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HIGH PERFORMANCE POLYMER FILM DIELECTRICS FOR AIR FORCE WIDE-TEMPERATURE POWER ELECTRONICS APPLICATIONS (Preprint)

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The focus of this research is the generation and dielectric evaluation, up to 350 ºC, of metallized free-standing thin films derived from high temperature polymer systems such as fluorinated polybenzoxazoles (6F-PBO) and fluorenyl polyesters incorporating diamond-like hydrocarbon units (FDAPE). The discussion will be centered mainly on variable temperature dielectric measurements of film capacitance, dissipation factor and insulation resistance, and the effects of thermal cycling on film dielectric performance. Initial studies clearly point to the dielectric stability of these films for high temperature power conditioning applications, as indicated by relatively minor variations (~ 2 %) in measured film capacitance over the entire range of temperatures studied. Comparison will also be made with the reported variable temperature dielectric properties of the state-of-the-art high temperature FPE films.

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High Performance Polymer Film Dielectrics for Air Force Wide-Temperature Power Electronics Applications

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ABSTRACT

Air Force currently has a strong need for the development of compact capacitors which are mechanically robust and thermally stable for operation in a variety of aerospace power conditioning applications. These applications demand better reliability and flexibility in the power system design as well as decreased thermal load for electronic system cooling. While power conditioning capacitors typically use polycarbonate (PC) dielectric films in wound capacitors for operation from -55ºC to 125ºC, future power electronic systems would require the use of polymer dielectrics that can reliably operate at elevated temperatures up to or even exceeding 350ºC.

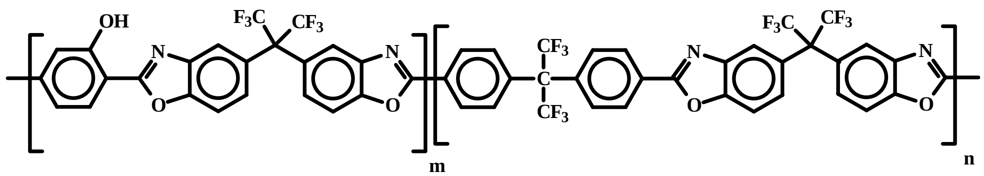
The focus of this research is the generation and dielectric evaluation, up to 350ºC, of metallized free-standing thin films derived from high temperature polymer systems such as fluorinated polybenzoxazoles (6F-PBO) and fluorenyl polyesters incorporating diamond-like hydrocarbon units (FDAPE). The discussion will be centered mainly on variable temperature dielectric measurements of film capacitance, dissipation factor and insulation resistance and the effects of thermal cycling on film dielectric performance. Initial studies clearly point to the dielectric stability of these films for high temperature power conditioning applications, as indicated by relatively minor variations (~ 2 %) in measured film capacitance over the entire range of temperatures studied. Comparison will also be made with the reported variable temperature dielectric properties of the state-of-the-art high temperature FPE films.

INTRODUCTION

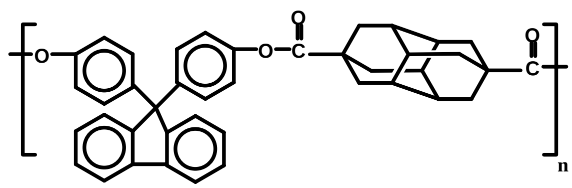
The general inadequacy of Commercial-Off-The-Shelf (COTS) capacitors in meeting the challenges of high temperature aerospace power electronic applications renders it necessary to design a new generation of dielectric materials for the development of high energy-density storage devices. Such applications demand better reliability and flexibility in the power system design as well as decreased thermal load for electronic system cooling. The demand for stable dielectric performance over a large temperature range arises from the closer proximity of aircraft power electronics to heat sources involving turbine engines, generators and motors. The primary focus of the Air Force high temperature dielectric film research is geared towards the development of compact capacitors which are mechanically robust and thermally stable for wide temperature aerospace and avionic power conditioning capacitor applications [1]. Polymer dielectrics comprise a class of materials for such wide-temperature capacitor applications because of their potential for high dielectric breakdown strengths, low dissipation factors and good dielectric stability over a wide range of frequencies and temperatures; their inherent drawback is that they have lower dielectric constants relative to inorganics and ceramics. Besides, they are also amenable to large area fabrication into films at a relatively lower cost. An added advantage of a metallized polymer film capacitor design is its self-healing capability [2, 3] that ensures a graceful rather than a catastrophic failure mechanism which is generally not the case with pure ceramic capacitors.

Aerospace power conditioning applications typically use polycarbonate (PC) dielectric films in wound capacitors for operation in the temperature range of -55ºC to 125ºC. For higher operating temperatures up to 200ºC, capacitors incorporating semi-crystalline poly(p-phenylenesulfide) (PPS) films have been evaluated because of their low dissipation factor and good dielectric strengths approximating 500 V/µm [4]. Recently, an amorphous high temperature fluorenyl copolyester (FPE) has been evaluated for space power conditioning [5] and is also being touted as a potential replacement for PC in aerospace power conditioning applications because of its relatively high dielectric strength (400 V/µm) as a thin film, a dielectric constant of 3.3 and a low dissipation factor of 0.03 % at 1 kHz and an operating temperature exceeding 250ºC. However, its cost and even more stringent (≥350ºC) thermal endurance requirements for future aerospace power conditioning capacitor applications necessitate further research in the area of high temperature polymer dielectrics. Enhanced energy dissipation occurring at higher operating temperatures can result in considerable heat rise within the dielectric, which, in turn, might cause dielectric degradation due to thermal runaway. It is also reasonable to assume that polymer dielectrics with high glass transition temperatures and high thermal/thermo-oxidative stability will play a significant role in providing electro-mechanical stability for wide-temperature power electronics applications.

As part of our capacitor research program toward meeting the requirements of dielectric stability and reliability in capacitor devices at temperatures as high as 350°C, we report herein the film fabrication and variable temperature dielectric testing of some high performance polymers synthesized at AFRL. The candidate high temperature polymers evaluated in this program are based on fluorinated polybenzoxazoles [6] as well as cardo-type (fluorenyl) polyesters incorporating diamond-like hydrocarbon structural units [7, 8] in the polymer backbone. The chemical structures of the polymer dielectrics examined in this study are shown below in Figure 1.



OH-6F-PBO-12F-PBO copolymer



FDAPE

Figure 1. Polymers for variable temperature dielectric testing in the current study

The fluorinated polybenzoxazole is a 1:1 random copolymer consisting of a hydroxyphenyl-6F-PBO unit as well as a 12F-PBO unit (referred to as OH-6F-PBO/12F-PBO copolymer) in this paper. FDAPE refers to a fluorenyl polyester incorporating a diamond-like hydrocarbon, i.e., 4,9-diamantyl unit in the polymer backbone.

EXPERIMENTAL

(a) Synthesis and general characterization of the polymers

The detailed synthesis of the fluorinated polybenzoxazoles has been described elsewhere in the context of micro-electronics applications [6]. The synthesis and characterization of the cardo-type polyester FDAPE has also been reported in earlier AFRL studies [7, 8]. The glass transition temperatures were determined by Differential Scanning Calorimetry (DSC) for the powder samples and by DMA (Dynamic Mechanical Analyzer) and TMA (Thermal Mechanical Analyzer) for the polymer films. The thermal and thermo-oxidative stabilities of the polymer were determined by Thermo-Gravimetric Analysis (TGA).

(b) Thin film fabrication

In all the cases, thin, unsupported (freestanding) films were fabricated by solvent casting. The 6F-benzoxazole copolymer film was cast from tetrahydrofuran (THF). FDAPE films were cast from chloroform. After slow solvent evaporation from the flat glass casting dishes placed inside a desiccator, the thin films were carefully isolated by the addition of a non-solvent such as de-ionized, distilled water. The films were dried at ~ 0.1 torr vacuum in an oven for several days at 30ºC. Circular films varying in diameter from 2” to 4” were fabricated and their film thickness was measured to be in the range of 3 μm to 8 μm by the thin film measurement system MP-100S (Mission Peak optics Inc).

(c) Metallized single film (M-I-M) device fabrication

Aluminum top and bottom electrodes with 100 nm thickness were deposited on both sides of the freestanding films using a thermal evaporator system. For small (2" diameter) dielectric films, circular electrical contacts with 3 mm diameter were deposited. In the case of larger polymer films (4" diameter), electrical contacts with 2 cm^2 area were deposited.

(d) Dielectric testing of metallized films

Film capacitance and dissipation factor were measured using Agilent HP4284A LCR characterization system at frequencies ranging from 20 Hz to 1 MHz with a 40 V DC Bias and 1 V AC applied voltage. Variable temperature LCR measurements were conducted in a high temperature test station with operating temperature capability up to 1000ºC. The test station has a 100V/10A power supply, a temperature controller as well as a vacuum controller. A vacuum of < 1 μ torr is achieved with a combination of a turbo pump and a scroll pump system. The sample probing is accomplished with 3-axis molybdenum probing rod test fixture. The system has also been modified with a 2nd thermocouple to monitor the sample temperature independent of the hot stage temperature. The dielectric measurements on the metallized films were conducted up to maximum temperatures in the 250-350ºC range in increments of 50ºC from room temperature. The film dielectric properties were monitored over a complete (ramp up and ramp down) thermal cycle and in some cases, over two complete thermal cycles.

Film insulation resistance (IR) measurements were also taken in the high temperature test station using a Keithley 6517A Electrometer/High Resistance Meter. There was a 100K-Ohm resistor in series with the sample to protect the electrometer input in case of voltage breakdown. A 40 V DC bias was applied to the sample for 60 seconds to charge it, and then the IR/leakage current was measured over a 60 s time period. Measurements were taken at 50ºC intervals from room temperature to 300-350ºC over two thermal cycles.

The breakdown strength of the metallized thin film polymer dielectric was evaluated using precision regulated high voltage power supply models Bertan 210-05 R and Spellman SR6 with operating voltage capability up to 5 kV and 30 kV respectively. The current was measured using a Hewlet Packard 4349A 4-Channel High Resistance Meter. There was a 200 Mega-Ohm resistor in series with sample to protect the meter input during voltage breakdown. The DC voltage was ramped at 50 volt intervals until breakdown.

RESULTS AND DISCUSSION

Besides the potential for electro-mechanical stability at high temperatures due to their high glass transition temperatures and thermal stability, some previously established criteria for micro-electronic packaging applications of fluorinated polybenzoxazoles [6] are equally relevant for their dielectric applications in wide-temperature power electronics. The incorporation of a diamond-like hydrocarbon unit in the fluorenyl polyester backbone (7, 8) (FDAPE) is expected to enhance its insulation resistance and film dielectric strength based on the rationale that the HOMO-LUMO band gaps in diamondoids are molecular counterparts of a large fundamental band gap in diamond itself [9].

(a) Thermal properties of the polymer systems

High glass transition temperatures (Tg) and thermal stability of the polymer dielectrics are an important prerequisite for electro-mechanical stability of the dielectrics utilized in power conditioning capacitor systems. Table 1 lists the T_gs and the thermal degradation (T_d) in nitrogen as well as in air, of the polymers under study.

Table 1. Thermal properties of the polymer dielectrics evaluated in the study

Polymer	Glass Transition Temperature (T_g, ºC)*	Thermal Degradation (T_d, ºC), Onset	
		In nitrogen	In air
OH-6F-PBO/12F-PBO copolymer	362	525	500
FDAPE	>400**	450	380

* By DSC unless otherwise mentioned
 **Not detected in DSC and Modulated DSC up to 400ºC but the DMA of the film showed a Tg of ~ 400ºC.

Figure 2 is illustrative of the thermal stability of the OH-6F-PBO/12F-PBO copolymer, showing the onset of degradation occurring at temperatures > 500°C in both nitrogen and air.

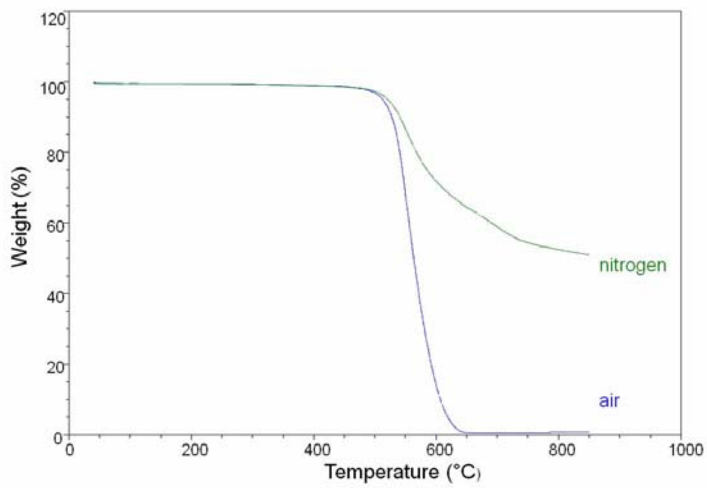


Figure 2. TGA traces of OH-6F-PBO/12F- PBO copolymer

(b) Dielectric film characterization

The solvent cast thin dielectric films were optically transparent, ductile and displayed good mechanical integrity. After metallization, the variable temperature dielectric properties of the films were characterized. Among the screening tests performed to assess the dielectric stability of the metallized polymer films, the most important measurements were those of film capacitance and dissipation factor, from room temperature up to a maximum temperature of 350ºC. The stability of the dielectric constant with temperature is defined by the temperature coefficient of capacitance (TCC) in the required range. The maximum temperature for testing was determined by the glass transition temperature (T_g) of the polymer which would affect the dimensional as well as the electro-mechanical stability of the dielectric. Typically, these films were tested up to temperatures 50°C below their T_gs and in some cases, close to their Tgs as well. Some breakdown voltage testing results are also reported for the metalized thin films. Most of the results discussed in this paper are based on dielectric measurements on metallized polymer films with 3 mm circular Al electrodes. The observations were also corroborated by the dielectric performance testing of metallized films with larger (2 cm^2) electrode areas. Representative single film (M-I-M) device structures with FDAPE as the dielectric are shown in Figure 3.

Figure 3. Single film device structures of FDAPE with circular Al contacts

(c) Metallized fluorinated polybenzoxazole copolymer film dielectric characterization

The initial variable temperature dielectric measurements were conducted up to 250°C in the high temperature LCR test station. Figure 4 shows measured capacitance (in Farads) as a function of frequency over RT-250ºC temperature range in increments of 50ºC during the ramp up cycle. Figure 5 depicts the corresponding % change in capacitance at the measurement temperature relative to the room temperature value (ΔC/C).

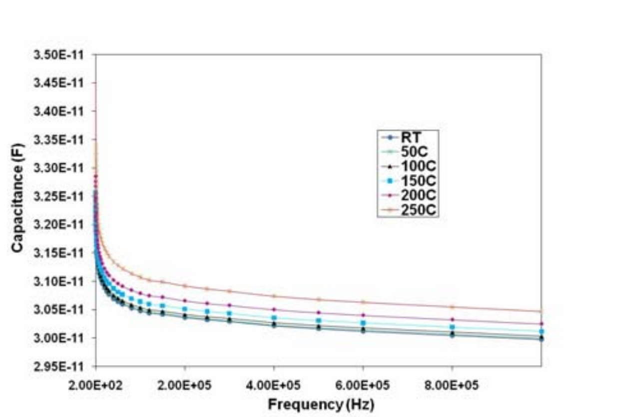


Figure 4. Variable temperature film capacitance of OH-6F-PBO/12-F PBO copolymer

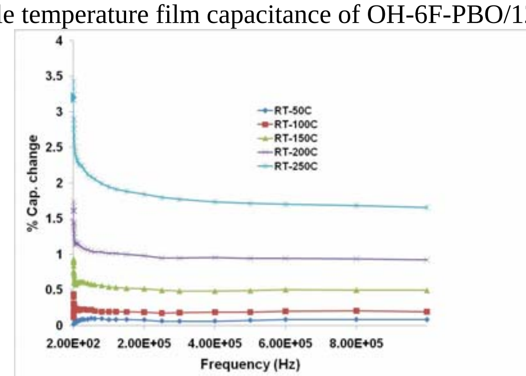


Figure 5. Capacitance change (%) as a function of temperature for OH-6F-PBO/12F-PBO copolymer film

Small incremental changes in film capacitance were measured as a function of temperature and the steady capacitance value over a large frequency range was found to increase by ~ 2 % in the RT-250ºC range. This corresponds to a temperature coefficient of capacitance of ~ 88 ppm/ºC, indicating stable capacitance over a wide temperature range. Capacitance (in Farads) measured over a complete heating (ramp up, RU) and a cooling cycle (ramp down, RD) at the frequency of interest (10 kHz) for power conditioning capacitor applications shows a slight hysteresis effect (Figure 6). Effect of thermal history as well as dimensional changes measured by film CTE (coefficient of thermal expansion) in the temperature range of RT-250ºC need to be considered in explaining the small, observed changes in film capacitance. A room temperature dielectric constant of 2.95 at 10 kHz was derived for the copolymer film.

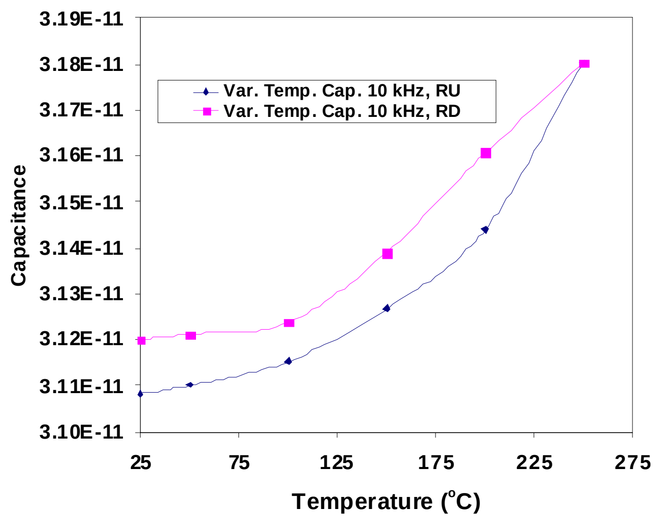


Figure 6. Capacitance in Farads (measured at 10 kHz frequency) as a function of thermal cycling for the OH-6F-PBO/12F-PBO copolymer film (RU and RD refer to ramp up and ramp down during one complete thermal cycle).

The dielectric properties of a metallized fluorinated polybenzoxazole film were also evaluated up to 350°C. Changes in film capacitance at 10 kHz, expressed in picofarads (pF), were monitored over two complete thermal cycles (Figure 7). The corresponding variations in film dissipation factor (expressed as %) were also monitored (Figure 8). While a marked increase in measured film capacitance and dissipation factor were indicated at 350°C, consistent reversion to lower values occurred during the cooling cycles, showing that no degradation of the dielectric had occurred due to thermal cycling in the RT-350°C range.

The sharp variations in the film dielectric properties measured at 350°C are presumably due to the operating temperature being close to the glass transition temperature, which might cause dimensional changes in the dielectric.

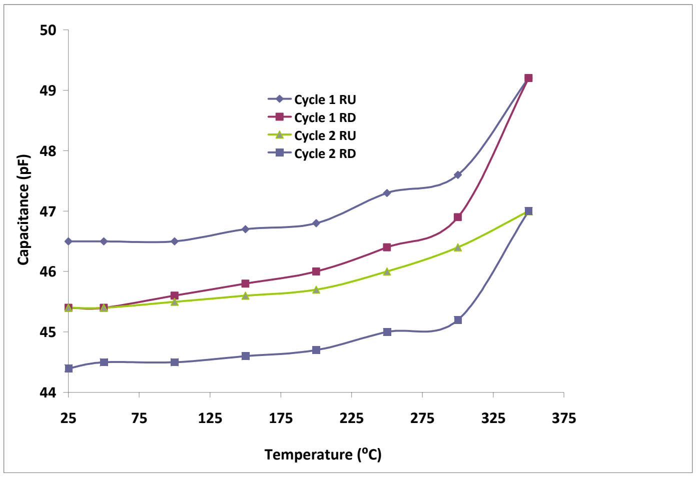


Figure 7. Film capacitance (10 kHz) over two complete thermal cycles up to 350°C for the OH-6F-PBO/12F-PBO copolymer

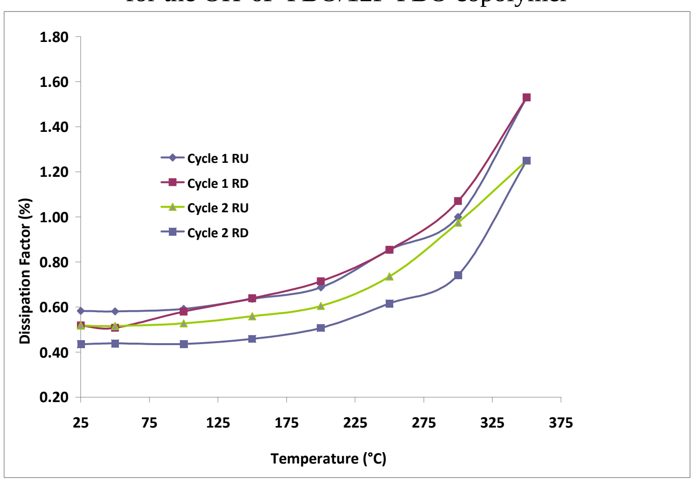


Figure 8. Film Dissipation Factor (10 kHz) measured over two complete thermal cycles up to 350°C for the OH-6F-PBO/12F-PBO copolymer

The leakage current of a fluorinated polybenzoxazole with 2 sq.cm deposited Aluminum contacts was also measured as a function of temperature in the RT-300ºC range, using 40 V DC as the test voltage. The derived insulation resistance, at 60 s acquisition time, was monitored over two complete thermal cycles and the results are shown in Figure 9. The insulation resistance (in ohms) was found to change by nearly two orders of magnitude in the RT-300ºC range and was ~ 18,500 Mega-ohms at 300ºC.

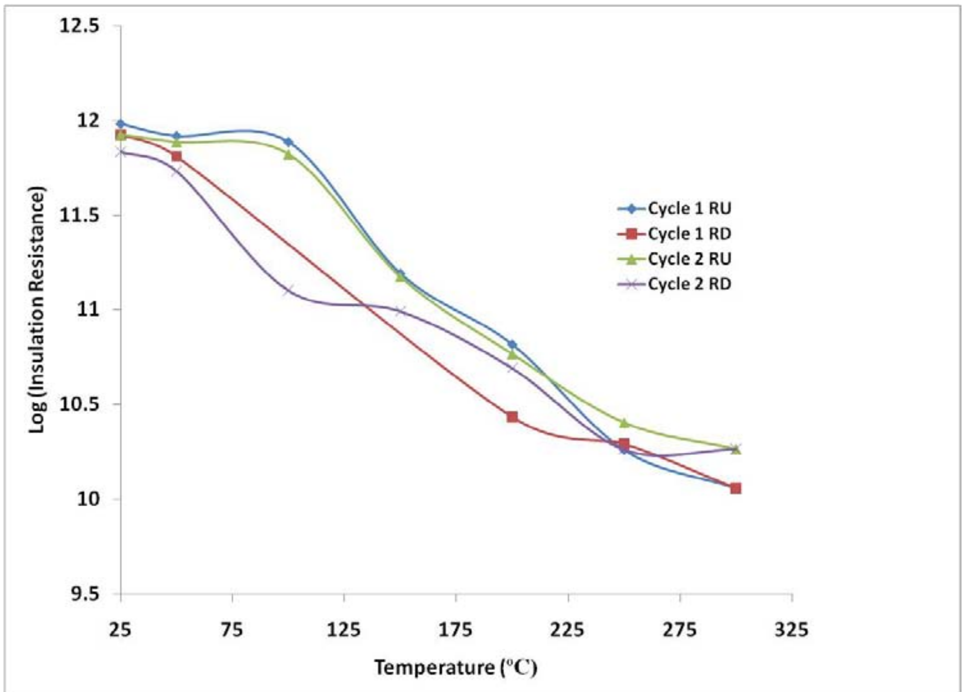


Figure 9. Insulation resistance of a metalized OH-6F-PBO/12F-PBO copolymer film as a function of thermal cycling in the RT-300ºC range

(d) Metallized fluorenyl polyester with 4,9-diamantyl unit (FDAPE) -film dielectric characterization

Minimal to negligible film capacitance changes were observed in the RT-350°C range for the FDAPE film. While the first heating and cooling cycles in this temperature range resulted in fluctuations of 1.9 % to 1.5 % in the capacitance values, the second heating and cooling cycles were found to cause even lower (0.8 % and 0.4 %) fluctuations in the measured capacitance in the entire temperature range. The dielectric stability of the FDAPE film in this temperature range was also reflected in the derived values of the dielectric constant of the film (Figure 10). Relative permittivity as a function of temperature during the second complete thermal cycle is shown in the figure and has a narrow range of 3.48-3.51 over the entire temperature range.

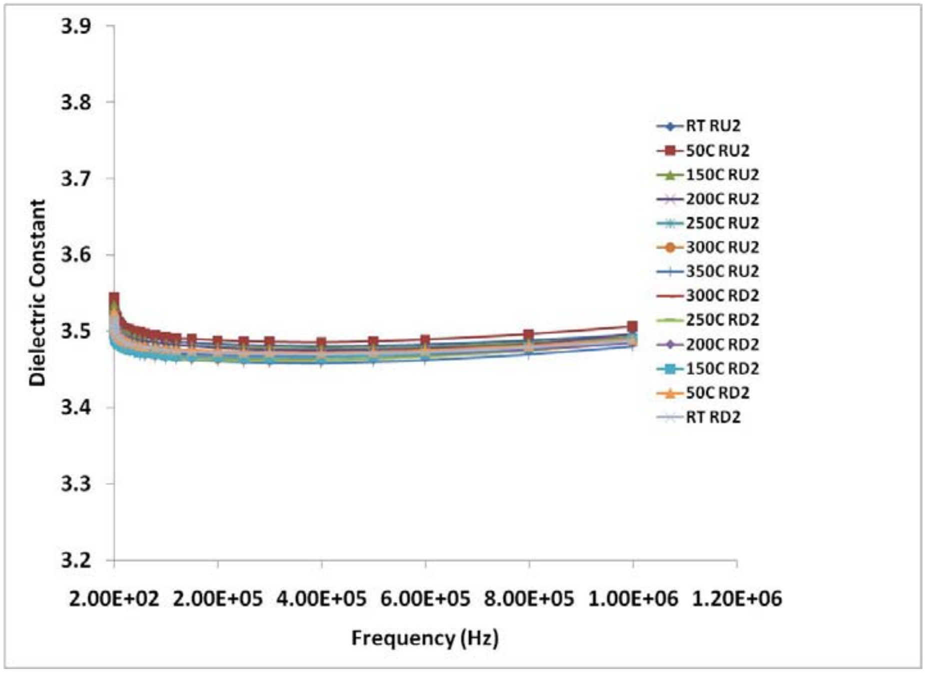


Figure 10. Variations in the dielectric constant of FDAPE film during the second complete thermal cycle (RU2 and RD2 refer to thermal ramp up and ramp down during the cycle)

The change in film capacitance in picofarads, measured at a constant frequency of 10 kHz over two complete thermal cycles is shown in Figure 11. Figure 12 shows the variable temperature cycling measurements of the dissipation factor (%) of the metallized FDAPE film up to a maximum temperature of 350°C.

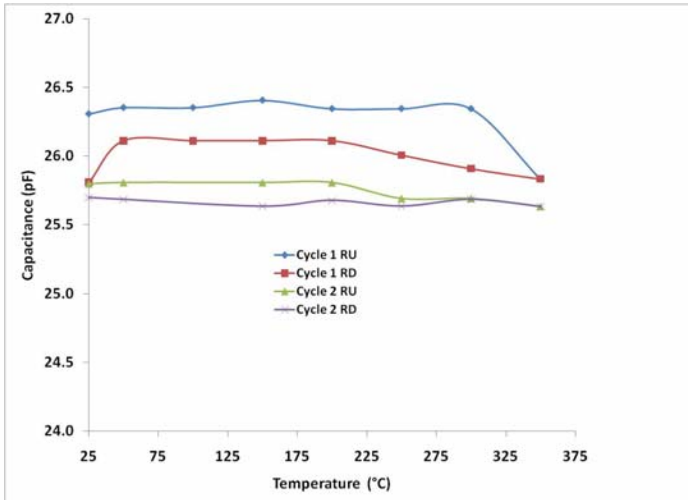


Figure 11. Film capacitance (10 kHz) over two complete thermal cycles up to 350°C for the FDAPE polymer

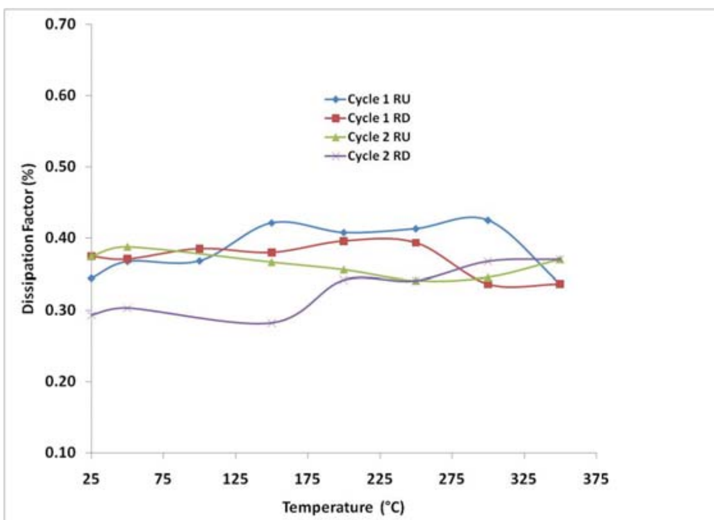


Figure 12. Film dissipation factor (10 kHz) over two thermal cycles up to 350°C for the FDAPE polymer

The FDAPE film capacitance has a maximum variation of 2 % occurring over the first thermal cycle and has even less of a variation (< 1%) during the second complete thermal cycle. While the overall changes during thermal cycling were small, the dissipation factor was down to < 0.003 (0.3 %) measured at room temperature after the second ramp down experiment. Ideally, dissipation factors of ≤ 0.1 % are desired for µs discharge rates in power conditioning capacitors. The dielectric stability of the metalized FDAPE film at temperatures as high as 350°C is borne out by these observations. The stability of film capacitance at 350°C can be presumably attributed to the chemical as well as dimensional stability of the polymer dielectric operating at temperatures well below its glass transition temperature.

The variations in the insulation resistance (in ohms) of the metalized FDAPE dielectric with 3 mm Aluminum contacts were monitored over two complete thermal cycles in the RT-350ºC range and are depicted in Figure 13. It was shown that the insulation resistance effectively decreased by just an order of magnitude relative to the room temperature value when the film was heated to 350ºC and was of the order of 10^7 Mega-ohms at 350ºC.

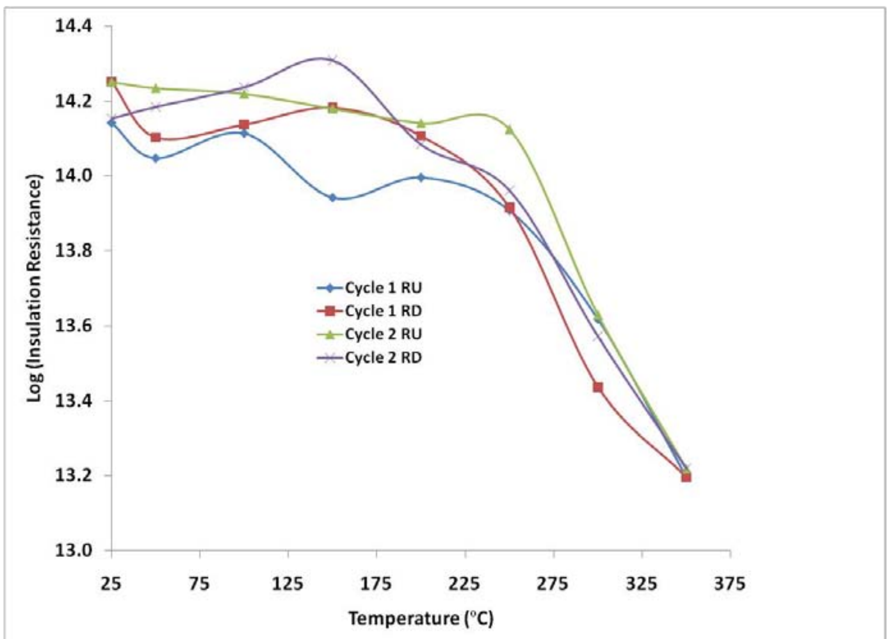


Figure 13. Insulation resistance of a metalized FDAPE film as a function of thermal cycling in the RT-350ºC range

(e) Film Breakdown strength measurements

While the main focus of the study was the evaluation of variable temperature dielectric stability of polymeric materials for wide-temperature capacitor applications, some preliminary high voltage (DC) dielectric breakdown measurements were performed on the films after thermal cycling. High dielectric breakdown strengths are desired for capacitive energy storage because of the quadratic dependence of stored energy density on the film breakdown voltage (BDV). Typical electrical breakdown behavior of the metallized FDAPE film is shown as current-voltage
(I-V) plots in Figure 14. Based on an average measured film thickness of 8 μ, the measured breakdown voltage ranged from 200-300 V/μ. However, optimization of the solvent –based film fabrication process can lead to potentially much higher film breakdown strengths for FDAPE.

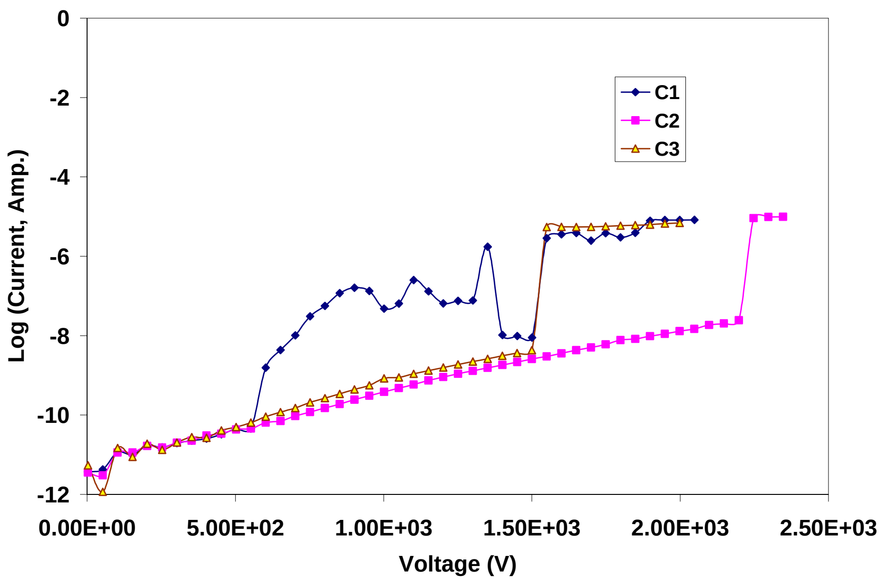


Figure 14. Voltage breakdown testing of a metalized FDAPE film (C1, C2 and C3 are the three capacitor areas tested)

(f) Comparison with FPE film

FDAPE belongs to the same class of amorphous polyesters as FPE and is a structural variant with a higher glass transition temperature (> 400ºC) compared to FPE, which has a Tg of 330ºC. Their thermal/thermo-oxidative stabilities and their solvent processing characteristics for thin film fabrication are also about the same. FPE has been established as a dielectric with stable performance in the -55ºC-275ºC range; the dielectric stability of the FDAPE film up to 350ºC, demonstrated in this study, points to its initial potential as a polymer dielectric with an operating temperature even higher than that of FPE. However, process optimization for the fabrication of continuous, large area, defect-free thin films similar to FPE would be required for enhancing its dielectric performance in terms of higher film dielectric strength and even lower dissipation factor.

SUMMARY

High temperature polymer dielectric films based on fluorinated polybenzoxazole copolymer and fluorenyl polyester (FDAPE) have been investigated with regard to their potential utilization in wide-temperature power electronics applications. Variable temperature film dielectric studies have revealed that while the fluorinated polybenzoxazole copolymer shows a low temperature coefficient of capacitance (88 ppm/°C) in the RT-250°C range, it is subject to marked increases in both capacitance and dissipation factor at temperatures close to 350°C. However, consistent reversion to lower values occurred during the cooling cycles, showing that no degradation of the polymer dielectric had occurred due to thermal cycling in the RT-350°C range. Results from variable temperature dielectric evaluation of FDAPE in the RT-350°C range show that small to negligible changes in film capacitance have occurred as a function of thermal cycling, indicating dielectric stability at high temperatures. The measured value of the film dissipation factor at 10 kHz as a function of thermal cycling was in the range of 0.3-0.4%. The dielectric constants of the high temperature fluorinated polybenzoxazole and FDAPE films were 2.95 and 3.5 respectively at 10 kHz. Variable temperature IR measurements up to 300ºC and 350ºC further confirmed the stability of these polymer films as high temperature insulating dielectrics. These studies indicate the initial potential of these high performance polymer dielectrics for AF wide-temperature power electronics applications.

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