

TEM Mechanical Testing in Liquid with Temperature Control

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Quantitative mechanical testing in the transmission electron microscope (TEM) has been commercialized for world-wide use with the Hysitron PI-95 stage [1], though there exist limitations in mechanical testing under varied environments. The combination of this holder with the Environmental TEM can provide mechanical quantification of nanowire and thin-film specimens under gaseous environments, but currently there is no TEM stage that has this level of control within a liquid environment [2]. Therefore, the Center for Integrated Nanotechnologies (CINT) at Sandia National Laboratories has developed a novel stage, the Mechanical-Environmental-Thermal (MET) TEM Discovery Platform to test materials under a hermetically-sealed liquid or gaseous environment at atmospheric pressure with elevated temperature control and displacement-sensing tensile testing.

This MET-TEM platform is a two-chip cell that allows for customization of the device for sample integration and for electrochemical experiments [Figure 1]. The sample is mounted in the scanning electron microscope/focused ion beam instrument onto the base platform, where the sample spans between two posts that position the thin sample 100 nm above the lower SiN membrane window. One of the posts is connected to a buried piston arm that is controlled by a thermal actuator. Surrounding the elongated SiN membrane window, varied sized heating elements have been integrated to control temperature from RT to 100°C.

Demonstration of this platform and details on its temperature and tensile control specifications will be presented. This instrumentation is available to the international research community, free of charge, through the CINT user program (cint.lanl.gov).

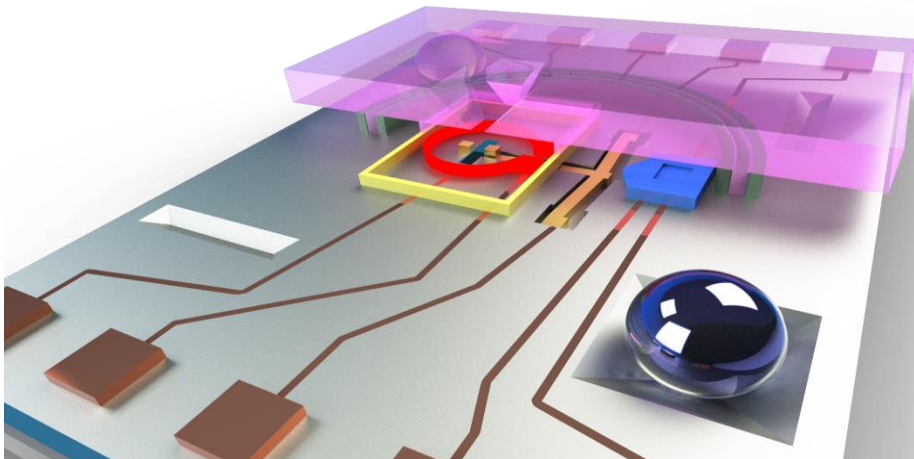


Figure 1. Mechanical-Environmental-Thermal (MET) transmission electron microscopy (TEM) Discovery Platform for quantitative mechanical testing of materials within a temperature controlled liquid environment inside the TEM. The base chip (colored in grey), with electrical leads (copper) buried to control/sense the electrodes (near window), heater (red), actuator and capacitive sensor (blue). The SiN membrane window is in the center of the

base chip with two posts (orange) on either side of the window to support a mounted electron transparent sample. An inner seal ring (yellow) segregates the liquid cavity within the specimen region from the air cavity where the actuator operates. An outer seal ring (green), sealed with epoxy, segregates the actuator area from the high-vacuum in the TEM. Surface etched regions serve to align the windows on the lid and base chips, using optical fibers and/or sapphire beads. The lid (pink) has three ports, a central port that contains the top SiN membrane window and two outer ports for liquid filling after the lid is affixed to the base.

1. C. Chisholm *et al.* (2012) *Acta Materialia* **60**, 2258-2264.
2. F.M. Ross (2015) *Science* **350**, aaa9886.

This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525.