High Temperature Dielectric Polyetherimide Film Development

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ABSTRACT

A new high temperature ultra-thin polymer film based on Polyetherimide (PEI) chemistry has been developed and electrically characterized and compared with other dielectric capacitor films. More specifically, intrinsic and extrinsic dielectric breakdown strength is compared and reported for multiple resins thus demonstrating feasibility of the new polymer film. In addition, results from a large area electrode test are presented and the importance of this test method in the development of high temperature capacitors for DC applications is discussed.

Index Terms — **Capacitors, Dielectric breakdown, Dielectric measurements, Electric breakdown, Insulators, Materials reliability, Power capacitors, Power electronics, Plastics, Prediction methods, Reliability, Solder reflow, Testing, Thin film capacitors, Thin film, Polyetherimide.**

1 INTRODUCTION

POLYMER film capacitors are an interesting technology for high voltage applications due to their capability to self-heal, fail open circuit, or fail gracefully over their operational lifetime. In the automotive and electronic markets, this self-healing nature is essential, as a catastrophic failure (failure resulting in a short circuit) could potentially increase the risk of a fire or explosion. Ceramic capacitors are known to have issues with shorting during failure as well as thermal shock. Electrolytic capacitors, which are cost effective with good energy density, were first specified for use in inverter applications but also have a catastrophic failure mode. Bi-axially oriented polypropylene (BOPP) is the preferred choice for automotive applications due to its excellent dielectric properties, self-healing capability, ease of manufacturing, and high breakdown strength. It can be bi-axially oriented to produce a highly crystalline polymer and is commercially available in 2.2 µm thickness. BOPP is not without limitations, however.

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The main limitation of BOPP is its lack of electrical performance at temperatures exceeding 105 ºC when wound into a capacitor as it tends to fail short due to low insulation resistance (IR). Other commercially available capacitor films currently being investigated for this application include polyethylene terephthalate (PET), polyethylene naphthalate (PEN), polyimide (PI), polycarbonate (PC), and polyphenylene sulfide (PPS) as well as others that are still under development including polyetheretherketone (PEEK), polytetrafluoroethylene (PTFE), polymethylpentene (PMP), and fluorene polyester (FPE)[1].

Thermoplastic materials such as PET, PEN, and PC with dielectric constant (Dk) > 3.0 are reasonable alternatives, however each material has other limitations restricting their use in the application. The materials are limited to operating temperatures less than 140^{o}C, a typical operating temperature requirement for the application. PET has a dissipation factor (Df) which increases exponentially at temperatures exceeding the materials glass transition temperature (Tg) of 92^{o}C. PET typically requires a voltage derating factor when used above 85^{o}C due to its poor insulation resistance (IR) [1]. PEN has a dissipation factor similar to PET and in addition has issues with particle

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additives, which are added to the material to provide surface texture to prevent blocking and enhance its processability [3]. The particle distribution can be larger than the final film thickness, which compromises the dielectric strength and mechanical strength in the regions near the particles. PC is an excellent material but is limited to a continuous use temperature of 125 ^{o}C and is no longer commercially available as an extruded thin film [3]. In addition, several materials, which meet the high temperature requirements such as polyphenylene sulfide (PPS), suffer from poor self-healing properties making them less desirable for use in capacitors [4]. However, polyetherimide (PEI) is a material for which there has been much attention, since it has high temperature capabilities near 185^{o}C with a Dk > 3.0, adequate breakdown strength, and low dissipation factor < 1%, which makes it ideal for use as a capacitor film [4]. In addition, PEI exhibits low ohmic conductivity up to 200 ^{o}C, which is shown by its high insulation resistance response at high temperatures in DC applications [5]. This high temperature capability coupled with low shrinkage also enables end use applications where a solder reflow process is used, such as DC-DC converters [6]. The limitation of PEI has been its inconsistency to self-heal when a thick metallization layer is used in the fabrication of a wound capacitor. This has been significantly improved by the use of thinner evaporated electrodes in the center of the capacitor. Thicker electrodes near the edges of the film are used to carry the large currents and ensure an optimal connection to the end spray. This metallization design is more commonly known as a 'heavy edge, light body'.

Upon failure of a plastic dielectric during breakdown, a film constructed with a polymer with a high [C/(O+H)] ratio will deposit graphite on the surface of a clearing and therefore it is desirable to minimize this ratio [7]. The deposited graphite creates conductive bridges on the surface of the cleared area that enable electrical shorts to form from the previously broken down area. Thus, instead of a slow decay in capacitance over time during self-healing events as effective area is reduced, electrical shorting occurs and the operational lifetime of the capacitor is significantly reduced. This is of high importance to capacitor manufacturers as many DC capacitors applications are generally expected to have an operational lifetime in the thousands of hours.

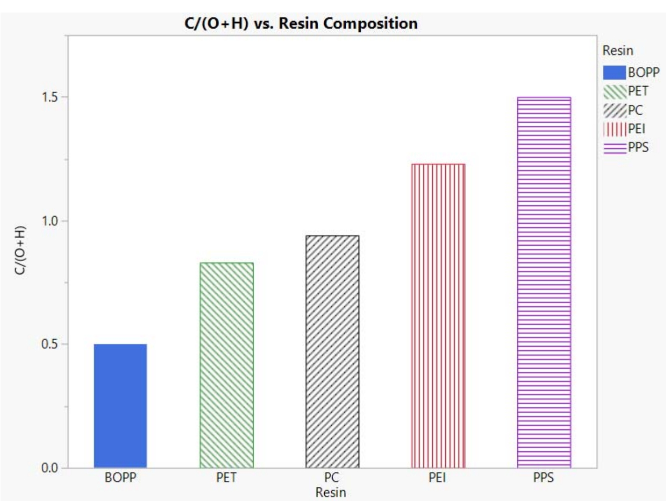

Figure 1. Self-healing capability of capacitor films based on [C/(O+H)] ratio.

The preferred method to analyze self-healing performance is through an insulation resistance measurement of a wound capacitor. Insulation resistance (IR) is the resistance the dielectric film creates between a voltage potential. Every dielectric insulator is leaky, meaning a small amount of current will flow between this potential difference when a voltage is applied. When a self-healing event occurs in a metallized film capacitor, graphite is deposited around the clearing location which provides a path for a current to flow and subsequently insulation resistance (IR) decreases. There are steps a capacitor manufacturing company can take to improve capacitor self-healing performance including using a burn-in procedure (slowly ramping voltage and temperature to remove any low voltage clearings in a low energy state), a patterned electrode, a reduction in electrode thickness [8], and an increase in the amount of trapped air found in between layers (i.e. tightness of capacitor wind).

Surface roughness is an important factor in film capacitors as web (or film) handling and winding procedures require roughness and minimum coefficient of friction (COF) for the film to track properly. It has been shown that a flat surface under electrical potential stress may have a more uniform electric field on its surface than a film with a surface roughness. This rough surface will cause the electric field to concentrate more strongly on any peaks or sharp protrusions. The concentrations enable an electrical breakdown to occur at a lower voltage due to the inhomogeneity of the electric field. As mentioned above, a surface roughness is necessary for dielectric films to be wound into capacitors so a compromise exists. Surface roughness could also have the effect of adding small air gaps in between the rolled layers, which could add oxygen into the (C/[O+H]) equation, potentially helping self-healing performance.

2 EXPERIMENTAL SETUP

2.1 DIELECTRIC CONSTANT AND LOSS FACTOR

Commercially available extruded film samples of ULTEM™ PEI (UTF-120) resin were provided by SABIC at 5 µm thickness and a uniformity of less than ± 4%. Capacitor grade 6 µm PPS thick film was supplied from Steiner Germany. Film samples were conditioned prior to testing by heating to approx. 25ºC below the material's glass transition temperature (Tg) to remove absorbed moisture (PPS was conditioned at 185ºC). Film samples of 5 µm BOPP and PET were supplied by MEHER Advanced Materials and conditioned under vacuum at 100ºC. All samples were conditioned for 10 hrs in a vacuum oven prior to testing.

Film thickness was measured using a Heidenhain Metro gauge accurate to ± 0.2 µm. Three locations in a 1.0 cm^2 area were chosen for film thicknesses measurement prior to metallization and their average was used for the dielectric constant calculations. For dielectric constant and loss measurements, aluminum (50 nm thickness) was deposited through thermal evaporation on 5 samples at 3 nm/second for each film type using a 10 mm diameter circular shadow mask. An Agilent E4980A Precision LCR Meter synced with a Tenney humidity and temperature chamber was used to measure dielectric constant and dielectric loss as a function of frequency at 25, 120, 160, and 200 ºC. The connection from the LCR meter was made with a Keysight 16048A test lead kit soldered to two spring probes.

2.2 BREAKDOWN STRENGTH

Breakdown strength (BDS) was measured following the ASTM D-149 standard (ramping at 500 V/s). This test utilizes a 6.35 mm stainless steel ball on a brass plate immersed in silicone oil to minimize the electric field non-uniformity and the chances of a film defect being present at the test location. ASTM D-149 returns a value that approaches the entitlement BDS of the sample. The breakdown strength thickness was measured in a 2 mm diameter circle drawn on each film using permanent marker and the respective thickness was recorded prior to breakdown. This was done so the ball in-plane measurement could be placed on the exact spot the thickness measurement was taken. Twenty measurements were made on each film and the dataset was fit using a 2-parameter Weibull distribution with a 95% confidence interval. The dielectric oil temperature was raised through a Fisher Scientific Isotemp circulator bath coupled with a cleaning filter. The oil temperature was maintained at each temperature for 5 minutes to reach equilibrium prior to breakdown testing.

2.3 LARGE AREA BREAKDOWN STRENGTH

Film samples with dimensions 30.5 cm by 20.32 cm were cut from the center of a film roll. Due to variation of film thickness across the roll, thickness was measured at four locations of each 30.5 cm x 20.32 cm sample, using the Heideinhain Metro thickness gauge, which is accurate to ± 0.2 µm. Thickness measurements were averaged to provide a per-sheet average.

In order to test overall quality of the films, samples were tested at room temperature on a 0.033 m^2 brass electrode connected to a high potential with a face down metallized bi-axially oriented polypropylene (BOPP) (metallized-side down) of dimensions 35.56 cm x 17.78 cm and a metallization surface resistivity of 4 to 8 ohms per square connected to the ground (low) potential. The films under test separate a low and high potential mimicking a capacitor under an applied electric field in order to determine extrinsic material performance, similar to a wound film capacitor. A schematic for the extrinsic "Large Area Electrode" test in shown in Figure 2 below.

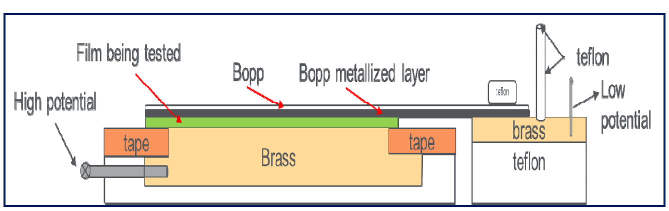

Figure 2. Extrinsic Large Area Electrode test setup.

High voltage was supplied to the bottom brass electrode using a BK precision programmable power supply, through a 20 kV Trek 20/20C amplifier. Highly insulating 2.54 cm wide and 1 mil thick 3M 1350F tape was used around the edges of the bottom brass electrode (positive potential) to remove the possibility of edge breakdown due to electric field concentrating at sharp edges. Ten samples (20.32 cm x 27.94 cm) of each material were tested (PPS had only 5 samples available to be tested). The results were statistically analyzed at a 95% confidence level using JMP 12 statistical software to test for measurement error, sample to sample variability, and statistical difference between samples.

The required voltage for the pre-amp was calculated for each electric field placed on the sample based on its average thickness measurement. The electric fields ranged from 20 V/µm to 500 V/µm. The voltage was held for 5 seconds at each voltage level and increased to the next voltage level until the test was complete. The goal was to observe and record, using an oscilloscope, all breakdowns that occurred at each voltage level. For each voltage, the cumulative counts across the 10 sheet samples were averaged and plotted with the maximum and minimum of the 10 samples being the bounds of error bars.

2.4 REFLOW SIMULATION

PEI film (UTF-120) samples were metalized using a vapor deposition method and capacitor samples were produced by Hitachi AIC Inc. with a PPS casing (20 mm x 22 mm x 12 mm), shown in Figure 3 below.

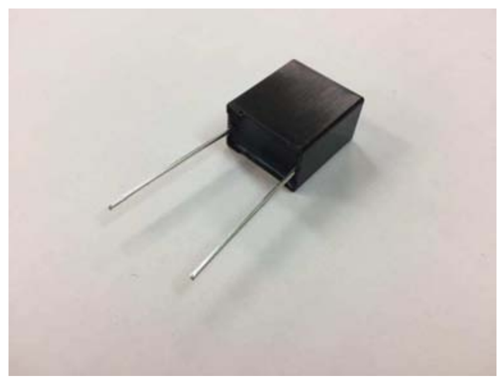

Figure 3. PEI film capacitor (Hitachi AIC Inc.).

Silicone glue (TSE3826, Momentive) was used to mount 5 PEI film capacitors on the printed circuit board (PCB), shown in Figure 4 below. In addition, 3 samples per each condition were prepared to evaluate data variance of the capacitor samples and reflow soldering conditions.

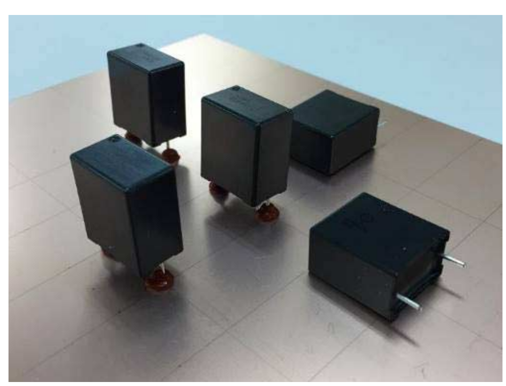

Figure 4. Reflow solder test capacitor orientation.

The lead-free reflow soldering process under hot air environment was simulated using an eight-zone reflow oven. Samples were transversely placed on the conveyor of a XPM3-820 (Vitronics-Soltec) continuous convection reflow chamber. The reflow soldering process was repeated three times and the shift of capacitance and insulation resistance (IR) between initial performance and the repeated cycles was determined.

The reflow soldering temperature profile was prepared following an IPC/JEDEC J-STD-020E standard and measured on the PCB and capacitor surfaces with a thermocouple fixed to the PCB using Kapton® tape. Figure 5 shows a 260ºC peak temperature for the tested profile.

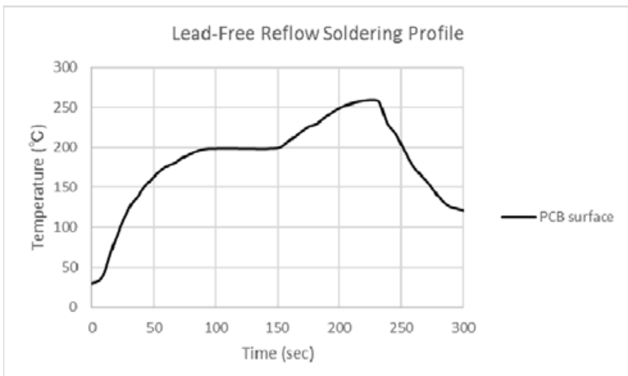

Figure 5. Lead-free reflow soldering temperature profile.

2.5 CAPACITANCE

Capacitance was measured using an LCR meter E4980A (Keysight) at room temperature. One side electrode of the capacitor was connected to high voltage and high current terminal of LCR meter and the other electrode of the capacitor was connected to a low voltage and low current terminal. A 1 V bias at 1 kHz frequency was applied to measure the capacitance value.

2.6 INSULATION RESISTANCE

Insulation resistance (IR) was measured using ultra-megohmmeter SM-8220 (Hioki) at room temperature. The electrodes of the capacitor were connected to terminals of the ultra-megohmmeter. A voltage of 100 V was applied to the capacitor for 60 seconds and IR was measured after 10 seconds.

3 RESULTS AND DISCUSSION

Figures 6-8 present the dielectric constant and loss at 20ºC, 120ºC, 160ºC, and 200ºC versus frequency ranging from a to b. The maximum measurement was dependent on the temperature capability of the individual polymers. The maximum operating temperature for BOPP is typically 100ºC whereas PET typically can be used to 125ºC. PEI and PPS were measured at 160ºC, and 200ºC, respectively, for the dielectric measurements as this is below the glass transition temperature for PEI and the melting temperature of PPS.

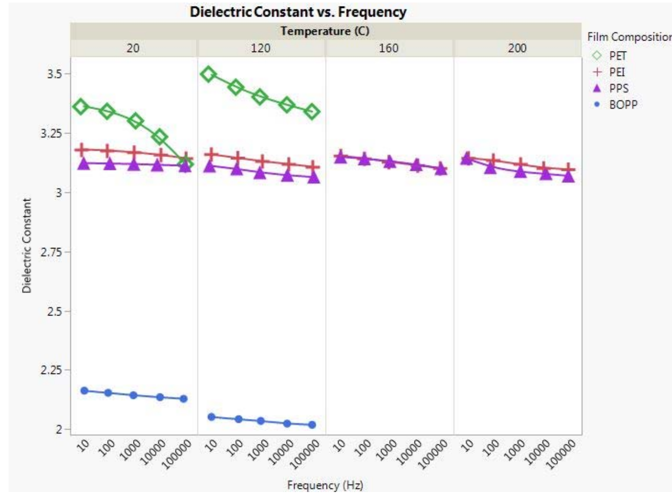

Figure 6. Dielectric constant as a function of frequency and polymer film type at 20ºC.

As presented in Figure 6, BOPP demonstrates a slight reduction in dielectric constant with temperature and remains relatively unchanged as a function of frequency. PEI has very similar dielectric performance as PPS throughout both temperature and frequency. PET demonstrates the highest dielectric constant of all of the films tested and ranges from 3.1 to 3.5 versus temperature and frequency.

Figures 7 and 8 present dielectric loss (also referred to as dissipation factor or loss tangent) at 20ºC, 120ºC, 160ºC, and 200ºC as a function of frequency depending on the maximum operating temperature of the polymer.

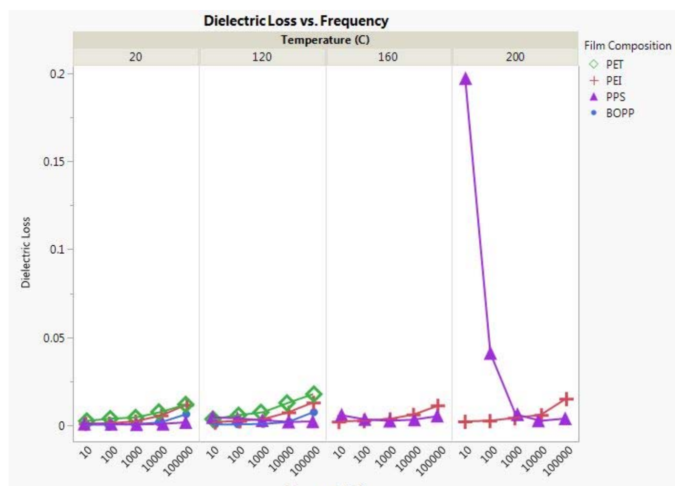

Figure 7. Dielectric loss (loss tangent) as a function of frequency and polymer film type at 20ºC through 200ºC zoomed out to show low frequency loss of PPS.

As can be seen in Figures 7 and 8, PPS has a very high low frequency dielectric loss at 200ºC due to ohmic conduction. This can lead to thermal runaway even though the polymer is thermally stable at elevated temperatures [4]. PEI does not exhibit this behavior and has very stable dielectric loss performance over temperature and frequency.

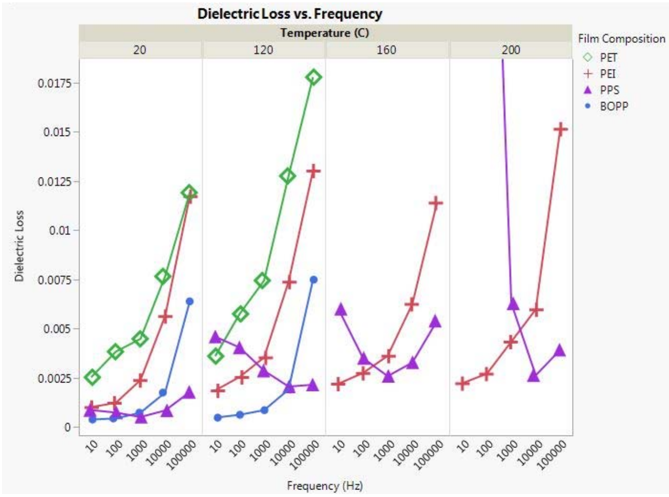

Figure 8. Dielectric loss (loss tangent) as a function of frequency and polymer film type at 20 ºC through 200 ºC zoomed in.

Figure 9 presents the 2-parameter Weibull breakdown strength with 95% confidence intervals using Minitab® via the ASTM D-149 standard method at 27ºC.

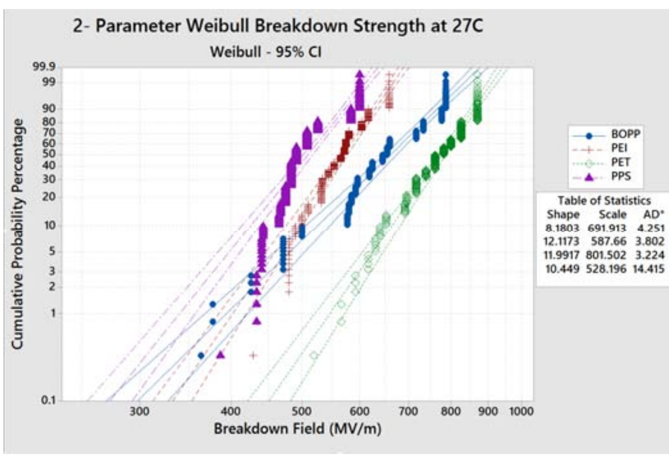

Figure 9. Room temperature ASTM D-149 breakdown strength of various polymer films. The Shape parameter describes how the Weibull data is distributed. The Scale parameter determines the spread of the Weibull distribution. The Anderson-Darling statistic (denoted AD*) measures the area between the Weibull distribution line of best fit and the empirical distribution function from the individual data points.

Figure 10 presents the 2-parameter Weibull breakdown strength with 95% confidence intervals using Minitab® via the ASTM D-149 standard method at 100 ºC for PET and BOPP films due to their max use temperature and 150ºC for PEI and PPS films.

A significant improvement in the quality of the PEI film can be seen by the slope of the Weibull breakdown strength as shown in Figures 9 and 10. Experimental grades of PEI as shown by Ho et. al. [4] showed greater variability in the Weibull breakdown strength and a reduction in the Weibull slope value compared to the commercial UTF-120 grade. Polyetherimide outperforms PPS in breakdown strength at room temperature and 150 ºC as well as having a higher slope value. As in Ho et. al. [4], a difference between the breakdown strength of PET and BOPP vs. the high temperature films is visible.

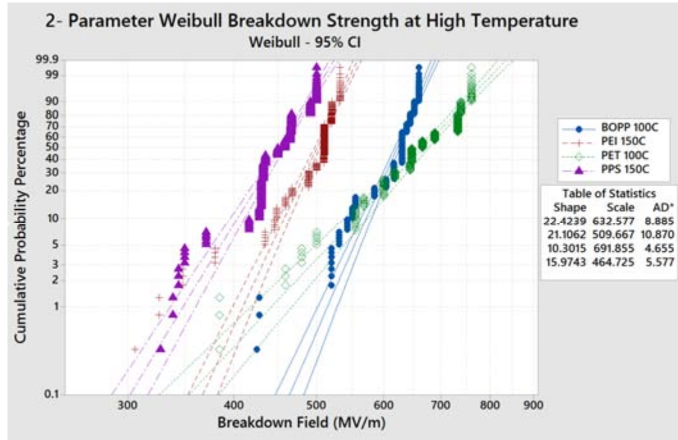

Figure 10. High temperature ASTM D-149 breakdown strength of various polymer films.

Figure 11 presents the cumulative clearing counts for the large area electrode test method.

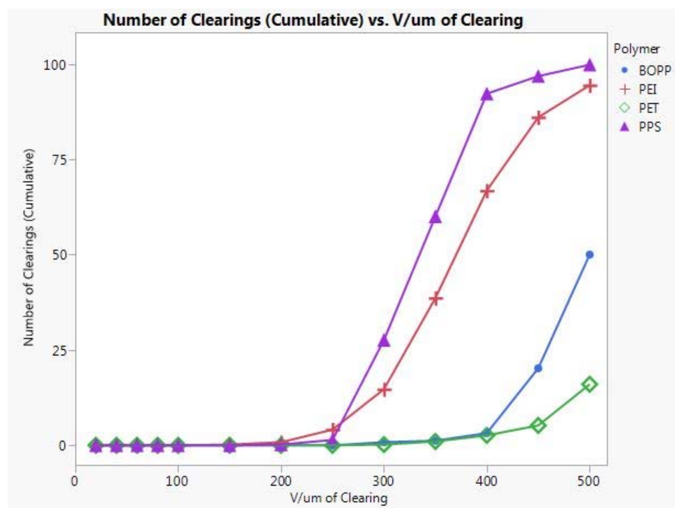

Figure 11. Extrinsic Large Area Electrode test cumulative clearing counts as a function of electric field and polymer film type.

Large area electrode (LAE) test results are best interpreted by reviewing the voltage take off point (change in slope) at which the film began to electrically break down. The take-off point corresponds to the initial peak observed in the average cumulative clearings graph. Furthermore, an analysis of variance (ANOVA) was performed at each voltage level, to evaluate the statistical difference in clearing counts between the two materials.

The ANOVA analysis showed significant differences in the data at 250 V/µm, with BOPP and PET having the lowest cumulative clearing counts versus PPS and PEI. PPS did not show many clearings at 250 V/µm with a mean value slightly above BOPP and PET. As electric field was increased to 300 V/µm, a large increase in clearing counts was realized for PPS. At 300 V/µm, PPS was statistically different from PEI, BOPP, and PET. PEI was statistically different from PPS, BOPP, and PET. BOPP and PET were only statistically different from one another at 500 V/µm. PET had the best overall LAE performance in terms of clearing counts and sample to sample variation. The LAE results showed similar trends in ASTM D-149 testing where PET had the highest breakdown strength versus temperature of all of the films evaluated.

While the large area electrode method allows for numerous breakdown strength events to occur in one film, caution must be taken in analyzing electric fields higher than the take-off point. The justification for this stems from the fact that once initial clearings occur, this removes effective area from the sample under test as the metallized film essentially open-circuits the failure point. This in-turn explains why the samples with higher cumulative clearing counts have a higher variability as they are strongly dependent on how many clearings previously occurred.

The large area electrode test method also makes a key assumption that the thickness uniformity of the sample under test is as small as possible. As the thickness non-uniformity of the sample increases in the 20.32 cm x 27.94 cm area, the electric field no longer is consistent throughout the entire sample, clouding the results.

The solder reflow results confirm the usability of PEI film capacitors. Figure 12 shows the surface temperature of the PCB and the capacitor during the reflow soldering process. PCB surface temperature is very similar to the lead-free reflow soldering temperature profile. The capacitor surface temperature does not increase the same as PCB surface temperature and it shows approximately 30ºC lower peak temperature than PCB surface temperature. Table 1 demonstrates the capacitance and IR shift before and after the reflow soldering profile. All capacitors tested slightly increased in both capacitance and IR indicating that there was no negative dielectric effects associated with the solder reflow process.

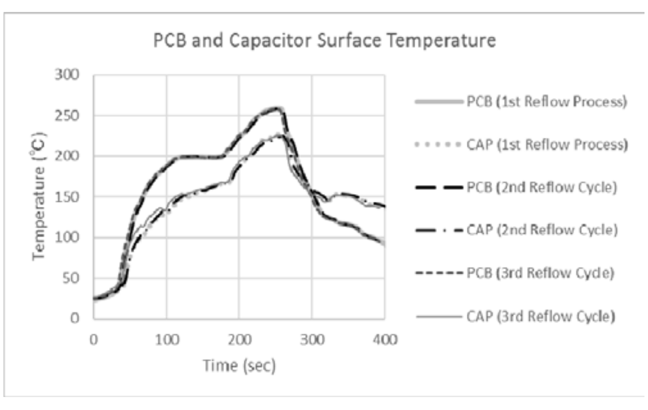

Figure 12. PCB and capacitor surface temperature during the reflow soldering process.

Table 1. Capacitance and Insulation Resistance change before and after the reflow soldering process.

Reflow condition	Initial Cp (μF)	% Change Cp (μF) after Reflow	Initial IR (MΩ)	% Change IR (MΩ) after Reflow
1st Reflow Process	1.272±0.01	0.0031	> 1.0×10^4	0.562
1 Repeat	1.275±0.02	0.0047	> 1.0×10^4	0.485
2 Repeats	1.274±0.01	0.0057	> 1.0×10^4	0.579

Figure 13 shows capacitance change before and after the reflow soldering process. The change in capacitance is quite stable with a shift of less than 0.01% after two repetitions, with the capacitors going through the reflow chamber a total of 3 cycles.

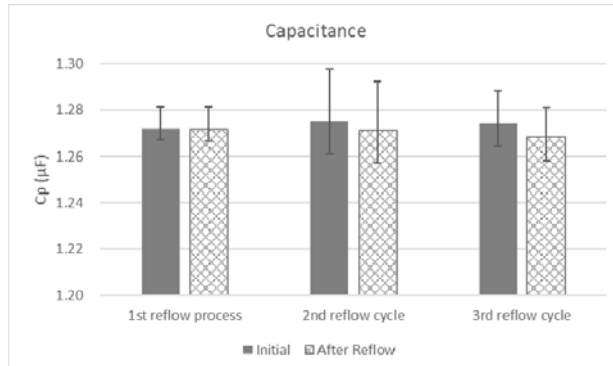

Figure 13. Capacitance change before and after reflow soldering.

Figure 14 shows insulation resistance (IR) change before and after reflow soldering. Insulation resistance is stable with a ΔIR < 0.1%. The IR data shows higher than $1.0\text{x}10^4$ MΩ even after cycling through the reflow chamber a total of three times.

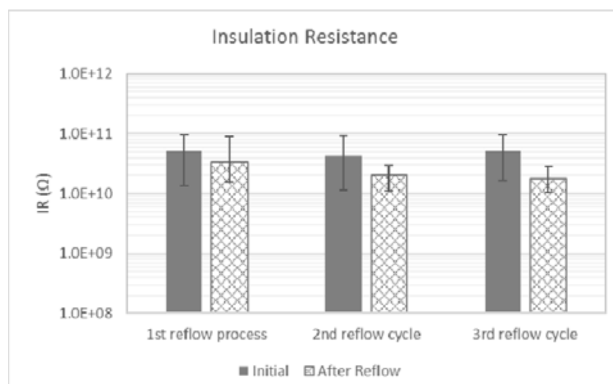

Figure 14. Insulation resistance change before and after the reflow process.

4 CONCLUSION

While numerous other researchers have developed metallized or metal-foil type large area breakdown strength test setups [9[13], this work is the first demonstration of a film quality ranking system from breakdown strength over a large area. This is quite useful for capacitor manufacturers as it demonstrates what electric fields the capacitor film can operate at over a large area. This data can then be directly translated to approximate working voltages for a wound capacitor instead of judging the film based on its intrinsic performance.

PEI film has been developed and optimized for capacitors in automotive and electronic applications. PEI has a use temperature of 185ºC with stable dielectric loss and dielectric constant creating the opportunity for useful high temperature capacitors that can pass the stringent solder reflow process. PEI film has an ASTM D-149 breakdown strength (BDS) greater than 550 V/μm at 22ºC, is very stable through 150ºC, and is melt processable into thin 5 μm films. This will enable PEI to meet the demanding requirements for high operating temperatures while still having the capability to fail gracefully when wound and pressed into a capacitor.

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