

# Hybrid Techniques for In-Situ Nanomechanics

Expanding the capabilities of the SEM and TEM PicoIndenter® Instruments

## Introduction

Pairing the high sensitivity of nanomechanical testing with the high spatial resolution of electron microscopy creates a powerful tool for direct-observation mechanical characterization of nanostructures and nanomaterials. The SEM and TEM PicoIndenter nanomechanical test instruments from Hysitron deliver quantitative load- or displacement-controlled nanomechanical tests (e.g., indentation, compression, bend, tensile). In addition to these standard testing modes, Hysitron now offers a variety of hybrid techniques that expand PicoIndenter capabilities in numerous new and exciting directions.

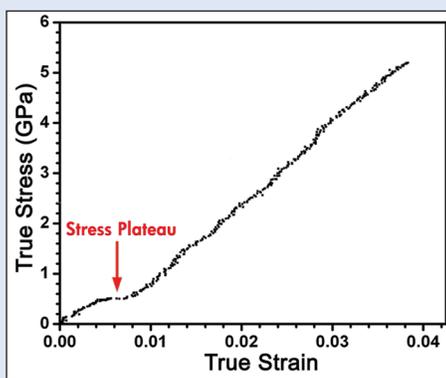


Figure 1: Stress-strain curve indicative of phase transformation in VO<sub>2</sub> nanowire during stress plateau (1).

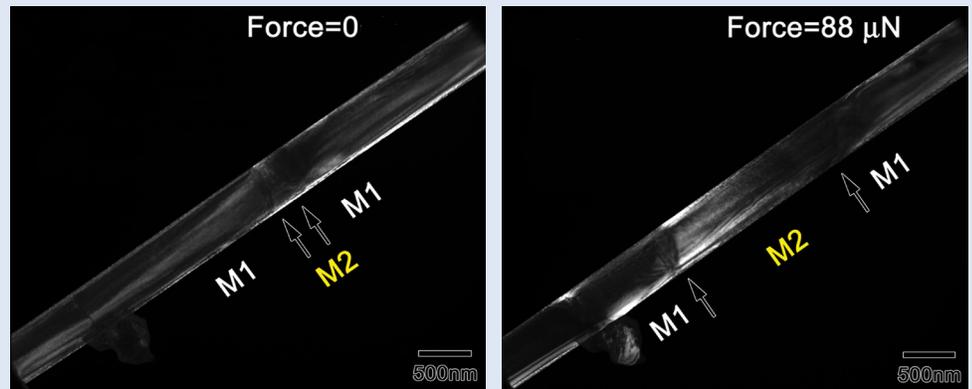


Figure 2: TEM images of VO<sub>2</sub> nanowire before and during strain-induced M1-M2 phase transformation (1). Phases have been identified by electron diffraction. Arrows point to domain walls.

## Push-to-Pull (PTP) and Electrical Push-to-Pull (E-PTP) Devices

Nanotensile testing is available on the PicoIndenter platforms through the use of a MEMS Push-to-Pull (PTP) device. Additionally, a MEMS Electrical Push-to-Pull (E-PTP) device enables tensile testing while simultaneously measuring sample resistivity using a standard four-point measurement. For both devices, true stress and strain can be determined instantaneously by monitoring and measuring specimen dimensions in the electron microscope.

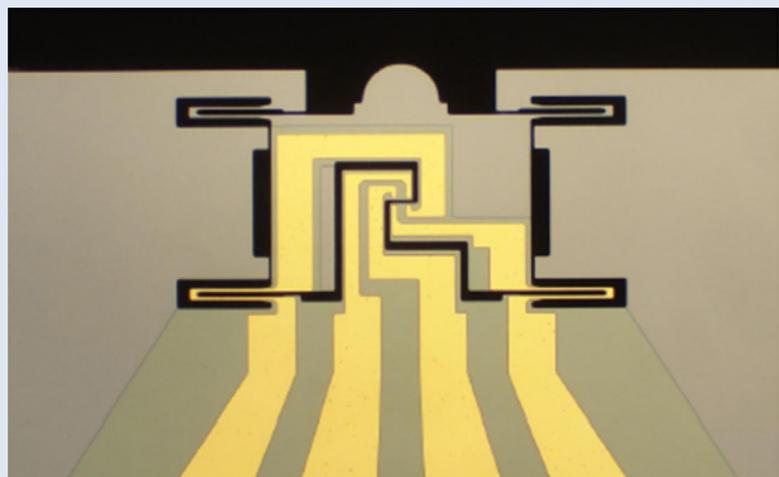
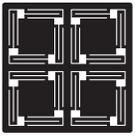


Figure 3: Optical image of Electrical Push-to-Pull (E-PTP) device.



### nanoECR® Electrical Characterization

Conductive nanoindentation involves passing a current through the indenter probe and sample interface. This technique has commonly been used to study stress-induced phase transformations, oxide fracture, MEMS contact switch resistance fatigue, nanoscale breakdown voltage, and piezoelectric material response. When performed *in-situ*, the combined electromechanical response can be characterized, while the current resulting from the electron beam of the microscope represents a constant offset which can be easily subtracted, leaving only the desired material response.

### 400°C MEMS Heater

*In-situ* heating is accomplished through the use of a resistive MEMS heater which facilitates measured temperatures up to 400°C. This activates a variety of deformation mechanisms for nanomechanical study, such as the brittle-to-ductile transition. Due to the small size of the heating element, the region of elevated temperature is highly localized which minimizes extraneous heating of system components and provides the maximum level of stability for mechanical testing. Temperature is actively measured and feedback-controlled to ensure that the desired value is achieved.

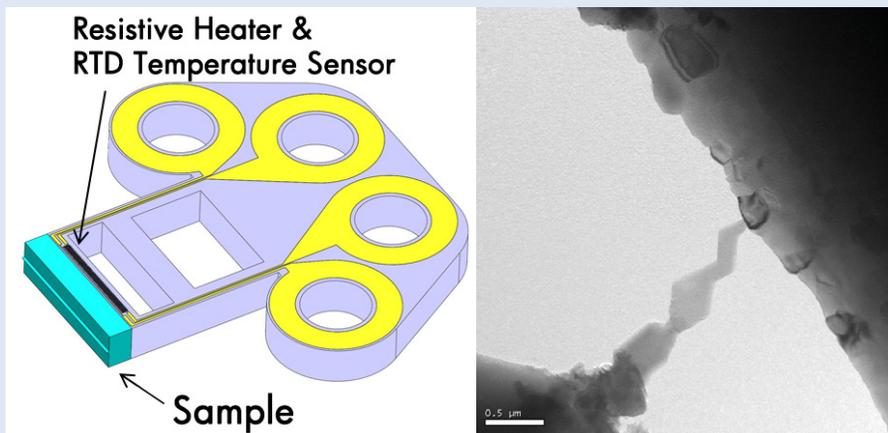


Figure 4: Schematic of MEMS heater and TEM image of mechanically assisted growth of aluminum nanowire at 400°C (2).

References:

1. Guo et al., *Nano Letters* (2011).
2. Research performed, in part, under U.S. DOE SBIR Grant No. DE-FG02-07ER84812 to Hysitron.
3. D.D. Stauffer, Ph.D. Thesis, "Deformation Mechanisms in Nanoscale Brittle Materials," University of Minnesota (2011) pp. 150-152.

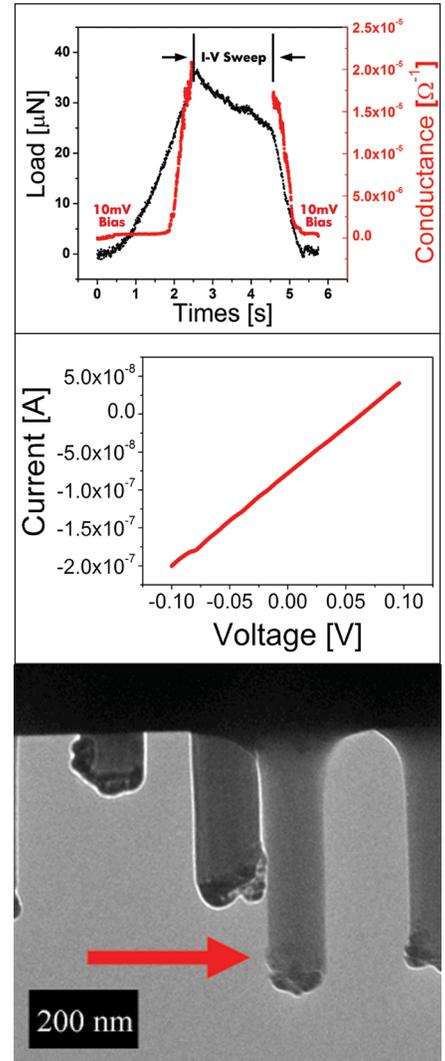


Figure 5: Electrical characterization during compression of n-doped VLS-grown silicon nanopillar (3).

### Conclusions

Complementary techniques are currently being developed to broaden the scope of nanomechanical testing that can be achieved in an electron microscope environment. These upgrade techniques continue to expand the range of testing options available on the SEM and TEM PicoIndenter platforms.