Understanding Mechanisms of Adhesion of SiO_2 Thin Film Deposited on a Polymeric Substrate

C. Ho [1,2], A. Dehoux [2], J. Alexis [1], L. Lacroix [1], O. Dalverny [1], S. Châtel [2], B. Faure [2]

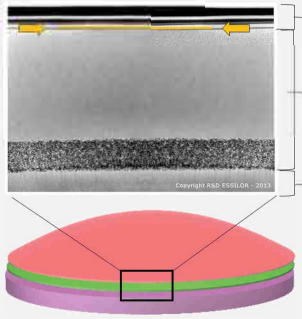
[1] Laboratoire Génie de Production ENIT-INP, University of Toulouse, Tarbes, France
[2] Thin Films Group, R&D Physico-Chemistry, Essilor International, Créteil, France

Contact :
caroline.ho@enit.fr

43rd ICMCTF
International Conference on Metallurgical Coatings and Thin Films
April 28-May 2, 2014
San Diego, CA, USA
http://www.avs.org/conferences/icmctf/

Abstract : A better understanding of mechanisms of adhesion between a 200 nm thick silicon dioxide layer and a 4.5 µm thick polymeric hardcoat is indispensable for an efficient adhesion at the interface. To reach this purpose, focus is placed on two axes: finding an applicable and effective method to quantify adhesion and in parallel, characterizing mechanical properties of materials composing the system. The second axis is needed to obtain data to feed modeling codes, enabling a better analysis of the adhesion experiment. Modulus of modified SiO_2 was found to be roughly 20% higher than reference SiO_2, by nanoindentation. AFM experiments showed no difference between modified and reference SiO_2. Currently, an investigation to detect cracks at the interface of interest for micro-tensile test is ongoing. Adhesion tests, such as micro-compression will be performed as well.

Context



Anti-Reflective Stack (nm) → REFLECTIONS
HardCoat (µm) → SCRATCHES
Substrate (mm)

To improve wearers' experience, ophthalmic lenses made of plastic polymeric substrates are coated with functional treatments, bringing anti-sratch and anti-reflective properties. These treatments are composed of 5 to 15 layers, ranging from micrometers to nanometers.

Challenge Insufficient adhesion between layers may lead to delamination

Delamination caused by Process Modification

To anticipate and prevent delamination
→ Understanding mechanisms of adhesion between Hardcoat and Anti-Reflective Stack

Methodology

Adhesion between SiO_2 layer and HardCoat

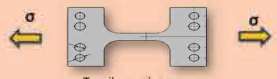
Mechanical Adhesion = *f* (Fundamental Adhesion + Other Factors)

Evaluation of Practical Adhesion & Localization of Mechanical Stress' Application

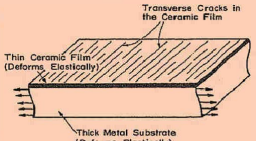
Characterization of Bulk Mechanical Properties of Constituents Composing the System

First Results

Micro-tensile test

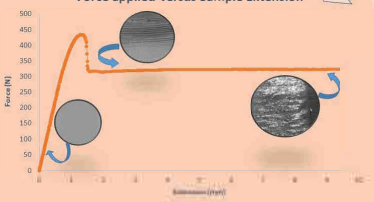


Tensile specimen



D.C. Agrawal, R. Raj, "Measurement of the ultimate shear strength of a metal-ceramic interface", Acta Metallurgica 37 (1989) 1625-1271

Force applied versus Sample Extension

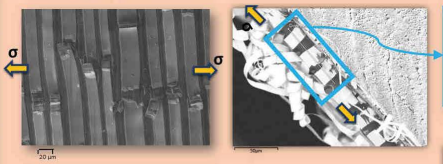


Agrawal and Raj model

$$\tau = \frac{\pi \delta \sigma}{\lambda}$$

τ : maximum interfacial shear strength in MPa
δ : film thickness in µm
σ : stress at first cracking occurrence in MPa
λ : mean inter-crack spacing at steady state in µm

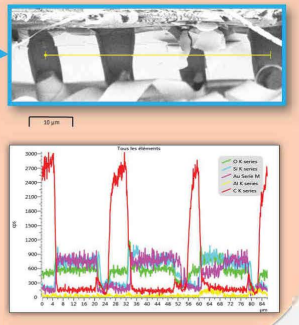
SEM micrographs of fractured tensile specimens



τ HardCoat / Substrate interface : [9 ; 14] MPa

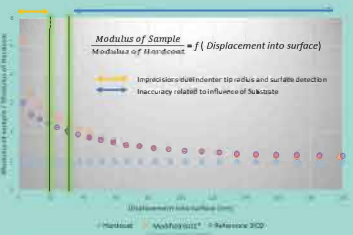
Variability due to :
- Determination of first Cracking occurrence
- Determination of Steady state

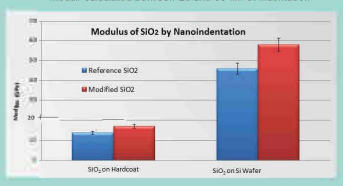
Identification of layers after tensile test by EDS



Nano-indentation

Experiments performed with a Berkovich tip on Nano-Indenter XPIII
Indentation Depth : 200 nm
Assumed Poisson Coefficient : 0.18
Moduli calculated between 20 and 30 nm of indentation

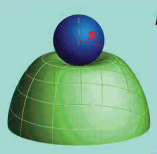




AFM

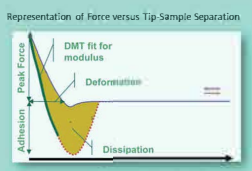
$$F - F_{adh} = \frac{4}{3}\frac{E}{(1-\upsilon^2)}\sqrt{R}\,\delta^{3/2}$$

F = Force in pN
F_{adh} = Adhesive Force in pN
E = Young's Modulus in MPa
υ = Poisson's ratio of the sample
R = Radius of the indenter in nm
δ = Indentation Depth in nm



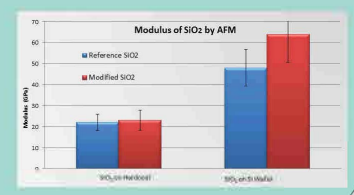
V. DERJAGUIN, V. M. MULLER et Yu. P. TOPOROV , Effect of Contact Deformations on the Adhesion of Particles, Journal of Colloid and Interface Science, 53(2):314-326, 1975.
D. Maugis, Contact, Adhesion and Rupture of Elastic Solids (Springer-Verlag, Berlin, 2000).

Representation of Force versus Tip-Sample Separation



Peak force QNM Application Note # 128 – Bruker

Experiments performed using Peak Force QNM on Dimension Icon
DNISPHS tip : 20 nm tip radius
Spring Constant : 436 N/m
Peak Force : 7100 nN
Indentation Depth : 10 nm



Acknowledgement : Mickael Febvre from Bruker for performing the AFM experiments

Conclusion and prospects

First micro-tensile experiments suggest that most noticeable cracks are located at the hardcoat/substrate interface. This was determined by EDS on fractured areas, after tensile stress. Modulus of modified SiO_2 was found to be roughly 20% higher than reference SiO_2 by nanoindentation. Considering the high standard deviation of moduli measured by AFM, no significant difference between moduli of modified and reference SiO_2 was observed by AFM. However, an important difference between moduli of SiO_2 on lens and on Si wafer was observed. This exposes unexpected influence of substrate on mechanical measurements using AFM, which has been hypothetically attributed to impact of Peak Force high frequency oscillations on viscoelastic substrates. Currently, an investigation to detect cracks at the interface of interest after micro-tensile test is ongoing. Other adhesion tests, such as micro-compression will be performed as well.