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MECHANICAL CHARACTERISTICS OF RHEONOMIC MATERIALS USING MICRO- AND NANOINDENTATION

Jiří Minster[1], Olga Bláhová[2], Romain Le Clere[3]

Summary: *The paper deals with the disspersion of instrumented indentation measurements caused by state and properties of tested samples. It demonstrates the important influence of the temperature of the tested material, its viscoelastic behaviour, techology and microinhomogeneities on the instantaneous elastic modulus and microhardness values measured using the Nano Indenter XP and the microindenter MHT 10V.*

1. Introduction

The mechanical behaviour of rheonomic materials is highly dependent on the test conditions, including the level of strain, the strain rate, temperature, moisture and other factors. The elastic modulus of metals, ceramics and other important structural materials with elastic-plastic behaviour can be estimated from indentation measurements with the unloading data. However, rheonomic behaviour does not allow this possibility directly. On the basis on results the of instrumented micro- and nanoindentations into an epoxy matrix, this paper documents the influence of loading conditions, temperature, technology of the samples and their non-homogeneity on instantaneous elastic modulus values and microhardness values, in order to obtain more authentic data for future modelling and simulation.

2. Methods

The Nano Indenter XP was used to obtain quantitative data on Young's modulus, conventional microhardness, contact depth, stiffness and area during loading-unloading, and also data about creep and relaxation properties during short- and medium-term dwell time intervals. In addition, the system allows (by so called continuous stiffness measurement (CSM technique)) quantitative measurements of hardness and modulus to be made during the loading cycle at every data point acquired (Bláhová (2007)). Experimental microindentation tests were performed with an Anton PAAR (MHT 10V) Vickers microhardness tester with a videomeasuring system.

[1] Ing. Jiří Minster, DrSc. ITAM AV ČR, v.v.i., Prosecká 76, 190 00 Praha 9

[2] Doc. Ing. Olga Bláhová, PhD., University of West Bohemia Plzeň

[3] Romain Le Clere, Ecole Polytechnique Univerisitaire de Montpellier, Dép. Materiaux

3. Material

The selected characteristic representative of the materials mentioned above is an epoxy resin mix consisting of solvent-free low-viscosity bicomponent pigmented systems on the basis of a low-molecular epoxy resin with a content of non-toxic reactive diluents, additives, pigments, fillers and auxiliary admixtures, hardened by a cycloaliphatic polyamide hardener (Coming, 2003). The polymer composition behaves differently from the neat resin due to confinement. The material is used for surfacing a range of building substrates, such as concrete, cement screed, plaster, asbestos cement, cement-and-chipboard, steel and stone. It is well suited for the manufacture of self-levelling flooring top layers and can be blended with fillers to form trowelled polymer mortar or polymer concrete mixes. The material is hygienically harmless, resistant to temperature loads within the range of -30°C and +90°C, with a reserve, and can be advantageously applied to light, medium and heavy duty plants requiring floors with high mechanical and also chemical resistance, among others.
The prismatic samples 4x4x12 cm and plates 7mm in thickness were made by mold casting from one mixing, cured at room temperature and postcured at 80°C for 4 h. This type of postcure followed by slow cooling to laboratory temperature is indicated in the text as rejuvenation (REJ).

4. Results

The following results were achieved in the course of micro- and nanoindentation instrumented measurements:
4.1. *The experiments proved an increase in microhardness values with a reduced level of loading*. Fig.1 demonstrates a weak dependence of microhardness values on levels of loading in the interval $12 \leq P \leq 100$ [gf]. More importantantly there are marked differences in microhardness caused by different temperature and technology.

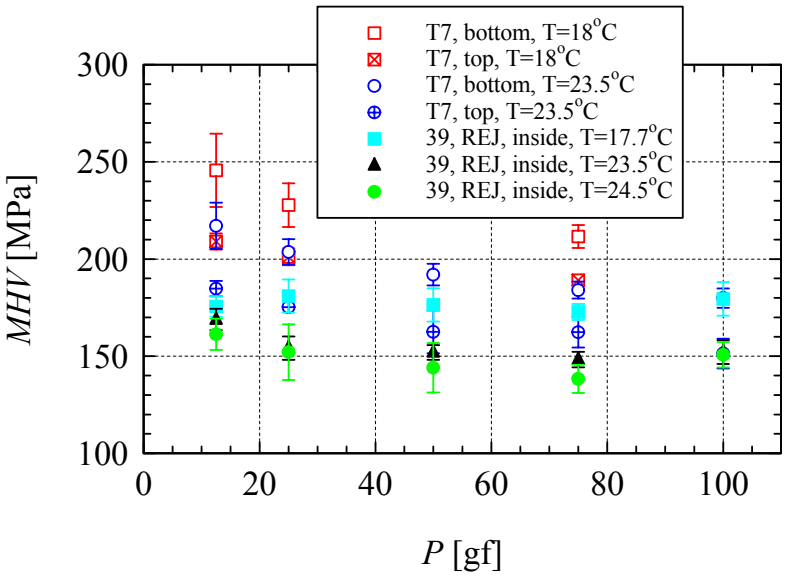

Fig.1 Influence of level of loading, temperature and casting technology on Vickers microhardnes values (epoxy matrix, A. Paar tester, t_d=20 [s], dP/dt=5 [gf/s])

4.2. *The constant-force indentation creep during dwell time causes a decrease in microhardness history values*. A comparison of the influence of dwell time intervals (peak

holds) on Vickers microhardness values measured continuously using the MTS XP nano tester, and conventionally using the Anton Paar microtester is shown in Fig.2. In the regression curve used here $MHV = A * t^{-K}$ constant A represents the microhardness before any recovery state of the material and coefficient K is a constant furnishing a qualitative measure for the recovery state of the material. The results presented in the figure show relatively good agreement between the types of measurement used, in comparison with nanoindentation, with higher levels of loading. The results are also well comparable with previously obtained experimental data (see Minster et al. (2004), Minster & Hristova (2005)).

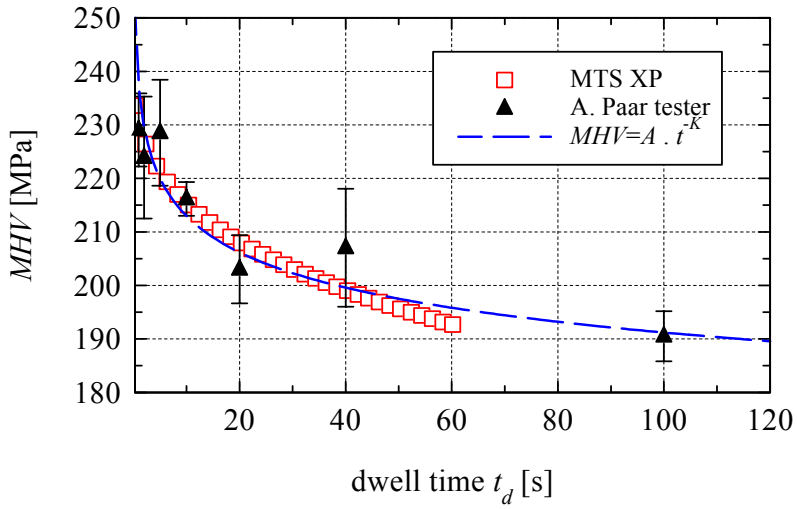

Fig.2 Comparison of the influence of dwell time intervals (peak holds) on Vickers microhardness values measured continuously using the MTS XP tester, and conventionally using the Anton Paar tester. (Epoxy matrix, MTS XP: maximum load 50 [gf], allowable drift rate 0.05 [nm/s], time to load 15 [s], peak hold 60 [s] ; A. Paar tester: P=25 [gf], dP/dt=5 [gf/s] ; T=21.7±0.6 [°C]; A=237, K=0.0469)

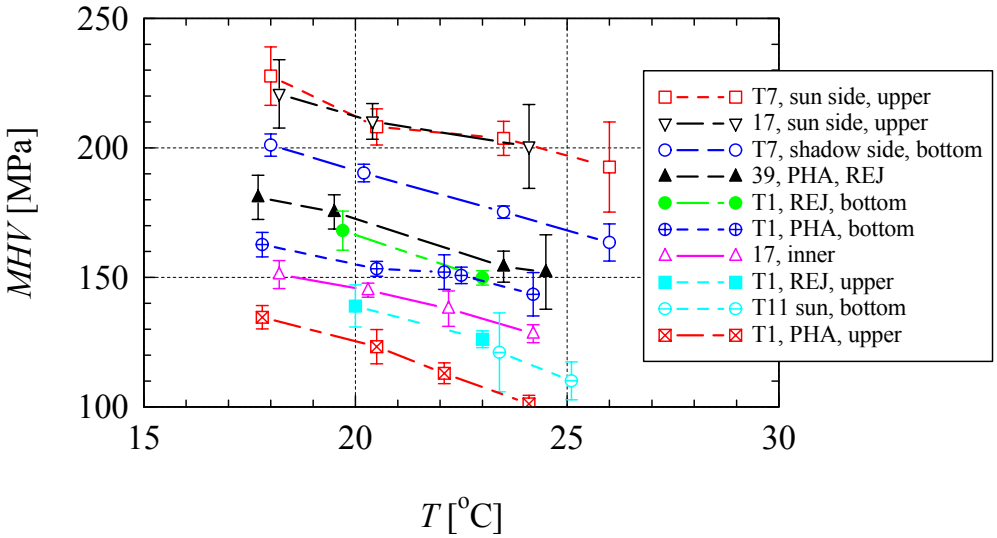

Fig.3 Influence of temperature on microindentation hardness of the Comflor epoxy composition (P=25 [gf], t_d=20 [s], dP/dt=1.25 [gf/s])

4.3. *The microhardness decreases by approximately 20% with rising temperature in the interval between 18 and 25 °C*, generally for all loading histories (including both physical

aging and rejuvenation). This predication is in agreement with results of previous standard measurements of instataneous elastic modulus (Minster and Hristova, 2004).

Similarly as in the case of the influence of loading level on microhardness values (see Fig.1), the casting technology and the nonmechanical loading history play a more important role.

4.4. C*asting technology gives rise to different microhardness values on the sample surface and in the bulk* (on the upper and bottom surfaces of the plate). Variations in Vickers´microhardness values due to the measurement location, according to different normal distances between a surface of the tested sample and indent mid points, are illustrated in Figs.4 and 5.

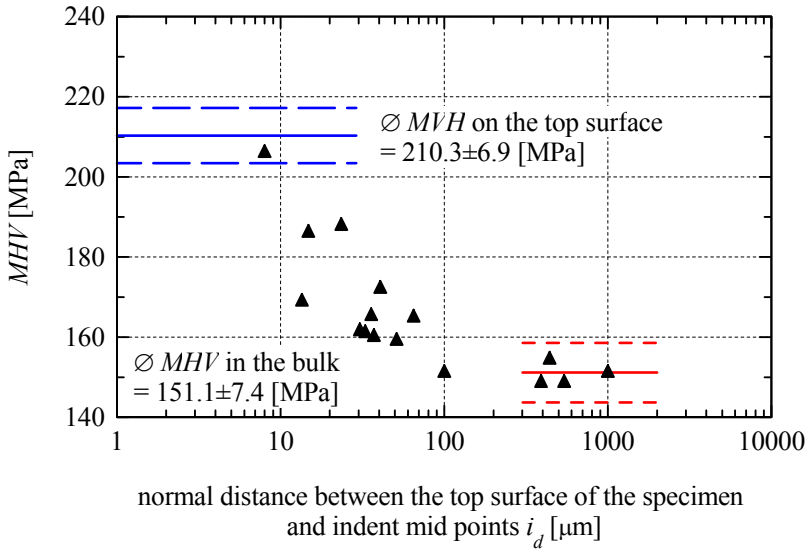

Fig.4 Vickers mikrohardness in the direction normal to the specimen surface (sample 17, T=21,7 °C], P=25 [gf], t_d=20 [s], dP/dt=1.25 [gf/s])

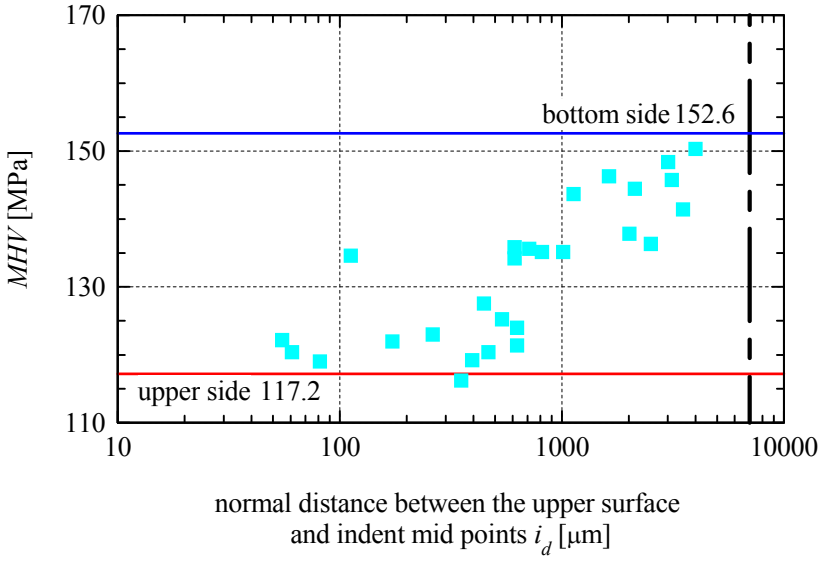

Fig.5 Vickers microhardness values between the upper and bottom side of a sample (sample T1, thickness 7 mm, 2 years physical ageing, T=21.5±1°C])

4.5. *The micro-inhomogeneity of the material is a source of sizeable experimental data dispersion*, particularly when the displacement into surface during nanoindentation is less than 2 μm. The average values are in agreement with expectations while standard deviations are relatively high (see Fig.6).

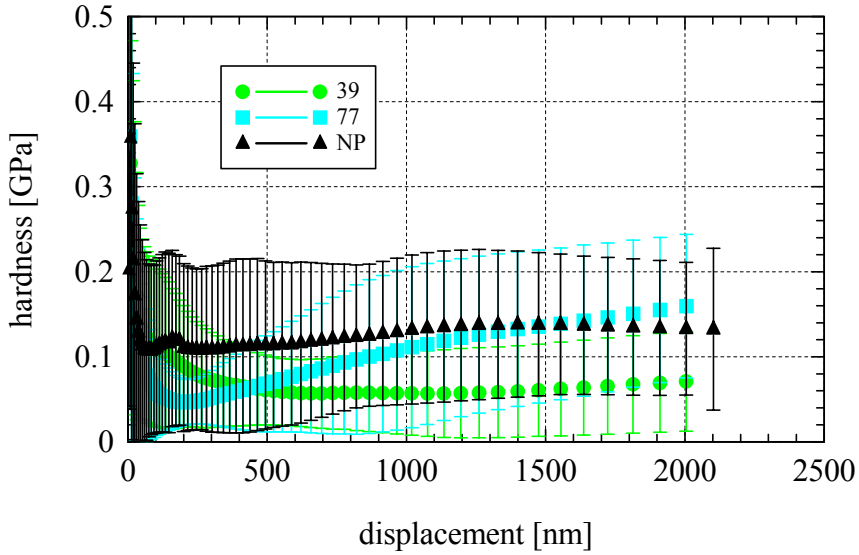

Fig. 6 Average Vickers´ microhardness values of three samples of the epoxy composition with different loading histories measured by the Nano Indenter XP (39: 4 years laboratory aeging, 77: rejuvenated, NP: 1 year of climate ageing)

4.6. *The initial part of the nanoindentation measurements (displacement into surface values less than 200 nm) is characterized by dispersion of data as well.*

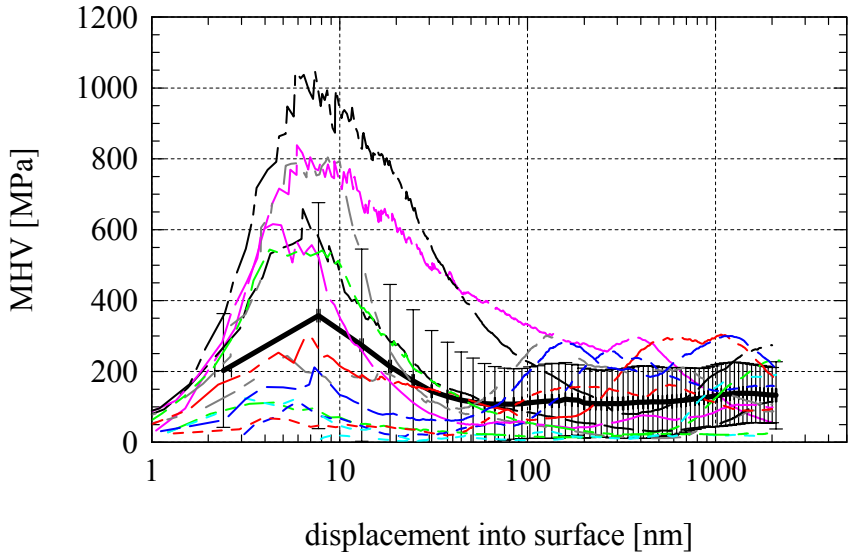

Fig.7 Vickers microhardness of the NP sample (individual measurements and average values with standard deviations) measured by the Nano Indenter XP

The measurement data with the Nano Indenter XP demonstrate that penetration depths of hundreds of nanometers or more can occur at very low force levels. This may be due to dificulties with identification the initial point of tip-sample contact (e.g. Vanlandingham et al. (2005)) or due to the quality and micro-inhomogeneous nature of the coats of the samples. All of these reasons can lead to artificial trends in measured modulus and hardness values (Cheng and Cheng (2004)).

5. Conclusions

Micro- and nanoindentation measurements of rheonomic materials are strongly influenced by test and sample conditions. The paper deals with measurement data disspersion due to the state and properties of tested samples. This demonstrates the important influence of temperature of the tested material, its viscoelastic behaviour, technology and microinhomogeneity on the instantaneous elastic modulus and microhardness values defined using instrumented indentation. In order to obtain reproducible data for future modelling and simulation, all possible influence factors have to be carefully taken into account.

Acknowledgement

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