INSPECTION OF TITANIUM NITRIDE COATINGS BY SCRATCH TESTING

Prepared by

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INTRODUCTION

The combination of high hardness, excellent wear resistance, corrosion resistance and inertness makes titanium nitride (TiN) an ideal protective coating for metal components in various industries. For example, the edge retention and corrosion resistance of TiN coating can substantially increase the work efficiency and extend the service lifetime of the machine tooling such as razor blades, metal cutting, injection molding and sawing. Its high hardness, inertness and non-toxicity make TiN a good candidate for applications in medical devices including implants and surgical instruments.

IMPORTANCE OF SCRATCH TESTING ON QUALITY CONTROL OF TIN COATINGS

Residual stress in protective PVD/CVD coatings plays a critical role in the performance and mechanical integrity of the coated component. The residual stress derives from several major sources, including growth stress, thermal gradients, geometric constraints and service stress[i]. The thermal expansion mismatch between the coating and the substrate created during coating deposition at elevated temperatures leads to high thermal residual stress. Moreover, the TiN coated tools are often used under very high concentrated stresses, e.g. drill bits and bearings. It is critical to develop a reliable quality control process to quantitatively inspect the cohesive and adhesive strength of the protective functional coatings.

MEASUREMENT OBJECTIVE

In this study, we showcase that the scratch mode of Nanovea Mechanical Tester provides an ideal tool to assess the cohesive/adhesive strength of the protective TiN coatings in a controlled and quantitative manner.

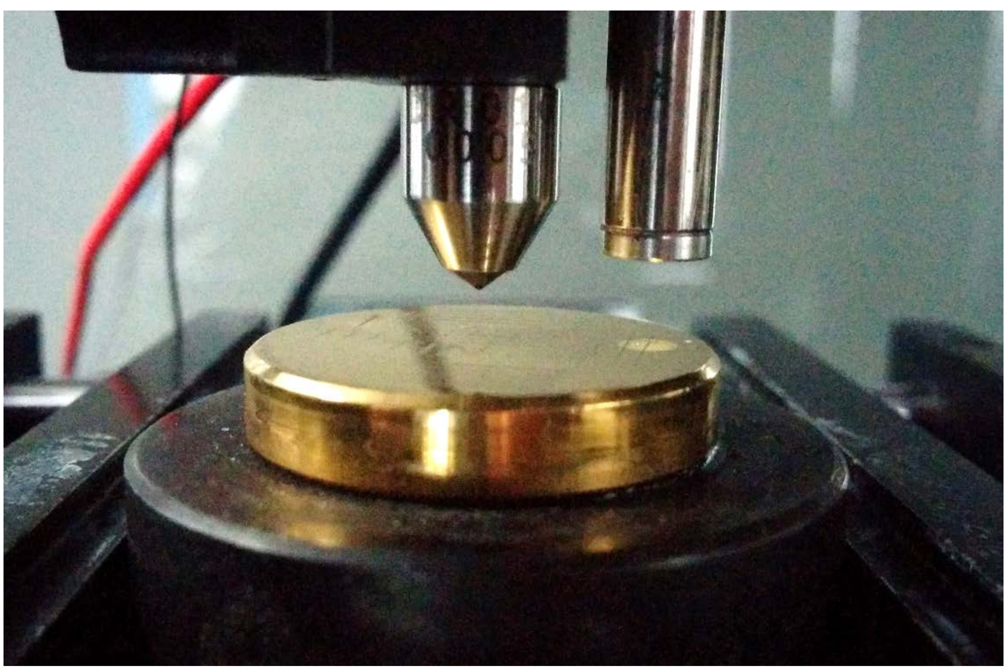


Fig. 1: Scratch tip on the TiN sample.

TEST PARAMETERS

The Nanovea Mechanical Tester was used to perform the scratch tests on three TiN coatings using the same test parameters as summarized in Table 1.

Loading mode	Progressive linear
Initial Load	0.02 N
Final Load	10 N
Loading Rate	20 N/min
Scratch Length	5 mm
Indenter Type	Sphero-Conical Diamond
Indenter Radius	20 µm

Table 1: Test parameters of the scratch tests on the TiN samples.

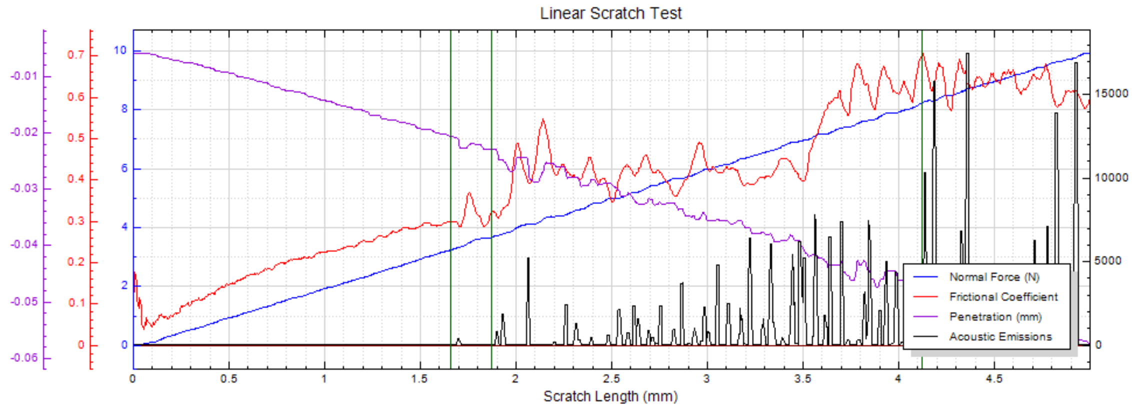
RESULTS AND DISCUSSION

Fig. 2 shows the recorded evolution of penetration depth, coefficient of friction (COF) and acoustic emission during the test. The full micro scratch tracks on the TiN samples are shown in Fig. 3. The failure behaviors at different critical loads are displayed in Fig. 4, where critical load L_{c1} is defined as the load at which the first sign of cohesive crack occurs in the scratch track, L_{c2} is the load after which repeated spallation failures take place, and L_{c3} is the load at which the coating is completely removed from the substrate. The critical load (L_c) values for the TiN coatings are summarized in Fig. 5.

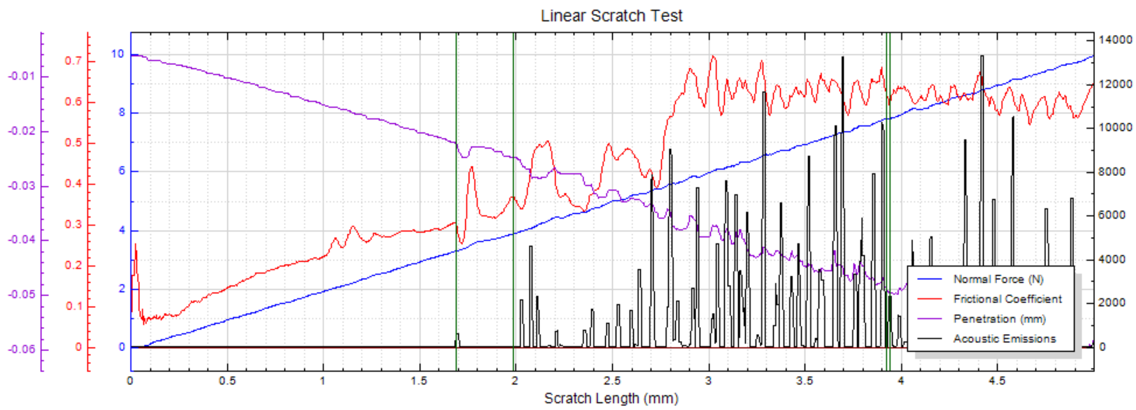
The evolution of penetration depth, COF and acoustic emission provides insight of the mechanism of the coating failure at different stages, which is represented by the critical loads in this study. It can be observed that Sample A and Sample B exhibit comparable behavior during the scratch test. The stylus progressively penetrates into the sample to a depth of ~0.06 mm and the COF gradually increases to ~0.3 as the normal load increases linearly at the beginning of the scratch test. When the L_{c1} of ~3.3 N is reached, the first sign of chipping failure occurs. This is also reflected in the first large spikes in the plot of penetration depth, COF and acoustic emission. As the load continues to increase to L_{c2} of ~3.8 N, further fluctuation of the penetration depth, COF and acoustic emission takes place. We can observe continuous spallation failure present at both sides of the scratch track. At the L_{c3}, the coating completely delaminates from the metal substrate under the high pressure applied by the stylus, leaving the substrate exposed and unprotected.

In comparison, Sample C exhibits lower critical loads at different stages of the scratch tests, which is also reflected in the evolution of penetration depth, coefficient of friction (COF) and acoustic emission during the scratch test. Sample C possesses an adhesion interlayer with lower hardness and higher stress at the interface between the top TiN coating and the metal substrate compared to Samples A and B. This study demonstrates the importance of proper substrate support and coating architecture to the quality of the coating system. A stronger interlayer can better resist deformation under a high external load and concentration stress, and thus enhance the cohesive and adhesive strength of the coating/substrate system.

(a) Sample A:



(b) Sample B:



(c) Sample C:

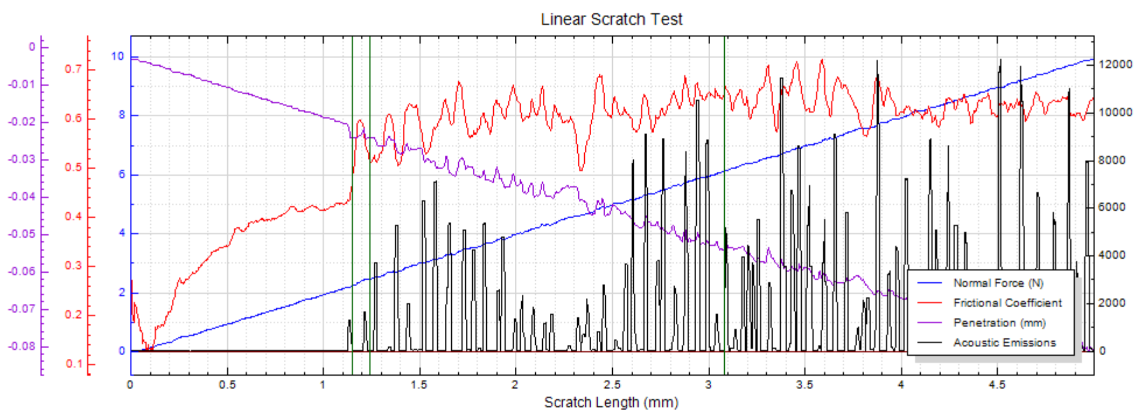


Fig. 2: Evolution of penetration depth, COF and acoustic emission of the TiN samples.

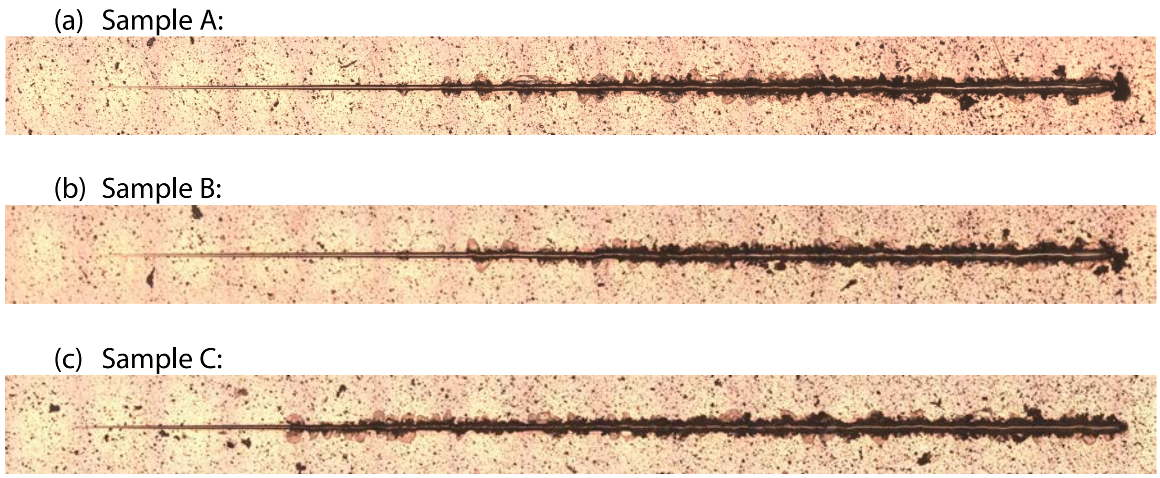


Fig. 3: Full scratch track of the TiN coatings after the tests.

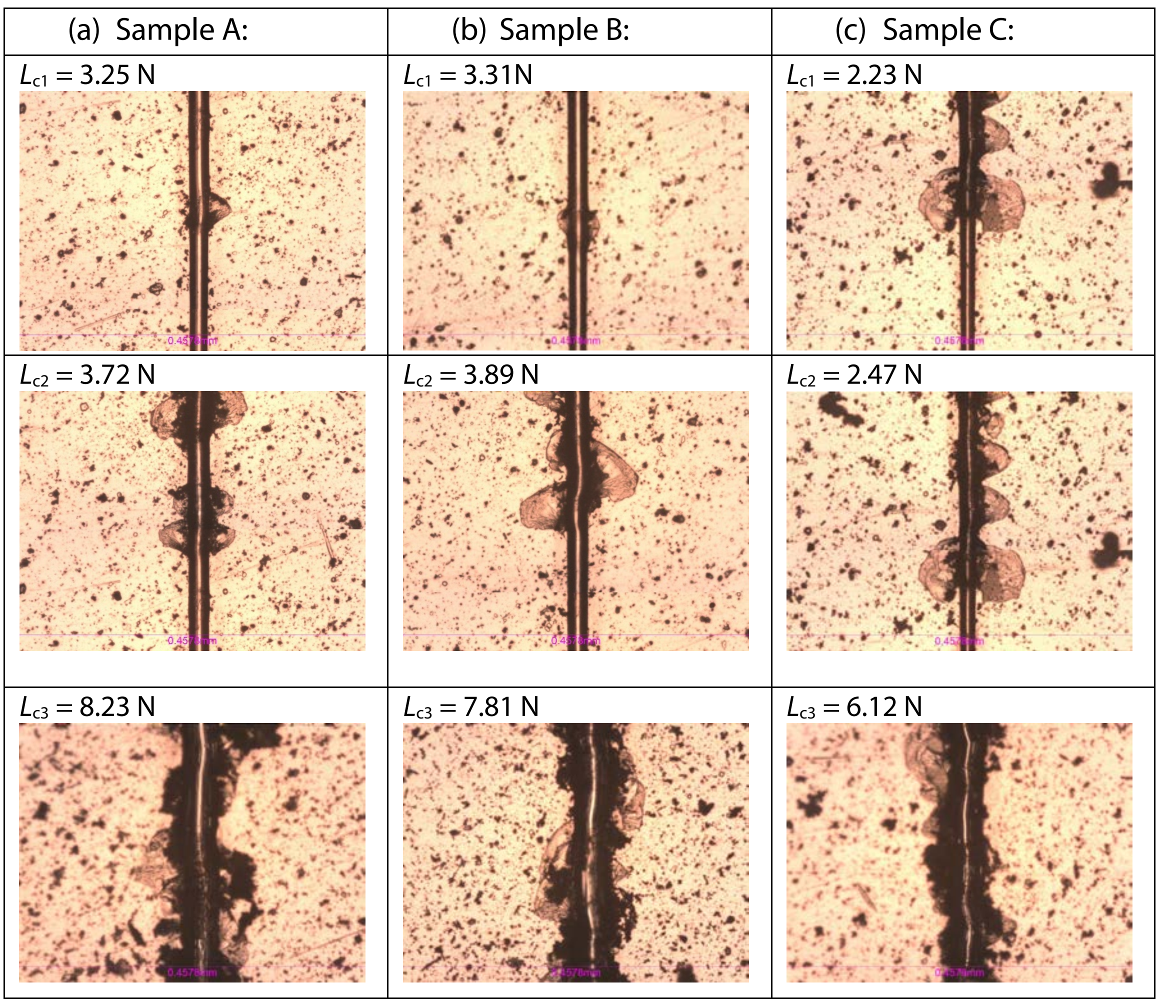


Fig. 4: TiN coating failures under different critical loads, L_c.

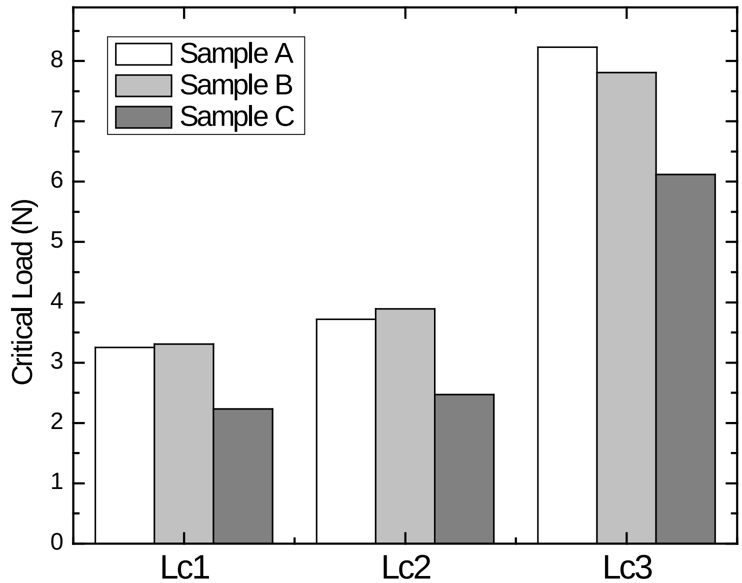


Fig. 5: Summary of critical load (L_c) values for the TiN coatings.

CONCLUSION

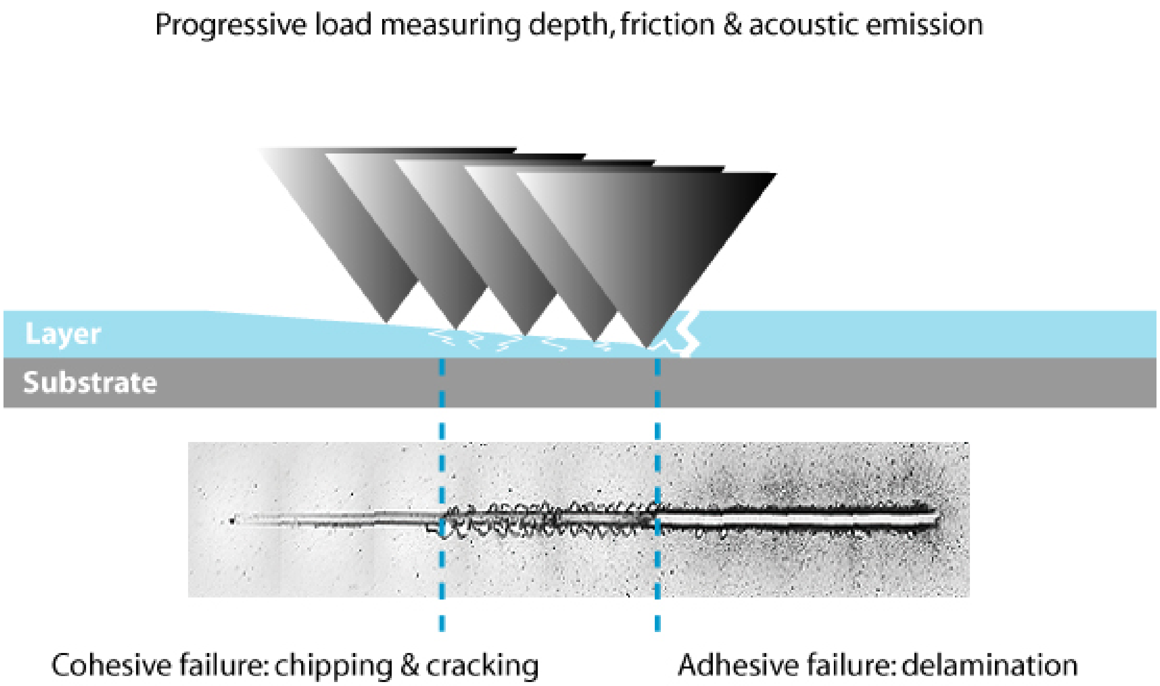
In this study, we showcased that Nanovea Mechanical Tester performs reliable and accurate scratch tests on the TiN coating sample in a controlled and closely monitored manner. The scratch measurement allows users to quickly identify the critical load at which typical cohesive and adhesive coating failure occurs. It provides a superior quality control tool to quantitatively inspect and compare the intrinsic quality of the coating and the interfacial integrity of the coating/substrate system. A coating with a proper interlayer can resist large deformation under a high external load and concentration stress, and enhance the cohesive and adhesive strength of the coating/substrate system.

The Nano, Micro or Macro modules of the Nanovea Mechanical Tester all include ISO and ASTM compliant indentation, scratch and wear tester modes, providing the widest and most user friendly range of testing available in a single system. Nanovea's unmatched range is an ideal solution for determining the full range of mechanical properties of thin or thick, soft or hard coatings, films and substrates, including hardness, Young's modulus, fracture toughness, adhesion, wear resistance and many others.

APPENDIX: SCRATCH TEST PRINCIPLE

The scratch testing method is a comparative test in which critical loads at which failures appear in the samples are used to evaluate the relative cohesive or adhesive properties of a coating or bulk material. During the test, scratches are made on the sample with a sphero-conical stylus (generally Rockwell C diamond, tip radius ranging from 20 to 200µm) which is drawn at a constant speed across the sample, under a constant load, or, more commonly, a progressive load with a fixed loading rate.

When performing a progressive load test, the critical load (L_c) is defined as the smallest load at which a recognizable failure occurs. The driving forces for coating damage in the scratch test are a combination of elastic-plastic indentation stresses, frictional stresses and the residual internal stresses. In the lower load regime, these stresses generally result in conformal or tensile cracking of the coating which still remains fully adherent. The onset of these phenomena defines a first critical load. In the higher load regime, one defines another critical load which corresponds to the onset of coating detachment from the substrate by spalling, buckling or chipping.



[i] V. Teixeira, Vacuum 64 (2002) 393–399