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Electrostatic Force Microscopy Measurement System for Micro-topography of Non-conductive Devices

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A home-made electrostatic force microscopy (EFM) system is described which is directed toward assessment of the microscopic geometry of the surface of specimens made of non-conductive material with a large thickness. This system is based on the variation in the electrostatic force between the conductive probe and the non-conductive specimen in order to get its surface morphology. First, based on the principle of dielectric polarization, the variation rules of the electrostatic force between the charged probe and the non-conductive specimen were studied. Later, a special tuning fork resonant probe unit made of quartz crystal was fabricated for measurement of the electrostatic force, and the scanning probe microscopic system in the constant force mode was constructed to characterize the three-dimensional micro-topography of the surface of the specimen. Finally, this system was used to perform scanning measurement experiments on the indented surface of the specimen made of the polyvinyl chloride (PVC) material with thickness 3 mm. In the present experimental system, when the external voltage was 100 V and the distance from the probe tip to the specimen surface approximately 100 nm, the variance in the resonant frequency of the probe unit was around 0.5 Hz. These results indicate that this home-made EFM system can effectively characterize the micro-topography of the non-conductive specimen with very large thickness which is above several millimeters.

Keywords: Electrostatic force microscopy, surface morphology, precise measurement, scanning probe microscopy.

1. Introduction

Scanning probe microscopes (SPMs), in particular atomic force microscopy (AFM), are the most effective type of tools to measure surface morphology. However, subject to the measurement principles of the AFM and the length-diameter ratio of the probe, when using the AFM to measure the surface morphology of optical devices, issues such as relatively poor stability, difficulty in achieving completely non-contact measurement, and difficulty in measuring the morphology of surface with large depth-width ratio exist [1]-[4].

Electrostatic force microscopy (EFM) is one type of scanning probe microscopy, and its measurement principles can be used to characterize a to-be-measured specimen by sensing the variance in the electrostatic force between the scanning probe and the to-be-measured specimen [5].

The EFM was first introduced by Y. Martin in 1987, which worked as AFM but detecting long range electrostatic forces [6]. At first, the EFM was used to measure the electrical properties, such as the distribution of surface charges [7]-[9] and surface potential [10]-[12]. As the investigations on the EFM became more in-depth, more researchers begun to use EFM for measurement of the dielectric constant of dielectric materials and to manipulate single charges on certain special carriers for transferring and storage [13]-[15]. Later, studies on the EFM began to focus on improving the measurement resolution and the detection methods of the electrostatic force [16]-[18]. The aforementioned studies on EFM are all subject to situations in which the EFM is used to measure the electrical properties of insulating materials or semiconductors, or the charge capture and release.

The research group led by Professor Gao at Tohoku University proposed using EFM to measure the surface morphology of the optical devices that are made of conductive materials, and they reported satisfactory measurement effects [19]. Compared to using AFM, measuring the surface morphology of microstructures via EFM demonstrates advantages, such as a rapid scanning rate, applicability to measurement of specimen surface morphology with large depth-width ratio, strong signal strength of the probe, high stability, and the capacity to realize true non-contact scanning (distance between the probe and the specimen can exceed 100 nm). In this way, EFM demonstrates advantages with respect to precise measurement for optical devices and high-precision mechanical components.

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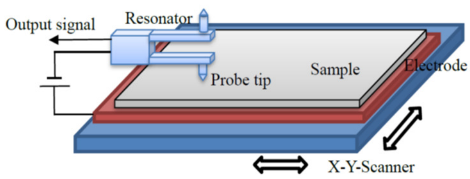
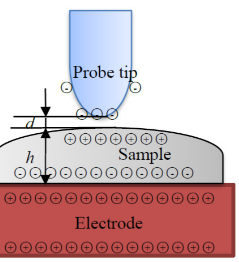
However, most optical devices are fabricated using dielectric materials and have large thickness above several millimeters, and when using the EFM system made by Prof. Gao for measurement, the specimen undergoes a conductive process. This process is relatively complicated and costly. Further, after the surface of the specimen is coated, the original properties of the specimen being measured are destroyed and cannot be restored. To address this issue, the use of an EFM system to directly measure the surface morphology of the insulating specimen with lager thickness was proposed and a series of early-stage theoretical and experimental studies were conducted; the experimental results indicated that the proposed approach was feasible to some extent [20], [21]. Based on the early-stage preliminary experiments, herein we utilized the established scanning measurement platform to perform partial measurement experiments on different non-conductive materials and different voltage-applying methods and compared the results of these measurements to the true morphology of the specimen. The results of measurement indicate that this approach can be used effectively to perform scanning measurement of the microscopic surface of the non-conductive specimen.

2. Measurement principles

The working principles of using the EFM to measure the surface morphology of non-conductive materials are shown in Fig.1. During measurement, the insulating specimen is placed between the metallic probe and the metallic electrode, a bias voltage is applied between the probe and the electrode, and an electrostatic force is generated between the probe and the specimen under the effect of the electric field. The strength of the electrostatic force varies with the distance between the probe and the specimen surface, and such variance can be characterized by the frequency variance of the resonator. By using the constant force mode for scanning, the surface morphology of the specimen can be depicted based on the trace of the probe.



a) measurement principle;

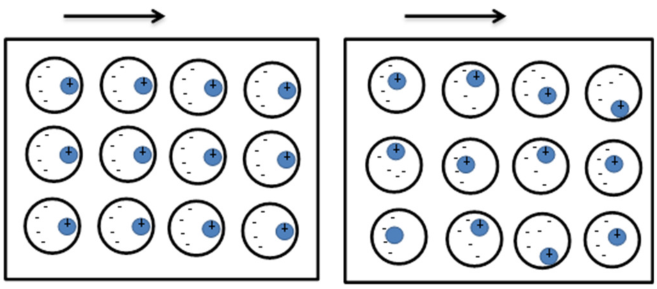


b) principle for the location of the probe tip

Fig.1. Working principle of EFM showing measurement of surface morphology of non-conductive material.

A. *Analysis of electrostatic force on the probe*

When a dielectric medium (i.e., insulating specimen) is placed in the electric field, under the effect of the electric field, the centers of the positive charges and negative charges in the dielectric medium formed by nonpolar molecules experience forces in opposite directions. In this way, the centers of a positive charge and a negative charge are pulled apart for a certain distance to form an electric dipole, which has a certain electric moment. The direction of the electric moment is the same as the direction of the external electric field, and the polarization of the medium formed by the nonpolar molecules (i.e., "electronic polarization") is thus illustrated, as shown in Fig.2.a). For the medium formed by polar molecules, because the electric field shows a torque effect on the electric dipole, the torque allows the inherent electric moment of each molecule to show a trend of rotating towards the direction of the electric field. Thus, the inherent electric moments of the molecules no longer counteract each other, and the entire medium displays electrical properties. This is the polarization of the medium formed by polar molecules, referred to as "orientation polarization", as shown in Fig.2.b).



a) electronic polarization; b) orientation polarization

Fig.2. Schematic view of polarization of a medium in an electric field.

The intensity of dielectric polarization is represented using the polarization strength P, and the relationship between P and the external electric field can be represented as follows:

$$P = \chi E_{\mathrm{e}} \tag{1}$$

In (1), E_e is the intensity of the external electric field; and χ is the polarizability of the dielectric medium, which reflects the difficulty degree of dielectric polarization.

Based on the polarization intensity, the density of the polarization charge on the surface of the dielectric medium in the electric field can be calculated, and the electrostatic force between the dielectric medium and the probe is expressed as follows [20]:

$$F = \frac{\varepsilon_0}{2}\left[1 - \frac{h}{\varepsilon_r (h+d)}\right]^2 U_{bt}^2 \frac{A}{d^2} \tag{2}$$

In (2), F is the electrostatic force on the probe; ε_0 is the dielectric constant of the specimen material; h is the thickness of the dielectric medium (specimen); d is the vertical distance from the surface of the dielectric medium (specimen) to the tip of the probe; A is the area of the probe tip projected onto the surface of the specimen; and U_{bt} is the voltage of the external electric field.

B. Variance in the probe frequency

The relationship between the resonant frequency of the probe under the effect of the electrostatic force is further studied. When the probe receives the electrostatic force in (2), the resonant frequency of the tuning fork resonator varies, and the variation can be represented as follows [19]:

$$\Delta f = \frac{f_0}{2k}F', \tag{3}$$

Here, k is the rigidity of the resonant beam, f_0 is the inherent resonant frequency (base frequency), F' is a first-order derivative of the electrostatic force with respect to the distance, which is calculated as follows: $F' = \partial F/\partial d$.

If the thickness of the sample h is far greater than the distance d, equation (2) can be written as:

$$F = \frac{\varepsilon_0}{2}\left(1-\frac{1}{\varepsilon_r}\right)^2 U_{bt}^2 \frac{A}{d^2} \tag{4}$$

Thus,

$$F' = \partial F/\partial d = -\varepsilon_0\left(1-\frac{1}{\varepsilon_r}\right)^2 U_{bt}^2 \frac{A}{d^3}. \tag{5}$$

Combining equation (3) and (5), the frequency shift can be expressed as:

$$\Delta f = -\frac{\varepsilon_0}{2}\left(1-\frac{1}{\varepsilon_r}\right)^2 U_{bt}^2 \frac{A}{d^3}\frac{f_0}{k} \tag{6}$$

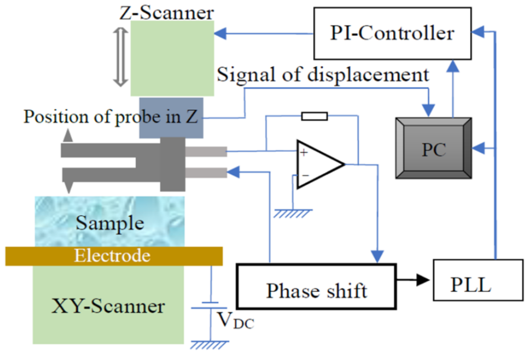
In one measurement system, ε_0, ε_r, f_0, k, and A in equation (6) are all constant, and if a stable bias voltage is applied between the electrode and the probe, the variance in the frequency of the tuning fork resonator (Δf) is a single-value function of the distance (d) from the tip of the probe to the specimen.

Based on the measurement principles shown in Fig.1., a certain bias voltage is applied between the probe and the electrode, such that when the probe approaches the surface of the specimen, the variance in the resonant frequency of the probe can be detected to depict the relationship between this variance and the distance (the vertical distance between the probe and the surface of the specimen).

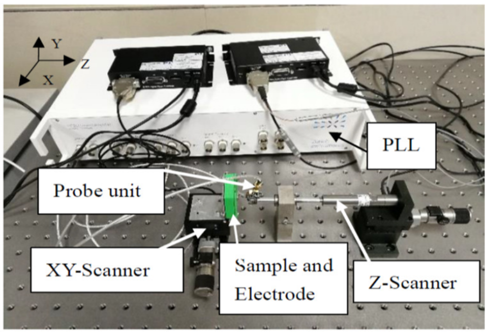
3. PROTOTYPE OF THE MEASUREMENT SYSTEM

A. Constitution of the measurement system

The EFM system for measuring the surface morphology of the specimen is shown in Fig.3. The measurement system consists of the probe and the resonator unit for detecting the electrostatic force, the piezoelectric scanner for scanning along the XY direction, the piezoelectric actuator for driving along the Z direction, and the sensor for displacement measurement. The principle of scanning measurement is similar to the AFM in the constant height mode, and the resonant approach is used to measure the electrostatic force.



a) principle of the measurement system



b) measurement system

Fig.3. EFM system for measurement of surface morphology.

B. Fabrication of the resonant unit of the probe

In order to measure the electrostatic force, the conductive resonant detection unit needs to be fabricated, and Fig.4. shows the scanning electron microscopic image of the fabricated detection unit. The probe tips are fabricated by applying the electrochemical etching method to the tungsten filament with a diameter of 0.7 mm and sticking the probe tips onto the resonant beam of the tuning fork resonator using a conductive paste. The tips of the probe at the resonant beam are distributed symmetrically, where one is used to balance the weight, and the other to assess the electrostatic force of the specimen. The resonator with the attached probe tips has a resonant frequency of approximately 23 kHz in the air. Its quality factor reaches 4000, and the radius of arc of the probe tip is approximately 200 nm.

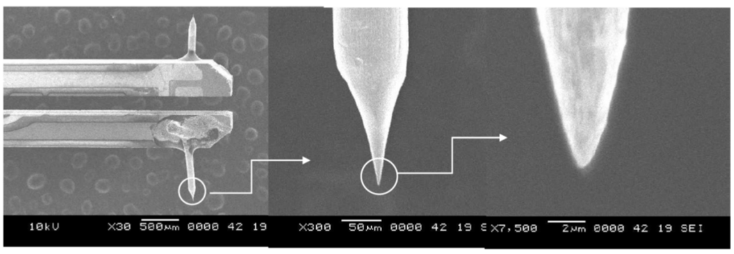


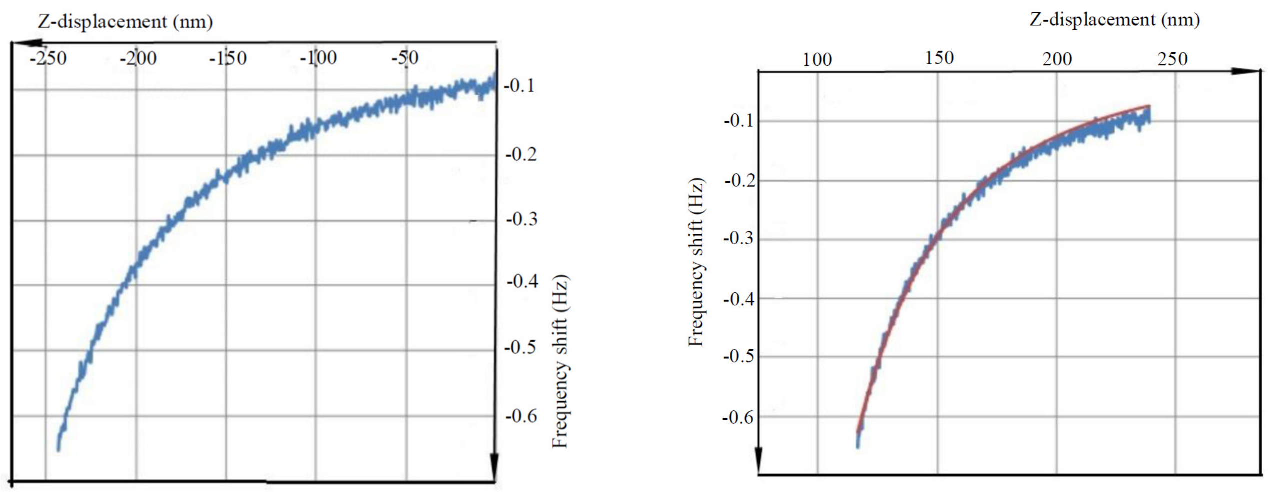
Fig.4. SEM images of the probe unit and tip for scanning.

4. EXPERIMENT AND RESULT ANALYSIS

A. Detection of electrostatic force

In experiments, the bias voltage applied between the probe and the electrode is 100 V, the distance between the probe and the specimen is adjusted through the Z-direction piezoelectric actuator, and the relationship between the frequency of the resonator and the displacement of the probe along the Z direction is recorded. These measurements have been performed in an atmospheric environment. The measurement results are shown in Fig.5. Fig.5.a) shows the variance in the resonant frequency recorded by the measurement system, where the horizontal presents the displacement of the probe along the Z direction recorded by the capacitive sensor of the measurement system. As can be seen in the figure, the estimated value of the vertical distance between tip and sample can be well fitted by an inverse cubic function by using the least squares method, which reflects the relationship shown in (6). The inverse cubic fitting curve can be used to estimate the absolute tip to sample distance based on equation (6), which are shown in Fig.5.b). In Fig.5.b), the horizontal axis (Z displacement) indicates the real distance between the probe tip and the test sample.

When the distance from the probe tip to the surface of the specimen is around 220 nm, the resonant frequency of the detection unit decreases by 0.1 Hz, and when the probe tip is 120 nm away from the surface of the specimen, the resonant frequency of the probe unit decreases by around 0.6 Hz. The measurement results describe the relationship between the resonant frequency of the detection unit and the distance, i.e., the relationship between the electrostatic force on the probe and the distance. As shown in Fig.5., the electrostatic force on the probe increases when the distance between the probe tip and the specimen decreases, while the resonant frequency of the detection unit decreases as the distance between the probe tip and the specimen decreases. The variance relationship fits equation (6) closely.



a) measurement results

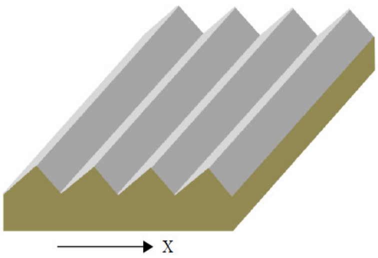
b) fitting result of the electrostatic force

Fig.5. Relationship between frequency shift and the distance of tip-sample.

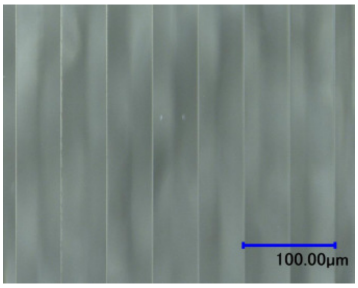
B. Scanning measurement result of the specimen

In order to verify the scanning measurement effects of EFM, a specimen was fabricated with the polyvinyl chloride (PVC) whose surface is indented with teeth at a height of 25 µm, where the teeth bottom is 50 µm wide at the base and symmetrical in shape. The thickness of the sample is 5 mm. The value of permittivity of the PVC is about 5.8 which was measured by dielectric spectrometer. The design diagram of the sample is shown in Fig.6.a), and Fig.6.b) is the optical microscopic image of the specimen. In the experiment, the specimen is coupled to the electrode, and a bias voltage is applied. The probe approaches the specimen until the resonant frequency of the probe begins to vary, and the variation (reduction) in the resonant frequency is configured to be 0.3 Hz by locking the feedback system, such that the distance between the probe tip and the specimen remains constant. Constant height scanning measurement is performed along the X direction, the displacement of the sensor along Z direction is recorded, and the surface morphology is depicted. Fig.7. shows the scanning measurement results at three areas of the specimen, which reflect the micro-topography at the slope, trough, and peak. The scanning length of the slope section is 5 µm, and the scanning length of the trough and the peak is 8 µm. The horizontal axis in Fig.7. indicates the scanning direction (X direction), and the vertical axis in Fig.7. is the height direction of the specimen micro-topography (Z direction). Fig.7. shows the three scanning results of the PVC sample, which reflect the micro-topography of the slope a), trough b), and peak c). Finally, using the same method, we measured a length of 200 µm along the X direction with the EFM system. Fig.8. shows the measurement result.

The measurement results of Fig.7. and Fig.8. are basically consistent with the design parameters of the specimen, which indicates that the EFM system can be used to assess the surface micro-topography of the non-conductive specimen with a thickness above several millimeters.

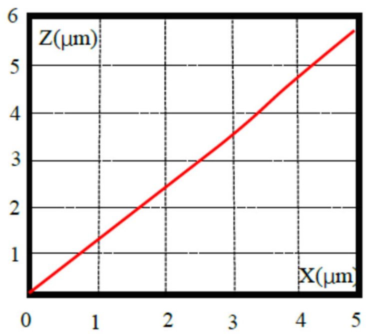


a) structural schematic view of the measurement specimen

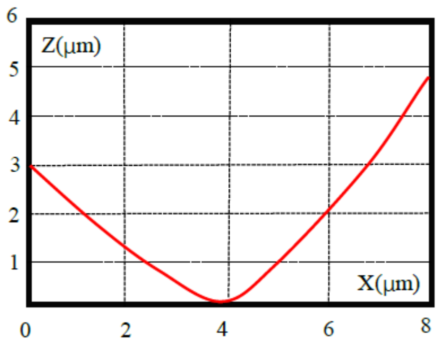


b) microscopic image of the measurement specimen

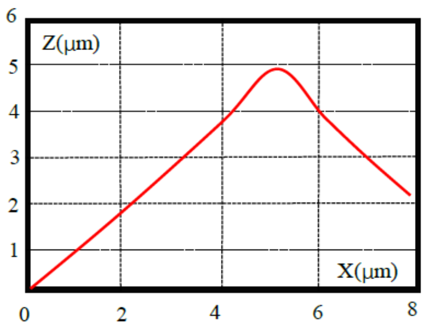
Fig.6. PVC specimen for the scanning experiment.



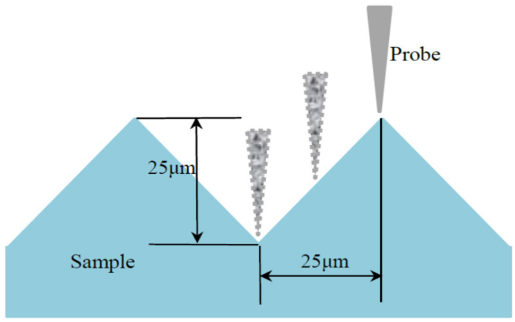
a) scanning result at the slope of specimen



b) scanning result at the trough of specimen



c) scanning result at the peak of specimen



d) schematic of scanning position on specimen

Fig.7. three different local measurement results of the PVC sample with EFM system.

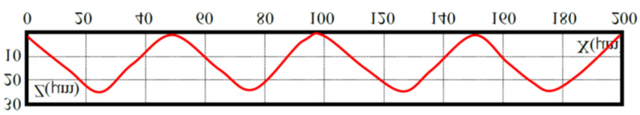


Fig.8. EFM measurement results with 200 μm scanning length.

5. CONCLUSION

The EFM assesses the measurement based on the variation of long-range electrostatic force, so the distance from the probe to the surface of the measured specimen is relatively large, the stability of the probe is satisfactory, and the application of the EFM to measure the surface morphology of a specimen with a large depth-width ratio has advantages. This paper designs the EFM system based on an investigation of the mechanism by which the electrostatic force between the conductive probe and the non-conductive specimen is generated. In this system, the quartz crystal resonator is adopted as the detection unit of the electrostatic force and the piezoelectric actuator is applied as the scanning unit. The EFM system is used to explore the relationship between the electrostatic force between the probe and the non-conductive specimen and the distance from the probe tip to the surface of the specimen, based on which the scanning measurement principles of the constant force mode are used to measure the surface morphology of the non-conductive specimen. The results of measurement indicate that this system can be used to perform microscopic characterization on the surface morphology of conductive and non-conductive specimens, which provides an effective non-contact scanning measurement approach for accurate characterization of optical devices, such as the gratings.

In addition, in all our experiments, the samples are homogeneous. If the samples are heterogeneous, a rather effective permittivity must be considered at the scanned region, which should be extracted in different ways, depending on the sample geometry and constitution.

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