Metrological method for determining the surface area function of the nanohardness testers tips

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Abstract. The paper describes a method for measuring the surface area function of the tips of the probes of nanohardness testers using a laser interferometer. The features of the developed system provide traceability of measurements to the standard of length through the wavelength of a stabilized source of laser radiation. In addition, it is possible to measure the parameters of the probe tip without removing it from the nanohardness tester, which allows the device not to be taken out of working condition and provides unambiguous measurements.

1. Introduction
To measure hardness on a submicron and nanometer scale, static loading methods are used: pressing a known-shaped tip (indenter) into a material (nanoindentation) [1] and scratching (sclerometry) [2]. The international standard ISO 14577: 2002 regulates the determination of hardness as the ratio of the measured value of the applied load to the surface area of the indenter. During indentation, the indenter footprint left in the test material when exposed with a given force is measured. The value of the surface area of the indenter is determined based on the shape of the tip used and is called the function of the area of the indenter. Typically, Berkovich diamond 3-sided pyramids (angle between the face and a height of 65 °), 4-sided - Vickers (angle between the sides of 136 °) or a pyramid in the form of a cube angle (angle between the face and a height of 35 °) are used for measuring indentation [3]. The choice of this form of indenters is due to the fact that when they are immersed in a homogeneous material, the strains and mechanical stresses remain constant at all penetration depths. Therefore, determining the surface area of the indenter knowing the area or depth of the print is not difficult. However, in the process of operation, the geometry of the indenter tip changes, the tip is rounded, edges are stitched, chips can occur. All this affects the function of the probe tip area and, as a consequence, the calculated values of the mechanical characteristics of the material. Conducting periodic measurements of the function of the area of the tips would solve these problems.

To determine the function of the surface area of the probe tips, two methods are mainly used: the method of measuring the areas of prints when indented with different loads in the reference material and [4], or direct measurement of the surface shape of the probe tip in a scanning probe microscope [5]. However, all these methods do not have direct traceability to the primary standard of length; therefore, they are not metrological. For metrologically correct measurement of the function of the surface area of the tips of the probes of nanohardness testers, a method was proposed for measuring the function of the surface of the tip using a three-axis linear displacement interferometer coupled with an atomic force microscope [6].
2. Laser interferometer
The developed interferometer has a number of features that made it possible to implement a system for measuring the parameters of nanohardness probes. A stabilized He – Ne laser with a power of 1mW with a relative instability of the radiation wavelength of 10–9 for 8 h of operation is used as a radiation source. Since the laser was an additional source of heat, which negatively affected accuracy, it was moved outside the optical circuit. The radiation was transported using optical fiber. An acousto-optic modulator (AOM) is installed in the path of the laser radiation introduced into the optical unit, which splits the laser beam into reference and measuring beams with mutually orthogonal polarization and provides a heterodyne frequency shift $f$ between them ($f = 36$ MHz). Further downstream, the polarizing beam splitter provides a spatial separation of the reference and measuring beams. A piezoelectric table, the movement of which is measured by an interferometer, is located below the plane of propagation of rays. A platform is fixed on it, in which three triple prisms are fixed. The peculiarity of their location allows you to control the movement of the table in three orthogonal directions and minimize the error of Abbe. As a result, the interferometer provides traceability of measurements to the length standard and has small dimensions that allow scanning the tip of the probe of a nanohardness testers without removing it from the working device.

Theoretical analysis and experimental studies of the magnitude of the errors associated with the operation of the device confirmed the possibility of measuring the surface of the probe with nanometer accuracy. Structural changes, such as the removal of the radiation source from the optical unit, the design of the triple prism holder and the compactization of the optical scheme, made it possible to reduce or completely eliminate the influence of such noise sources as thermal drift, non-orthogonality of the interferometer axes, and Abbe error. A detailed description of the optical scheme can be found in [6]

3. Measurement of probe tip parameters
The procedure for measuring the indenter geometry consists in scanning the probe with the known test structure (regular periodic lattice) in so many directions that the indenter does not reach its bottom during scanning.

For measurements on a platform with triple prisms mounted on a piezoceramic table, a tuning fork type piezoceramic probe is included, which is included in the oscillator excitation circuit. The offset of the measuring probe is carried out by a piezoelectric table. A rotary disk is used to rotate the probe around a vertical axis. To perform measurements on the probe, the necessary test structure is fixed, for example, lattices of the TGF, TGX, TGT, TGZ type.

Mathematical processing of scans allows you to determine the basic geometric characteristics of the tip, and to calculate the function of the dependence of the cross-sectional area and surface area on height. The geometrical characteristics of the tips of nanohardness testers include: a function of surface area, radius of curvature of the tip, angles at the apex, and defects in faces.

The function of the surface of the probe tip can be determined from the data obtained by scanning both the test structure TGT and TGZ. In this case, the surface functions of the tips were calculated based on the data obtained by scanning the probe tips with a TGT lattice using the triangulation method (approximating the surface with triangular plates). The use of TGT lattices for this procedure is explained by the independence of the result obtained from the orientation of the test lattice and the ability to use statistical averaging over the set of obtained images. In addition, according to the scanning data of TGT test structures, it is possible to characterize the structure of the tip by determining the presence of chips or other distortions in the shape of the cannula.

To measure the radius of curvature of the tip and the angles at the apex, test structures of the TGZ type were used. The use of TGZ structures for measuring these parameters is explained by the fact that only for this type of measures the shape of the elements of the lattice structure is precisely determined, and
for the calculation of these parameters accurate information about the angles between the pyramid face and the lattice face is needed.

Using the described measuring system, the area functions of four different indentors of nanohardness were determined (Figure 1). Deviations of the shape of the dependence of the surface area of the tip No. 3 and No. 4 from the theoretical one were revealed, indicating its wear. Small deviations in the form of indenters No. 2 and No. 1 from the theoretical dependence were also found (tip of the Berkovich pyramid), which is associated with the standard deviation during production and orientation with respect to the normal when installed in a nanoscale hardness tester (the reason why it is necessary to scan the indenter without removing it from the nanohardness testers).

![Graph of the surface area of the probe tips on the height](image)

**Figure 1.** Graph of the surface area of the probe tips on the height

4. Conclusion

The developed system for measuring the function of the probe tips of nanotrhdomeres based on a laser interferometer allows one to diagnose the indenter without removing it from the nanohardness tester and, accordingly, eliminate the error associated with a change in the angle of inclination of the tip during rearrangement, and also provides traceability of results to a unit of length. The implemented technique is operational and technically simple and can be used for periodic verification of the surface area function of indenters of nanohardnesses.

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