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FACTORS INFLUENCING MICROINDENTATION MEASUREMENTS

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Abstract: *The factors influencing microhardness measurements are listed and their influence on the distortion of experimental results is discussed. Some of them which are widely known and commented in many articles, they are only mentioned. Others are discussed in details and their limits are changed as the particular study on their influence has been performed. Some factors are considered as parameters which influence the values to be determined giving additional information about the structural and mechanical properties of the materials.*
Key words: *Microhardness, Microindentation, Imprint, Polymer structure.*

INTRODUCTION

The microindentation measurements are very important because they give information about the technological and exploitation characteristics of the materials and their applicability for different purposes. The increasing information and experience in the field of microindentation investigating methods give the idea and opportunity to consider the micro- and nano-mechanical investigation as adequate tools for elucidating the structural peculiarities of polymeric materials. Unfortunately, the complexity of the molecular and supramolecular structure of the polymers as well as the structural imperfections, macro-, micro and nano-defects, the relations between standard mechanical properties and structure are difficult to determine and interpret. From the stress-strain test at constant deformation rate or creep test at constant load or whatever static or dynamic mechanical experiment we could not obtain direct information about the supramolecular structure because the standard mechanical approaches evaluate the material as a whole, including all types of its defects. Microindentation methods occupy a medium position between standard mechanical measurements (dependencies stress-strain at constant load, creep, relaxation etc.) and classical structural investigations (Wide Angle X ray Scattering, Differential Scanning Calorimetry, Light and Electronic Microscopy, Positron Annihilation Lifetime Spectroscopy and Dynamic Mechanical Thermal Analysis).

For example, until recently, microhardness was considered as one of the standard, routinely measured mechanical characteristics. Now the microindentation experiments are developed as a method for material investigation. This qualitatively new approach to microindentation experiments can be compared with the development of the tensile test. From a simple break measurement, it was developed up to registration of the stress - strain diagram and many characteristics could be obtained from it: modulus of elasticity, yield stress, yield strain, shape of the maximum in the yield point, strength and deformation at the break etc.

Now microhardness has a leading place in the studies and control over materials mechanical properties. Its wide application as an investigation method is determined not only by the simplicity of the measurements but also by the fact that microhardness parameters give an idea of the overall picture of the mechanical

properties of the material as well as of the its structural peculiarities. Microhardness owns this especial position amongst other mechanical properties to its physical nature. In reality hardness is a complex internal characteristic; dependent on the basic mechanical properties of the material and in this sense characterizes completely its elastic-plastic properties.

MICROINDENTATION METHODS DEVELOPED IN OUR GROPE

Taking into consideration the rapid development of the microindentation experiment and its extensive applications as a scientific method our group contributed to this development inventing some new microindentation methods as definition and determination:

Total microhardness [1]

Total Microhardness can be considered as a measure for the local total material resistance against penetration and is related to the total deformation, including elastic, plastic and viscoelastic components. This parameter is defined by the analogy of Vickers microhardness:

(1) $$MHT= kP/D^2,$$

where P is the applied load, D is progected diagonals in loaded sate, k is a constant depending on the geometry of the pyramid.

It includes also the behaviour of the amorphous phase and together with Vickers macrohardness gives information about the elasticity of the material.

Penetration curves [1,2]

The penetration curves present dependences of the penetration depth changes (Δh) as a function of time (t)

(2) $$\Delta h=f(t), (P=const)$$

This experiment is similar to creep experiment as a trend of the curves as well as a physical meaning. In both measurements the material is simultaneously subjected to tension and pressure. The difference is that at constantly applied load in the case of penetration the cross section increases during the experiment, while in the case of the creep, cross section decreases.

Microhardness profiles [3]

Microhardness profiles are dependences of Vickers microhardness or Total microhardness on the applied load (P), respectively, on the penetration depth (h). They are sensitive to non-uniformity of the structure in the depth of the sample.

MHV=f(P); MHV=f(h); MHT=f(P); MHT=f(h).

This method is suitable for characterizing the laminated materials and coatings or for tracing changes in the surface layer structure and properties upon chemical, physical or mechanical treatment.

Imprint relaxation [4]

Imprint relaxation gives information about the viscoelastical component of deformation. This method is important for studying of materials when load/time dependencies on the deformation are strongly pronounced.

DICUTION ON FACTORS INFLUENCING MICROINDENTATION MEASUREMENTS

Many authors deal with the factors influencing the accuracy and unification of the microhardmess measurements. The next most frequently factors are commented:

Temperature. Of course the impact of temperature on microhardness experiments is very pronounced because the solid changes its structure passing through some very important temperature characteristic: the main glass transition, Tg (α-transition) and some low temperatures relaxation transitions, which usually are marked as β, γ transition. For example we have studied the sensibility of LDPE on the temperature in the zone of Tg. Fig. 1 illustrates these dependencies. That is why it is very important to specify the temperature of the measurement providing

Figure 2. R. Benavente *et al.*

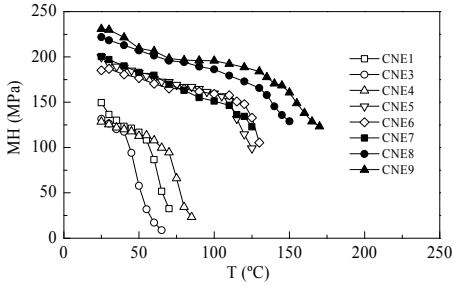

Fig.1 Temperature dependence of the Vickers microhardness [5]

Loading time. Loading time could not be very short because in this case the loading would not be considered as a static experiment but as a dynamic one. On the other hand the very slow loading could allow some time dependent processes as a creep or relaxation to take place.

Usually the time in the range of 5-15 sec. depending on the material and on the applied load is reasonable. In the modern testers the loading is automatic.

Time in loaded state.

This factor is very disputable. Many authors insist on that the loading time could be short to avoid a possible vibration. Moreover the majority of standard Vickers microhardness testers are construed in the way ensuring automatically constant loading time but without possibility to change it. Our previous investigations of the so called penetration curves [6] (The dependences of penetration depth changes with time at constant applied load) have shown that the time interval when the indenter slows down or stops its penetration is different for different materials. We think that the time interval when the penetrations curves change their initial fast increasing with smoother ones has to be the time interval in loaded state. If it is smaller, the inaccuracy would be larger because of the rapidly change in indentation shape. This time interval is different for different materials (fig. 2).

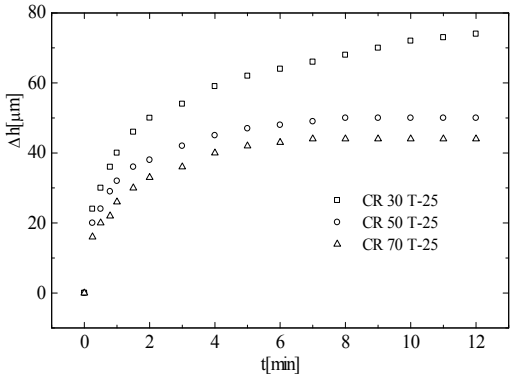

Fig. 2 Penetration curves for samples of chloroprene rubber modified with different kaolin contents [6]

Unloading time

Unloading time interval has to be short so that it could not hinder elastic and viscoelastic imprint relaxation. Also some casual factors as mechanical vibrations, stress fluctuations, changes in friction coefficient, adhesion between the indenter and sample etc, could provoke distortion of the measured microhardness characteristics. Some of these factors could not be avoided but their relative influence could be diminished provided the measurement is performed at higher loads. Their influence could be partly eliminated by statistic methods as well [7].

The minimal distance between the imprints

To avoid the results distortion it is necessary the distance between the imprints to be at least in the range of the imprint dimensions

The relation between indentation depth and sample thickness

In many guidance for Vickers microhardness measurement there is the requirement that the indentation depth, h, should be 10 times smaller than sample thickness, b, in order to guarantee .that the substrate does not distort the experimental data. When measuring the varnish layer with different thickness we have established that for harder materials, respectively materials with higher elastic modulus, the relation between indentation depth and sample thickness is smaller and vice versa: for softer materials this relation is bigger [8].

But there are some factors that are not mentioned in most articles though affecting the final results.

The type the material.

For evaluating how the nature of the materials could distort the results the model suggested by Oliver and Pharr[9] has to be commented. They offer a method for evaluating the so called contact imprint surface, respectively contact indentation depth. An illustration of imprint shape after indenter releasing is given in fig. 3. In some materials wherein the plastic deformation prevails and they are not shrinkable it is possible during the penetration a part of the material under the indenter to be pushed out on the surface thus forming a small pile around the imprint and making the sides. The imprint looks larger with the sides outside bulged. For elastic-plastic materials the part of the material around the indentation is dragged down and the imprint looks smaller with sides concaved inside. This microhardness behavior is tipical for many polymer materials especially for those with loose cross linking or physical entanglements. The authors who calculate the microhardness on the basis of the contact surface have made some corrections in the standard formula for Vickers microhardness. We calculate the Vickers microhardnesss on the base of microscopically measured diagonals that remain the same independently of the deformation of the imprint sides. Measuring the total microhardness is on the basis the indentation depth as a distance that the top of the indenter passes from moment of the surface touching till the equilibrium in loaded state. In this approach the deformation of the zones around the indenter does not influence the calculated values.

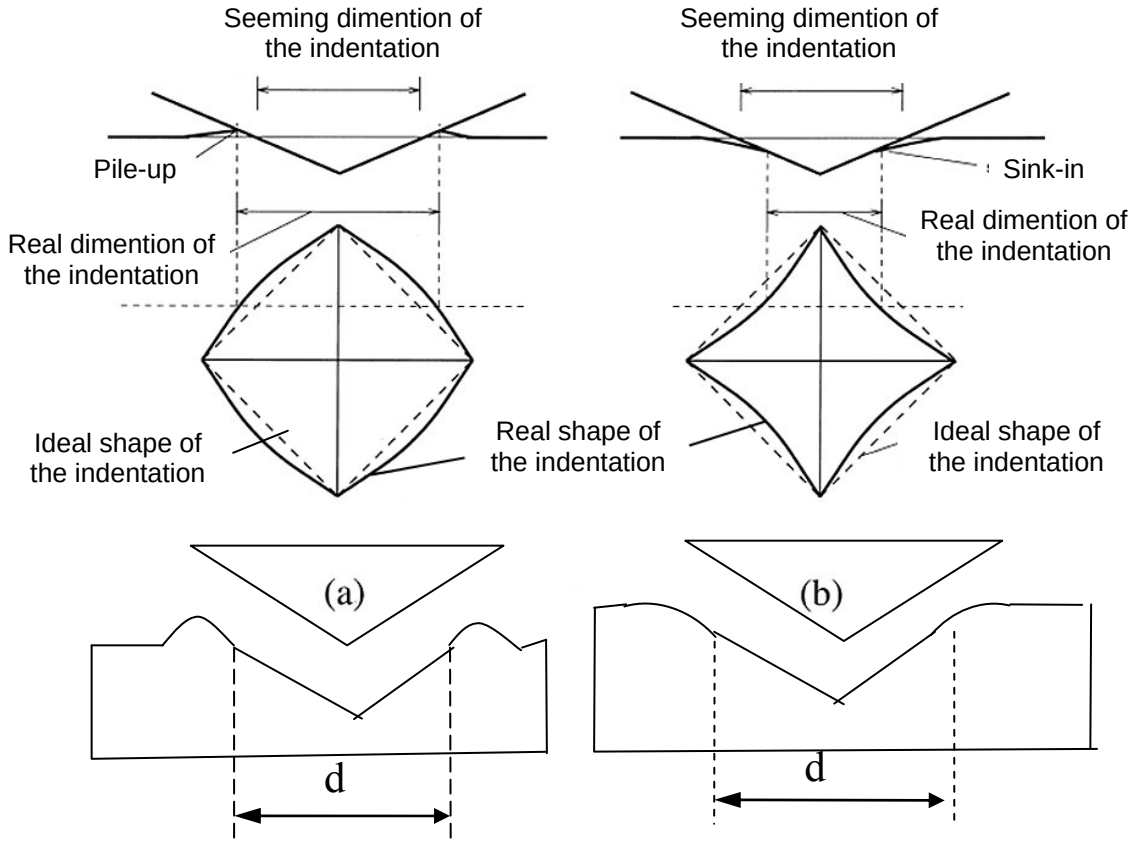

Fig. 3 Indentation shape a) pile-up effect, b) sink –in effect [10 -74]

Surface roughness

Surface roughness influences the contact surface especially at small applied loads, because the indenter does not cling close to the whole supposing area (fig. 4). The surface roughness also increases the error concerning to the moment when the indenter touches the sample surface, which causes the inaccuracy especially for determination the total microhardness. In the article [11] the samples with different roughness has been measured and it has been established that after certain number of measurements the average microhardness value is almost the some as if the measurement would be made on a polished surface. If the average surface roughness deviation is Ra = 0.7; 1.5or 5 μm, it is necessary to make about 15-20, or respectively 30, measurement for eliminating the influence of the surface roughness.

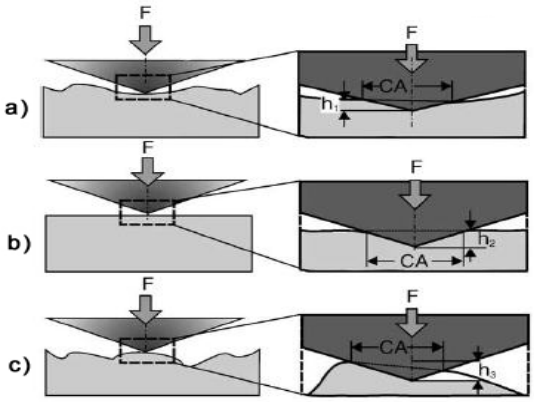

Fig. 4 Relation between surface roughness, indentation depth, h, and contact surface a) concave profile, b) ideal polish surface, c) bulged profile, CA is the diagonal of the imprints [11].

Volume nonhomogeneity of the samples

Nonhomogeneity of the materials leads to arising of casual errors. The nonhomogeneities could be some structural components in the blends or composition, vacancies, defects, different orientation etc. This type of the errors could be determined and reduced by statistic methods [10].

Unhomogeneity of the samples in direction perpendicular to the sample surface.

Unhomogeneity in direction perpendicular to the sample surface is very frequently, almost typical for polymer materials. Always sample preparation and especially the cooling regime of details preparation changes more or less the surface layer structure. Usually it is more amorphous for semicrystal plastics or less packaged for amorphous materials. When the goal is to study this factor, Mayers lines and microhardness profiles should be taken. The first ones give information about the tendency of microhardness changes: increasing or decreasing. Microhardness profiles give information concerning to the genuine changes in Vickers or total microhardness in the different indentation depth. When the unhomogeneity is not a goal of the measurement, then all the measurement could be provided at constant applied load, usually in a larger range.

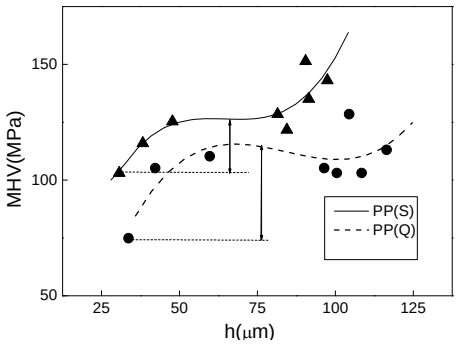

Fig. 5 MHV profiles for PP sample cooled slowly and quickly

CONCLUSIONES

Some factors influencing the microindentation measurements could not be avoided, but their precisely controlling would increase the experimental accuracy. These factors are: temperature; time parameters for loading, loaded states and unloading; distance between the imprints; sufficient sample thickness; surface roughness, etc.

Other factors, as time factors, (penetration curves, imprint relaxation) not only influence the microindentation parameters but could be applied for studying some time dependent mechanical processes as a creep and relaxation.

There are factors which could contribute to structural peculiarities investigation as defects or unhomogeneity (Mayers lines, microhardness profiles) existing in materials or arising during the sample preparation or some treatment. Moreover the microindentation methods allow to distinguish their origin in some cases.

ACKNOWLEDGEMENT

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ФАКТОРИ ВЛИЯЕЩИ ВЪРХУ МИКРОТВЪРДОСТНИТЕ ИЗМЕРВАНИЯ

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Ключови думи: *Микротвърдост, Микропроникване,Отпечатък,Структура на полимерите*
Резюме**:** *Разгледани са факторите влияещи върху микротвърдостните измервания и е дискутирано тяхното действие за изкривяване на експерименталните резултати. Някои от тях, които са широко известни и коментирани в много статии са само споменати. Други са детайлно дискутирани и техните граници са променени, базирайки се наши конкретни изследвания за тяхното влияние. Някои фактори са разглеждани като параметри, чието влияние върху изследваните микротвърдостни характеристики дава допълнителна информация за структурните и механични свойства на материалите.*