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MODELLING AND EXPERIMENTAL VERIFICATION OF NANOINDENTATION TESTS ON COATING-SUBSTRATE SYSTEMS

MODELOWANIE I EKSPERYMENTALNA WERYFIKACJA TESTÓW NANOINDENTACJI DLA UKŁADÓW POWŁOKA–PODŁOŻE

Key words:

nanoindentation, FEM modelling, thin coatings

Słowa kluczowe:

nanoindentacja, modelowanie MES, cienkie powłoki

Summary

The nanoindentation method is widely used to study the mechanical properties of thin films and surface layers. But the results of such a tests are difficult to interpret, especially for coatings with a thickness less than 1 μm. A new method of analysis of nanoindentation results are presented in this paper. It is based on simultaneous studies of experimental results by transformation indentation curves into stress-strain curves and FEM modelling. This method was compared with a few models provided in published literature. The influence of substrate

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properties and coating thickness on the deformation of the system and the critical depth of penetration to onset substrate yield are analysed.

INTRODUCTION

Analysis of the mechanical properties of materials at the micro- and nanoscale has become a necessity as a result of the development of thin coatings which are now widely used in tribological applications. Determination of hardness and elastic modulus of coatings is a very important issue because of the decisive influence of both of these parameters on the deformation mechanism of coating-substrate systems and their wear. For thin coatings milinewton range have to be used as testing loads. This follows from the demand of restriction of deformation only to a coating, what exclude the influence of substrate on the measurement results. In this area of mechanical testing the instrumental indentation technique (micro, nanoindentation) based on continuous measurement of the load and penetration depth during the test is commonly used. Nevertheless the analysis of the test results are still not sufficiently developed and the literature shows a much controversy on this field **[L. 1]**. This paper presents the problems associated with indentation testing of coating--substrate systems and the mathematical models used to interpret the test results. On this background, the authors' own method of test results analysis based on the procedure of transformation of indentation curves into stress-strain curves is presented. The method is described in details in previous paper **[L. 2]**. Indentation tests were also modeled using finite element method. Experimental verification was performed for ZrN coatings deposited on various substrates.

INDENTATION TESTING

Indentation tests were performed on MCT-CSM Instruments machine using a Berkovich geometry diamond and 5–500 mN load range. 2 μm thick ZrN coatings deposited by PLD (Pulsed Laser Deposition) technique on X10CrNi18-8 austenitic steel, X6Cr13 ferritic steel and silicon substrates were examined. Substrates' hardness was 4, 8 and 11GPa respectively, while ZrN coatings have hardness 26–27 GPa. The indentation hardness were compared to FEM modeling results and results obtained by applying models presented in the literature and authors' own model. All this analysis are presented in the following parts of this work.

MODELS OF COATING–SUBSTRATE COMPOSITE HARDNESS

Commonly accepted for indentation of the coating-substrate systems 1/10th rule recommends that the penetration depth should not be greater than 10% of coating thickness in order to measure the intrinsic coating properties. Hence the tests must be carried out under few milinewtons or even lower loads. Results of such a tests are usually characterized by a large scatter arising from errors of indenter shape calibration and sample roughness. In addition, substrate yield can occur before the coating reach fully plastic regime of deformation. In this case, the only one possibility remains. Large number of tests with a wide range of loads must be performed. And then the hardness results changing with penetration depth must be analyzed by applying one of the hardness models of coating-substrate composite H_k. The most frequently cited in the literature models and equations for calculations of composite hardness are summarized in **Table 1**. These relationships usually involve hardness of coating H_C, substrate H_P and the relative penetration depth RID = P_d/t (penetration depth/coating thickness). By using an inverse analysis hardness of the coating can be calculated from results of measurements performed under higher load when the substrate is also plastically deformed.

Table 1. Summary of hardness models for coating-substrate systems
Tabela 1. Zestawienie modeli twardości dla układów powłoka–podłoże

Model and designation	Equation for coating-substrate hardness calculation	Symbols
Jonsson i Hogmark [3] *J-H*	$H_k = H_p + \left[\frac{2 \cdot C}{7 \cdot RID} - \left(\frac{C}{7 \cdot RID}\right)^2\right] \cdot (H_c - H_p)$	C – coefficient, values range 0,5–1
Burnett i Rickerby [4] *B-R*	$\mathrm{H_k = H_p + 3 \cdot (H_c - H_p) \cdot \left(\frac{H_c}{E_c}\right)^{\frac{1}{2}} \cdot \frac{1}{7RID} \tan \xi^{\frac{1}{3}}}$	ξ – indenter apex angle, E_c – coating elastic modulus
Korsunsky [5] *Kor*	$\mathrm{H_k = H_p + \frac{H_c - H_p}{1 + k \cdot RID^c}}$	k, c – coefficients
Chicot i Lasage [6] *Ch-L*	$\mathrm{H_k = H_p + \left\{\frac{3}{14 \cdot RID} \cdot \left[\left(\frac{H_c}{E_c}\right)^{\frac{1}{2}} + \left(\frac{H_p}{E_p}\right)^{\frac{1}{2}}\right] \cdot \tan \xi^{\frac{1}{3}}\right\} \cdot (H_c - H_p)}$	E_P – substrate elastic modulus
Bhattacharya i Nix [7] *Bha*	$\mathrm{H_k = H_p + (H_c - H_p) \cdot e^{-a \cdot RID^n}}$	a, n – coefficients

The curves fitted using these models to the indentation results of 2 μm ZrN coatings on X10CrNi18-8 steel substrate are shown in Figure 1a. The square markers correspond to the experimental results. The best approximations were obtained for *Bha* and *Kor* models. Figure 1b shows the approximation using the authors' method (σ/ε model) involving complex analysis of indentation and FEM modeling results **[L. 3]**. Modeled changes of a mean pressure in the contact area with a high compliance match to hardness results.

FEM MODELLING

FEM modeling was performed using the ADINA System v.8.6 within elastic-plastic deformation regime of the coating and substrate **[L. 8, 9]**. Model of the analyzed indenter-coating-substrate system is shown in **Figure 2a**. It was assumed that the coating and substrate are perfectly connected. Rigid conical indenter with an angle α = 140.6°, tip radius R = 150 nm (equivalent to Berkovich geometry) was pressed into the coating surface with increasing load from 0 to 15 mN, and then was unloaded.

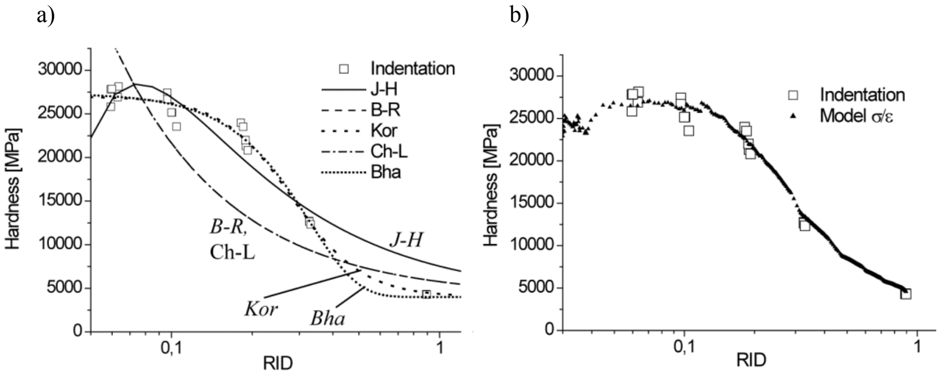

Fig. 1. The modeled and measured hardness changes of 2μm ZrN/X10CrNi18-8 system as a function of RID using: a) literature models - Table 1, b) σ/ε model

Rys.1. Zmiana mierzonej twardości układu ZrN-2μm/X10CrNi 18-8 w funkcji RID przy użyciu: a) modeli zestawionych w **Tabeli 1**, b) modelu σ–ε

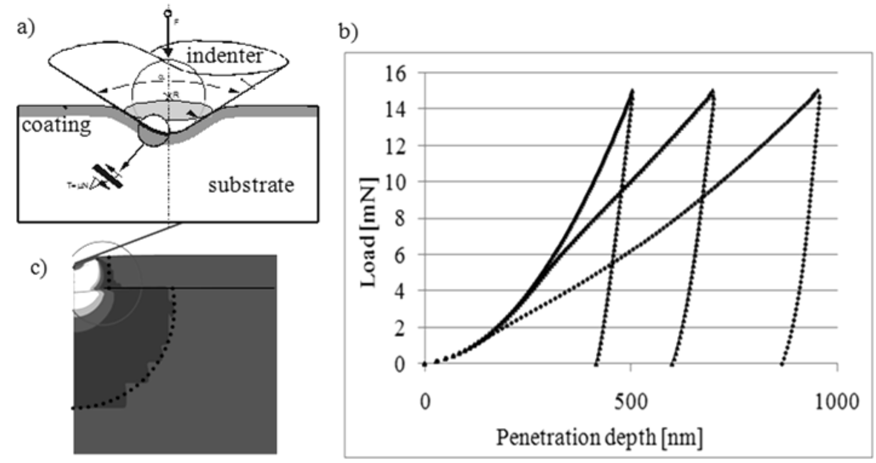

Fig. 2. Results of FEM modeling: a) model, b) indentation curves, c) range of plastic deformation

Rys. 2. Modelowanie indentacji układu powłoka–podłoże: a) model, b) krzywe indentacyjne, c) zakres odkształceń plastycznych

Typical modeled indentation curves for coatings with thicknesses of 1, 2 and 5 μm are shown in **Figure 2b**. The effect of coating thickness on the deformation range of the whole system is clearly visible. Initially, the deformations of all systems are the same, what indicate the lack of substrate deformation. The load increase causes that the plastically deformed zone reaches the substrate, and the indentation curve changes its slope. For t = 1 μm thick coating this change of slope was found at 120 nm penetration depth , while for thicker coating t = 2 μm at 230 nm. Both of these values are slightly higher than 10% of coatings thickness. Range of coating and substrate yield for t = 2 μm coating under 15 mN load is shown in **Figure 2c**. The values of RID to onset substrate yield are presented in **Figure 3**. These values can be regarded as a critical parameter to obtain intrinsic properties of coatings. The strong impact on this parameter has a ratio of substrate to coating yield strength Y_P/Y_C. The lower the ratio, the substrate yield occurs at lower RID **(Fig. 3a)**. For the softest substrate $Y_P/Y_C = 0{,}033$ plastic deformation starts at RID = 0.035, while for $Y_P/Y_C = 0{,}5$ the indenter must be pressed much deeper to RID = 0.15. Whereas, the coating thickness does not play a meaningful role on the RID critical value **(Fig. 3b)**. For each loading steps the contact area and mean pressure was calculated.

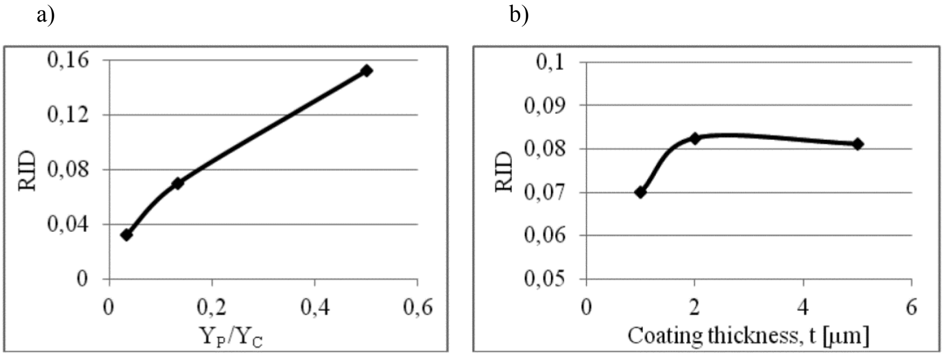

Fig. 3. Critical RID to onset substrate yield as a function of: a) Y_P/Y_C, b) coating thickness
Rys. 3. Krytyczna wartość RID w funkcji: a) R_{eP}/R_{eC}, b) grubości powłoki

Changes of contact stresses were analyzed as a function of RID. The graphs were normalized to 0–1 range (0-substrate hardness, 1-coating hardness) by calculating relative hardness RH = $(H_k - H_p) / (H_c - H_p)$. The similar character of hardness changes with increasing RID for all coatings thickness are shown in **Figure 4a**. Changes at low RID < 0.1 indicate that for thinner coatings it is more difficult to induce the fully plastic regime and the plateau on the RH/RID curve is getting narrower. In the same way the results of indentation tests were normalized for ZrN coatings on all tested substrates **(Fig. 4b)**. At the beginning of indentation the changes of RH for all systems are the same regardless the substrate. That shows a lack of substrates effect on the deformations in this range. For coating on austenitic soft steel hardness begins to fall when RID excides 0.08 and at RID = 1it reaches the substrate hardness. For harder ferritic steel and silicon substrates initial hardness decrease is observed at higher RID = = 0.13 and 018 respectively, that confirms the FEM modeling results **(Fig. 3a)**. The curve for 2 μm coating in Fig. 4a obtained from modeling corresponds with a high accuracy to σ–ε curve **(Fig. 4b)**.

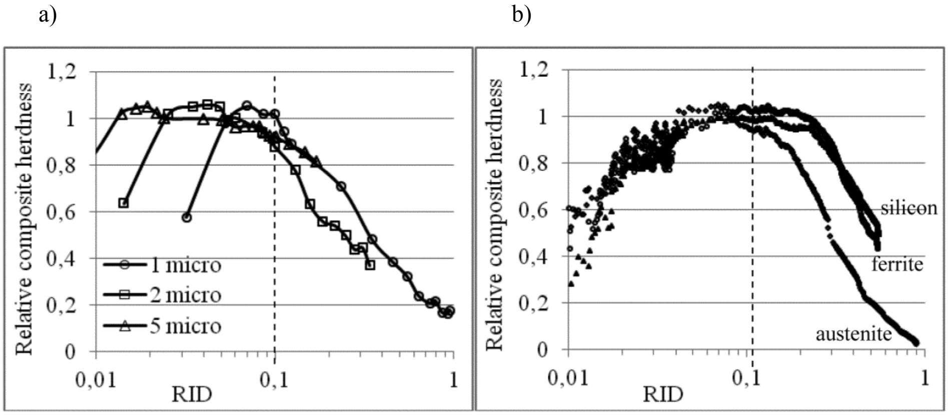

Fig. 4. Relative composite hardness of coating-substrate systems changes as a function of RID from: a) FEM modelling, b) indentation results of ZrN coatings

Rys. 4. Jednostkowe zmiany twardości układu powłoka–podłoże w funkcji RID wyznaczone z: a) modelu MES, b) z testów indentacyjnych powłok ZrN

Conclusion

Complex analysis of indentation tests based on the FEM modeling and according to the method of transformation indentation curves into stress-strain curves allows proper interpretation of test results. This authors' method shows possible measurement errors as a result of too high and also too low load. The former leads to substrate yield and lower hardness values obtained for hard coatings deposited on softer substrates. The critical RID at which the substrate begins to affect the measurement results depends mainly on the ratio of plastic properties of the coating and substrate Y_P/Y_C. For analyzed range of Y_P/Y_C=0,03–0,5 critical RID increases from 0.03 to 0.15. On the other hand, too low load results in too low pressure in the contact zone to induce coatings yield. Of course, the results of indentation tests in both conditions can be used to compare different coatings but do not provide exact values of coatings plastic properties. From both of these limits derives a possibility of substrate yield before the fully plastic regime of coating is reached. In such a case the results interpretation can be based on one of the models presented in the literature, but their application requires performing a lot of tests at wide range of RID. At this background analysis of indentation results using authors' method and plotting stress-strain curves seems to be more effective, less time consuming and gives information on deformations in a whole RID range.

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Streszczenie

Metoda instrumentalnej indentacji jest powszechnie stosowana do badania właściwości mechanicznych cienkich powłok i warstw wierzchnich. Wciąż jednak wyniki testów nanoindentacyjnych są trudne do interpretacji zwłaszcza dla powłok o grubości poniżej 1 μm. W pracy przedstawiono metodę analizy wyników badań indentacyjnych w oparciu o badania rzeczywistych układów powłoka–podłoże oraz wyniki modelowania MES. Badano wpływ właściwości podłoża i grubości powłoki na deformacje układu i krytyczną głębokość penetracji, przy której podłoże nie ma wpływu na pomiar. Do analizy wyników eksperymentów zaproponowano metodę transformacji krzywych indentacyjnych do układu naprężenie–odkształcenie. Metodę tę porównano z modelami podawanymi w literaturze.