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FEM application in studies of mechanical properties of the C-Pd film

JOANNA RYMARCZYK

Tele & Radio Research Institute, Ratuszowa 11, 03-450 Warsaw, Poland
E-mail address: joanna.rymarczyk@itr.org.pl

ABSTRACT

Comparison of numerical simulation and experimental data for nanoindentation studies of nanostructural carbonaceous-palladium films (C-Pd) is presented.

Finite Element Method (FEM) and ANSYS program (Ansys, Inc) were used. Traditional Oliver–Pharr method for cone and spherical shaped indenter were applied for FEM modelling of nanoindentation experiment. FEM simulations showed that Pd nanograins are moved in carbon matrix toward the film surface due to an external stress. Distribution of palladium grains in the film volume influence on the Young modulus and nanohardness was also found.

1. INTRODUCTION

Mechanical properties of new nanomaterials are particularly interesting due to the possibility innovative applications. Properties of such materials on the nanometer scale might differ from those of a bulk material of the same composition. Nanoindentation investigations of films containing Pd nanograins are difficult because topography and/or structure of films and contents of Pd in the films significantly affect their nanomechanical properties. Such films could be applied as an active layer in many types of sensors. C-Pd films chemical, mechanical and physical properties depend on palladium nanograins and carbonaceous matrix structure [1, 2]. One of new methods of investigations of nanomechanical properties is nanoindentation method. This method is designed to measure the nanomaterial's mechanical properties such as nanohardness and reduced modulus of elasticity [3]. In this technique, the indenter is placed on very small depth into materal.

Finite Element Method (FEM) is useful to analyze mechanical behavior of nanocomposites, thin films and biomaterials. In this paper a structural model of film composed of palladium nanograins embedded in carbonaceous matrix is presented. The model allows predicting some mechanical properties of the film. The preliminary FEM studies demonstrated that this method can be applied to simulation of nanoindentation experiment and is used to fit experimental load-displacement data for nanoindentation test.

2. SAMPLE PREPARATION

Composite C-Pd films were obtained by Physical Vapour Deposition process (PVD). This process was performed from two separated sources, one containing C_{60} fullerenes and other containing palladium acetate. PVD process was carried out in a dynamic vacuum of $\sim 10^{-5}$ mbar. The deposition rate was about 5 nm/min and the temperature of the substrate was 180°C [1]. Fig.1 presents typical TEM image of C-Pd film. Palladium nanograins have an oval shape with a diameter 10 to 15 nm. The thickness of the film is ~300 nm. Pd nanograins are of *fcc* type. In Fig.1b Pd <111> planes for such *fcc* structure are visible.

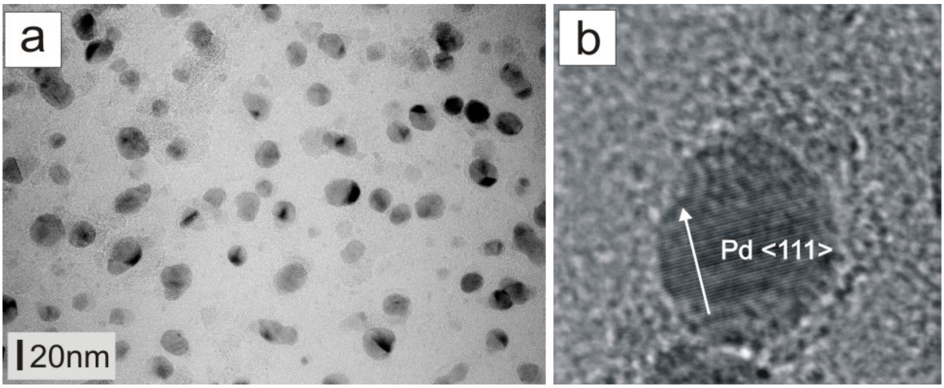

Fig. 1. a) TEM image of C-Pd film, b) high resolution image of Pd grain.

3. THE NANOINDENTATION TECHNIQUE

Nanoindentation experiments were performed using the electrostatic transducer of the Hysitron Nanoindentor Triboscope attached to a scanning force microscope (SFM). The indenter can be used for scanning over the sample surface for imaging. If nanoindentation is combined with surface imaging, then nanomechanical properties can be locally determined. Traditionally, the load of indenter tip causes penetration into a specimen and in this way tip reaches a desired depth. At this point, the load of tip could be stabilized for a moment or removed. In nanoindentation small loads value and small sizes of tip are used. Then, the indentation area may have a few square micrometres or even nanometres. The depth of penetration versus a force is obtained and then the area of the indent is determined with the known geometry of the indenter tip. A record of these values can be plotted on a graph to create a force-depth curve. The shape of the curve provides information on mechanical properties of material (nanohardness, elastic modulus, etc.).

The indenter tip is made of diamond and has 90° pyramid shape. Schematic geometry of the indenter tip is shown in Fig. 2. The angle between (001) and (111) planes is 54°44'08" [4].

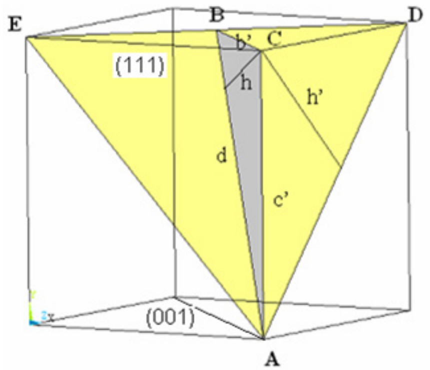

Fig. 2. Schematic geometry of the indenter tip.

In this case the cross-section area (plane AED in Fig.2) is given by:

$$A_c = \frac{3\sqrt{3}}{2}h^2 = 2.598 \cdot h^2 \tag{1}$$

Force versus depth curve (Fig.3) provides much more information than a microscopic image (elastic and plastic deformation with increasing and decreasing load) and permits to calculate nanohardness and reduced modulus as a function of penetration depth [8].

From the F-D plot one obtain following parameters: maximum indentation depth (h_{max}) which includes elastic and plastic deformation, depth of the remaining impression after complete unloading (h_f) and maximal applied force (F_{max}). Based on this data and Oliver–Pharr theory it is possible to determine the nanohardness (NH) and reduce the modulus of elastic (E_r) [3].

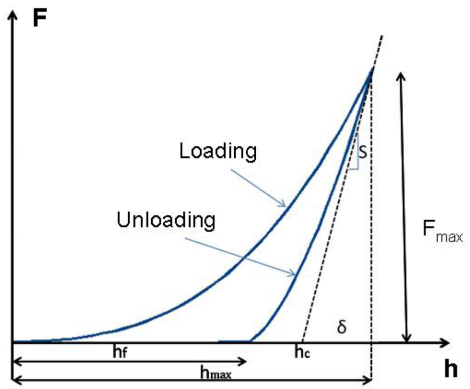

Fig. 3. Plot of force-depth (F-D plot) dependence.

The nanohardness of the sample (*NH*) is obtained from the formula [5, 6]:

$$NH = \frac{F_{\max}}{A_c(h_c)} \tag{2}$$

where F_{max} is the maximum applied force and A_c is the cross-sectional area corresponding to the contact depth h_c. The contact depth h_c is given by:

$$h_c = h_{\max} - 0{,}75\,\frac{F_{\max}}{S} \tag{3}$$

where *S* is the contact stiffness:

$$S = \frac{dF}{dh} \tag{4}$$

with *dF/dh* being the slope of the unloading curve at the initial point of unloading.

The reduced modulus E_r is a measure of the elastic properties of the tip-sample system and can be calculated from the force-depth curves according to the formula:

$$E_r = 1/2\sqrt{\pi / A_C(h_C)}\, dF/dh\,. \tag{5}$$

4. RESULTS

For numerical modelling nanoindentation experiment FEM and ANSYS program (Ansys, Inc) were applied. In our model C-Pd film is deposited on a glass substrate and it is indented by means of a diamond indenter as shown in Fig. 4. In the carbon layer spherical objects, representing Pd nanograins, were located.

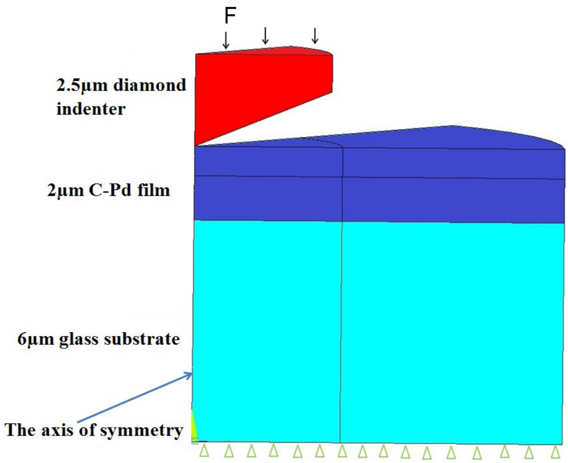

Fig. 4. Axisymmetric model of the C-Pd film and tip.

The thickness for the model of C-Pd film is 2 μm and the glass substrate is 6 μm. This thickness was much higher than for real C-Pd film, but it was necessary for our model to eliminate the influence of substrate effects on results. In order to properly simulate the nanoindentation experiment a contact analysis was applied. For this purpose, contact elements were placed along the top surface of the film and target elements were used along the bottom surface of the tip. The contact is assumed to be frictionless. The indentation experiment was simulated by applying displacement boundary conditions in the y-direction to the nodes along the upper surface of the tip. The top surface of the tip was constrained in all directions. Since the problem possesses y-axis symmetry then the model was analyzed in 1/8 of the geometry. Displacements along the symmetry planes were constrained.

Ratio of nanograins size and tip diameter determines a scale of the problem. The plastic deformation after unloading for the model of C-Pd film is shown in Fig. 5a. In Fig. 5b the displacement of palladium nanograins due to tip reaction is presented. The colorful spherical shapes show palladium nanograins position after this reaction. Object with a gridding shows the position of Pd grains before the reaction indenter (preliminary position).

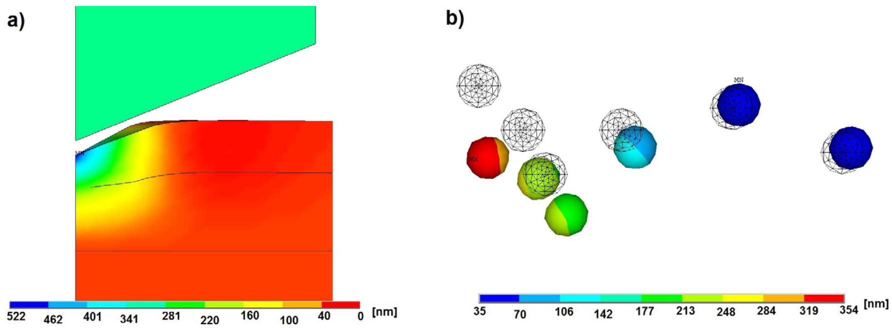

Fig. 5. Model of C-Pd film, a) displacement vector sum for the whole film b) displacement vector sum Pd nanograins.

Spherical shape of the tip becomes important when one wishes to perform nanoindentations on thin films (with a thickness of less than 500nm) and when the maximum depth of penetration of tip is ~50 nm [6]. A real indenter can therefore be modelled as a spherical indenter. The scheme of the spherical shaped tip in the simulated nano-indentation experiment is shown in Fig. 6.

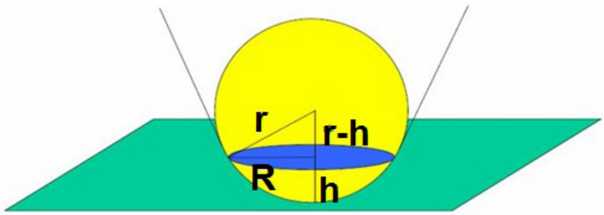

Fig. 6. Scheme of spherical shaped tip in the simulated nanoindentation experiment.

In nanoindentation experiment the SFM is used to give the contact area of the tip with the surface using the bearing function mode. In the case of a *cone* indenter with a perfect *spherical tip* the total cross-section area A_c is given by:

(5) $A_c = -\pi h^2 + 2\pi r h$

For $r >> h$, $A_c \approx 2\pi r h$ according to [7]

In FEM mode the sphere-conical tip was replaced by a sphere. The location of Pd grains in the carbonaceous layer is not symmetric. The deformation of the C-Pd film model in the FEM simulated nano-indentation experiment with the spherically shaped tip is shown in Fig. 7.

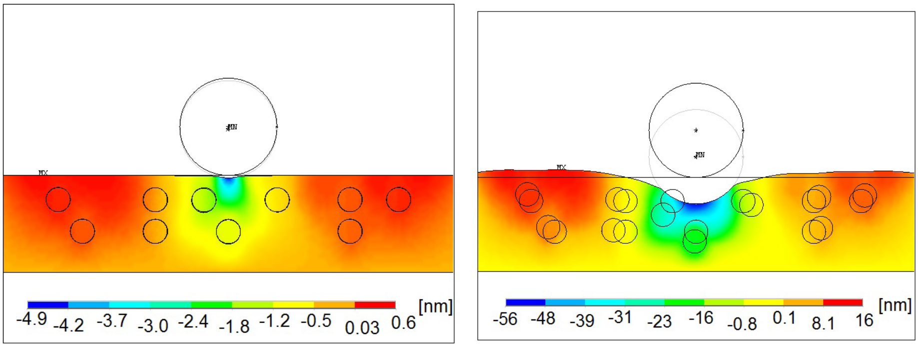

Fig. 7. Plastic deformation of C-Pd film in time.

Fig. 7 shows that the deformation for a carbonaceous-palladium film is not symmetrical. Nanograins are moved in the carbonaceous matrix toward the film's surface and create piling-up on the surface of the film in a contact area of the tip's penetration region. Deformation inside the film's depends on the distribution of grains in this film.

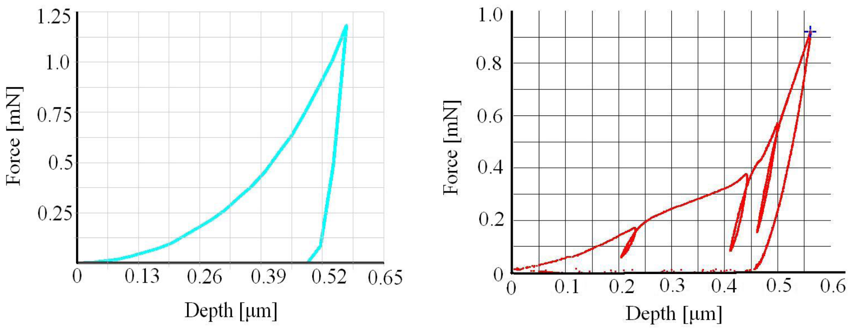

Fig. 8. Force-depth curve a) FEM simulation, b) experimental test.

Nanoindentation analysis results for C-Pd film are presented in Fig. 8. F-D plots were obtained as a result of the FEM simulation (Fig.8a) and nanoindentation experiment (Fig. 8b). The calculated plot reflects experimental curve. The analysis of these curves shows that hardness of C-Pd film is ~1 GPa and reduced modulus is ~20 GPa.

This compatibility of experimental and simulated data allow to predict of Pd nanograins behavior inside the C-Pd film (palladium displacement in carbonaceous matrix, stress) during the nanoindentation test.

4. CONCLUSIONS

We can deduce from our modeling experiment that the mechanical properties of obtained C-Pd films are connected to the nanostructure of these films. The C-Pd carbonaceous film has completely different mechanical properties in comparison to pure C_{60} films as well as to pure palladium film. From FEM results one can conclude that Pd nanocrystals are moving in carbon matrix toward the film surface due to an external stress.

FEM simulation also shows that deformation of the model of the C-Pd film is not symmetrical. Pd nanograins move toward the film's surface in the carbonaceous matrix. They also create piling-up on the surface of the film in a contact area of the penetration region. Changes in the form of film undergoing in the film volume depend on the distribution of grains in this film.

Observed effects are important because of practical applications of films as MEMS devices. Various mechanical properties are connected to differences in the structure and the composition of obtained films.

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