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MODELLING OF NANOMATERIALS – SENSITIVITY ANALYSIS TO DETERMINE THE NANOINDENTATION TEST PARAMETERS

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Abstract

Analysis of nanomaterials is challenging in modeling and in experiments. Due to many difficulties in measuring their properties and a large scatter in results, there is a necessity to use computational methods to obtain better data. Thin film is applied in this paper to model the nanoindentation test and finally to predict the force-depth curve. The nanoindentation test with round tip indenter is simulated and remeshing problem is solved in the program. Numerical graph representing force versus depth, which is an output from the program, is performed to optimize deformation process. Sensitivity analysis is a numerical method used here, which is set in the present work to determine parameters in the deformation process in nanoindentation test and to specify the influence of elastic material model on the test results.

Key words: nanoindentation test, sensitivity analysis, finite element method (FEM)

1. INTRODUCTION

Thin hard coatings are difficult in experimental and numerical tests, because of a very small scale and their physical properties. Other significant numerical difficulties rise from die-specimen contact region, which produces necessity of remeshing on a very small distance. Finite element method is helpful in solving the designing problems, in particular it allows to control deformation of hard coatings and to anticipate their behaviour. Thus, the general objective of this paper is a creation of the direct numerical model for nanoindentation test and further for inverse analysis. Inverse analysis concept for interpretation the mechanical nano test is presented in (Kopernik and Pietrzyk, 2006). Using this model, the second purpose is sensitivity analysis of nanoindentation test and specifying, which of the process and material model parameters is the most influential on the nanoindentation test results.

2. MATERIAL PROPERTIES AND MATERIAL RHEOLOGICAL MODELS

2.1. Material properties

In this paper thin hard monocoating deposited using PVD (physical vapour deposition) is investigated. Here are obtained: material behaviour under loading conditions and material properties in simulation of nanoindentation test. Thin material - purely elastic monolayer, is 400nm thick and 2600nm wide. The FEM model settings related to material are: Poisson ratio $v = 0.177$ and four elastic moduli $E_1 = 20$GPa, $E_2 = 22$GPa, $E_3 = 28$GPa, $E_4 = 30$GPa, which are set in simulations. Four elastic moduli are chosen, because the sensitivity analysis requires different values of analysed parameters. The values of mentioned mechanical properties are comparable to elastic moduli obtained in inverse analysis for hard nanocoating by the Authors (Kopernik and Pietrzyk, 2007a).

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2.2. Material rheological model

The FEM program is designed for forging simulation in various conditions. This FEM based computing procedure solves numerical problems connected with large deformations, which occur in mesh elements during process. Remeshing is applied, when it is necessary (mesh elements are too distorted). Considered case needs new mesh generation in the die-specimen contact area during deformation process and it is a reason that the program FORGE 2 is chosen. The description of the rheology of the material in this program is based on the Norton-Hoff flow rule written in the following form:

$$\boldsymbol{\sigma} = 2K(T, \bar{\varepsilon}, ...)\left(\sqrt{3}\dot{\bar{\varepsilon}}\right)^{n-1} \dot{\boldsymbol{\varepsilon}} \tag{1}$$

This relation links the deviatoric stress tensor $\boldsymbol{\sigma}$ to the strain rate tensor $\dot{\boldsymbol{\varepsilon}}$ through the consistency $K(T, \bar{\varepsilon}, \ldots)$ and the sensitivity to the strain rate n. The theoretical behavior corresponding to the value $n = 1$ also called Newtonian behavior, can be integrated in FEM program. In this case, the set of equations describing the problem of mechanical equilibrium is linear. In equation (1) T is temperature, $\bar{\varepsilon}$ is effective strain and $\dot{\bar{\varepsilon}}$ is effective strain rate.

Part of the material deformation is represented by a reversible elastic behavior. This material behavior is idealized through the linear elasticity law. For purely small strains, this law is written in the following form:

$$\boldsymbol{\varepsilon} = \frac{1+\nu}{E}\boldsymbol{\sigma} + \frac{3\nu}{E} p\mathbf{I} \tag{2}$$

where $\mathbf{I}$ - unit tensor, $\boldsymbol{\varepsilon}$ - the elastic small strains tensor, $\boldsymbol{\sigma}$ - the stress tensor.

Friction law. Coulomb friction law, called „Tresca limited Coulomb", is expressed as follows:

$$\tau = \mu\sigma_n \text{ if } \mu\sigma_n < \frac{m\sigma_0}{\sqrt{3}}, \tag{3}$$

and $$\tau = \frac{\sigma_0}{\sqrt{3}} \text{ if } \mu\sigma_n > \frac{m\sigma_0}{\sqrt{3}}, \tag{4}$$

where:
τ – shear stress, μ – friction coefficient, m - friction factor, σ_n - normal stress, σ_0 - flow stress.

Coulomb friction law was used in simulations and the following friction coefficients and factors were set as contact conditions:
a) $\mu = 0.1$, $m = 0.05$; b) $\mu = 0.15$, $m = 0.05$; c) $\mu = 0.2$, $m = 0.1$; d) $\mu = 0.25$, $m = 0.1$.

2.3. Experiment - nanoindentation test

The International Organization for Standardization has produced an international standard ISO 14577, which can be applied to instrumented indentation testing (figure 1c). Indentations are many-cycle load-controlled load-partial unload experiments from 10μN to 500mN maximum load. An uncoated substrate wafer is tested for comparison. The data are analyzed with the Oliver and Pharr method (Oliver & Pharr, 1992) and repeated load-partial unload experiments are performed on each sample.

The nanoindentation test for thin films (20nm to ~ 5 micron thick) can be controlled either in force or in depth. This test gives the following parameters: mechanical properties (hardness, elastic modulus), creep resistance and temperature-dependent properties. No other technique provides information about both the elastic and plastic properties of thin films. The four most commonly used pyramidal indenter shapes are such as Vickers (figure 1b) (α = 68°), Berkovich (α = 65.03°), modified Berkovich (α = 65.3°) and cube corner (α = 35.26°). Berkovich (figure 1c) is deformable, elastic diamond (E = 1141GPa, ν = 0.07), with tip radius R assumed to be 100-150nm. The Martens hardness is defined in ISO 14577 for Vickers and Berkovich indenter geometries. Nanoindentation testing is performed in load-controlled mode (figure 1a) using a Nano Test System platform (Beake and Lau, 2005) with indentation module, which has high-temperature countershaft, high resolution microscope, atomic force microscope, nanopositioner, indenters of different shapes and relevant software.

3. NUMERICAL TESTS

Conditions for numerical simulation of nanoindentation test are similar to those in laboratory, but die[*] shape simplification is made (specimen cross-section is taken in simulation). Twelve Berkovich shape indenters are set to specimen surface. They have four tip radii equal to 100, 110, 150 and 160nm, as well as three tip vertex angles: 65.3, 67.5 and 70.32°, all with round tip (figure 1d).

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[*] *die, probe, tool, indenter* have the same meaning in nanoindentation test; *tip* is the end of the die, probe, tool, indenter in nanoindentation test.

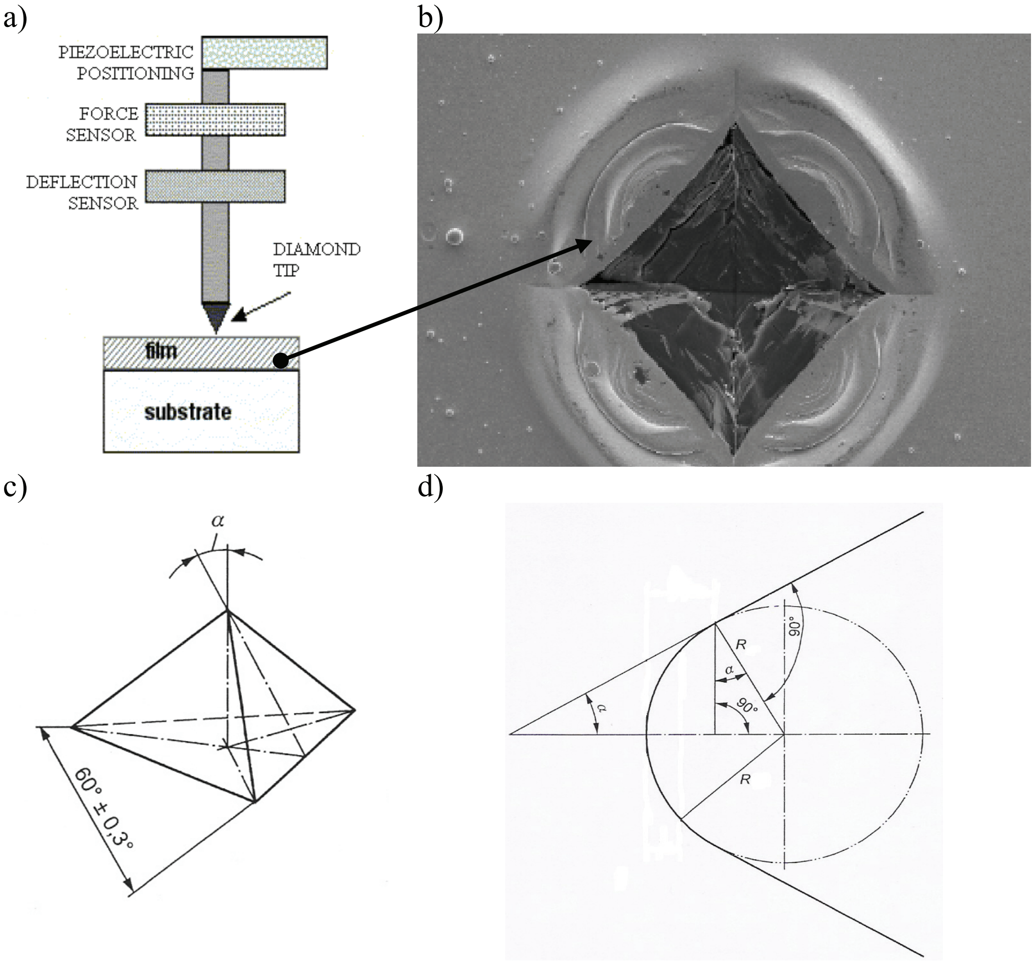

Fig. 1. *a) Schematic illustration of nanoindentation test (Albrecht et al., 2005), b) Example of Vickers indent, SEM image of Au/Si probe, c) Berkovich indenter [PN-EN ISO 14577- 2], d) Berkovich round tip geometry.*

Tip round geometry is based on spherical indenter geometry (figure 1d) and it is necessary, when remeshing is activated. The work (Kopernik and Pietrzyk, 2007b) shows that sharp indenter tip and remeshing procedure cannot be together set in the FEM model. In such case, especially when multistage deformation process for multilayer is executed in deep nanoindentation test, one contact point (sharp indenter tip) leads to wrong solution in remeshing procedure. It is manifested as sticking the moving die into specimen and when round tip is made, even in late stage of deformation process, specimen behaves as in laboratory test – the maximum depth is only twenty percent$^{\infty}$ of layer and bottom layers are not cut through.

Berkovich shape deformable probe is used as a die in the present work and it is simulated as a semi-cone cross-section. Die is moving into specimen and is set as a constant velocity press in program settings. Knowing velocity (10 nm/s) and displacement (100 nm) as an input data, force versus depth is calculated, as well as average stress, equivalent strain and equivalent stress distributions. Number of indentations steps is fixed to one in each simulation, because specimen material is a thin monolayer, elastic medium, and there is no need to repeat indentation process. Remeshing is introduced when elements are too distorted. After each loading step, elastic unloading is made. There is a difference between experimental test and numerical one. In experiment, only partial unloading is made and in simulation there is a total unload. In numerical test whole specimen is able to respring and every load step starts exactly from the specimen surface. Moving die is a deformable one and has its own mesh. Finally, 196 FEM simulations for 196 variants were performed: for four friction laws, four material models and twelve indenter shapes.

Assuming 2D and axisymmetric model is an often made simplification (decrease of computing costs) and here it is also set. Such model is justified and it does not cause a lost of important information. The full 3D FEM (wedge/brick) results in the nanoindentation test are compared to those obtained in the axisymmetric 2D FEM model (cross-section of wedge/brick) and good agreement is observed (Albrecht et al., 2005). Beyond this, numerical and experimental results have good conformability in nanoindentation test, what is proved by (Chollacoop et al., 2003).

In this work some examples of the nanoindentation test simulations were performed, which proved that the full 3D FEM results are comparable to those obtained from the axisymmetric 2D FEM model

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[∞] 20 percent of layer as the maximum depth is set in experiments and provides to no substrate effects.

(figure 2). Specimen was 400 nm thick and 1000 nm wide, purely elastic monolayer was assumed (E = 200 GPa and ν = 0.3). Coulomb friction law was used and the friction coefficient and factor were equal to μ = 0.1 and m = 0.05. Deformable, elastic modified Berkovich (α = 65.3°) indenter with tip radius R = 150nm was set to specimen surface.

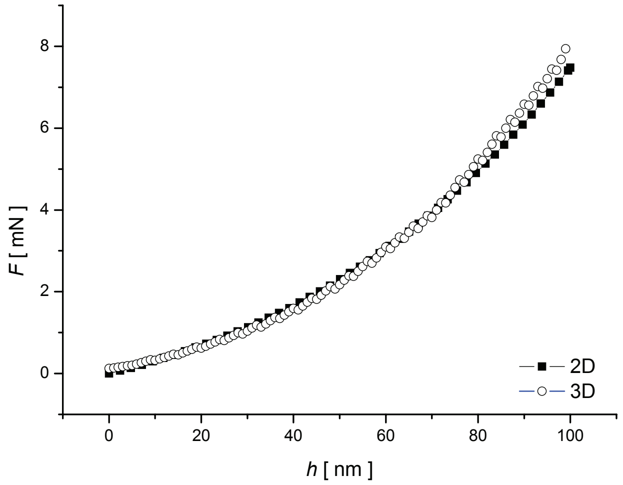

Fig. 2. Force versus depth results for 2D FEM axisymmetric and 3D FEM models.

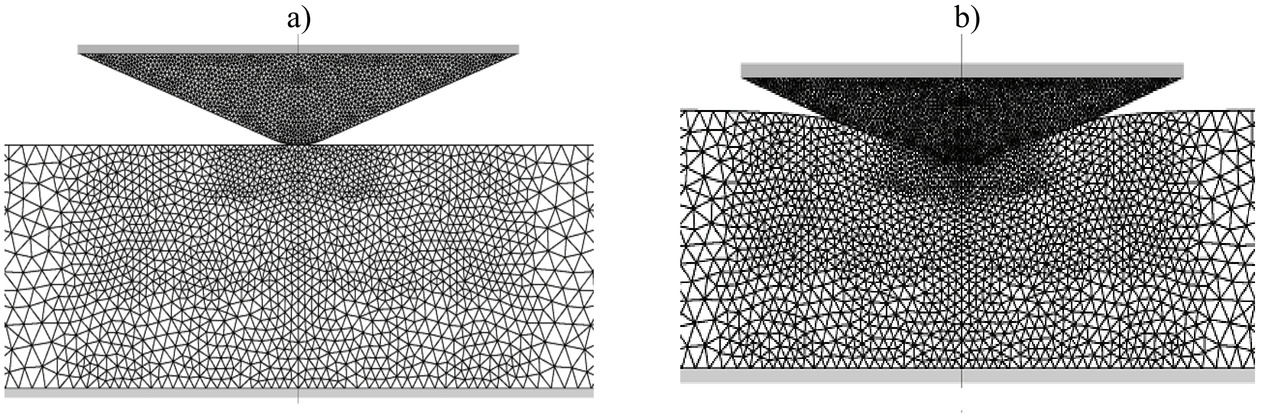

***Fig. 3.** a) Specimen and die discretization for FEM analysis at the beginning of the test; b) Deformed mesh of specimen and die at the end of test.*

4. FEM MODEL – DIRECT PROBLEM

In 2D axisymmetric FEM simulations the average number of nodes is about 1500 and number of triangular elements is 3000.

Specimen and die discretization for FEM analysis is presented in figure 3a. Numerical, axisymmetric FEM model with deformable Berkovich indenter is used in the test and the initial mesh is shown as well as lower fixed, rigid die. Local mesh refinement in program leads to fine mesh in the contact region, what gives good results. Deformed mesh of specimen and die after loading, at the end of the test, is presented in figure 3b.

5. SENSITIVITY ANALYSIS

The most advanced procedure for thin films and nanoindentation test with dual sharp indenter, using the inverse algorithm and performing the sensitivity analysis, was presented in (Chollacoop et al., 2003). The previous study, with single sharp indenter, was made by (Dao et al., 2001). Many investigations were introduced in (Chollacoop et al., 2003; Dao et al., 2001) and they gave good results.

In authors opinion (Kopernik and Pietrzyk, 2007), what was indicated above in this paper, sharp indenters are not proper for multilayer nanoindentation numerical test – multistage one, because of incorrect result in remeshing solution. Usually remeshing is necessity, because of distorted mesh in multilayer, multistage case, especially in the die-specimen contact region. Moving, sharp indenter is driven into material, whole specimen is not pressed - is penetrated through by the die. Round tip leads to correct remeshing solution – whole specimen is pressed and it behaves exactly like in the experiment. It is caused by the fact, that more than one contact node occurs between the round tip and the specimen. In paper (Dao et al., 2001) only monolayer, shallow sharp indentation test was analyzed and the FEM model without remeshing is correct, but it cannot be further applied to the multilayer, multimaterial FEM model.

The purpose of this work is to design the FEM model with remeshing and round tip indenter. Following this the sensitivity analysis was performed. The analysis was based on the finite difference approximation (brute force method). The main advantages of this method is that no modification to the original model is needed. It should be remembered, that this method is not the most accurate, but as the first investigation gives satisfying results.

The comparable value for nanoindentation test and the simulation of this experiment is the load.

This parameter is directly measured at the test and the goal function defined in the inverse analysis applied to the identification of the process parameters will be based just on the load. Following this, the sensitivity of the total load with respect to the process parameters was determined. To compare the sensitivities for various process parameters, the relative, dimensionless, sensitivities coefficients φ_{p_j} were defined:

$$\varphi_{\mathrm{p_j}} := \left.\frac{\partial F_{\mathrm{av}}}{\partial p_{\mathrm{j}}}\right|_{\mathbf{p}*} = \frac{p_{\mathrm{j}}^{*}}{F_{\mathrm{av}}(\mathbf{p}^{*})} \frac{F_{\mathrm{av}}(\mathbf{p}^{*} + \Delta p_{\mathrm{j}} \cdot \mathbf{e}_{\mathrm{j}}) - F_{\mathrm{av}}(\mathbf{p}^{*})}{\Delta p_{\mathrm{j}}} \tag{5}$$

where $p \in \{R, \alpha, \mu, E\}$ vector composed of the tip parameters R and α, friction coefficient μ and Young modulus E, $\mathbf{e}_{\mathrm{i}}$ - vector of the canonical basis, Δp – variation of the parameter p, F_{av} – average value of the total load, calculated as follows:

$$F_{\mathrm{av}} = \frac{1}{T}\int_{0}^{T} F(t)dt \tag{6}$$

where $F(t)$ - the load at the time t of the process, T – total time of the process.

Equation (6) defines sensitivities independently of the absolute values of variables, therefore, these sensitivities can be directly compared to each other. The accuracy of the sensitivities depends on the selection of the parameter variation Δp. For nonlinear models too large parameter variations could damage the assumption of local linearity. On the other hand, the round-off error could be high for too small perturbations. The trial-and-error procedure was performed to set the acceptable variations Δp.

6. RESULTS

Sensitivity analysis was performed for the total load with respect to the geometrical tip parameters: the radius R and the angle α, friction coefficient μ and Young modulus E. Two points were analyzed for each parameter, defined as the lower and upper limit of the appropriate parameter. The parameters limits were identified based on the experimental results and they are applied to the penalty function of the inverse algorithm to estimate the process parameters. The values of all parameters were described in chapters 2 and 4. The sensitivity coefficients φ_R were calculated for each value of the parameter for changing values of remaining parameters to verify if the sensitivity coefficients are independent or not of other parameters.

The results of the sensitivity analysis are presented in figure 4. The total load of the process is the most sensitive to the tip angle α and the highest discrepancies between the values of φ_α are observed (see figure 4 and table 1).

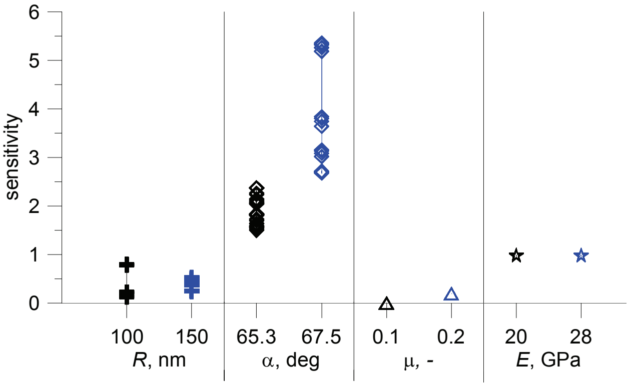

Fig. 4. *Load sensitivity with respect to the geometrical tip parameters R and α, friction coefficient μ and Young modulus E.*

The total load is sensitive to the tip radius R, as well, and the values of the coefficient φ_R vary for various values of the parameters α, μ and E. Further analysis of the coefficients: φ_R (figure 5) and φ_α (figure 6) shows that the jumps of these coefficients are caused first of all by the changes of the tip angle α for the φ_R (figure 5a) and the tip radius R for φ_α (figure 6a). Beyond this, the arrangement of the sensitivity values φ_R and φ_α for the parameters μ and E is similar for various values of these parameters (figures 5b-c and 6b-c). The load sensitivities φ_μ are close to zero and φ_E are close to 1 for all combinations of the remaining parameters (see table 1). Moreover, for upper and lower limit established for μ and E the coefficients φ_μ and φ_E are very close to each other, what proved the linear load behavior with respect to μ and E.

7. CONCLUSIONS

The results of the sensitivity analysis show that the nanoindentation test is the most sensitive to the geometrical parameters of the tip. The behavior of the process (in the terms of the total load) is strongly nonlinear with respect to the tip angle and further analysis of the process, more accurate, should be performed. The sensitive analysis proved that in the modeling of the nanoindentation test and in the inverse analysis of this process, based on the total load, geometrical tip parameters and Young modulus are the main factors, which decide of the process character. The friction coefficient does not require special consideration and it can be omitted in the inverse analysis.

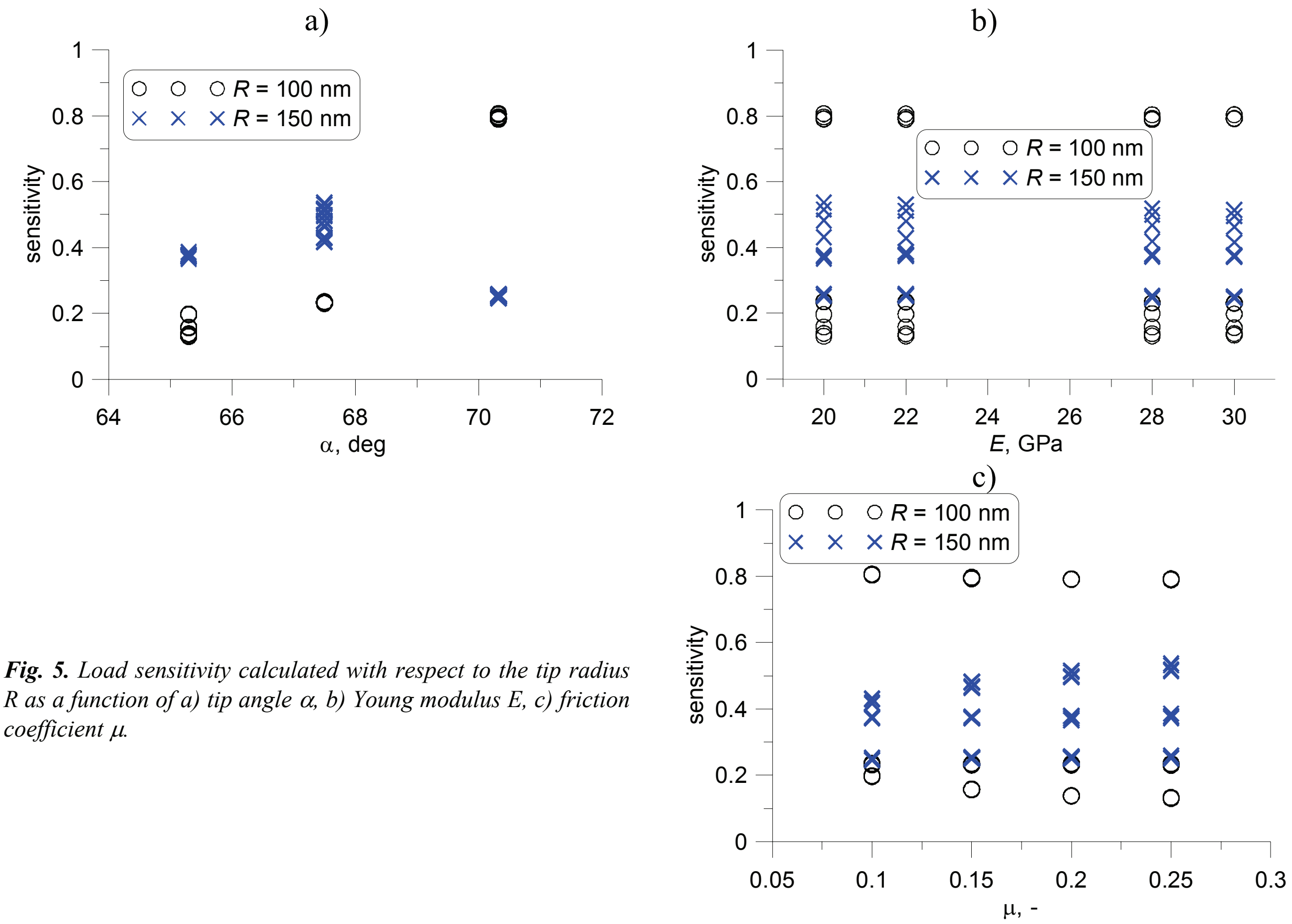

Fig. 5. Load sensitivity calculated with respect to the tip radius R as a function of a) tip angle α, b) Young modulus E, c) friction coefficient μ.

Table 1. Maximum and minimum values of the sensitivity coefficients φ_{p_j} calculated with respect to parameters R, α, μ and E.

$\max(\varphi_{R=100nm}) = 0.80683$	$\alpha = 70.32^o$	$\mu = 0.1$	$E = 20$ GPa
$\min(\varphi_{R=100nm}) = 0.12997$	$\alpha = 65.3^o$	$\mu = 0.25$	$E = 20$ GPa
$\max(\varphi_{R=150nm}) = 0.53665$	$\alpha = 67.5^o$	$\mu = 0.25$	$E = 20$ GPa
$\min(\varphi_{R=150nm}) = 0.24521$	$\alpha = 70.32^o$	$\mu = 0.1$	$E = 30$ GPa
$\max(\varphi_{\alpha=65.3^o}) = 2.384$	$R = 110$ nm	$\mu = 0.2$	$E = 30$ GPa
$\min(\varphi_{\alpha=65.3^o}) = 1.4998$	$R = 150$ nm	$\mu = 0.25$	$E = 20$ GPa
$\max(\varphi_{\alpha=67.5^o}) = 5.3653$	$R = 110$ nm	$\mu = 0.25$	$E = 30$ GPa
$\min(\varphi_{\alpha=67.5^o}) = 2.6795$	$R = 160$ nm	$\mu = 0.25$	$E = 20$ GPa
$\max(\varphi_{\mu=0.1}) = 0.0089$	$R = 100$ nm	$\alpha = 65.3^o$	$E = 30$ GPa
$\min(\varphi_{\mu=0.1}) = 0.0006$	$R = 110$ nm	$\alpha = 65.3^o$	$E = 28$ GPa
$\max(\varphi_{\mu=0.2}) = 0.0061$	$R = 160$ nm	$\alpha = 67.5^o$	$E = 20$ GPa
$\min(\varphi_{\mu=0.2}) = 0.0004$	$R = 110$ nm	$\alpha = 67.5^o$	$E = 30$ GPa
$\max(\varphi_{E=20GPa}) = 0.99036$	$R = 100$ nm	$\alpha = 70.32^o$	$\mu = 0.2$
$\min(\varphi_{E=20GPa}) = 0.9696$	$R = 150$ nm	$\alpha = 65.3^o$	$\mu = 0.25$
$\max(\varphi_{E=28GPa}) = 0.99032$	$R = 110$ nm	$\alpha = 70.32^o$	$\mu = 0.25$
$\min(\varphi_{E=28GPa}) = 0.97139$	$R = 160$ nm	$\alpha = 65.3^o$	$\mu = 0.15$

The above numerical observations can be interpreted using the knowledge regarding the indentation test gained from experiments. The size of contact area between indenter and specimen decides about the response of deformed material and its properties. For example, hardness of the coating is calculated using the relation of force-indent area. The most influential geometrical parameter should be the tip angle, what is proved by the sensitivity analysis. The tip radius only changes the volume of the indenter. The behaviour of elastic material under loading is specified by the elastic properties and they also have great meaning in the model output. They determine character and values of the force-depth curve.

The next step of the work will be to perform the procedures for multilayer, multistage deep nanoindentation test and the identification of the model parameters. The identification will be based on the sensitivity analysis results and on the inverse method developed by the authors (Szeliga and Pietrzyk, 2002). The algorithm of the inverse analysis will be adapted to present test conditions, developed FEM models with remeshing solution and the tip round indenters.

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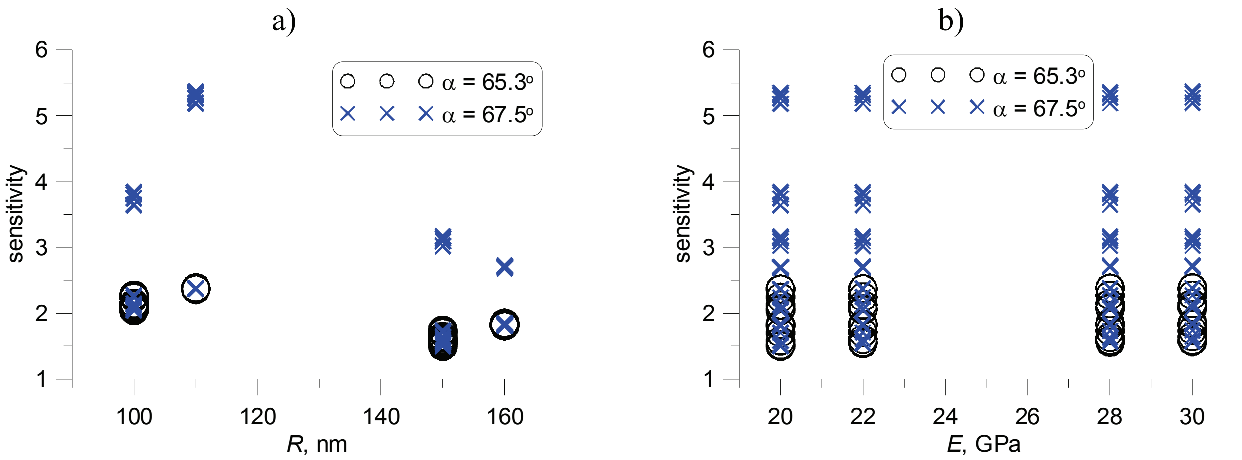

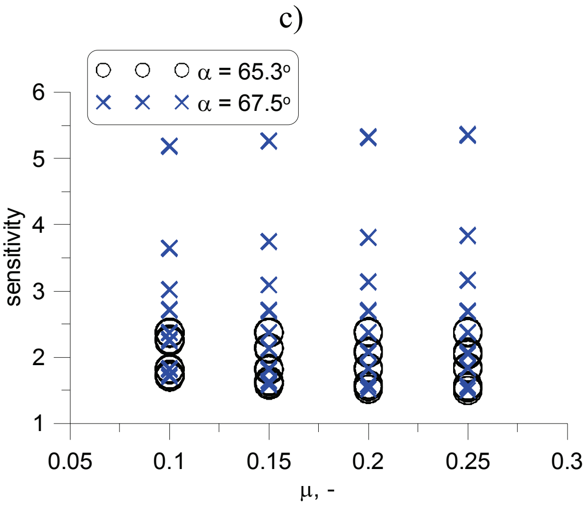

Fig. 6. *Load sensitivity calculated with respect to the tip angle α as a function of a) tip radius R, b) Young modulus E, c) friction coefficient μ.*

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MODELOWANIE NANOMATERIAŁÓW – ANALIZA WRAŻLIWOŚCI W CELU WYZNACZENIA PARAMETRÓW INSTRUMENTALNEJ PRÓBY WCISKANIA WGŁĘBNIKA

Streszczenie

Badania doświadczalne nanomateriałów są wyzwaniem dla naukowców. Ze względu na trudności związane z interpretacją wyników pomiarów, na podstawie których wyznaczane są własności nanomateriałów, i ich duży rozrzut, wykonywane są komputerowe symulacje doświadczenia w celu dokładnej analizy zachodzących zjawisk. W niniejszej pracy analizowano cienkie powłoki dla modelowania instrumentalnej próby wciskania wgłębnika i przewidywania zależności siły od głębokości wciskania. Doświadczenie symulowano metodą elementów skończonych, przyjmując zaokrągloną końcówkę wgłębnika i stosując aktualizację siatki (remeshing). Do optymalizacji próby używana jest zależność siły od głębokości wciskania. W pracy przeprowadzono analizę wrażliwości siły względem parametrów procesu dla identyfikacji tych parametrów, które mają istotny wpływ na przebieg doświadczenia. W wyniku analizy określono wpływ parametrów sprężystych i kształtu wgłębnika na wyniki symulacji.

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