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IMPROVING THE ACCURACY OF MICROHARDNESS MEASUREMENT OF NANOELECTRONIC ELEMENTS BY THE SILICIC PROBES OF ATOMIC-FORCE MICROSCOPY, THAT IS MODIFIED BY CARBON COVERAGE

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ABSTRACT:
Possibility of measuring of microhardness of different surfaces of elements of nanoelectronics by means of method of atomic-force microscopy is considered in the article. Possibility of application of silicic probes that is modified by carbon coverage is first shown, that allows to conduct complex researches of surfaces and ultrathin coverages. Dependence of measuring exactness is shown on the microhardness of the investigated material. The range of measuring of microhardness of elements of nanoelectronics is set by such probes. Measuring exactness is megascopic to 20-28%, which is a range from 100 MPa to 39 GPa. It extends the nomenclature of materials at determination of its microhardness by atomic-force microscopy.

Keywords: *atomic-force microscopy, microhardness, nanoelectronics, silicic probe, accuracy*

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1. INTRODUCTION

The nanoindentation method has become more and more popular in recent years due to a decreaseof components size and an increased interest in the mechanical characteristics of elements in the nanometric range [1]. The peculiarity of the manifestation of the mechanical properties of materials at the nanoscale is associated with the manifestation of size effects (the theory of W. Nixon and H. Gao), as well as the possible diversity of the material in phase composition at the nanoscale and a sharp elastic-plastic transition in single crystals of materials (especially metals) [2, 3].
At the same time, methods for measuring the microhardness and elastic properties of materials, as well as thin coatings along the depth of the nanoindentor imprint, are generally recognized in the materials science society. These methods are based on the analysis of the diagram of nanoindentor penetration into the sample (for example, the methods proposed by W. Oliver, J. Farom [4] (using a pyramidal indenter); J. Field, M. Swain, A. Fisher-Crips [5] (for spherical indenters).
A significant number of methods for processing primary data based on the results of nanoindentation using the Berkovich indenter, developed in recent years, are based on the use of scanning probe microscopy (SPM) methods, which makes it possible to determine more than two dozen different mechanical characteristics of materials

(microhardness, wear resistance, tribological properties, etc.). Thus, the use of methods for determining microhardness using SPM, namely [6]: microsclerometry, restoration of the "tension-compression" curve of the probe when it is brought to the surface, restoration of the indenter unloading curve without obtaining an image of the imprint, and others greatly simplify the acquisition and processing of test results. However, this does not take into account the possible deviation of the indent from the shape of a regular triangle due to the formation of piles and / or the subsidence of the sample surface on the sides of the indent, which can distort the study results.

The expediency of using the atomic force microscopy (AFM) method for studying the microhardness of dielectric surfaces lies in the similarity of the principle of operation of a nanohardness tester and an atomic force microscope, which allows them to be combined in one complex, thereby significantly expanding the capabilities of probe methods and making them one of the most popular methods of metrological research in nanotechnology.

At the same time, the application of the atomic force microscopy method for nanoindentation is one of the most promising in the complex study of the mechanical properties of dielectric materials and thin coatings (less than 100 nm) on their surface [7]. With such a study, during scanning of the sample relief, selective local measurement of interfacial zones or inclusions of micro- and nanometric sizes in the sample surface in one scanning cycle is possible [4].

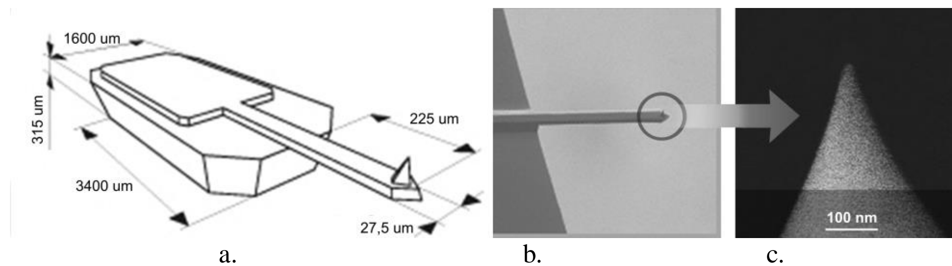
However, the main problem of the active application of this approach to study the mechanical characteristics of dielectric materials is the need for the correct choice of a probe for AFM. Typically, a silicon probe is used for scanning a sample, and a diamond probe is used for nanoindentation. None of them are suitable for scanning and indentation in one cycle, since a silicon probe is too fragile for nanoindentation, and the use of a diamond probe for scanning dielectric materials is not rational due to its high cost and high hardness, which leads to the destruction of soft samples and, as a result, to distortion of scan results.

The most promising approach to solve this problem is the modification of the surfaces of conical silicon probes with a carbon functional coating used to scan solid materials in the contact mode. In this case, an effective method for obtaining such coatings is the method of thermal evaporation in vacuum, the deposition technology of which is described in the author's work [8].

The purpose of this work is to determine the accuracy of microhardness measurement by the AFM method using silicon probes modified with a carbon coating by comparing these results with the data obtained by the certified Vickers microdentation method.

2. EXPERIMENTAL PROCEDURE

Tests of the microhardness of dielectric surfaces were made using an NT-206 atomic force microscope (Mikrotestmashiny, Belarus). The main tool in the research was silicon conical probes (tip radius of 8 nm) of the CSC18 brand (NT-MDT, Russia), modified with a thin carbon coating (Fig. 1).



a. b. c.

Fig.1. External view of the console (a), diagram of the sensitive element of an atomic force microscope (b) and micrograph of the tip of the CSC18 probe (NT-MDT, Russia) (c), modified with a thin carbon coating

Modification is based on the formation of ultrathin (up to 10 nm thick) carbon coatings obtained by thermal deposition in vacuum $(5...6)\cdot10^{-3}$ Pa according to the method described in [8]. Previous studies held in [9] showed high strength, hardness and wear resistance of such coatings.
Nanoindentation of the surfaces of the sample materials with a silicon conical probe modified with a thin carbon coating using the AFM method is used in the following sequence.
The sample is scanned to select the nanoindentation site (nanoindentation site in a clean, homogeneous area, without inclusions and abrupt relief changes).
The process of nanoindentation of the surface is carried out in the selected place of the investigated surface of the coatings (thickness 20 nm) under a gradually increasing load and under the following modes: for SiO_2 coating - at a maximum load of up to 0.8 mN in 5 seconds; for HfO_2 coating - at a maximum load of up to 0.6 mN in 7 seconds; for Au coating - at a maximum load of up to 0.5 mN in 7 seconds.
From the unloading curve of the indenter - a silicon conical probe (the general view of the curve is shown in Fig. 2), the projection area of the indent under the maximum load is determined when the probe penetrates the surface using the following formulas:
- when the probe is introduced to a depth of $h \leq 10$ nm:

$$A_c = \pi \cdot r \cdot h_c \quad (1),$$

- when the probe is introduced to a depth of $h > 10$ nm:

$$A_c = \frac{\pi \cdot (r_1 + r) \cdot (h_c - r)}{\cos\varphi} + \pi \cdot r^2 \quad (2),$$

where r is the radius of the tip of the probe; h_c - indentation depth under maximum load; r_1 is the radius of the probe base; $r_1 = h_c \cdot tg\varphi$; φ is the slope angle of the tip of the conical probe (for probes CSC18: $\varphi = 25°$).

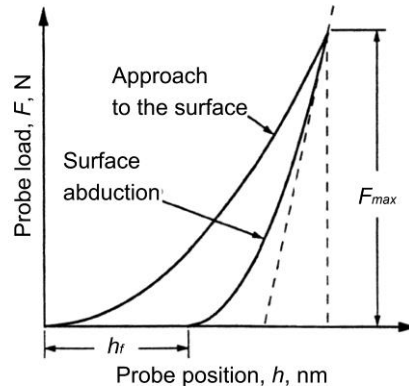


Fig.2. General view of the curves of loading and unloading of the probe - nanoindenter: F_{max} - maximum load of the probe on the surface; h_f - distance in height from the point of penetration of the probe into the surface to the point of its retraction

Further, substituting the value of r_1 into the formula (2):

$$A_c = \frac{\pi \cdot (h_c \cdot tg\varphi + r) \cdot (h_c - r)}{\cos\varphi} + \pi \cdot r^2$$

or, after simplification (cos φ = 0,906; tg φ = 0,466):

$$A_c \approx 1{,}1 \cdot \pi \cdot (0{,}466 \cdot h_c + r) \cdot (h_c - r) + \pi \cdot r^2 \qquad (2')$$

Calculate the microhardness of the sample material under study:

$$H = \frac{F_{\max}}{A_c}, \qquad (3)$$

where F_{max} is the maximum probe load on the surface; A_c is the projection area of the probe imprint on the surface of the material under study.

The accuracy of the results obtained is determined by comparing the data with the certified Vickers microindentation method using a diamond pyramid on the device DuroScan-10/20.

3. DISCUSSION OF THE EXPERIMENTAL RESULTS

As a result of the experiments conducted to measure the microhardness by atomic force microscopy for various materials, which have found application in nanoelectronics and their further comparison with the certified Vickers microindentation method, the dependences of the accuracy of determining the microhardness of various materials for modified and unmodified AFM probes on the depth of probe penetration into the investigated surface, fig. 3.

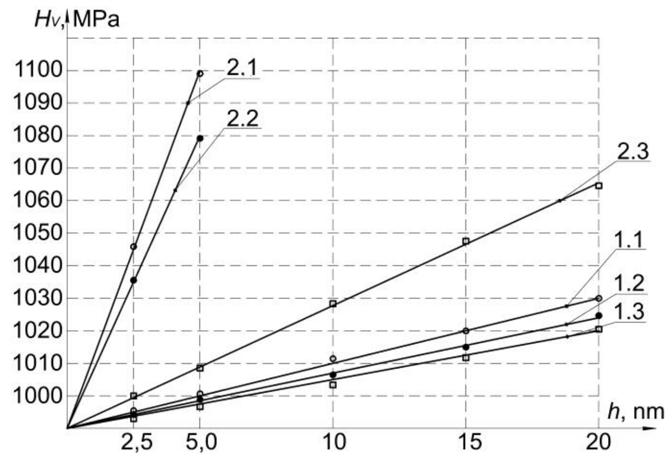


Fig.3. Dependences of the deviation of the microhardness of thin coatings on a silicon substrate on the depth of penetration of the nanoindenter into the surface:
1.X - for a probe modified with a carbon coating;
2.X - for unmodified silicon probe.
○, 1 - respectively, obtained experimentally and approximated data for SiO_2 coating;
●, 2 - respectively, obtained experimentally and approximated data for HfO_2 coating;
□, 3 - respectively, obtained experimentally and approximated data for Au coating.

As can be seen from the dependences shown in Fig. 3, with the increase in the penetration depth of the unmodified silicon probe, the deviation of the microhardness of thin coatings increases from the initial value of 1.00 to 1.063 (which corresponds to + 6.3% deviation of the microhardness measurement accuracy) for the Au surface according to an exponential law, as well as for the results obtained for probes modified with a carbon coating (the maximum value of 1.03 (deviation of microhardness measurement accuracy by 3%) corresponds to SiO_2 coating). At the same time, the accuracy of measuring the microhardness for SiO_2, HfO_2 coatings using unmodified probes is only reduced to values of the nanoindentor penetration depth into their surface of 5 nm. This is due to the occurrence of critical mechanical loads, leading to the destruction of such a probe when it penetrates to a great depth.
The limiting values when using the nanoindentation method in the study of dielectric surfaces with conical AFM probes modified with a thin carbon coating were calculated based on the load range of the order of 0.05-1.25 mN (for silicon probes 0.05-0.09 mN), which was applied to the probe in the process of measuring the microhardness, as well as the maximum penetration depth, which was about 60 nm (for a silicon probe 180 nm). Based on a formula (3), the range of microhardness measurements by probes modified with a thin carbon coating was obtained: 100 MPa - 39 GPa, and for silicon probes: 477 MPa - 11 GPa.

4. CONCLUSION

The deviation of the microhardness of thin coatings measured by the considered method of atomic force microscopy (NT-206) using CSC18 silicon probes modified with a carbon coating in comparison with the Vickers microindentation method on the DuroScan-10/20 device increases from 2.1% (for the coating surface Au) up to 3% (for SiO_2 coatings) almost linearly.
The range of microhardness measurements by silicon probes modified with a thin carbon coating is increased by 20-28% and amounts to 100 MPa - 39 GPa, which makes it possible to expand the range of materials under study when determining their microhardness.

5. REFERENCES

[1] A.R. Karimzadeh, S.S.Koloor, M.R.Ayatollahi,(2019)Assessment of Nano-Indentation Method in Mechanical Characterization of Heterogeneous Nanocomposite Materials Using Experimental and Computational Approaches,*Sci.Rep.*, 9 : 15763.

[2] W.D.Nix, and H.Gao (1998) Indentation size effects in crystalline materials: A law for strain gradient plasticity, *J. Mech. and Phys. of Solids*, 46 (3): 411-425.

[3] Mao Liu, Jhe-yu Lin, Cheng Lu, Kiet Anh Tieu, Kun Zhou, and Toshihiko Koseki, (2017) Progress in Indentation Study of Materials via Both Experimental and Numerical Methods, *Crystals*, 7 (10) : 258.

[4] W.C.Oliver, and G.M.Pharr (1992) An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, *J. Mater. Res.,* 7 (6) : 1564-1583.

[5] Fischer – Cripps A.C. (2002) *Nanoindentation*,Springer-Verlag, New-York, USA.

[6] C.A. Charitidis, D.A. Dragatogiannis, Elias P. Koumoulos, and D. Perivoliotis (2016) *Mechanical, Tribological Properties, and Surface Characteristics of Nanotextured Surfaces*, in book: *Nanomaterial Characterization*, John Wiley & Sons, Athens, Greece.

[7] J. Notbohm, B. Poon, and G. Ravichandran(2011) AnalysIis of nanoindentation of soft materials with an atomic force microscope,*J. Mater. Res.*,27 (1) : 229-237.

[8] V.S.Antonyuk, S.O.Bilokin, M.O.Bondarenko, Yu.Yu.Bondarenko, and Yu.I.Kovalenko (2015) Formation of Wear-Resistant Coatings on Silicon Probes for Atomic Force Microscopy by Thermal Vacuum Evaporation, *J. Superhard Mater.,*37 (2) : 112-119.

[9] V.S.Antonyuk, Yu.Yu.Bondarenko, S.O.Bilokin’, V.O.Andrienko, and M.O.Bondarenko (2019) Research of microhardness of thin ceramic coatings formed by combined electron-beam method on dielectric materials, *J. Nano-Electron. Phys.*, 11 (6) : 06024-1–06024-5.