

xSol[®] High Temperature Stage

Nanomechanical Characterization at Elevated Temperatures Up to 800°C and Beyond

To develop high temperature materials capable of reliable performance in extreme operational environments, the ability to understand and tailor nanoscale mechanical properties is required. Hysitron's **xSol High Temperature Stage** enables high resolution nanomechanical measurements to be performed over a broad temperature range. The thermally stable **xSol** stage design provides superior feedback-controlled temperature accuracy, fast stabilization times (under tight PID control), and a thermally stable stage design that enables quantitative, accurate, and reliable nanomechanical characterization at elevated temperatures up to 800°C and beyond. Dual resistive heating elements eliminate temperature gradients within the sample for a uniform temperature to the outermost testing surface. **xSol**'s exclusive heating element architecture and proprietary probe design provides passive tip heating for isothermal tip-sample contact. The tip-sample-thermal equilibrium in a uniform micro-environment allows the fundamental relationships between composition, microstructure, temperature, and mechanical behavior of materials to be quantitatively characterized at the nanoscale.

Hysitron's **xSol High Temperature Stage** has been specifically designed to enhance core nanoscale characterization capabilities of **TI Series** instrumentation. The **xSol** stage can be utilized in conjunction with *in situ* SPM imaging, nanoindentation, nanoscratch, and nanowear to obtain a comprehensive knowledge of nanoscale mechanical and tribological behavior at non-ambient temperatures. It can be further extended to include precise control of humidity and cooling. Combined with **nanoDMA[®] III**, time-temperature-superposition studies of viscoelastic materials and prolonged, elevated temperature creep experiments can be accurately and reliably performed.

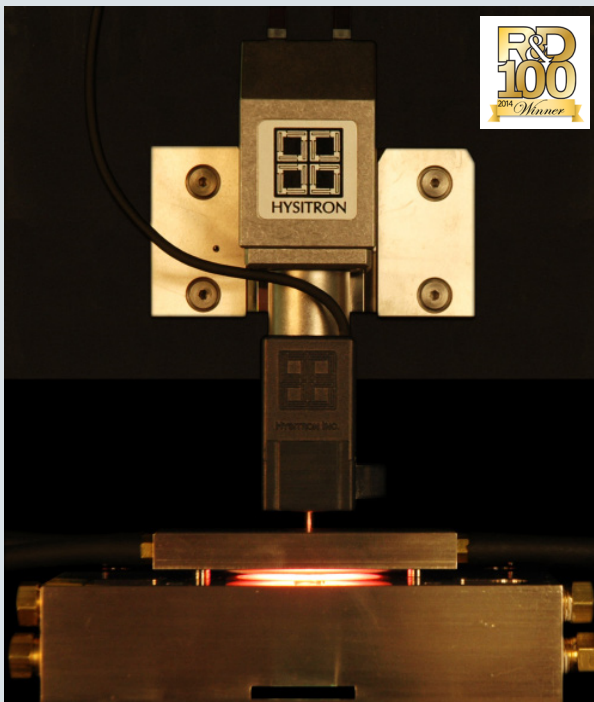


Figure 1. Hysitron's **xSol High Temperature Stage** integrated into the **TI 950 TriboIndenter[®]**.

HIGHLIGHTS

- Nanomechanical characterization capabilities up to 800°C and beyond enabled by the thermally stable stage design
- No thermal gradient within the sample due to the dual heating element design
- No need for UHV: Atmospheric control with a built-in micro-environment enables testing to be conducted in specialized gaseous environments to reduce the effects of reactive chemistries, such as oxidation
- Sub-angstrom/sec. drift rates at 800°C due to an innovative thermal expansion cancellation design and stable temperature feedback control algorithms
- Easy mechanical sample mounting between the heating elements eliminates the need for high-temperature adhesives
- High temperature SPM imaging capabilities for precise test positioning accuracy and surface topography measurements
- Compatible with Hysitron's microscale and nanoscale characterization transducers
- *In situ* drift compensation provided by the reference frequency algorithms (**nanoDMA[®] III**) provide accurate results over a broad range of temperatures and extended time durations

Testing Stability

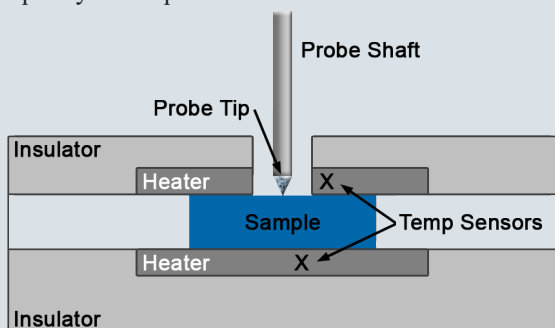
Hysitron's **xSol** stage utilizes a proprietary design constructed with a unique combination of low thermal expansion and thermally insulating materials to achieve minimal thermal drift during testing. PID feedback loops and high precision resistive heating elements assure tight temperature control with fast equilibration times. Insulating ceramics surround the heated core of the stage, creating an internal region of uniform temperature. Dissipated heat is transported outside of the instrument enclosure through the **xSol**'s liquid-cooled metal base. The coolant is held at a constant temperature, ensuring dimensional stability in the base and preventing heat from dissipating into other areas of the system, assuring ultra-precise measurements at all times.

Temperature Uniformity

To achieve accurate nanomechanical measurements as a function of temperature, precise control and knowledge of the sample surface temperature is required. The **xSol** stage incorporates a dual, independently-controlled heating element architecture that heats from the top and bottom of the sample, forming a micro-environment in the stage's interior. The test probe is designed to maximize the thermal resistance of the shaft so that heat conduction through the shaft is negligible. When the probe tip enters the heated test chamber prior to performing a measurement, thermal equilibrium between the sample surface and the probe develops within seconds. Sample temperature uniformity combined with isothermal tip-sample contact allows for quantitative, high resolution nanomechanical measurements to be performed over a broad range of temperatures.

Elevated Temperature nanoDMA[®] III

Hysitron's solution is complex, including not only hardware, but also methodology for high temperature nanomechanical testing. Hysitron's nanoscale dynamic mechanical analysis, **nanoDMA III**, used in combination with the **xSol High Temperature Stage** vastly expands the capabilities of traditional nanomechanical testing. **nanoDMA III** performed at various testing frequencies and temperatures allows time-temperature-superposition studies to be performed. Additionally, drift-insensitive elevated nanoscale creep measurements can be performed over long time durations utilizing **nanoDMA III**'s reference frequency technique.



Schematic view of the xSol High Temperature Stage.

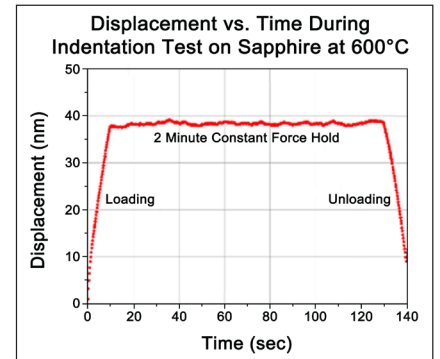


Figure 1: High thermal stability of the xSol heating system over an extended period of time.

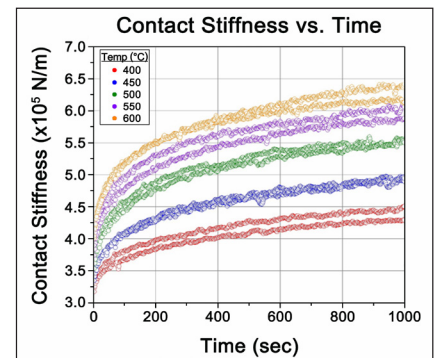


Figure 2: Creep measurements performed on Gold confirm superior stability of the system. Repeating each measurement twice demonstrates high repeatability.

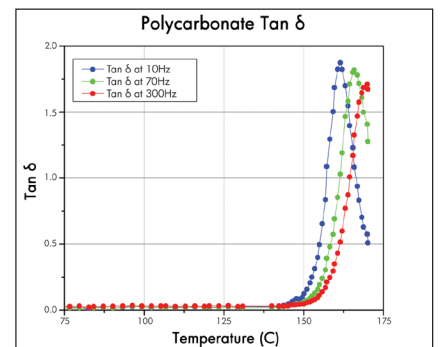


Figure 3: Polycarbonate: nanoDMA III tan delta in a function of temperature and frequency.

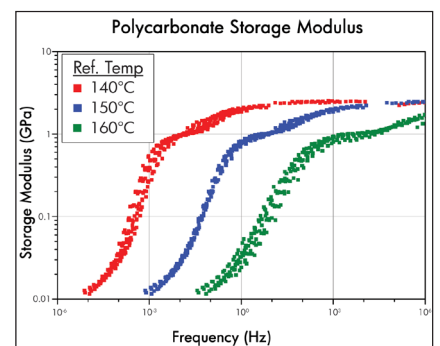


Figure 4: Polycarbonate storage modulus Time-Temperature Superposition.