Thermal Stability of Poly(vinyl Fluoride) Film

INTRODUCTION

Poly(vinyl fluoride) is reported in technical brochures to have a dielectric constant of about 9 and a dissipation factor of 1% to 2% (at 1 kHz and 23°C), and an electric strength of 1.5×10^6 volts/cm (at 23°C). It is also reported to be thermally stable up to 300°C, although 107°C should not be exceeded in continuous use.

This material appears to be an excellent candidate for a capacitor dielectric when used at temperatures below 107°C. However, many capacitive devices experience temperatures above 107°, and, since we had a need for a high dielectric constant insulator to be used in a device which would operate at temperatures up to 150°C, we carried out a preliminary investigation of the effects of relatively elevated temperatures on poly(vinyl fluoride) film. This was done in the absence of oxygen to approximate more closely the conditions existing in the device after the film is sandwiched between electrodes and then encapsulated in a plotting compound.

EXPERIMENTAL

Strips of poly(vinyl fluoride) film ($10 \times 15 \times 0.0012$ cm) were heated at 145°C in a desiccator under a continuous dynamic vacuum of about 10^{-3} torr. Similar strips were sealed in break-seal tubes after evacuating the tubes to about 10^{-3} torr. These tubes were then aged at 145°C and examined after various time intervals.

Electric breakdown was the property of the film chosen as the criterion of insulation deterioration.[1] The electric breakdown was measured at a number of points on each film, at room temperature, by placing the film between two flat electrodes (5 cm in diameter), and applying an increasing voltage (d.c.) until dielectric puncture. The volatile contents of the sealed tubes were examined by mass spectrometry.

RESULTS

The films in the sealed tubes were examined visually. After seven days, the films showed a noticeable brown discoloration. After 14 days, they became black. At the end of 21 days, they had started to disintegrate, and after 28 days, they were so fragile that no electrical measurements could be made, while the films in the dynamic vacuum showed only slight discoloration at the edges after this time.

The electrical properties of poly(vinyl fluoride) before aging were: electric breakdown = 1460 volts (average of ten readings); capacitance = 0.0106 μF (1 kHz, 25°C); dielectric constant = 7.5 (1 kHz, 25°C); dissipation factor = 2.0% (1 kHz, 25°C). All of these values were measured at 25°C on 0.0012-cm-thick films. These values confirm that the material is a good candidate for a capacitor dielectric.

After 14 days in the dynamic vacuum, the electric breakdown was 1590 volts (average of ten values); and after 21 days, this had risen to 1950 volts. The final value of 1927 volts, after 28 days, indicated that an upper limit had been reached.

The above results were subjected to an analysis of variance. The standard F-test was used to show that there was, indeed, a significant increase in the electric strength over the 28-day period. Several low values were also obtained, between 400 and 600 volts. These occurred when a fold or crease in the film was between the electrodes.

Mass-spectrometric analysis of the volatile contents of the seven-day-aged tubes indicated that these gases contained a large number of very small molecules, many of which were highly fluorinated. Some of the smaller fragments identified were H_2, CH_4, C_2F_3, and $C_3F_3H_3$. The tubes were badly etched, and the formation of silicon tetrafluoride indicated that HF had been liberated.

DISCUSSION

When poly(vinyl fluoride) film is heated to 145°C in a continuous vacuum, there is an initial increase in the electric strength of the film. However, it is our experience that many systems exhibit a similar initial increase in electrical properties but that further aging causes a deterioration of these properties.

In a static vacuum, the small amount of thermal degradation apparently induces autocatalytic decomposition which completely degrades the film, destroying its useful properties. This, then, precludes the use of poly(vinyl fluoride) in capactive devices designed for use at temperatures above the recommended 107°C, where the dielectric is enclosed in any manner which would prevent the escape of evolved gases.

Reference

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