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NECESSITY OF RUSSIAN NANOINDENTATION STANDARD BASE DEVELOPMENT

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Abstract – Over the past years a large number of papers have been published on the application of the nanoindentation method. These works describe various aspects of the nanoindentation method and provide a detailed overview of the characteristics of different hardware and software tools implementing this method. However, measurements taken under the same conditions and on the seemingly same samples often provide a significant discrepancy in the results on different instruments functioning according to the same principle. This paper discusses the issues of metrological support for the nanoindentation method in Russia.

Keywords: nanoindentation method, Martens hardness**,** primary standard machine

1. INTRODUCTION

Rapid advancement in nanotechnology has made the nanoindentation method [1], developed several decades ago, particularly sought-after in recent years. This has led to a fast increase in the number of domestic and foreign instruments implementing this method. The essence of the nanoindentation method lies in combined measurements of the force F, applied to the indenter, and the indenter displacement h while penetrating into the tested material. Such combined measurements help to determine the correlation between force and displacement F (h) during the processes of load application and removal. The analysis of these correlations enables to get data on both the hardness of the tested object and its other mechanical properties.

2. METROLOGICAL ASPECTS OF THE NANOINDENTATION METHOD

The application of the nanoindentation method is most efficient while studying mechanical properties of nano-objects – thin films and coatings, one- or multilayered, having the thickness of tens to hundreds nanometers. From the metrological viewpoint, the peculiarity of nanoindentation, that distinguishes it from kinetic indentation at the macro-level [2], lies in the following:

- small scales of tested force (mN and μN units) and displacement (nm units and tens);
- stricter requirements to the precision of these values measurements. This is explained by the fact that while analyzing the F(h) correlation and calculating the final values (Martens hardness, elastic modulus, etc.), it is necessary to use not only the measured values, but also their increments, which leads to a significant increase in the distortion factors influence;
- necessity to reliably determine not only the indenter shape, but also its deviation from the ideal shape caused by chips, cracks, and other defects close to its edge, i.e. within the lengths comparable with the indentation depth.

The peculiarities of nanoindentation require a more thorough preparation of the measurement process than the one required by micro- and macroindentation. In order to get reliable measurement results, such preparation concerns both measurement equipment and the measuring object. Moreover, the results of this preparation should be confirmed by special additional investigations helping to receive preliminary data about the object. In particular, it is necessary to make sure the surface is sufficiently clean and the object is homogeneous within the limits of the supposed penetration zone, as well as to determine the parameters of the surface roughness, the anisotropy and the rheological model. Sample mounting conditions during measurements and the substrate parameters may affect the measurement results as well. It is also necessary to conduct trial measurements to make sure the measurement process is stationary and ergodic, and the measuring object is representative and sufficiently homogeneous in its various parts. Of no less importance is the determination of the dispersion scale. Even if the measurement process is fully automated, it is essential to have a certified document for each type of equipment – that is the procedure of measurements that guarantees measurement results with standardized metrological characteristics. It is common knowledge that the most precise measurement results are obtained by the comparison against a test block or the substitution with a test block. These measurement methods help to get accurate measurement results, i.e. values with minimum systematic error, and to eliminate the measurement results drift in time and the influence of slowchanging factors (e.g. anisothermality of the measurement complex). In order to implement the method of the comparison with a test block, it is necessary to have the corresponding test blocks. Mass, length and roughness test blocks are currently available for testing the instruments that implement the nanoindentation method. However, these test blocks are not sufficient (e.g. for the control of indenter parameters). Besides, the afore-mentioned test blocks allow to perform just an element-by-element check of the measurement equipment status. Moreover, the check is carried out under conditions that differ from those of the instruments application, since the very process of indentation doesn't take place. To make sure the measurement results are accurate, it is not enough to develop the system of measurement instruments calibration by force and displacement and to define the functions of the indenter surface area. It is also important to elaborate the system of comparison with using hardness test blocks that are presently missing in the national State Register of Measuring Equipment.

All the described factors indicate that up to now there is no system for insuring the uniformity of measurements, and the uncertainty of the measurement results is unknown. Underestimation of the metrological aspects of investigation leads to the impossibility of an adequate estimation of nanoindentation results. This can be avoided by taking organizational, technical and legal measures that ensure the uniformity of measurements, creating an orderly metrological system of obtaining accurate measurement results in this field, as well as providing traceability to the state standards [3, 4]. For this end, FSUE "VNIIFTRI" has taken the following steps to create the national nanoindentation standard base:

- standardization of Martens hardness scales through the harmonization of ISO 14577 "Metallic materials - Instrumented indentation test for hardness and materials parameters";
- creation of the national primary hardness machine for Martens scales and the national verification schedule;
- elaboration and creation of hardness reference test blocks in the field of nanoindentation for the transfer of Martens hardness scales from the national primary hardness machine to working measuring instruments.

The structural diagram of the created primary standard machine for Martens scales is given below.

TI-750 Ubi **G 200** **GET – 31-06**

I__________II__________III__________IV

F=50 nN **F=100-200 nN** **F=100 mN** **F=980 N**

(1) **(2)** **(3)** **(4)** **(5)**

Here the hardness scale is split into 3 parts (I-II, II-III and III-IV), the two first are Martens scales, and the third is the Vickers scale. Hardness scale transfer occurs from national primary standard machine for Vickers scales GET - 31-06 [5] to the nanoindenter G 200 [6] by sets of reference blocks (4). G 200 transfers the scale to the nanomechanical test instrument TI-750 Ubi [7] by sets of reference blocks (2).The two last mentioned devices have been chosen proceeding from the criterion of more stable work depending on the magnitude of the applied forces. Sets of reference blocks (1), (3) and (5) are designed for calibration of the primary standard machine in the scale ranges given in the above diagram, and also for the scale transfer to the working measuring instruments.

Indentation modulus is largely independent of indentation depth, but this is not the case for indentation and Martens hardness. In the latter case the indentation depth/force has also to be considered at calibration of reference blocks.

3. CONCLUSIONS

The creation of the standard base in the field of nanoindentation will contribute to a significant increase in the level of national developments in the field of nanotechnologies and nanoindustry, as well as an increase in their quality, importance and competitiveness.

REFERENCES

[1] W. C. Oliver, G. M. Pharr, "An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments", *J. Mat. Res*., vol. 7, pp. 1564-1583, 1992.

[2] S. I. Bulychev, V. P. Alekhin, M. Kh. Shorshorov, A. P. Ternovskii. and G. D. Shnyrev "Young's modulus determination by the indenter penetration diagram", *Manufacture laboratory*, vol. 41, № 9, pp. 1137-1140, 1975

[3] E. G. Aslanyan, "Necessity of uniform system for materials nanohardness measurement", *Proceedings of MEPhI scientific session*, vol. 7, pp. 195-196, 2008.

[4] P. A. Krasowski, E. G. Aslanyan, V. M. Borovkov, B. V. Yuryev, "Present-day Questions of Metrological Support of the Nanoindentation Method", *Proceedings of VNIIFTRI*, part 55 (147), pp. 8-10, 2009.

[5] Interstate standard GOST 8.063-2007. State system for ensuring the uniformity of measurements. State verification schedule for means measuring the metals and alloys hardness on Vickers scales.-Standardinform, Moscow, 2007.

[6] http://www. agilent .com/

[7] http://www.hysitron.com/