

High Temperature, High Throughput Nanoindentation

Quantitative Mechanical Property Mapping and Statistics at Elevated Temperatures

Introduction

Understanding the intrinsic mechanical properties of microstructural features within materials that operate in extreme environments is necessary for developing improved performance. Hysitron's xSol® High Temperature Stage in conjunction with the XPM™ accelerated property mapping technique enables a fast and reliable method to measure mechanical properties at elevated temperatures.

XPM is a technique for performing a large quantity of quantitative nanoindentation measurements in a short period of time, up to six per second. Indents are performed in a grid pattern to quickly gather property distribution statistics and generate spatial distribution maps of localized mechanical properties. The speed of XPM alleviates several critical complications in high temperature nanoindentation, such as chemical reactions, which can dull the probe and degrade samples. XPM enables 500x faster testing than traditional nanoindentation routines, minimizing the effect of potential chemical reactions and maximizing measurement reliability. Additionally, the impact of thermal drift becomes negligible during the ultra-fast testing cycles provided by XPM.

Procedure

A Hysitron TribolIndenter® equipped with a xSol high temperature stage were used in combination with XPM to characterize the mechanical properties of a silicon wafer and a silicon carbide fiber-matrix composite at 400°C and 800°C. A diamond Berkovich probe was used and each sample was kept in a 95% argon, 5% hydrogen atmosphere throughout testing. A tip area function was performed on fused quartz before and after testing; no measurable tip degradation was observed.

Silicon: A 10 x 10 grid of indents was performed at each temperature with a 7mN peak load and 5 μm indent spacing. Each grid of 100 indents took 30 seconds to complete.

Silicon Carbide: A 20 x 20 grid of indents was performed at each temperature with a 4mN peak load and 750 nm indent spacing. Each grid of 400 indents took 100 seconds to complete. The grid location was chosen such that both the matrix and fibers were indented during a single test.

A combination of optical imaging and scanning probe microscopy (SPM) was used for precise positioning of the grid as shown in Figure 1.

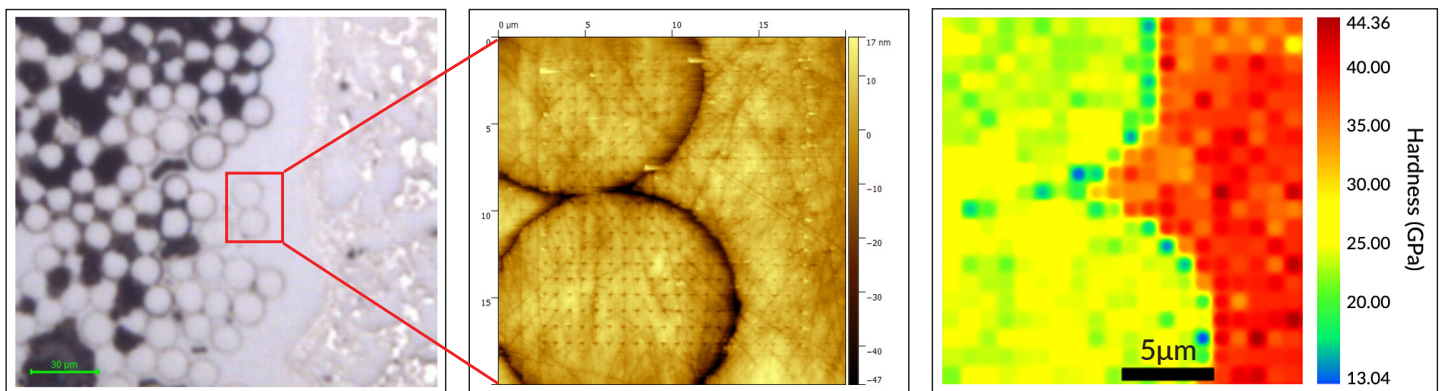


Figure 1. Optical image of 400°C testing location (left) used to find a suitable testing location. SPM image post XPM testing (center) where the circular regions are SiC fibers embedded within the SiC matrix. 400 indents were performed in both the fiber and matrix material. The resulting property map of hardness (right) shows a higher hardness in the matrix material and edge effects around the peripheral of the fibers.

Results

The measured elastic modulus and hardness of silicon at 400°C and 800°C are shown in Table 1. The hardness of silicon at 400°C is close to that of room temperature, but decreased dramatically at 800°C. An in-situ SPM image of silicon taken at 800°C after an XPM test is shown in Figure 2.

Sample	Temperature	Elastic Modulus (GPa)	Hardness (GPa)
Silicon	400°C	168.9 ± 2.9	10.67 ± 0.24
	800°C	163.7 ± 8.6	2.350 ± 0.035
SiC Fiber	400°C	278.4 ± 11.5	24.38 ± 1.27
	800°C	259.6 ± 8.5	17.53 ± 0.25
SiC Matrix	400°C	466.1 ± 27.3	38.08 ± 2.63
	800°C	383.0 ± 27.6	22.74 ± 2.71

Histograms of the reduced modulus and hardness of the SiC fiber-SiC matrix composite are shown in Figure 3. The matrix was observed to have a higher hardness and modulus at both temperatures. However, the hardness of the matrix decreased by a much greater magnitude with increasing temperature. At 800°C the hardness of the fiber and matrix are almost indistinguishable. The hardness and elastic modulus were obtained by fitting a double Gaussian to the histogram results. Adjusted R squared values ranged between 0.91 and 0.98. The resulting values are shown in Table 1.

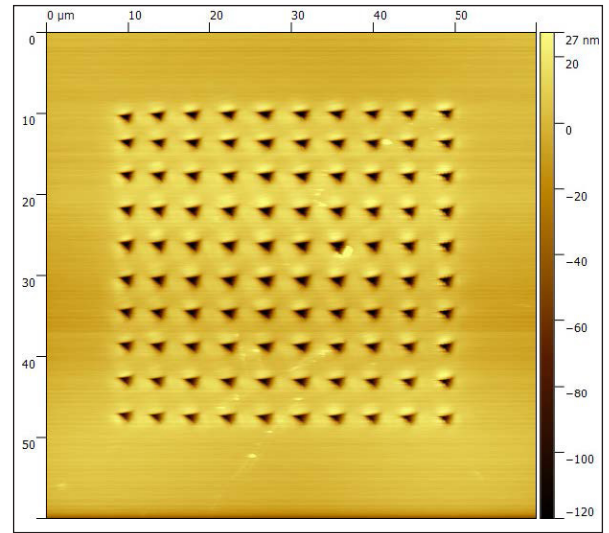


Figure 2. SPM image of silicon taken at 800°C following XPM testing.

Conclusions

The hardness of silicon decreases dramatically at high temperature as the material transitions from a brittle to ductile material. For SiC, the properties of the fiber and matrix change at different rates, with the fiber retaining more of its mechanical strength at increasing temperatures. Hysitron's xSol heating stage in combination with XPM allows for rapid determination of mechanical properties, allowing localized material properties to be thoroughly characterized and tuned for their intended operating temperature.

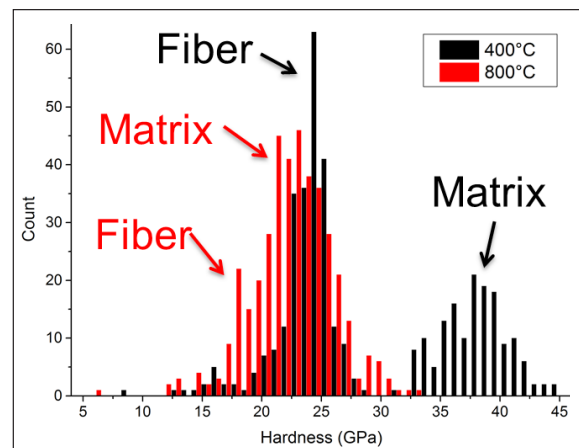
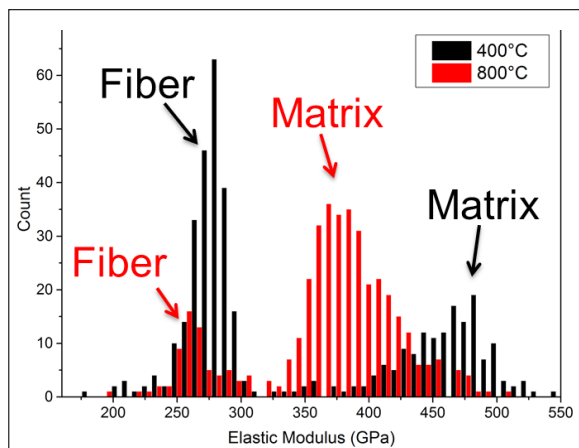


Figure 3. Histograms of silicon carbide fiber-matrix composite hardness and elastic modulus results obtained from XPM indentation testing at 400°C and 800°C. Fiber properties remain relatively constant over the temperature range, while the modulus and hardness decrease by approximately 18% and 40%, respectively.

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