Surface charge behaviour of corona-charged thin polymer films - simultaneous LIPP and TSDC measurement

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Abstract. A simultaneous Thermally Stimulated Discharge Current (TSDC) and LIPP (Laser-Induced Pressure Pulse) signal measuring system was constructed to identify the charge behaviour in/on the dielectric film. For 50 or 100 µm thick corona-charged PTFE sheets, the correlation between the spatial charge distribution change with the temperature and TSDC peaks was examined. Corona-charging temperature dependence is also examined.

1. Introduction
The authors have been engaged in making the long-time stable electret and the charge stability of the polymer films has been studied. As Thermal Stimulated Discharge Current (TSDC) measurement is very effective in understanding the charge stability on/in the polymer film, the authors established an automatic digital TSDC analyzing system which was also used for TSDC analysis of the fly ash collected from the combustion flue gas [1]. For the understanding of the charge behaviour, TSDC is very sensitive to the charge, but the correlation between the exact spatial position and the stability of the charge is not yet clear, which is a very important parameter to estimate the stability of the electret. The Laser-Induced Pressure Pulse (LIPP) [2] method is also a very effective technique in understanding the charge in-depth profile. The authors also observed UV-irradiation charge relaxation current which is also very sensitive to the electric field between the metal and the insulator (insulator side). We compared the TSDC spectra and LIPP signals for the same film to identify each TSDC peak corresponding to the position of that charge. After the LIPP measurement, TSDC measurement was done for the same sample but the direct relation between the TSDC peak and LIPP observed signal was not yet clear because of the time delay and the different setting changed the positioning and so on. Finally we designed the new TCDC and LIPP simultaneous measuring system where the LIPP measurements are repeated during TSDC measurement. After large TSDC signal, LIPP signal suggested the charge position of TSDC current source.

2. Experimental
A block diagram of a new simultaneous TSDC and LIPP signal measuring system is shown in Fig. 1 where an ultra low noise pico-ammeter (Keithley 617 or similar high sensitive pico-ampere or femto-
ampere current meter) is connected to the detecting electrode, and the amplified low current signal by the pico-ammeter is digitalized, digitally averaged, and stored in the personal computer memory at each temperature. During TSDC measurement, the signal connector (switch in the Figure) is switched to the high speed amplifier (bandwidth: several GHz) which is connected to the high speed digital oscilloscope (bandwidth: DC to GHz) and data are also transferred to the personal computer as LIPP signal. The charged-sample film is inserted into two metal discs whose one side of the electrode is applied black non-glossy paint to avoid infra-red reflection. That disc is rather thick to remove the electric laser irradiation noise without impedance matching grease because shock-wave reaches to the sample surface with rather large time delay. The β sample film holder is kept in the metal 10 container which has a laser beam inlet aperture of 8 mm in diameter, the electric heater and thermocouple wirings, and BNC connector for the signal analysis connected with the pico/femto-ampere amplifier or a low noise high frequency amplifier. The sample holder temperature can be controlled from the room temperature to 300°C by the digital temperature controller. Typical temperature increase rate for TSDC measurement is 3.6°C/min.

Sample films are 50 or 100 µm thick PTFE (polytetrafluoroethylene supplied by Nichiasu) sheets. Those films are cut 2 or 3 square cm and charged under the needle corona discharge for 10 minutes. The surface potential of the film is usually controlled by the mesh electrode inserted between the film and the corona needle electrode. After the corona charging, surface charge distribution is rather uniform but very high. Typically that film is kept between aluminium foils which reduce the surface charge density greatly but realize the stable charge.
3. Results and Discussions

3.1 PTFE thin film Corona-charged at the room temperature

One example is corona-charged 100 µm thick PTFE film at the room temperature where the corona-needle voltage is – 30 kV and the voltage of the mesh (grid) is – 5 kV during charging period of 10 minutes. TSDC of that sample is shown in Fig. 2. In that figure, three TSDC peaks, β (at 225°C), γ (at 150°C), δ (at 80°C) are identified. If the thickness of the film is thin, much apparent TSDC peaks are observed as shown in Fig. 3 where another new peak α (at 275°C) is also detected. The charging electric field is about double for 50 µm thick film and stable charge, α, is stored. On the other hand, unstable TSDC current peak, δ, is not so clear. That current peak may be dependent on the surface condition (not the electric field).

At 25°C intervals during the TSDC measurements, LIPP signals are also observed. For 100 µm thick PTFE film, those signals are as shown in Fig.4. Peak A is corresponding to the corona-charge surface and Peak B is at the back side of the film. The positive peak should be the counter charge of the negative charge in the electrode surface. Peak position (time from peak B) increases with the increasing temperature, which may be due to the thermal expansion of the film and the change of the elastic wave propagation velocity. Below 75°C, there is no apparent change concerning the peak A. From 75 to 100°C, a slightly larger signal decrease is observed but the 0.02 peak value continues to be constant from 100 to 125°C. From 125°C to 200°C, peak A decreases significantly. Finally peak A disappears completely at 250°C. It may be that final charge decay at 250°C corresponds to TSDC peak α in Fig. 3. Charge decay from 200 to 250°C may cause TSDC peak β although the temperature values for TSDC and LIPP are not equal in both figures. Another LIPP signal peak B may correspond to TSDC peak γ. That tendency is also observed for corona-charged 50 µm thick PTFE film as shown in Fig. 5 where above 175°C the tendency is very similar to Fig.4 and abbreviated. Peak B decreases from 75 to 150°C indicating the TSDC peak γ may be corresponding to that charge. That means γ signal is caused by the relaxation of the positive charge induced inside the insulator from the counter electrode.

Figure 5. LIPP signal of a corona-charged 50 µm PTFE film at room temperature. Observed at 75, 100, 125 and 150°C.

3.2 Corona-charged PTFE film at high temperature

If the sample temperature is high during the corona-charging, TSDC and LIPP signals change significantly. TSDC signals from 100 µm thick PTFE film corona-charged at 100°C is shown in Fig.6. Comparison with Figs. 3 and 6, there is no large difference. δ peak disappears and γ peak temperature decreases. However, the LIPP signal shown in Fig.7 is very different from Fig. 4. Peak A, which was the dominant charge peak in Fig.4, is very small and peak B which was a small signal in Fig.4 is very large and counter charge in the electrode is shown as peak C. A new peak just to the right side of peak B may be caused by the amplifier specific character (that is only a high frequency range amplifier) and
Figure 6. TSDC of a corona-charged 100μm thick PTFE film at 100°C.

Figure 7. LIPP signal of a corona-charged 100μm PTFE film charged at 100°C.

Figure 8. LIPP signal of a corona-charged 100μm thick PTFE film at 160°C. Observed at room temperature.

sharp peak signal causes such parasitic signal. During TSDC measurement, peak B decreases gradually from below 100°C to 200°C.

If the PTFE thin film is corona-charged at more than 160°C, those peaks change drastically. LIPP signal at the room temperature shows in Fig. 8 where the film is charged at 160°C. Peak A becomes very wide indicating the charge distributes not only over the film surface but diffuses into the film inside. TSDC peak is larger than 150°C and charge in LIPP also decays at more than 150°C which is easily understood because of 160°C charging.

4. Conclusions
The correlation between TSDC measurement and space charge measurement by LIPP method was examined for corona-charged PTFE films. Four different TSDC peaks were identified but the space charge is mostly located near the interface and exact position has not yet identified.

References
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