In-Situ Mechanical Testing at Extreme Temperatures

Nanoindentation + Heating up to 800°C

SEM PicoIndenter®

The PI Series SEM PicoIndenter instruments are depth-sensing mechanical test systems designed to easily interface with a wide range of scanning electron microscopes. These systems couple the ability to acquire quantitative nanomechanical data with the power to precisely control the test position, as well as observe the sample before, during, and after each test for a more complete understanding of deformation and failure processes.

Extended Range PicoIndenter®

The **PI 88 SEM PicoIndenter®** offers an enhanced load and displacement range, combined with the ability to tilt and rotate the sample for superior tip-sample alignment, or align with additional detectors for supplemental analysis without having to break vacuum.

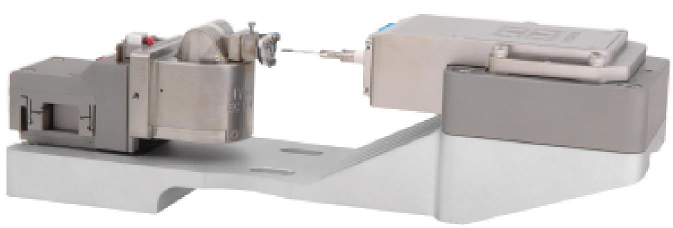

Figure 1: PI 88 with 800°C heating module.

800°C In-Situ Heating

By equipping the PI 88 with the 800°C heating option, researchers now have the ability to conduct nano- and micro- mechanical tests on advanced materials in conditions that mirror their extreme operating environments, while directly observing the tests with SEM imaging. The 800°C heating module has been carefully designed to allow for **active heating of both the tip and sample** for test stability, while minimizing thermal flow to other parts of the instrument.

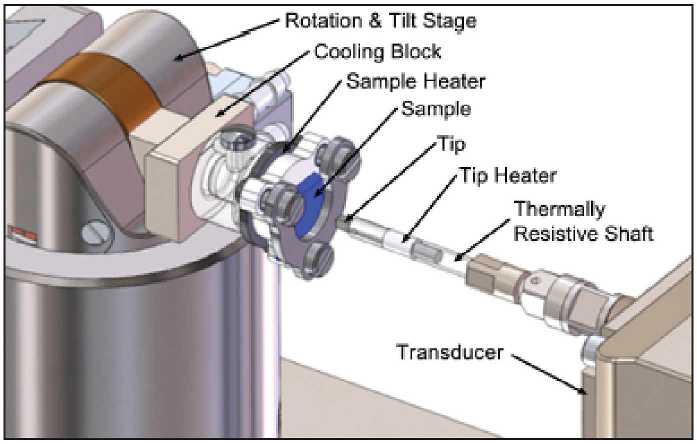

Figure 2: Components of the 800°C Heating Module on a PI 88 SEM PicoIndenter.

Features:

- Actively heated tip and sample for isothermal contact and maximum test stability.
- Wide variety of tip *materials* and *geometries* available.
- Mechanically secured sample for simplified test setup.
- Low power, localized heating.
- Integrated thermal management system through active cooling.
- Vacuum environment minimizes reactive chemistries and oxidation of sensitive samples.
- Optional tilt and rotation capabilities enhance sample positioning to ensure correct tip/sample alignment.
- Easily replacable long-lived, reusable heaters.

Case Study: Indentation on (001) Si

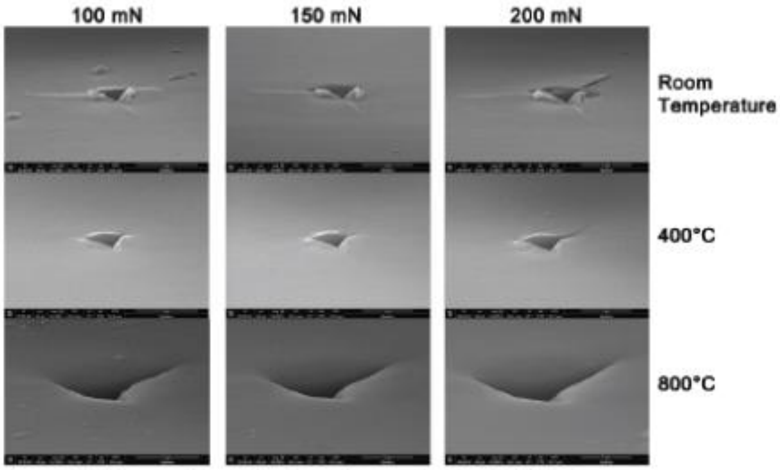

Figure 3: SEM images of the residual impressions resulting from the indentation tests.

The influence of temperature on mechanical stability has long been an area of interest across the materials spectrum. Silicon, for example, exhibits a remarkable resistance to softening at moderate temperatures (<500°C), with only a small reduction in measured hardness. The objective of this study is to understand the mechanical properties and softening effect of Si at much higher temperatures.

To evaluate the effect of temperature, a PI 88 equipped with a cube-corner indentation probe was used to perform a series of quasi-static indentation tests on (001) Si at room temperature, 400°C & 800°C.

Figure 4 shows the force versus displacement curves from the indentation testing at each of the temperatures. The data collected at 400°C shows a repeatable increase in the hardness of the (001) Silicon at this temperature relative to room temperature, possibly caused by the suppression of observable fracture at 400°C, as shown in the SEM images of the residual indent impressions in Figure 3. The data from identical tests performed at 800°C indicates significant softening of the (001) Silicon, as the curves at this temperature achieve a much greater displacement relative to those collected at room temperature and 400°C.

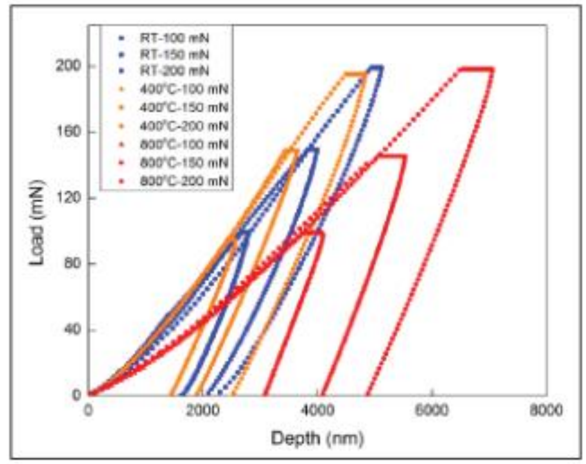

Figure 4: Load-displacement curves from the indentation tests performed on Si at room temperature, 400°C, and 800°C.

Effect of Temperature: Brittle to Ductile Transition

- At room temperature, large radial cracks can be seen emanating from the corners of the impression in addition to significant spallation along the edges.
- At 400°C, the fracture resulting from indentation appears to be suppressed, indicating an increase in fracture toughness.
- The large, crack-free impressions at 800°C indicate significant softening and the transition to primarily ductile behavior.

Other Applications of the SEM PicoIndenter® & 800°C Heating Module

- Superalloys
- Automotive Engine and Exhaust Components.
- Thermal Barrier Coatings
- Irradiated Materials
- High Temperature Fuel Cells
- Metals & Alloys
- Ceramics and Ceramic-Matrix Composites

9625 West 76th St. Minneapolis, MN 55344 TEL: 1-952-835-6366 FAX: 1-952-835-6166 WWW.BRUKER.COM/NI