_o} Sin\phi$ where f is the frequency at which the test was performed.

---

[i] **Y.C. Liu , J.W.R. Teo, S.K. Tung, K.H. Lam, Journal of Alloys and Compounds 448 (2008), 340–343**
[ii] **M.J. Mayo, W.D. Nix, Acta Metallurgica 36 (1988) 2183–2192.**
[iii] **W. Bolton, Engineering Materials Technology, Butterworth-Heinemann (1998), p. 435**