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ESTIMATION OF YOUNG'S MODULUS AND ADHESIVE FORCE OF POLYMERIC FILMS BY USE OF ATOMIC FORCE MICROSCOPE

Nowadays a big interest exists in microengineering forces to study and test microsystems and their components. There exist a lot of methods and tools to perform this kind of investigations. Most of them need good skills, a lot of time and expensive equipment.

We would like to present a simple and quick method to determine such material properties as Young's modulus and adhesive (pull-off) force of thin films applied on e.g. working surfaces of microgrippers.

Keywords: Atomic force microscope, Young's modulus, adhesive (pull-off) force, polymeric films, microhandling, microgripper.

1. INTRODUCTION

A key point of the engineering development during the last decades is miniaturization of the objects of manufacture and it will be of even more importance in future. From this point of view new instruments and methods are needed to analyze and study microstructures [1, 2].

For a long time such instruments as the scanning electron microscope (SEM), scanning tunneling microscope (STM), atomic force microscope (AFM) and etc. are used [3, 4].

In this paper we would like to present some methods that allow us to receive easily and quickly information about such material properties as Young's modulus and adhesive (pull-off) force in the case of thin polymeric films by the use of results of AFM experiments.

The purpose of the experiments was also to determine the influence of environment conditions (temperature) on the material properties of polymeric films useful to be deposited on working surfaces of microgrippers used in manipulations and in an microassembly process [5, 6].

2. MEASUREMENT METHOD

2.1. Atomic force microscopy

The atomic force microscope (AFM) is one from the number of scan probe microscopes [7, 8]. The principle of operation of such microscopes is based on measuring the tunneling current (STM) or e.g. atomic force interactions (AFM). In difference with traditional ones these microscopes do not use lenses. The idea of measurement on an AFM is to use the cantilever, the rectangular or triangular flat spring, which is fixed on one side and has a silicon or diamond tip with a height of 10 - 20 μm and a radius of about 10 nm on the other side.

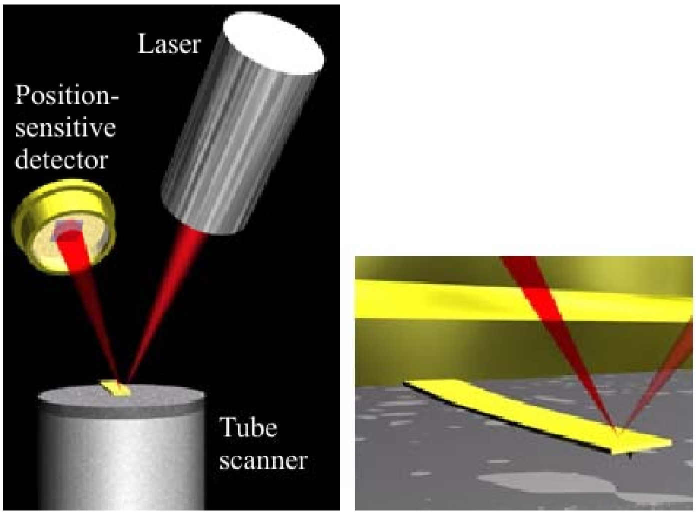

Fig. 1. Concepts of AFM work. Left: schematics of AFM. Right: AFM cantilever reflecting a laser beam. [9].

An AFM can work in two different modes: contact and non-contact. In the contact mode the instrument makes a cantilever lightly "touch" the sample by the tip. In this case the vertical deflection of the cantilever can be measured by a position-sensitive detector which shows the local height of the sample. In the non-contact mode the tip does not touch the sample and determines the surface topography through measurement of the attractive forces between the tip and the sample.

Another possibility to use the AFM is to make single indentations. In this case the cantilever's tip makes a single penetration into the sample. The information about the displacement of the tip is collected by an optoelectronic, position-sensitive detector. As a result of this experiment a force-distance curve is obtained.

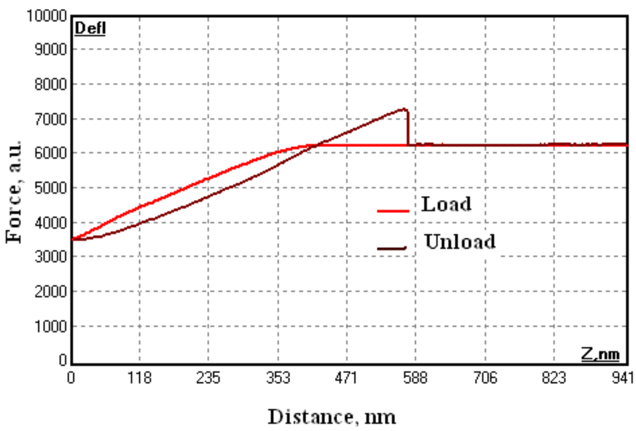

Fig. 2. Force-distance curve as a result of the indentation test.

From the results of indentation tests such sample's mechanical properties as Young's modulus and adhesive properties (pull-off force) can be determined.

2.2. Estimation of Young's modulus

For estimation of the elasticity (Young's) modulus the part of the force-distance curve that is indicated in Fig. 3 is needed. This part of the graph describes the process of penetration of the tip into the sample material.

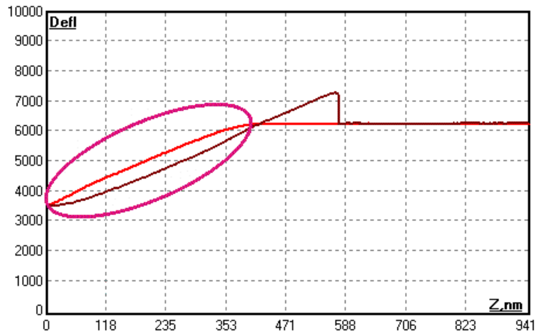

Fig. 3. Part of the force-distance curve which is used for the determination of Young's modulus.

As the result of an AFM indentation test the deflections of the cantilever are given in arbitrary units (a. u.). To use in calculations these data should be calibrated. In our work we use the following calibrating procedure. We suppose that during the indentation of a stiff sample, deformation of the material does not occur and the deflection of the cantilever will be equal to the displacement of the AFM scanner in normal *Z* direction.

The calibration coefficient *C* can be calculated as follows:

$$C = Z'/Defl', \tag{1}$$

where *Z*' - displacement (in nm) of the scanner, *Defl*' - deflection (in a. u.) of the cantilever in the case of a stiff sample.

To calculate Young's modulus we use the dependence given by Hertz theory [9]

$$E = 3/4(1-\nu^2)*P/(R^{1/2}\delta^{3/2}), \tag{2}$$

where ν - Poisson ratio, *P*- applied force, *R*- tip radius, δ - penetration into material.
P can be determined as

$$P = k*Zdef, \tag{3}$$

where k- stiffness of the cantilever, *Zdef* = *C** *Defl*, *Defl* – deflection of the cantiever.

Penetration δ can be found basing on the geometry of the contact, in our case *Zpoz* = *Z*.

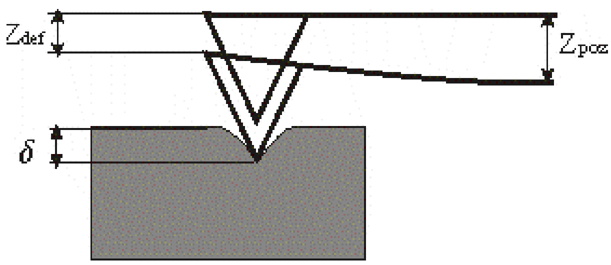

Fig. 4. Geometrical model of penetration.

$$\delta = Zpoz - Zdef. \tag{4}$$

The resulting equation is taking the form

$$E = 3/4(1-\nu^2)*k*Zdef / (R^{1/}2(Z - Zdef)^{3/2}). \tag{5}$$

For the force-distance curve shown in Fig. 1, the dependence of Young's modulus on the penetration depth δ is presented in Fig. 5.

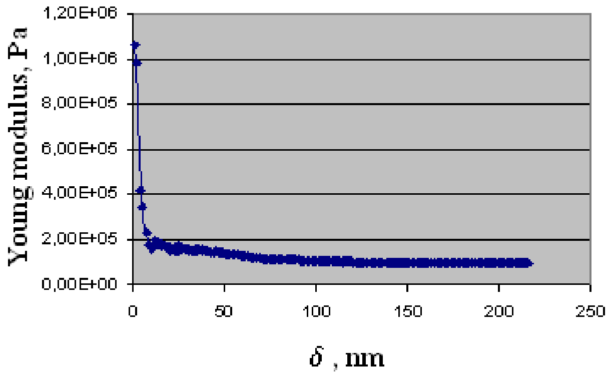

Fig. 5. Dependence of Young's modulus on penetration depth.

2.3. Estimation of pull-off force

For estimation of the pull- off force the part of the force-distance curve that is illustrated in Fig. 6 is needed.

To find the value of the pull-off force we need to find the height Δ of the peak on the calibrated force- distance curve during the unloading process.

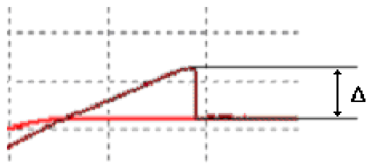

Fig.6. Geometrical determination of height of the peak on the force-distance curve.

The value of the pull-off force can be found as follows:

$$F = k * \Delta, \qquad (6)$$

where k - stiffness of the cantilever.

3. SOME PROBLEMS OF MICROHANDLING

From the late 1990s, researchers have been actively developing micro-manipulation systems .In spite of the research in this area, most of the micrograppers developed up to now cannot perform such fundamental function as a stable release. This lack of success can be attributed to the fact that most grippers have been designed in the same fashion as grippers for macro-objects, despite the large differences between the behaviors of micro- and macro-objects. Fig. 8 illustrates some of the effects which can be seen when attempting to manipulate micro parts. As the gripper approaches the part, electrostatic attraction may cause the part to jump off the surface into the gripper, with an orientation dependent on initial charge distributions. When the part is placed to a desired location, it may adhere better to the gripper than to the substrate

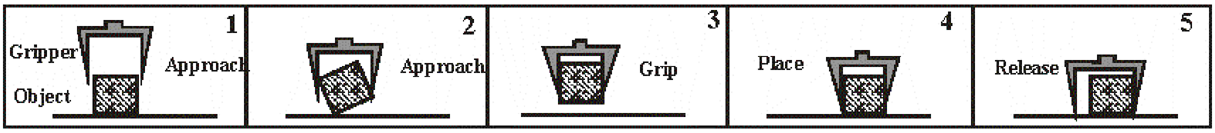

Fig. 7. Typical pick-and-place assembly in microscale. 1). A gripper approaches to a component. 2). The component sticks to the gripper. 3). The gripper grasps the component. 4). The component is transported to a desired location. 5). The component is placed at inaccurate position, component sticks to the gripper.

For smaller objects, sticking effects become dominant. Actually, compared to the adhesion and electrostatic forces, the weight of the particle can be neglected (Fig. 9).

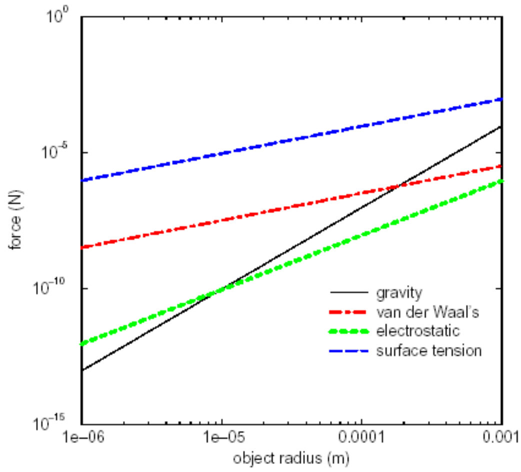

Fig. 8. The size dependence of the gravitational, electrostatic, Van- der-Waals, and surface tension forces, which appear during the contact of the sphere and plane [11].

As a consequence, the main problem in handling microscopic objects is no more the gripping aspect (most of the time the particles stick without any additional effort) but it is rather the controlled release phase that is not solved yet.

In our work we offer to solve the sticking problem by coating the microgripper with a solution of a polymer that will decrease the influence of the surface forces.

4. EXPERIMENT DETAILS

The purpose of our experiments was to obtain the Young's modulus and value of the pull-off force of eight different polymeric films. Another goal was to examine the influence of environment conditions on micromechanical properties of these coatings.

Samples of polymers were prepared by dipping a clean silicon (100) wafer in the following silicone resin solutions:

Sample 1 and 2: - methylsilicone resin solution in aliphatic and aromatic hydrocarbons cured thermally at 180 C deg. during 2 hours.

Sample 1' and 2': - methylsilicone resin solution in aliphatic and aromatic hydrocarbons cured with a catalyst improving the adhesion to the substrate.

Sample 3 and 4: - methylphenylsilicone resin solution in aliphatic and aromatic hydrocarbons cured thermally at 180 C deg. during 4 hours.

Sample 3' and 4': - methylphenylsilicone resin solution in aliphatic and aromatic hydrocarbons cured with a catalyst improving the adhesion to the substrate.

The polymeric films were tested on an AFM NT-206 by "Microtestmachines", Belarus in controlled environment conditions (clean-room, temperature: 21°C, relative humidity: 50%). In the experiments two different types of cantilevers were used: a standard silicon cantilever by “MicroMasch” and a special cantilever (Fig. 9). This cantilever was made from beryllium bronze; the upper side was polished very precisely and covered with 300 nm of gold. The length of this special cantilever was 6 mm, width 1 mm, thickness 50 µm. On the bottom side of this cantilever a steel ball with 0.7 mm diameter was glued. The experiments with this indenter were made at different temperatures of the sample: 20, 30, 40°C.

Young’s modulus and pull-off force were calculated using that techniques.

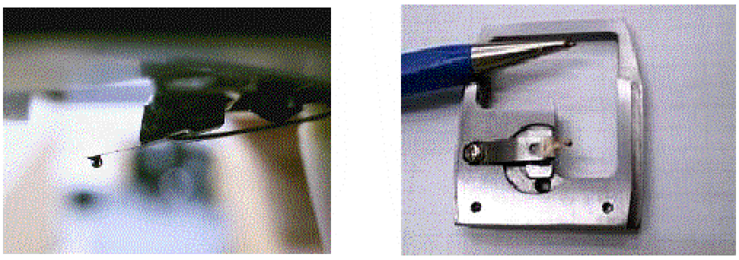

Fig. 9. Special cantilever for indentations in the holder.

5. RESULTS AND CONCLUSIONS

One of the finds of this work was the procedure of preparation of the samples. This technique was successfully used to coat a microgripper (Fig. 10). This method seems to be very quick, easy and reliable.

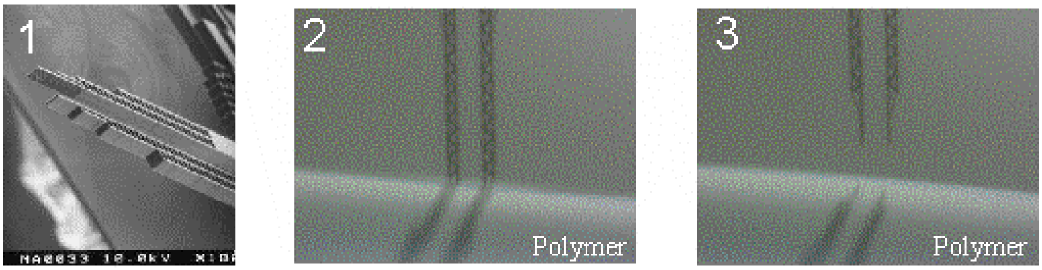

Fig. 10. Piezoelectric microgropper by Nascatec company (Germany).
1). Clean microgripper. 2). Microgripper dipped into the polymer.3). Microgripper coated with the polymer.

The results of indentation tests are presented in Table 1.

Table 1. Results of indentation experiments.

Sample	Young modulus	Pull-off force			
	AFM, Pa	AFM, nN	Special indenter, au		
	20^0 C	20^0 C	20^0 C	30^0 C	40^0 C
1	10.3 E 6	13.03	879.8	2048	2334.6
1’	20.4 E 6	11.06	1543.3	1960	1809
2	26.7 E 6	11.03	185	209	1769
2’	27 E 6	32.15	1512	1186	742
3	57 E 6	36.6	1454	----	----
3’	78 E 6	13.07	1484	2310	2821.3
4	40 E 6	43.67	991.2	----	----
4’	39.6 E 6	7.65	758	1183	1967.8
Silicon		50.8	540		

According to results of indentation using a standard cantilever, the value of the pull-off force of a polymeric film is at least several times smaller than in the case of silicon.

The pull-off force was found to be several times greater in the case of tests with a special cantilever as compared with the use of a standard silicon cantilever. It can be explained by several factors. The materials of the cantilever's tip are different and adhesion of steel to this coating is greater. The dimension of the contact area is much bigger in the case of the special indenter and this fact can cause the insignificant growth of the influence of capillarity forces. We can say that in the case of tests with a standard cantilever the local properties were measured but with the use of a special cantilever the deformed area is greater and the local properties of the material were averaged.

In difference with other samples, the value of the pull-off force in the case of Sample 2 was decreasing when the special cantilever was used. This polymer solution might improve work of the microgrippers and facilitate the release process.

One of the purposes of these experimental investigations was to determine the influence of topography and temperature on the value of the pull-off force. An increase of temperature in the case of most of polymeric films caused an increase of the pull-off force.

In the case of sample 2' the pull-off force was decreasing. This effect can be used in microgrippers with the possibility of variation of temperature of the working surface for better control of the picking up - releasing process.

We can say that the pull-off force is the value that depends on a lot of different parameters and by proper choice of topography and material properties of the surface this value can be minimized.

It is interesting to apply the tested films e.g. on the working surface of microgrippers.

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WYZNACZANIE MODUŁU YOUNGA I SIŁY ADHEZJI WARSTW POLIMEROWYCH PRZY UŻYCIU MIKROSKOPU SIŁ ATOMOWYCH

Streszczenie

W pracy opisano metodę wyznaczania modułu Younga i siły adhezji warstw polimerowych przy użyciu mikroskopu sil atomowych (AFM). Wartości te oblicza się korzystając z krzywych siła - przemieszczenie wyznaczonych przy użyciu AFM. Przedstawiono wyniki badań ośmiu warstw polimerowych silikonowych nałożonych na podłoże krzemowe. Zbadano także wpływ temperatury na moduł Younga i siłę adhezji. Z badań wynika, że siła adhezji zależy od materiału polimerowego i warunków otoczenia. Umożliwia to zmienianie tej siły np. w procesach chwytania i uwalniania mikroelementów przy użyciu mikrochwytaków stosowanych w mikrotechnice do manipulacji lub montażu. Badane warstwy polimerowe mogą być interesujące w zastosowaniu na powierzchnie robocze takich mikrochwytaków.