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MICROINDENTATION OF SOLIDS WITH VISCOELASTIC RESPONSE

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Summary: *This paper presents the results of the introductory part of a proposed project aimed at identifying long-term time-dependent mechanical characteristics of viscoelastic materials on the basis of microindentation data. Indentation simulations using ANSYS FEM probabilistic modelling and some experimental data based on MTS XP system are introduced.*

1. Introduction

The purpose of microindentation hardness testing is to study fine scale changes in hardness, either intentional or accidental. The applied load and the resulting indent size are small relative to bulk tests. Microindentation testing methods have a number of advantages as a modern static nondestructive investigation approach to the determination of mechanical properties: small amounts of material are subjected to investigation, and the technique and its implementation are simple. Microindentation parameters are sensitive to structural parameters and also to mechanical behaviour. In recent years, micro and nanoindentation techniques for measuring the Young's modulus of linearly elastic materials, hardness and yield strength of elastic-plastic materials have been very well established for time-independent materials (Giannakopoulos, 2006).

However, the techniques developed for time-independent materials cannot be suitably used in the case of time dependent solids. Cheng et al. (2005) derived analytical solutions for linearly viscoelastic deformation, and provided a method for measuring viscoelastic properties using a flat-punch and spherical tip indenters. In the paper by Lu et al. (2003), techniques for measuring the linear creep compliance of solid polymers in the glassy state using nano-indentation with Berkovich and spherical indenter tips were validated. The Berkovich indenter is modelled as an axisymmetric conical indenter with an effective cone angle that provides the same area-to-depth relationship as the actual pyramidal indenter. Then Sneddon´s elastic solution for the relation between indentation depth h and indentation load P on an axisymmetric indenter can be used to solve this moving boundary problem in linear viscoelasticity theory. Instead of the correspondence principle between a linearly viscoelastic solution and a linear elastic solution, hereditary integral operators are used based on Sneddon´s associated solution (Riande et al. (2000)). This approach enables us to find the creep compliance in shear $J(t)$ under prescribed loading histories for a conical indenter with the angle between the cone generator and the substrate plane α and a spherical indenter with the radius R

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$$J(t) = \frac{8h}{\pi(1-\nu)\tan\alpha} \frac{dh}{dP} \qquad \text{conical indenter} \qquad (1)$$

$$J(t) = \frac{4\sqrt{R}\,.\,h^{1/2}(t)}{(1-\nu)} \frac{dh}{dP} \qquad \text{spherical indenter} \qquad (2)$$

Both cases require computation of derivative of indentation depth with respect to indentation load, which evokes the elastic solution for Young's modulus with all inherent problems.

In addition to the representation of viscoelastic properties in the time-domain, the material properties of viscoelastic materials can also be represented by complex material functions in the frequency domain. Loubet et al. (1995) proposed a method for computating the complex modulus of viscoelastic materials. The method uses data acquired from an MTS nano indenter XP system installed with a continuous stiffness measurement (CSM). CSM allows cyclic excitation in load or displacement, and records of the resulting displacement or load. In this way the CSM system provides information on the dynamic viscoelastic behaviour in a time domain usually less than 1s.

Numerical simulations of indentation processes were first developed for higher loadings and spherical indenters. Many authors learned about negligible influence of wear and adhesion between indenter and sample on simulation results for different forms of indenters (see for example *Huang et al*. (2005), *Bláhová* (2007)).

The aim of this paper is to point out some preliminary steps for converting microindentation data to mechanical viscoelastic properties by experimentally checked numerical simulations. The results introduced here represent the introductory part of a wider project being prepared by the authors.

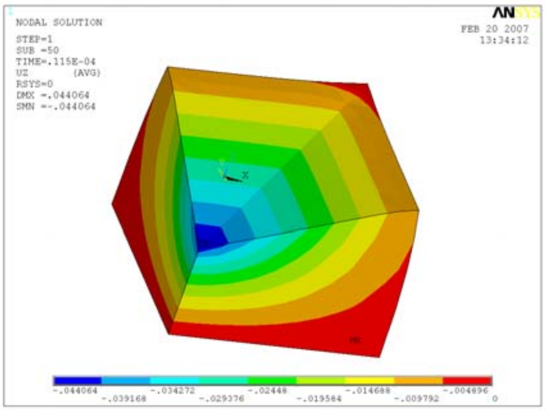

Figure 1. Indentation simulation by ANSYS FEM modelling

2. Numerical simulations

ANSYS offers the complete range of linear and nonlinear material models required for accurate representation of material behaviour. An advanced curve fitting toolkit offers accurate conversion of experimental nonlinear material data from to a mathematical model for analysis. A comprehensive list of material models supported by ANSYS includes also rate-dependent materials with viscoelastic and creep processes. This part of the package was used for parametric modelling in 3D.

Indentation of a bulk material can be considered as a process of indenting a half-space with a rigid indenter (Figure 1). The strains and stresses during indentation were analyzed by FEM using a probabilistic approach. Ideal kinematic and mechanical behaviour was assumed during the test, and so the condition of axisymmetry was applied. The wear and adhesion between indenter and sample were neglected. Viscoelastic isotropic material of the half-space was assumed and the indenter was characterized as a rigid material. An epoxy matrix with known mechanical characteristics (see e.g. Minster & Hristova (2005), (2006)) was chosen as a model material. Long-term mechanical characteristics were defined in the form of the Prony series (see Figure 2). Scatter of the input variables (±10%) was assumed in the sensitivity analysis of the scatter of the input variables to the deformation of the half-space in the *z* direction (Figure 3).

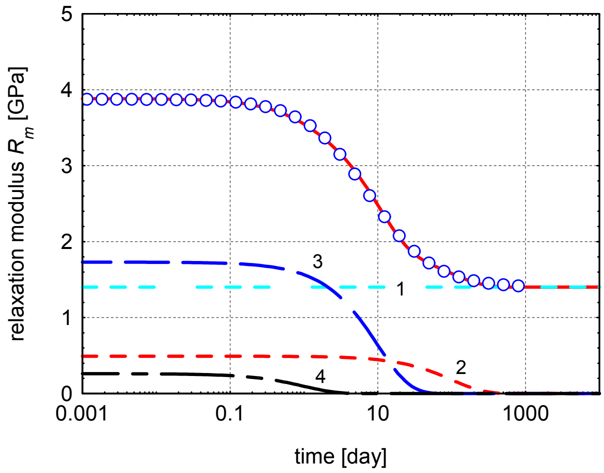

Figure 2. Relaxation modulus of the epoxy matrix R_m *(t)* as four-term Prony series

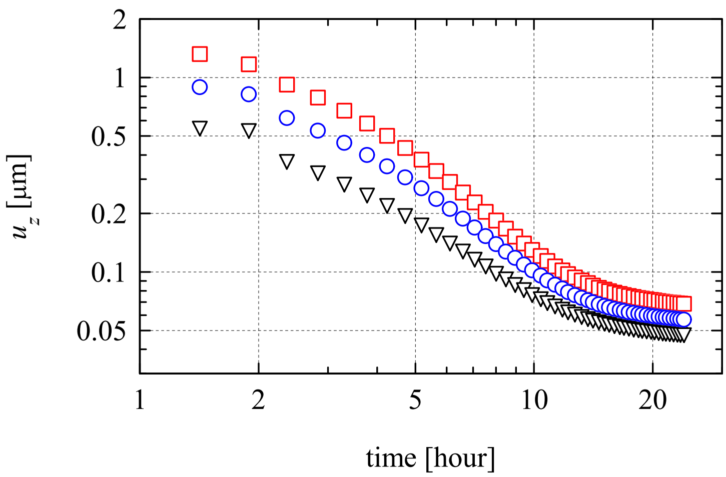

Figure 3. Simulation of the depth histories u_z (*t*) for Vickers indent and unit unloading of the epoxy matrix characterised by three different sets of input mechanical characteristics: (i) average, (ii) average +10%, and (iii) average – 10%

Indentation is a three-dimensional problem and can induce deformations in both the linearly and nonlinearly viscoelastic regimes. Practically full recovery in a time interval of one day (see Figure 3) can serve as an indicator of linearity (Lu et al. (2003)) corresponding with small deformation regime.

3. Measurements

Application of the Nano Indenter XP enables us to receive quantitative data on Young's modulus, conventional microhardness, contact depth, stiffness and area during loading-unloading, and also data on creep and relaxation properties during dwell time intervals. In addition, by so called continuous stiffness measurement (CSM technique)), the system allows quantitative measurements of hardness and modulus to be made during the loading cycle at every data point acquired.

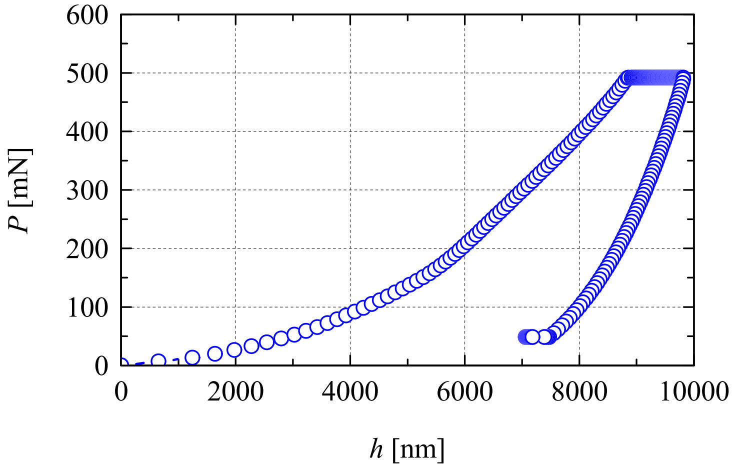

Figure 4. A typical ramp loading-unloading diagram using MTS XP (Epoxy matrix, maximum load 50 [gf], allowable drift rate 0.05 [nm/s], time to load 15 [s], peak hold 60 [s], T= 22^{o}C)

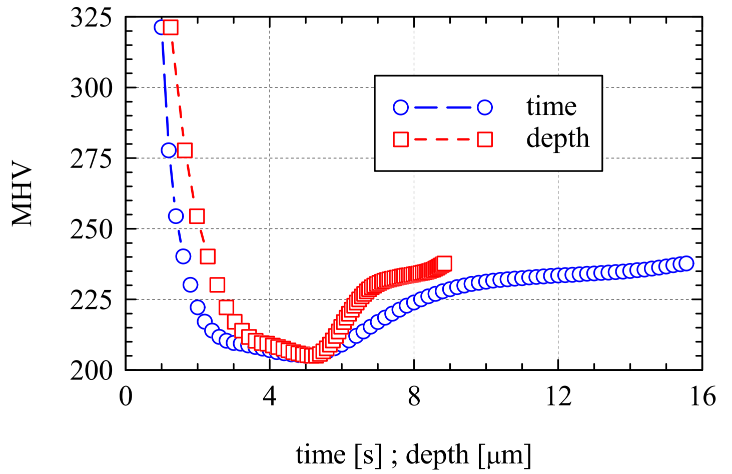

Figure 5. Dependence of Vickers microhardness on indent depth (time) during loading (MTS XP, epoxy matrix)

With the CSM method, the indenter applies a load to the indenter tip and simultaneously superimposes an oscillating force with a force amplitude several orders of magnitude smaller than the nominal load. The resulting amplitude and phase relation between load and displacement oscillation then provides direct and accurate measurements of the contact stiffness at all depths and also of the uniaxial storage modulus and the loss modulus for viscoelastic materials (i.e. information on the viscoelastic behaviour in a time domain less than 1s). Typical results of the epoxy matrix examination using the MTS XP system are shown in Figures 4-6. Fig. 4 shows a ramp loading-unloading diagram, and Fig. 5 shows the dependence of Vickers microhardness on indent depth (time) during loading shows.

Figure 6 illustrates the history of relaxation modulus *E (t),* derived with the use of equation (1) for creep compliance in shear, and the history of depth *h (t)* during loading. The results show evidence of surface layer (thickness about 6 μm) of investigated sample with less microhardness and a lower relaxation modulus. Figure 7 compares the influence of dwell time intervals (peak hold) on Vickers microhardness values previously measured conventionally using the Anton PAAR tester and continuously using the MTS XP tester. As in this case, the results presented in Figures 4-6 are also well comparable with previously obtained experimental data (see Minster et al. (2004), Minster & Hristova (2005)).

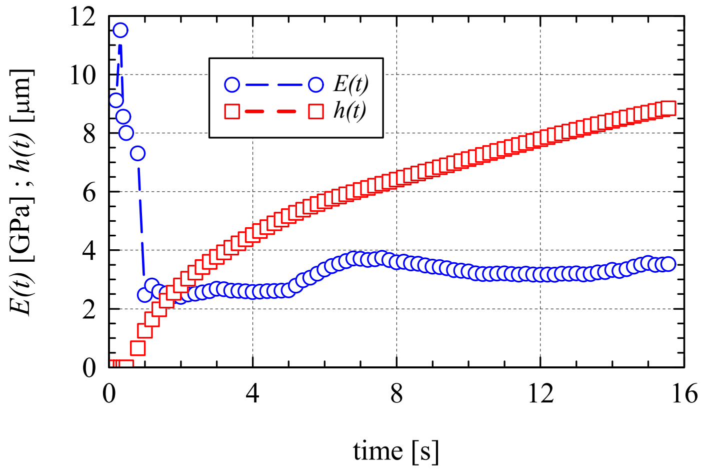

Figure 6. Histories of relaxation modulus *E* (*t*) and indent depth *h (t)* during loading (MTS XP, epoxy matrix)

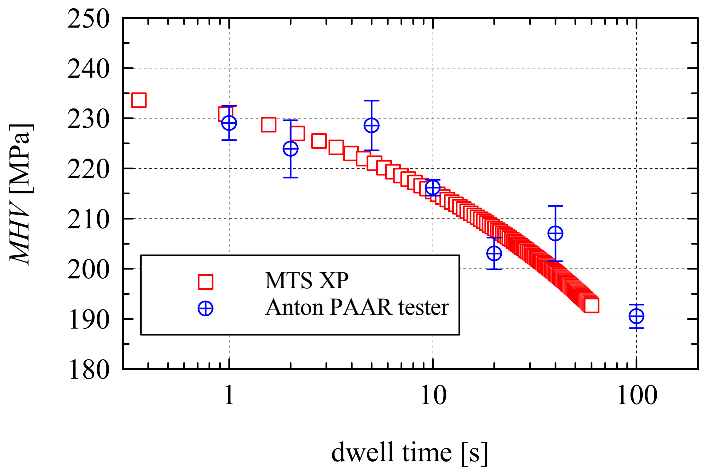

Figure 7. Influence of dwell time intervals on Vickers microhardness values (Epoxy matrix, *P*=25 [gf], *dP/dt*=5 [gf/s] (A. Paar tester), *T*=21.7±0.6 [^{o}C])

Conclusion

A combination of instrumented measuring of hardness and material parameters during indentation, providing us with the whole time-dependent story (properties versus depth), and Monte Carlo numerical parametric simulations using FEM modelling can be used to find optimal long-term viscoelastic characteristics of solids. Our future efforts will be aimed at this goal.

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