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EXPERIMENTAL-NUMERICAL HYBRID APPROACH TO INDENTATION EXPERIMENT

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Abstract: *The directly measured indentation parameters as well some additional information concerning the mechanical properties obtained by depth-sensing indentation method (DSI) have been discussed. Some limitations of the solely experimental approach have been critically listed. On the other hand, taking into account the opportunities of commercial software MSC.MARC basic principles, when apply numerical simulation using the finite element method in micro- and nanoindentation have been described, namely, material and geometric nonlinearity. The different approach to resolving engineering and scientific problems has been distinguished. The Experimental-numerical hybrid method in indentation experiment combining microindentation experiments with numerical simulation using FEM has a number of advantages: enables determination of some valuable mechanical properties (yield strength, distribution of equivalent Von Mises stress, distribution of the equivalent plastic strain in the zone under indenter and etc), reduces the time and facilitates the trial-error procedure in post-processing. A block diagram showing the steps in the experimental-numerical hybrid approach in indentation experiment has been suggested.*

INTRODUCTION

Micro- and nano-indentation is an effective and promising method for determining the mechanical properties of materials characterizing their behavior in micron or nano scale. Besides the classic way to hardness determining from the size of the imprint after subtracting the indenter, recently so-called DSC method [1] has been widely used.

Numerical modeling using the finite element method is applied to solve problems ranging from relatively simple linear analysis to challenging nonlinear simulations.

Therefore, the aim of this article is to discuss the advantages of combining the experimental results from DSI method with computer modeling and simulation of indentation processes. These two approaches have to be considered complementary in order to understand comprehensively the micromechanical processes during indentation.

DEPTH SENSING INDENTATION

Depth sensing indentation (DSI) or instrumented indentation testing (IIT) is a method based on a controlled penetration of the standard shaped indenter in the investigated material. At given constant penetration rate, the measuring device renders an account of the load and

indentation depth at every moment of the experiment. As a result the dependence of applied load vs. indentation depth (indentation curve) has been drawn.. According to this method the Young's modulus and hardness are main characteristics determined.

In fig. 1 a typical indentation curve (a) are shown. As a result the following characteristic dimensions could be determined:

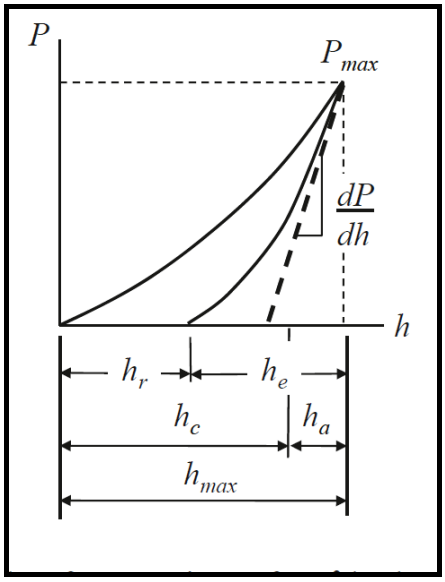

Fig. 1 Typical indentation curve (a) and corresponding changes in the imprint (b)

h_{max} – maximal indentation depth in loaded state;

h_r – residual indentation depth after unloading;

h_c – depth corresponding to the intersection of the tangent at the unloading curve with the abscissa;

h_e – elastic lifting of the bottom of the imprint.

Indentation experiment could be performed in different regimes:

- Only load up to defined maximal load or up to defined maximal depth;
- Loading-unloading. From the unloading part of the curve the indentation elastic modulus could be determined;
- Loading-hold-unloading. From the holding part of the indentation curves the creep processes could be investigated;
- Cyclic load-unload test, which is convenient for studying hysteresis;
- Step-load-hold test, enabling study inhomogeneity in the depth and especially creep;
- Step load-hold-unload test. Similar to the previous regime, but also gives a possibility to determine the indentation modulus at a different depth.

The indentation characteristics that could be measured are (ISO 14577-1):

- Dynamic hardness (DH):

(1) $$DH = \frac{aF}{h^2},$$

where (F) is the value of the instant load at loading and unloading testing regime, (a = 3,8584) is a constant which depends on the shape of the indenter and (h) is an indentation depth. This characteristic reveals how the material responds to plastic, elastic and viscoelastic deformation during the test.

- Martens hardness (HMs) was determined from the slope (m) of the increasing force/indentation depth curve in the 50% ÷ 90% P interval and characterizes the material resistance against the penetration:

(2) $$HMs = \frac{1}{26{,}43m^2} \ .$$

- Indentation hardness (Hit) according to the model of Oliver-Pharr [2] is a measure for the resistance to permanent deformation:

(3) $$H_{it} = \frac{P}{24{,}50h_c^2} \ ,$$

where (h_c) is the depth of contact of the indenter with the test piece.

- Indentation Elastic Modulus (E_{it}) calculated from unloading part of the dependence :

(4) $$\frac{1}{E_r} = \frac{1-\nu_s^2}{E_{it}} + \frac{1-\nu_i^2}{E_i} \ ,$$

where (E_r) is the experimental converted elastic modulus based on indentation contact, (ν_s) is the Poisson ratio of specimen, whereas (E_i) and (ν_i) are the Young's modulus and Poisson's ratio for indenter, respectively. Elastic modulus could be used for determining the stiffness or compliance of the specimen.

- Indentation creep (C_{it}) which is a relative change in the indentation depth at constant test force:

(5) $$C_{it} = \frac{h_2 - h_1}{h_1} \ ,$$

where (h_1) and (h_2) are indentation depths at the beginning and the final of the creep measurement.

- Plastic and elastic part of indentation work (ηit) determined from the areas under loaded and unloaded part of the load unload test ($W = \int Pdh$):

(6) $$\eta_{it} = \frac{W_{el}}{W_{el} - W_{pl}} \ .$$

DSI approach can give some additional information concerning cracking and when measuring laminates or coating one can evaluate delamination. The ratio of modulus and hardness (E/H) also provides valuable information about the spatial extent of the elastic deformation that occurs under loading before the point of yield strength. Also by these tests could be determined strain hardening, phase transformation, fracture toughness, energy absorption, anisotropy, manifestation of scale factor and some time-dependent mechanical characteristic as relaxation of the imprint and creep processes mentioned above.

However, there are some purely experimental limitations of the approach, namely:

1. Totally indentation experiment could not determine the yield strength. The hardness is not a defined property of the material, but its value depends on the method and the experimental conditions under which it is determined, including the type of the indenter. Therefore, the yield strength is a well-defined characteristic of the material and is used more often as an important parameter in many models;

2. When the samples or coatings are thin, it is relatively difficult to measure the elastic modulus and the hardness because of the influence of the substrate or the base material. There are a number of models developed to avoid the influence of the base material (substrate), but they are also based on various assumptions and limitations as well as on a certain mathematical apparatus that is not always appropriate for a wide variety of mechanical properties and behavior of the two materials;

3. One of the conditions for the direct measurement and eliminate substrate effect is the depth of indenter penetration should not be greater than 1/10 of the thickness of the layer tested. This automatically limits the measurement of samples thinner than 50 nm because the testing instruments are not able to measure penetration less than 5 nm;

4. The independent indentation experiment allows to determine the tendency of the material to cracking, but it cannot give an idea of tensile strength;

5. Indentation measurements do not give information of the complicated complex stress state and deformation under the penetrating indenter;

6. When measuring some indentation characteristic at the certain depth, the obtained value does not characterize this parameter exactly in this depth. It includes all surface layers up to this depth due to the superimposition of the effects of all the layers;

7. It is impossible to obtain exact information about the magnitude of the contact area and the occurrence of the so called pill-up and sing-in effects, respectively, although there are authors who suggest some semiempirical approach to determining the contact depth.

NUMERICAL SIMULATION

There is much commercial software specialized in numerical simulation by applying the finite element method. In scientific papers dealing with the numerical simulation the indentation, programs used most often are MSC.Nastran, MSC. MARC, ABAQUS.

Indentation simulations have two sources of nonlinearity: material and geometric nonlinearity. Simulation by the finite element method [4,5] is performed by considering that:

- The indenter as a rigid body penetrates the half infinite space. The indenter half of the space is modeled as a rotationally symmetric.
- The indentation process is considered as quasi-static problem, which does not take into account the time and speed effects.
- No stress-strain prehistory is taken into account
- The friction forces in the contact area are neglected;
- The deformable axisymmetric specimen is composed of an isotropic linear elastic-plastic with linear hardening material;

The constitutive model used in the finite element model of the investigated materials is based on the Von Misses yield criterion in combination with a linear hardening law.

The material model is elastic-plastic with linear hardening. The hypothetic “linear hardening” provides accurate results in the analysis of load, although actually irreversible plastic deformation occurs after reaching the yield strength of the surface layers.

The influence of large deformations is included in the analysis by geometric nonlinearity. The criterion of von Mises for cold flow is used in the calculations. This is an engineering criterion that indicates whether a combination of three-dimensional stresses σ_1, σ_2, σ_3 in the body will cause reaching of the yield strength in a microvolume of the subject.

$$\sigma_{VM} = \sqrt{\frac{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2}{2}} . \quad (7)$$

The model involves the following material parameters: Young modulus, Poisson ratio, yield strength, strength coefficient.

In structural engineering where the aim is to determine whether a material is suitable for specific parts or coatings literature data can be used for incoming material constants. For example, certain steel grades or alloys that would meet the technological and mechanical requirements for the surface properties of the object under study. In this case it is not necessary to carry out an indentation experiment.

But when the goal is the characterization of the new material, it is necessary to combine the DSI experiments with FEM. This is especially true for polymeric materials and composites based on them. The same polymer may have different mechanical properties in accordance with the polymerization conditions (molecular weight, degree of branching or crosslinking, degree of crystallinity), the melting temperature and the cooling rate during molding of the specimen. All these factors influence the supramolecular structure and hence

the mechanical properties of the material. Some researchers practice the classic method for determining the input mechanical parameters from the stress-strain curves. However, this method requires a large amount of research material for a statistical processing of the data. Moreover, the simplified regime of uniaxial loading is not unequivocal on the complicated deformation behavior of the material under the indenter at the indentation experiment.

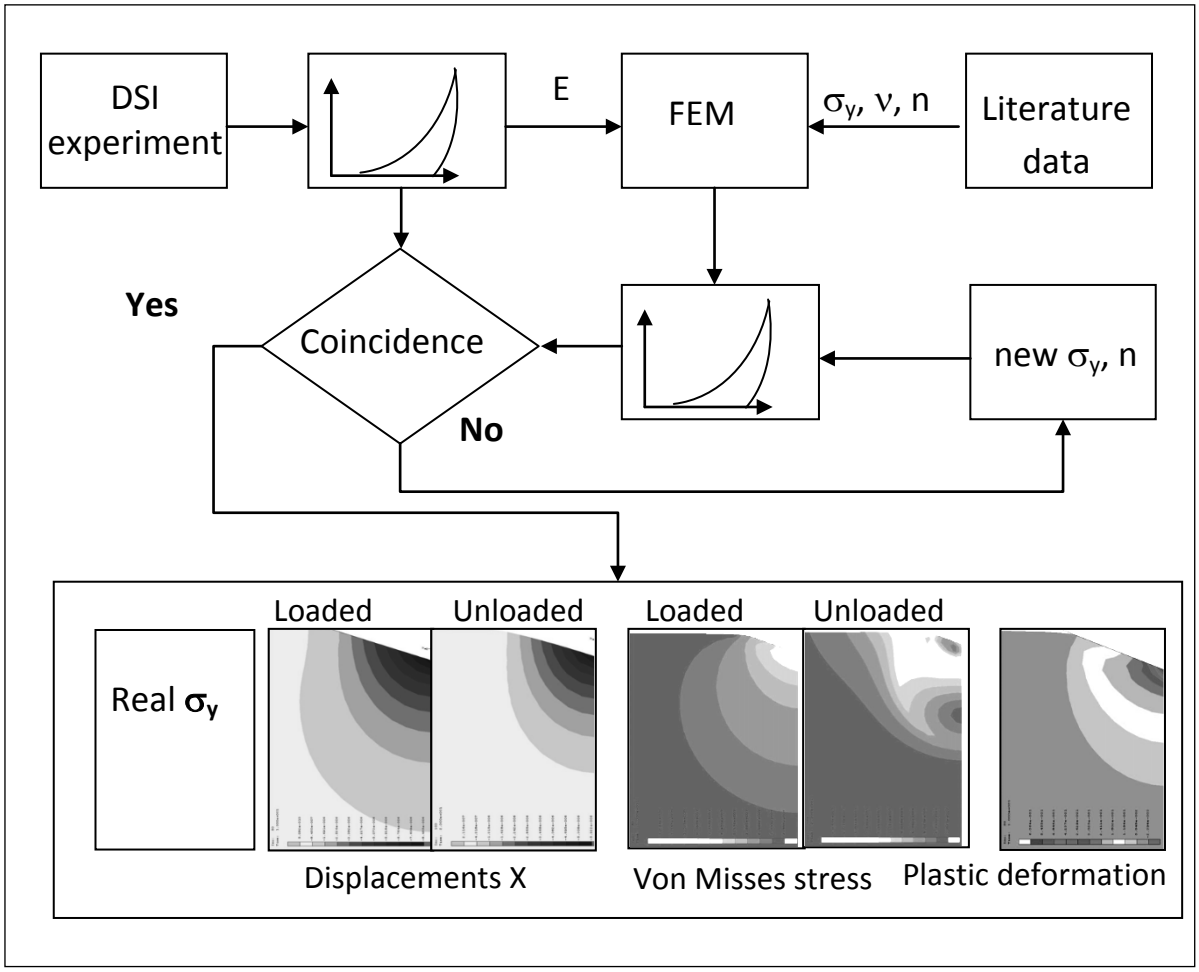

Fig.2 Block diagram of the experimental-numerical hybrid approach in indentation experiment and additional information from FEM

The full analysis by the finite element method is usually consisting of three distinct stages:

- pre-processing, when we have to obtain reliable material parameters for starting the simulation;
- simulation itself;
- post-processing, which consists of a comparing the experimental and numerical load-displacement curves. If there is a discrepancy, we perform a trial-error procedure in order to obtain the material parameter set that gives the best fit to the experimental data.

The advantage of the hybrid indentation model is that we vary only the yield strength and strength coefficient, because the elastic modulus is real, determined under the indentation condition, Poason's ratio for polymers varies between 0,35 and 0,45 and it is taken the same as that we use for modulus calculating. Fig. 2 plots the block diagram showing the steps in the experimental-numerical hybrid approach to the indentation experiment.

CONCLUSION

Experimental-numerical hybrid method to the indentation experiment combines microindentation experiments with numerical simulation using FEM. Thus microindentation characteristic are supplemented by additional information about the mechanical characteristics of the investigated material: yield strength, distribution of equivalent Von Mises stress, distribution of the equivalent plastic strain in the zone under indenter and etc., which cannot be obtained only by means of a microindentation experiment.

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ЕКСПЕРИМЕНТАЛНО-ЧИСЛЕН ХИБРИДЕН ПОДХОД ПРИ ИНДЕНТАЦИОННИЯ ЕКСПЕРИМЕНТ

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Ключови думи: *контролирано проникване на индентора, еластичен модул, числена симулация, МКЕ*

Резюме: *Дискутирани са индентационните параметри, които могат да се измерят директно, както и допълнителна информация за механичните свойства получени по метода на контролирано проникване на индентора (DSI). Изброени са някои ограничения на чистия експериментален подход. От друга страна имайки впредвид възможностите на софтуерния продукт MSC.MARC са описани основните принципи при прилагане на числената симулация по метода на крайните елементи (МКЕ)за процеса на индентация, основно материалната и геометрична нелинейност. Направено е разграничаване в подхода при решаване на инженерни и научни проблеми. Експетиментално-числения хибриден подход при индентационния експеримент, който съчетава микроиндентационния експеримент с числената симулация прилагайки МКЕ има редица предимства: позволява определяне на някои ценни механични свойства (граница на пластично течение,разпределение на еквивалентните напрежения по фон Мизес и пластичната деформация в зоната под индентора и др.), намалява времето и улеснява процедурата „проба-грешка". Предложена е блок-схема на стъпките при експетиментално-числен хибриден подход при индентационния експеримент.*