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Samples

Four samples were supplied by Morgan Technical Ceramics for determination of the mechanical properties of the coating using nanoindentation testing. Two of the samples were polymer substrates coated with DIAMONDSHIELD® coatings; the other two samples were the bare substrate materials consisting of CR39 and polycarbonate. The four samples are shown in Figure 1 mounted and ready for testing. The sample descriptions are listed in Table 1.

Figure 1: Four diced wafer samples mounted on a sample puck ready for testing.

Table 1: Sample descriptions

Samples
CR39 Substrate
Polycarbonate Substrate
4.4 µm DIAMONDSHIELD coating on CR39
5.4 µm DIAMONDSHIELD coating on PC

Test Protocol

All of the samples were tested at the Nanomechanics' Analytical Services Laboratory using the Nano Indenter G200 with the Continuous Stiffness Measurement (CSM) and Dynamic Contact Module (DCM) options. The CSM option was used to superimpose a small harmonic oscillation on top of the loading curve and it provided the data required to evaluate the evolution of mechanical properties as the diamond tip penetrated the sample. A test method titled "DCM CSM Hardness, Modulus for Thin Films" was used to test all of the samples, even though the bulk substrate samples were not coated – this test method has an option for specifying the sample as a bulk material as opposed to a thin film sample. The

Thin Films test method provides substrate independent results for thin films up to 40% of the film thickness by using the Hay-Crawford model to account for substrate influences [1]. All of the tests were performed using the inputs listed in Table 2.

Table 2: Test Parameters for the *Thin Films* Test Method

Test Parameter:	
Depth Limit	500 nm
Strain Rate Target	0.1 1/s
Harmonic Displacement Target	1 nm
Poisson's Ratio, film	0.25
Poisson's Ratio, substrate	0.35
Young's Modulus, substrate	2.91, 4.79 (CR39, PC) GPa
Film or bulk (1 for film; 0 for bulk)	1 for coated samples 0 for bulk substrates
Frequency Target	75 Hz
Film Thickness	5400, 4400 (CR39, PC) nm

Results

Ten tests were performed on each sample and any aberrant tests were excluded from the results. The tabulated results for the four samples are shown in Table 3. Tests results were obtained by averaging the measured properties over a small range of penetration depth. For the substrate materials, the range over which the properties were averaged was between 95 nm and 105 nm of penetration. Results for the coated samples were averaged over different ranges because the properties of hardness and elastic modulus are affected differently by substrate influences; the elastic modulus measurement is affected very early on during an indentation test because this results is a direct effect of the elastic stress field which propagates endlessly - to a limit at which it can be assumed negligible - throughout the sample during an indentation test. The hardness result is affected by the plastic stress field which is confined to the small volume of material around the tip of the indenter. Therefore, hardness is much less affected by underlying substrate material than is elastic modulus and is typically measured at deeper penetration depths. The results for elastic modulus of the coated samples were averaged between 0.5% and 1.0% of the film thickness while the hardness was averaged between 80 nm and 100nm of penetration.

Figures 1 and 2 show the results for the elastic modulus and hardness, respectively, for the two substrate materials and the two coated samples as a function of penetration. Both substrate materials show excellent repeatability by examination of the one standard deviation error bars shown on the graphs. The polycarbonate sample shows a slight decrease in the measured elastic modulus as the surface was penetrated, which indicates that there is a “skin” effect on this sample due to processing; Figure 4 shows that the drop in elastic modulus occurs after 150 nm for the polycarbonate sample. These samples were analyzed as bulk samples and the results of elastic modulus for the CR39 and polycarbonate samples were used as inputs for the thin film samples.

The results for the coated samples are also shown in figures 2 and 3. The results for the elastic modulus of the sample are clearly being affected by the substrate material after 80nm of penetration; this drop is caused by an error in the calculation of the contact area during the tests because of the extreme differences in the properties of the coating and substrate. The Hay-Crawford model works well with the standard Oliver-Pharr analysis for determination of the contact area for all soft films on hard substrates and for hard films on soft substrates when the film’s elastic modulus is not more than 2X the substrate modulus [1], [2]. The advantage for these coated samples is that the coatings are very thick and, therefore, the properties at shallow penetration depths less than 80 nm are minimally affected by the substrate material. The results reported in Table 3 for the coated samples are averaged over ranges less than 80nm of penetration.

Table 3: Averaged test results for the elastic modulus and hardness of the four samples.

Sample	Elastic Modulus	Hardness Average
	GPa	GPa
PC - Substrate	4.82 ± 0.14	0.4 ± 0.02
DiamondShield - PC	11.61 ± 1.6	1.24 ± 0.26
DiamondShield - CR39	11.04 ± 0.78	1.09 ± 0.13
CR39 - Substrate	2.91 ± 0.02	0.16 ± 0.002

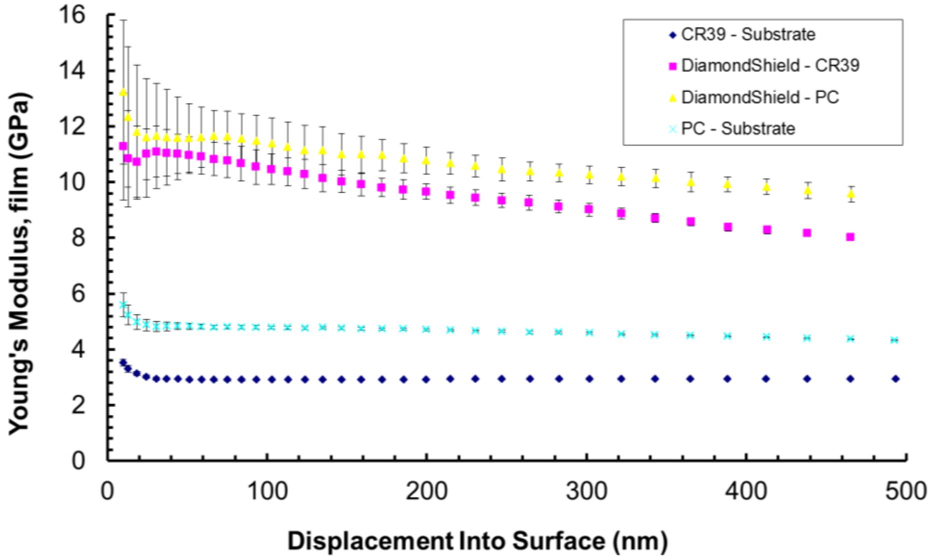

Figure 2: Young's modulus (elastic modulus) of the four samples (coated and uncoated) as a function of penetration into the test surface.

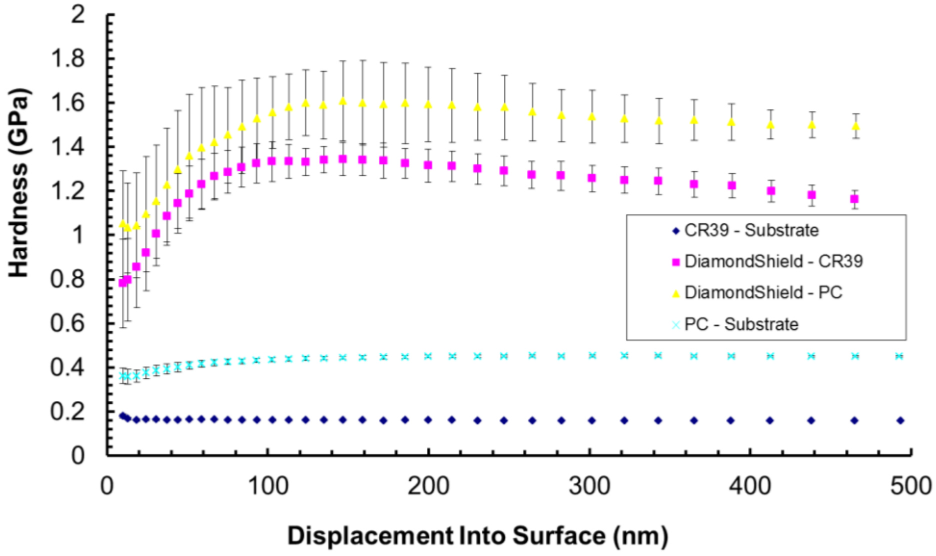

Figure 3: Hardness of the four samples (coated and uncoated) as a function of penetration into the test surface.

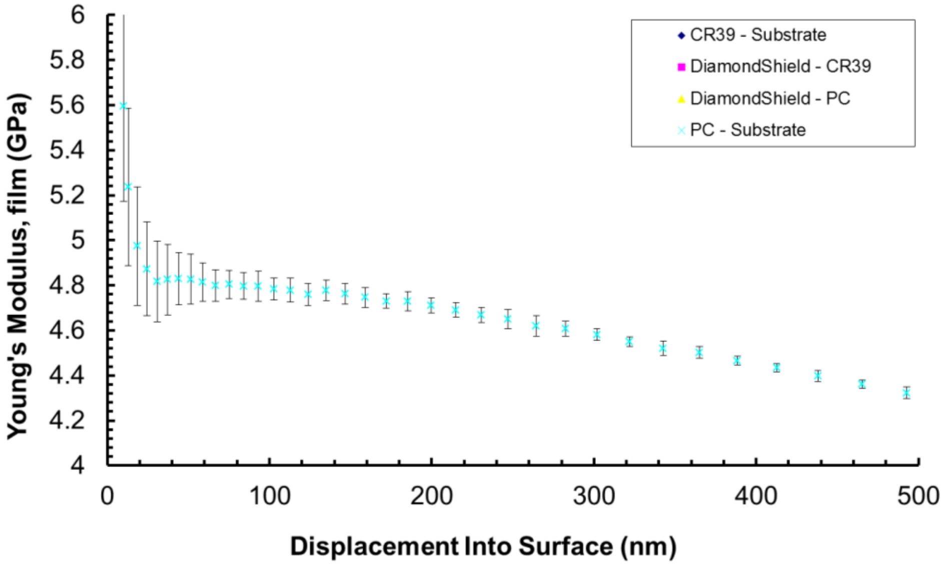

Figure 4: Drop in the elastic modulus for the polycarbonate sample after 150 nm of penetration showing a "skin" effect probably caused by processing.

References

[1] J.L. Hay, "A new model for measuring substrate-independent Young's modulus of thin films by instrumented indentation," Agilent Technologies application note (2010).

[2] W.C. Oliver and G.M. Pharr, "An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments," *J. Mater. Res.,* 7(6): 1564–1583 (1992).