Development of methods for mechanical properties measurements using the scanning probe microscope-nanohadrness tester NanoScan

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Introduction

People have been investigating the elastic properties of materials for the last several centuries. The famous experiment of Robert Hook dates back to 1678. He strained the uniaxial isotropic bars to find out the dependency between the load applied and the length of the bar. A lot of time has passed since then. The recent development of electronic equipment allows us to investigate the elastic properties on a nanometric scale.

The NanoScan measuring system is represents a scanning force microscope of original construction. The major distinction of the SFM NanoScan is the use of piezoresonant probe embedded in the self-oscillating schema. The main features and operating modes of the NanoScan system are described in details in the review [1]. The SFM NanoScan makes it possible to obtain the picture of the surface relief with extra resolution and the image of mechanical properties redistribution map simultaneously with relief scanning. In addition, the NanoScan system allows performing micro- and nanoindentation and sclerometry of the samples with subsequent surface scanning in the indentation area, measuring materials hardness on a submicron scale and investigating their breakdown nature.

During the last years, the SFM NanoScan has been successfully used in investigation of mechanical properties of hard alloys, thin films [2] and superhard materials [3].

Almost since the birth of SFM, the measuring mode has been widely used in which the tip is pressed into the sample surface with simultaneous recording of the one of probe parameters. Generally, this parameter is selected to be the pressing force value and the recorded dependency is called force-distance curve. In general, force-distance curves are used for investigation and measuring of different forces that arise during the interaction of the tip and the sample surface. The meniscus forces, Coulomb, Van der Vaals, adhesion, solvation, hydration, hydrophobic, steric, depletion forces represent only a small part of the entire forces variety. All these experiments deal with the quite soft materials. Measuring the elastic properties of hard and superhard materials using the standard force curves is an intractable or even impossible task. The common obstruction is that increase in the load hampers extraction of the effective signal and leads to deformation of the tip [4]. All of this makes it impossible to obtain the information on the properties of investigated material.

Measuring of elastic properties with the NanoScan system

Hardware. The investigation using loading curves is implemented in the SFM NanoScan. The probe operation mode in which surface is scanned and curves are measured is to a certain extent similar to the Tapping Mode. The difference is that in this case the resonant frequency and amplitude of probe oscillations are measured. The recorded curves represent the dependencies of the amplitude and frequency from the displacement of the tip. That is why we call our dependency the “approach curves”.

For measuring the approach curves we used the cantilever with the high spring constant (about $10^4 - 10^5$ N/m). This allowed us to reduce the unwanted effects and obtain high loads. Besides, we could avoid considering the surface and adhesion forces due to their small contribution to the tip-sample interaction.

The tip is made in the form of triangular diamond pyramid with a vertex angle of about 60 degrees. The tip radius is no more than 100 nm. The use of the diamond tip allows not to take into account the deformation of the tip itself. Together with very a stiff cantilever it gives the possibility to apply the Herz model to calculate the tip-sample interaction.

The curves were measured in the open air, with no special sample preparation.

Theory. The oscillating tip is moved down to the sample surface. When it begins to interact with the surface, the resonant frequency and the amplitude of oscillation change. To describe the tip-sample interaction we can use the standard Herz approximation [5]. The tip can be treated as a hemisphere with the radius R and the surface can be assumed flat and absolutely elastic. In addition, we assume the Young modulus of the tip to be much greater than that of the tested materials. The probe frequency shift during tip-sample interaction can be described [6] as:

$$f - f_0 = \frac{f_0}{k_0}\cdot\sqrt{R}\cdot\frac{E}{(1-\nu^2)}\cdot\sqrt{h} \qquad (1)$$

Here k_0 is the elastic coefficient of the oscillating system (cantilever), E and ν are the Young modulus and Poisson ratio of the sample, R is the tip radius, and h is the displacement of the cantilever. Hence, if we draw the line that represents the frequency shift square dependency from the displacement, the angle between this line and an abscissa axis will be proportional to the coefficient $\frac{f_0^2}{k_0^2}\cdot R\cdot\left(\frac{E}{(1-\nu^2)}\right)^2$. One can see that this coefficient depends on the cantilever and tip parameters and also on the elastic properties of the sample. Therefore, if the cantilever and the tip were calibrated using a sample with the well-known properties, then the difference in the line slope can be correlated with the values of materials elastic properties.

Results

The common shape of the approach curves recorded using the SFM NanoScan is indicated on the fig. 1. The curve can be divided into four sections. The first section corresponds to the tip oscillation out of the contact with the sample, so the amplitude and the frequency of its oscillation don't change with the distance. At the beginning of the second section the tip gets into contact with the viscous contamination layer adsorbed on the surface in the open air. In some cases the line in the second section can have some deflections. This can be due to the surface undulations or other artifacts. The third section corresponds to the elastic interaction between the tip and the sample surface atoms. This section allows to determine the elastic properties of the material. The fourth section shows the plastic deformation of the sample. This section can be eliminated at all if we stop further loading before the beginning of plastic deformation (fig. 2).

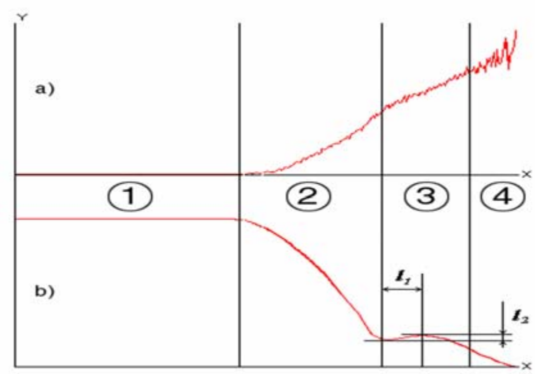


Fig. 1 The common shape of the approach curve; a) the frequency curve b) the amplitude curve

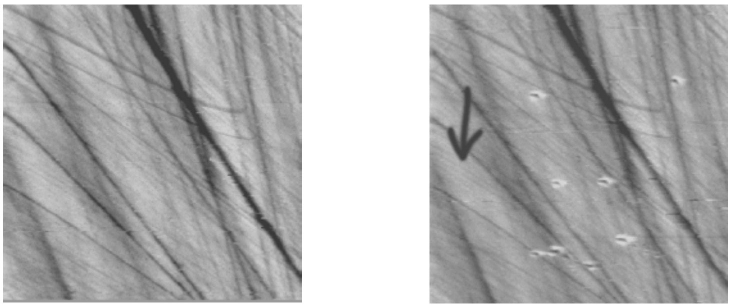
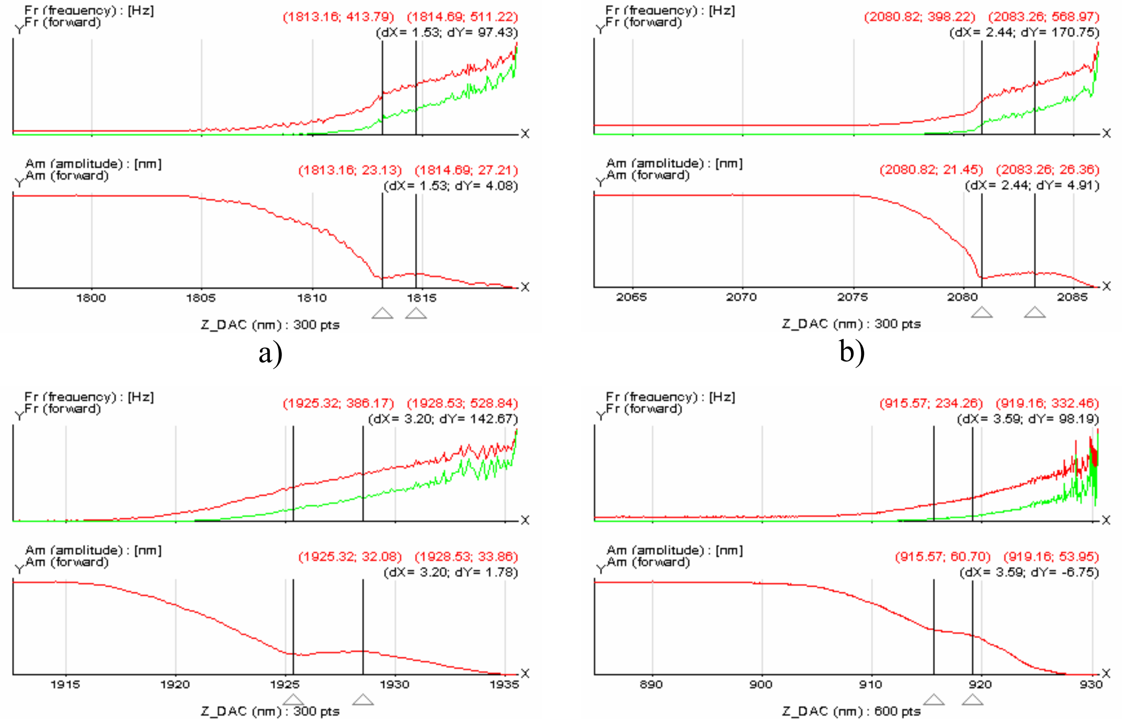


Fig. 2 The zirconium oxide surface.
Left – before, right – after curve measuring. The right image shows the marks at the points of tip interaction. The arrow indicates the place with no mark after curve recording

During experiment the curves were measured for four different materials: garnet, zirconium oxide, ruby and quartz glass (see fig. 3). As figure 3 shows, all curves have the same shape. The distinctive features for every material are the distances l_1, l_2 (fig. 1) and also the slope of the line $\left[f(Z)\right]^2$ in the third section.



a) b)

c) d)

Fig. 3 The approach curves: a) garnet b) zirconium oxide c) ruby d) quartz glass. The lower lines on the frequency curves are the square of the frequency shift.

The question of samples preparation remains open. We didn't apply any special preparation, so the curves selection for the rough surfaces is quite a painstaking job. In many cases the tip hits the hills or holes on the surface and the resulting curves give the erroneous picture. For final analysis we accepted only that curves which measured in the areas with roughness of no more than 1 nm.

Conclusions

The results obtained using the SFM NanoScan show the concise dependency between the elastic properties of materials and the parameters of approach curves measured. The curves with larger slope in the working section 3 correspond to the materials with larger value of elastic modulus. The values of l_1, l_2 distances (fig. 1) also can provide information about the mechanical properties of investigated materials. Further experiments in this direction will cast light upon these and many other questions. The final goal of such experiments is to develop the complete method of measuring the elastic properties of the materials using the approach curves.

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