AFRL-RZ-WP-TP-2010-2129

THERMALLY ROBUST POLYMER DIELECTRIC SYSTEMS FOR AIR FORCE WIDE-TEMPERATURE POWER ELECTRONICS APPLICATIONS (Postprint)

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JULY 2009
Final Report

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AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE ***(DD-MM-YY)***	**2. REPORT TYPE**	**3. DATES COVERED** ***(From - To)***
July 2009	Conference Paper Postprint	01 February 2009 – 24 July 2009

4. TITLE AND SUBTITLE

THERMALLY ROBUST POLYMER DIELECTRIC SYSTEMS FOR AIR FORCE WIDE-TEMPERATURE POWER ELECTRONICS APPLICATIONS (Postprint)

5a. CONTRACT NUMBER
FA8650-04-D-2403-0016

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
62203F

5d. PROJECT NUMBER
3145

5e. TASK NUMBER
13

5f. WORK UNIT NUMBER
31451312

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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Air Force Research Laboratory
Materials & Manufacturing Directorate

Power Division, Electrical Technology Branch (AFRL/RZPE)
Air Force Research Laboratory
Propulsion Directorate

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory
Propulsion Directorate
Wright-Patterson Air Force Base, OH 45433-7251
Air Force Materiel Command
United States Air Force

10. SPONSORING/MONITORING AGENCY ACRONYM(S)
AFRL/RZPE

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)
AFRL-RZ-WP-TP-2010-2129

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES
PAO Case Number: 88ABW-2009-3432; Clearance Date: 24 July 2009. Presented at IMAPS High Temperature Electronics Network (HiTEN 2009). The U.S. Government is joint author of the work and has the right to use, modify, reproduce, release, perform, display, or disclose the work.

14. ABSTRACT

Thermally stable, mechanically robust, compact capacitors are the technology driver for high performance power systems. The proximity of power electronics to heat sources demands that the thermal load for electronic system cooling be reduced or eliminated in the new generation aircraft power systems. While aerospace power conditioning capacitors typically use polycarbonate (PC) films in wound capacitors for operation in the -55°C to 125 °C range, there is a current need for high temperature polymer film dielectrics with dielectric stability up to 350 °C. As part of our program toward meeting the objective of dielectric stability and reliability in capacitor devices at temperatures as high as 350 °C, we designed and evaluated high strength polymer films with high glass transition temperatures (375-450 °C) as well as high thermal stabilities (470-520 °C). Variable temperature dielectric properties of metallized thin films in the RT-350 °C range are reported for high temperature polymer systems such as fluorinated polybenzoxazoles (6F-PBO) and a fluorenyl polyester incorporating a diamond-like hydrocarbon unit, known as FDAPE. A comparative dielectric evaluation of the state-of-the-art fluorenyl polyester film FPE, with a glass transition temperature of 330 °C, has also been performed. The focus of the study is on wide temperature dielectric measurements of film capacitance, and dissipation factor as well as insulation resistance and the effects of thermal cycling on polymer dielectric stability. Possible correlations between the thermo-mechanical properties of the polymer films and their high temperature dielectric properties are examined, from the viewpoint of their electro-mechanical stability for long-term operation in wide-temperature power electronics applications.

15. SUBJECT TERMS high temperature, polymer films, dielectric stability, aerospace, wide-temperature power electronics

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT:**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON** (Monitor)
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			Jeffery T. Stricker
Unclassified	Unclassified	Unclassified	SAR	14	**19b. TELEPHONE NUMBER** *(Include Area Code)* N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

Thermally Robust Polymer Dielectric Systems for Air Force Wide-Temperature Power Electronics Applications

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Abstract

Thermally stable, mechanically robust, compact capacitors are the technology driver for high performance power systems. The proximity of power electronics to heat sources demands that the thermal load for electronic system cooling be reduced or eliminated in the new generation aircraft power systems. While aerospace power conditioning capacitors typically use polycarbonate (PC) films in wound capacitors for operation in the -55°C to 125°C range, there is a current need for high temperature polymer film dielectrics with dielectric stability up to 350°C. As part of our program toward meeting the objective of dielectric stability and reliability in capacitor devices at temperatures as high as 350°C, we designed and evaluated high strength polymer films with high glass transition temperatures (375-450°C) as well as high thermal stabilities (470-520°C). Variable temperature dielectric properties of metallized thin films in the RT-350°C range are reported for high temperature polymer systems such as fluorinated polybenzoxazoles (6F-PBO) and a fluorenyl polyester incorporating a diamond-like hydrocarbon unit, known as FDAPE. A comparative dielectric evaluation of the state-of-the-art fluorenyl polyester film FPE, with a glass transition temperature of 330°C, has also been performed. The focus of the study is on wide temperature dielectric measurements of film capacitance, and dissipation factor as well as insulation resistance and the effects of thermal cycling on polymer dielectric stability. Possible correlations between the thermo-mechanical properties of the polymer films and their high temperature dielectric properties are examined, from the viewpoint of their electro-mechanical stability for long- term operation in wide-temperature power electronics applications.

Key words: High temperature, polymer films, dielectric stability, aerospace, wide-temperature power electronics.

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Introduction

Thermally stable, mechanically robust, compact, energy storage capacitors are the technology driver for a new generation of aerospace power conditioning systems [1]. The increasing proximity of power electronics to heat sources makes it imperative that the current thermal load for electronic system cooling be reduced or even eliminated in the new generation aircraft power systems. While the overall design should address increased reliability, and durability arising from wide-temperature dielectric stability, a graceful failure mode, characteristic of a metallized film capacitor, is also an important consideration. The benefits of the high temperature capacitor design are geared towards the AF INVENT (Air Force Integrated Vehicle Energy Technology Demonstration) program for the support of increasingly "more electric" aircraft configurations comprising robust electrical power systems and high temperature electric actuators. While we will focus on aerospace power conditioning as an area of emphasis, this will also be an enabling technology to other applications such as electric utilities, deep oil/well drilling, aircraft and automotive engine ignition systems and hybrid electric vehicle (HEV) technology.

High temperature polymer dielectrics are suitable candidates for wide-temperature capacitor applications because of their good dielectric stability over a wide range of frequencies and temperatures as well as their potential for high dielectric breakdown strengths and low dissipation factors. Furthermore, they are also amenable to large area fabrication into thin 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 ensures a graceful rather than a catastrophic failure mechanism which is generally not the case with pure ceramic capacitors.

Aerospace power conditioning capacitors typically use polycarbonate (PC) dielectric films in wound capacitors for operation in the temperature range of -55°C to 125°C. For higher operating temperatures up to 200°C, 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, amorphous high temperature fluorenyl polyester (FPE) films [5] have been considered as potential replacement for PC in aerospace power conditioning applications, with an operational temperature capability in the -55°C to 275°C range. FPE films are also reported to have good dielectric strength (400 V/μm), a low dissipation factor of 0.03 % at 1 kHz and good insulation resistance. However, thermal management for future aerospace power conditioning capacitor applications requires such polymer dielectrics to have operational capability up to or even exceeding 350°C. It is evident that polymer dielectrics with high glass transition or softening temperatures and high thermal/thermo-oxidative stability will play a significant role in providing the needed electro-mechanical stability for wide-temperature power electronics applications.

The main objective of the study is the design, synthesis, film fabrication and dielectric evaluation of some high performance polymer dielectrics for meeting the goals of aerospace power system conditioning capacitor requirements of stable dielectric performance up to 350°C and above. The variable temperature dielectric properties of metallized thin films in the RT-350°C range and the effects of thermal cycling on polymer dielectric stability are reported for high temperature polymer systems such as a fluorinated polybenzoxazole copolymer (OH-6F-PBO-12F-PBO, also referred to as 6F-Co-PBO) and a fluorenyl polyester incorporating a diamond-like hydrocarbon unit, known as FDAPE. The wide-temperature dielectric evaluation of the current high temperature candidate fluorenyl polyester (FPE) film has also been performed in the same temperature range to provide some comparison. Possible correlations between the thermo-mechanical properties of the polymer films and their high temperature dielectric properties are examined, from the viewpoint of assessing their electro-mechanical stability for long-term operation in Air Force wide temperature power electronics applications.

Experimental

(a) Synthesis and general characterization of 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]. Preliminary findings on the variable temperature dielectric properties of a range of high performance polymer films from AFRL have been reported elsewhere [9]. FPE films, provided by Brady Coatings, Inc., to AFRL/RZPE, were evaluated for comparison. The thermal and thermo-oxidative stabilities of the polymer were determined by Thermo-Gravimetric Analysis (TGA).

(b) Thermo-mechanical and mechanical properties of polymer films

The thermo-mechanical properties of solvent-cast films of 6F-Co-PBO, FDAPE and FPE were investigated by dynamic mechanical analysis (DMA) and thermo-mechanical analysis (TMA).

Glass transition temperatures (T_g) of the polymer films were determined by both DMA and TMA via tan δ and dimensional change measurements respectively. The storage modulus of the films was also measured in DMA over a wide temperature range. Film mechanical properties such as tensile strength and tensile modulus were performed using a Tinius tensile tester at a strain rate of 1 mm/min. The above mentioned tests were performed on solvent cast, relatively thick polymer films (30-90 μm).

(c) Thin film fabrication for dielectric measurements

Thin, freestanding polymer films were fabricated by solvent casting. 6F-Co-PBO films were cast from tetrahydrofuran (THF) as solvent while FDAPE films were generated from chloroform solutions of the polymer. After slow solvent evaporation from the flat glass casting dishes placed inside a desiccator, the thin films were carefully released from the glass dish by the addition of de-ionized, distilled water. The films were dried at ~ 0.1 torr vacuum and 80-85°C in an oven for several days. Circular films varying in diameter from 2" to 4" were fabricated and their average film thickness, measured by the thin film measurement system MP-100S (Mission Peak optics Inc.), was typically in the range of 3 μm to 6 μm. The average thickness of the FPE film was 5.7 μm.

(d) 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 Kurt J. Lesker 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), circular aluminum contacts with 2 cm^2 area were deposited. The wide temperature dielectric data reported in this paper are based on films with the larger (2 cm^2) aluminum contacts.

(e) Dielectric evaluation 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 1 V AC applied voltage. Variable temperature LCR measurements were conducted in a high temperature test station with operating temperature capability up to 1000°C. The test station has a 100V/10A power supply, a temperature controller and 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 a 3-axis molybdenum probing rod test fixture.. The dielectric measurements on the metallized films were conducted up to 350°C as the upper limit in increments of 50°C from room temperature. The film dielectric properties were typically monitored over two complete (ramp up and ramp down) thermal cycles.

Film insulation resistance (IR) was also measured 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 data were acquired over a 60 s time period. Measurements were taken at 50°C intervals from room temperature to 350°C 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 Hewlett 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

(a) General

The chemical structures of the high temperature polymer films 6F-Co-PBO, FDAPE and FPE films are shown in Figure 1. 6F-Co-PBO is a 1:1 random copolymer consisting of a hydroxyphenyl-6F-PBO unit as well as a 12F-PBO unit; both the glass transition temperature and the dielectric constant of the polymer can be tweaked by varying the copolymer composition [6]. The fluorenyl polyester FPE is also a 1:1 random copolymer consisting of ester links based on the relatively rigid 1,4-phenylene and the more flexible 1,3-phenylene moieties. FDAPE refers to another fluorenyl polyester composition incorporating a diamond-like hydrocarbon, i.e., 4,9-diamantyl unit in the polymer backbone. 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 diamond-like hydrocarbons (also referred to as diamondoids) are molecular counterparts of a large fundamental band gap in diamond itself [10].

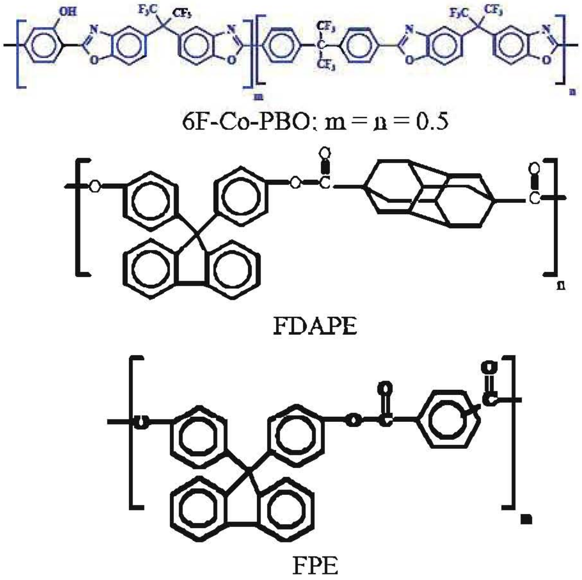

Figure 1. Chemical structures of the high temperature polymer dielectrics in this study

Comparative thermal stability of the three polymers is shown by the TGA traces in nitrogen in Figure 2. It was found that both FPE and FDAPE have nearly the same thermal degradation profiles (475°C onset of degradation for FPE and 470°C onset of degradation for FDAPE) while the onset of degradation for 6F-Co-PBO polymer was higher (~ 490°C). This shows the potential for these polymer dielectrics to operate reliably in the high temperature environment of the power conditioning capacitors.

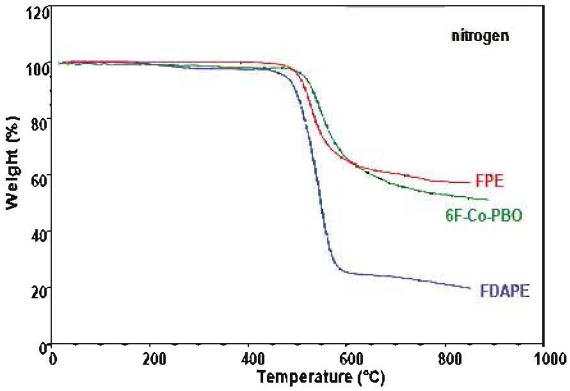

Figure 2. Comparative thermal stability data in nitrogen for FDAPE, 6F-Co-PBO and FPE films

(b) Film thermo-mechanical and mechanical properties

Thermo-mechanical profiles of the three polymer films FDAPE, 6F-Co-PBO and FPE are shown in Figure 3 as DMA plots. Since the limiting factor for the dimensional stability of the polymer dielectric is its glass transition temperature (T_g), glass transition temperatures well in excess of 350°C are potentially required for dielectric/electro-mechanical stability at operational temperatures in the vicinity of 350°C. From the tan δ and storage modulus plots, the onset of T_g is 450°C for FDAPE, 375°C for 6F-Co-PBO and 330°C for FPE, taken as the points of intersection of the storage modulus drop with the tan δ peaks . The TMA results also confirm the high glass transition temperatures of these polymer films (Figure 4). While FPE and FDAPE are found to undergo considerable dimensional changes at their T_gs, 6F-Co-PBO was found to undergo relatively less dimensional change due to stretching at its T_g. These differences will likely impact the long-range wide-temperature dielectric stability of these films in power system conditioning capacitors.

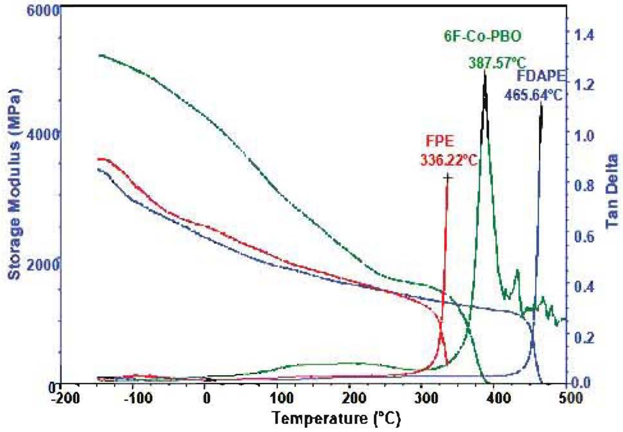

Figure 3. Thermo-mechanical profiles, by DMA, for FDAPE, 6F-Co-PBO and FPE films.

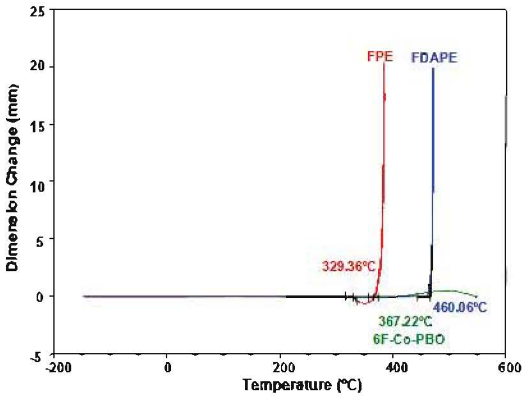

Figure 4. Thermo-mechanical profiles, by TMA, for FDAPE, 6F-Co-PBO and FPE films.

Room temperature tensile stress-strain profiles of these polymer films are shown in Figure 5. The average tensile properties of FPE and FDAPE films (2.14-2.15 GPa tensile modulus and 54-64 MPa tensile strength) are very similar. 6F-Co-PBO polymer film exhibited somewhat better mechanical properties with 2.3 GPa tensile modulus and a tensile strength of 68 MPa. The higher tensile properties of 6F-Co-PBO films can impart good mechanical integrity to thin films (5μm or less), ideally required for power conditioning capacitor applications. The average elongation-at-break (~14 %) is higher for 6F-Co-PBO than those for both FPE and FDAPE films (9.7 % and 8.2 % respectively). Another interesting feature of the stress-strain plot for 6F-Co-PBO is that the film exhibits an elongation-at-yield as well at 7.5 %. This indicates that the 6F-Co-PBO film exhibits a relatively more ductile failure mode under a tensile load compared to FPE and FDAPE. Such differences in modes of film mechanical failure and factors such as yield behavior and plastic deformation could play a crucial role in influencing continuous, large scale film processing as well as packaging of the wound capacitor device utilizing these films.

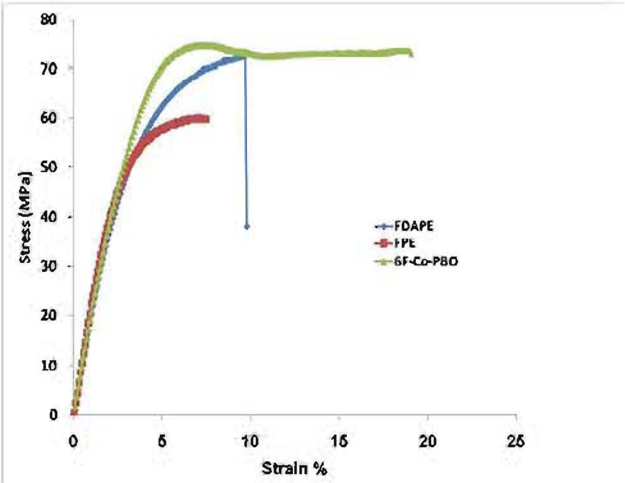

Figure 5. Stress-strain profiles of the three high temperature polymer films

(c) Dielectric evaluation

(1) Temperature coefficient of capacitance (TCC) and Dissipation Factor (DF)

The typical device fixture used in the high temperature, high vacuum test chamber for variable temperature LCR measurements is shown in Figure 6. The polymer film is fully aluminized on the bottom face and 2 cm^2 aluminum contacts deposited through contact masks serve as the top electrodes in the measurement.

Figure 6. Test fixture used in the variable temperature LCR measurements on metallized polymer films

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. Figures 7 and 8 illustrate the variable temperature LCR characteristics in the temperature range of RT-350°C for the three metallized polymer films. The measured film thickness was 5.7 μm for FPE, 5.1 μm for FDAPE and 3.3 μm for 6F-Co-PBO. The measured film capacitance for the films with the 2 cm^2 testing area was typically of the order of 1-1.5 nF. The derived values of dielectric constant at 10 kHz from the capacitance measurements were 3.4, 3.5 and 2.9 for FPE, FDAPE and 6F-Co-PBO films respectively. Figure 7 depicts TCC as a function of temperature for the three metallized films and Figure 8 shows the Dissipation Factor (DF, %) for the three films at various temperatures. The variable temperature profiles shown in Figures 7 and 8 are based on measurements at 10 kHz frequency during the second heating cycle. It is found that FPE and FDAPE films undergo minimal changes in TCC as a function of temperature (2.0 % and -2.4 % respectively) while only a marginally higher TCC (3.0 %) is observed in the case of 6F-Co-PBO in the RT-350°C range. However, at temperatures in the 300-350°C range, relatively sharper variations in measured film capacitance are observed for 6F-Co-PBO. An analysis of the DF-temperature profiles of the three polymers reveals the following. FDAPE film showed the lowest DF among the films tested, with the DF increase from 0.08 % at RT to 0.12 % at 350°C. In the case of FPE, the DF was found to fluctuate in the 0.3 %-0.6 % range. For the 6F-Co-PBO film, DF was found to increase threefold from 0.3 % at RT to 0.9

% at 350°C. Again, pronounced increase in DF values at 300°C and above was clearly discernible in the case of 6F-Co-PBO.

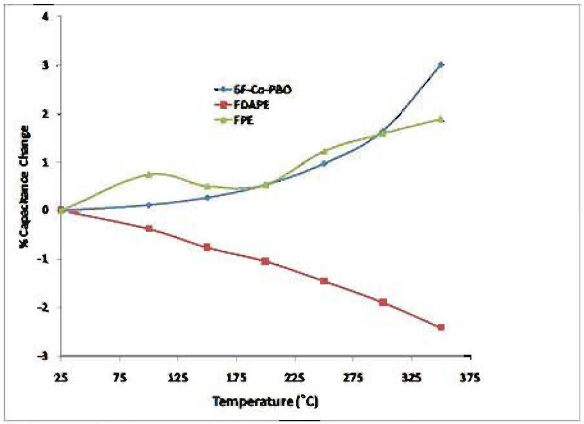

Figure 7. Capacitance variation vs Temperature at 10 kHz for the polymer dielectrics.

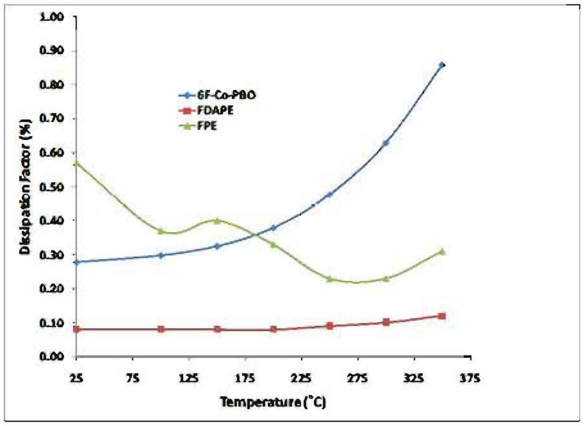

Figure 8. Film dissipation factor (%) as a function of temperature at 10 kHz for the polymer dielectrics

Since FPE has a rated stability for utilization up to 250°C, it was somewhat surprising that we observed a low TCC for the FPE film tested up to even 350°C. Presumably, short-term excursions into thermal environments somewhat exceeding the glass transition temperature (330°C) of FPE did not result in significant dimensional changes that would affect its wide-temperature dielectric stability in the RT-350°C range. Its dielectric stability was comparable to that of FDAPE film at 350°C which was clearly much lower than the T_g of FDAPE (~ 450°C). However, comparative assessment of long-term reliability of these films would require thermal aging studies and dielectric stability evaluation in an even wider temperature range (up to or exceeding 400°C). It will be interesting to examine the FPE film at temperatures well above its T_g, in order to observe the effect of enhanced polymer chain mobility on its dielectric behavior.

The significant enhancement in DF (%) values and the relatively sharp variations in % capacitance change at the higher temperatures of 300°C and 350°C for 6F-Co-PBO can be tentatively ascribed to a change in the polarization of the polymer dielectric. A plausible explanation is based on the dissociation of the intra-chain hydrogen bonding involving the hydroxyl group in 6F-Co-PBO at these high temperatures; this can potentially enhance both dissipation factor and dielectric permittivity due to the presence of free, polar hydroxyl groups attached to the polymer backbone. Presumably, since 6F-Co-PBO film is still in its glassy state (T_g~375°C) at the operational temperatures of 300°C and 350°C, dimensional changes occurring in the metallized polymer dielectric may be too insignificant to cause any sharp variations in film dielectric behavior at these temperatures.

(2) Variable temperature film insulation resistance data

Variable insulation resistance data up to 350°C, with 40VDC as the test voltage for 6F-Co-PBO, FDAPE and FPE films are shown in Figure 9. The measured values of room temperature volume resistivity of FPE and FDAPE are very similar, 1.2 x 10^{17} and 1.7 x 10^{17} ohm.cm respectively. In the case of 6F-Co-PBO, the room temperature resistivity is somewhat lower (2.7 x 10^{15} ohm.cm). For comparison, the reported room temperature resistivity values of some commercial polymer films evaluated for capacitor applications are also shown.

Interesting variations in wide-temperature insulation resistance characteristics were observed for the different polymer films in the RT-350°C range. In the case of FPE, there was an overall decrease in volume resistivity by nearly four orders of magnitude from 1.2 x 10^{17} ohm.cm at RT to 3.2 x 10^{13} ohm.cm at 350°C. 6F-Co-PBO showed resistivity decrease from 2.7 x 10^{15} to 3 x 10^{13} ohm.cm and FDAPE exhibited considerably less variation in volume resistivity, with a decrease from 1.7 x 10^{17} to 8.7 x 10^{16} ohm.cm in the entire RT-350°C range. The high temperature insulation resistance of FDAPE was the highest among the three polymer films tested.

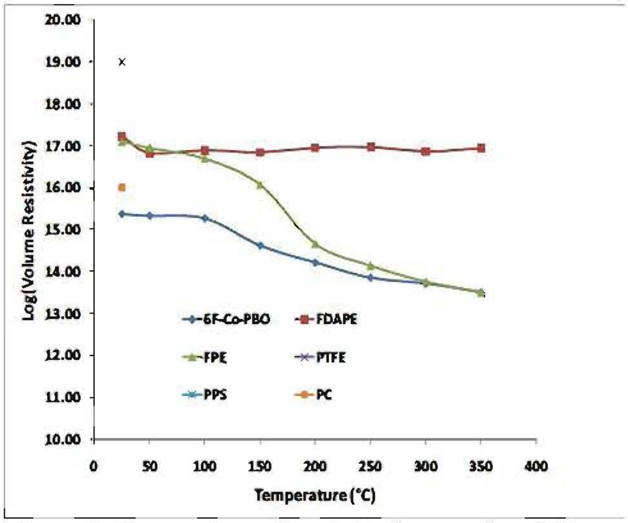

Figure 9. Comparison of variable temperature IR of the films (also shown: RT resistivity of some common commercial films)

(3) Film dielectric breakdown

While the main focus of the investigations was the evaluation of high temperature dielectric stability of polymeric materials for wide-temperature capacitor applications, some preliminary room temperature, high voltage (DC) dielectric breakdown measurements were performed on metallized films with 3 mm aluminum electrodes after thermal cycling up to 350°C. 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 measured average breakdown strengths for FDAPE film and 6F-Co-PBO films were 300 V/µm and 280 V/µm respectively while it was measured to be 400V/µm for the FPE film. However, optimization of the current solvent–based laboratory scale thin film fabrication process can lead to potentially higher film breakdown strengths for both FDAPE and 6F-Co-PBO films.

Summary and Conclusions

Variable temperature dielectric properties of high performance polymer films with high glass transition temperatures and excellent thermal stabilities were evaluated in the RT-350°C range from the viewpoint of potential utilization in wide temperature power conditioning capacitor applications. The temperature coefficient of capacitance (TCC) ranged from 2-3.5 % in the RT-350°C range for the three films, FPE, FDAPE and 6F-Co-PBO. While film thermo-mechanical properties are indeed important for electro-mechanical stability at higher operational temperatures, measured TCC indicated that the short-term dielectric stability of FPE at temperatures even slightly exceeding its Tg was not affected. Besides a low TCC, FDAPE also exhibited a desirably low DF in the range of 0.08-0.1 % in the RT-350°C temperature region. Overall, all these polymer films exhibited good high temperature insulation resistance with measured volume resistivity in the range of 10^{13}-10^{16} ohm.cm at 350°C.

Ongoing and future studies will focus on assessment of long-term reliability of these polymer dielectric systems via thermal aging and dielectric evaluation up to even higher temperatures (400°C). Besides thermal endurance, the influence of voltage stress on wide-temperature dielectric stability will also be studied, by biasing the sample under AC or DC conditions during thermal cycling.

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