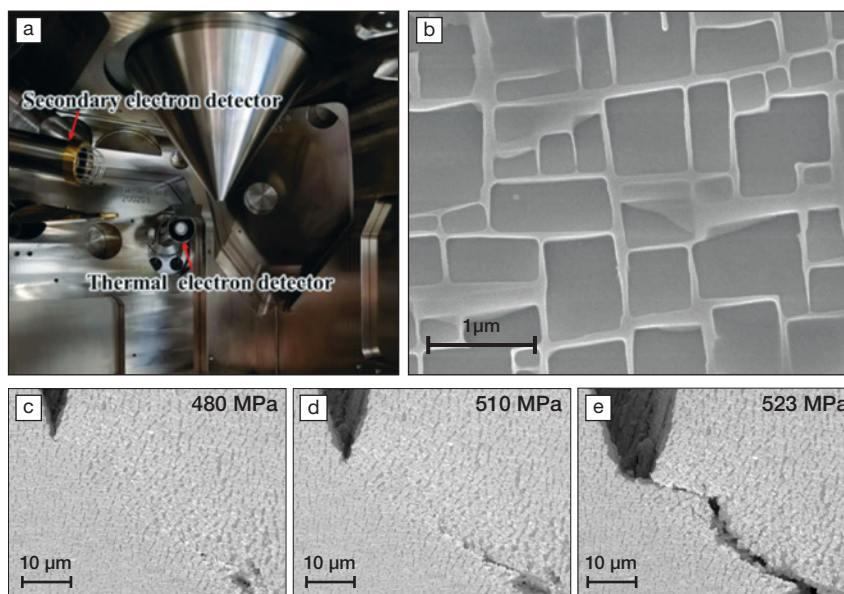


In situ mechanical testing in an SEM performed at 1150°C with submicron resolution

Metals and ceramics are widely used in high-temperature and other extreme environments; for example, boron carbides are used in nuclear reactors and nickel superalloys in turbine blades. For these applications, the materials need not only to withstand high temperatures but also to perform while retaining their mechanical properties. Although mechanical testing can be done at high temperatures, so far this has only been achieved on macroscopic specimens where the failure mechanisms are investigated post-mortem. To get nanoscopic and microscopic details, compression, tensile, or crack propagation tests can also be conducted inside a scanning electron microscope (SEM). However, simultaneously heating, deforming, and observing a sample within an SEM is challenging, where the thermal electrons emitted by a hot sample and the heating wire interact with the imaging electron beam and blur the images.

To retain the high resolutions in an SEM during heating, the research teams of Yuefei Zhang and Ze Zhang from Beijing University of Technology and Zhejiang University, China, developed a special *in situ* tensile setup that heats up to 1150°C while retaining force and displacement resolutions of 1 N and 0.08 μm, respectively. This development is detailed in an article published earlier this year in the *Review of Scientific Instruments* (doi:10.1063/1.5142807).

What makes the setup special is a multilayer tantalum metal shield that blocks the thermal radiation from the heated stage, as well as a procedure to suppress the interference between the thermal and the imaging electrons. Typically, a bias control is applied to the thermal shield plate using feedback from a thermal electron detector placed close to the secondary



(a) View of the interior of the scanning electron microscope (SEM) indicating the secondary thermal electron detectors. Credit: Yuefei Zhang. (b) Sub-micrometric resolution micrograph of the surface of a nickel superalloy at 1150°C (credit: AIP Publishing). (c–e) Snapshots of a notched superalloy sample deformed at 1150°C showing the crack propagation features under increasing stress. Credit: AIP Publishing.

electron detector of the SEM (Figure a). The local heating of the sample is done using a tungsten filament, whereas the setup is cooled using water.

After calibration procedures to correct for thermal expansion of the device itself, control tests were done using standard specimens made of carbon steel. The yield strength measured, 1129 MPa, showed an error of less than 2% compared to the reference value.

Finally, the setup was used to stretch at 1150°C a single-crystal Ni superalloy specimen with a notch on its side. The crack propagation and surface features were clearly revealed during the experiment (Figure b–e). The device was used to study the oxidation of nickel superalloys under thermo-stressed conditions in an SEM, and the results were published earlier this year in *Scientific Reports* (doi:10.1038/s41598-020-59968-3).

But the testing is not limited to metallic alloys. “Of course, it can be

used for nonmetallic samples too,” says Zhang. “When we observe ceramic samples at high-temperature conditions, the yield of secondary electron emission increases. This improves the image quality without the need for a conductive coating.”

The capabilities offered are exciting for many. “The developed instrument paves the way for exploring new mechanisms, which could serve as guidelines for designing ultra-tough ceramic nanocomposites for demanding environments,” says Christos Athanasiou from Brown University, who is investigating such materials but was not part of the study. “For example, silicon carbide ceramics reinforced with 2D-hexagonal boron nitride fail at the interface between matrix and reinforcement. This occurs during loading and crack bridging, but no one has looked at the failure mechanism at high temperatures.”

Hortense Le Ferrand

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