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TECHNICAL REPORT

Reference number			
Title	*Dielectric Measurements on Eight Composite Materials*		
Customer:	**CRP TECHNOLOGY S.r.l.** ***Via Cesare della Chiesa 150/C*** ***41126 Modena (MO)***		
Reference:	Offer nr. ***CB-2024/001***		
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INDICE

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1 Purpose

The aim of the work is the determination of the dielectric properties of eight different materials shaped as thin disks, supplied by the Customer. Dielectric parameters to be measured are:

- dielectric constant at the frequency of 1 kHz of each material.
- dielectric strength at the frequency of 50 Hz of each material.

2 Equipment

(1) Quorum Mod. Q150TES Sputter Coater

(2) Combined Newtons4th Phase Sensitive Multimeter/IAI2 Impedance Analysis Interface 2/IAI Kelvin Probe frequency response analyzer (Serial Numbers: 233-1185/92414552/925-14349/92114563, resp., Certificate of Calibration Newton4th Ltd 233-11185 on 6th July 2022), operated under the control of the PSMComm2v1_2 proprietary software.

(3) KEYSIGHT Mod. 33511B Series Waveform Generator, Serial Number MY8000596.

(4) TReK MODEL 609E-6 HIGH VOLTAGE AMPLIFIER, Serial Number 2154).

(5) KEYSIGHT Mod. DSOX2012A 2-Channel 100Mx Digital Oscilloscope, Calibration Certificate N. LAT109 0487-21.

Equipment items (2) and (5) were also used as internal calibration references to check the good working order of equipment items (3), (4). NI LabView applications were purposely developed for the coordinated control of said equipment according to the needs mandated by the reference Norms.

3 Relevant documents

Measurements were carried out with reference to ASTM D 150 (dielectric constant measurements) and ASTM D149 (dielectric breakdown measurements) norms.

4 Sample description

Samples were provided by the Customer as triplicate 40 mm nominal diameter flat disks of variable thickness, about 1. mm. For each material, six samples were provided, three for dielectric constant measurements and three for dielectric strength tests. All samples were clean and dust-free as required and were only handled with tweezers or gloved hands to avoid contaminations.

Triplicate samples to be used for dielectric constant measurements were electrodized on both circular flats by sputter deposition of 200 nm thick films of silver. Careful masking of the rim of the disks was done to avoid formation of conductive Ag bridges between the electrodes. Samples to be used for the determination of the dielectric strength were used as received.

Naming of the samples in the Tables is self-explanatory.

5 Experimental procedures, parameters, operators

For dielectric constant measurements, FRA scans were performed to determine the capacitance of the electrodized disks taking into account electrodized area and thickness. Primary data were recorded as impedance (magnitude and phase) of each sample, both at 1 kHz and over the 100 Hz-1 MHz frequency range. A 0.5 V amplitude voltage sine wave was applied. For measurements at 1 kHz, 30 (thirty) experimental points were acquired in sequence, whereas FRA scans over the 100 Hz - 1 MHz frequency range were acquired as series of 500 experimental points. In both cases, electric impedance values were automatically converted to capacitance values by the measuring equipment, and these were used to calculate the dielectric constant of the material of each sample.

For dielectric strength tests, 50 Hz AC sinusoidal high voltage waves in the amplitude range from 0 to 4 kV were applied across the sample. A solid copper sphere-to flat geometry was employed, polished to a mirror finish, and mounted in a Teflon rig. Dielectric strength tests were performed at with both the samples and the sphere fully immersed in BLUESIL FLD 604V50 silicone oil fluid (SILICONI Italia Srl, Limena, Italy, dielectric strength > 15.75 kV/mm, nominal resistivity 10^{15} Ω cm) to prevent discharging from

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causes other than dielectric breakdown through the material, and/or spurious events such as flashover or discharging other than through the sample. The oil was baked for 2 hrs. at 150 °C to boil off humidity and brought back to room temperature in a closed vessel prior to use for dielectric strength tests. The dielectric strength of the oil was tested using the same geometry with a 1 mm gap between the sphere and the flat; no discharge events were observed at the highest applied voltage.

Dielectric strength tests consisted in applying several high amplitude 50 Hz sinusoidal voltages in succession, each amplitude being maintained constant for either 1 s (rapid voltage rise tests) or 20 s, all the while watching for dielectric breakdown, spurious discharges, or flashover events, none of which were ever observed. The current threshold above which the instrument would interrupt the HV wave in response to the occurrence of any of the above events was set at 4 mA for all tests. All dielectric strength tests were performed at 22 °C and 48% to 52% relative humidity.

6 Results

6.1 Dielectric Constant Measurements

Dielectric constant measurements results are shown in **Table 1**. Of the eight materials tested in triplicate, only three were found to be dielectrics (i.e. electrical insulators). The remaining five materials were found to be conductive, albeit to different degrees. The conductive nature of these samples was made clear by the very small (of the order of a fraction of a degree) value of the phase angle of their impedance at all frequencies investigated (an ideal conductor's phase angle is exactly zero). While it is indeed possible to provide dielectric constant values for any materials having measurable impedance (electrical conductors included), dielectric constants of conducting materials are of little value when the main focus is on their electrical insulation properties. Accordingly, for the five materials that were found to be conductive, the electrical resistivity is given in the table instead of the dielectric constant.

Relative dielectric constant values of the materials that were found to be true dielectrics are of the order of a few units, as shown in **Table 1**. Combined with the standardized geometry of the samples, such low values of the dielectric constant leads to values of the measured capacitance of the samples of the order of a few tens of pF, which is of the same magnitude order as the parasitic capacitance of the measuring system, including the capacitance of the sample holder, the proprietary Kelvin probe, and all electrical connections. Therefore, the parasitic capacitances were measured in exactly the same physical configuration of the measuring system as during the measurement, but without any sample being present. A value of 11.3 pF was thus found for the parasitic capacitance, fully corroborated by additional measurements performed on a series of precision capacitors having capacitances between 4.7 and 180 pF. This value was subtracted from the measured capacitance to obtain the true capacitance of the dielectric samples, used to determine the dielectric constant.
The main result here is that the electrical regime of all material is consistent over all triads, meaning that all of the samples in the same triad were either conductors or insulators, no triad containing both insulating and conductive samples.

6.2 Dielectric Strength Tests

Dielectric strength measurements were performed only on the materials that were found to behave as electrical insulators. Dielectric strength measurements results are summarized in **Table 2**.
Results show that materials subjected to dielectric strength tests all withstood the highest applied 50 Hz AC sinusoidal high voltage (4 kV amplitude). There was no indication of even incipient dielectric breakdown at regime for all applied voltages. Transients occurring at the switch between two consecutive values of the applied voltage amplitude (which are known to prime electric discharge at high voltages) were observed to be instantaneous, fully consistent with the small *RC* time constant given by the combination of the output impedance of the high-voltage amplifier and the capacitance of the measuring rig (several tens of pF in full measurement configuration, sample's capacitance included).

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7 Experimental Errors

7.1 Relative Dielectric Constant

The uncertainty on the relative dielectric constant ε_r values **of each single sample** is of the order of ±0.5 units (the reader is reminded that the *relative dielectric constant* is a dimensionless number. The *dielectric constant* can be obtained as $\varepsilon_0\varepsilon_r$, where $\varepsilon_0 = 8.854\times10^{-12}$ F m^{-1}). The spread of the three ε_r values listed in the table is representative of the uncertainty on the average value **over the triplicate samples**, i.e., the dielectric constant **of the material**, due to fluctuations of the material's properties from one sample of the same material to the next one. However, dielectric constant values display little scattering (see **Table 1**).

7.2 Conductivity

The uncertainty on the value of the conductivity **of each single sample** is quite small (estimated as **2%** as most) due to the resistivity at 1 kHz being calculated according to:

$$\rho = R_{Series} \cdot (Area/Thickness) \qquad [1]$$

All three quantities appearing in [1] were precisely known due to the very regular geometry of the provided samples (diameter and thickness were known within ±0.1 mm and a couple of hundredths of mm, resp.). The uncertainty on the value of R_{Series} is also quite small (about 0.1%).

7.3 Dielectric Breakdown Voltage

Parameters defining the dielectric testing protocols (applied sinusoidal AC voltage amplitude, frequency, timing) were known with high precisions (0.5% at most percent error) due to the features of the instrumentation used.

8 Opinions and interpretations

The **electrical resistivity** ρ of the conductive samples, i.e., materials **WINDFORM XT 2.0 (ρ = 71 Ω cm)**, **WINDFORM SP (923 Ω cm)**, **WINDFORM RS (1244 Ω cm)**, **WINDFORM FR1 (2508 Ω cm)**, and **WINDFORM SL (1664 Ω cm)** (average values over the triplicate samples) is typical of composite materials in which an insulating matrix hosts particles of some conductive phase. In such cases, the final resistivity of the composite material is difficult to reproduce (as shown by the large spread of ρ values over each triad), because conduction depends as much on the amount of conductive phase dispersed in the host phase, as it does on the details of the production process. In such materials, the way in which the conductive particles establish conductive chains across the material by e.g., mutual contact cannot be reproduced by simply keeping the production parameters constant and may vary significantly over different regions of the same specimen.

The **relative dielectric constant** ε_r of the three electrically insulating materials **(WINDFORM LX 30, ε_r = 4.3)**, **WINDFORM GT (ε_r = 3.9)**, and **WINDFORM FR2 (ε_r = 4.4)** (average values over the triplicate samples) is better defined than the resistivity of the conducting materials above, as shown by the little spread of ε_r values over the triplicate set (see **Table 1** below).
The three electrically insulating materials listed above behave as strong dielectrics under 50 Hz sinusoidal AC high-voltage excitation. No indications of dielectric breakdown were observed under the conditions detailed above, even after prolonged application of the maximum (4 kV_p, or 2.83 kV_{RMS}) or at amplitude switches, neither during rapid rise (1s/step) nor during slow rise (20 s/step) tests. Additional long-duration dielectric breakdown tests carried out at constant 4 kV_p amplitude could not cause dielectric breakdown either. While the results of the dielectric breakdown tests only allow to define a lower bound (about 2450 kV_p / mm), see **Table 2** below, for all electrically insulating samples, with little variations between the triplicate sets, indications are there that the actual dielectric strength of these materials is significantly higher.

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9 Operators

Summary of activities and operators involved in this Technical Report, in the order they were carried out:

Activity	Name
Preliminary characterizations and procedures, i.e., inspection of provided samples; masking as required; geometrical and physical characterization (diameter, thickness, mass); deposition of 200 nm-thick sputtered Ag electrodes as required.	Carlo Baldisserri
Evaluation of stray/parasitic impedances of the FRA measuring system. Explorative FRA over the 100 Hz – 1o kHz frequency range. 30-point FRA measurements at the frequency of 1 kHz. Conversion of FRA data to relative dielectric constant data.	Carlo Baldisserri
Set-up of sample holder/sphere-to-flat geometry assembly in high voltage Teflon holder for dielectric strength measurements. Writing of dedicated NI LabView code for the control and coordination of the instrumentation used for the application of the HVAC application protocols. High-voltage dielectric strength tests under sinusoidal AC voltage excitation at the frequency of 50 Hz.	Carlo Baldisserri
Data curation and interpretation and quantification of experimental errors.	Carlo Baldisserri
Writing/editing of the Technical Report	Carlo Baldisserri

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10 Tables

Sample	Material	Diameter	Thickness	Mass	Density	Area	Area	Thickness	Thickness	C @ 1 kHz	ε_r @ 1 kHz	R_{serie} @ 1 kHz	ρ @ 1 kHz	1 kHz FRA Datafile	FRA 100 Hz-10 kHz Scan Datafile	Electric Behavior
1/8_A	WINDFORM XT 2.0	39,89	1,54	2,0404	1,060	12,50	1,250E-03	0,154	1,54E-03	n.a.	n.a.	0,753	61,1	1-8_A_1 kHz	1-8_A_100 Hz-10 kHz	Conductor
1/8_B	WINDFORM XT 2.0	39.93	1,53	2,0564	1,073	12,52	1,252E-03	0,153	1,53E-03	n.a.	n.a.	1,103	90,3	1-8_B_1 kHz	1-8_B_100 Hz-10 kHz	Conductor
1/8_C	WINDFORM XT 2.0	39,96	1,53	2,0393	1,063	12,54	1,254E-03	0,153	1,53E-03	n.a.	n.a.	0,764	62,6	1-8_C_1 kHz	1-8_B_100 Hz-10 kHz	Conductor
2/8_A	WINDFORM LX 30	40,06	1,62	2,5900	1,268	12,60	1,260E-03	0,162	1,62E-03	31,6	4,59	n.a.	n.a.	2-8_A_1 kHz	2-8_A_100 Hz-10 kHz	Dielectric
2/8_B	WINDFORM LX 30	40,06	1,72	2,7815	1,283	12,60	1,260E-03	0,172	1,72E-03	26,6	4,10	n.a.	n.a.	2-8_B_1 kHz	2-8_B_100 Hz-10 kHz	Dielectric
2/8_C	WINDFORM LX 30	40,04	1,62	2,6205	1,285	12,59	1,259E-03	0,162	1,62E-03	29	4,21	n.a.	n.a.	2-8_C_1 kHz	2-8_C_100 Hz-10 kHz	Dielectric
3/8_A	WINDFORM SP	40,08	1,56	2,1898	1,113	12,62	1,262E-03	0,156	1,56E-03	n.a.	n.a.	13,22	1069	3-8_A_1 kHz	3-8_A_100 Hz-10 kHz	Conductor
3/8_B	WINDFORM SP	40,10	1,60	2,2191	1,098	12,63	1,263E-03	0.16	1,60E-03	n.a.	n.a.	10,21	806	3-8_B_1 kHz	3-8_B_100 Hz-10 kHz	Conductor
3/8_C	WINDFORM SP	40,09	1,68	2,3384	1,103	12,62	1,262E-03	0,168	1,68E-03	n.a.	n.a.	11,91	895	3-8_C_1 kHz	3-8_C_100 Hz-10 kHz	Conductor
4/8_A	WINDFORM GT	40,21	1,63	2,4586	1,188	12,70	1,270E-03	0,163	1,63E-03	26,5	3,84	n.a.	n.a.	4-8_A_1 kHz	4-8_A_100 Hz-10 kHz	Dielectric
4/8_B	WINDFORM GT	40,30	1,63	2,4455	1,176	12,76	1,276E-03	0,163	1,63E-03	27,2	3,93	n.a.	n.a.	4-8_B_1khZ	4-8_B_100 Hz-10 kHz	Dielectric
4/8_C	WINDFORM GT	40.15	1,70	2,5533	1,186	12,66	1,266E-03	0.17	1,70E-03	25,6	3,88	n.a.	n.a.	4-8_C_1 kHz	4-8_C_100 Hz-10 kHz	Dielectric
5/8_A	WINDFORM RS	40,29	1,54	2,1496	1,095	12,75	1,275E-03	0,154	1,54E-03	n.a.	n.a.	21,30	1763	5-8_A_1 kHz	5-8_A_100 Hz-10 kHz	Conductor
5/8_B	WINDFORM RS	40.23	1,45	1,9854	1,077	12,71	1,271E-03	0,145	1,45E-03	n.a.	n.a.	10,78	945	5-8_B_1 kHz	5-8_B_100 Hz-10 kHz	Conductor
5/8_C	WINDFORM RS	40,33	1,53	2,1326	1,091	12,77	1,277E-03	0,153	1,53E-03	n.a.	n.a.	12,28	1025	5-8_C_1 kHz	5-8_C_100 Hz-10 kHz	Conductor
6/8_A	WINDFORM FR1	40,15	1,69	2,4128	1,128	12,66	1,266E-03	0,169	1,69E-03	n.a.	n.a.	36,33	2722	6-8_A_1 kHz	6-8_A_100 Hz-10 kHz	Conductor
6/8_B	WINDFORM FR1	40,05	1,66	2,3386	1,118	12,60	1,260E-03	0,166	1,66E-03	n.a.	n.a.	25,64	1946	6-8_B_1 kHz	6-8_B_100 Hz-10 kHz	Conductor
6/8_C	WINDFORM FR1	40,13	1,70	2,4324	1,131	12,65	1,265E-03	0,17	1,70E-03	n.a.	n.a.	38,37	2855	6-8_C_1 kHz	6-8_C_100 Hz-10 kHz	Conductor
7/8_A	WINDFORM FR2	40,08	1,61	2,4388	1,201	12,62	1,262E-03	0,161	1,61E-03	30,6	4,41	n.a.	n.a.	7-8_A_1 kHz	7-8_A_100 Hz-10 kHz	Dielectric
7/8_B	WINDFORM FR2	40,13	1,63	2,5106	1,218	12,65	1,265E-03	0,163	1,63E-03	30,3	4,41	n.a.	n.a.	7-8_B_1 kHz	7-8_B_100 Hz-10 kHz	Dielectric
7/8_C	WINDFORM FR2	40,08	1,70	2,5997	1,212	12,62	1,262E-03	0,17	1,70E-03	27,9	4,25	n.a.	n.a.	7-8_C_1 kHz	7-8_C_100 Hz-10 kHz	Dielectric
8/8_A	WINDFORM SL	40,05	1,68	1,8222	0,861	12,60	1,260E-03	0,168	1,68E-03	n.a.	n.a.	29,62	2221	8-8_A_1 kHz	8-8_A_100 Hz-10 kHz	Conductor
8/8_B	WINDFORM SL	39,96	1,62	1,7504	0,862	12,54	1,254E-03	0,162	1,62E-03	n.a.	n.a.	16,02	1240	8-8_B_1 kHz	8-8_B_100 Hz-10 kHz	Conductor
8/8_C	WINDFORM SL	40,17	1,65	1,8065	0,864	12,67	1,267E-03	0,165	1,65E-03	n.a.	n.a.	19,92	1530	8-8_C_1 kHz	8-8_C_100 Hz-10 kHz	Conductor
		[mm]	[mm]	[g]	[g cm^{-3}]	[cm^2]	[m^2]	[cm]	[m]	[pF]	[-]	[Ω]	[Ω cm]			

Table 1 Physical and electrical, and dielectric parameters (measured on all materials, by frequency response analysis)

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Sample	Material	Thickness	Max. 50 Hz AC Voltage Amplitude Without Breakdown	Dielectric Strength ≥	Dielectric Strength ≥
2/8_D	WINDFORM LX 30	1,62	4	2469	1746
2/8_E	WINDFORM LX 30	1,72	4	2326	1645
2/8_F	WINDFORM LX 30	1,62	4	2469	1746
4/8_D	WINDFORM GT	1,63	4	2454	1735
4/8_E	WINDFORM GT	1,63	4	2454	1735
4/8_F	WINDFORM GT	1,70	4	2353	1664
7/8_D	WINDFORM FR2	1,61	4	2484	1756
7/8_E	WINDFORM FR2	1,63	4	2454	1735
7/8_F	WINDFORM FR2	1,70	4	2353	1664
		[mm]	[kV]	[kV_{peak}/mm]	[kV_{RMS}/mm]

Table 2 Dielectric strength under 50 Hz sinusoidal high-voltage AC excitation.

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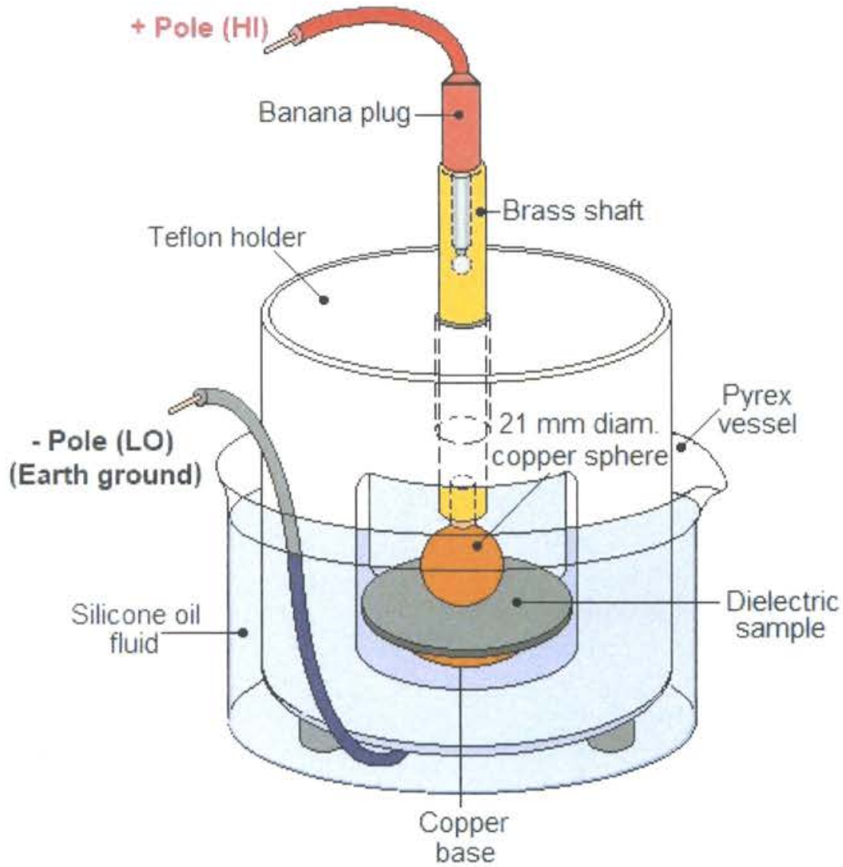
11 Figures



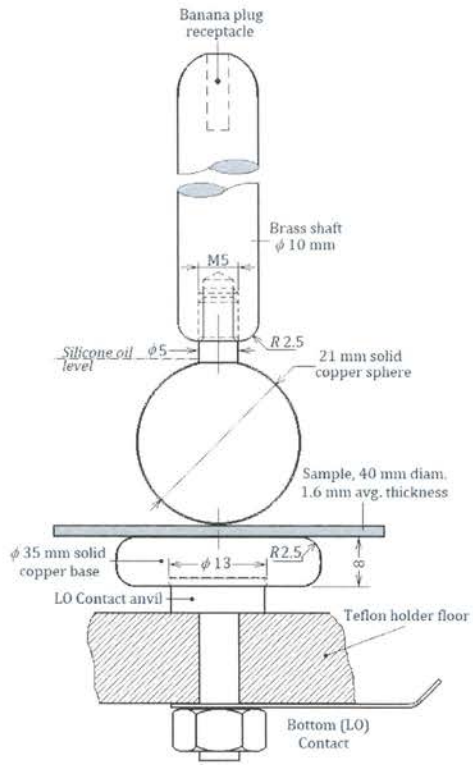


Fig. 1 Full setup (left) and sphere-to-flat geometry (right) employed for dielectric for breakdown tests. All dimensions are in mm.

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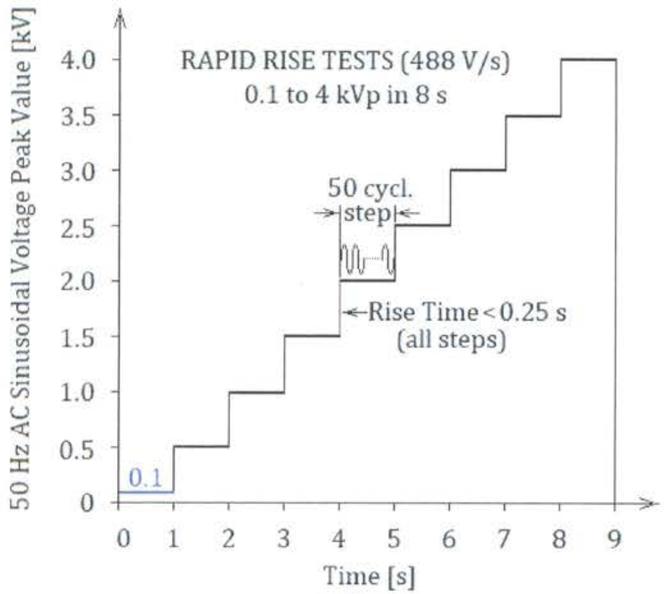

Fig. 2 High-voltage amplitude profile used for rapid voltage rise tests on all dielectric samples. Slow-rise tests were also carried out, with the duration of each voltage step increased to 20 s. Tests were performed on all triplicate WINDFORM LX 30, WINDFORM GT, and WINDFORM FR2 samples. Each sample was tested at multiple locations on their circular surfaces. A number of long-duration (about 10 min.) tests were also performed. No indications of dielectric breakdown through any of the materials were ever observed in any of the tests, even at the highest applied voltage amplitude (Vpeak = 4 kV).

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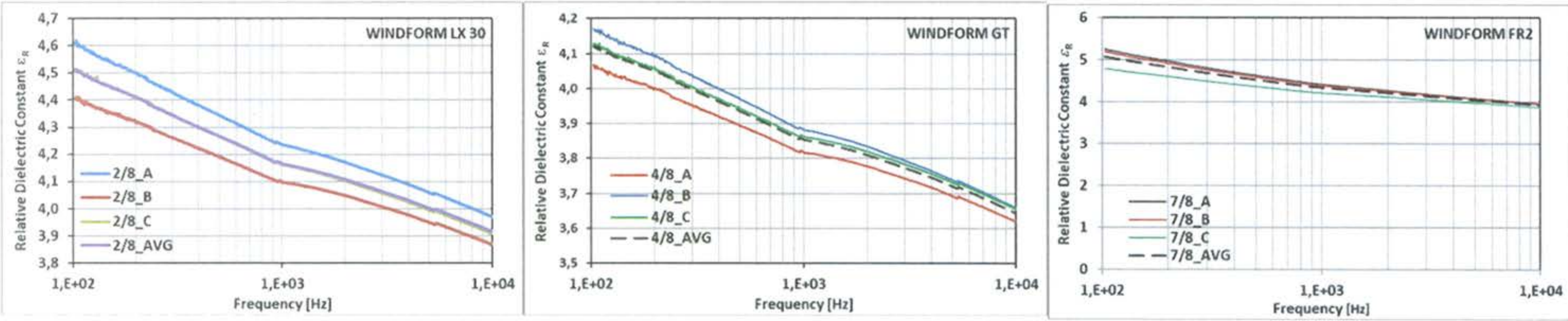

Fig 3 Dependence of the dielectric constant ε_R of the three materials that were found to behave as dielectrics on frequency, calculated based on preliminary explorative frequency response analysis scans performed over the 100 Hz to 10 kHz frequency range, and sample's geometry parameters. AVG curves are obtained as the average of the dielectric constant of triplicate samples at each frequency. The decreasing trend of the dielectric constant vs. frequency is typical of most electrically insulating materials. Dielectric constant data shown in Table 1 above were not calculated from these scans, but from sets (30 experimental points for all samples) of consecutive FRA measurements carried out at the fixed frequency of 1 kHz.

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12 Disclaimer

The present results only refer to the described samples and procedures. Any extrapolation or extension to other samples and procedures is beyond the scope of this document.

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