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Ultra low load indentation studies of creep and time-dependent properties of elastomers and polymers

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Abstract – Indentation experiments on time-dependent and creep properties of several types of soft materials such as polymers and gels have been performed using a new low load indentation system with extremely low thermal drift. Thermal stability of the indentation instrument is a key factor that strongly affects measurement of viscoelastic properties and therefore its elimination is of primary importance. The results of the presented study show that the new indentation system can be used for characterization of viscoelastic properties of various soft and very soft materials where long term indentation is required.

Characterization of soft materials such as polymers and elastomers by indentation has always been a challenging task because of their time-dependent mechanical behavior. Their viscoelastic properties are characterized by storage and loss moduli whereas response of the material to constant load is described by its creep properties. Use of experimental techniques with perfect thermal stability is required so that results are not discredited by the most important source of error in indentation which is thermal drift [1], i.e. change in displacement signal while a constant load is applied on a stable material. CSM Instruments has recently developed a depth-instrumented indentation system with extremely low value of thermal drift, commercially named Ultra Nanoindentation Tester (UNHT). It is based on a unique patented active surface referencing system [2] and is perfectly suited for long term measurements.

This paper presents the results of a study on several types of polymers and elastomers that explain the new methodology used for creep and viscoelastic properties measurements. Experiments were performed in force controlled mode with maximum load varying between 50 µN and 1000 µN with Berkovich or spherical indenters. Materials were selected so that a broad range of properties are covered, from rather hard polymers (PMMA, PC and PVC) to elastomers and gels. In addition, characterization of the elastomer with polymer layer of various thicknesses was performed using spherical indenters. The indentation procedure consisted of controlled indentation at given loading rate, hold on the maximum load for several minutes and unloading at the same rate. The loading rate was varied and its effect on creep was studied. The thermal drift of the instrument was determined by indentation at several loads on fused silica. It was found that increasing of the loading rate leads to increase of creep during the hold period of both polymers while there was only minor influence on the creep of the elastomers and gels. Hardness and elastic modulus could be relatively well calculated using the standard formulae for both polymers but different approaches had to be used for the elastomer and gels since the residual imprint depth was almost immeasurable. Spherical indentation on the elastomer with various polymer coatings was closely related to this problem and solution was found in comparing the indentation force-displacement plots and calculating 'apparent' hardness by dividing the maximum force by the maximum depth. Such criterion allowed characterization of even such complicated systems as the very soft elastomer with hard polymeric coating. The results show the easy analysis of time-dependent and creep properties of a wide range of soft and very soft materials. The methodology was further developed for evaluation of hardness of more complicated coated systems where the two components have radically different mechanical properties.

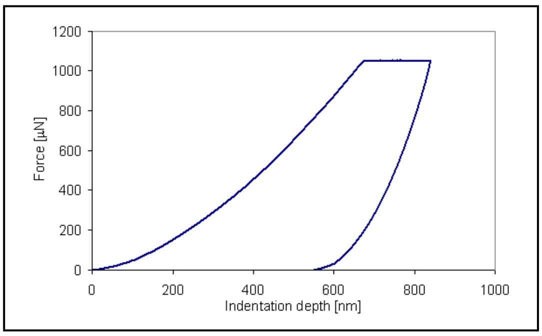

Figure 1: Creep of PMMA polymer during indentation with 120 s hold at 1 mN force.

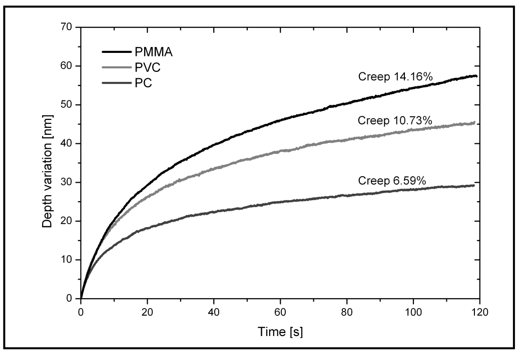

Figure 2: Different level of creep for the PMMA, PVC and PC polymers during 120 s holad at 1 mN.

[1] T. Chudoba and F. Richter, *Surf. Coat. Technol.* **148**, 191 (2001)
[2] J. Woirgard, B. Bellaton and R. Consiglio, US Patent No. WO 2006/069847 A1, July 6, 2006