PLASMA CLEANING OF SILICON SURFACE OF ATOMIC FORCE MICROSCOPY PROBES*

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Silicon is the most common material used in fabrication of commercial *atomic force microscopy* (AFM) probes. However, the surfaces of the AFM probes surface are rarely clean due to adsorption of airborne and package-released hydrophobic organic contaminants. Cleaning of silicon AFM probes removes the contaminants and renders very hydrophilic probe surfaces. This work presents a simple method of cleaning silicon AFM probes. The method consists in immersing the AFM probes in the negative glow plasma of a glow discharge in air at low pressure. To avoid destruction of the AFM probes, they are kept floating. Cleaning for ten minutes in air plasma at pressure of 6 Torr and cathode current density of $50\mu A\cdot cm^{-2}$ provides a very hydrophilic character to the AFM probe surfaces. The cleaning effectiveness is evaluated by water contact angle measurements of the AFM probe surfaces, which showed a drastic decrease from about 90° to less than 5°. Cleaning of the silicon surfaces of AFM probes and flat samples resulted in significant increase of adhesive and friction forces measured in air on hydrophilic surface of a glass sample.

Key words: plasma cleaning, silicon surface, Atomic Force Microscopy, surface forces.

1. INTRODUCTION

Silicon is the most common material used in fabrication of commercial *atomic force microscopy* (AFM) probes. The surfaces of the as-received AFM probes surface are rarely clean due to adsorption of airborne and package-released hydrophobic organic contaminants [1, 2]. The contaminant molecules are hydrophobic and they render a hydrophobic character to the surface on which they are adsorbed. Thus, a simple way to check the cleanness of the AFM probes is to determine the water contact angle of their surfaces. Cleaning of silicon AFM probes removes the contaminants and renders very hydrophilic probe surfaces [1].

This work presents an efficient and simple method of cleaning AFM probes. The method consists in immersing the AFM probes in the negative glow plasma of a glow discharge in air at low pressure. To avoid destruction of the AFM probes,

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they are kept floating. Cleaning of the probe surface is attributed to the positive ion bombardment of the floating probe surface and oxidizing species of air plasma (oxygen radicals). AFM probe cleaning provides hydrophilic character to the probe surfaces. This has also the effect of increasing of adhesive force measured by the AFM probes in air on hydrophilic samples. Therefore, the cleaning effectiveness is evaluated indirectly by measurements of water contact angle and by adhesive force. The efficiency of the present plasma cleaning method is estimated in comparison with a previously published plasma cleaning method [1].

2. EXPERIMENT

2.1. PLASMA CLEANING

Before plasma cleaning, the AFM probes were pre cleaned by immersing them in organic solvents (10 minutes in acetone, and 10 minutes in ethanol) at 50°C. Then, the probes were washed with deionized water, dried and cleaned by plasma. Two plasma cleaning methods were tested: (I) the AFM probes were placed on the cathode surface of a *dc* discharge in air at low pressure, the probes being protected by an electric shield against the high electric field of the cathode sheath [1]; (II) the AFM probes were immersed in the negative glow plasma of a dc discharge in air at low pressure, the probes being placed on a thick glass slide placed on the cathode. Sketches of the two plasma cleaning methods are given in Fig. 1. Both methods use the negative glow discharge plasma of a dc discharge in air at pressure of 900 Pa, discharge voltage of 350 V, and discharge current intensity of 5 mA in a commercial plasma sputtering/cleaning system (Edwards Sputter Coater S150B) working in the cleaning mode. The cathode was a tantalum disk with diameter of 10 cm. In the method I, the shield created an electric field-free volume where the oxygen radicals produced in the negative glow plasma in air can diffuse in through the open sides. In method II, the probe surface was maintained floating in negative glow plasma by a thick glass slide. In the latter case, the cleaning is attributed to radical oxygen species and positive ion bombardment of the floating probe surface.

2.2. AFM PROBE INTEGRITY

Plasma cleaning of the AFM probes may harm the AFM probes by altering the cantilever elasticity, tip geometry, and quality of metallic reflex coating. The integrity of the AFM probe cantilevers after the cleaning was checked by measurements of thermal noise spectra. No significant changes in cantilever resonant frequency was observed, which proves that the elasticity of the cantilevers was not affected by the cleaning procedure. The integrity of the probe tips after cleaning was checked by analysis of images of sharp edges (nominal curvature radius of 5 nm) of a grating sample (TGG1 from NT-MDT). Cleaning resulted in no significant change of tip curvature radius. The damage caused by cleaning procedure to the reflex coatings of the AFM probes have been evaluated by measurements of the total intensity of the laser light beam reflected by the as-supplied and cleaned AFM probes. It was found that the reflectivity of the aluminum reflex coating decreased to about 50% of the initial values, while the reflectivity of the gold reflex coating decreased to no less than 90% of the initial values.

2.3. COMPARISON BETWEEN DIFFERENT CLEANING METHODS

States of AFM probe surfaces before cleaning, after pre cleaning, and after cleaning by the two methods were evaluated by measurements of water contact angle and adhesive force. Figure 2 presents results of water contact angle measurements

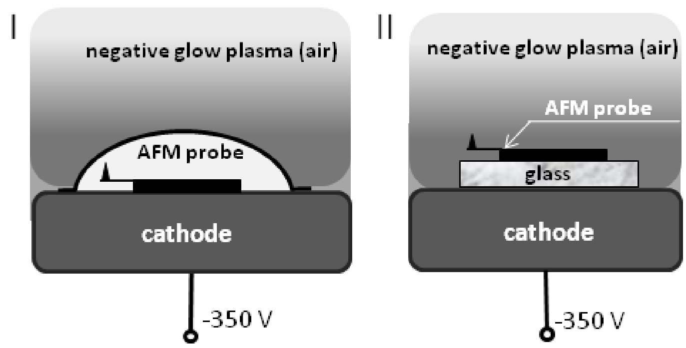

Fig. 1 – Sketches illustrating the two methods used for plasma cleaning of the AFM probes. The dimension of the AFM probe is exaggerated for clarity.

as function of treatment time. Both plasma cleaning methods are efficient in removing contaminants from AFM probe surfaces, a treatment time of 10 minutes resulting in a decrease of water contact angle from 90° to about 5°. Also, the water contact angle measurements showed that cleaning by organic solvents is not efficient. The water contact angle measurements are confirmed by measurements of the adhesive force between ATM tips and hydrophilic glass surface in air (Fig. 3). Increase of the probe surface hydrophilic property results in a corresponding increase of adhesive force between the AFM tip and a hydrophilic sample surface. The adhesive force between an AFM tip and a sample surface in air is caused mainly by the water bridge formed by capillary condensation of water vapor from atmosphere. Increase in AFM tip surface hydrophilicity results in an increase of water bridge volume and adhesive force [3]. The adhesive force increased by 50%

as result of the AFM probe cleaning by ethanol and acetone, and by 200% as result of plasma cleaning of the AFM probes. Our measurements (results not shown) revealed that plasma cleaning of the AFM probes had drastic effects also on friction force measurement in contact AFM and on the phase lag in tapping AFM.

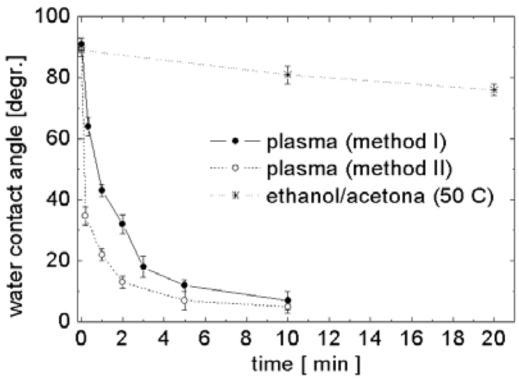

Fig. 2 – Water contact angle measured at different treatment time values (silicon probes NSG 11 from NT-MDT).

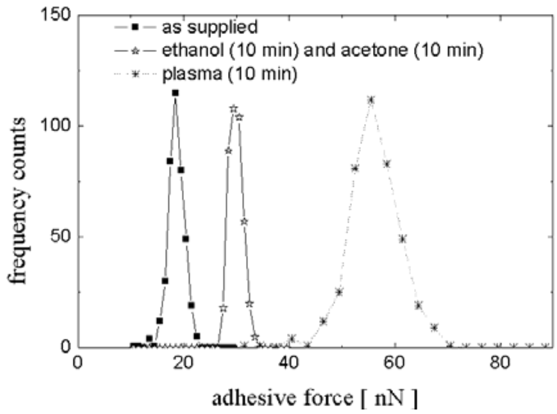

Fig. 3 – Histograms of adhesive force measurements measured with AFM probes before and after cleaning.

3. CONCLUSION

The present work proposes a simple plasma cleaning method for commercial AFM probes. The probes were kept for 10 minutes in the negative glow plasma of a dc glow discharge in air at pressure of 6 Torr. The cleaning effectiveness was evaluated by water contact angle measurements of the AFM probe surfaces, which showed a drastic decrease. Checks on the integrity of the cleaned AFM probes proved that this plasma cleaning method does not harm the cantilevers or tips of the AFM probes.

Cleaning of the silicon surfaces of AFM probes and flat samples resulted in significant significant increase of adhesive force in air. This increase of the adhesive force is attributed to the increase of surface hydrophilicity and, consequently, to the increase of the size of the nanoscopic water bridge formed at tip-sample contact.

Generally, the increase of the adhesive and friction forces is not desirable for the propose of contact AFM imaging of samples in air. However, the effect is useful for increasing of the chemical contrast in friction and adhesive force images taken on samples with hydrophobic-hydrophylic surface patterns at nanoscale [4]. Moreover, cleaning and hydrophilization of the AFM probes has the benefic effect of decreasing of adhesive and friction forces in water [1].

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