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Mechanical Performance of UV/Ozone-Treated PDMS by AFM and JKR Testing Across the Length Scales

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Abstract – The Young's modulus of cross-linked poly(dimethylsiloxane) (PDMS) surface was quantitatively investigated across the length scales as a function of UV/ozone treatment time. The modulus value increased with increasing treatment time as observed by AFM as well as by JKR contact mechanics (Adhesion Testing Device, ATD), which we explained by the gradual formation of a silica-like layer at the sample surface with increasing treatment dose. For all specimens tested, the modulus values obtained were highest from AFM, lower from ATD, and lowest from bulk tensile experiments for the same UV/ozone dose. These results demonstrate the effect of the probed length scale of the tests used on the mechanical performance observed.

Cross-linked PDMS finds applications in several areas such as thin films and coatings, soft lithography, biomedical applications, optical systems, nanotribology, nanofluidics, MEMS/NEMS, etc.[1] These applications require an understanding and precise, quantitative characterization of the mechanical properties of PDMS from the nanometer to the macroscopic length scales. In our previous studies,[2] the changes of surface chemistry and ionization state of PDMS exposed to UV/ozone irradiation have been reported. It has been shown that oxidative cross-linking via Si-O bridges caused the gradual formation of a continuous "silica-like" barrier layer. This thin silica-like layer should affect the mechanical properties of the treated PDMS surface. In addition, as the characteristic dimensions of the mechanical tests are reduced, the observed effective surface properties may greatly vary. Despite the widespread use of untreated and surface treated PDMS, the characteristic length scale effects on mechanical properties have not been quantitatively studied. Hence, in this work we address the variation in modulus related to the indentation size, indentation depth, and treatment dose.

In this study, two indentation instruments, i.e. atomic force microscopy (AFM) and adhesion testing device (ATD) were chosen to investigate the influence of surface treatment upon the elastic modulus of PDMS obtained from nano and micro indentations. Advances in AFM enable the quantitative examination of surface mechanical properties with substantially improved force and spatial resolution. Here, an analysis method, partly derived from the Sneddon model, and taking into account adhesion by using a hyperboloid tip, was applied. The ATD technique provided data on the elastic modulus at the micrometer length scale. The value of the average bulk modulus was obtained by tensile tests. Values of the Young's moduli obtained with measurements with different techniques, were summarized in Table 1. The elastic moduli varied with the size of the characteristic dimensions of the experiments. In addition, the values of surface elastic moduli increased with increasing treatment time. The variations in the mechanical performance were interpreted on the basis of a structural model assuming the formation of a stiff silica-like layer at the top of the treated films. In general, indentation tests of surface mechanical properties are reasonably robust. However, the results must be interpreted with care and attention to the limitations of the analysis and instrument, keeping also in mind the type of material, and the length scale of the study.

Table 1. Young's elastic modulus obtained across different length scales. Si_3N_4 tip and lens were used as indentor.[3]

Treatment Time (min)	**Nano Indentation by AFM**		**Micro Indentation by ATD with Si_3N_4 lens**		**Tensile Test** (MPa)
	Young's Modulus (MPa)	Maximum Indentation Depth (nm)	Young's Modulus (MPa)	Maximum Indentation Depth (μm)	
0	6.6 ± 0.4	86	3.1 ± 0.6	3.0	2.5 ± 0.2
15	24.7 ± 4.0	19	8.6 ± 0.8	2.8	-
30	49.7 ± 4.7	13	9.0 ± 1.2	2.5	-
60	110. ± 6.	9	13.1 ± 2.5	2.4	2.4 ± 0.3

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