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**HIGH TEMPERATURE POLYMER FILM DIELECTRICS FOR
AEROSPACE POWER CONDITIONING CAPACITOR
APPLICATIONS (Postprint)**

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14. ABSTRACT Polymer dielectrics are the preferred materials of choice for capacitive energy-storage 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, despite having inherently lower dielectric constants relative to ceramic dielectrics. They are also amenable to large area processing into films at a relatively lower cost. Air Force currently has a strong need for the development of compact capacitors which are thermally robust for operation in a variety of aerospace power conditioning applications. While such applications typically use polycarbonate (PC) dielectric films in wound capacitors for operation from -55 °C to 125 °C, future power electronic systems would require the use of polymer dielectrics that can reliably operate up to elevated temperatures in the range of 250–350 °C. The focus of this research is the generation and dielectric evaluation of metallized, thin free-standing films derived from high temperature polymer structures such as fluorinated polybenzoxazoles, post-functionalized fluorinated polyimides and fluorenyl polyesters incorporating diamond-like hydrocarbon units. The discussion is centered mainly on variable temperature dielectric measurements of film capacitance and dissipation factor and the effects of thermal cycling, up to a maximum temperature of 350 °C, on film dielectric performance. Initial studies clearly point to the dielectric stability of these films for high temperature power conditioning applications, as indicated by their relatively low temperature coefficient of capacitance (TCC) (~2%) over the entire range of temperatures. Some of the films were also found to exhibit good dielectric breakdown strengths (up to 470 V/μm) and a film dissipation factor of the order of <0.003 (0.3%) at the frequency of interest (10 kHz) for the intended applications. The measured relative dielectric permittivities of these high temperature polymer films were in the range of 2.9–3.5.					
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High temperature polymer film dielectrics for aerospace power conditioning capacitor applications

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ABSTRACT

Polymer dielectrics are the preferred materials of choice for capacitive energy-storage 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, despite having inherently lower dielectric constants relative to ceramic dielectrics. They are also amenable to large area processing into films at a relatively lower cost. Air Force currently has a strong need for the development of compact capacitors which are thermally robust for operation in a variety of aerospace power conditioning applications. While such applications typically use polycarbonate (PC) dielectric films in wound capacitors for operation from -55°C to 125°C , future power electronic systems would require the use of polymer dielectrics that can reliably operate up to elevated temperatures in the range of 250 – 350°C . The focus of this research is the generation and dielectric evaluation of metallized, thin free-standing films derived from high temperature polymer structures such as fluorinated polybenzoxazoles, post-functionalized fluorinated polyimides and fluorenyl polyesters incorporating diamond-like hydrocarbon units. The discussion is centered mainly on variable temperature dielectric measurements of film capacitance and dissipation factor and the effects of thermal cycling, up to a maximum temperature of 350°C , on film dielectric performance. Initial studies clearly point to the dielectric stability of these films for high temperature power conditioning applications, as indicated by their relatively low temperature coefficient of capacitance (TCC) ($\sim 2\%$) over the entire range of temperatures. Some of the films were also found to exhibit good dielectric breakdown strengths (up to $470\text{ V}/\mu\text{m}$) and a film dissipation factor of the order of <0.003 (0.3%) at the frequency of interest (10 kHz) for the intended applications. The measured relative dielectric permittivities of these high temperature polymer films were in the range of 2.9–3.5.

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1. Introduction

The inadequacy of Commercial-Off-The-Shelf (COTS) capacitors in meeting the challenges of high temperature aerospace power electronics applications renders it absolutely necessary to design a new generation of dielectric materials for the development of capacitive energy-storage devices. These applications require better reliability and flexibility in the power system design as well as high temperature stability to lower the demand for the cooling system. The increasing 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 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 are the preferred materials of choice for such capacitor applications because of their potential for high breakdown strengths, low dissipation factors (dielectric loss factors) and good dielectric stability over a wide range of frequencies and temperatures, despite having inherently lower dielectric constants relative to ceramic capacitors. Besides, they are also amenable to large area processing into films at a relatively lower cost. An added advantage of a metallized polymer film capacitor design is its 'self-healing' or 'clearing' capability [2,3] that facilitates a graceful 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

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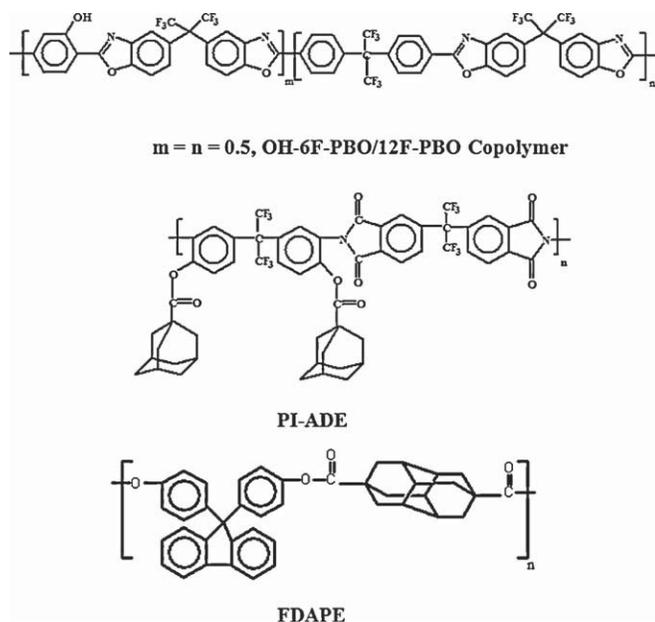


Fig. 1. High temperature polymer dielectrics evaluated in this study.

from -55°C to 125°C . For higher operating temperatures up to 200°C , capacitors incorporating poly(*p*-phenylenesulfide) (PPS) films have been evaluated because of their low dissipation factor and dielectric strengths approximating $500\text{ V}/\mu\text{m}$ [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; besides an operating temperature capability up to or exceeding 250°C , it is reported to have a relatively high dielectric strength ($400\text{ V}/\mu\text{m}$) as a thin film, a dielectric constant of 3.3 and a low dissipation factor of 0.0003 (0.03%) at 1 kHz. However, its cost and current, even more stringent ($\sim 350^{\circ}\text{C}$) thermal management requirements for future aerospace power conditioning capacitor applications necessitate further research in the area of high temperature polymer dielectrics. High energy dissipation occurring at higher temperatures can result in considerable heat rise within the dielectric, which, in turn, might cause dielectric degradation due to thermal runaway. Thus, it is reasonable to assume that polymer dielectrics with high glass transition temperatures and high thermal 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 at high temperatures, we report herein the film fabrication and variable temperature dielectric evaluation of some high performance polymers synthesized in our laboratories. The candidate high temperature polymers evaluated in this program are based on fluorinated polybenzoxazoles and polyimides as well as cardo-type (fluorenyl) polyesters incorporating diamond-like hydrocarbon structural units in the polymer backbone. The structures of the polymers are depicted in Fig. 1.

The fluorinated polybenzoxazole is a 1:1 random copolymer consisting of a hydroxyphenyl-6F-PBO unit as well as a 12F-PBO unit (OH-6F-PBO/12F-PBO copolymer). PI-ADE refers to a fluorinated polyimide post-functionalized with an adamantyl ester pendant and FDAPE refers to a fluorenyl polyester incorporating another diamond-like hydrocarbon group, i.e., 4,9-diamantyl unit in the polymer backbone. These polymers have high glass transition temperatures as well as good thermal stabilities and fulfill the

requirements for potential evaluation in wide-temperature power electronics applications of interest to the Air Force.

2. Experimental

2.1. Synthesis and general characterization of the polymers

The detailed synthesis of the fluorinated polybenzoxazoles has been described elsewhere [6]. PI-ADE was synthesized from the post-polymer reaction of a hydroxy polyimide with adamantane-1-carboxylic acid chloride [7]. The synthesis and characterization of the fluorenyl polyester FDAPE have also been reported in our earlier studies [8,9]. The glass transition temperatures of the solvent-cast polymer films were determined by DMA (Dynamic Mechanical Analysis). The thermal and thermo-oxidative stabilities of the polymer films were determined by Thermo-Gravimetric Analysis (TGA). The DMA and TGA measurements were performed on relatively thick ($\sim 90\ \mu\text{m}$) solution-cast polymer films.

2.2. Thin film fabrication

In all the cases, free-standing films were fabricated by solvent casting. The 6F-benzoxazole copolymer film was cast from tetrahydrofuran (THF). FDAPE films were cast from chloroform. PI-ADE was cast from *N,N*-dimethylacetamide (DMAc) as solvent, *in vacuo*, from a vacuum desiccator over 2 days. The thin films were carefully isolated from flat glass casting dishes 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 $65\text{--}75^{\circ}\text{C}$. Thin circular films varying in diameter from 2 in. to 4 in. were fabricated and their film thickness was measured to be in the range of $3\text{--}8\ \mu\text{m}$ by the thin film measurement system MP-100S (Mission Peak Optics Inc.).

2.3. Metallized single film device fabrication

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

2.4. Dielectric testing of metallized films

Film capacitance and dissipation factor (DF) were measured using Agilent HP4284A LCR characterization system at frequencies ranging from 20 Hz to 1 MHz with a 1 V AC applied voltage. Variable temperature LCR measurements were conducted in a high temperature dielectric test station with operating temperature capability up to 1000°C . The test station has a 100 V/10 A power supply, a temperature controller as well as a vacuum controller. A vacuum of $<1\ \mu\text{Torr}$ is achieved with a combination of a turbo pump and a scroll pump system. The sample probing is accomplished with a three-axis molybdenum probing rod test fixture. The dielectric measurements on the metallized films were conducted up to maximum temperatures in the $250\text{--}350^{\circ}\text{C}$ range in increments of 50°C from room temperature. The film dielectric properties were monitored over a complete (ramp up (RU) and ramp down (RD)) thermal cycle in the preliminary stage of the program. However, the variable temperature dielectric properties of the polymer films were examined over two complete thermal cycles in the later stage of the investigations.

The breakdown strength of the metallized thin film polymer dielectric was evaluated using precision-regulated high voltage

Table 1

Thermal properties of the polymer films in this study.

Polymer	T_g^a (°C), N ₂	T_d^b (°C), onset, N ₂	T_d^b (°C), onset, air	T_d^b (°C), 5 wt% loss, N ₂	T_d^b (°C), 5 wt% loss, air
OH-6F-PBO/12F-PBO copolymer	375	525	500	550	520
PI-ADE	305	400	350	425	380
FDAPE	450	470	400	490	465

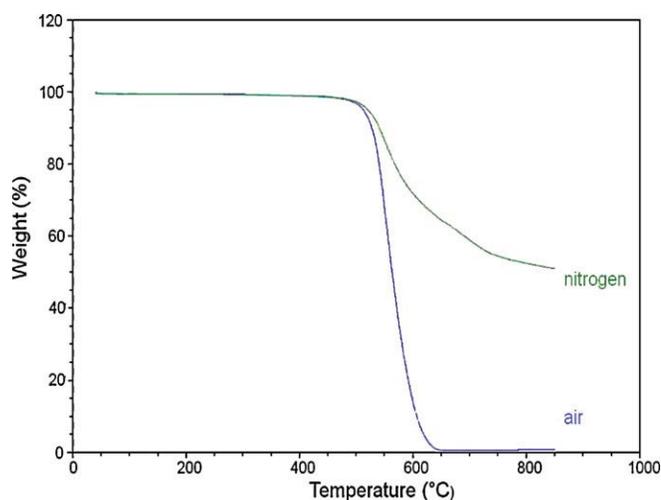
^a DMA of the film (tan δ measurement).^b Degradation temperature, TGA measurement.

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

power supply models Bertran 210-05 R and Spellman SR6 with operating voltage capability up to 5 kV and 30 kV respectively.

3. Results and discussion

Besides the potential for electro-mechanical stability at high temperatures due to their high glass transition temperatures and thermal stability, some established criteria for micro-electronic packaging applications of fluorinated polybenzoxazoles [6,10] and fluorinated polyimides [10,11] are equally important for their dielectric applications in wide-temperature power electronics. The versatility of the hydroxyl pendant in fluorinated polyimides can be

utilized to develop novel, tailorable dielectrics such as PI-ADE by post-functionalization reactions. The incorporation of a diamond-like hydrocarbon unit or diamondoid in the fluorenyl polyester backbone [8,9] (FDAPE) or as a pendant in the fluorinated polyimide (PI-ADE) structure [7] is expected to enhance film insulation resistance and dielectric strength based on the rationale that the HOMO-LUMO band gaps in diamond-like hydrocarbons are molecular counterparts of a large fundamental band gap in diamond itself [12,13].

3.1. Thermal properties of the polymer systems

High glass transition temperatures (T_g) and thermal stability of the polymer dielectrics are an important prerequisite for electro-mechanical stability of the dielectrics utilized in the power conditioning capacitor systems. Table 1 lists the T_g s and the thermal degradation (T_d) in nitrogen as well as in air, of the polymers under study. Fig. 2 is illustrative of the thermal/thermo-oxidative stability of the OH-6F-PBO/12F-PBO copolymer.

The high glass transition temperature of 450 °C for the FDAPE film can be ascribed to the presence of the relatively stiff 4,9-diamantane structural unit in the fluorenyl polyester backbone. Among the three polymer systems examined, the fluorinated benzoxazole copolymer had the highest thermal as well as thermo-oxidative stability.

3.2. Thin film fabrication for dielectric studies

As stated in Section 2, circular thin films with film thickness in the 3–8 μm range were solution-cast from solvents such as THF, chloroform and DMAc. It is recognized (as one of the referees has commented) that compared to melt processed films, solution-cast films may lead to more defects such as pin-holes. However, melt processing of polymers is not generally amenable to fabrication of very thin films, unless they are stretched in the extrusion process [14]. In addition, the high glass transition temperatures of the polymers described in the study (305–450 °C) preclude the option of melt processing the polymers into films.

3.3. Dielectric film characterization

The solvent-cast thin dielectric films were optically transparent, ductile and displayed good mechanical integrity. After metallization, the dielectric properties of the films were characterized, with regard to their variable temperature capacitance and dissipation

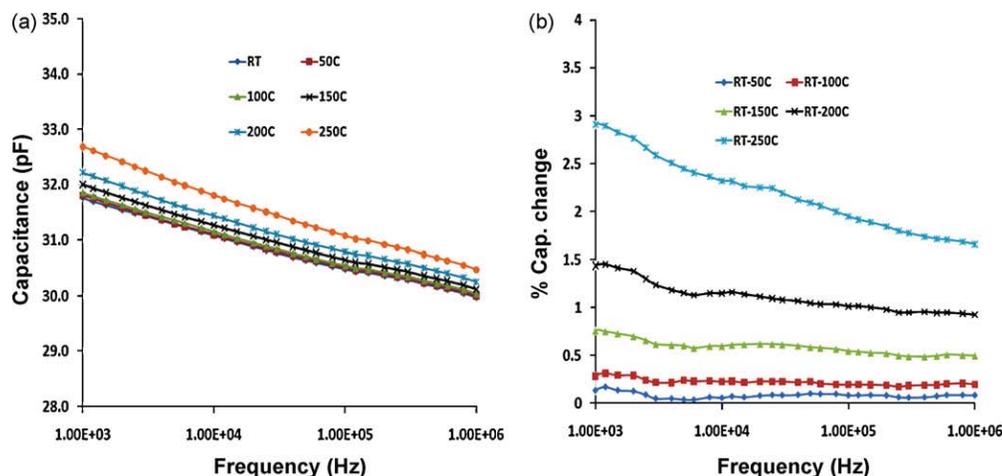


Fig. 3. (a) Variable temperature capacitance of OH-6F-PBO/12F PBO copolymer film and (b) the corresponding % variation in capacitance at different temperatures (the frequency is on logarithmic scale).

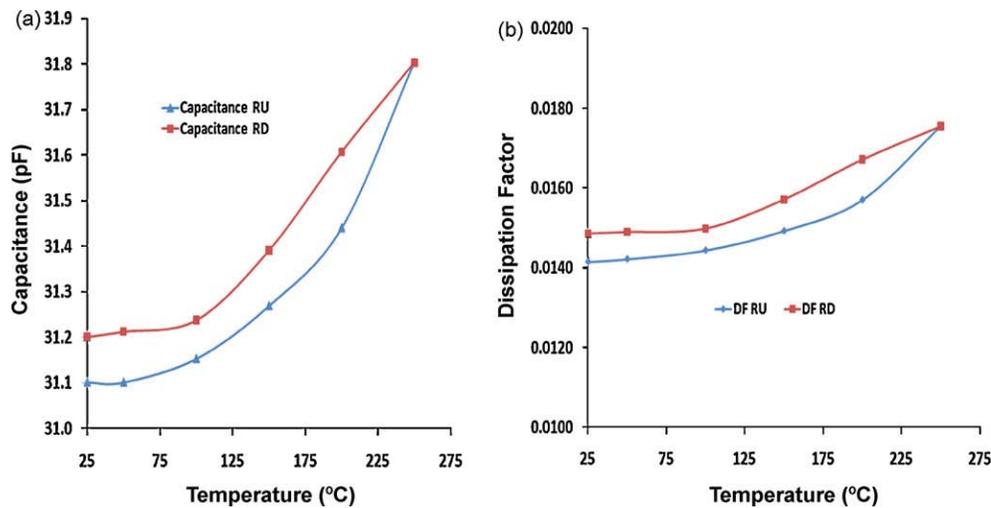


Fig. 4. (a) Film capacitance and (b) DF as a function of temperature for the OH-6F-PBO/12F-PBO copolymer at 10 kHz frequency.

factor. Room temperature DC breakdown voltage measurements were also performed on the films.

3.3.1. Variable temperature film capacitance and dissipation factor

Among the screening tests performed to assess the dielectric stability of the metallized polymer films as a function of temperature, the most important measurements were those of film capacitance and dissipation factor, from room temperature up to maximum temperatures in the range of 250–350 °C. Temperature Coefficient of Capacitance (TCC), expressed as % change in film capacitance at any given temperature relative to room temperature, is the signature of wide-temperature dielectric stability for capacitors. The maximum temperature for testing was determined by the glass transition temperature (T_g) of the polymer which would limit the dimensional and electro-mechanical stability of the dielectric. Typically, these films were tested up to temperatures which were 50–100 °C below their T_g s. The results discussed in this paper are based on dielectric measurements performed on metallized polymer films with 3 mm circular Al electrodes.

3.3.1.1. OH-6F-PBO/12F-PBO copolymer film. The metallized film was tested up to 250 °C in the high temperature LCR test station

for measuring capacitance and dissipation factor. Fig. 3(a) shows variable temperature capacitance (in pico-Farads (pF)) in 1 kHz to 1 MHz applied frequency range from RT to 250 °C in increments of 50 °C during the ramp up cycle. Fig. 3(b) depicts the corresponding % change in film capacitance at the measurement temperature relative to the room temperature value.

Small incremental changes in film capacitance were measured as a function of temperature and the capacitance value over a large frequency range was found to increase by ~2–3% at 250 °C relative to RT (Fig. 3(b)). This small change in film capacitance demonstrates the stability of the dielectric over a wide-temperature range. Capacitance measured over a complete heating (RU) and cooling cycle (RD) at the frequency of interest for power conditioning capacitor applications (10 kHz) shows a slight hysteresis effect (Fig. 4(a)) and it will be of interest, in the future, to further examine the film dielectric properties as a function of repeated thermal cycling. Thermal history as well as the film CTE (coefficient of thermal expansion) in the temperature range of RT–250 °C has to be considered in explaining the small, measured changes in film capacitance. A room temperature dielectric constant of 2.95 at 10 kHz was derived for the copolymer film from the measured capacitance.

A similar hysteresis effect is also observed during the complete thermal cycle for the measured film dissipation factor (DF)

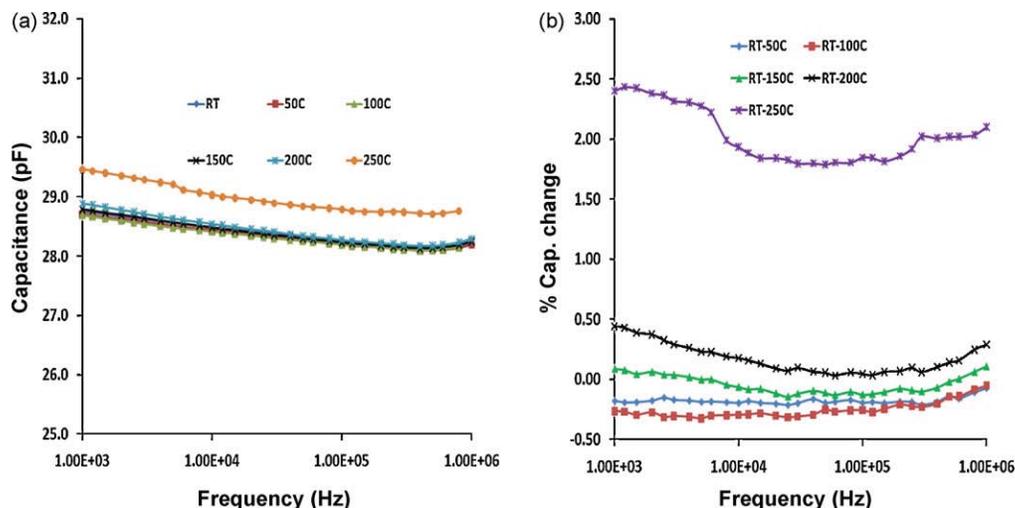


Fig. 5. (a) Variable temperature capacitance of PI-ADE film and (b) the corresponding % variation in film capacitance at different temperatures (the frequency is on logarithmic scale).

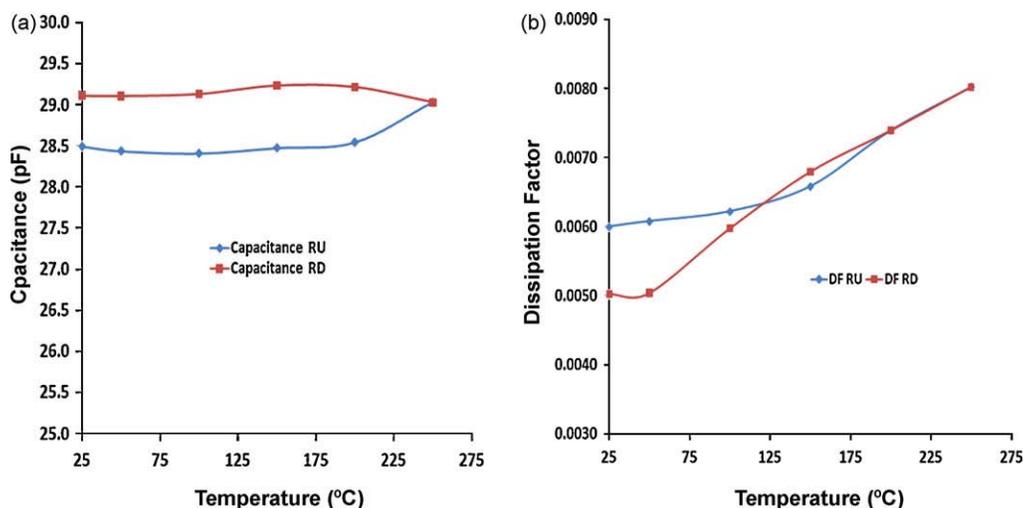


Fig. 6. (a) Film capacitance and (b) DF measured as a function of temperature for PI-ADE film at 10 kHz frequency.

(Fig. 4(b)). Over a complete thermal cycle in the RT–250 °C range, an increase in DF from 0.014 to 0.017 was noted. While a gradual increase in film dissipation factor was evident, no sharp region of increased dielectric loss was noticed since the highest temperature of measurement was well below the polymer glass transition temperature.

3.3.1.2. *PI-ADE (fluorinated polyimide with adamantyl ester pendant)*. The metallized PI-ADE film was also tested in the temperature range of RT–250 °C. The measured film capacitance at various temperatures over a range of frequencies is shown in Fig. 5(a). The high temperature dielectric stability of the film was indicated by minimal changes in the measured values of film capacitance as a function of temperature. The observed capacitance variation of ~1.8–2.5% at 250 °C relative to RT (Fig. 5(b)) is similar to that of the OH-6F-PBO/12F PBO copolymer film. A relatively sharp increase in film capacitance, observed at 250 °C, in comparison to capacitance at the lower temperatures, can be tentatively attributed to the proximity of the measurement temperature to the polymer T_g of 305 °C. Film dielectric constant was derived from measured film capacitance based on a circular electrode of 3 mm diameter and a mean dielectric film thickness of 7.6 μm

and ranged from 2.85 to 2.91 in the RT–250 °C region at 10 kHz frequency.

As in the case of OH-6F-PBO/12F-PBO copolymer, a slight hysteresis effect is observed in measured film capacitance at 10 kHz frequency (Fig. 6(a)) over a complete thermal cycle. The measured film dissipation factor at 10 kHz (Fig. 6(b)) ranged from 0.005 (0.5%) to 0.008 (0.8%) for a complete thermal cycle in the RT–250 °C range. Relatively sharp variations in film DF were found to occur at 200 °C and above.

3.3.1.3. *Fluorenyl polyester with 4,9-diamantyl group (FDAPE)*. The dielectric stability of the metallized polyester film was examined up to a maximum temperature of 350 °C over two complete thermal cycles. The measured capacitance as a function of temperature at 10 kHz frequency showed a maximum variation of 2% occurring over the first thermal cycle and had even less of a variation (<1%) during the second complete thermal cycle in the RT–350 °C range (Fig. 7(a)). The corresponding dielectric constant values were found to vary in the 3.50–3.55 range over the entire temperature span.

The film dissipation factor, measured at 10 kHz as a function of thermal cycling, is shown in Fig. 7(b). While the overall changes in DF were not significant, the room temperature dissipation factor

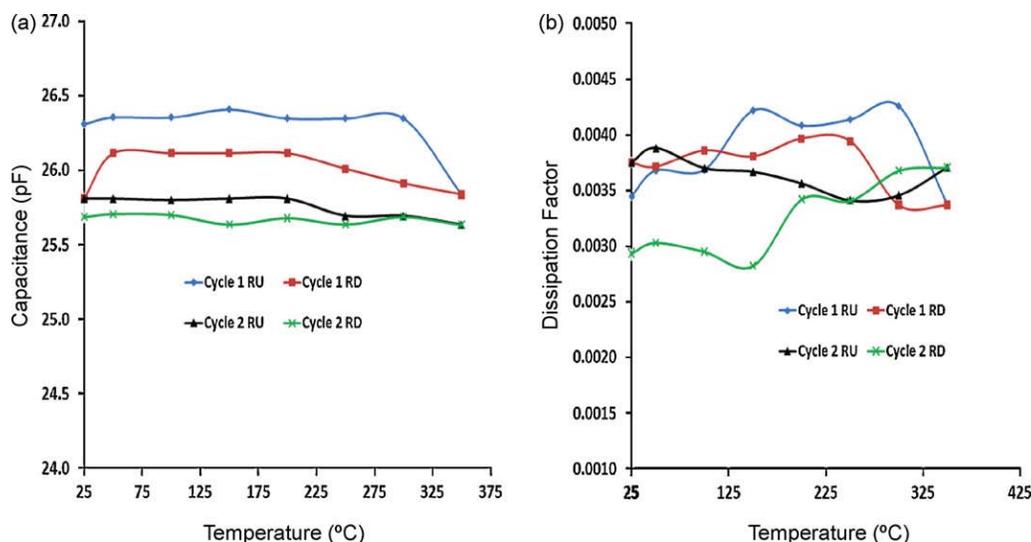


Fig. 7. (a) Film capacitance and (b) DF vs. temperature for FDAPE at 10 kHz frequency during two complete thermal cycles.

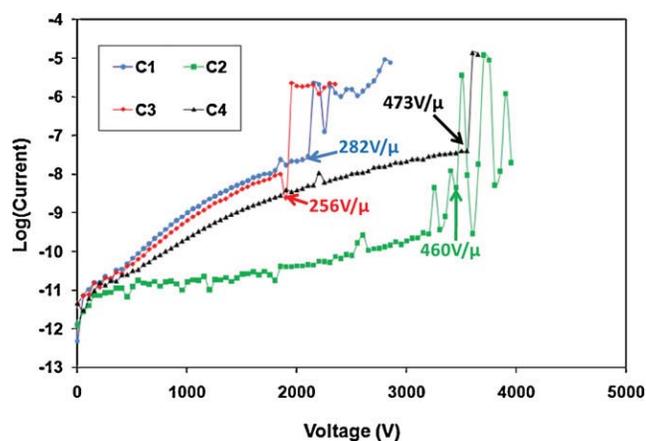


Fig. 8. High voltage breakdown measurement of metallized PI-ADE film (C1, C2, C3 and C4 are different capacitor areas tested in the same film).

was <0.003 (0.3%) after the second ramp down experiment. This is somewhat close to dissipation factors of ≤ 0.001 (0.1%) desired for μs discharge rates in power conditioning capacitors. FDAPE thus displays excellent wide-temperature dielectric stability due to its low TCC up to 350°C ; its relatively low DF can be attributed in part to the presence of the 4,9-diamantyl structural unit in its polymer backbone.

3.3.2. Film breakdown strength

High voltage (DC) breakdown measurements were performed on the metallized films. 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 room temperature electrical breakdown behavior of the metallized films is shown as current–voltage (I – V) plots and is illustrated for the PI-ADE film (Fig. 8) which was already subjected to LCR measurements in the RT– 250°C range. Testing of four different capacitor areas (referred to as C1, C2, C3 and C4) in the same film reveals the highest observed breakdown voltage to be $473\text{ V}/\mu\text{m}$ and the average breakdown voltage to be $367\text{ V}/\mu\text{m}$ for a $7.6\text{ }\mu\text{m}$ thick film. The breakdown voltages of various capacitor areas, marked on the I – V curves, correspond to the first surge with a flow of current of the order of $1\text{ }\mu\text{A}$ and more. The spikes in the current, seen in some of the I – V traces, are due to ‘clearing’ or ‘self-healing’ events occurring in the M–I–M (Metal–Insulator–Metal) device prior to complete electrical breakdown.

Preliminary breakdown results on the OH-6F-PBO/12F-PBO copolymer and FDAPE films show average room temperature

breakdown voltages to be in the range of 280 – $300\text{ V}/\mu\text{m}$. Further film breakdown measurements are in progress.

4. Conclusions

A few classes of high temperature polymer dielectric films have been investigated with regard to their potential utilization in wide-temperature aerospace power conditioning applications. Preliminary studies have demonstrated their dielectric stability for operation at high temperatures in the 250 – 350°C range. Variable temperature dielectric studies have shown that their temperature coefficient of capacitance over a wide-temperature range has been relatively small (1–3%). A relatively low dissipation factor (<0.003 (0.3%)) at 10 kHz has also been measured for the fluorenyl polyester (FDAPE) film after two complete thermal cycles conducted in the RT– 350°C range. The dielectric constants of the various polymer dielectrics in this study were in the range of 2.9–3.5.

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