Measurement of contact potential difference and material distribution by using an SEFM

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Abstract:
This paper presents a novel method for the measurement of the contact potential difference (CPD) and material distribution over the sample surface using scanning electrostatic force microscope (SEFM). Since the intensity of electrostatic force generated between the probe and the sample surface is relied on the differences of the CPD, the CPD can be calculated by using the frequency shifts of the probe oscillation when two different bias voltages are applied between tip and the sample. The detection sensitivity corresponding to the experimental conditions has been confirmed by simulation. In addition, the basic characteristic of the CPD measurement system has been evaluated.

Keywords: Scanning electrostatic force microscope, Electrostatic force, Noncontact measurement, Contact potential difference, Nano metrology

1. Introduction
Scanning probe microscope (SPM) represented by scanning tunneling microscope (STM) and atomic force microscope (AFM), has been applied to measure various surface conditions of the device. In addition to the surface observation, SPM has been conventionally used to measure not only the surface topography but also the surface charges, the material distributions, magnetic characteristic, and many other physical properties [1,2]. The distribution of the physical properties in the surface of the functional devices will cause great influence on the reliability of the product, and thus it has been required to evaluate the local distribution of the surface physical properties with nanometer scale accuracy. The surface physical properties such as the composition of the surface and the local distribution of surface contamination are important parameters for the characterization of the materials.

SPM has many advantages in precision measurement of these devices, such as noncontact measurement, high spatial resolution, and the possibility of measurement in air atmosphere. In particular, the ability of mapping the local distribution of the physical properties will allow the applications to the fields from surface science to the development of new electric devices. On the other hand, there is a trade-off for SPM measurement systems. In SPMs, a local interaction between probe and the sample such as a tunneling current or an atomic force can be detected to scan the sample surface. In order to detect the local interaction efficiency, it is necessary to maintain the probe-to-sample distance less than 10 nm during the measurement [3]. This small distance causes a risk for collision between the probe tip and the sample owing to the feedback response limitation. Efforts have been made to improve the stability of SPMs, such as increasing the feedback response or optimizing the scanner structure [4], although the scan range and the sample flatness are often limited and it is still difficult to realize an SPM without collision. A further improvement of measurement stability for a faster scanning is desired to expand the application of the SPM measurement.

In our previous research, an SEFM has been developed for surface profile measurement in noncontact condition [5]. The electrostatic force is detected at two different tip heights with respect to the sample surface to calculate the absolute tip-to-sample distance. Since the electrostatic force is detectable in a large distance, the tip-to-sample distance can be extracted over 200 nm by applying the measuring principle. The principle is effective to avoid the collision of the probe tip and the measuring surface.

Since the intensity of the electrostatic force is depended on the contact potential difference (CPD) of the both electrode material, the SEFM will be able to applicable for the measurement of CPD on the sample surface. The CPD over the sample surface can be calculated by cancelling the influence of the surface profile. Unlike the conventional SPMs, the proposed SEFM has an advantage that the CPD can be measured by maintaining a large tip-to-sample separation during the measurement, and thus it has a possibility to realize crash-free and fast measurement.

It will be possible to estimate the kinds of material over the sample in noncontact condition because the distribution of the CPD can be detected by modulating bias voltage between the tip and the sample. The principle of measuring the CPD can be applied in the fields of semiconductor fabrication and micro-electro mechanical systems.

In this paper, the characteristic of the proposed method is evaluated by the simulation. Also, an experimental result to evaluate the basic characteristic of the proposed measurement principle by utilizing the developed prototype microscope is presented.

2. Principle
In order to obtain the CPD value, it is necessary to
Figure 1. Measurement principle of tip-to-sample distance

Figure 2. Measurement principle of contact potential difference

The principle of measuring CPD is shown in Fig. 2. First, bias voltage between tip and the sample is set to $V_{bias1}$ and the tip-to-sample distance $h_1$ will be calculated by utilizing the equation (4). Then, the bias voltage is changed to $V_{bias2}$ and the tip-to-sample distance $h_2$ will be calculated in the same way. The detected frequency shift $\Delta f_1$ and $\Delta f_2$ corresponding to the applied bias voltage $V_{bias1}$ and $V_{bias2}$ can be expressed by equation (6) and (7), respectively.

$$\Delta f_1 = \frac{1}{2} f \frac{\varepsilon_0 \varepsilon_r (V_{bias1} + V_{opt})^2 R}{h_1^3}$$

$$\Delta f_2 = -\frac{1}{2} f \frac{\varepsilon_0 \varepsilon_r (V_{bias2} + V_{opt})^2 R}{h_2^3}$$

By utilizing equations (6) and (7), The CPD can be calculated by equation (8).

$$V_{opt} = \frac{h_1 \cdot \sqrt{\Delta f_1} \cdot V_{bias2} - h_2 \cdot \sqrt{\Delta f_2} \cdot V_{bias1}}{h_2 \cdot \sqrt{\Delta f_2} - h_1 \cdot \sqrt{\Delta f_1}}$$

In constant height mode, tip-to-sample distance $h$ can be cancelled, so equation (9) can be simplified as;

$$V_{opt} = \frac{\sqrt{\Delta f_1} \cdot V_{bias2} - \sqrt{\Delta f_2} \cdot V_{bias1}}{\sqrt{\Delta f_2} - \sqrt{\Delta f_1}}$$

3. Simulations

In this work, CPD is calculated from the detected frequency shift of QTF. The expected frequency shift detected in different materials has been estimated by simulation. Table 2 shows the example of measurement condition and parameters used in this simulation. The frequency shift was calculated by using the value of work function of different materials versus Tungsten, by using equation (6).

The result is shown in Fig. 3. The horizontal axis is the CPD between each material and W tip. The vertical axis is the estimated frequency shift using equation (6). As shown in this result, the frequency shift would be varied by the different materials, and the difference of the
frequency shift between different materials was calculated to be less than 5 mHz in this measurement condition.

The sensitivity of the detection of CPD will be greatly influenced by the bias voltage and the actual tip-to-sample distance. In order to confirm the influence of measurement condition towards the frequency shift, the parameters included in equation (6) has been changed and the frequency shift has been compared by simulation. The result is shown in Fig. 4. Two horizontal axes are the tip-to-sample distance and bias voltage, respectively. The vertical axis indicates the frequency shift. The simulation result shows that the sensitivity gradient can be expressed as the function of the distance and the bias voltage. With the increase of the bias voltage and the decrease in the tip-to-sample distance, the amount of the frequency shift would be decreased.

Note that a discharge between tip and the sample will occur when the electric field is larger than 1.0 V/nm [9]. For example, when the bias voltage is -50 V, the tip-to-sample distance should be set larger than 50 nm in a practical sense.

4. Experimental procedure

4.1 Schematics of the measurement system

Schematic of the measuring system is shown in Fig. 5. Sharpened Tungsten wire is used as the probe to detect the electrostatic force. A Tungsten wire was electrochemically etched and the probe tip radius was sharpened to be about 100 nm. The probe has mounted on the QTF by conductive epoxy. The QTF is oscillated in its resonant frequency by self-oscillation circuit.

When the probe detects the electrostatic force from the sample, the resonant frequency of the QTF will be shifted according to the amount of the force. The value of the frequency shift is detected by a phase-locked-loop circuit (PLL), and recorded by the PC. Also, the frequency shift is kept constant by controlling the Z scanner displacement using PI controller. When the bias voltage is constant, the electric potential will change due to the distance, thus in order to maintain the tip-to-sample distance constant, the frequency shift is controlled to be constant. The resolution of the Z scanner with linear encoder is 0.5 nm, and the nonlinearity is less than 10 nm over a stroke of 50 μm.

The relationship between the frequency shift of the probe oscillation and the probe-to-sample distance has been taken over two different materials. The result is shown in Fig. 6. The horizontal axis is the estimated tip-to-sample distance calculated from equation (6), and the vertical axis is the frequency shift of the probe oscillation. The bias voltage was set to -50 V. It can be confirmed from the results that the frequency shift is inversely proportional to the tip-to-sample distance, as is shown in equation (6). The $R^2$ values of the inverse fitting curve of each force curves were calculated to be 0.98.
both over 0.999. The result of two curve had a slightly difference and the shift value of Au was larger than the Al. This tendency can be explained from the difference of the CPD of Au and Al towards Tungsten, which is shown in Fig. 4.

The CPD value has been calculated from the curve in Fig. 6 and equation (8). The result is shown in Fig. 7. The horizontal axis is the tip-to-sample distance and the vertical axis is the calculated CPD value. The average value of the calculated CPD of Au and Al can be distinguished and the values tend to be more stable when the tip-to-sample distance was set to be smaller. On the other hand, each value of the CPD had a large difference compared to the experimental value in previous researches (Al-W: +99.3 mV, Au-W: -202.3 mV). The change of the work function of the sample surface or the probe tip, due to the meniscus layer of the surface or the oxidation of the probe tip can be considered as the error factor. A better measurement condition such as measuring in the stable temperature or in a lower degree of humidity condition is necessary.

5. Conclusion
A new method for calculating CPD value by using SEFM has been proposed. The sensitivity of CPD detection has been evaluated by using a numerical simulation. The sensitivity of detecting CPD were expected to be better when the tip-to-sample distance is smaller and the bias voltage between the tip and the sample is larger. Experiment of the CPD measurement has been carried out to evaluate the CPD calculation method. The relationship between the frequency shift of the probe oscillation and the probe-to-sample distance over two different materials had different gradients, which can be explained from the simulation result. The calculated CPD value for different material has confirmed to have difference, although the value itself had a large difference compared to the theoretical value. The meniscus layer over the sample or the oxidation of the probe tip has been considered to be error factor. Improvement of the experimental environment is necessary.

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References