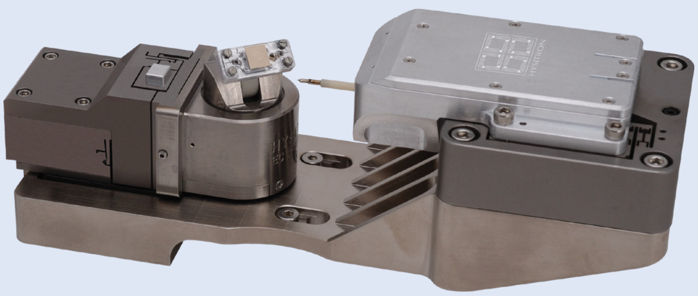
# Heating up to 800 °C

High Temperature Mechanical Characterization on the SEM PicoIndenter®



**PI 87 SEM PicoIndenter®** instrument equipped with 800 °C resistive heater.

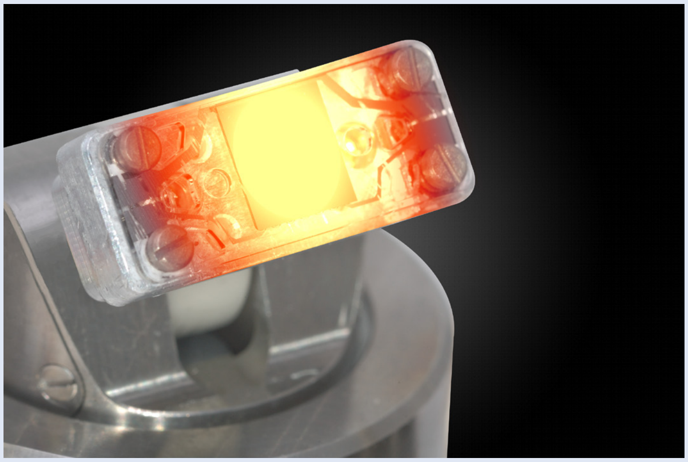
Recent advances in instrumentation have, for the first time, enabled *in situ* nanomechanical materials characterization at high temperatures (>400 °C). Such testing benefits applications (aerospace, automotive, nuclear, fusion, etc.) which demand high temperature materials capable of reliable performance in extreme operational environments. For example, turbines and combustion engines exhibit maximum thermodynamic efficiency when operating at the highest possible temperature, while reactivity and mass transport in fuel cells and catalysis are exponentially dependent on temperature. The limits of these operational temperatures are completely determined by the materials in use. Thus, the ability to measure mechanical properties at high temperature and correlate them to real-world performance is critical for evaluating engineering materials for improved energy efficiency.

The newly developed 800 °C heating option for Hysitron's **SEM PicoIndenter®** instruments enables high resolution *in situ* nanomechanical measurements to be performed over a broad temperature range, facilitating complete characterization of mechanical properties as a function of temperature. Multiple available test modes (indentation, compression, bend, tensile) accommodate diverse sample geometries while simultaneous SEM imaging reveals the mechanisms behind high temperature materials deformation.

## Active Tip and Sample Heating

The 800 °C stage features feedback-controlled closed-loop heating for accurate temperature, fast stabilization times (under PID control), and superior thermal stability for quantitative nanomechanical characterization at high temperatures. Sample heating is accomplished through an easily replaceable consumable resistive heater, while tip heating features an integrated resistive coil and thermocouple. An exclusive heating element architecture and proprietary probe design provides active tip heating for isothermal tip-sample contact. The test probe is designed to maximize the thermal resistance of the shaft so that heat conduction through the shaft is negligible.

The system utilizes a proprietary design constructed with a unique combination of low thermal expansion and thermally insulating materials to minimize thermal drift during testing. PID feedback and high precision resistive heating elements assure tight temperature control with fast equilibration times. Excess heat is transported to a liquid-cooled heat sink for dissipation. The coolant is held at a constant temperature, ensuring thermal stability and preventing heat transfer into other areas of the system. Upon reaching thermal equilibrium, the system exhibits total drift rates less than 0.5 nm/sec.



Resistive heating element with integrated thermocouple.

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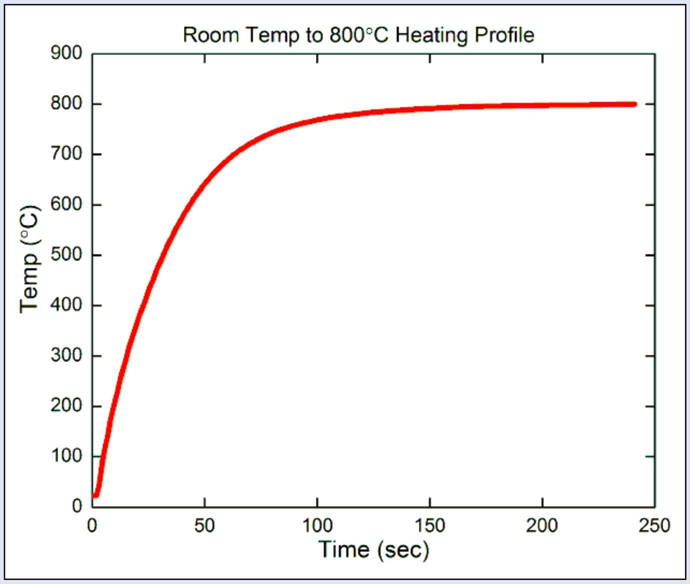


In addition, the vacuum environment of the Scanning Electron Microscope (SEM) provides the added benefit of mitigating the effects of reactive chemistries such as oxidation which are typically accelerated at high temperatures.

## System Features

The system is available in either 3-axis (X, Y, and Z) or 5-axis (X, Y, Z, tilt, and rotation) stage configurations, depending on the positioning capabilities needed. The transducer side features a variable load frame for mechanical testing that bridges the gap between nano- and micro-scale. Both the instrument and its heating architecture are designed to maintain high frame stiffness for superior mechanical testing with minimal compliance.

The system's **TriboScan**™ v.9 control software incorporates a flexible and intuitive graphical user interface for simplified test setup and execution, enhanced data analysis and plotting capabilities, and drift correction capabilities for accurate results during long test durations.

## Specifications

- Maximum Operation Temperature: > 800°C
- Drift: < 0.5 nm/sec
- Sample positioning (when used with 5-axis stage): > 90° rotation and > 20° tilt

## HIGHLIGHTS

- Quantitative, accurate, and reliable nanomechanical characterization at elevated temperatures up to 800 °C
- Ideal for applications (aerospace, automotive, nuclear, fusion, etc.) which demand materials capable of reliable performance in extreme operational environments
- Active heating of both the tip and sample provides greater stability and isothermal tip-sample contact
- A liquid-cooled heat sink maximizes thermal stability of the entire system
- Available in 3-axis (X, Y, and Z) or 5-axis (X, Y, Z, tilt, and rotation) stage configurations
- Easily replaceable resistive heaters
- Feedback-controlled closed-loop heating for accurate temperature and fast stabilization times
- Vacuum environment of SEM minimizes the effects of reactive chemistries such as oxidation
- Variable load frame for mechanical testing that bridges the gap between nano- and micro-scale
- Heater design maintains high frame stiffness for superior mechanical testing
- A variety of probe materials and geometries available

## APPLICATIONS

- Superalloys
- Thermal Barrier Coatings
- Nuclear Cladding
- Metals and Alloys
- Ceramics/Glasses