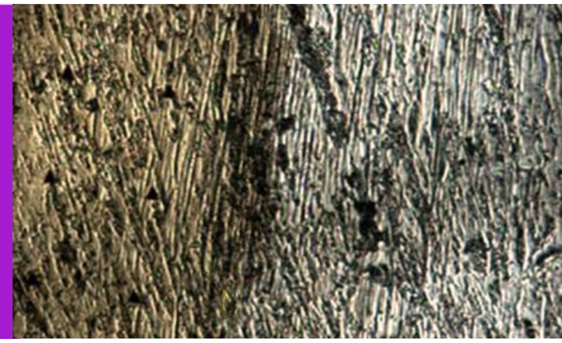
KLA INSTRUMENTS

Mechanical Evaluation of Titanium Nitride Coated Tool Steel



Introduction

Many products are often subject to environments where friction and wear can degrade performance. In such environments, hard coatings can both enhance performance and extend product life. In tooling applications, hard coatings are often used to protect cutting edges and extend the useful tool life. In automotive applications, hard coatings lower the friction between moving parts, thus reducing energy requirements and extending part life. Other applications for hard coatings include computer hard-drives and displays for portable electronic devices. This application note explains the methods and results of testing a titanium nitride (TiN) coating intended to improve the performance and extend the life of a utility blade.

Theory

Instrumented indentation testing (IIT) is a technique for measuring the mechanical properties of materials. It is a development of traditional hardness tests such as Brinell, Rockwell, Vickers, and Knoop. Instrumented indentation testing is similar to traditional hardness testing in that a hard indenter, usually diamond, is pressed into contact with the test material. However, traditional hardness testing yields only one measure of deformation at one applied force, whereas during an IIT test, force and penetration are measured for the entire time that the indenter is in contact with the material. Nearly all advantages of IIT derive from this continuous measurement of force and displacement. Instrumented indentation testing is particularly well-suited for testing small volumes of material such as thin films, particles, or other small features. It is most commonly used to measure Young's modulus *(E)* and hardness *(H)*[1,2]. The Young's modulus for a material is the relationship between stress and strain when deformation is elastic. By knowing the Young's modulus for a design material, strain for a known stress can be predicted, and vice- versa. In metals, hardness depends directly on the flow stress of the material at the strain caused by the indentation. In other words, hardness is an indirect but simple measure of flow stress; within a class of metals, the metal with the higher hardness will also have the higher flow stress.

Using the continuous measurements of force and penetration from a single instrumented indentation test, hardness *(H)* is calculated as:

$$H = P/A \qquad (1)$$

where P is the applied force and A is the contact area. Young's modulus *(E)* depends directly on the reduced modulus (E_r), which is calculated as:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \qquad (2)$$

where S is the elastic stiffness of the contact. The elastic stiffness of the contact may be determined in two different ways. It may be determined semi-statically as the change in force with respect to displacement when the indenter is first withdrawn from the sample, as this part of the test manifests purely elastic recovery. It may also be determined dynamically by oscillating the indenter[3]. If S is determined by the first (semi-static) method, then hardness and Young's modulus can only be realized at the maximum penetration. But if S is determined by the second (dynamic) method, then these same properties can be determined as a continuous function of penetration depth. Both types of tests were performed in this work.
It is often the case that actual product surfaces are so rough that the contact area, A, cannot be determined with sufficient accuracy. In such circumstances, the parameter S^2/P is useful, because it depends directly on the square of reduced modulus divided by hardness, but is independent of contact area[4,5]. This relationship is easily shown by dividing Equation 2 by Equation 1:

$$\frac{(E_r)^2}{H} = \frac{\pi}{4}\frac{S^2}{A}\cdot\frac{A}{P} \propto \frac{S^2}{P} \qquad (3)$$

It should be noted here that the parameter S^2/P has the dimensions of stress, and the value of this parameter is expressed in units of GPa. Dimensional analysis and finite-element analysis have revealed that in contact mechanics, it is not the modulus or flow stress that independently determines permanent damage as a result of stress, but rather the *ratio* of the two[6]. Therefore, the parameter S^2/P is expected to be a good predictor of resistance to permanent damage; lower values of S^2/P correspond to higher resistance to permanent damage.

Experimental Method

For this work, utility blades, shown in Figure 1, were purchased from a local hardware store and tested to determine the extent to which a TiN coating should be expected to improve the performance and longevity of the blade. The base material of the blade is M42 tool steel. The cutting edge of the blade has a titanium-nitride coating that is gold in color, and this coating extends onto the main body of the blade, as shown in Figure 2. Tests of the coating were placed on this part of the blade where the coating extends up onto the flat surface. For comparison, uncoated parts of the blade were also tested.

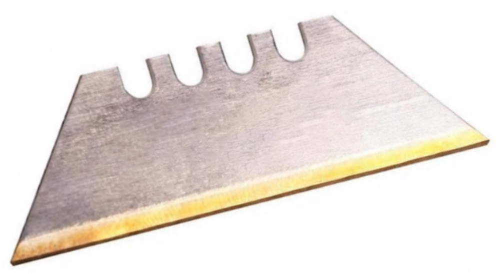


Figure 1. Commercial utility blades were tested.

A KLA Nano Indenter® G200 fitted with a diamond Berkovich indenter was used for all testing. Semi-static tests were performed according the method prescribed by ISO 14577[7]. For all ISO 14577 tests, the force-time algorithm was as follows: 10 second load, 5 second hold, 10 second unload. The CSM option was employed for dynamic testing in order to generate depth profiles of mechanical properties. For all CSM tests, loading was controlled such that the loading rate divided by the load (P'/P) remained constant at 0.1/s. The excitation frequency was 45Hz, and the excitation amplitude was controlled such that the displacement amplitude remained constant at 2nm. Table 1 summarizes all tests that were performed on the utility blade.

Every G200 system is supplied with two reference materials: Pyrex and fused silica. To ensure that the system is functioning properly, it is good practice to test at least one of these materials every time the instrument is used. In this work, Pyrex was tested using both the semi-static (ISO 14577) and dynamic (CSM) methods, and these results are also presented.

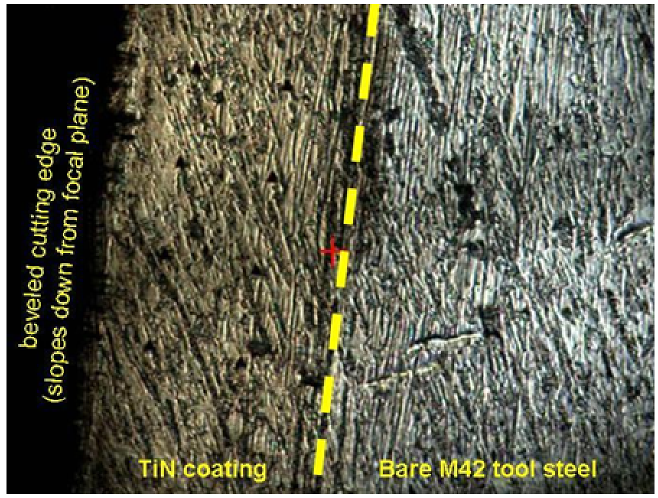


Figure 2. Image of test surface (10X objective lens). Eight residual test impressions are visible.

Table 1. Summary of Utility Blade Tests

Material	Method	P_{max}, mN	Number of tests (N)
Bare M42 steel	CSM	650	20
TiN coating	CSM	650	15
TiN coating	ISO 14577	0.5	20
TiN coating	ISO 14577	5.0	15
TiN coating	ISO 14577	50.0	15
TiN coating	ISO 14577	500.0	15
Bare M42 steel	ISO 14577	0.5	20
Bare M42 steel	ISO 14577	500.0	20

Results and Discussion

The results for Pyrex are shown in Figures 3 and 4, and confirm that the testing instrument is in good working order:

- The two techniques (ISO 14577 and CSM) give results that are sufficiently close to each other;
- Both hardness and Young's modulus are sufficiently constant with depth;
- The measured Young's modulus is sufficiently close to the literature value for Pyrex (62GPa).

The results in Figure 3 and Figure 4 demonstrate the benefits of good surface preparation. The CSM data points each represent a 20-test average. Error bars representing one standard deviation are shown but are small enough to be contained within the data points. Note that ISO 14577 data points do not have error bars because each data point represents only one test; nevertheless, strong repeatability is evident. The slight discrepancy at small depths is due to thermal drift; CSM tests are more sensitive to thermal drift than ISO 14577 tests.

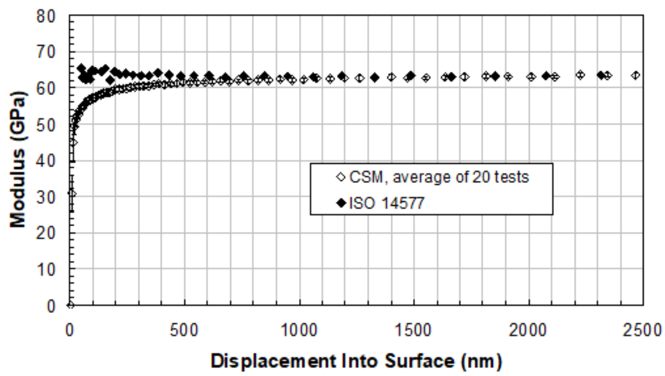


Figure 3. Modulus of Pyrex as a function of surface penetration using the CSM and ISO 14577 test methods. The area function for the Berkovich diamond tip is $A(d) = 24.5037d^2 + 281.7d$, where d is the distance along the diamond axis from the apex to the contact plane.

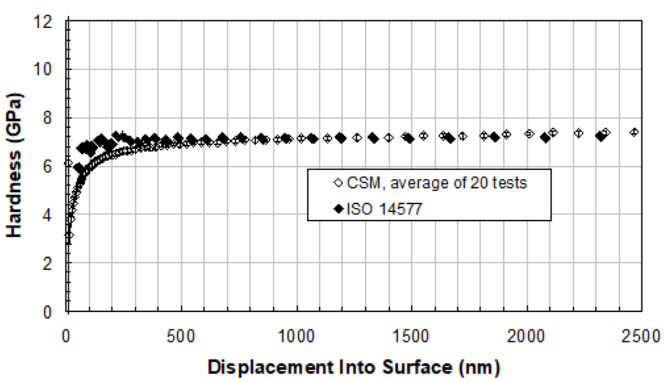


Figure 4. Hardness of Pyrex as a function of surface penetration using the CSM and ISO 14577 test methods.

Figure 5 shows a residual impression from one CSM test on the TiN coating. The peak force for this test was 650mN, yet it is difficult to distinguish the residual impression amid other surface features. Because of this difficulty, the degree of scatter in Figure 6 and Figure 7, which show modulus and hardness for the blade, respectively, is not surprising. For the near-surface data, the error bars span a range that is comparable to the measured value. Such scatter makes it impossible to draw meaningful conclusions about the benefits of the TiN coating based solely on independent measurements of Young's modulus and hardness. At a force of 500mN, the ISO 14577 test method gave a modulus of 238 ± 6.5GPa for the uncoated surface. Steels normally have Young's moduli between 200GPa

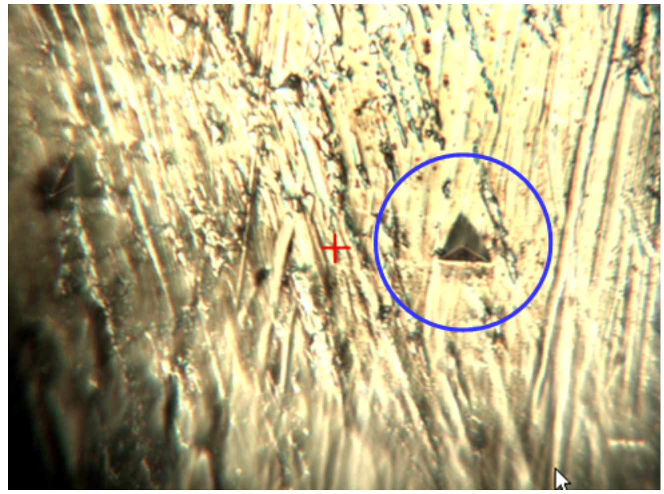


Figure 5. Residual impression from 650mN indent on the TiN coating (40X objective).

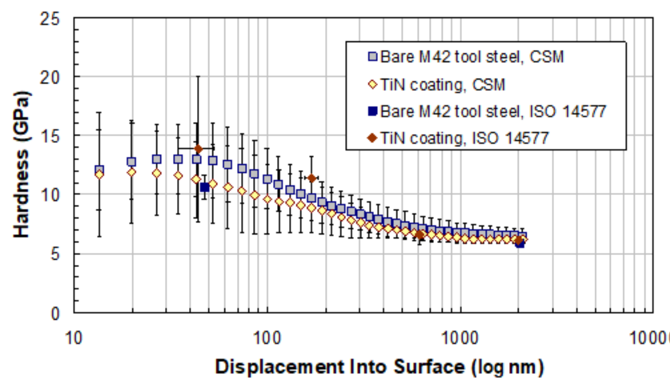


Figure 6. Modulus vs. penetration depth for coated and uncoated regions of a utility blade. Error bars span one standard deviation.

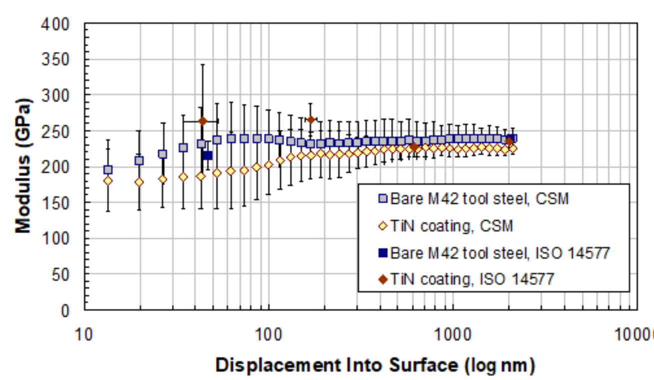


Figure 7. Hardness vs. penetration depth for coated and uncoated regions of a utility blade. Error bars span one standard deviation.

and 220GPa, so this value is slightly higher than expected. However, AISI M42 is a complex alloy of iron and nine different alloying metals, and these alloying metals comprise about 25% of the material by weight[8].

However, the parameter S^2/P does allow us to draw meaningful conclusions about the benefit of the TiN coating, because this parameter is independent of contact area. Figure 8 shows the parameter S^2/P plotted against surface penetration, where the scatter in S^2/P is much lower than the scatter in Young's modulus and hardness. It is reasonable to attribute the scatter in Figure 8 to real point-to-point variation in material properties.

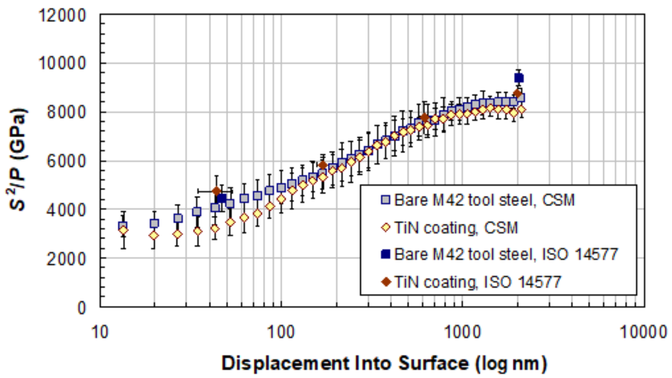


Figure 8. S^2/P vs. penetration depth for coated and uncoated regions of the utility blade. Lower values of this parameter indicate greater resistance to damage. These results do not reveal any mechanical advantage conferred by the TiN coating.

Two important conclusions can be drawn from the data in Figure 8. First, the blade surface does have an improved resistance to damage, as evidenced by the significantly lower value of the parameter S^2/P at small penetration depths. However, this resistance does not seem to depend on the presence of the TiN coating, as both the coated and uncoated regions manifest similar depth profiles for the parameter S^2/P over the range of 10nm to 2μm. Other mechanical tests might reveal an advantage conferred by the coating. For example, the lateral force option of the G200 might be used to measure the friction coefficient of the coated and uncoated regions. However, based solely on the tests performed in this work, no mechanical advantage is conferred by the TiN coating.
Both the CSM and ISO 14577 test methods are useful in their own way. CSM is useful, because every individual test returns complete depth profiles of modulus, hardness, and S^2/P. Testing according to ISO 14577 is useful, because it is a standardized test method, and because scatter is generally lower relative to CSM under identical conditions. If it is important to measure properties according to a standard test method, then CSM can be used for preliminary testing in order to determine the ideal peak force or depth that should be used for the standard measurements.

Conclusion

As manufactured, the surface of this product was too rough to draw meaningful conclusions about the value of the TiN coating solely from the independent measurements of hardness and Young's modulus. However, the parameter S^2/P, which is proportional to the parameter E_r^2/H but independent of contact area, measured much lower scatter. This parameter revealed no mechanical advantage conferred by the TiN coating. Instrumented indentation testing (IIT) is an essential tool for evaluating films, coatings, and surface layers, which are used to improve mechanical performance and longevity. Although the contact-mechanics theory behind instrumented indentation testing is complex, the KLA Nano Indenter G200 makes IIT one of the simplest and fastest types of mechanical testing, because sample preparation is relatively easy, and hundreds of tests can be performed on a single sample. Continuous stiffness measurement (CSM) is a valuable addition to basic IIT and is used to measure properties of thin films and surface layers as a continuous function of penetration depth.

References

1. I.N. Sneddon, "The Relation Between Load and Penetration in the Axisymmetric Boussinesq Problem for a Punch of Arbitrary Profile," International Journal of Engineering Science, vol. 3, pp. 47-56, 1965.
2. W.C. Oliver and G.M. Pharr, "An Improved Technique for Determining Hardness and Elastic Modulus Using Load and Displacement Sensing Indentation Experiments," Journal of Materials Research, vol. 7 (No. 6), pp. 1564–1583, 1992.
3. W.C. Oliver and J.B. Pethica, "Method for continuous determination of the elastic stiffness of contact between two bodies," U.S. Patent 4848141, 1989.
4. D.L. Joslin and W.C. Oliver, "A new method for analyzing data from continuous depth-sensing microindentation tests," Journal of Materials Research, vol. 5 (No. 1) pp. 123-126, 1990.
5. T.F. Page, G.M. Pharr, J.C. Hay, W.C. Oliver, B.N. Lucas, E.G. Herbert and L. Riester, "Nanoindentation characterization of coated systems: P:S2—A new approach using the continuous stiffness technique," Fundamentals of Nanoindentation and Nanotribology, MRS Symposium Proceedings, vol. 522, pp. 53-64, 1998.
6. Y.T. Cheng and C.M. Cheng, "Relationships between hardness, elastic modulus, and the work of indentation, Applied Physics Letters, vol. 73 (No. 5), pp. 614-616, 1998.
7. "Metallic materials - Instrumented indentation test for hardness and materials parameters," ISO 14577, 2002.
8. http://www.efunda.com/Materials/alloys/tool_steels/show_tool.cfm?ID=AISI_M42&show_prop=all&Page_Title=AISI%20M42.

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