APPLICATION NOTE

Ultimate Solution for Ultra-Thin Film Systems

i^{TF} Modulus Solution with xProbe Applications for 10nm (and below) Thin Films

Introduction

Reliable measurements of the Elastic Modulus of thin films is particularly challenging due to the so called substrate effect. The prevalent rule of limiting indentation depth to 10% of the coating thickness to avoid the substrate influence in the mechanical properties is challenging to assure, especially when the film thickness goes below 200nm. The tip radius can be one of the many factors limiting the application of Oliver-Pharr model [1] on the elastic modulus calculation, so as the surface roughness.

Figure 1: Sample diagram.

With the newly developed ultra-low noise **xProbe** transducer combined with the **Intrinsic Thin Film Property Solution** (i^{TF}) [2], quantitative mechanical properties from nanoindentation tests on 10nm thin film systems become possible.

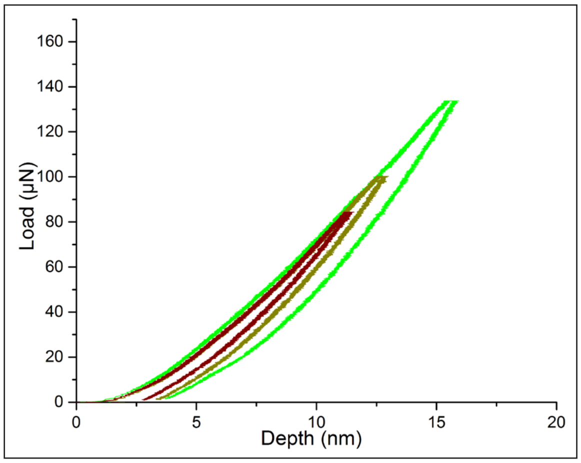

Figure 2: Representative load displacement nanoindentation curves of on 10nm ultra-thin film with an **xProbe**.

Procedure

The experiment was conducted using Hysitron's **TI 950 TriboIndenter®** equipped with an xProbe and Cube Corner probe in quasi-static mode. The xProbe is a MEMS based transducer with a noise floor similar to that of a contact mode Atomic Force Microscope (AFM). The linear actuation allows for direct and fully quantitative measurements without the need of modeling, which leads to more precise mechanical properties estimation and higher analysis throughput. Nanoindentation tests (Figure 2) were performed on the ultra-thin film sample using load control feedback mode.

Results

Unloading segments of each indentation were analyzed using the Oliver-Pharr model [1], where the stiffness is calculated from the unload segment. Based on that, as well as probe calibration, the elastic modulus of nanoindentation can be directly calculated with non-linear substrate effect to the thin film based on the equation:

$$E_r = (S\sqrt{\pi})/(2\sqrt{A}) \quad [1]$$

Where S is the unloading stiffness and A is the projected contact area.

Reduced elastic modulus and hardness are plotted in a function of contact depth (Figure 3). The initial increase in the mechanical properties can be related to the surface roughness and the interfacial surface layer on the surface.

Applying i^{TF} analysis to the stiffness, depth and load profile, the intrinsic elastic modulus of the film will be calculated almost instantaneously. i^{TF} analysis utilizes the known area function, the contact geometry, and the load and stiffness as an input for the three equations between the elastic deflection and the contact radius.

Unlike finite element analysis, the model does not need any presumed modulus for the thin film. By numerically solving these equations, the film modulus (Figure 3) and plasticity parameter are calculated.

Conclusions

By combining the ultra-low noise xProbe transducer and analytical intrinsic thin film solution (i^{TF}), we quantitatively determine elastic properties of the ultra-thin film systems of 10nm or below*.

**Contact your local Hysitron sales representative/sales agent for a demonstration.*

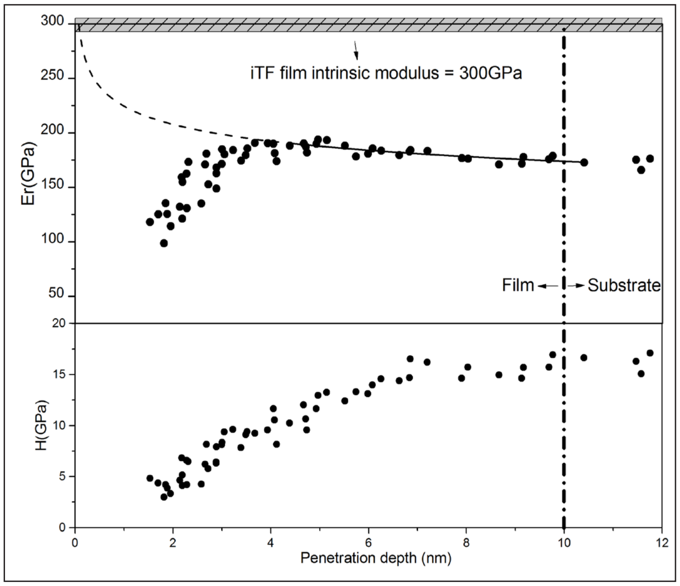

Figure 3: Reduced elastic modulus and hardness are plotted in terms of nanoindentation penetration depth. The interface between the film and substrate can be seen in the graph shown as dash dot line. The Estimated film modulus analyzed by i^{TF} analysis is marked in the shadowed area (300GPa). The approximation of the reduced elastic modulus of the whole system is marked in dash line.

References

1. Li H. and Vlassak J. J. 2009 Determining the elastic modulus and hardness of an ultra-thin film on a substrate using nanoindentation Journal of Materials Research 24 1114–26.
2. Oliver W. C. and Pharr G. M. 1992 An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments J. Mater. Res. 7 1564–83.