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High Temperature and Ageing Test Methods to Characterize the Dielectric Properties of BOPP Capacitor Films

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Abstract- A large-area high temperature breakdown measurement and an ageing test method are presented. These methods facilitate the development of reliable higher energy density film capacitors by exploiting large measurement areas to provide information on weak point formation and subtle changes in breakdown behavior after electro-thermal or thermal ageing. The test methods were used to characterize two types of highly isotactic biaxially oriented polypropylene capacitor films, which had similar breakdown behavior at room temperature, but different breakdown properties at high temperature and out of which one was more susceptible to electro-thermal DC ageing.

I. INTRODUCTION

Large-area breakdown tests and electro-thermal ageing tests during material development are used to evaluate effects, which limit the energy density of assembled capacitors far below any theoretical values calculated from small-area breakdown strength and permittivity. These mechanisms include the decrease of breakdown strength when insulation area [1], temperature [2] or duration of applied voltage [3] is increased.

Today state-of-the-art power capacitors utilize biaxially oriented isotactic polypropylene (BOPP) as the main insulation. By using high purity base materials a low dielectric loss factor ($\tan\delta \sim 10^{-4}$) [4] and with optimized film processing [5] a small-area breakdown strength above 700 V/ μm are achieved. Moreover, the capacitance of BOPP capacitors remains constant over a wide temperature range. These convincing dielectric properties however come at a cost of larger physical size, as the available energy density is constricted by the relatively low dielectric constant of BOPP (2.22–2.25) [6].

There is a continuing effort to manufacture higher energy density capacitors demanded for high energy- and power density applications, such as voltage source converters (VSCs) in wind turbines and offshore AC/DC converter stations. Energy density can be increased either by developing materials which can withstand higher electric fields during operation or by using higher permittivity dielectrics [7]. Increasing the operating field improves the energy density in a power of two, while for increased permittivity the increase is only linear. Moreover, a look at a list of common dielectric

plastics reveals that higher permittivity often comes at a cost of increased dielectric losses, which limits the energy density through the limited service temperature [8].

This paper presents our recent progress in developing and evaluating suitable and reproducible test methods to rank the dielectric performance of different capacitors films. In this study, two BOPP films from highly isotactic PP grades were evaluated, yet similar methods could also be used to characterize other dielectric films with self-healing capability. Properties of the films were determined using room- and high temperature large-area multiple breakdown measurements and DC electro-thermal and thermal ageing tests. The high temperature test method is further developed from [9] and the ageing test methodology is featured in a recent TDEI article [10]. The effects of ageing were characterized with wide-angle x-ray scattering (WAXS), differential scanning calorimetry (DSC) and large-area multiple breakdown measurements.

II. EXPERIMENTAL DETAILS

A. Capacitor materials tested

Two industrially produced BOPP films based on highly isotactic polypropylene grades with different molecular weights were characterized. The films were provided as film rolls and later on referred as PP-1 and PP-2. The average thicknesses of the both film were approximately 5.6 μm with a standard deviation of 0.1 μm .

B. Large-Area Multiple Breakdown Measurement

The large-area DC breakdown strength of the films was measured at room temperature and 100 °C using methodology detailed in e.g. [5]. Similar measurements at room temperature were also done on intact portions of electro-thermally and thermally aged films, as changes in breakdown behavior is an indicator of insulation ageing [11], with large measured areas revealing possible weak point formation or localized degradation. All breakdown measurements were done in ambient air.

Reference and high-temperature breakdown measurements were done on 12 samples and 6 samples of aged materials were measured. Each sample had an active area of 81 cm^2 . A commercial 12- μm Zn-Al metallized BOPP film was used as

electrodes and a voltage ramp of 30 V/s was applied to the “test capacitor” until no more discharges were detected.

Proper and independent breakdowns were selected from all recorded discharge events using a self-healing energy and voltage –based data qualification procedure [5]. Breakdown fields were calculated using the average thickness of the film samples and fitted with either single two-parameter or additively mixed two-subpopulation Weibull distributions. The first breakdowns from the reference measurements were also reviewed using video recordings and any possible non-breakdown discharges were manually removed in order to ensure highest reliability BDS results.

C. High Temperature Breakdown Measurement

Breakdown strength measurements at 100 °C were conducted in a 0.125 m³ test chamber, heated with 800 W PID-controlled heaters. As an improvement to our earlier high temperature large-area measurements [9], the temperature accuracy was improved by using an 800 W hot plate to heat the test fixture. The temperature of the test fixture was monitored in real time, and temperatures on top of the hotplate and test fixture were used to evaluate temperature gradient. The temperature accuracy was estimated to be better than ± 1.5 °C. The warm-up period before measurements was approximately 15 minutes.

C. DC Electro-Thermal Ageing Test

The ageing test was designed to simulate real capacitors where thermal and electrical stresses are present and the exposure to oxygen and moisture is limited. Oxidative degradation has been linked to decreasing breakdown strength of BOPP films [12], [13], against which capacitors are protected with vacuum impregnated oil or epoxy encapsulation. Based on IEC 60871-2 [14] the ageing test duration was 1000 hours (41 ⅔ days), and target stress levels were 100 °C and 100 V/μm for half and 100 °C and 200 V/μm for the rest of the samples. These were in the range of operating fields of metallized film PP capacitors [15] and in the < 1% probability range in 100 °C breakdown measurements. At the same time sheets of film were also aged thermally in the oven.

Eight parallel-plate test capacitors, as depicted in Fig 1a, were constructed from both materials. Metallized polyethylene naphthalate (PEN) films (1) were used as electrodes and the assembly was supported by 100 μm polyester films (2). The capacitors were sandwiched between two 3 mm × 400 mm × 400 mm glass plates and installed in test racks. The connections to Keithley 2290-5 power supplies and ground were realized by clamping the protruding edges of the electrode films between two aluminum bars (3).

An initial preconditioning step was done to expunge air from between the film layers. This consisted of energizing at 40 V_{DC}/μm for 24 hours during which the chamber was flooded with inert nitrogen gas. A nitrogen flow was maintained during ageing to retain overpressure and prevent air from leaking in. Nevertheless, the presence of trace air cannot be ruled out, as some air may have been left between the supporting polyester and glass layers. However, a limited

supply of air and moisture may be trapped between the layers of dry-type film capacitors during winding [16].

The test voltages of 561 and 1122 V_{DC} were applied after preconditioning and the chamber was heated to 100 °C. Temperature recorders inside the chamber indicated that the temperature stabilized to 96–100 °C after 8 hours, but after a cross-flow fan inside the oven broke at 600 hours the temperatures dropped to 90–94 °C. After ageing the capacitors were short-circuited and let to cool for a several days before further characterization.

III. RESULTS AND DISCUSSION

The Weibull distributions from large-area DC multiple breakdown measurements at room temperature (RT) and 100 °C (HT) measurements are displayed in Fig. 2a with results from thermo-electrically and thermally aged films displayed in Fig. 2b. Only films aged at 100 V/μm could be characterized, as those aged under 200 V/μm were destroyed by discharge activity, and no samples could be prepared from them. In the first days of the measurement, relentless self-healing activity in 200 V/μm capacitors was suspected since the supply current remained unstable for the first days of measurement, after which occasion current spikes were detected. Visual post-ageing inspection confirmed separation of active electrode areas caused by extensive demetallization, which was probably followed by occasional DC corona discharges. The damage is illustrated in Fig 1b. The results were rather surprising, since the 200 V/μm is still below those encountered in high energy density PP capacitors [15].

The materials had high initial crystallinity, which increased further due to thermally induced secondary crystallization [17] as evident in WAXS and DSC measurements summarized in Table I. The increased degree of crystallinity is also reflected in increasing melt peak temperatures, but presumably due to dominating ageing mechanism did not result in increasing breakdown strength. WAXS verified that the BOPP films contained only monoclinic α-form, as expected since β-form crystallinity in cast films, if any, transforms to α-form during biaxial orientation process [18].

Both non-aged films had similar breakdown behavior at room temperature and their characteristic 63.2% breakdown strength of 792–794 V/μm was high compared to other commercial and pilot-scale films measured using similar techniques, reported in e.g. [5]. Single Weibull distributions fit the RT data well, which suggest that one breakdown mechanism was operating [19].

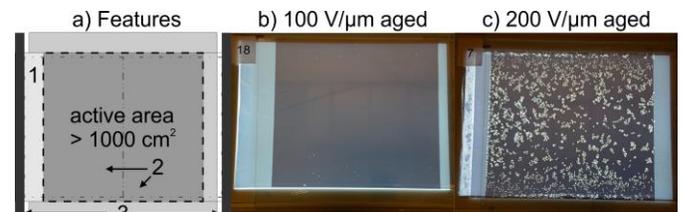


Fig 1a: Features of test capacitors for electro-thermal ageing (for labels please see text) and 1b: example photos of electro-thermally aged test capacitors. Capacitors aged at 200 V/μm were destroyed by self-healing breakdowns while those aged at 100 V/μm had a few isolated clearing.

TABLE I
THERMAL, MORPHOLOGICAL AND DIELECTRICAL FEATURES OF CAPACITOR INSULATION FILMS TESTED

	Melt onset °C	Melt peak °C	Crystallinity X _c (DSC)%	Crystallinity X _c (WAXS)%	63.2% breakdown strength (V/μm)	Breakdown points
PP-1 reference RT	154.5	166.6	60.3%	67.5%	792	277
PP-1 100 °C					633	351
PP-1 thermal	168.0	168.5	71.5%	76.3%	747	153
PP-1 thermo-electrical 100 V/μm	165.8	168.0	69.1%	73.7%	677	234
PP-2 reference RT	157.0	166.2	60.3%	65.5%	794	325
PP-2 100 °C					607	504
PP-2 thermal	167.4	169.0	66.8%	74.7%	721	126
PP-2 thermo-electrical 100 V/μm	169.6	170.0	68.8%	73.1%	718	245

At 100 °C the characteristic breakdown strength of both films decreased by approx. 18%, and differences between the films became evident, with PP-2 demonstrating lower breakdown fields in the 5–50% probability range. Nevertheless, both films still displayed similar breakdown behavior in the few percent probability region that may be the most interesting for practical capacitor applications.

The ~18% decrease in characteristic breakdown strength from RT to 100 °C is high compared to literature value of 11% presented in [2] or in our earlier studies [9]. This does not seem to be related to the degree of crystallinity as the relative degrees of crystallinity, both from DSC and WAXS measurements is close to the 61% reported in [20]. The DSC-based crystallinity is also high compared to 53% reported for high crystallinity BOPP film in [21]. In this sense, the relationship between crystallinity and high temperature breakdown strength seems to differ from HDPE, as reported in [22] and summarized in [17], lower crystallinity HDPE demonstrated higher breakdown strength at room temperature

decreasing rapidly with increasing temperature and eventually falling below that of higher crystallinity materials.

Both electro-thermally aged materials had decreased characteristic breakdown strength compared to room temperature reference measurements and even more interestingly the Weibull distribution was curved, suggesting that ageing had introduced a second defect-dominated breakdown mechanism [17], [19]. A similar phenomenon had occurred in thermally aged PP-2, and possibly also in PP-1, but the scarcity of data points in the low probability regions makes statistical analysis unreliable. These findings are in line with our earlier electro-thermal ageing tests, where a defect subpopulation appeared in laboratory-scale BOPP and nanosilica materials aged for 1000 hours at 80 °C and 100 V/μm [10]. Similar weak point formation has been reported for thermally aged BOPP films in [23], and for capacitor-grade films aged under AC voltages in oxygen-statured oils [24]. Thus, degradation accelerated by trace oxygen cannot be ruled out.

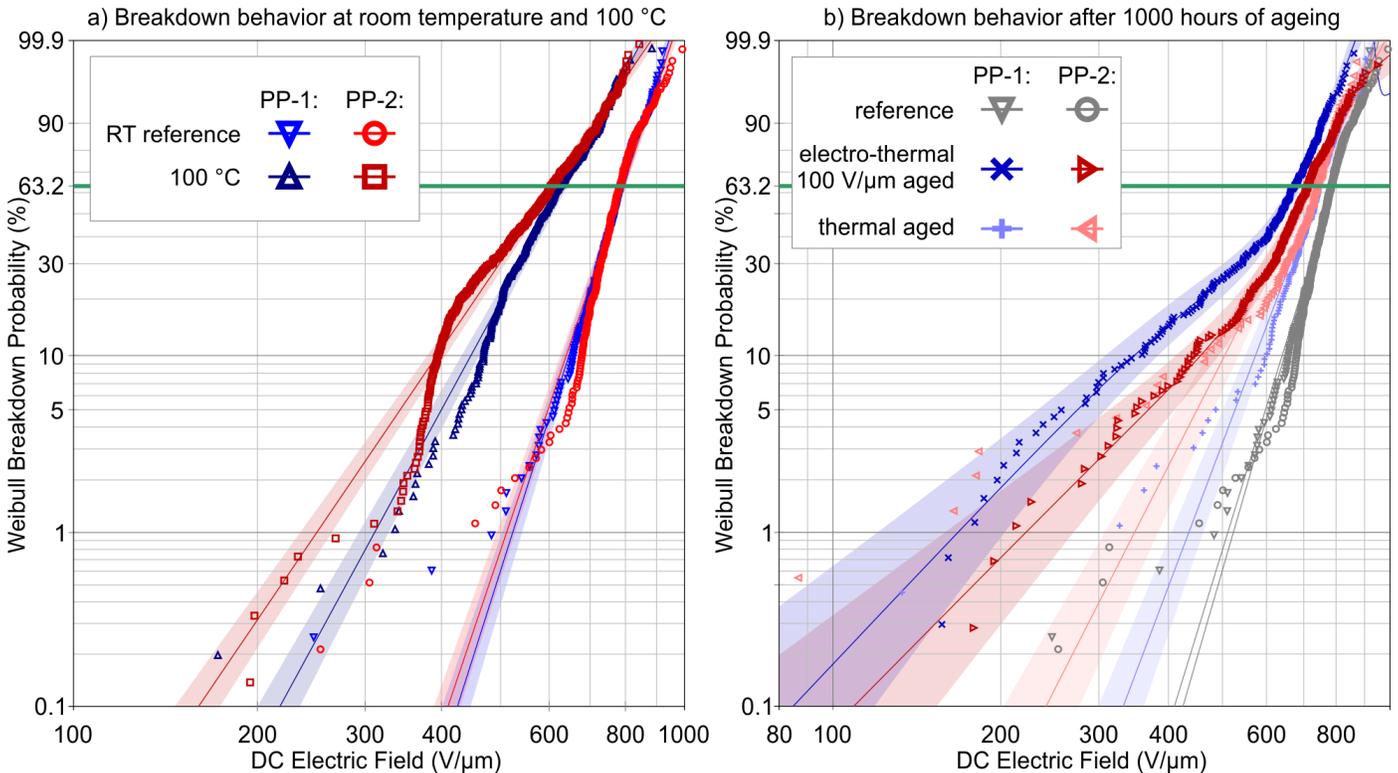


Fig. 2 Weibull distribution of breakdown voltages at a) room temperature, 100 °C and b) after thermo-electric and thermal ageing. Shaded areas represent 90% two-sided confidence intervals.

TABLE II
CAPACITANCES AND NUMBER OF BREAKDOWNS IN AGED CAPACITORS

		Capacitance (nF)	BD holes
Capacitors PP-1	1	293	53
	2	309	10
	3	294	9
	4	314	8
Capacitors PP-2	1	273	28
	2	289	26
	3	288	10
	4	286	22

The degree of ageing, as indicated by the number of self-healing breakdowns during long-term test was heavily dependent on the electric field stress: the 100 V/ μm capacitors had relatively few breakdowns while the 200 V/ μm units were destroyed by them. In literature [25] voltage stresses have also been shown to have a more negative effect on insulation life than temperature. The breakdowns were distributed rather evenly on the active areas, which calls into question major field enhancements at electrode edges. The number of breakdowns and capacitances in 100 V/ μm test capacitors are reported in Table II. The capacitances were line in with the film thicknesses and active areas, from which it was deduced that no air gap existed between the film under test and electrodes. The capacitances were measured several days after voltage was turned off, revealing that the electrodes had adhered to the BOPP films. Lastly, no change in film thicknesses after ageing was detected in measurements with a precision thickness gauge (accuracy of 0.1 μm).

IV. CONCLUSIONS

BOPP films from highly isotactic base PP grades were compared. Breakdown measurements at high temperature and both thermal and thermo-electrical ageing tests brought forward differences in two BOPP films which displayed similar breakdown performance at room temperature. In breakdown measurements at 100 °C PP-1 film demonstrated slightly higher breakdown voltages but in thermo-electric ageing tests this “order of goodness” reversed, with aged samples of PP-2 material having higher breakdown strength. This highlights the importance of both high temperature and ageing tests during material research and development.

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