Advanced Nanoindentation of Viscoelastic Properties in Soft Biomaterials

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Introduction: Understanding the mechanical behavior of soft biomaterials is essential to the development of these materials and the devices in which they are used. In recent years, investigating these systems at a degree beyond the traditionally available macroscopic methods has become a great focus. Various methods are currently used to characterize the mechanical behaviour of biomaterials, commonly either dynamic tests or quasi-static tests (such as creep or stress relaxation tests). Such methods have major drawbacks: (i) the sample often needs to be of a specific shape and (ii) sample fixation can be problematic, especially for materials with high viscoelasticity. Nanoindentation is particularly appropriate because it is unaffected by these two limitations and allows the characterization of very small material volumes. However, the main drawback of nanoindentation tests is linked to the low thermal stability of most instruments. These instabilities introduce an uncontrollable penetration drift which, when coupled with the viscoelastic deformation of the sample, produces a composite response which is a mixture of that of the instrument and that of the sample. For many biomaterials, such limitations induce significant error in the measured mechanical properties. This paper presents alternative methods of performing nanoindentation in order to gain quantitative and meaningful data on such materials.

Methods: Nanoindentation measurements were performed on a range of challenging soft biomaterials using a CSM Instruments Ultra Nanoindentation Tester (UNHT). Various methodologies were used to demonstrate how the true mechanical properties of materials having significant viscoelasticity can be measured. Materials tested comprised hydrogels, PDMS gels, bulk polymers and canine cartilage. Both pyramidal (three-sided Berkovich) and spherical diamond indenters were used with indentation loads in the < 10 mN range.

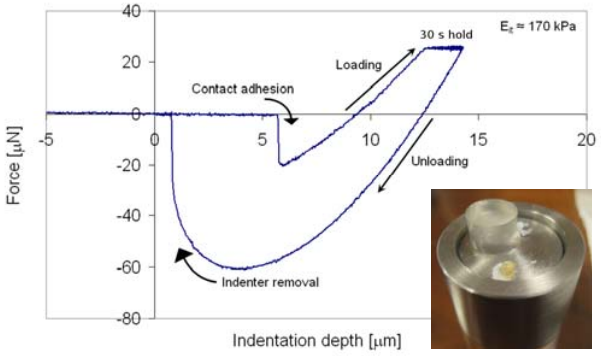

Figure 1. Example of a nanoindention result on a very soft hydrogel (E = 170 kPa) showing the adhesive "snap-in" upon contact and the "pull-off" upon retraction. Inset photo shows a fully hydrated hydrogel sample next to a fully dehydrated sample (95% shrinkage).

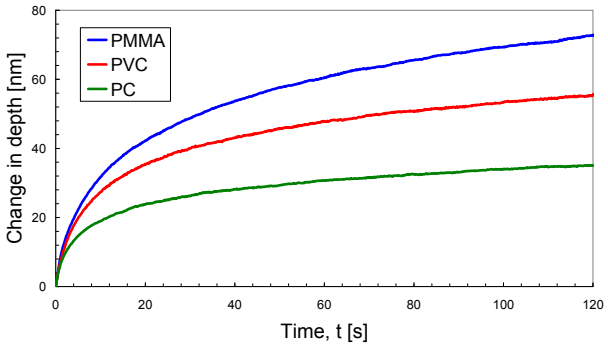

Figure 2. Creep curves measured for three different polymers over a 2 minute pause under a 1 mN constant load.

Results: Nanoindentation results are presented for different combinations of loading/unloading rate, maximum applied load and pause time at maximum load. Fig. 1 shows a typical result on an ultra soft hydrogel where adhesive forces at contact are significant, both during loading and unloading. In this case, a spherical indenter with radius of 100 µm was used in order to investigate this phenomenon with a large contact area. Fig. 2 shows a series of creep curves on three different bulk polymers. These curves represent the change in the depth signal over a 2 minute period whilst maintaining the indentation load at its maximum ramped value (1 mN). The exponential response of such creep curves allow the viscoelastic response of each material to be measured in an accurate manner and therefore provide useful information about creep dissipation in the material. Such results are made possible thanks to a quasi elimination of the thermal drift by the use of dedicated materials with very low thermal expansion coefficients and a unique design of the measurement head. Furthermore, the influence of frame deformation has been eliminated by an active top referencing system which continuously monitors the position of the surface of the sample through a reference applying a very small and controlled pressure. The indenter penetration depth is thus measured relative to that reference.

Conclusions: A series of relatively long term quasi static tests on several viscoelastic coating materials has been used to demonstrate the efficiency of a new nanoindentation instrument design and its ability to almost totally eliminate thermal drift. This study therefore demonstrates that nanoindentation testing, when performed in good conditions with appropriate apparatus, constitutes a reliable tool to study the time-dependent mechanical properties of biomaterials.

References:

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