Effects of Heating and Discharge on Adhesive Strength Improvement of Polytetrafluoroethylene by Heat-assisted Atmospheric Pressure Glow Plasma

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Abstract: The effects of treatment temperature and discharge power on the improvement of the adhesive strength of polytetrafluoroethylene (PTFE) were independently investigated. The helium plasma treated PTFE (200 W, 250 °C) showed high adhesive strength with an epoxy glue (about 1400 Nm^{-1}). The heating effect for the improvement of adhesive strength began to appear at about 240 °C or more. And the effect of plasma (discharge power) affected the reduction of treatment time over the adhesive strength.

Keywords: Atmospheric Pressure Glow Plasma, Polytetrafluoroethylene, Adhesive Strength

1. Introduction

Fluorinated polymers have taken an important role in industry development. The most typical fluorinated polymer, polytetrafluoroethylene (PTFE) has excellent properties such as high chemical resistance, heat resistance, electric insulation, and low friction coefficient. Thus, PTFE is applied in various fields such as chemical plants, biomaterial, electric wires and cookware.

Since the poor adhesiveness of PTFE prevents its use, the treatments such as wet process with metal sodium or low pressure Ar plasma are often used for surface modification of PTFE. However, each treatment has its own disadvantages such as waste liquid treatment and low production efficiency, respectively. Therefore, a new drying process is expected.

The atmospheric pressure glow (APG) plasma treatment is the most effective alternate method because of the high density of excited species, low temperature and uniform surface treatment [1-3]. And we have succeeded in obtaining higher adhesive strength between the PTFE treated by boron/He plasma treatment and epoxy glue than that of the PTFE treated by wet process in our previous study [4]. Although various plasma treatments were tried, even higher adhesive strength, over 1000 Nm^{-1}, have not been obtained.

Recently, Ohkubo et al. reported a method of significant improving adhesive strength of PTFE, named "heat-assisted plasma" [5]. However, the effects of temperature and discharge power on the improvement of the adhesive strength have not been independently evaluated. Therefore, in this study, we prepared a new discharge chamber, and investigated these effects.

2. Experimental

Fig. 1 shows the standard dielectric barrier discharge chamber with the shower head electrode. The chamber was first vacuumed with a rotary pump, and then the pressure was restored with helium gas to atmospheric pressure. The plasma was generated with a 13.56 MHz power supply. Table 1 shows the plasma treatment conditions.

The lower electrode temperature was increasing during discharge without some copings. Thus, to keep the electrode temperature constant, the lower electrode was heated by sheath heater (very thin tube type heater) first, and then the cooling liquid (Fluorinert, FC-40, 3M Co.) was introduced into the lower electrode just prior to the discharge start. The lower electrode surface temperature was measured by an optical fiber thermometer.

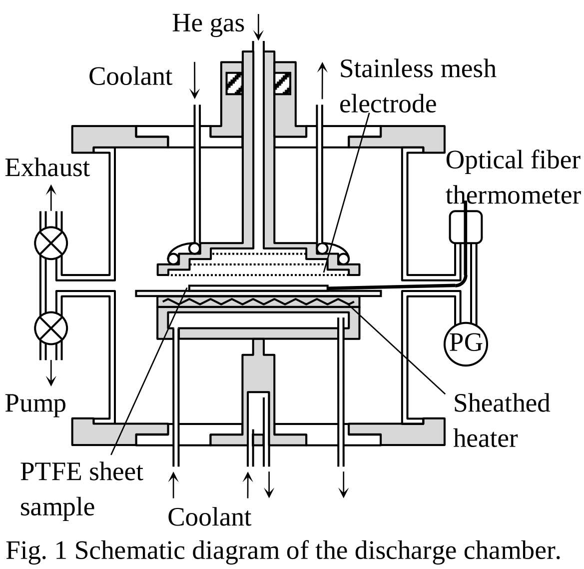

Fig. 1 Schematic diagram of the discharge chamber.

Table 1 The plasma treatment conditions.

Discharge frequency	13.56 MHz
Discharge power	100 ~ 200 W
Discharge gap	3 mm
Treatment time	0 ~ 15 min
He flow rate	2 slm
Treatment temperature	110 ~ 270 °C

The dimension of PTFE sheet was 25 mm × 130 mm × 0.2 mm, and it was washed with trichloroethylene and deionized water in an ultrasonic cleaner before treatment.

The samples for the peel test were prepared as follows: first, a treated PTFE sheet was glued on a stainless-steel plate with an epoxy glue (NICHIBAN Inc. Araldite), and was pressed for 24 hours. The adhesive strength was measured by 180° peel tester at 30 mm·min^{-1} peel speed.

3. Result and discussions

The typical electrode temperature profiles are shown in Fig. 2. The electrode temperature was increasing during the discharge with only the electrode heating without the coolant. On the other hand, when the heater and coolant (heat temperature, 175 °C; coolant flow rate, 50 ml·min^{-1}) were used at the same time, the electrode temperature rapidly increased at 15 seconds and then exhibited a constant value.

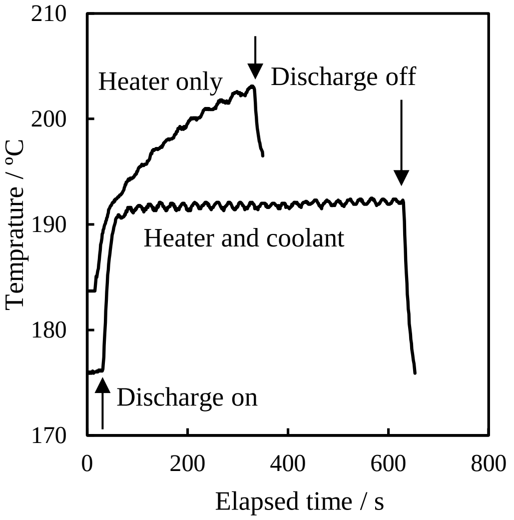

Fig. 2 The relationship between elapsed time and electrode temperature. The discharge power was 100 W.

Fig. 3 shows the relationship between electrode temperature and adhesive strength at 100 W discharge power. Until the electrode temperature reached 240 °C, the adhesive strength depended only on the treatment time. The adhesive strength also began to depend on the electrode temperature at above 240 °C. Eventually, the highest adhesive strength, 1323 Nm^{-1}, was obtained at 15 min treatment time. This adhesive strength value was significantly larger than the value of our previous study (900 Nm^{-1}) [4].

Fig. 4 shows the relationship between electrode temperature and adhesive strength at 200 W discharge power. Until the electrode temperature reached 210 °C, the adhesive strength depended only on the treatment time. Consequently, the maximum adhesive strength was obtained before reaching 240 °C for 100 W. These traits were seen even for earlier treatment times, as seen in 5 min and 10 min.

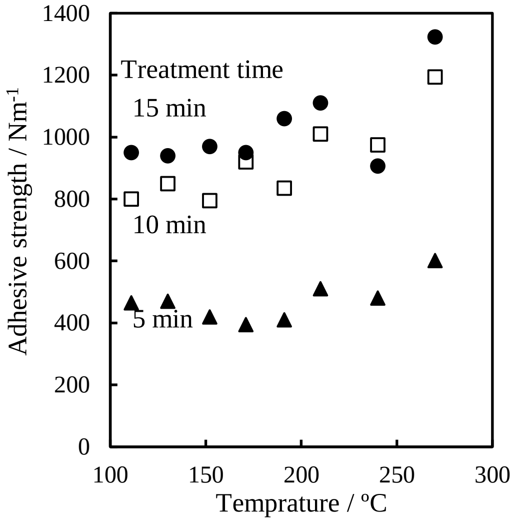

Fig. 3 The relationship between electrode surface temperature and adhesive strength at 100 W.

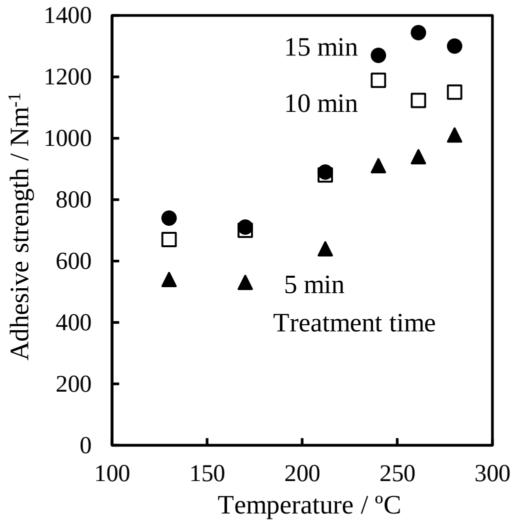

Fig. 4 The relationship between electrode surface temperature and adhesive strength at 200 W.

Fig. 5 and 6 show the relationship between electrode temperature and atomic ratio F/C measured via XPS at 100 and 200 W, respectively. Fluorine atoms were removed only by He plasma treatment. And the amount of fluorine reduction was not dependent on the electrode temperature but was dependent on the discharge power and the treatment time. This result indicated that the cross-linking inside the PTFE was progressing at high temperature (over 200 °C).

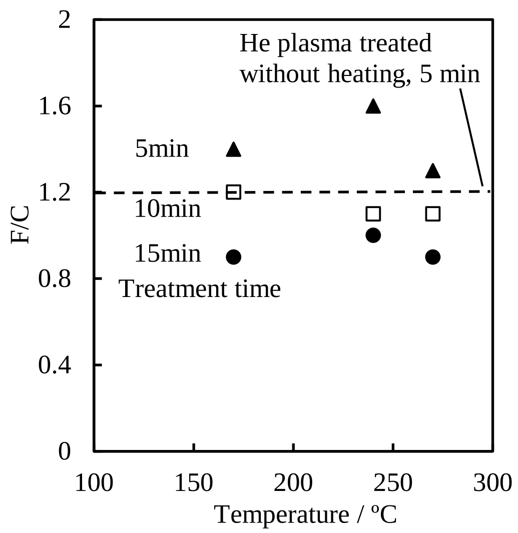

Fig. 5 The relationship between electrode surface temperature and atomic ratio F/C at 100 W.

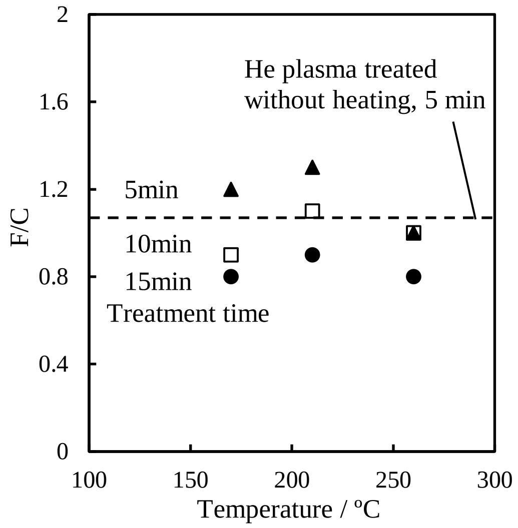

Fig. 6 The relationship between electrode surface temperature and atomic ratio F/C at 200 W.

4. Conclusion

Heat assisted plasma treatment improved adhesive strength of PTFE. The heating effect for the improvement of adhesive strength began to appear at about 200 °C or more. And the effect of plasma (discharge power) affected the reduction of treatment time over the adhesive strength.

5. References

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