NANOVEA®
A Better Measure.

MICRO-SCRAPE TEST OF POLYMERIC COATING ON SILICON WAFER

Prepared by
Duanjie Li, PhD

6 Morgan, Ste156, Irvine CA 92618 · P: 949.461.9292 · F: 949.461.9232 · nanovea.com

INTRODUCTION

Polymeric coatings are extensively used to enhance the mechanical, tribological, optical and chemical properties of different substrates in a wide variety of industries, such as semiconductor, solar energy, optics and biomedicine. The intrinsic quality of the coating and the interfacial integrity of the coating/substrate system determines the service performance and lifetime of the polymeric coating.

The scratch and micro-scratch testing has developed to be one of the most widely applied methods to evaluate the cohesive and adhesive strength of the coatings. The critical load, at which a certain type of coating failure occurs as the applied load progressively increases, is widely regarded as a reliable tool to determine and compare the adhesive and cohesive properties of the coatings. The most commonly used indenter for scratch testing is the conical Rockwell diamond indenter. However, when the scratch test is performed on the soft polymeric coating deposited on a brittle substrate such as silicon wafer, the conical indenter tends to plough through the coating forming grooves rather than creating cracks or delamination. Cracking of the brittle silicon wafer takes place when the load further increases. Therefore, it is vital to develop a new technique to evaluate the cohesion or adhesion properties of soft coatings on a brittle substrate.

MEASUREMENT OBJECTIVE

In this application, the cohesive and adhesive strength of a polymeric coating on the silicon wafer substrate is evaluated by micro-scrape test using the Nanovea Mechanical Tester.

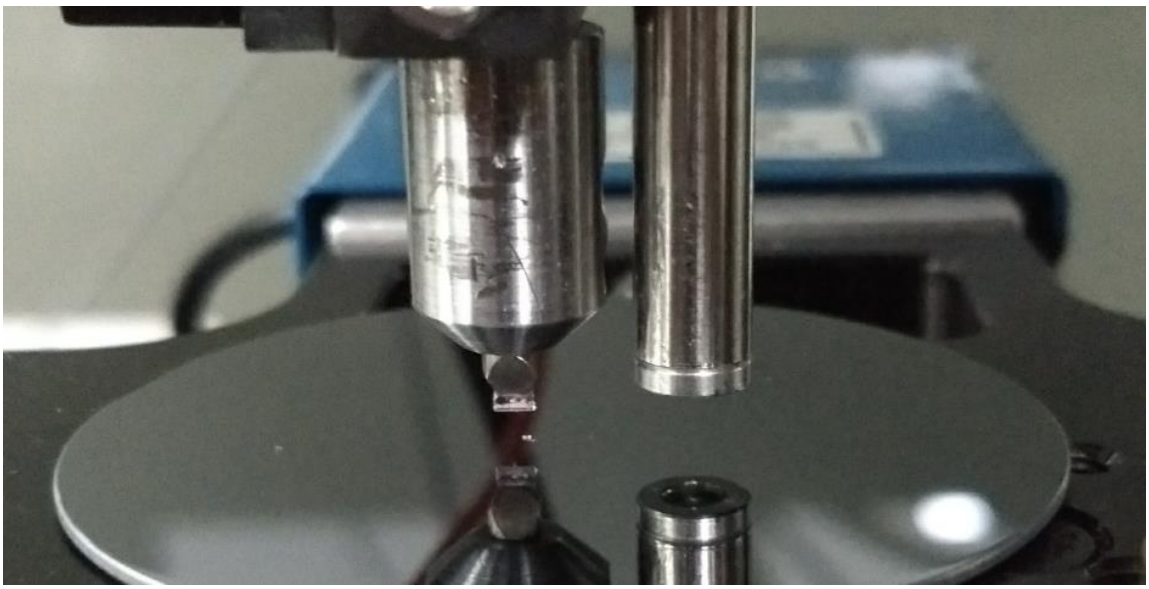

Fig. 1: Micro-scrape knife diamond tip and depth sensor on the polymer coating.

TEST CONDITIONS

The Nanovea Mechanical Tester was used to perform the micro-scrape tests on a polymer coating sample on the silicon wafer substrate using the test parameters summarized in Table 1. A new sample is used for the micro-scrape tests at each load.

Test parameters	Value
Load type	Constant
Loads	0.25, 0.5, 1, 2, 3, 3.5, 3.75 and 4 N
Sliding speed	3 mm/min
Sliding distance	1 mm
Indenter geometry	Knife
Indenter material (tip)	Diamond
Indenter tip angle	90°
Atmosphere	Air
Temperature	24°C (room)

Table 1: Test parameters of the micro-scrape measurements.

RESULTS AND DISCUSSION

The adhesion of the polymer coating on the silicon substrate is measured in this study. Due to the brittle characteristics of the silicon wafer, a diamond stylus of a horizontal knife-shape edge is used to avoid penetration of the silicon substrate that may be caused by the sharp conical indenter. The full scrape tracks on the polymer coating sample at different constant loads are shown in Fig. 2. The polymer coating has a 90° triangle shape pattern on the silicon wafer substrate.

It can be observed that the polymer coating shows signs of cohesive failure at the tip of the triangle shape at a normal load of 0.25 N. As the constant normal load during the micro-scrape test increases from 0.25 to 2.0 N, the cohesive failure region on the coating progressively expands from the tip of the triangle to its bottom as indicated by the increased size of the dark area. When the load further increases from 2.0 to 3.75 N, signs of delamination of the polymer coating starts to appear at the tip of the triangle. The coating completely delaminates from the substrate at a normal load of 4.0 N. Such critical loads at which typical forms of coating failures occur can be used to quantitatively compare the cohesive and adhesive strength of the coating to select the best candidate for targeted applications.

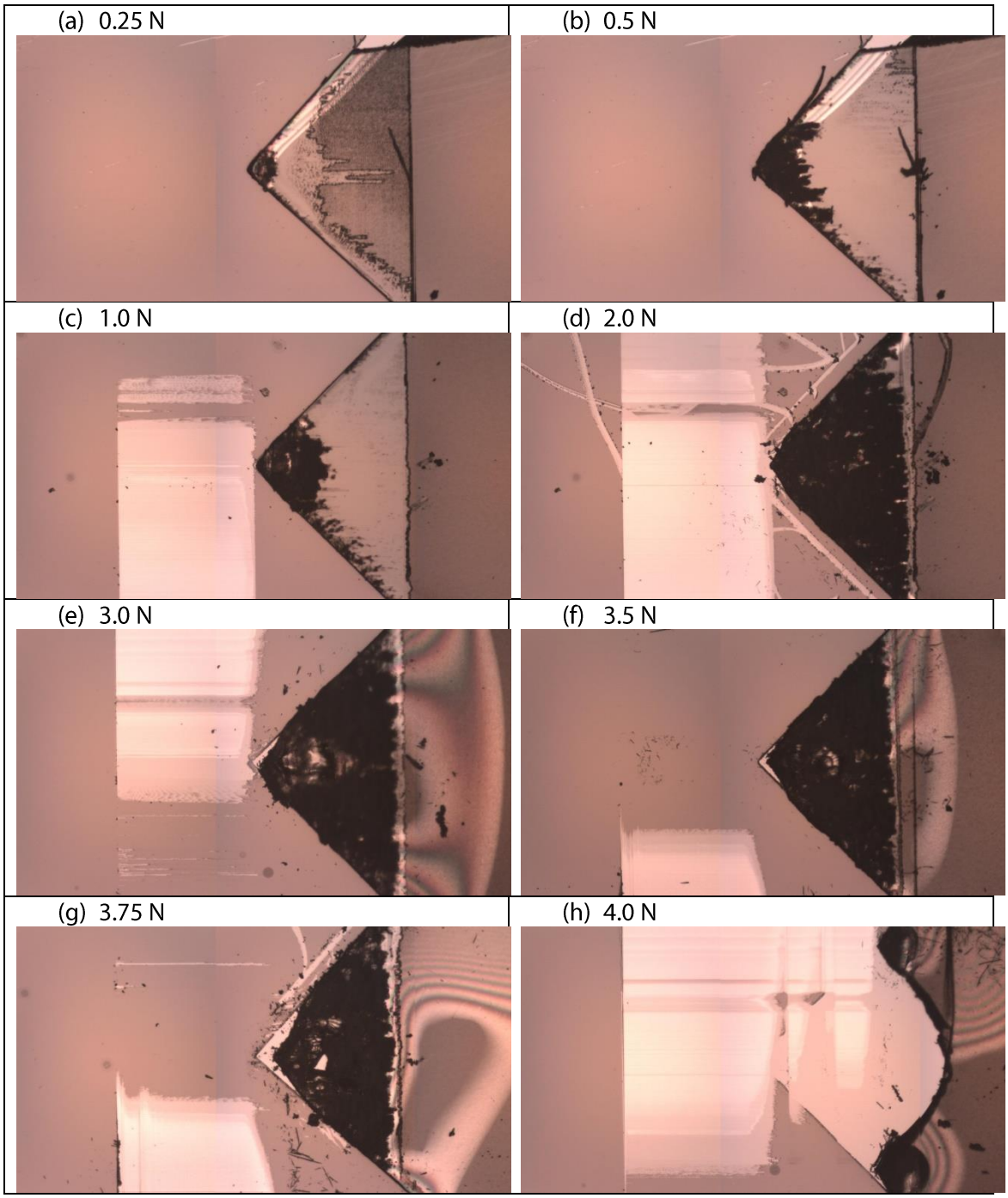

Fig. 2: The coating after the micro-scrape tests at different loads.

Fig. 3 plots the evolution of COF during the micro-scrape test that may provide more insight in the progression of coating failures. At a low load of 0.25 N, the COF remains constant as the knife tip scrapes in the silicon substrate and the polymer coating areas. However, when the knife tip scrapes at the tip of the triangle, i.e. the edge of the coating, a high COF peak is measured due to the elevated height by the coating. A small cohesive failure of the polymer coating takes place at the same time. At a higher

load of 2.0 N, cohesive failure takes place on the whole surface of the polymer coating, leading to higher COF value measured in the coated area. As the normal load further increases to 4.0 N, the polymer coating is completely scraped off from the silicon substrate.

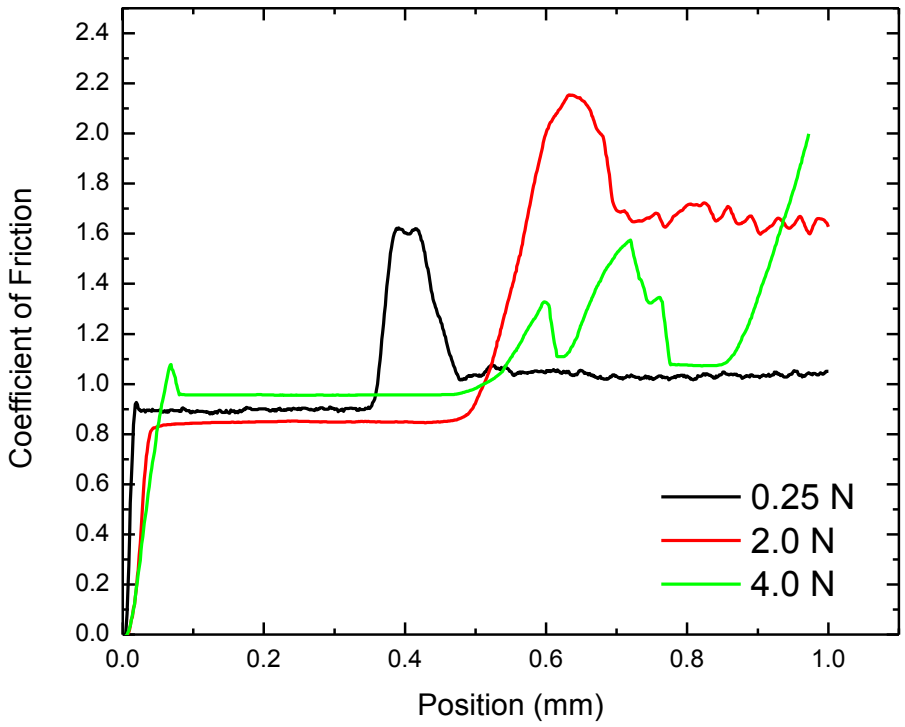

Fig. 3: Evolution of coefficient of friction during the micro-scrape tests.

CONCLUSION

In this study, we showcased that Nanovea Mechanical Tester performs reliable micro-scrape tests on a polymer coating deposited on a silicon wafer substrate. A knife-shape diamond tip of a horizontal edge is selected to perform the test so as to avoid penetration through the brittle silicon substrate.

By applying different constant loads, the micro-scrape measurement allows users to identify the load at which typical cohesive and adhesive coating failure occurs. It provides a superior tool to quantitatively evaluate and compare the intrinsic quality of the polymer coating and the interfacial integrity 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.

To learn more about Nanovea Mechanical Tester or Lab Services.

APPENDIX: MEASUREMENT PRINCIPLE

Principle of scratch test

The scratch testing method is a very reproducible quantitative technique. Critical loads at which failures appear are used to compare the cohesive or adhesive properties of coatings or bulk materials. During the test, scratches are made on the sample with a sphero-conical stylus (tip radius ranging from 1 to 200 μm) 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. Sphero-conical stylus is available with different radii (which describes the "sharpness" of the stylus). Common radii are from 20 to 200 μm for micro/macro scratch tests, and from 1 to 20 μm for nano scratch tests.

When performing a progressive load test, the critical load is defined as the smallest load at which a recognizable failure occurs. In the case of a constant load test, the critical load corresponds to the load at which a regular occurrence of such failure along the track is observed.

In the case of bulk materials, the critical loads observed are cohesive failures, such as cracking or plastic deformation of the material. In the case of coated samples, the lower load regime results in conformal or tensile cracking of the coating which still remains fully adherent (which usually defines the first critical load). In the higher load regime, further damage usually comes from coating detachment from the substrate by spalling, buckling or chipping. Fig. 1 illustrates the principle of scratch testing.

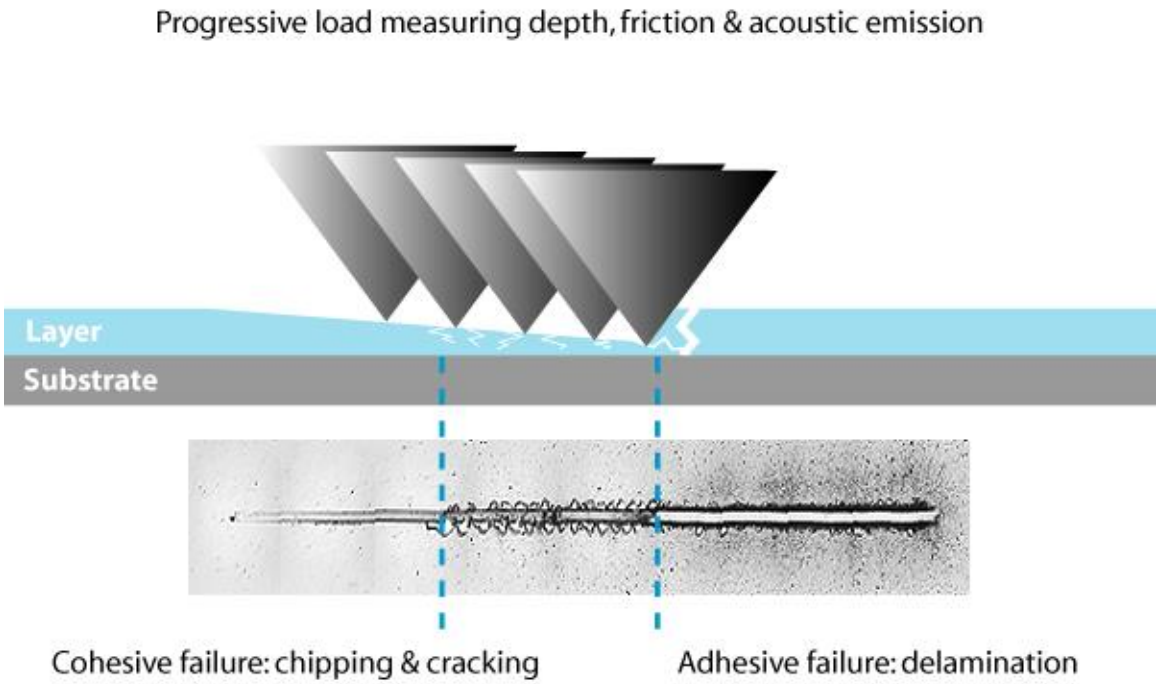

Fig. 1: Principle of scratch testing.

Comments on the critical load

The scratch test gives reproducible quantitative data that can be used to compare the behavior of various coatings. The critical loads depend on the mechanical strength (adhesion, cohesion) of a coating-substrate composite but also on several other parameters: some of them are directly related to the test itself, while others are related to the coating-substrate system. The parameters that determine the critical loads are summarized in Table 1.

Test specific parameters	Sample specific parameters
Loading rate Scratching speed Indenter tip radius Indenter material	Friction coefficient between surface and indenter Internal stresses in the material for bulk materials Material hardness & roughness for coating-substrate systems Substrate hardness and roughness Coating hardness and roughness Coating thickness

Table 1: List of parameters that determine the critical loads.

Means for critical load determination

Microscopic observation
This is the most reliable method to detect surface damage. This technique is able to differentiate between cohesive failure within the coating and adhesive failure at the interface of the coating-substrate system.

Tangential (frictional) force recording
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses to failures are very specific to the coating-substrate system in study.

Acoustic emission (AE) detection
Detection of elastic waves generated as a result of the formation and propagation of microcracks. The AE sensor is insensitive to mechanical vibration frequencies of the instrument. This method of critical load determination is mostly adequate for hard coatings that crack with more energy.

Depth Sensing
Sudden change in the depth data can indicate delimitation. Depth information pre and post scratch can also give information on plastic versus elastic deformation during the test. 3D Non-Contact imaging such as white light axial chromatism technique and AFMs can be useful to measure exact depth of scratch after the test.

Other possible measurements by Nanovea Mechanical Tester:
Hardness and Young's Modulus, Stress-Strain & Yield Stress, Fracture Toughness, Compression strength, Fatigue testing and many others.