Atomic force microscopy as applied to materials with anisotropic nanostructure

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Abstract

Creation of new nanostructured materials is impossible without serious study of their internal structure at the nanoscale level (when it is necessary to consider the effects associated with the features of the molecular structure of substance, even though the material can still be considered as a continuous medium). Atomic force microscopy (AFM) is one of the most promising tools for such studies. Its main advantage over traditional electron microscopy is that the atomic force microscope allows obtaining information not only on the topology of the internal structure of the material, but also on its local physical properties (which may differ significantly from the macroscopic characteristics).

Standard software supplied to decrypt the atomic force scanning (AFM), based mainly on the models using a classical solution of the Hertz contact of two linearly elastic spheres (or a sphere and a flat half, if one of them has infinite radius). In most cases this is enough. However, there are situations where the Hertz solution should be used with great caution. For example: 1) a very "soft" materials, when the AFM probe is pressed into the sample to a greater depth, 2) the sample surface has microasperities commensurate probe tip radius, 3) the test material is anisotropic at nanoscopic level. This work is devoted to the theoretical study of last version.

Modeling processes in contact AFM probe, the surface was considered as a brittle elastic medium crumbling in the interaction with the probe (tooth enamel). It is known that the tooth enamel is a complex of heterogeneous media consisting of parallel long and fragile prisms with characteristic transverse dimension of the order of 2 to 8 mm. Typically, these prisms are perpendicular to the work surface of the tooth and quite firmly connected to each other at the sides. Such a material must have anisotropic properties. Enamel can be regarded in terms of mechanics as a transversely isotropic body, in which the stiffness in direction perpendicular to the surface is different from tangential stiffness.

The problem of repeated indentation of AFM probe into the same place the damaged brittle elastic transversely isotropic material has been solved. Is a growing hole remained in the sample after each contact (as a result of brittle fracture of the material). That is, changing the geometry of the sample surface occurred without permanent plastic deformation. Repeated indentation into the same place allows you to collect the necessary information about its true mechanical properties and modeling should help in adequate explanation of the data. Power dependences of the response of the AFM probe on the depth of its indentation and degree of anisotropy of material were obtained as a result. These data will be used in experimental studies of the nanostructure of enamel at different stages of tooth decay.

Design and development of new nanostructured materials is impossible without serious research of their internal structure at nanoscale level (i.e. when it is necessary to consider the effects associated with the features of the molecular structure of matter, even though the material can still be regarded as a continuous medium).

As of today atomic force microscopy (AFM) is one of the most perspective tools for such studies. Its main advantage over traditional electron microscopy is that the atomic force microscope allows to receive information not only about the topology of the internal structure of material but also on its local physical properties (which may differ significantly from the macroscopic characteristics) [1, 2, 3, 4]. The atomic force microscope allows direct observation of micro-processes such as the appearance of dislocations, the occurrence a shear instability, phase transitions, and many other phenomena that are not available for the previously known techniques [5].

Cantilever as a console steel beam with a silicon probe at the free end is a core element of an atomic force microscope. Typically, this probe (indenter) has a conical shape with a rounded top.

AFM probe scans the selected surface of the sample during the experiment. Obtained thereby data are the relationship between the coordinates of the points of the scan, reaction force F acting on the probe, and the depth of the probe top indentation in the material under study u [6, 7]. These results in themselves (without additional knowledge of the subject of research) have little information. And the involvement of various physical and mechanical models required for their further theoretical interpretation.

Standard software supplied to decrypt the atomic force scanning (AFM), based mainly on the models using a classical solution of the Hertz contact of two linearly elastic spheres (or a sphere and a flat half, if one of them has infinite radius). In most cases this is enough. However, there are situations where the Hertz solution should be used with great caution. For example if the tested material is anisotropic at micro or nanoscopic level. This work is devoted to the theoretical study of last version.

Modeling elastic interaction of AFM probe with a flat anisotropic surface was carried out for this purpose. Probe was taken as a rigid cone with rounded top. Contact boundary value problem of the indentation probe solved numerically — finite element method was used. The solution was sought in the axisymmetric formulation, for both linear and nonlinear elastic medium (Neo-Hook). As a result dependence of the force of elastic response on the depth of the indentation u, mechanical properties of the sample material and geometric characteristics of the probe: nose radius R and the cone angle α were received.

Carried out by the authors comparison of the obtained numerical nonlinear elastic solution with the classical Hertz formula (case rigid sphere contact with a flat linear-elastic half-space) [8] showed that the discrepancy between them begins with $u/R > 0.4$ (with nonlinear elastic solution is below). At lower values it is quite possible to use Hertz formula [9].

In the simulation of processes occurring upon contact probe of an atomic force microscope with an anisotropic surface, was accepted that the sample — is destructible brittle elastic when interacting with the probe medium (tooth enamel). It is well known that the microstructure of tooth enamel is composed of parallel arranged long and brittle prisms with characteristic transverse size of about from 2 to 8 microns. Typically, these prisms are perpendicular to the work surface of the tooth and quite firmly connected to each other along the lateral sides [10]. This material should have anisotropic properties [11]. From the point of view of continuum mechanics tooth enamel can be considered as a transversely isotropic body, in which the rigidity in the direction perpendicular to the surface is different from the tangential stiffness.

Contact boundary problem of hard probe pressing in a transversely isotropic medium was solved by finite element method in the axisymmetric formulation in the frame of linear theory of elasticity. It was assumed that in the perpendicular direction to the surface of the elastic Young's modulus of z is E_z, and in a transverse plane — $E_r = E_\theta$.

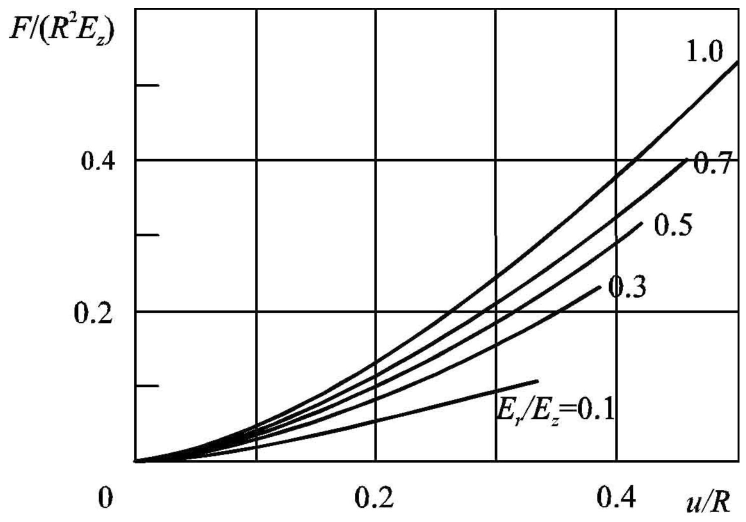

Figure 1: AFM probe indentation in an anisotropic intact surface ($\delta/R = 0$), $E_r/E_z = 0.1, 0.3, 0.5, 0.7, 1.0$. $\alpha = 30°, 40°, 60°, 90°$

Moreover E_r is always less or equal to E_z. Poisson's ratio was chosen to satisfy standard conditions for "engineering" constants, providing "physicality" of the solution of the problem for a transversely isotropic medium (non-negative elastic energy of an arbitrary method of loading) [12]. Coefficients ν_{ij} for the anisotropic case were taken equal $\nu_{rz} = \nu_{z\theta} = 0.03$, $\nu_{r\theta} = 0.3$ (at isotropy $\nu_{ij} = \nu_s = 0.3$).

The problem of repeated pressing of the AFM probe in the same place elastic brittle damaged material (isotropic and transversely isotropic) was solved by means this approach. Ever increasing hole depth δ remained in the sample after each contact. Hole was a result of brittle fracture of the material under the AFM probe action, i.e., changing the geometry of the sample surface occurs, but there is no residual plastic deformation. Such processes are typical at AFM studies of tooth enamel, which can have different mechanical properties in thickness (on the surface, there may be various damage, it can be covered with a thin coating of another material, etc.). Repeated indentation in the same place the tooth surface can gather the necessary information about its true mechanical properties and theoretical modeling should help in adequate decoding of the data.

Model studies on indentation differing in geometry of AFM probes in transversely isotropic material with varying degrees of anisotropy were conducted. Ratio E_r/E_z ranged from 0.1 to 1 (the isotropic case, the curve coincides with the solution of Hertz). Cone angle of the indenter α taken to be 30°, 40°, 60° and 90°. Force dependences to the first indentation AFM probe in transversely isotropic material (still intact) are shown in Fig. 1. Since the depth of the indentation is relatively small — less than or close to the height of the spherical part of the probe — the parameter α has almost no effect on the effort. The larger the anisotropy of the material, the less reaction indentation probe.

Similar relations for the case when the indenter AFM already "punched" hole depth R and $2R$ are shown in Fig. 2 and 3, respectively. Dependence on only to the two extreme values of the cone angle α — be 30° and be 90° shown. It is clear from the graph that for large cone angles of the probe depth of the well, done by the previous contacts, much greater effect on the reaction force F, than for small values of α.

Calculated dependences of F on u and α for the limit values E_r/E_z equal to 0.1 and 1.0, are shown in Fig. 4 and Fig. 5. The depth of wells is R and $2R$, respectively. The graphs show that the greater the depth, the greater the effect of material anisotropy on

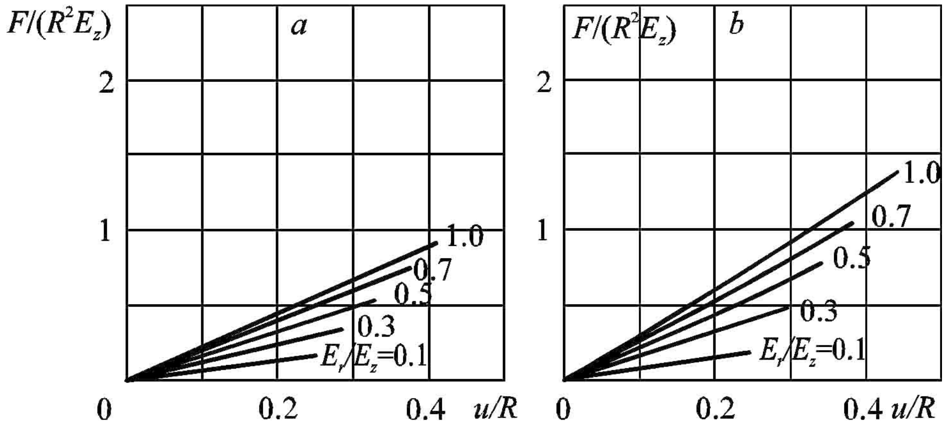

Figure 2: AFM probe indentation in an anisotropic surface with the hole ($\delta/R = 1$), $E_r/E_z = 0.1, 0.3, 0.5, 0.7, 1.0$. $\alpha = 30°$ (a), $\alpha = 90°$ (b)

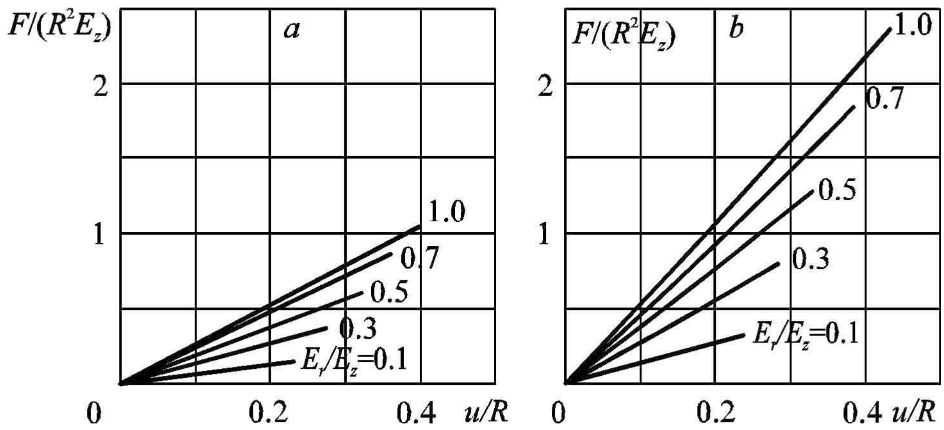

Figure 3: AFM probe indentation in an anisotropic surface with the hole ($\delta/R = 2$), $E_r/E_z = 0.1, 0.3, 0.5, 0.7, 1.0$. $\alpha = 30°$ (a), $\alpha = 90°$ (b)

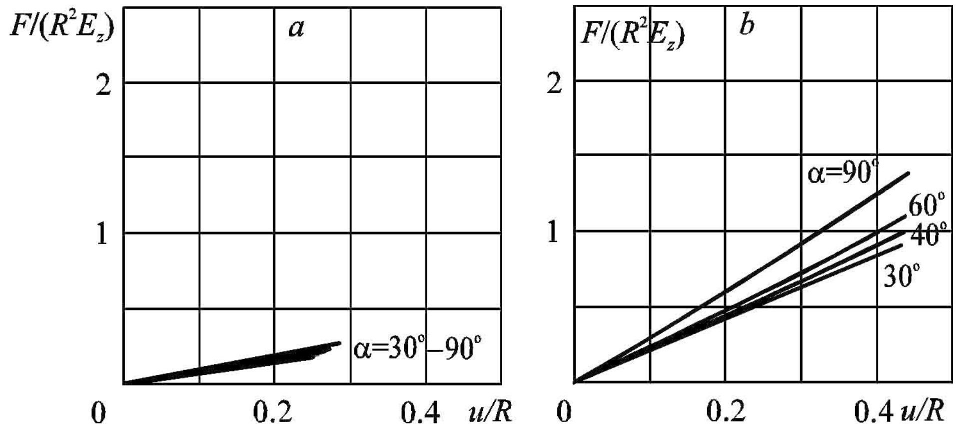

Figure 4: AFM probe indentation in an anisotropic surface with the hole ($\delta/R = 1$), $\alpha = 30°, 40°, 60°, 90°$. $E_r/E_z = 0.1$ (a), $E_r/E_z = 1.0$ (b)

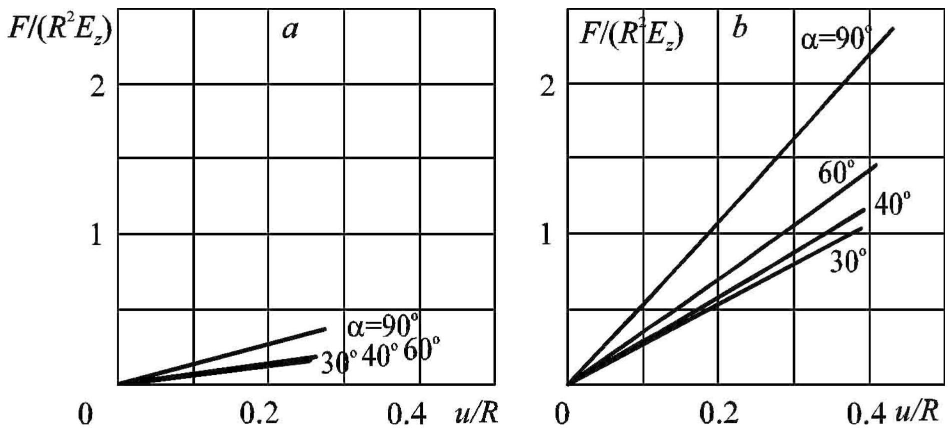

Figure 5: AFM probe indentation in an anisotropic surface with the hole ($\delta/R = 2$), $\alpha = 30°, 40°, 60°, 90°$. $E_r/E_z = 0.1$ (a), $E_r/E_z = 1.0$ (b)

the force of reaction F.

Based on the data, the conclusion should be that anisotropy of the surface can very significantly affect the reaction force F. In this case, the correct interpretation of adequate obtained by AFM scan data should be very careful to use the standard models based on the solution of Hertz.

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