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Large-area Approach to Evaluate DC Electro-thermal Ageing Behavior of BOPP Thin Films for Capacitor Insulation Systems

Mikael Ritamäki, Ilkka Rytöluoto, Kari Lahti

Tampere University of Technology
Department of Electrical Engineering
P.O. Box 692
FI-33101 Tampere, Finland

Torvald Vestberg

Borealis Polymers Oy
P.O. Box 330,
FI-06101 Porvoo, Finland

Satu Pasanen and Timo Flyktman

VTT Technical Research Centre of Finland Ltd
P.O. Box 1300
FI-33101 Tampere, Finland

ABSTRACT

A large-area electro-thermal ageing test setup was developed and utilized to age several laboratory-scale biaxially oriented polypropylene (BOPP)–hydrophobic silica nanocomposite films. The films were aged in test capacitors with self-healing metallized film electrodes, which enabled the ageing test to continue beyond the first breakdowns. Eight different films were aged for 1000 hours under 100 V/ μm DC stress at 75 °C, and large-area DC breakdown measurements, dielectric spectroscopy, gel permeation chromatography (GPC), differential scanning calorimetry (DSC) and dielectric spectroscopy were used to detect localized and bulk degradation after the ageing period. The effects of antioxidant contents, different PP grades and compounder screw speed were evaluated. Material characterization indicates no bulk degradation had occurred during ageing, which was associated with moderate temperature stress and inert nitrogen atmosphere. On the other hand, low-field breakdowns (weak points) were observed in all but two of the aged materials, indicating that ageing was dominated by localized degradation which may have introduced new breakdown mechanisms. Weak points were also measured in a similarly aged commercial capacitor-grade BOPP film aged at a lower field, supporting this conclusion. The importance of long-term characterization in material development is demonstrated, and it is shown that long-term properties of the evaluated nanocomposites were at least on the same level compared to neat BOPP films.

Index Terms — Aging, films, polymers, electric breakdown, nanocomposites, dielectric measurement, dielectric spectroscopy, thermal analysis, molecular weight.

1 INTRODUCTION

AGEING of electrical insulation, as presented by Fothergill in [1] is a continuous process taking place throughout insulation during its service life. Ageing may lead to degradation, which in turn can lead to electric breakdown during service life, and so the three are mutually linked. In addition to changes in breakdown behavior [2], [3] ageing of polymeric capacitor insulation systems has been associated

with chemical and physical changes, examples of which include oxidation and changing morphological properties, elongation [4, 5], molecular-weight distribution and crystallinity [6, 7]. Another important factor is space charge accumulation, which can be seen both as a result and a cause of ageing [8]. For power capacitors individual breakdowns of the film dielectrics are not always fatal, as the insulations can be made defect-tolerant by the use of self-healing metallized electrodes [9], [10]. However the causal link between ageing and breakdown makes it possible to use changes in breakdown behavior as an indicator of ageing progression. In

[11] it has been reported that in general the breakdown strength of insulation remains rather unchanged, only to decrease drastically in the final moments before breakdown. More specifically for capacitor dielectric films increasing or decreasing overall breakdown strengths and weak point formation have been reported in [5]. While the breakdown strength of insulation tends to follow the Weibull distribution [12], weak point formation justifying the use of multiple Weibull subpopulations indicates ageing may introduce new breakdown mechanisms [13].

Appearance of low-probability low-field breakdowns or 'weak points' in AC electrothermally aged polypropylene (PP) film capacitor elements has been demonstrated in the recent work of Guillermin et al [14]. In their work this was associated with partial discharging (PD) at the film edges above an electric field threshold due to field enhancement effects. In general accelerated ageing experiments should be conducted using stress levels which do not introduce new ageing mechanisms, such as PD, which has been reported to be the fastest ageing mechanism of polymeric insulation [15, 16]. Appearance of weak points in PP film combined with an increase in its characteristic breakdown strength has been reported by Umemura et al [5] for films aged in PP/kraft paper (KP)/PP multilayer model capacitors for 370 days at 1.4× nominal voltage and 90 °C, in which the weak points were associated with a non-specified morphological change. On the other hand Sebillotte et al [2] report no significant changes in the breakdown behavior of PP sheets AC aged in pure impregnating liquids for 1800 hours at 80 °C. Presence of oxygen and/or additives however decreased the breakdown strength depending on the liquid used. The effect of impregnation on the ageing behavior of PP films is hardly surprising, since physical interactions such as swelling and dissolution between impregnating liquid and PP have been reported in literature [4, 9, 17]. For low and medium voltage capacitors thin metallized electrodes are often used [10]. This provides defect-tolerance via electrode self-healing, but degradation, principally corrosion of this thin metallization has been reported [10, 18, 19] as one of the dominant ageing phenomena in such capacitors. Because of different ageing phenomena in different types of capacitors, and also due to the effects of capacitor manufacturing, the ageing of film insulation systems cannot be determined solely by the film property, necessitating endurance testing of finalized capacitor products, as outlined in IEC 60871-2 [20].

At present at least two unknowns have been introduced in the field of ageing in polymeric capacitor insulations: new insulating materials [21], such as polymer nanocomposites (PNCs) [22] and nonsinusoidal waveforms, such as DC and repeating voltage transients [16]. Research on polymer nanocomposite systems [23, 24] have shown different ageing behavior compared to neat materials and the (nano)particle interfaces may introduce new degradation mechanisms [25, 26]. Ongoing research [27] at TUT has also revealed differences in the DC voltage endurance coefficient of BOPP-hydrophobic silica nanocomposites compared to neat materials. PNCs have also shown improved electrical properties, viz. enhanced partial discharge resistance [28, 29]. Improved partial discharge resistance is especially beneficial as electrical insulations, capacitors included, are nowadays

utilized in applications where repetitive voltage transients from power electronics are present, which have been associated with non-conventional and highly accelerated ageing behavior [15], [16, 30].

Material development would benefit from a practical way to evaluate the long-term properties of laboratory scale materials before expensive and time-consuming pilot-line production. Accurate evaluation of the long-term properties of a polymer-based film is however complicated, requiring knowledge of which type of capacitor it will be used in and also to what kind of stresses it will be subjected to. During material development these are rarely feasible. Properly wound up model capacitors would also require more film than can be produced using laboratory-scale film stretchers. The use of traditional metal foil is also problematic, as ageing tests cannot usually continue beyond the first permanent breakdowns.

The need to evaluate the long-term properties of laboratory-scale materials has led to the development of an ageing test system using stacked plate-type capacitors with self-healing metallized film electrodes. The system was used to age several BOPP-silica nanocomposites under 100 V/μm DC stress at 75 °C for 1000 hours, followed by extensive material characterization to detect ageing-induced changes. Two types of isotactic PP were compounded with hydrophobic fumed silica, and for one PP type compounding screw speed and antioxidant contents were varied.

2 EXPERIMENTAL DETAILS

2.1 MATERIAL DETAILS

The laboratory scale materials were produced at VTT Technical Research Centre of Finland Ltd. Two grades of isotactic polypropylene provided by Borealis Polymers Oy, PP1 and PP2, were used as the base materials. 0.9 wt% of hydrophobic fumed silica Aerosil® R 812 S was added in the nano-silica compounds. Aerosil® R 812 S is a fumed silica aftertreated with hexamethyldisilazane (HMDS). 0.35 wt% of Irganox 1010 powder antioxidant and 0.0075 wt% of calcium stearate was added to all compounds. In antioxidant series the effects of low (0.20 wt%, PP1-LoAO) and high (0.60 wt%, PP1-HiAO) antioxidant contents were evaluated. The raw materials were dried at 70 °C and vacuum treated before processing. All the materials were compounded similarly with a Berstorff ZE 25/48D twin-screw compounding using a highly mixing screw configuration and same processing parameters, therefore the materials share the same thermal history. In one of the trials, the compounding screw speed was varied from the nominal 300 1/min, resulting in the low (PP1-LoSS, 200 1/min) and high (PP1-HiSS, 350 1/min) screw speed variants. The materials details and processing parameters are summarized in Table 1.

The compounding strands were cooled, pelletized and dried and thereafter extruded into cast films with a Brabender Plasticorder extruder using a flat die. To prevent contamination, compounding and cast film extrusion were done in a clean room environment and the machines were cleaned before processing. All the cast films were similar in appearance. Cast films were biaxially oriented (stretching ratio of 5.8 × 5.8) with a Brückner KARO IV biaxial stretcher

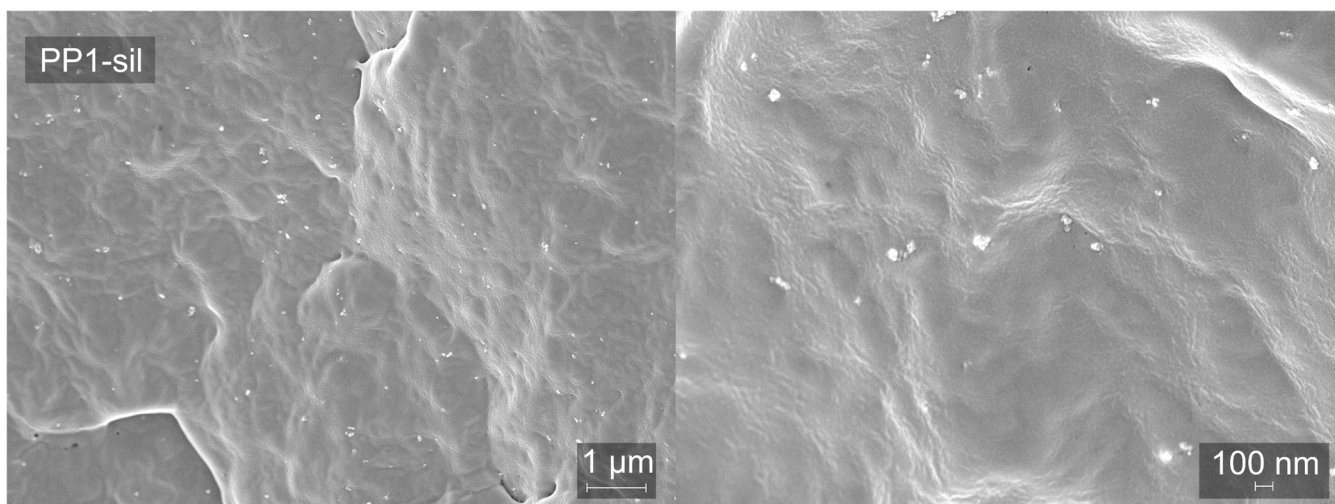


Figure 1. Cross-sectional SEM images of cryofractured PP1-sil (0.9 wt% silica) cast film at two zoom levels, taken at VTT Technical Research Centre of Finland Ltd. These images were representative of the nanoparticle distribution and dispersion in all of the evaluated nanocomposites. The effects of screw speed variation were not clear. Nanoparticle distribution and dispersion were adequate and similar to what has been measured in our earlier nanocomposite research projects using the same hydrophobic fumed silica.

at the set temperature of 161 °C, except films PP1-HiSS and PP2-LoSS, which were oriented at the set temperature of 157 °C. PP1-HiSS oriented at 161 °C had a significantly decreased large-area breakdown strength and it was decided to re-do PP1-LoSS at 157 °C as well for reference purposes. This was associated with excessive melting during orientation, which had also manifested in significantly decreased stretching forces. Material details are presented in Table 1. The average thicknesses of the reference samples demonstrate that the target thickness of $\leq 10 \mu\text{m}$ was reached. Cast film samples of the nanocomposite materials were cryofractured in liquid nitrogen and the nanoparticle dispersions were evaluated by cross-sectional imaging of the samples by Zeiss® Merlin-42-63 field-emission scanning electron microscope (FE-SEM). There were no clear differences between the materials, and the overall dispersion was considered adequate and in line with our previous results [31] using the same hydrophobic silica. Representative SEM images of PP1-sil are presented in Figure 1.

In addition to the laboratory-scale films, a commercial 14.4 μm capacitor-grade BOPP film was aged both thermally and electro-thermally. This film is the same used in our earlier thermal ageing experiments in [32], [33]. Film rolls were stored under normal room temperature conditions.

2.2 SAMPLE PREPARATION AND TEST CAPACITORS

Film samples were prepared for use as dielectrics in test capacitors. The test capacitor dielectric was either four 110 mm \times 145 mm samples from laboratory-scale films or a 300 mm \times 400 mm sheet of the commercial capacitor-grade film.

Four 380 mm \times 90 mm sheets of commercial Zn-Al metallized capacitor-grade 12 μm BOPP film were used as electrodes, the self-healing capability of which enabled the test capacitors to tolerate breakdowns during ageing [3, 34]. The film arrangement resulted in an active area of approximately 400 cm². This also left an at least 10 mm free margin around the film samples. The test capacitors were sandwiched between two 3 mm thick 400 mm \times 400 mm glass plates. In preliminary high temperature tests in ambient air the metallized BOPP electrode film adhered to the glass plates. To prevent this, A4-sized 100 μm polyester sheets were placed between test capacitor and the glass plates. The ageing test capacitor structure is depicted in Figure 2a.

20 test capacitors were produced; 16 using laboratory-scale materials and 4 using the commercial capacitor-grade film. The test capacitors were loaded into two custom-made test racks and the protruding metalized film edges were sandwiched between two flat aluminum bars. To evaluate the effects of thermal stress only, additional sheets of the commercial capacitor-grade film were placed on top of the test rack assembly in paper envelopes.

2.3 AGEING SETUP

The ageing test circuit, illustrated Figure 2b, was designed to minimize the demetallized area during self-healings which is related to the inverse of self-healing energy, which in turn is inversely proportional to the capacitance partaking in the self-healing process [3]. This capacitance was decreased by connecting the high voltage bar electrodes to a common feedthrough via 10 k Ω low inductance thick film resistors. Test voltage was supplied via Keithley 2290E-5 high voltage

Table 1. Specifications of the laboratory-scale materials evaluated. All the materials were produced at VTT Technical Research Centre of Finland Ltd.

Material	Nanosilica (wt%)	Antioxidant (wt%)	Screw speed	Bi-axial stretching ratio	Bi-axial orientation set temperature (°C)	Film thickness (μm)	
						Mean	SD
PP1-ref	-	0.35	normal	5.8 \times 5.8	161	9.97	1.01
PP2-ref	-	0.35	normal	5.8 \times 5.8	161	10.24	0.87
PP1-sil	0.9	0.35	normal	5.8 \times 5.8	161	8.47	0.79
PP2-sil	0.9	0.35	normal	5.8 \times 5.8	161	9.49	0.78
PP1-LoAO	-	0.2	normal	5.8 \times 5.8	161	8.96	0.99
PP1-HiAO	-	0.6	normal	5.8 \times 5.8	161	10.31	0.84
PP1-LoSS	0.9	0.35	low	5.8 \times 5.8	157	9.59	0.76
PP1-HiSS	0.9	0.35	high	5.8 \times 5.8	157	9.16	0.65

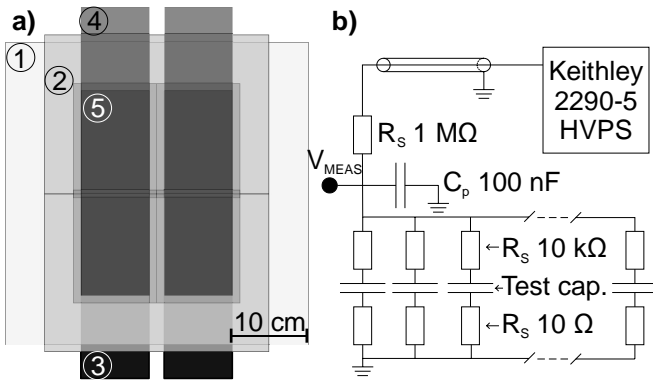


Figure 2. Ageing experiment test capacitor (a) and circuit (b). Parts: (1) glass plates (2) polyester support films, two on each side (3) bottom electrode BOPP films (4) top electrode films (5) four film samples. For commercial capacitor-grade film four samples were replaced with one large sheet. Voltage was measured from V_{MEAS} .

power supply (HVPS) having a manufacturer reported maximum output ripple of $0.1 V_{RMS}$. An external RC-filter was used to smooth out transients. Test voltage was ramped up in 50 V increments, giving the HVPS current, monitored via the HVPS current meter (resolution of 0.001 mA), time to stabilize after each step. This was done to limit the inrush current to the highly capacitive load and to prevent degradation of the metallized electrode film – aluminum electrode interface caused by Joule heating in the resistive aluminum oxide layers.

The test capacitor racks were placed in a gas-tight environmental chamber or ‘oven’ detailed in [33], and a constant nitrogen flow of approximately 40 ml/min was fed into the oven through an Aalborg flowmeter to prevent ambient oxygen and moisture from seeping in. During the first 72 hours over 500 liters of nitrogen was fed into the oven to displace air inside. To push air out from between the film layers a voltage of 340 V was applied to the test capacitors during this period, which in preliminary tests had been deemed sufficient to remove visible air bubbles from between film layers. Temperature was maintained with two 400 W heaters and a cross-flow fan, the latter being necessary in reducing the temperature gradient [33]. The ageing setup is illustrated in Figure 3.

After ageing the capacitances of the individual test capacitors were measured (at 1 kHz and 2 V_{AC}) with GW Instek LCR-8101G LCR meter. The results are reported in Table 2. The measured capacitances were in line with the film thicknesses, which is interpreted to indicate proper adhesion

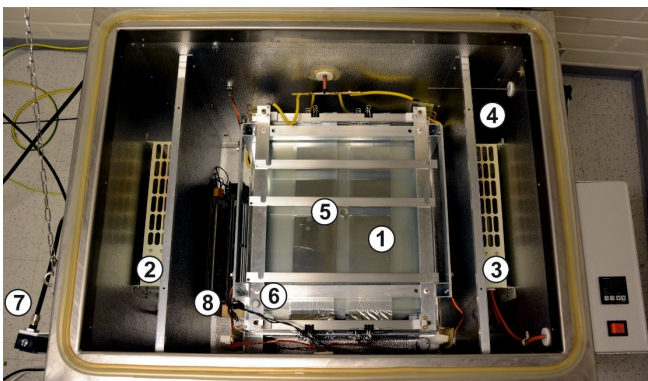


Figure 3. Ageing test setup, parts shown: (1) test racks; (2 & 3) heaters; (4) controller PT100 sensor; (5 & 6) temperature loggers placed on top; (7) nitrogen regulator and flowmeter; (8) cross-flow fan.

of electrode and sample films. The series resistance of the test capacitors varied considerably not only between samples but also between consecutive measurements on the same test capacitors. This is presumed to result from contact issues caused by aluminum oxidation.

2.4 ELECTRO-THERMAL STRESS LEVELS

Target electro-thermal stress levels were 100 V/μm for laboratory-scale films and 75 °C. Oven temperatures were recorded from bottom, middle and top of the test racks with iButton temperature loggers every three hours. Based on these measurements the temperature accuracy was estimated to be better than 0.5 °C and temperature variations after initial stabilization were minimal.

The test voltage of 950 V was determined based on the average thickness (9.52 μm) of the reference laboratory-scale samples, as the thickness of aged samples was determined after ageing. This was done to avoid any extra film handling before the ageing experiment. Voltage was measured with a North Star High Voltage PVM-1 1:2000 probe and a Fluke 179 multimeter, which after stabilization indicated a voltage of 948.6 V. Realized electric field stress levels were determined from the 81 cm² samples to be measured using the large-area breakdown measurement [35–37], more detailed in subsection 2.6. LE1000-1 high-precision thickness gauge with a manufacturer-reported accuracy of 0.1 μm and a ball-point measurement tip was used for all the thickness measurements, and only the thicknesses from areas with breakdowns during the breakdown measurement were used for the calculation of mean thickness. The material-specific electric field stress levels are reported in Figure 4.

2.5 AGEING TEST PROGRESSION

The test duration of 1000 hours at the target stress levels was loosely based on IEC TS 60871-2 [20]. The initial 72-hour gas exchange period took place at room temperature, after which the heating was turned on together with the full test voltage. Temperature logs indicate that the oven temperature stabilized to 75 °C after 5 hours. The PID controller had been tuned beforehand and thus no significant overshoot was expected.

After 1000 hours the supply side of the RC filter was grounded and the oven was let to cool for several days. Grounding was done via its 1 MΩ resistance to give space charge time to dissipate as based on literature rapid grounding of energized XLPE cables has been associated with electrical

Table 2. Features of test capacitors after ageing: number of self-healing breakdowns and series capacitances and -resistances. Electrical parameters were measured at 1 kHz and 2 V_{AC}. Resistive component varied between measurements, which is interpreted to indicate imperfect contact at the bar electrode – metallization interface.

Cap.	Material	BDs	C_s (nF)	R_s (Ω)	Cap.	Material	BDs	C_s (nF)	R_s (Ω)
1	PP1-ref	0	95	212	11	PP1-HiAO	0	93	93
2	PP1-ref	0	88	73	12	PP1-HiAO	0	98	165
3	PP2-ref	1	84	173	13	PP1-LoSS	1	98	75
4	PP2-ref	0	82	133	14	PP1-LoSS	2	92	86
5	PP1-sil	5	107	40	15	PP1-HiSS	1	90	23
6	PP1-sil	0	110	89	16	PP1-HiSS	1	92	73
7	PP2-sil	0	89	386	17	cap-BOPP	0	67	86
8	PP2-sil	0	92	47	18	cap-BOPP	0	64	122
9	PP1-LoAO	2	92	185	19	cap-BOPP	0	66	130
10	PP1-LoAO	2	100	50	20	cap-BOPP	0	63	76

degradation (treeing) [38]. Nitrogen flow remained on until room temperature was reached. Logs indicate that the gas temperature had dropped below 30 °C in 20 hours

The test capacitors were visually inspected for breakdowns. Laboratory-scale materials had a total of 15 breakdowns and the commercial capacitor-grade film had none. The breakdowns were located seemingly randomly on the film surface, with no breakdowns at electrode edges. The amount of breakdowns in each test capacitor has been presented in Table 2.

The test circuit was fitted with discharge logging system capable of measuring μs -scale current pulses in individual test capacitors. As a discharge or a breakdown took place, the dissipated energy would be replaced by a fast current pulse. These pulses would be detected by actively comparing the voltage difference across the 10 Ω low-inductance resistors to an adjustable voltage reference. The 20-channel measurement circuit consisted of peak hold rectifiers, comparators and a FPGA-based detector. Based on initial testing this setup would be capable of detecting pulses with amplitude of approximately 100 mA.

Analysis of the recorded data indicated excessive discharging in test capacitors 2 (80 events) and 4 (105 events). Visual inspection of these aged samples however revealed that neither of these samples had any breakdown holes. This discrepancy indicated that discharging may have taken place in the aluminum bar – metallized film electrode. This was further supported by discernible demetallization in the film-bar interface regions.

2.6 MATERIAL CHARACTERIZATION

Large-area DC breakdown behavior of all the materials and the complex dielectric permittivity of the laboratory-scale materials was measured before and after ageing. Additionally, differential scanning calorimetry (DSC) and gel permeation chromatography (GPC) measurements were conducted for all the PPI-based materials.

Breakdown measurements were conducted using large-area multiple breakdown methodology detailed in e.g. [35]–[37]. 12 μm metallized BOPP film was used as electrodes and measurements were conducted in dielectric oil (Shell Oil Diala). A fast voltage ramp-up of 400 V/s up to 2 kV followed by a slower ramp at 30 V/s was used, and measurement was continued until no breakdowns were detected.

A self-healing discharge energy and breakdown voltage – based data qualification procedure detailed in e.g. [37] was used to validate each recorded event. For laboratory-scale films the breakdown fields were determined manually by combining the 25-point thickness data with a video recording of the measurement. To reach a both statistically and visually adequate fit the qualified events were fitted to either single two-parameter or additively mixed two-subpopulation Weibull-distributions [13]. For a few materials individual outliers were excluded from Weibull parameter estimation to reach an adequate fit with the large majority of data points.

The complex dielectric permittivity of reference and aged laboratory scale films was measured using Novocontrol Alpha A –analyzer. Measurements were done using a shielded sample cell BDS 1200 with a test voltage of 1 V_{RMS} , the sample being sandwiched between gold-plated 40 mm \varnothing

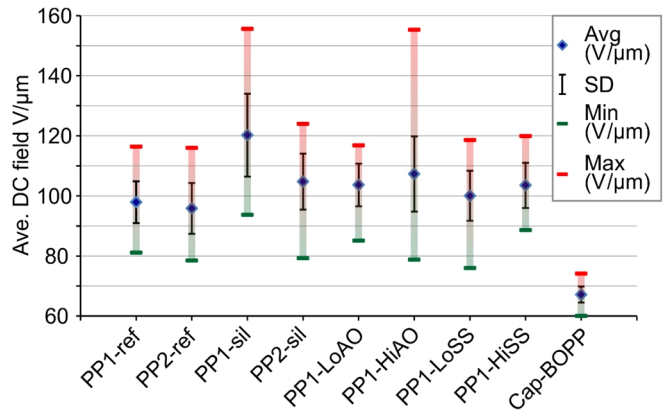


Figure 4. Electric field stresses for different material, with averages, extreme values and standard deviations. Target field for laboratory-scale materials was 100 V/μm.

bottom and 10 mm \varnothing top electrodes. Golden electrodes ($d=50$ nm) with a diameter of 12 mm were evaporated on two approximately 32 \times 32 mm film samples using Leybold L560E E-Beam evaporator. Before evaporation the samples were dried overnight in a vacuum flask at 35 °C, then washed twice in 2-propanol and fastened to a metal sample holder, with the latter two steps conducted in a clean room to prevent contamination by airborne particles. For accurate determination of the relative permittivity, the thicknesses of the samples were measured from four points around the evaporated electrode.

To evaluate the repeatability of the measurement two samples of each film were prepared and measured twice. The average and standard deviation of these four measurements 50 Hz are reported. For some materials one of the two samples had a hole, recognized by indentures and loss characteristics typical of surface conduction [39] (other sample displaying rather flat loss levels in the measured frequency range). The measurement data from the broken samples was not used in any of the analyses.

3 RESULTS

3.1 LAB-SCALE MATERIALS

The Weibull distributions of the large-area multiple breakdown measurement results of laboratory-scale materials are presented in Figure 5 and their distribution parameters are given in Table 4. The absolute values of relative permittivity and the loss tangent ($\tan \delta$) at 50 Hz together with the results from DSC and GPC measurements on PP1 materials are presented in Table 3. Of the DSC measurement melting T_m and crystallization T_{cr} temperatures and the calculated degree of crystallinity X_c are presented. Of the GPC results the number- M_n , weight- M_w , z- M_z and viscosity- M_v averaged molar masses, the polydispersity PD and antioxidant contents in ppm are reported.

3.2 COMMERCIAL BOPP FILM

The Weibull distributions from large-area multiple breakdown measurements on reference, electro-thermally aged and also thermally aged commercial capacitor-grade film are presented in Figure 6 and the Weibull distribution parameters are shown in Table 5. The reference measurements consist of 30 samples while the thermal and electro-thermally aged results are from 12 and 11 samples respectively. Due to the uniform thickness profile in the

commercial capacitor-grade films no manual thickness correction was used. The reference measurement results have been previously presented in [32], [33].

4 DISCUSSION

4.1 LABORATORY-SCALE MATERIALS

The large-area DC breakdown strength of the neat PP and the nanocomposite films was in line with our earlier studies [31, 37] using the same hydrophobic silica. The overall large-area breakdown strength was also in line with the commercial BOPP film, and also largely in line with what is reported for eight non-metallized BOPP films in [37]. The breakdown behavior of all materials was homogeneous, despite the one statistically insignificant outlying low-field breakdown in PP1-sil and PP2-sil references. Of the nanosilica series (Figure 5a) PP2-sil had the highest breakdown characteristic strength, yet this apparent improvement was accompanied with a decreased distribution homogeneity compared to the other materials of the series. From the antioxidant series the low antioxidant variant had slightly decreased breakdown strength, but more interestingly the breakdown strength of the high-AO variant was in line with the reference. This was also the case in the screw speed series, where the low variant was slightly lower. All in all the overall differences between the materials were considered small compared to for example the effects of increasing silica content beyond 1 % [31].

The small amount (0.9 wt%) of silica was chosen since increasing nanosilica filler loading has been associated with decreasing characteristic breakdown strength in BOPP-silica nanocomposite films [31] and also in polyethylene matrices [40]. The hydrophobicity of the silica limits water absorption, recognized as one of the main challenges in nanodielectrics research and which has been associated with deteriorating dielectric properties [25] and sources therein. A commercial product available in large quantities was used for its suitability for pilot and industrial-scale manufacturing. The compounder screw speed trial was based on our earlier studies with 4.5 wt% hydrophobic silica BOPP-nanocomposites [41], where decreased screw speed resulted in significantly higher overall breakdown strength. In this

study however no such clear effect was observed, which may be associated with lower silica loading.

The complex dielectric permittivity of the silica- and screw speed series reference materials was similar, indicating that neither the addition of 0.9 wt% nano-silica nor the screw speed markedly affect the loss levels. However PP1-LoAO from the antioxidant series, both reference and aged, had the lowest dielectric loss levels in the frequency range of 0.1–100k Hz. The decrease in the loss levels with a lighter antioxidant loading was in line with the study of Umemura *et al.* [42], based on which more pronounced differences could be expected if measurements were repeated at elevated temperature.

Notable differences can be seen in the DSC-results of the screw speed series, with both variants, but especially PP1-HiSS displaying a lower degree of crystallinity accompanied with lower melting and crystallization temperatures. This explains the difficulties in successfully bi-axially orienting these films at 161 °C (Subsection 2.2). Worth mentioning is also that a similar, albeit slighter, decrease was measurable between PP1-ref and PP1-sil. The degree of crystallinity does not however correlate with the dielectric loss levels or the DC large-area dielectric breakdown strength, contrary to [43] where lower dielectric loss levels are reported for a high crystallinity BOPP film. The GPC results however do not indicate significant changes in the averaged molecular weights, which may indicate that the differences in DSC results originate from morphological changes. It is therefore postulated, that in future the orientation parameters should be optimized individually for each material to obtain the best and most representative dielectric performance for each material.

4.2 EFFECTS OF AGEING

Distinct differences in the DC large-area breakdown behavior were seen between the electro-thermally aged and the reference films. The characteristic 63.2 % breakdown strength of all the materials had decreased after electro-thermal ageing, and all of them except PP1-HiAO and PP1-HiSS displayed curvature towards lower fields in the low probability region. The two aforementioned materials in turn displayed increased scatter in breakdown voltages, and thus significantly decreased Weibull β . For capacitor applications

Table 3. Physical and electrical properties of the laboratory-scale films: melting and crystallization temperatures and total crystallinity from DSC measurements; molar masses, polydispersity and amount of antioxidants from on GPC measurements and complex dielectric permittivity at 50 Hz. Electro-thermal DC ageing did not significantly affect these bulk material properties. *) *only one sample was measured.*

<i>Not aged</i>	T_m °C	T_{cr} °C	X_c %	M_n /1000	M_w /1000	M_z /1000	M_v /1000	PD	Antioxidants (ppm)	$ \epsilon_r $ (50 Hz)	SD	$\tan \delta (\times 10^{-4})$ (50 Hz)	SD ($\times 10^{-4}$)
PP1-ref	159.9	115.5	48	56.4	257	600	223	4.57	2350	2.10	*	7.0	0.1
PP2-ref										2.13	0.04	5.8	0.7
PP1-sil	159.6	114.6	47	58.3	256	596	221	4.39	2140	2.11	*	5.9	0.1
PP2-sil										2.12	0.01	4.5	0.3
PP1-LoAO	159.0	116.3	47	47.0	232	509	202	4.93	840	2.15	0.01	3.6	0.2
PP1-HiAO	158.9	114.2	45	59.6	260	608	224	4.36	3430	2.18	0.01	6.6	0.2
PP1-LoSS	156.3	113.1	44	55.1	263	646	226	4.78	2910	2.11	0.00	5.0	0.2
PP1-HiSS	156.7	112.6	41	53.5	234	551	202	4.36	3600	2.06	0.02	7.4	0.1
<i>Aged</i>													
PP1-ref	160.6	115.6	44	57.9	259	596	221	4.47	2340	2.15	0.03	6.0	0.7
PP2-ref										2.18	0.02	4.3	0.9
PP1-sil	160.8	114.6	44	59.6	260	595	225	4.35	2320	2.19	0.03	5.6	0.5
PP2-sil										2.16	0.01	4.5	0.1
PP1-LoAO	158.6	116.9	48	58.3	239	529	209	4.09	1180	2.18	0.01	4.2	0.3
PP1-HiAO	158.3	114.7	45	61.8	268	642	231	4.33	4130	2.20	0.01	5.6	0.1
PP1-LoSS	157.7	113.4	43	54.6	272	697	232	4.98	3040	2.16	0.02	4.7	0.3
PP1-HiSS	155.0	113.4	41	53.8	229	528	198	4.26	2950	2.18	0.00	5.4	0.6

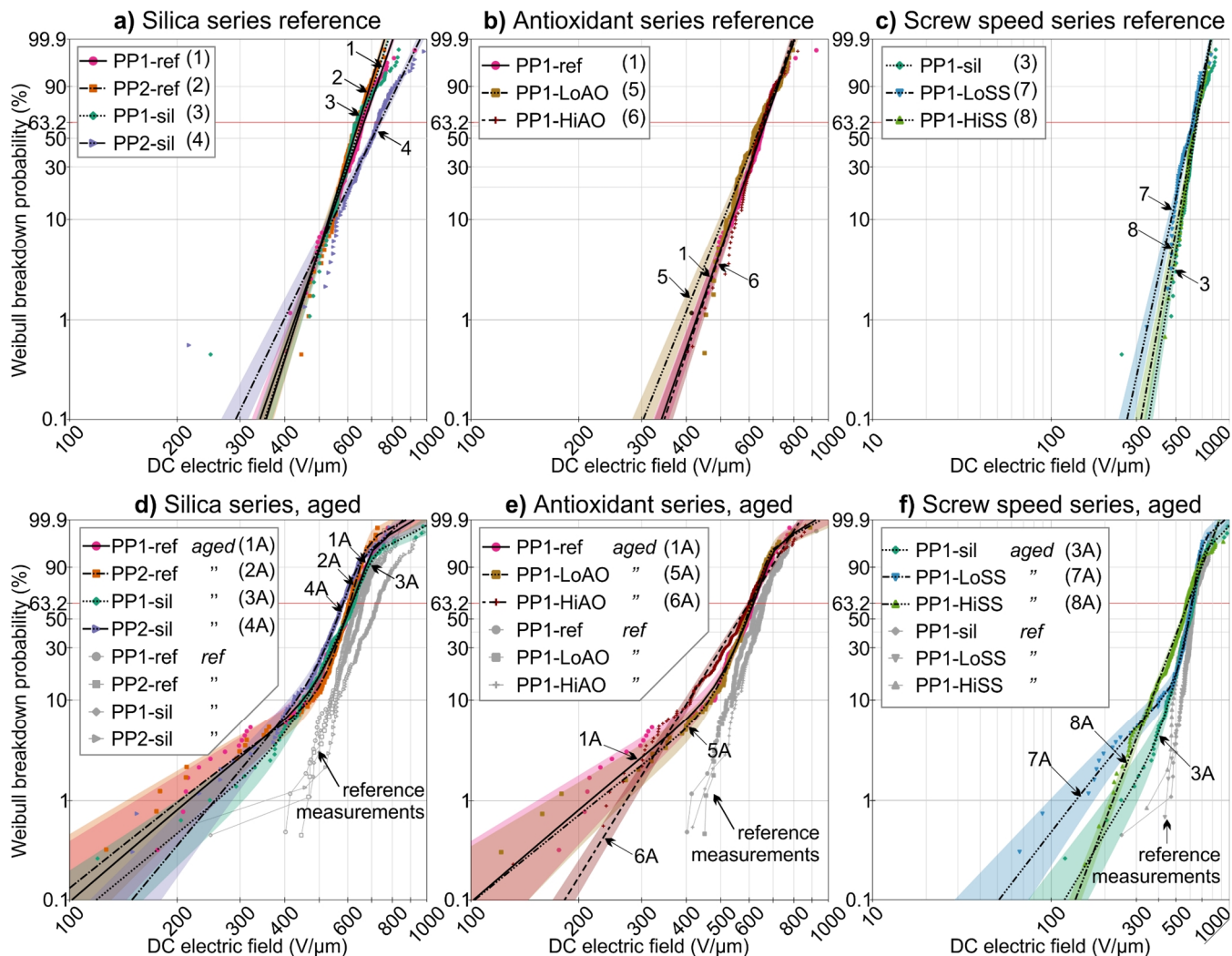


Figure 5. Large-area DC multi-breakdown performance of aged and reference films. a & d) The effects of two grades of PP base polymer and 0.9 wt-% of hydrophobic nano-silica. Low-field weak points are seen in all four aged material, and in comparison with the other three the characteristic breakdown strength of PP2-sil decreased rather notably. b & e) The effects of high and low antioxidant contents and c & f) the effects of high and low compounder screw speed.

the weak points are the most interesting, albeit difficult to measure in statistically significant quantities using traditional small-area single-breakdown methods. However, the large-area measurement method used is capable of detecting these statistically rare events. Similar ‘weak point’ formation has been reported in neat BOPP films thermally aged using a 1000 hour step stress from 50 to 110 °C [32, 33], in which however weak points were not measured after the highest stresses, raising questions whether the changes were ‘ageing’ or thermal conditioning or if they were related to varying film quality.

It is remarkable that the PP1 based nanocomposites displayed similar breakdown behavior compared to its un-filled reference, even though referring to Figure 4 PP1-sil was aged in a higher mean electric field. This may indicate that PP1-silica is more resistant to ageing compared to its un-filled reference. One possible explanation could be improved partial discharge (PD) resistance in silica-filled material [29], but this cannot be verified since the PD levels in the test capacitors were not measured, and since although silica filler has been associated with improved PD resistance, it is not known if this benefit remains with small (0.9 wt%) amounts of silica. On the contrary the breakdown strength of the aged PP2-sil film was slightly lower than its un-filled reference, in comparison with reference measurements in which PP2-sil

demonstrated highest breakdown strength. This illustrates the importance of both long-term testing and evaluating different PP grades when preparing nanocomposites.

No changes in the bulk material properties were detected in the aged film samples, as demonstrated by the essentially unchanged GPC and DSC measurement results. GPC measurements would have indicated if the average molecular weight had changed, which would have indicated that chain scission or crosslinking had occurred. However, based on the results in Table 3 it is evident that neither of the aforementioned had occurred in significant quantity. The variations seen may as well result from measurement uncertainty, caused by a limited quantity of aged film used in the analyses. This is also suspected to be the prime reason behind the apparent *increase* of antioxidant contents in the majority of the aged materials. Nevertheless, it is clear that non-significant antioxidant consumption took place during electro-thermal ageing. The DSC measurements indicate that the degree of crystallinity was not affected by electro-thermal DC ageing.

The complex dielectric permittivity over a broad frequency range (~0.1–100 kHz) of the laboratory-scale materials remained unchanged, and was essentially the same for all the materials apart from PP1-LoAO which displayed lower

Table 4. Weibull distribution parameters for large-area DC breakdown measurements on the laboratory-scale films.

Material	Reference			Electro-thermally aged						
	Points	α	β	Points	Portion	α_1	β_1	Portion	α_2	β_2
PP1-ref	143	666	10.3	217	0.18	542	3.1	0.82	622	11.0
PP2-ref	154	644	11.6	215	0.14	504	2.9	0.86	612	14.4
PP1-sil	154	655	11.1	265	0.14	623	3.0	0.86	617	9.3
PP2-sil	124	738	7.4	227	0.34	572	4.3	0.66	585	12.5
PP1-LoAO	149	652	9.0	228	0.15	554	3.0	0.85	612	11.6
PP1-HiAO	128	662	10.7	302		599	5.8			
PP1-LoSS	102	645	9.8	228	0.18	465	2.4	0.82	620	12.8
PP1-HiSS	82	617	8.2	304		583	4.8			

overall loss levels. The differences between the two measurements on individual samples were minimal, but the differences between the two samples measured were in many occasions in the same scale as the differences between materials. As such it can be concluded that with the possible exception of the antioxidant contents neither of the process variations had major effect on the real permittivity or dielectric loss levels. This confirms that ageing, at least within this scope, does not markedly affect the dielectric loss levels. Nevertheless, together the aforementioned results assert that the ageing had no significant effect on the bulk material. In comparison significant morphological changes have been reported [6] in BOPP films thermally aged for 60 days at 150 °C in SF₆ gas. These changes were detected using DSC and GPC measurements and were also associated with changed dielectric response. This confirms that such changes *can* be measured using the methods used, but also that 75 °C was probably not high enough to induce such changes.

None of these measurements can however provide information on any localized degradation which has been associated with polymer ageing [3, 32]. Limited and localized degradation could explain the ‘weak points’ measured in the aged films, but on the other hand the decrease of the characteristic breakdown strength, which could indicate changing intrinsic properties, remains unaccounted for.

Low-probability low voltage breakdowns or ‘weak points’ were also measured in the electro-thermally aged commercial BOPP film. This suggests that localized degradation similar

to the laboratory-scale films had taken place. On the other hand, the thermally only aged commercial film samples displayed an increase in overall breakdown strength, similar behavior has been reported for one thermally aged commercial BOPP film in [33]. Worth mentioning is also that the film thickness was not affected by the thermal stressing, indicating that no shrinkage had occurred. This behavior may however be related to annealing effects rather than true ‘ageing’, especially considering the relatively short duration of 1000 hours when compared to capacitor service lives, often measured in decades.

4.3 AGEING TEST

The function of the ageing test system used was not to perfectly imitate conditions found in real capacitors, but to subject several different films to similar stresses for a prolonged period, and to use the differences in material properties after ageing as a guideline in further material development. The ageing test system fulfilled these purposes, since the differences in the breakdown behavior between aged and reference samples, especially the weak point formation clearly indicate measurable ageing had occurred, despite no changes in bulk material property were measured in DSC and GPC. The next step is to apply the methodology to AC ageing, although care is needed to prevent partial discharging whose deleterious effects on BOPP films have been demonstrated recently at TUT [44] and also in related literature [45].

One possible source of uncertainty in the ageing system used is the success in removing the ambient air. Atmospheric air containing oxygen and moisture may have been ‘trapped’ between the metalized electrode films and the dielectrics. The presence of oxygen has been linked with accelerated degradation and the appearance of low-field weak points [2], [46]. In the latter degradation has been associated with radical-oxidation reactions progressing non uniformly, resulting in localized degradation and the weak points. Worth mentioning is also that in [47] it has been proposed that ageing of polymeric insulation in air would be representative (albeit accelerated) of ageing in oxygen-free environments. However in [46] oxygen-accelerated reactions have been also associated with *notably* lower mean molecular weight due to chain scissions, contrary to what was measured in this study. This, combined with the result that no antioxidant depletion had occurred seem to indicate the air removal was indeed

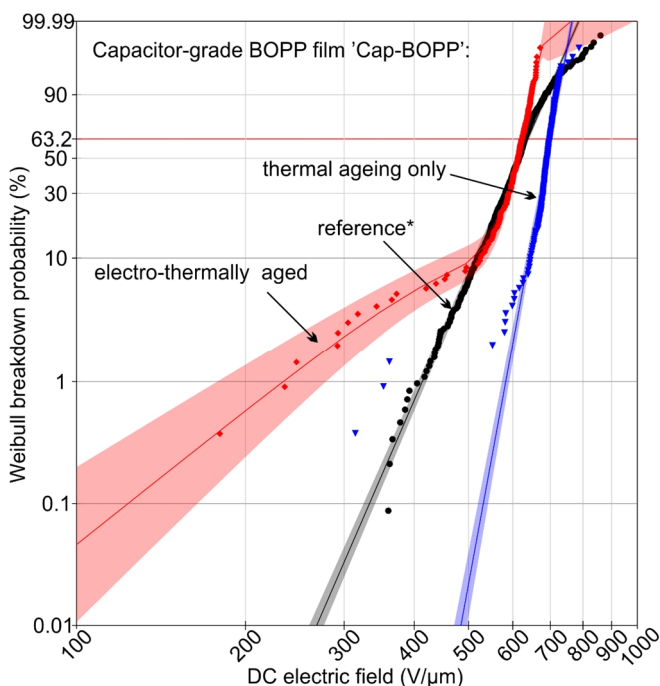


Figure 6. Large-area breakdown behavior of a commercial BOPP film: breakdown strength increased with thermal ageing while electro-thermal ageing resulted in weak point formation. Reference results are from [32], [33].

Table 5. Weibull distribution parameters for Cap-BOPP:

	Points	Portion	α_1	β_1	Portion	α_2	β_2
Reference	793	-	-	-	1	638	10.6
Electro-thermally aged	185	0.11	448	3.7	0.89	627	23.5
Thermally aged	183	-	-	-	1	700	25.1

successful. Based on [46] FTIR measurements would however be useful in confirming this further.

5 CONCLUSIONS

A new type of large-area electro-thermal ageing test system for polymer thin films was designed, assembled and evaluated successfully. Self-healing metallized electrodes enabled the ageing test to continue beyond the first breakdowns, and in principle would have enabled collection of times-to-breakdown data, although in practice this was hampered by possible non-breakdown discharging elsewhere in the system.

The ageing test system was used in a long-term DC ageing test, the results of which suggest that localized degradation in BOPP and BOPP-silica nanocomposite films during ageing results in the formation of low-field breakdowns or “weak points”. No changes in bulk material properties were measured with DSC, GPC and dielectric spectroscopy, as such the large-area breakdown methodology from [31, [35–37] was proven especially suitable in evaluating the effects of thin film ageing.

Eight laboratory-scale films were stressed for 1000 hours at 75 °C and 100 V/μm. In multiple cases the relative order of goodness of the films reversed after ageing, demonstrating the importance of long-term characterization during material development. The effect of two PP grades, the addition of 0.9 wt% of hydrophobic nano-silica, compounder screw speed and antioxidant contents were evaluated. Slight differences were detected between materials, and more importantly it was proven that it was possible to reproduce the results from earlier projects, in which laboratory-scale BOPP-nanocomposites having large-area DC breakdown strength comparable to neat BOPP films and commercial capacitor-grade products were produced successfully. Moreover, the oriented nanocomposite films had similar dielectric loss levels to the neat PP materials. Together these results suggest that BOPP-silica nanocomposites may have long-term properties comparable to neat BOPP films.

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Mikael Ritamäki (S’15) was born in Tampere, Finland in 1990. He received the M.Sc. (Tech.) degree in electrical engineering from Tampere University of Technology, Tampere, Finland in 2014. Since 2015 he has been working as a Researcher in the high voltage research group of the Department of Electrical Engineering at TUT, with aim towards the D.Sc. (Tech.) degree. His current research interests include electro-thermal

characterization of thin films.



Ilkka Rytöluoto (S’13-M’16) was born in Tampere, Finland on 17 April 1985. He received the M.Sc. (Tech.) and Doctoral degrees in electrical engineering from Tampere University of Technology (TUT), Tampere, Finland in 2011 and 2016, respectively. Since 2011 he has been working at the Department of Electrical Engineering at TUT, currently as a post-doctoral researcher. His main research interests are in the field of high voltage and insulation material

engineering, including e.g. dielectric characterization of polymeric thin films and establishing processing–structure–dielectric property relationships in polymer nanocomposite- and capacitor films.



Kari Lahti (M’01) was born in Hämeenlinna, Finland in 1968. He received the M.Sc. and Doctoral degrees in electrical engineering from Tampere University of Technology in 1994 and 2003, respectively. Since then he has worked at the Department of Electrical Engineering at TUT, currently as a Research Manager and Adjunct Professor. He is the head of TUT’s research group on high voltage insulation systems. He has also been responsible for the high voltage

laboratory services at TUT since 2002. His research interests are in the area of high voltage engineering including surge arresters, nanocomposite insulation systems, environmental testing of high voltage materials and apparatus, high voltage testing methods and dielectric characterization of insulating materials.



Torvald Vestberg was born Petalax, Finland in 1957. He received the M.Sc degree in chemical engineering and Doctoral degree in polymer technology from Åbo Akademi University in 1983 and 2013, respectively. He has been working with Borealis the last 20 years where he holds a group expert position. His area of interest is Ziegler-Natta catalysed polymerisation of propylene as well as effect of polymerization conditions on polymer structure and properties.



Satu Pasanen was born in Tampere, Finland in 1977. She received the B.Sc. (Tech.) in chemical engineering from the Tampere University of Applied Sciences, Tampere, Finland in 2001 and M.Sc. (Tech.) in Plastic and elastomer technology (including biomaterials) from Tampere University of Technology (TUT), Tampere, Finland in 2010. Since 2001 she has worked at VTT first as a Research Engineer and later as a Research Scientist. Her main

research interests are nanocomposites, functional polymer composites, polymer film applications, electrical applications and recycling & reuse of waste materials.



Timo Flyktman was born in Tampere, Finland in 1977. He received the B.Sc. (Tech.) degree in electrical engineering from Tampere University of Applied Sciences, Tampere, Finland in 2003. Since 2002 he has been working as a Research Engineer at VTT. His main research interests are plastic processing, plastic film applications, sensor technology and electrical applications.