Measurement of wettability for polymer materials using non-contact surface resistivity tester

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Abstract—**The wettability of polymer materials can be enhanced by irradiation with plasma or ultraviolet light due to the introduction of hydrophilic groups onto the surface. The wettability can be typically measured by the contact angle of a water droplet on the material surface. We propose a new method to characterize and evaluate wettability using a non-contact surface resistivity tester. The tester measures the time variation of the surface potential for a test sample charged by corona charging. The surface potential increases faster to lower the surface resistivity of the test sample. The relationship between the contact angle and the surface potential is measured for silicon rubber and polyethylene samples with various levels of deterioration from plasma treatment. The results suggest a negative correlation between the surface potential and the contact angle. The enhancement of wettability of the surface plays an important role to both reduce the contact angle and the surface resistivity.**

I. Introduction

Surface resistivity is one of the most important characteristics of materials when contact charging or tribocharging of a material may cause electrostatic problems such as deposition of dust, spark to the human body and so on. The motion of the surface charge and decay of the surface potential are functions of the surface resistivity. Surface charges on higher surface resistivity materials can remain for longer times and maintain a high surface potential, which results in electrostatic problems. Therefore, the choice of a material and its surface treatment are important to avoid such problems.

The surface resistivity of glass or ceramic materials is lower than that of typical polymer materials, especially under humid conditions. Glasses or ceramics have hydrophilic groups on the surface, whereas polymers have hydrophobic groups. The difference in the surface properties causes the difference in the surface resistivity. For polymers, hydrophilic groups can be introduced by irradiation with plasma or ultraviolet light, which changes the surface resistivity.

Surface resistivity can be characterized by leakage current measurement using two or four electrodes in contact with the surface [1]. The typical surface resistivity of insulating

materials is greater than 10^{12} Ω. Measurement of the higher resistivity range is difficult in practice because the leakage current flowing on the surface is very small and the contact area between the electrode and the surface have a significant effect on the leakage current measurement. We have developed a non-contact surface resistivity tester for the higher surface resistivity range [2]. The tester includes a corona charger and surface voltmeter and measures surface potential next to the corona charged area. The rate of charging up due to the corona charger is a function of the surface resistivity. The measurement range is approximately from 10^9 to 10^{16} Ω [3]. The sensitivity of this tester is very high, even for surface resistivities of over 10^{12} Ω.

Wettability is also an important characteristic of polymer materials, especially with respect to electrical insulation. Silicon rubber is typically used as a non-ceramic insulator because of its light weight and high insulation performance under wet conditions. The difference in insulation performance between ceramic and silicon rubber comes from the difference in the wettability of their surfaces [4-6]. The low wettability of the silicon rubber surface means that the leakage current flowing on the silicon rubber surface is significantly lower than that with glasses or ceramics. However, if the silicon rubber insulator is exposed to an outdoor environment, then the wettability can be enhanced due to the introduction of hydrophilic groups on the surface and the contact angle is reduced. The reduction in the contact angle may cause a reduction in the surface resistivity to that observed for glasses or ceramics.

In this work, the relationship between the contact angle and surface resistivity was investigated for polymer materials deteriorated by plasma treatment. Once the relationship can be determined, the wettability of a polymer material can then be evaluated by measuring the reduction in surface resistivity without the placement of a water drop and visual analysis of the contact angle.

II. NON-CONTACT SURFACE RESISTIVITY TESTER

A. Principle

Figure 1 depicts the non-contact surface resistivity measurement system, including the probe and a test sample. The probe is composed of a corona charger with a needle electrode and a surface voltmeter. The corona charger provides surface charge to the sample placed below the probe with a gap of δ. The charged potential just below the needle V_0, is con-
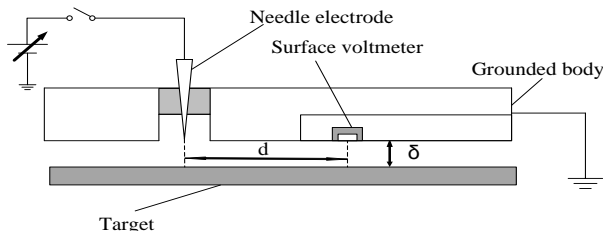

Fig. 1. Non-contact surface resistivity tester. Target is polymer material.

trolled by the voltage applied to the needle electrode. The surface charge on the sample extends over the surface, depending on the surface resistivity; therefore, the change in the surface potential outside of the charged area is also dependent on the surface resistivity. The time variation of the surface potential, $v(t)$, is measured with the surface voltmeter, and the normalized surface potential, $v(t)/V_0$, can be calculated. From a simple one-dimensional model, the theoretical normalized surface potential is a function of the surface resistivity, ρ_s, as shown in Eq. (1), which includes the complementary error function, $erfc$ [2]:

$$\frac{v(t)}{V_0} = erfc\left\{ \left(\frac{\varepsilon_0 \rho_s}{4t\delta} \right)^{\frac{1}{2}} \cdot d \right\}, \tag{1}$$

where ρ_s, ε_0, t, δ, and d are the surface resistivity of the test sample, the permittivity of air, the time from charging, the gap between the probe and sample, and the distance between the charged spot and measured spot. The value of $erfc$ changes from 0 to 1 with an increase in t, so that the normalized surface potential also changes from 0 to 1. The predicted surface resistivity can be derived as Eq. (2) using the measured surface potential $v(T_m)/V_0$ at $t=T_m$:

$$\rho_s = \frac{4\delta}{\varepsilon_0 d^2} T_m \left\{ erfc^{-1}\left(\frac{v(T_m)}{V_0} \right) \right\}^2 = \frac{4\delta}{\varepsilon_0 d^2} T_m V_m, \tag{2}$$

where V_m is the value that corresponds to Eq. (3):

$$V_m = \left\{ erfc^{-1}\left(\frac{v(T_m)}{V_0} \right) \right\}^2. \tag{3}$$

Figure 2 shows the relationship between the normalized surface potential $v(T_m)/V_0$ and V_m. V_m decreases with an increase in the normalized surface potential, and becomes zero when
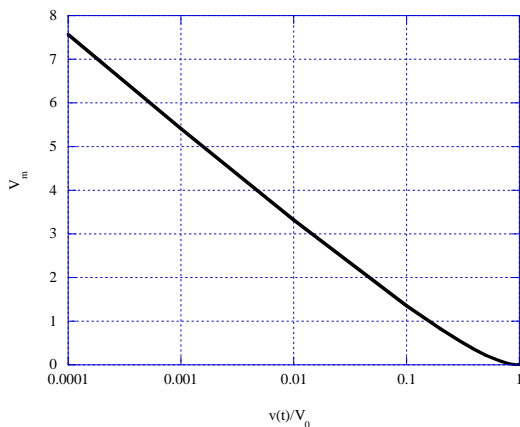

Fig. 2. Non-contact surface resistivity tester. Target is polymer material.

the normalized surface potential is increased to 1. As predicted from Eq. (2), the surface resistivity is a function of the product of T_m and V_m, both of which are determined by measuring the time variation of the surface potential. If T_m is determined from the time when the normalized surface potential becomes a fixed value to obtain a constant value of V_m, then only T_m can be a direct measure of the surface resistivity. However, if V_m is measured from the normalized surface potential at fixed T_m to obtain a constant value of T_m, then only V_m can be a direct measure of the surface resistivity.

It should be noted that Eq. (2) can only be applied for the simple one-dimensional model, which suggests how the measurement system works. The shape of the probe for a practical setup is three-dimensional and the surface charge is spread two-dimensionally. The relationship between the predicted surface resistivity and the measured surface potential should be calibrated using standard test samples with definite surface resistivities. However, standard sheet samples are difficult to produce because the surface resistivity is easily varied with the deposition of dust or a change in the humidity. In this study, the normalized surface potential at a fixed T_m is used as a measure of the surface resistivity. Thus, a larger normalized surface potential indicates a smaller surface resistivity.

B. Tester setup

Figure 3 shows the probe used in this study. The body of the probe is a grounded cylinder, so that the surface charge supplied to the test sample just below the needle can travel linearly to below the surface voltmeter under the influence of an opposite charge induced at the bottom of the grounded body. The distance between the needle and the surface voltmeter is 35 mm. The gap between the bottom of the probe and the test samples δ, is 2 mm. A dc voltage of +3.5 kV is applied to the needle electrode. The measured surface potential for a sample with a conducting surface layer is the same as that of the charging spot, V_0. In the present setup, V_0 was +500 V. The time variation of the normalized surface potential was measured just after application of the dc voltage to the needle electrode. An ionizer was used to eliminate surface charge before and after the measurement.
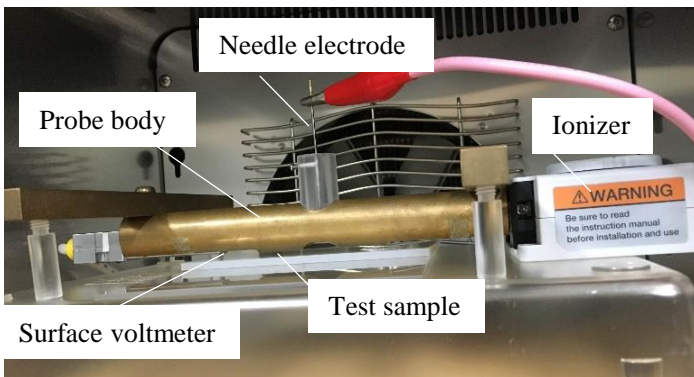

Fig. 3. Experimental setup for normalized surface potential measurement.

III. Experimental

Silicon rubber and polyethylene plates with dimensions of $50 \times 100 \times 5$ mm^3 were used as test samples. The surfaces of the samples were deteriorated with a plasma surface treatment system (Kasuga Co., TEC -4AX) using barrier discharge in air. The degree of deterioration was adjusted according to the exposure time and input power of the system. The time variation of the normalized surface potential was measured for samples with various degrees of deterioration. The surface potential measurement system was installed in a bench-top type temperature and humidity chamber (ESPEC Co., SH-222). After the measurement, the contact angles of water droplets (1.5 µl) at five points on the sample were measured with a wettability evaluation system (Nick Co., LSE-ME2).

IV. Results and Discussion

A. Deterioration of test samples

Figures 4(a) and (b) show the relationship between the contact angle and the plasma input power for deterioration of the silicon rubber and polyethylene samples, respectively. The initial contact angle of the silicon rubber was 100° and was decreased with an increase in the input power. The contact angle became almost 0° at an input power of 65 kJ/m². The contact angle of the polyethylene sample was also decreased with an increase in the input power, although it became saturated at approximately 40° with an input power of 40 kJ/m². The change in the contact angles is caused by the introduction of hydrophilic groups to the surface by the plasma treatment. The difference in the contact angle between the deteriorated silicon rubber and polyethylene samples is due to the difference in the energies of bonds destroyed by the plasma treatment. The contact angles also vary depending on the position of the water droplet placed on the same samples. Non-uniform barrier discharge is different from glow plasma [7], which could be one reason why the contact angle varies. In this study, all contact angle data for each sample were used except when the mean value of the contact angle was required.
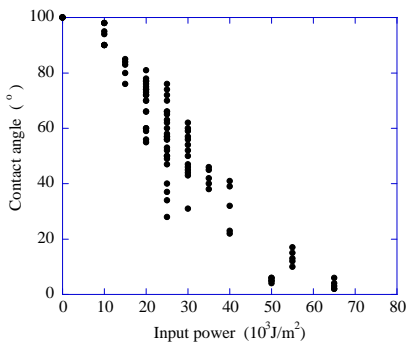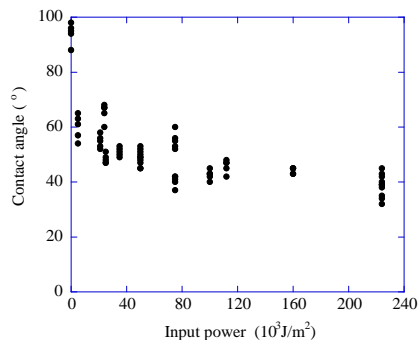
(a)　Silicone rubber　　　　(b) Polyethylene
Fig. 4. Contact angles of test plates under plasma treatment.

B. Time variation of normalized surface potential

Figure 5 shows the normalized surface potential for silicon rubber samples with different contact angles (mean value). The normalized surface potential for the sample before deterioration by plasma treatment and with a contact angle of 100° increased quickly to 0.05 because of detecting the potential of the needle electrode. The potential increase is independent of the traveling of the surface charge. The normalized surface potential is then gradually increased to 0.1 at t = 15 s due to the slow motion of the traveling surface charge.

The normalized surface potential increases faster depending on the decrease in the contact angle. The normalized surface potential at t =15 s becomes more than 0.5 for contact angles less than 61.8°. For fully deteriorated samples with a contact angle of 0°, the normalized surface potential rapidly became close to 1 within a few seconds. The surface charge can spread quickly over the surface. As predicted from Eq. (2), the normalized surface potential at a fixed time $t = T_m$ is a theoretical measure of the surface resistivity. In this study, the normalized surface potential V_{10}/V_0 at $T_m = 10$ s was selected as a measure of the surface resistivity for both silicon rubber and polyethylene samples.

C. Relationship between the normalized surface potential and contact angle

Figures 6(a) and (b) show the relationship between the normalized surface potential and the contact angle for silicon rubber and polyethylene, respectively. There is a strong negative correlation between the normalized surface potential and the contact angle. For silicon rubber, the contact angle becomes smaller than 50° for normalized surface potentials greater than 0.7. For polyethylene, the contact angles become smaller than 50° for normalized surface potentials greater than 0.2. The surface resistivity of polyethylene is significantly decreased even with contact angles larger than approximately 40°. Although the correlation factor is dependent on the material, the normalized surface potential measurement can be a measure of wettability for polymer materials after the correlation factor can be experimentally determined with this type of test.
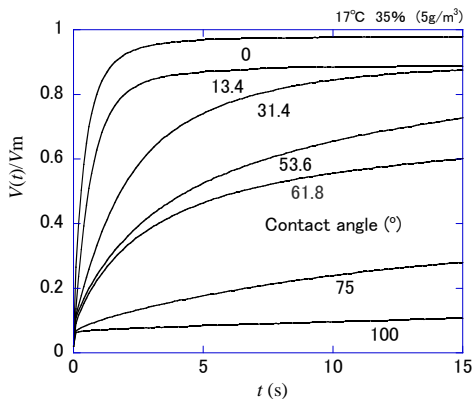

Fig. 5. Time variation of the normalized surface potential for silicon rubber with various contact angles.

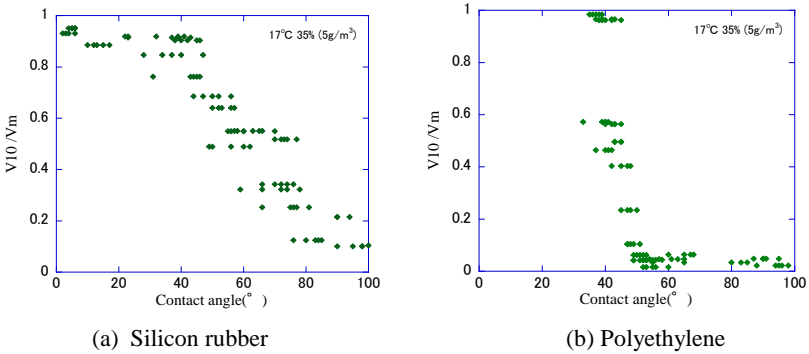(a) Silicon rubber　　　　　　　　　　　　(b) Polyethylene

Fig. 6. Relationship between the normalized surface potential and the contact angle.

The increase in the normalized surface potential for the smaller contact angle samples may be due to an increase in the deposition of water vapor. To confirm this effect, the humidity in the chamber was changed during the surface potential measurements. Figure 7 shows that relationship between the normalized surface potential and the contact angle for absolute humidities of 3 and 10 g/m^3, compared with that of 5 g/m^3 shown in Fig. 6(a). The higher humidity results in a larger surface potential. The water vapor deposited on the surface thus plays an important role to reduce the surface resistivity and increase the normalized surface potential.
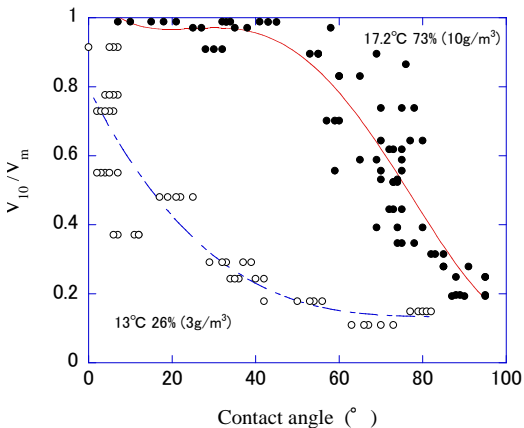

Fig. 7. Relationship between the normalized surface potential and the contact angle for silicon rubber samples under various humidity conditions.

V. CONCLUSION

The contact angles of silicon rubber and polyethylene samples were decreased due to deterioration by plasma treatment. The decrease in the contact angle, i.e., enhancement of the wettability, is considered to be caused by the introduction of hydrophilic groups onto the surface. The wettability also affects the normalized surface potential or the surface resistivity. A negative correlation was confirmed between the normalized surface potential and the contact angle. Surface resistivity measurements of deteriorated polymer samples can thus be used to predict the wettability of samples without the placement of a water droplet to measure the contact angle, after the correlation factor is experimentally determined.

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