

Keysight Technologies

Nanotribology of Hard Thin Film Coatings: A Case Study Using the G200 Nanoindenter

Application Note

Introduction

Thin film coatings are extensively used in various applications, such as microelectronics, data-storage media, surgical tools, biomedical prosthetic components, machining tools, turbine blades etc. Along with some functional improvements, these thin coatings also significantly enhance the mechanical reliability of the components. Hard thin films provide the resistance to mechanical deformation that the components need during their application. With the advancements in nanomaterials research, new thin film systems — often only a few 100's of nm — are being developed to improve the performance of thin coatings in all of these technologically important areas. Hence, it is becoming more and more important to design new test methods that addresses the relevant deformation mechanisms in thin films, which are not suited to be characterized by the conventional test methods and standards.

Nanoindentation has been a method of choice to characterize mechanical properties of small volumes of materials, such as thin films. This is especially true since an analytical method has been proposed to measure the properties of the films without the substrate effect. Besides measuring the elastic modulus and hardness of thin films, it is also important in many applications to understand the deformation behavior of the film due to lateral movement of sharp contacts, which is referred to as nanotribology. Most thin film hard coatings should possess two important characteristics, low coefficient of friction and a high strength adhesive bonding with the underlying substrate. In some applications a low wear rate under cyclic scratching may also be important, but it was not a critical concern for the thin film with extremely high hardness, discussed herein. Nanoindentation with lateral force measurement capability is an ideal tool to address the tribological characteristics of such thin film materials at nano length scales.

Before we discuss the tribological properties of a thin hard coating, it is important to differentiate the measurements using a nanoindenter with those performed at micro/macro-scale. Tribology being a subject of interest for long time, there are quite a few international standards for tribological measurements, some of which also include measurement on thin coatings. However, according to all the standards, the measurements are performed with a probe of much larger length scale (usually 100's of microns) compared to the coating material. Although the larger probes enable an optical investigation of the scratched region, the forces required for scratching a hard coating often surpasses any realistic values of forces these coatings encounter during their operation. The measurements with the larger probes are also affected by significant deformation of the substrate material. On the other hand, a sharper contact can damage the thin film coating at much smaller force and should be used to characterize

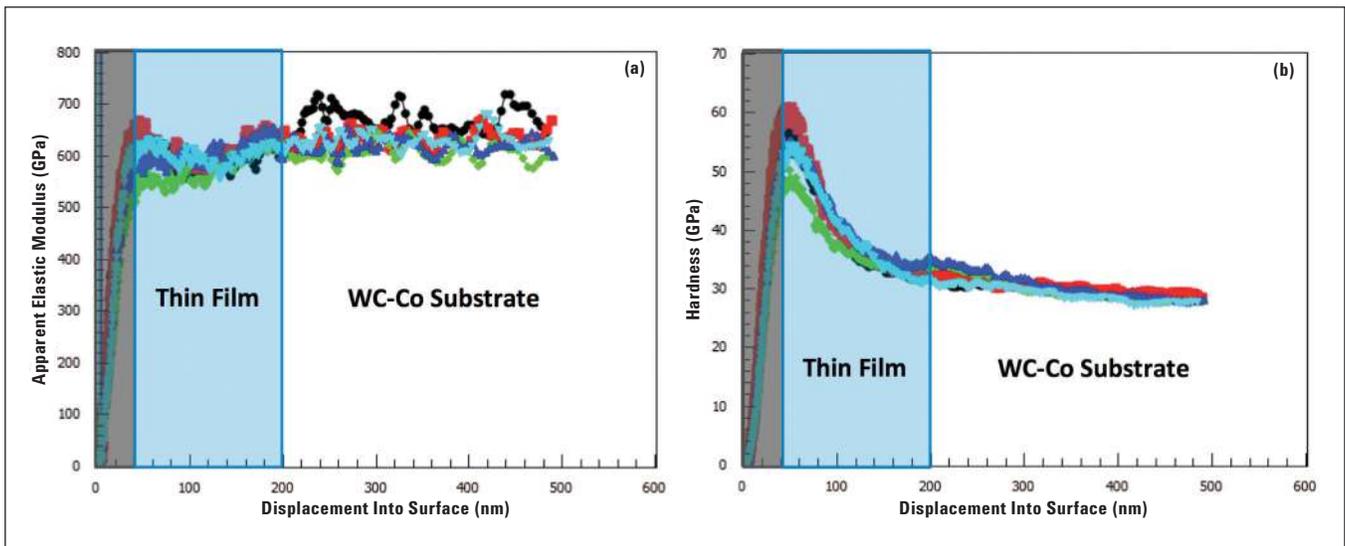


Figure 1. Continuous stiffness measurement of (a) elastic modulus and (b) hardness for thin film coating on WC-Co substrate. Note the thin film and substrate have similar elastic modulus, but the thin film is much harder than the substrate. Measured properties in the greyed region (below 50 nm) are artifacts of the tip shape.

critical parameters. It also enables us to understand the deformation of the thin film from a more fundamental length scale. There is another, probably a more important, reason to move towards smaller dimensions for tribological measurements of thin films. Larger contact probes often contains sharp asperities on the surface. So, even if it requires a larger force to observe significant deformation, the coating may start to get critically deformed at those asperities at much smaller forces. However, reducing the size of tribological probes to smaller size also requires force and displacement measurements at smaller length scales. It is important to note here that international standards are important and they provide a basis for comparison. But, with the advent of novel nanomaterials the coatings on various components are becoming thinner (often only 10's or 100's of nm), and the existing standards may not be adequate to measure the critical parameters for these thin films. Hence, a nanoindenter, where the force and displacement are calibrated in compliance with international standards is more suitable for addressing this need for the future thin hard coatings applications. Note the G200 nanoindenter is compliant with ISO 14577 standards for measurement of normal force and displacement.

Experimental

The sample that has been used during this study came from a company, producing high-precision carbide tools. It is a hard thin film coating of about 200 nm thickness on a WC-Co substrate. As mentioned above, the tribological properties of this thin film include determination of two key parameters – coefficient of friction and critical adhesion force. To characterize these parameters a Nanoindenter G200 with lateral force capability was employed.

Before the scratch tests, the elastic modulus and hardness of the thin film coating was characterized using a diamond Berkovich indenter. Indents were made with 500 nm penetration depth, and the change in modulus and hardness were measured using continuous stiffness measurement as a function of penetration into the surface. Details of continuous stiffness measurement (<http://literature.cdn.keysight.com/litweb/pdf/5990-4183EN.pdf>) can be found elsewhere [Hay et al., *Experimental Techniques* 34, 86–94 (2010)].

The nano-scratch tests were performed using a diamond cono-spherical tip with 90° included angle and 2 μm tip

radius. The reason behind selecting a cono-spherical tip was mainly two-fold. First, the geometry is symmetric about the cone axis, and hence the measurements are independent of scratch direction. Second, the spherical part of the tip provides measureable elastic to plastic transition regime that is useful to measure the coefficient of friction more accurately (discussed in the next section). It is also important to note here that most of the established standards in the tribology community are based on similar geometry of probes, and it should be easier to scale-down the standards for nano-scratch tests.

Nanoindents with increasing maximum force were first made to measure the range of normal force for elastic and plastic deformation of the thin film. Single-pass constant load scratch tests were performed at 5, 10, 20 and 50 mN. Scratches of 100 μm lengths were made with speeds of 5, 10 and 25 μm/s. To determine the critical adhesion force between the thin film and substrate, ramp load scratch tests were carried out at a speed of 10 μm/s, where the normal force was ramped from 0 to 100 mN over a length of 150 μm. The measured friction force (or, lateral force) in all the tests were normalized by the applied normal force for comparison.

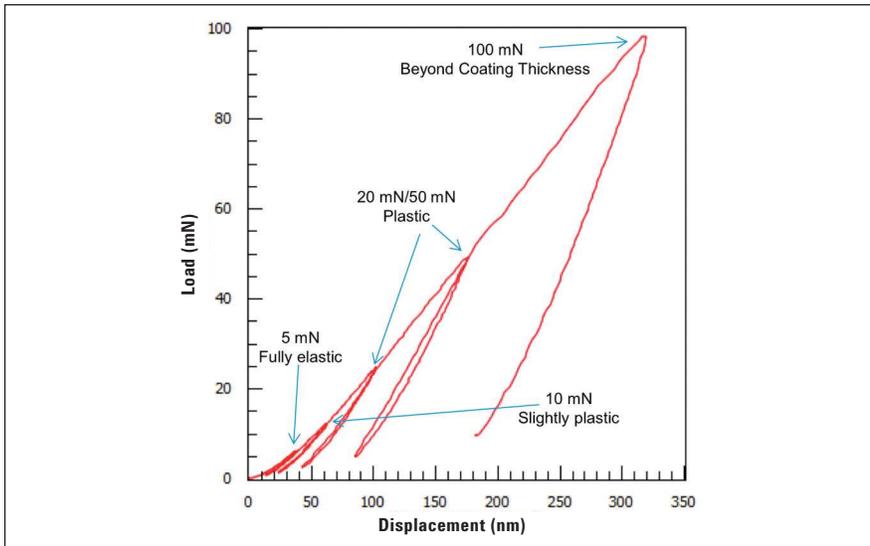


Figure 2. Nanoindentation response of the thin film coating on carbide substrate when indented with the cono-spherical diamond tip used for nanotribology experiments.

The scratches were investigated with the optical microscope installed in the nanoindenter as well as a stand-alone Nikon optical microscope. To obtain detailed surface features, the scratched regions were scanned in the nanoindenter using a diamond Berkovich tip, DCM transducer and the nanopositioning stage.

Results and Discussion

The apparent elastic modulus and hardness as a function of displacement into surface are plotted in Figure 1. The term ‘apparent’ is used for elastic modulus because the substrate effect could not be subtracted from the film response for lack of information on substrate properties (<http://literature.cdn.keysight.com/litweb/pdf/5990-9003EN.pdf>). However, from Figure 1a, it is evident that the modulus value does not change significantly when the indenter moves from the film into the substrate. The modulus value of ~ 600 GPa is close to the nominal value of WC, and hence, it is reasonable to infer that the thin film has similar elastic modulus as the substrate. In many thin coating applications it is an important criterion for material design to ensure better mechanical and thermal stability.

On the other hand, the hardness is considerably high for the film (Figure 1b) and it gradually decreases to a constant value inside the substrate, which is again close to the nominal value of WC. The large difference in hardness between the film and substrate most likely caused the gradual decrease observed in the thin film region in Figure 1b.

As discussed above, the nano-scratch tests were performed using the cono-spherical tip. However, before the scratch tests it is important to know the loads where the coating behaves elastically. The load vs. displacement response from the nanoindentation using the cono-spherical tip is shown in Figure 2. It is clear that the nanoindentation response is almost completely elastic at about 5 mN load, and the deformation shows significant plastic deformation beyond 10 mN load.

In Figure 3, the displacement into the surface and the normalized friction force during the constant-load scratch tests at 10 $\mu\text{m/s}$ are plotted. As expected, the displacement into the surface increases with increasing normal force (Figure 3a) from 5 mN to 50 mN. There is a slight change in corresponding normalized friction force for 5, 10 and 20 mN (Figure 3b). However, there is a significant change in both displacement and friction force for 50 mN normal force. During the initial part of the scratch at 50 mN, the thin film is still adhered to the substrate. After about 20 μm , the film starts to chip off or delaminate from the substrate causing a sharp increase in displacement and friction force. This behavior shows that 50 mN of normal force is critical for adhesive failure of

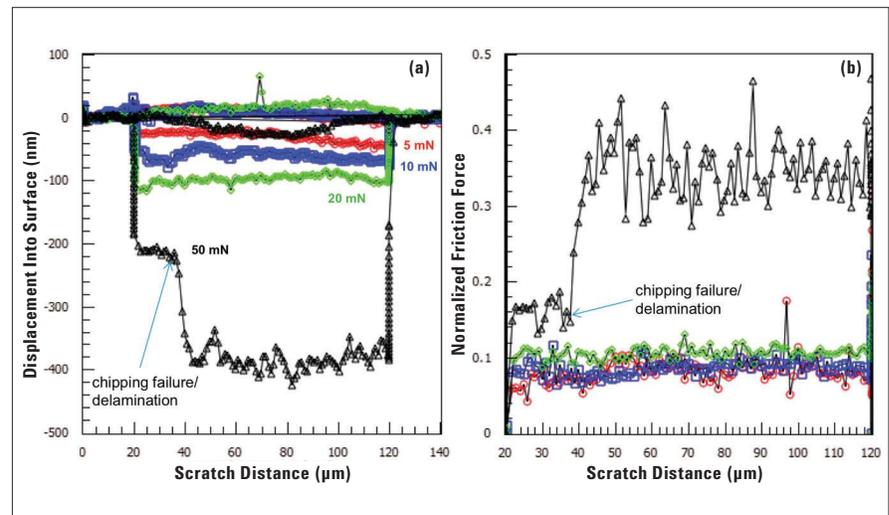


Figure 3. (a) Displacement into surface, and (b) normalized friction force along the scratch distance during constant load nano-scratch tests at different applied normal forces (5, 10, 20 and 50 mN) at a speed of 10 $\mu\text{m/s}$.

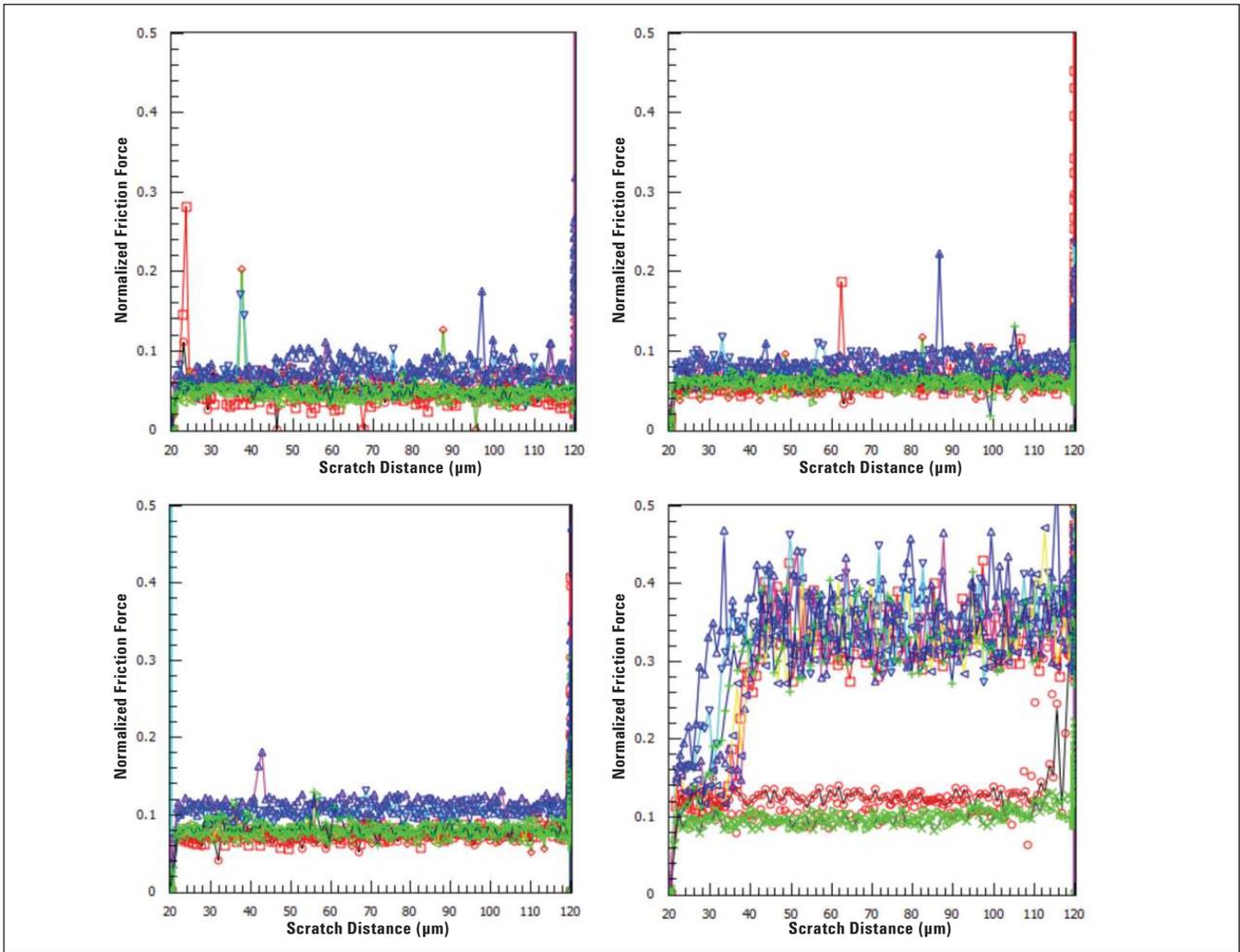


Figure 4. Normalized friction force during constant-load nano-scratch tests as a function of different normal force – 5 to 50mN – and scratch speed – 5 to 25 $\mu\text{m/s}$. Note the increasing trend in normalized friction force with increasing normal force.

this thin film coating under the current test conditions.

The coefficient of friction is defined as the ratio of friction force and normal force. So, from definition, it is same as the normalized friction force defined above. However, from Figure 4, it becomes clear that the normalized friction force depends on the normal force and to some extent on the speed. Note that some locations (more than half of the total number of tests) showed

chipping failure of the thin film at 50mN normal force. We need to consider the friction force measurements only from tests without any failure of the film to measure the coefficient of friction.

The increase in friction force with increasing normal force is due to the plastic deformation of the material. Therefore, if we represent normalized friction force measured at any randomly chosen normal force as the friction coefficient, it becomes a qualitative tool at best to compare between

two materials because the plastic deformation of the two thin films may be significantly different. Hence, to measure the coefficient of friction accurately, we need to consider the friction force measurement only when the deformation is elastic or have small plastic deformation. Accordingly, Figure 5 plots the normalized friction force as a function of applied normal force. The coefficient of friction (μ) is calculated to be 0.06 ± 0.01 as the average of the normalized friction

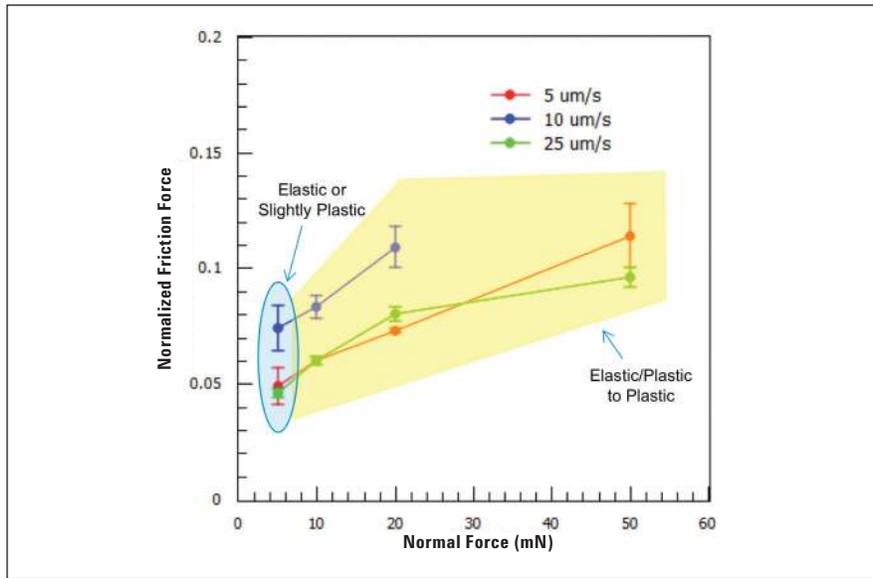


Figure 5. Normalized friction force as a function of applied normal force. Note the coefficient of friction is calculated as the average normalized friction force at 5 mN normal force.

force at 5 mN normal force, where the deformation is almost fully elastic (Figure 2). This value of coefficient of friction agrees well with the expected value (< 0.1) of the company who manufactured this coating. It is important to note here that the coefficient of friction is not a material property but rather a property of the

system consisting of both the thin film and the diamond tip.

The displacement-distance profile and the corresponding friction force profile during the ramp-load scratch test are shown in Figure 6. The critical point for the thin film failure can be easily identified from the sudden change in either the displacement or the

friction force profiles. Interestingly, the normal force corresponding to that critical failure point is about 50 mN, and corroborates the constant-force scratch measurements discussed before. More interestingly, the displacement corresponding to the critical point is close to 200 nm, indicating that the substrate is not deforming significantly

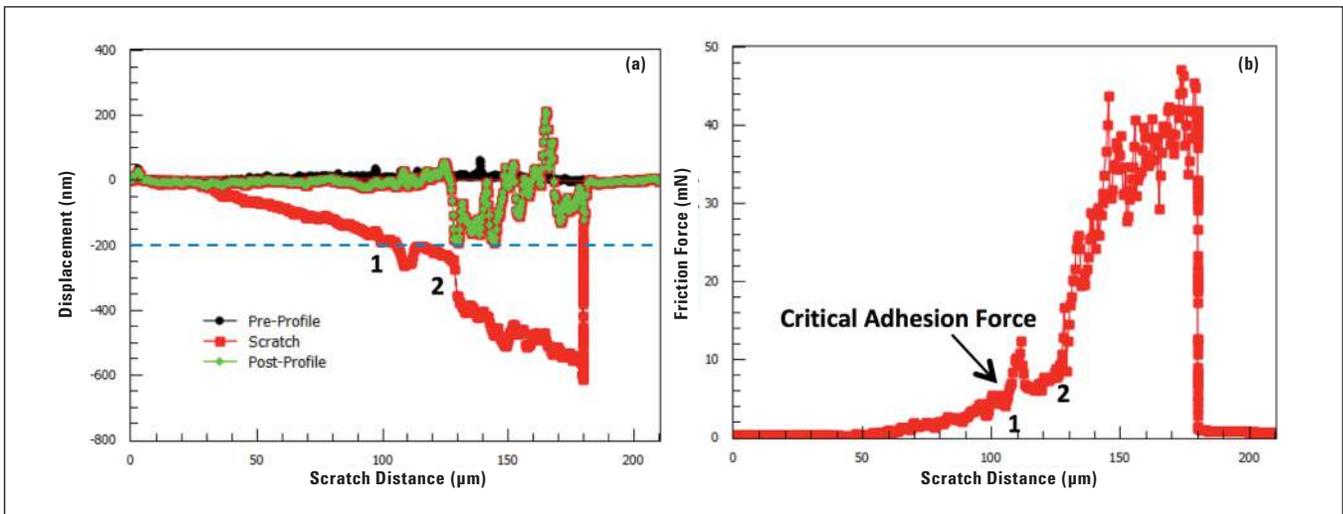


Figure 6. (a) Displacement profile during a ramp-load nano-scratch test on the thin film coating on WC-Co substrate. The normal force was increased linearly from 0 to 100 mN. The dotted line is showing the thin film coating thickness. (b) Corresponding change in friction force during the ramp-load nano-scratch test. The arrow indicates the critical adhesion force.

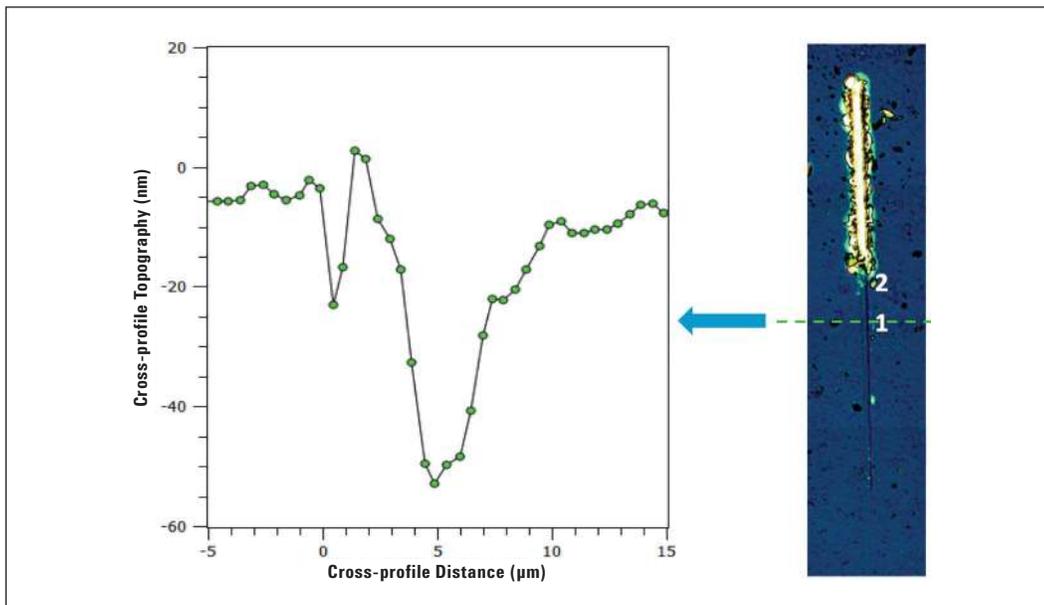


Figure 7. The cross-profile of the ramp-load nano-scratch at the location where the normal force was 50 mN (about the critical failure point). Optical micrograph on the right shows approximately where the cross-profile is taken.

prior to the coating failure. The critical friction force from Figure 6b is about 6 mN. Although there is not a high residual deformation before the critical point (Figure 6a), the uneven residual profile after the critical point clearly indicates severe chipping failure of the coating. Figure 7 shows the cross-profile at the point where the normal force was 50 mN, i.e. almost at the critical point. The absence of any noticeable pile-up suggests that the film did not deform in a ductile way. The optical micrograph, taken post-test, also shows that the positions 1 and 2 corresponding to the same numbers as marked in Figure 6.

This brings us to the other challenging part of performing a nano-scratch

experiment — how to visualize the deformation? The nano-scale thin films requires measurement of small forces and displacements to determine the relevant critical parameters, but at the same time the deformation produced by these tests are also significantly smaller compared to the ones produced by larger probes at much higher loads. So, although we can obtain a picture in an optical microscope (Figure 7), most deformation features are not visible. If it is important, in some cases, the deformation may have to be investigated *ex-situ* in a SEM or an AFM. This is part of the challenges nanomaterials and nanostructures bring with them as we put them in many applications.

An easier solution will be a scanning capability inside the same nanoindenter. The Keysight Technologies, Inc. Nanoindenter G200 can be equipped with a more sensitive DCM transducer along with the XP transducer (needed for lateral force measurement) for better scanning capability. The sample was placed on the nanopositioning stage in order to scan the scratch using the DCM (with a sharp Berkovich tip) to generate high-resolution 3D images. In the scanned 3D image of the scratch made with 50 mN of normal load (critical load in this case) some buckling and conformal cracking (arrows in Figure 8b) can be observed prior to the chipping failure. These images also provide more

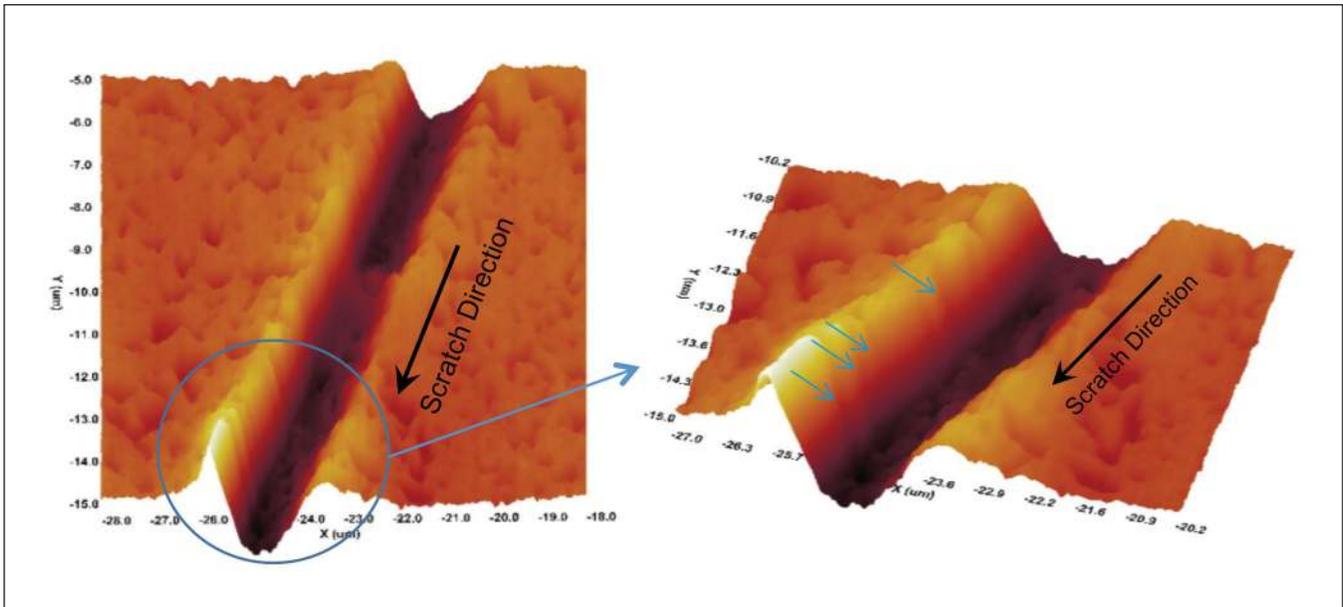


Figure 8. High resolution scan of the nano-scratched region using the same nanoindenter instrument. The 3D images show the surface morphology along with the scratch, made with a normal force of 50 mN. The arrows indicate surface marks, which are most likely caused by buckling and conformal cracking of the thin film during scratching.

details about the thin film surface to interpret the deformation mechanisms.

Summary and Conclusions

The nanotribological properties of a hard thin film coating of about 200 nm thickness, for carbide tools, were investigated. The changes in elastic modulus and hardness of the thin film were measured using the nanoindenter and the continuous stiffness measurement option, which measures the mechanical properties as a continuous function of depth. The thin film modulus is close to that of

the carbide substrate. The coefficient of friction between the thin film and the diamond tip — as determined from nano-scratch tests in the elastic regime — is quite good for machining tool applications. Note that plastic deformation, and speed of scratching, may affect the friction force and should be taken into account when calculating the coefficient of friction. The ramp-load nano-scratch tests were used to characterize the critical adhesion force of the thin film coating under contact loading. From high resolution 3D scans in the same nanoindenter, the deformation mechanisms during the nano-scratch experiments were identified to be buckling and conformal cracking prior

to severe chipping failure at the critical adhesion force.

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