DETERMINATION OF THE ADHESION PROPERTIES OF MICA VIA ATOMIC FORCE SPECTROSCOPY

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This paper addresses the Adhesion of mica surface employing Atomic Force Microscope (AFM) as a surface force apparatus. AFM is commonly used for atomic and nano-scale surface measurements. Based on the relations between cantilever responses and tip-sample interaction, methods for quantitative evaluation of a sample's mechanical parameters are described and issues concerning the use of AFM are discussed. The measurement of the Force-Distance curve was performed implementing Atomic Force Spectroscopy (AFS). During these measurements, the static deflection of the cantilever is monitored as a function of piezoelectric element displacement. The recorded plot

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is then used to quantitatively measure the mechanical properties like adhesion and elastic modulus. Forces were measured by multiplying the distance by force constant of the cantilever thorough Hook's law. It was necessary to calibrate the force constant of the cantilever to perform a precise force measurement. Force-Distance curves were obtained in three different points on the surface of mica and Distant Dependant Measurement (DDM) was conducted 10 times per each point by 1.5s interval. Adhesion force was then calculated in every single curve and the final data was the mean of thirty different curves.

Keywords: Atomic force spectroscopy; adhesion; distance dependent measurement.

1. Introduction

Scanning probe microscope (SPM) is one of the newest branches in the field of microscopy. It is known as the complement of optical and electron microscopes. In this microscopy technique, the surface of the sample is scanned by a sharp tip¹. By recording the signals, resulting from the interactions of the tip and the surface, the topography image from the surface of the sample can be obtained¹.

Atomic Force Microscope (AFM) is one of the most important members of the SPM family. The surface is scanned by a tip usually made from silicon nitride. Its diameter is around 5-30 nanometers and it is attached to a bar shaped spring named cantilever. The cantilever's length is usually in the order of 100-200 microns.

In addition to the excellent resolution of topography images inherent in this technique, AFM is capable of performing force measurements in nanometer range. These small forces are the result of interaction between the surface of the sample and the tip of the cantilever. Atomic Force Spectroscopy (AFS) is one of the most promising working modes of AFM that is typically utilized to analyze these interaction forces². The foundation of AFS is based on its ability to deliver force-distance curves which are the direct results of approaching and retracting of the cantilever to and from the surface of the sample. The cantilever approaches to the surface as the attractive forces overcome its spring constant. By the time the cantilever contacts the surface, repulsive forces overwhelm and make the cantilever to retract from the surface³. The quantity of the interaction force can be measured by multiplying the force constant of the cantilever and the distance between the cantilever and the surface, horizontal axis of the force-distance curves, based on the Hook's law².

According to the Hook's law, for precise measurement of the force, the force constant of the cantilever must be calibrated. There are several calibration methods like geometry of the cantilever², reference cantilever³, added mass and thermal noise.⁴

The cantilever deflection is induced by the interaction force between the tip and the sample⁵. Upon recording the deflection of the cantilever while it's scanning the surface, topography image of the sample can be obtained. Fig. 1 shows different parts of the so called force-distance curves.

Fig. 1. Different parts of the force-distance curve in clean mica sample

According to Fig. 1a, the cantilever is far from the surface, located in its equilibrium position, and the cantilever is undeflected⁴. As a result of attractive forces, the cantilever jumps into contact with the surface (Fig. 1, point b). Further movement towards the surface results in bending of the cantilever due to the stiffness of the surface (Fig. 1, point c). The slope of this line can be considered as Young's modulus of the material. The path between points (d) and (e), appears when the tip is withdrawn and the cantilever retracts from the surface. While the cantilever is trying to jump off the contact, adhesion forces may occur and cause the cantilever to adhere to the surface (Fig1, points (f) to (g)). Adhesion force is measured based on the difference between these two curves. Finally, by overcoming the adhesion force, the cantilever is no longer in touch with the surface and will go back to its equilibrium position (Fig. 1, point h).

The aim of this study is to measure the adhesion force, as one of the interaction forces. This is done through force-distance curves obtained from the surface of Mica. In order to measure this force quantitatively, the force constant of the cantilever was calibrated by the thermal noise method. After the calibration of the force constant and obtaining the right force-distance curves, adhesion force was measured.

2. Materials and Methods

2.1. Materials

In this study, mica film, purchased from Danish Micro Engineering (DME) Company, was used to measure the adhesion forces employing AFM, Dualscope/ Rastroscope C26, DME, Denmark.

2.2. Methods

2.2.1. Topography imaging

The first step in the measurement of adhesion forces was to obtain the topography image of the surface of mica. The topography images were recorded with AFM, Dualscope/Rastroscope C26, DME, Denmark. The non-contact working mode of AFM was used with a DC probe and a force constant between 0.07-0.4 N/m according to the manufacturer. All experiments were conducted in air. The scanning area of the images was 500×500 nm² and the resolution was 128×128 pixels. Fig. 2 is the topography image of clean mica obtained with a DC probe.

Fig. 2. Topography image of the surface of clean mica

2.2.2. Distance Dependant Measurements

The second step was to obtain the force-distance curves in order to perform the quantitative measurements of adhesion forces. Calibrated force-distance curves were obtained at each point of the topography image performing the distance dependant measurements (DDM). In order to get the best force-distance curve; one should change the external force at each point during DDM.

In this study, different amounts of external forces were applied at different points of the sample. The appropriate force-distance curve was then acquired according to the conditions mentioned in Table 1.

Table 1. External applied force for mica samples in order to obtain the appropriate force curves

Sample	External Force
As-prepared Mica	15.3 nN
Clean Mica	8.3 nN

The experiments were performed at 3 random points on the surface of the mica. 10 force-distance curves were obtained with a time interval of 1.5 second at each point. Adhesion force was measured in all 30 curves and reported as the mean of these 30 curves. Fig. 3 is an example of force-distance curves coming from the AFS for mica without any additional corrections.

Fig. 3. Measured force-distance curve of as-received mica sample

It has been reported that the presence of adsorbents or oxide layers could change the adhesive behavior of the surfaces⁵. Therefore, it is essential to have the surfaces chemically clean during the measurement of the adhesion force. In order to study the influence of the surface cleanliness, the experiments were conducted under two different conditions on the mica surfaces; as-prepared and cleaned with acetone.

2.2.3. Adhesion force measurement

As mentioned earlier, adhesion force is the result of the difference between the approaching and retracting force-distance curves. While the cantilever is trying to snap-out from the surface, adhesion force between these two makes the cantilever stay in contact with the surface. According to Fig. 4, the adhesion force can be measured by subtracting the two curves.

Fig. 4. Adhesion force in force-distance curve from clean mica

3. Results and Discussion

3.1. Adhesion force and surface cleanliness

The mean adhesion force measured based on 30 force-distance curves obtained for mica without the cleaning process is given in Table 2.

Table 2. Measured adhesion force from 30 force curves obtained for mica

Mica	Adhesion Force (mean)	Standard deviation
Point 1	0.1786 nN	0.04284
Point 2	0.4955 nN	0.1197
Point 3	0.2655 nN	0.08389

Fig. 5 is an example of the 30 different force curves obtained in point 1.

Fig. 5. An example of 30 force curves in point 1 on the mica surface.

Table 3 presents the results for the mica sample which its surface was cleaned with acetone. This data is also the mean of 30 force curves obtained from 3 different points on the mica surface.

Table 3. Mean of the measured adhesion forces in 30 force curves for clean mica

Mica (cleaned)	Adhesion Force (mean)	Standard deviation
Point 1	2.426 nN	0.46373
Point 2	2.671 nN	0.16482
Point 3	4.284 nN	0.70505

Fig. 6 is an example of the 30 different force curves obtained in point 1.

Fig. 6. An example of thirty force curves in point 1 on clean mica surface

It can be observed that the adhesion force increases from about 0.312 nN for the as-prepared mica to 3.1 nN for the cleaned mica surface. Accordingly, it can be concluded that the adhesion force is extremely sensitive to the condition of the surface. Fig. 7 is a comparison between the mica samples in these two different conditions.

Fig. 7. Investigation of the effect of surface cleanliness on the adhesion force

4. Conclusions

It was demonstrated that Atomic force Spectroscopy is a proper force measurement technique for probing surface interactions. These measurements can be performed on different kinds of the samples with the least need of sample preparation.

Adhesion forces are extremely sensitive to surface conditions. Small amounts of adsorbents and contaminations lead to decrease in the adhesion force. The effect of surface cleanliness was studied on mica samples. It was observed that cleaning the sample surface with acetone can increase the adhesion force from 0.312 nN to 3.1 nN.

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