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Application of different methods for measuring modulus of elasticity of nanostructured materials on the example of titanium

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The article presents the results of a comparative study of various methods for determining the Young's modulus of nanostructured titanium obtained by equal-channel angular pressing followed by drawing. The approaches based on the Hertz model, the Oliver-Farr method of instrumental indentation, and dynamic mechanical analysis (DMA) are considered. It is shown that all methods allow to obtain the values of elastic modulus, which are in agreement with each other and close to the tabular data, while the differences are related to the phase of the loading process, which is taken into account in a particular model. The peculiarities of the spherical indenter application, which allows to restore the true stress-strain diagram, are analyzed. It is shown that at low loads the roughness of the indenter and the investigated surface, as well as the noise level, is the determining factor of accuracy. It is concluded that it is reasonable to measure Young's modulus by the method of instrumental indentation with subsequent use of this value in reconstructing the full stress-strain diagram from the data of indentation with a spherical tip.

Keywords: stress-strain diagram, modulus of elasticity, tool indentation, nanostructured titanium.

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Introduction

The assessment of the elastic characteristics of a material, primarily Young's modulus, plays a key role in engineering materials science, contact interaction mechanics, micro- and nanoelectronics, as well as in functional coating technologies. The elastic modulus determines not only the initial response of a material to external loads, but also is directly involved in calculations of durability, vibration resistance, and strength characteristics in structures of various scales.

Classical mechanical tests for uniaxial tension or compression are certainly the benchmark for constructing a stress-strain

diagram, as they allow obtaining a complete picture of the material's mechanical behavior from the elastic to the fracture stage. However, the implementation of such tests requires samples of a specific shape and size that comply with standards (e.g., GOST 1497–84, ASTM E8/E8M) [1], which practically excludes the possibility of directly testing objects with a characteristic size less than 1 mm, thin coatings, gradient structures, or individual components of microelectromechanical systems (MEMS/NEMS). For such objects, local mechanical probing methods, in particular instrumented indentation, prove to be the most suitable [2].

One of the urgent tasks in this area is not just extracting individual mechanical parameters (such as hardness or elastic modulus), but also reconstructing the complete stress-strain curve from indentation data. Today, there are a number of methods for transitioning from the indentation diagram to the tensile hardening curve. In particular, the study [3] shows that the suitability of some simplified analytical models describing the complex stress state of bodies of non-conforming shape during contact has been established. Another approach, implemented within the ABI method or in multi-step loading-unloading cycles, allows extrapolating macromechanical characteristics to the micro level. However, the reliability of such reconstructions depends on many factors: calibration of the contact area, accuracy of force and depth measurement, choice of the contact interaction model (Hertz, Oliver-Pharr, etc.), as well as the correctness of assumptions about the stress distribution in the vicinity of the imprint.

Comparison of diagrams reconstructed from indentation data with reference diagrams obtained from macro-tests represents a critically important step for validating the contact methods and models underlying the processing of experimental data. Without such verification, it is impossible to reliably assert the equivalence of the obtained mechanical characteristics, especially under conditions where the stress and strain gradients in the local region significantly exceed the corresponding values in macroscopic tests. Such systematic studies are particularly important from the perspective of further investigation of gradient structures, layered and topocomposite materials [4], characterized by anisotropy of mechanical properties.

Thus, the development and testing of methods for measuring Young's modulus based on spatially localized measurements represents an important direction in experimental solid mechanics.

Methods

A common method for reconstructing the stress-strain diagram from indentation results is the automated ball indentation (ABI) technique [5]. Within this approach, a series of sequential loading-unloading cycles is performed with an

increasing maximum applied normal force. The elastic and plastic characteristics of the material are determined from the reconstructed stress-strain diagram obtained by analyzing the accumulated indentation. A feature of this method is the possibility of directly reconstructing equivalent stresses and strains without prior knowledge of the cross-sectional area, which makes it particularly valuable for micro-volumes and inhomogeneous structures [6].

When transitioning to sub-micrometer indentation depths, correctly constructing the elastic portion of the stress-strain diagram using the ABI method becomes extremely difficult, because the measurement error becomes too large due to the small depths and applied loads. Therefore, to construct this portion, a strategy can be chosen in which Young's modulus, which characterizes the slope of the σ - ε diagram in the elastic deformation region, is determined by one of the alternative methods based on quasi-static or dynamic instrumented indentation.

Indentation with a spherical tip using the Hertz model

The first approach to estimating the elastic modulus is based on analyzing the loading curve when indenting with a spherical indenter. According to the classical Hertz theory, the contact between an elastic half-space and a rigid sphere is described by the analytical expression:

$$P = \frac{4}{3} E^* R^{\frac{1}{2}} h^{\frac{3}{2}},$$

where P is the load, h is the indentation depth, R is the radius of curvature of the indenter, and E^* is the reduced elastic modulus.

$$\frac{1}{E^*} = \frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2},$$

where E_1 and E_2 are the elastic moduli, and ν_1 and ν_2 are Poisson's ratios of the contacting bodies (indenter and specimen).

Approximating the experimental loading curves with the Hertz function allows determining E^* , and then, considering the elastic properties of the indenter material, Young's modulus of the material under study.

Instrumented indentation method

GOST R 8.748-2011 standard [7] regulates the procedure for determining the elastic modulus based on the analysis of the unloading segment during an indentation cycle. This uses the model proposed in the papers of Oliver and Pharr [8, 9], which relates the contact stiffness $S = dP/dh$ to the value of the reduced elastic modulus:

$$E^* = \frac{\sqrt{\pi}}{2\beta} \cdot \frac{S}{\sqrt{A}},$$

where A is the contact area, and β is a factor depending on the indenter geometry. After accounting for the indenter properties, Young's modulus of the material under study is calculated. For a spherical indenter geometry, $\beta = 1$.

Dynamic mechanical analysis (DMA)

The dynamic indentation mode is based on superimposing a harmonic excitation of small amplitude onto the main quasi-static indentation of the indenter into the material under study. The dynamic operating mode of the indenter allows recording the dependence of hardness, elastic modulus, and loss on the penetration depth in a single cycle mode.

The model of such interaction can be described in the first approximation as a system with forced harmonic oscillations, analogous to a damped oscillator. The external force signal has the form:

$$F(t) = F_{dc}(t) + F_{ac} \cdot \sin(\omega t),$$

where F_{dc} is the quasi-static load component, F_{ac} is the amplitude of the harmonic component, and ω is the angular excitation frequency.

The indenter displacement:

$$h(t) = h_{dc}(t) + h_{ac} \cdot \sin(\omega t + \delta),$$

where δ is the phase shift between excitation and response, determining the presence of a viscous material response.

Different materials can resist the applied load as elastic or viscous substances. The former store energy during deformation and return to their initial state upon load removal. The latter, which remain compressed after load removal, dissipate all the work of their deformation as heat.

When using the dynamic method, the deformation ε of the body depends on the frequency ω of the applied force as follows:

$$\varepsilon = \varepsilon_0 \sin(t\omega).$$

For a purely elastic body, Hooke's law holds, and the stress σ is proportional (with coefficient E – Young's modulus) to the strain:

$$\sigma = E\varepsilon.$$

Consequently, load and strain are in phase. In a viscous material, where there is no elastic deformation, the stress is given by Newton's law: $\sigma = \eta \dot{\varepsilon}$, and the stress arising in response to the load differs from it in phase by $\delta = 90^\circ$ [10]:

$$\sigma = \sigma_0 \sin(t\omega + \delta).$$

However, absolutely elastic and absolutely inelastic bodies do not exist in nature. The imaginary and real components of Young's modulus $E^* = E' + iE''$ have the form:

$$E' = \frac{\sigma_0}{\varepsilon_0} \cos \delta, \quad E'' = \frac{\sigma_0}{\varepsilon_0} \sin \delta.$$

From the analysis of the phase and amplitude of the response, the complex elastic modulus is measured, including the real part (storage modulus) E' and the imaginary part (loss modulus) E'' . In this case, the storage modulus E' approximately corresponds to Young's modulus for linearly elastic materials.

Experimental procedure

Commercially pure titanium Grade 4, subjected to severe plastic deformation by equal-channel angular pressing using the Conform scheme (ECAP-C) followed by drawing, was used as the material for the comparative study. This method enables obtaining an ultrafine-grained structure through the accumulation of large plastic strains without changing the external geometry of the sample. ECAP-C was carried out using the KV-048.IFPM.043911001 setup (Research Institute of Physics of Advanced Materials of the Ufa University of Science and Technology and Limited Liability Company NanoMeT):

deformation temperature – 250 °C, channel intersection angle – 120°, processing route Bc, number of passes – 4. For uniform deformation, the billet was rotated by 90° around its axis before each pass. Drawing of the billets after ECAP-C was carried out to a rod diameter of 6 mm over 17 passes with a die diameter change step of 0.5 mm. The drawing temperature was 250 °C, and the deformation rate was 0.3 m/s.

Based on the results of X-ray diffraction analysis using the broadened lines method (Williamson–Hall analysis) [12], the average equivalent grain size was determined to be about 41 ± 2 nm, which confirms the formation of a nanostructured state. The sample surface was mechanically polished, providing a roughness level $R_a < 5$ nm, sufficient for high-precision indentation tests.

Measurements were carried out on a NanoScan-4D nanoindenter [13]. A spheroconical diamond tip made from a single-crystal diamond was used as the indenter. The radius of the spherical part of the tip was 2.5 μm , and the height of the working part was 1 μm . The tip area function was obtained from data from an Ntegra Prima scanning probe microscope.

Measurements using the static indentation method were carried out at a rate of 50 nm/s until a maximum load of 30 mN was reached. DMA tests were carried out up to the same maximum load and at the same loading rate, with an oscillation frequency of 10 Hz and amplitude of 20 nm, respectively.

Discussion of the results

The table presents the averaged measurement results obtained by different methods. The instrumented indentation data were obtained with a maximum load starting from 1 mN (Fig. 1).

Table

Elastic modulus values

Measurement method	Young's modulus value, GPa
Hertz model	107 ± 5
Instrumented indentation	104 ± 4
Dynamic mechanical analysis	97 ± 3
Tabulated value [11]	110 ± 10

The key difference in the approaches based on the Hertz model and the Oliver-Pharr method is that the elastic properties of the surface are determined in different phases of loading. The Hertz model describes the elastic response of the initial, undeformed surface in a small local area of the contact spot under the indenter. The Oliver-Pharr model describes the elastic response of the surface contour upon unloading at the end of the indentation procedure.

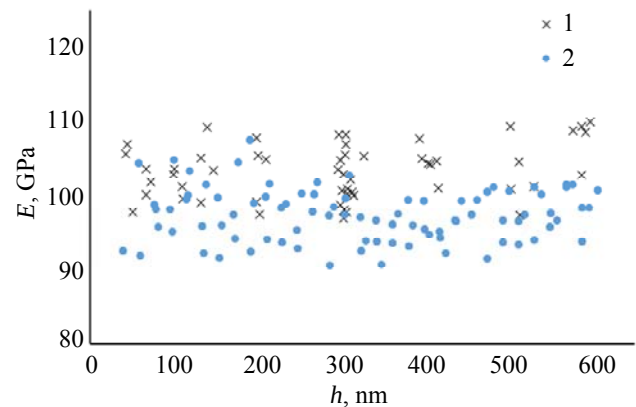


Fig. 1. Dependence of the elastic modulus on depth in instrumented indentation tests (1) and DMA (2)

To calculate the elastic modulus according to the Hertz formula, the initial section of the loading curve under static loading was approximated by a power function (Fig. 2). In this case, the load range in the elastic deformation region was less than 1 mN, which is consistent with the experimentally obtained dependence, as well as with the value calculated by the formula given in [5].

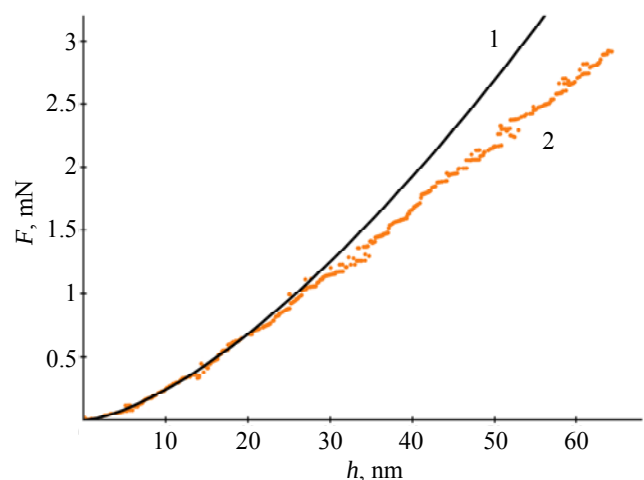


Fig. 2. Dependence of force on indentation depth: 1 – approximation of the elastic deformation section according to the Hertz model, 2 – experimental data

The Hertz theory was applied to describe the indentation of an ideal sphere into an elastic half-space. This model does not account for surface roughness. Moreover, the considered elastic deformation region is comparable to the roughness of the indenter. According to the Greenwood-Williamson theory for elastic deformation of micro-asperities (plasticity index $< 2/3$), the arithmetic mean surface roughness should be 1.1 nm. Exceeding this value by the actual surface roughness leads to an overestimated measured value and an increased measurement error.

The DMA method demonstrates lower elastic modulus values. During the DMA test, an oscillating load acts at a given loading rate. Therefore, the strain rate turns out to be higher than the rate of increase of the mean load and amounts to ~200 nm/s. To exclude the influence of strain rate for this material in this range of values, comparative tests were carried out using the instrumented indentation method with a maximum load of 15 mN and loading rates of 10, 50, 100, and 200 nm/s. No dependence of properties on loading rates was found in this range. An increase in the strain rate during dynamic tests of titanium leads to an increase in strength characteristics but does not affect the elastic modulus.

Conclusion

When constructing the stress-strain diagram in the elastic deformation region using a small-diameter spherical indenter, large errors arise related to the roughness of the indenter and the investigated surface, as well as to seismic noise.

In this study, various methods for determining the elastic modulus were investigated: using the Hertz model, the instrumented indentation method, and dynamic mechanical analysis. These methods demonstrate values that are close within the margin of error. To eliminate inaccuracies when constructing the σ - ϵ diagram in the elastic deformation region, it is proposed to use the value of the elastic modulus determined at large indentation depths in the region of plastic deformation of the material, for which instrumented indentation appears to be the most reliable method.

An advantage of using a spherical tip is the construction of the true stress diagram. Tensile test results of materials allow constructing an engineering stress diagram, which does not account for the change in cross-sectional area during tension.

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