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NANOTRIBOLOGICAL INVESTIGATIONS ON ADESION EFFECT APPLIED TO MEMS MATERIALS

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Abstract: *The roughness effect on adhesion of different MEMS materials is investigated and presented in this paper. Asperities of rough surfaces can either reduce or increase the contact area of interacting MEMS components. As a consequence, roughness of substrates cannot be eliminated from tribological behavior of MEMS materials, and this surface characteristic must be taken into consideration in the interpretation of stiction and friction. Stiction is one of the main failure causes of microsystems with compliant structural members. It is influenced by the material properties, surface characteristics and environmental conditions. Experimental investigations are performed using the spectroscopy in point mode of an atomic force microscope (AFM) in order to measure the adhesion force between AFM probes and MEMS materials with different roughness.*

Key words: *MEMS materials, roughness, interaction forces, stiction, atomic force microscope.*

1. INTRODUCTION

Tribological behavior of MEMS materials depends on the topography of the two contacting surfaces. Based on the surface topographies importance to many fields besides tribology, a wide variety of techniques have been developed over the years for topography characterization including contact mode (profilometry, scanning probe microscopy, atomic force microscopy) or non-contact mode (optical interference, optical scattering, scanning electron microscopy).

Microtribology and nanotribology are the techniques to characterize surface topography, adhesion, friction, wear on a micro/nano-meter scale. The components used in micro/nano-structures are very light, on the order of a few micro/nano-grams, and operate under very low loads, of a few micro/nano-newton. As a result, friction on nanoscale is highly dependent on the surface interactions as work of adhesion or adhesion force. Work of adhesion is the energy per unit area required to separate two flat surfaces in vacuum from contact to infinity.

Adhesion is the maximum force needed to separate two samples. A well known technique used to measure adhesion forces is atomic force microscope (AFM). In the AFM measurements the maximum negative force upon separation of the tip and sample is the adhesion force, often referred to the pull-off force. Measuring the pull-off force does not directly give the work of adhesion. To measure the surface topography with very high resolution and relatively small area (<100 μ m across) an AFM can be used.

The technique of atomic force microscopy (AFM) was invented by Binnig *et al.* in 1986 [1, 4]. The operational principle and setup of AFM is presented in figure 1. Briefly, a cantilever is used as a force sensor to detect force between tip and sample surface. The cantilever is fixed at one end and free at the other end where there is a tip, gently contacting the sample surface. A laser and a detector are used, forming an optical beam deflection system to detect the bending and/or rotational deflections of the cantilever. When the sample is scanned the cantilever moves up and down in the vertical direction, or left and right in the

lateral direction to the surface. Commercial AFM cantilevers are typically made of silicon or silicon nitride with a tip radius or curvature on the order of nanometers.

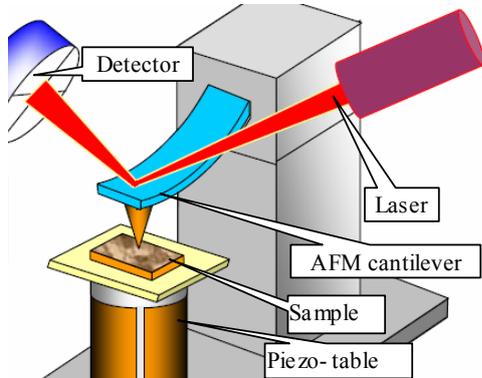


Fig. 1. Operational principle of the atomic force microscope used in experiments.

During experimental tests, vertical and lateral deflection signals detected by photodetector are proportional to the deflection of the cantilever. The photodetector sensitivity (eV) needs to be calibrated in order to measure accurately the nano-deflection (nm) of AFM cantilever [4]. For calibration, the AFM tip is pushed on a smooth and hard sample (made of diamond or silicon) by moving the piezo-table in vertical direction over a known distance. The deflections of AFM cantilevers (eV) can be assumed to be the same as the displacement of piezo-table (nm). The photodetector sensitivity for cantilever setup is determined as the ratio between deflections of AFM cantilevers and displacement of piezo-table (eV/nm). During measurements, when the AFM tip interacts with investigated materials, the monitored deflections of AFM cantilever (eV) is divided with the calculated photodetector sensitivity (eV/nm) resulting the deflection of AFM cantilever in nanometers. This deflection is used to determine the adhesion forces between the AFM tip and investigated MEMS materials.

2. THEORETICAL APPROACH OF SURFACES INTERACTION

When the surface to volume ratios of structure become large, adhesion is important. The adhesion force is the source of stiction. Adhesion forces arise from van der Waals

forces, and capillary forces acting between two contacting surfaces. Adhesion forces depend on the surface characteristics and the affinity to water of the interacting surfaces.

In AFM pull-off force measurements, continuum contact mechanism model are commonly used to describe the interaction between the AFM probe and substrate. In this model a spherical tip end with radius R that interacts with sample is assumed. This interaction is proportional with the tip radius.

Meniscus interaction force is given by the relative humidity which arises from capillary condensation of water vapor from environment. For adhesion in air due to liquid, meniscus force can be computed as [1]

$$F_{meniscus} = 4\pi\gamma R \cos \varphi \quad (1)$$

where R is the tip radius, γ is the surface energy (for water $4\pi\gamma \approx 0.88\text{N/m}$) and φ is the contact angle of the meniscus with the AFM tip and sample.

For van der Waals interaction the force is described by

$$F_{vdW} = \frac{6AR}{z} \quad (2)$$

where A is the Hamaker constant and z the tip-sample distance.

Two contact mechanisms models for adhesion developed by Johnson et al (1971) and Derjaguin et al (1975) is JKR model and DMT model. These models are frequently used by researchers to interpret the pull-off forces measured by AFM [2, 5]. The difference between JKR and DMT models occurs in assuming the nature of forces acting between AFM tip and substrate. JKR model assumed attractive forces act only inside the tip-substrate contact area, whereas DMT model include long-range surface force operating outside tip-substrate contact area. In the JKR and DMT models, the adhesion force (pull-off force) is related to the work of adhesion as [2, 5]

$$F_{adhesion} = c\pi\gamma R \quad (3)$$

where γ is the work of adhesion (J/m^2), R is the radius of the AFM tip and c is a constant equal by 2 in DMT model and equal by 1,5 in JKR model. The JKR and DMT models were derived based on the Hertz theory (from 1896).

If rough substrate is considered, the pull-off force is changed as the result of change in the tip-substrate contact area. Equation (3) corresponds to simple contact mechanics model but it can be applied to samples with rough surfaces if a single contact point between the AFM tip and substrate is established. For such situation, equation (3) is modified as

$$F_{adhesion} = c\pi\gamma \frac{R_1 R_2}{R_1 + R_2} \quad (4)$$

where R_1 and R_2 are the radii of AFM tip and asperity in contact. This equation is valid for asperities that can be treated as spherical in their upper section.

The tip of AFM can be coated with different materials of interest in order to estimate the adhesion effect between different contacting surfaces. The adhesion force in general decreases with increasing surface roughness because the true contact area of interaction between the surfaces decreases [3].

Adhesion force affects friction at micro/nano scale. As the size of component decreases to micrometers and nanometers, surface - related properties like roughness, adhesion, capillarity and electrostatic forces play a major role in affecting the friction force, unlike in macroscale components. Adhesion force acts as an additional force to the applied external force and thereby results in higher friction. The following relationship exists at micro/nano-scale for friction force [1, 4]:

$$F_f = \mu(F_{applied} + F_{adhesion}) \quad (5)$$

where μ is the friction coefficient, $F_{applied}$ is the normal force and $F_{adhesion}$ is the adhesion force.

In the case of MEMS components that operate at low pressure, the roughness greatly affects adhesion and therefore friction. If the contact area decreases, the interaction zone between surfaces becomes small and the adhesion reduces.

3. EXPERIMENTAL INVESTIGATIONS

The materials investigated in our work are thin solid films of gold (Au) and aluminum (Al) with thickness of 300 nm which are deposited one-layer on a glass as substrate. Gold and aluminum as MEMS materials are often used in optical applications as a reflective coating thin layer deposited on the top of a basic structure. Mechanical and tribological properties of thin films often differ from those of bulk materials. This can be partially explained by the nanostructures of thin films and the fact that these films are attached to a substrate [1, 4].

Adhesion forces between different MEMS materials can be measured using the spectroscopy in point mode of atomic force microscope (AFM). The adhesion between the AFM tip material and MEMS materials can overwhelm the other forces at play.

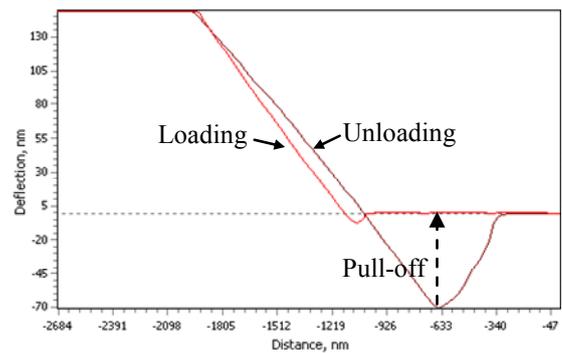


Fig. 2. Loading - unloading AFM curve obtained using spectroscopy in point mode.

Based on the spectroscopy in point mode, AFM experimental curves are obtained as presented in figure 2. "Distance" is the vertical displacement of piezo table and "deflection" is the bending deflection of AFM probe. The deflection-distance curves give the direct measurement of tip-sample interaction forces as a function of the gap between the tip and sample. Using the known stiffness of AFM probe, experimental dependance between force and displacement of piezo-table can be computed as presented in figure 3. The adhesion between tip and sample is characterized by so-called pull-off force. The pull-off force is related in current continuum contact mechanics model to the work of adhesion.

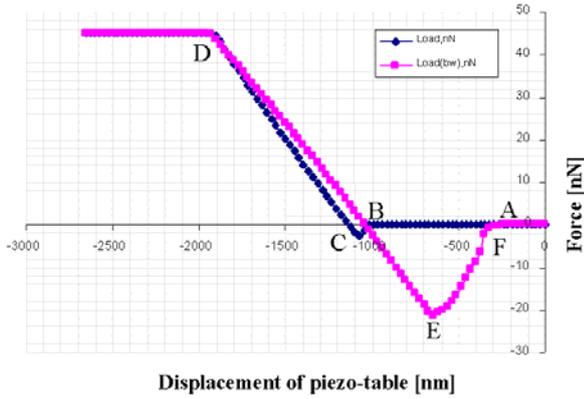


Fig. 3. Force versus AFM piezo-table displacement during experimental test (the contact between AFM tip Si_3N_4 and a gold material).

During the approach (A–B) no interactions occur between the tip and the sample surface (Fig.3). As the tip-surface distance becomes sufficiently small, the gradient of the attractive force overcomes the cantilever spring constant and brings the tip in contact with the sample surface (position C). Further approaching causes a deflection of the cantilever (C–D). The unloading part of the force–displacement curve starts from position D, the deflection of the cantilever is decreased as the sample surface retracts from the tip. When the sample surface is further withdrawn from the tip, the cantilever is deflected owing to adhesive forces. At position E, the elastic force in the cantilever overcomes the force gradient and the tip snaps off from the surface (position F). From position F to A, the cantilever returns to its equilibrium position.

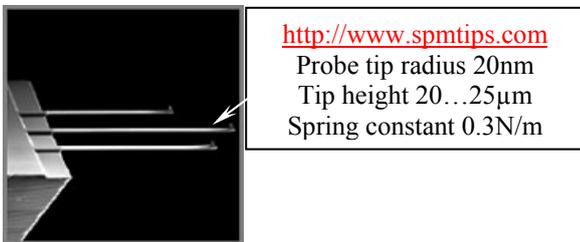


Fig. 4. AFM probe used in experimental investigations.

Figure 4 shows the AFM probe using in our experiment with a bending stiffness of 0.3N/m. Using the experimental AFM values the dependence between force of AFM probe and displacement of piezo-table was computed for investigated materials: gold and aluminum. The maximum adhesion force between the AFM tip

(Si_3N_4) and the samples are: 21nN for gold and 13nN for aluminum.

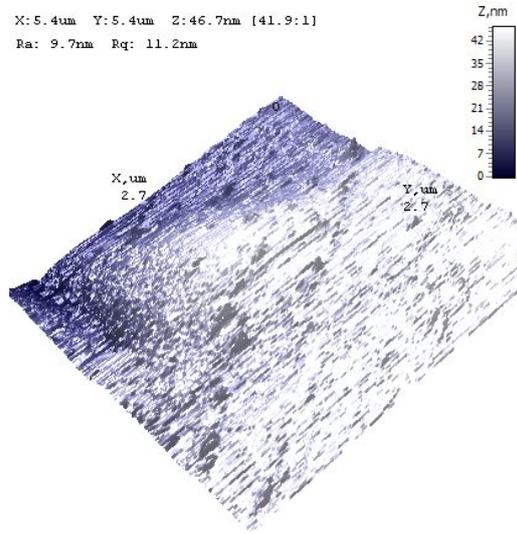


Fig. 5. AFM topography of an aluminum surface ($R_a=9,7\text{nm}$).

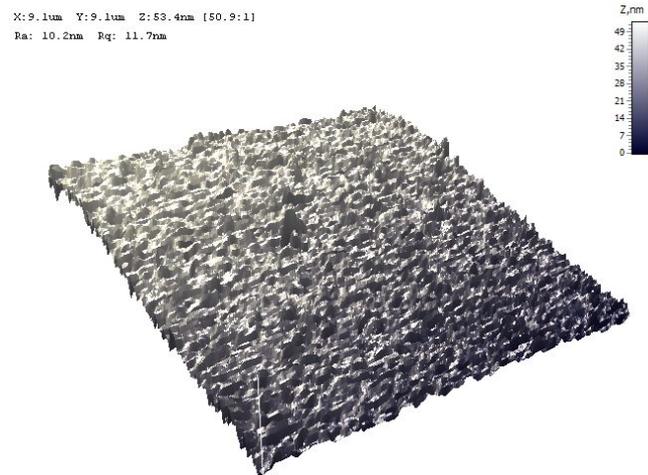


Fig. 6. AFM topography of a gold surface ($R_a=10,2\text{nm}$).

Based on the scanning mode of AFM, the surfaces topographies of investigated samples are obtained. In figure 5 an aluminum surface with R_a of 9,7nm is plotted and figure 6 shows a gold surface with R_a of 10,2nm.

The other gold surfaces with different roughness are identifies. Figure 7 shows a gold surface with a roughness R_a of 27,2nm, in figure 8 a gold surface with R_a of 53,2nm is illustrated and the other gold surface with R_a of 72,7nm is presented in figure 9. For each of them the spectroscopy in point is taken in order to estimate the roughness effect on the adhesion force between the AFM tip and investigated samples.

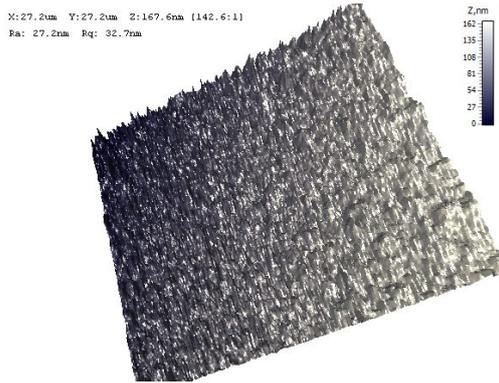


Fig. 7. AFM topography of a gold surface ($R_a=27,2\text{nm}$).

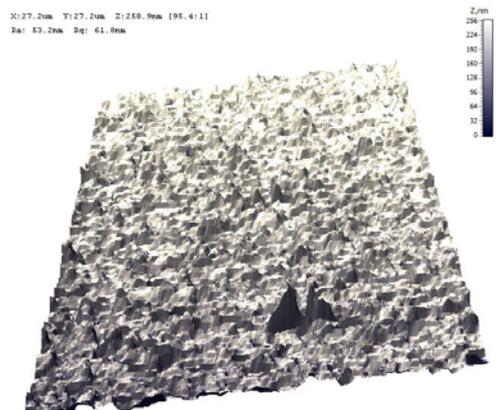


Fig. 8. AFM topography of a gold surface ($R_a=53,2\text{nm}$).

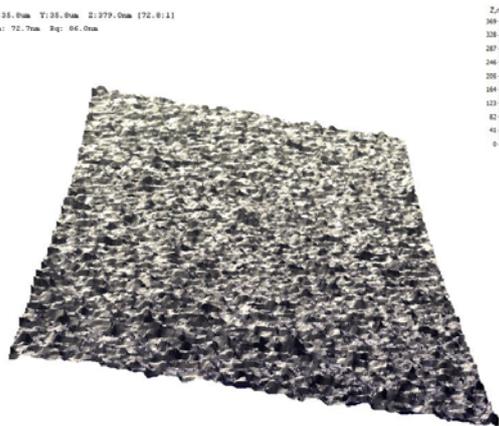


Fig. 9. AFM topography of a gold surface ($R_a=72,7\text{nm}$).

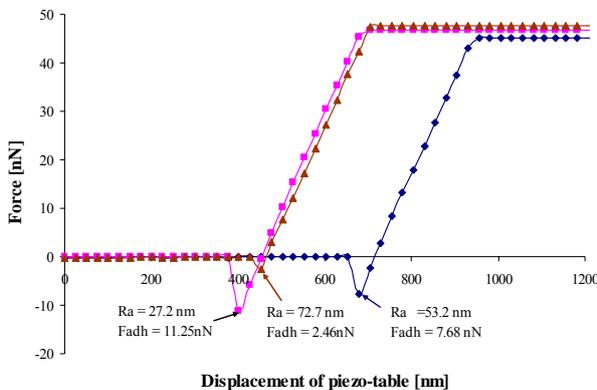


Fig. 10. Adhesion forces between AFM tip Si_3N_4 and gold surfaces with different roughness.

Figure 10 presents the roughness effect on the adhesion force between the AFM tip (Si_3N_4) and investigated gold samples. Because the adhesion forces are given by the unloading part of AFM curves only these parts are considered in figure 10.

The same experiments were performed using a gold coated AFM tip in contact with investigated gold samples. The cantilever used in the second experiment is Cr-Au coating formed as a 20-nm Au film with a 20-nm Cr sublayer, which is deposited for better adhesion of Au. The results of gold-to-gold adhesion forces as a function of roughness are presented in figure 11.

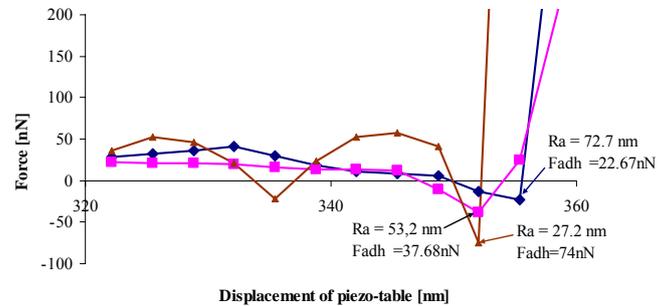


Fig. 11. Adhesion forces between gold coated AFM tip and gold surfaces with different roughness.

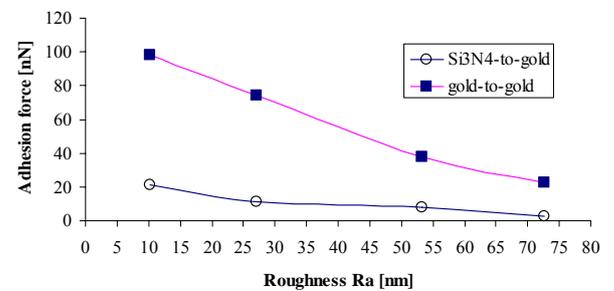


Fig. 12. Adhesion force versus roughness between the AFM tips and investigated gold surfaces.

Figure 12 presents the experimental dependence of adhesion forces versus surface roughness for two interacting surfaces: Si_3N_4 as tip material to gold surfaces; gold coated of the AFM tip to gold surfaces. The adhesion forces decreases as the roughness increases.

4. CONCLUSIONS

In this paper, experimental investigations of the interaction forces between different MEMS materials are presented. The adhesion between the AFM tips in contact with gold and

aluminum surfaces is determined. The adhesion effect can be easily investigated using the AFM force spectroscopy mode. Two materials for the AFM tip were chosen: Si₃N₄ in the first experiment and gold coated in the second experiment. Gold surfaces with different roughness were identified for tests. Contact area between AFM tip and samples depends by the roughness and it is larger for small surface roughness. The results from laboratory indicate that the adhesive forces increase with decreasing of the surface roughness. The nano-scale adhesion force is influenced by the surface energy and the roughness of surface. Based on different surface energy, different adhesion forces were obtained using the same AFM tip (Si₃N₄) in contact with gold and aluminum surfaces with close roughness. Materials with small surface energy are characterized by small adhesion forces occurring during interaction.

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Investigatii tribologice asupra efectului de adeziune la materialele MEMS

Rezumat: Efectul rugozitatii suprafetelor asupra adeziunii la diferite materiale MEMS este analizat si prezentat in acest articol. Asperitatile suprafetelor pot reduce sau creste aria de contact la interactiunea dintre diferite componente MEMS. Prin urmare, efectul rugozitatii trebuie sa fie luat in considerare in cazul analizei tribologice la materialele MEMS. Adeziunea este unul dintre principalii factori de iesire din uz a componentelor cu miscare relativa din structura unui microsystem. Adeziunea este influentata de proprietatile materialelor, caracteristicile suprafetei si conditiile de lucru. Investigatii experimentale sunt realizate prin utilizarea unui microscop de forta atomica prin care se determina adeziunea dintre cantileverul AFM aflat in contact cu suprafete din aluminiu si aur avand rugozitati diferite.

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