Application Note

KLA INSTRUMENTS

Nanoindentation of Physical Vapor Deposition Hard Coatings at Elevated Temperatures

Introduction

Grown by physical vapor deposition (PVD), hard coatings are typically applied as a protective covering for engineering components. They are designed for improving the lubricity and hardness of parts, improving wear resistance, reducing friction, improving appearance, and achieving other performance enhancements[1]. A zirconium nitride (ZrN) coating provides a hard ceramic-like refractory material that has a high heat resistance. The evolution of the ZrN hard coating material system is motivated by continuous demands from the cutting tool, automotive, aerospace and hard disk industries. Typically, failures of these hard coating components include decrease of wear resistance, corrosion of the base materials, and cracking in the coating that propagates to and/or beyond the interface between coating and substrate[2]. Hardness measured by nanoindentation is a good predictor of wear resistance for hard coatings, based on the generic proportionality between these two terms. Nanoindentation is also the fastest and most straightforward method for quantifying wear resistance. The aim of this research is to extend the knowledge on how temperature variation influences the nanomechanical properties of ZrN-based nanostructured coatings.

Experimental Method

Zirconium nitride coatings on steel substrates were purchased from an advanced coating service. The measured nano-hardness of the white-gold ZrN coating measured 22GPa, the average thickness of the coating ranged from 1-4µm, and its maximum usage temperature is around 550°C. The ZrN sample came in the form of a disk, and all testing was conducted using a KLA InSEM® HT nanoindenter system. The InSEM HT was used with the CSM option, and an InForce 50 actuator was fitted with a Berkovich indenter to perform room temperature nanoindentation. For indentation testing under elevated temperature, the NanoBlitz 3D technique using a tungsten carbide (WC) Berkovich tip was used to analyze 900 indents per temperature. NanoBlitz 3D is a high-speed mapping technique, where each indent typically takes less than one second, allowing for increased statistical information across the material surface. After performing an area function calibration on fused silica samples and correcting for load-frame compliance, the hardness and elastic modulus for every indent was calculated using the standard Oliver-Pharr method.

Results and Discussion

A typical nanoindentation load-displacement curve of a ZrN coating at room temperature by constant strain rate method is shown in Figure 1. The hardness was calculated by the Oliver-Pharr method to be 23GPa.

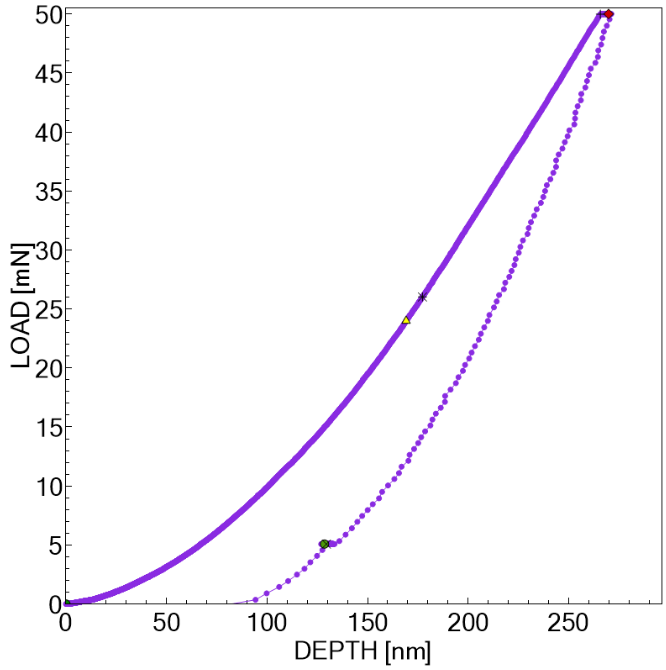

Figure 1. Typical nanoindentation load-depth curve of a ZrN coating at room temperature.

A NanoBlitz 3D test was performed on the same sample at room temperature using diamond and tungsten carbide (WC) Berkovich tips. For the diamond Berkovich tip, the ZrN coating hardness histogram in Figure 2a shows the peak of the distribution in the 20-26GPa range, centered at 23GPa. Similarly for the WC Berkovich tip, shown in Figure 2b, the peak of the distribution is in the 19-26GPa range, centered at 22GPa. These results indicate that the NanoBlitz 3D measurement data agrees with the constant strain rate indentation data.

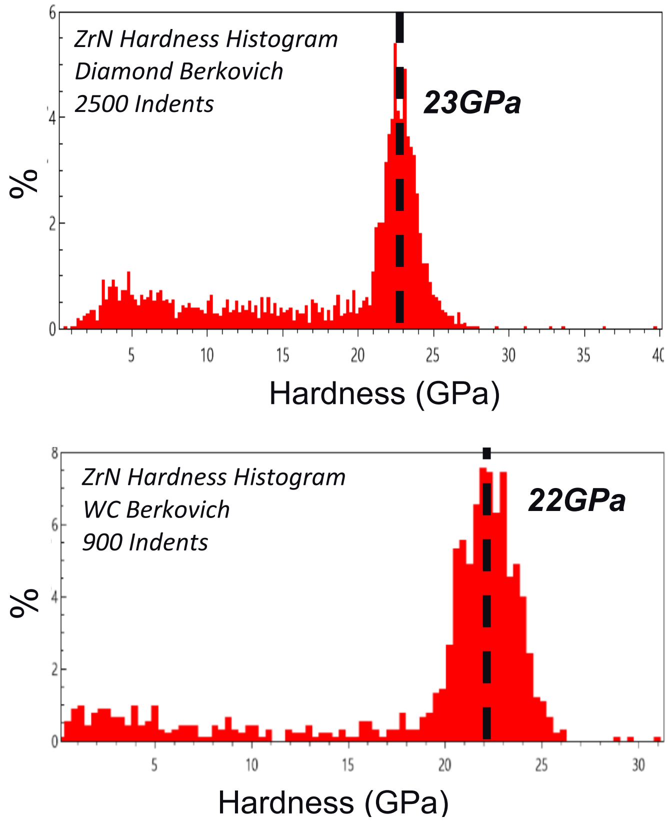

Figure 2. Nanoindentation hardness histogram for ZrN coating at room temperature, using (a) diamond Berkovich tip, and (b) WC Berkovich tip.

The KLA InSEM HT mechanical properties microprobe system combines robust performance with simplicity of operation, service and installation. It utilizes the InForce 50 actuator for testing samples up to 800°C and includes a sample mount for isothermal heating of both tip and sample to 800°C. Figure 3 (top) shows the InSEM HT system with fused silica clip mounted on the sample stage. In this work, we used a tungsten carbide (WC) indentation tip for measuring the properties of both elastic modulus and hardness on ZrN coatings. The WC tip enables indentation at operational temperatures up to 800°C.

The hardness and elastic modulus maps of ZrN at elevated temperature are shown in Figure 4. Each map contains 900 data points collected at 400°C and 660°C, with each pixel in the image representing one indentation measurement result. The color bars represent hardness and Young's modulus in GPa.

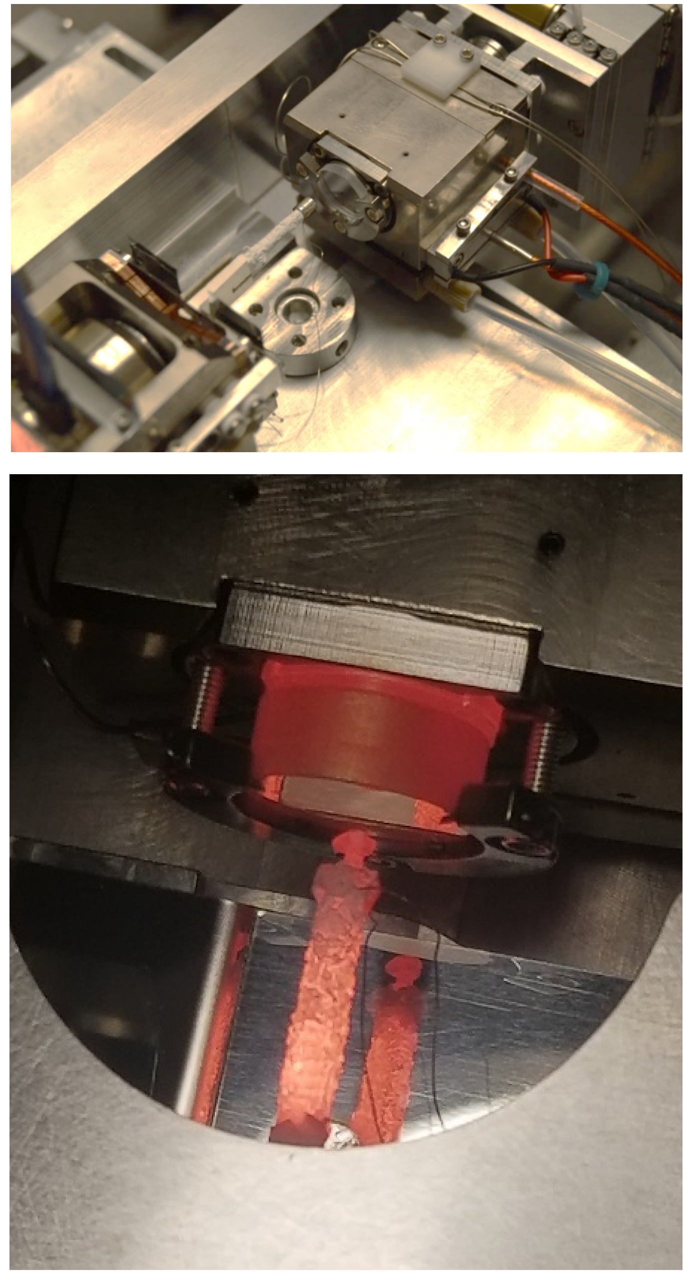

Figure 3. The KLA InSEM HT system (top), with the PVD coating sample mounted on the InSEM HT sample stage heated to a high temperature (bottom).

Surface roughness is an important coating characteristic that influences coating performance. For example, roughness can influence the friction level and material pick-up behavior of the cutting tool[3]. Many factors can affect the surface roughness, both during the coating processes, as well as post-coating treatment. The NanoBlitz 3D mapping data in Figure 4 show significant scatter due to the surface roughness, which

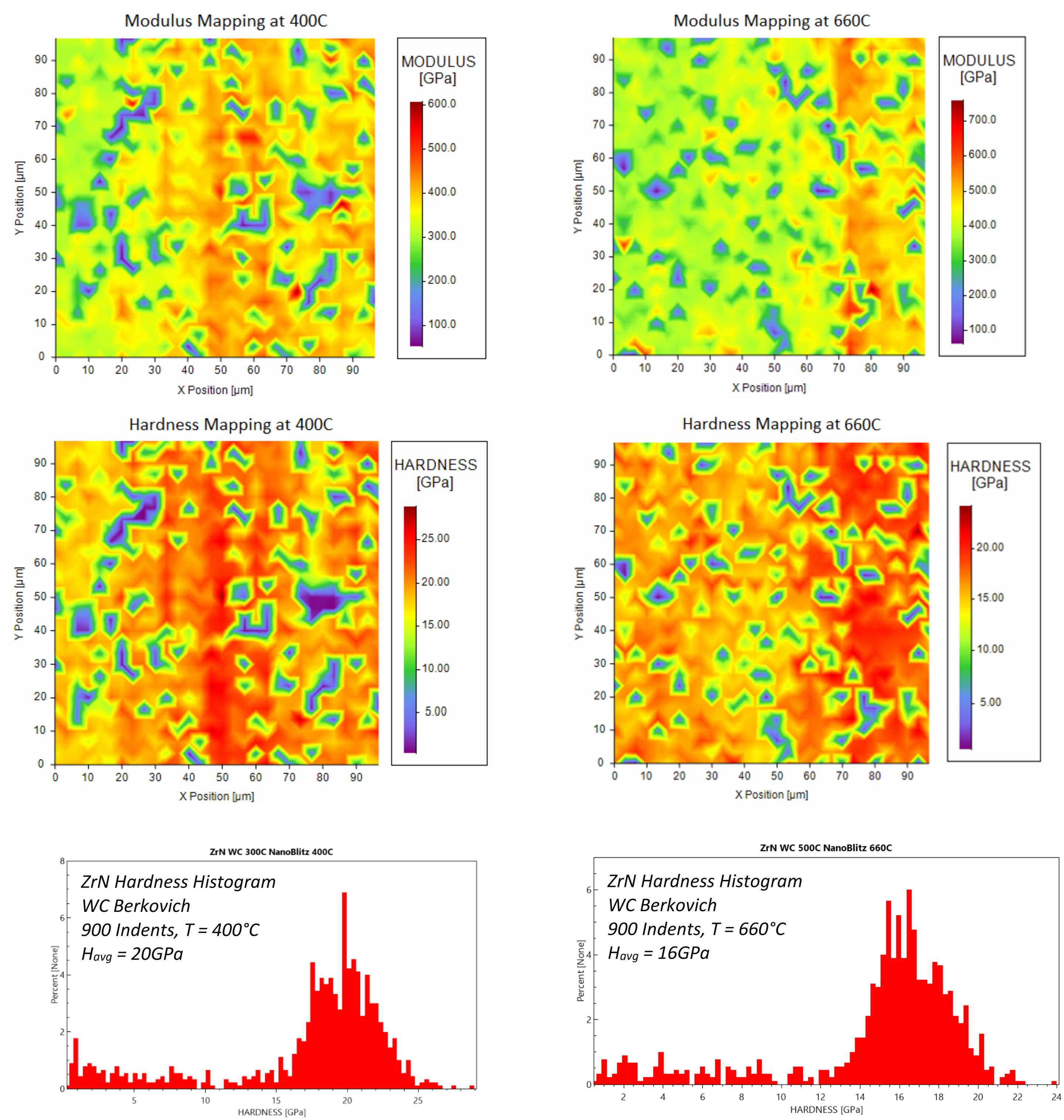

Figure 4. ZrN coating nanomechanical property maps and corresponding histograms at elevated temperatures: modulus mapping at 400°C and 660°C (top); (b) hardness mapping at 400°C and 660°C (middle); and hardness histograms at 400°C and 660°C (bottom).

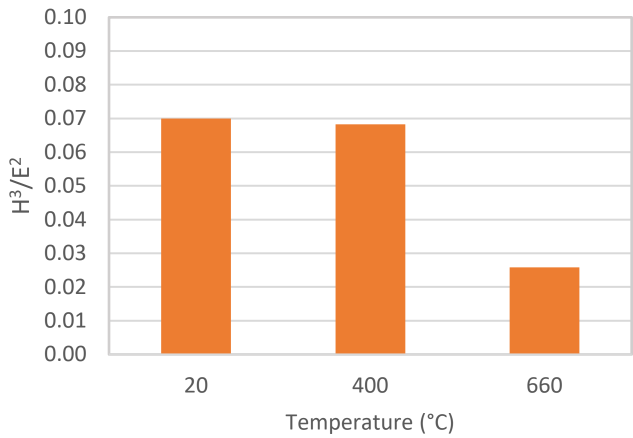

Figure 5. H^3/E^2 ratio of ZrN coating as a function of elevated temperature.

are on the order of the size of the indentation tests. Compared to the data at room temperature, the average hardness of the ZrN hard coating drops from 22GPa at 20°C to 20GPa at 400°C and 16GPa at 660°C. The 400°C and 660°C hardness histograms follow a skewed left normal distribution, with the distributions' highest peaks at 20GPa and 16GPa, respectively. Used with the InSEM HT, the NanoBlitz 3D technique provides high-throughput data and meaningful statistics in a short period of time.

The ratio of H^3/E^2 is considered a key parameter in evaluating the wear resistance of coatings[4]. In this case, Figure 5 shows the H^3/E^2 ratio as a function of temperature, where H^3/E^2 ratio shows a significant decrease at 660°C, suggesting a marked decrease in wear resistance at very high temperature.

Conclusions

The combination of NanoBlitz 3D nanoindentation mapping and the InSEM HT enable investigation of the nanomechanical properties of PVD hard coatings at elevated temperature. The KLA InSEM HT nanoindenter is the industry choice for these measurements because of its high precision, speed, ease-of-use and fast mapping techniques. Nanoindentation measurements at elevated temperature allows for a better understanding of the relationship between the nanomechanical properties and the PVD hard coating structures, which facilitates process optimization for the hard coating industry.

References

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