Analysis of Soft Materials Using Microindentation and Nanoindentation

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Abstract

Although generally displaying a non-linear behavior, biological soft tissues are commonly characterized using mechanical material parameters such as hardness and elastic modulus. This simplified approach is extremely useful in the clinical routine since the analysis of the properties of soft tissues can lead to better methods of detection and treatment of disease. Examples of this approach are arterial elastography and arthroscopy indentation testing.

In the present work a method is investigated for measuring soft materials' response to microindentation. The feasibility of said method was tested on a soft material whose material properties mimic those of soft tissues and in particular those of arteries. The tissue response was characterized by means of digital image analysis, and the behavior observed was further validated by comparison with nanoindentation tests.

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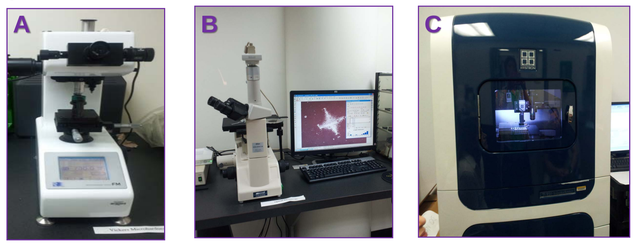

Figure 1. Equipment used: A) Microindenter; B) Microscope; both at NYU-Poly; C) Nanoindenter, at NYU Dental School.

Materials and Methods

All dry and treated samples were tested on substrates consisting of 30mm-diameter discs of between 12mm and 18mm thickness of PolyFast Phenolic hot mounting resin. Discs were machined to remove wells 20mm in diameter and 1.0 mm to 2.0 mm deep (Fig. 2A). Samples of FDA-Compliant 40A durometer rated silicone rubber were cut to fit in wells, and cyanoacrylate was used to affix samples into substrate wells (Image 2C).

Sample treatments consisted of 15-18 hr immersion in PBS (Phosphate Buffered Saline) at 5 C, followed by 1-2 hr desiccation at 25 C. This simple procedure's result is a microfine salt residue layering the surface of the sample. Microindentations performed subsequently on the treated samples produce a highly visible, repeatable and measurable imprint which digital imaging methods can demonstrate correlate with applied load and dwell time.

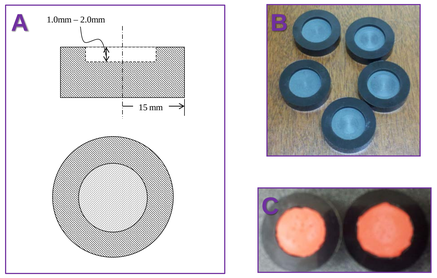

Figure 2. Specimen holder design and realization: A) technical drawing; B) empty holders; C) specimen ready for testing.

Treated samples underwent microindentations by a Future-Tech Digital Microhardness Tester FM-700 with loads ranging from 491mN to 9820mN at dwell times between 60s and 100s.

Treated and untreated samples were also analyzed using a Hysitron TI-950 Triboindenter applying trapezoidal loading functions with hold times of 80s and loading and unloading times of 10s. Peak electrostatic forces of 70μN and 100μN resulted in peak loads of 15 μN and 20 μN respectively. This mechanical testing procedure is consistent with Ebenstein's. Digital imaging analysis was performed by special code developed for MatLab Compiler Runtime.

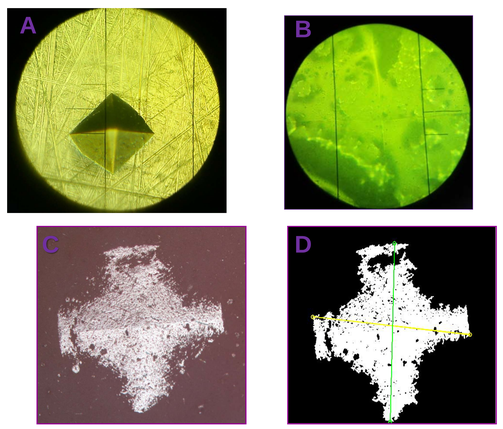

Figure 3. Image capture from microindenter tests: A) normal microindentation test on metal; B) microindentation on a dry silicone sample; C) microindetation of a treated sample; D) digital image analysis of a treated sample, the lines connect the extremal points in the horizontal and the vertical direction, respectively.

Results

Digital image analysis of saline solution-treated samples showed that for soft materials, it is possible to track the effects of microindentation. The relationship established between the loading increase and the larger area measured suggests the possibility of a linkage between behavior under micro-scale conditions and nano-scale conditions.

Also of note is clear evidence of adhesion between sample and indenter tip, a property of viscoelastic materials (see Figure 4A). Further tests applying different variations of trapezoidal loading function may reveal additional details in viscoelastic nature of the sample.

These patterns combine to suggest the possibility of a linkage between behavior under micro-scale conditions and nano-scale conditions. Data which explains the effects of the treatment process on the material properties would be necessary to draw future conclusions, however.

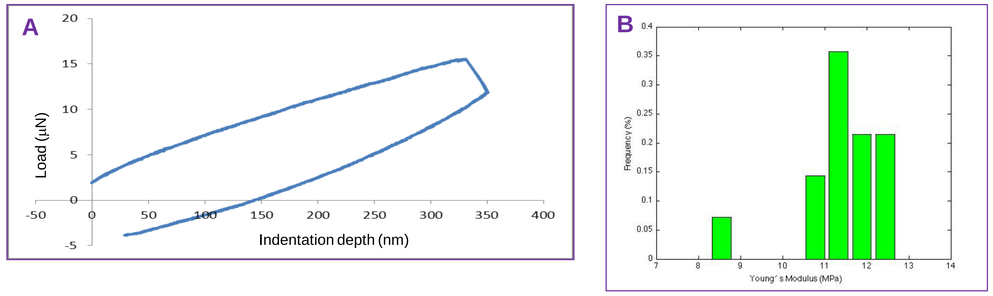

Figure 4. Nanoindenter results for a 70μN electrostatic force: A) force vs. depth of indentation graph; B) histogram showing the distribution of the results.

	N of samples	E (MPs)	H (GPa)	Peak electrostatic force (μN)	Peak load (μN)
Dry, untreated sample	14	11.4 ± 1	3.9 ± 1.4	70	9 ± 2

Table 1. Summary of the nanoindenter results for a dry, untreated sample.

Discussion and Conclusion

Adhesion between the indenter tip and sample has been discussed in numerous studies on soft material analysis and clearly presents a complication in each of the data. As the compliance method uses data from the peak of the unloading portion of the curve, this "negative force" behavior does not play a role in this analysis. Understanding that adhesion has been shown to confound the compliance method by reporting a larger value of sample modulus, more complex models must be applied to achieve better estimates of modulus.

The adhesion behavior is evidenced in nanoindenter testing of agarose, gelatin and arterial tissue. The results of this study therefore indicate that this silicone rubber may be a useful control in which to observe adhesion behavior, albeit on a different scale of force than typically evidenced by soft tissue in vivo.7

The correlation between micro-scale loading force and area of salt residue in treated samples gives indications that the imaging method of hardness testing may be possible for soft materials. Once data is gathered for the effects of treatment on soft materials, correlations can be drawn between the results of this novel approach to soft material imaging and material properties of the samples.

Bibliography

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Acknowledgements

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