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TEST METHODS FOR THE EVALUATION OF DYNAMIC HYDROPHOBICITY PROPERTIES OF POLYMERIC INSULATING MATERIALS FOR HIGH-VOLTAGE APPLICATIONS

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1 INTRODUCTION

Polymeric insulating materials are applied as housing materials of composite insulators in different design variations and for all voltage levels. They are known for their advantageous properties such as the resistance to UV weathering and the excellent dynamic hydrophobic properties (retention, recovery and transfer of hydrophobicity). These specific characteristics result in favourable electrical properties in wet and polluted conditions, e.g. significantly higher flashover voltages and lower leakage currents compared to inorganic materials (glass, ceramic) [1]. Though service experiences of composite insulators show that hydrophobic properties are generally maintained for long periods [2], they may be temporarily reduced by certain stresses (electrical micro-discharges at water droplets, corona at fittings, water absorption etc.). Hence an evaluation of the dynamic hydrophobic properties is required. Even though there are no standardized test methods yet [3], many investigations with different test procedures have been conducted evaluating the retention and recovery of hydrophobicity e.g. the salt fog test [4], the modified rotating wheel dip test [5] as well as the

Dynamic Drop Test (DDT) [6], [7]. For an evaluation of the hydrophobicity transfer onto pollution layers on polymeric materials test methods were developed amongst others in [8], [9], [10]. The dynamic hydrophobicity properties have been found to be determined and influenced by the following factors:

- Composition of base polymer [11], type and content of fillers [12]
- Reorientation and diffusion of low molecular weight components of silicone elastomers [13]
- Roughness and surface structure [14]

The relevant stress factors subject to influence the status of hydrophobicity are voltage type and magnitude, type and content of pollution layer, climate conditions [15]

Based on test methods according to [16] (DDT) and [17] the aim of the present study is to increase the robustness of the procedures to variations of the influencing parameters, achieving eventually reproducible test methods for the dynamic properties of hydrophobicity. Thus, this work contributes to a potential standardization of

these promising but not yet matured test methods. For the DDT the influence of the conductivity of the electrolyte as well as the inclination angle of the test arrangement on the retention of hydrophobicity is studied. For the transfer of hydrophobicity the most suitable pollution layer base materials in respect to processability of the pollution layer suspension are identified. The temperature dependence, the time requirement of the transfer process, contact angle measurability as well as the robustness of the procedure to variations of grain size and different material brands are evaluated.

2 EXPERIMENTAL TEST SETUP

2.1 Test Materials

In these investigations specimens made from different silicone elastomers (LSR, HTV-SIR, RTV-2-SIR) and a special epoxy resin HCEP with a uniform roughness of $Rz=5~\mu m$ (averaged roughness height) are used. Prior to testing the specimens are cleaned with isopropanol and distilled water and stored for minimal 24 hours under laboratory conditions.

2.2 Retention of Hydrophobicity

The DDT permits an accelerated evaluation of the retention of hydrophobicity to pollution initiated surface discharges. The pollution is caused by an electrolyte with defined conductivity. The evaluation criterion is the time to the loss of hydrophobicity. It is the time to the development of a conductive path between high voltage and ground electrode indicated by a defined leakage current [16]. In **Figure 1** the experimental test setup is given. Test parameters are noted in **Table 1**.



- 1 Upper electrode
- 2 Bottom electrode
- 3 Specimen
- 4 Peristaltic pump
- 5 Container for electrolyte
- 6 Earth contact
- 7 High voltage contact
- 8 Photoelectric sensor

Figure 1: Photograph of the DDT setup

Table 1: Test Parameters of the DDT

Test Parameter	Value
Distance of electrodes	50 mm
Conductivity of electrolyte	(1,0-10) mS/cm at 20 °C
Inclination angle α	30°-90°
Flow rate	(1 ± 0,2) ml/min
Failure criterion	Leakage current > 2 mA for
	a duration of 1 s
Drop frequency	(12 ± 1) drops/min at $\alpha = 60^{\circ}$
Test voltage (50 Hz - AC-	(0 – 4) kV AC (rms)
and +DC-stress)	(0 - 5,67) kV DC
Number of specimens	12

For the evaluation of the retention of hydrophobicity under AC and DC stress two different voltage sources are applied (AC: transformer (35 kV/ 4,4 kVA); DC: Electronic Switching Power Supply (ESPS, 12 kV, 300 mA)). For a comparison of different voltage types peak values are chosen as common basis.

During these tests the electro hydrodynamic behaviour of the moving droplets is observed with the use of a high speed camera (Olympus, I-Speed 3). The sliding performance of the droplets is evaluated by geometric parameters such as droplet volume, top length (moment in which the droplet tears off the upper electrode) and bottom length (moment in which the droplet contacts the bottom electrode) (**Figure 2**)

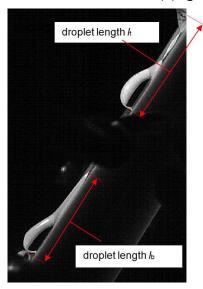


Figure 2: Droplet length during the sliding process

2.3 Hydrophobicity Transfer

The transfer of hydrophobicity is investigated by applying a suspension of a solid base material and distilled water (in some cases mixed with isopropanol) onto the surface of an insulating material specimen (dimensions 50 mm x 50 mm x 6 mm). Using a mask of self-adhesive foil (**Figure 3**) the thickness of the wet layer approximately corresponds to the thickness of the mask (0,36 mm). Unless otherwise stated all the suspensions are produced of 7,5 g of the base material and as much distilled water as is necessary to solve it (2,5 ml – 3,5 ml).

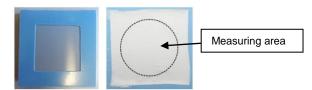


Figure 3: Specimen (left); with layer applied (right)

Once the pollution layer is applied the samples are stored in a climate chamber at a standard climate of 23°C and 50% relative humidity. After

a short drying process of a few minutes, the dry pollution layer is by itself hydrophilic and any drops applied onto it will be absorbed. If there are hydrophobic components in the specimen which are able to pass through the pollution layer, its surface will gradually increase its hydrophobicity and drops will exhibit increasing contact angles over time. It is accepted that the low molecular weight components (LMWs) of the silicone rubber are responsible for that phenomenon. Static and dynamic contact angles are measured using a contact angle goniometer (**Figure 4**). For measuring the dynamic contact angles the volume of the droplets is increased or decreased.

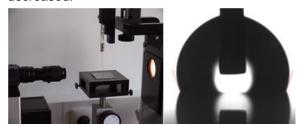


Figure 4: Contact angle goniometer (left) and drop on hydrophobic surface (right)

3 RESULTS AND DISCUSSION

3.1 Retention of Hydrophobicity

First investigations focus on an optimization of the test arrangement to improve the stability of parameter settings and thus the scatter of the test results. One important parameter to achieve this aim is the droplet volume which should be kept independent from the applied voltage type and magnitude. The droplet volume may have an influence on the intensity of micro discharges. Especially the corresponding droplet length accounts for the bridging degree of the test arrangement. The higher the bridging degree the more intense are the hydrophobicity reducing electrical micro discharges.

These studies result on the one hand in a slight modification of the used upper electrode (additional millings to limit the droplet width (**Figure 5**)).

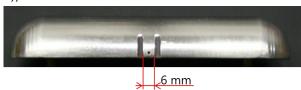


Figure 5: Slightly modified upper electrode

On the other hand the high voltage potential is applied to the bottom electrode and the top electrode is grounded in contrast to specifications in [7] (see Figure 1). This significantly reduces the dependence of the droplet volume on the applied voltage type and magnitude (**Figure 6**).

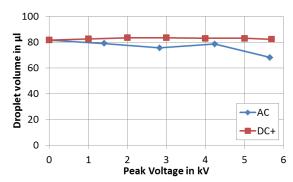


Figure 6: Droplet volume depending on the voltage type and magnitude

In the following a parametric study with respect to these modifications is presented in order to specify test parameters and test conditions for the DDT. The influence of the electrolyte's conductivity on the retention of hydrophobicity of two selected silicone elastomers under AC and DC stress is shown in **Figure 7**.

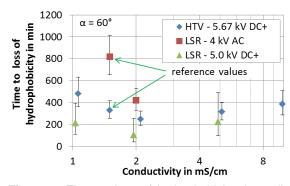


Figure 7: Time to loss of hydrophobicity depending on the conductivity of the electrolyte; 63% quantiles and confidence intervals with 5 % error probability

For a conductivity of 1 mS/cm up to 2 mS/cm at AC and DC stress decreasing times to the loss of hydrophobicity are achieved and a tendency to increasing times to failure for a higher conductivity up to 10 mS/cm under DC stress are determined. It is assumed that with an increasing conductivity more intense micro discharges appear which result in an accelerated reduction of hydrophobicity as also investigated in [16]. Visual observations during the tests reveal a different behaviour of water droplets at 5 mS/cm and 10 mS/cm. In both cases there is a significant decrease in the droplet frequency with the test time.

The influence of the inclination angle on the retention of hydrophobicity is investigated for HTV-SIR under DC stress (**Figure 8**). The time to the loss of hydrophobicity decreases with increasing inclination angle. If the inclination angle is increased a reduction of the droplet volume can be noticed (**Figure 9**). This results in an increasing frequency of the sliding droplets if the flow rate of the electrolyte is kept constant. An increased droplet frequency leads to a more frequent stress by pollution initiated surface

discharges which may cause a reduced retention of hydrophobicity.

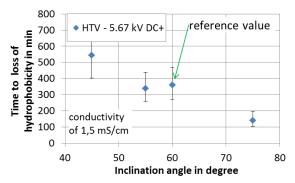


Figure 8: Time to loss of hydrophobicity depending on inclination angle; 63% quantiles and confidence intervals with 5 % error probability

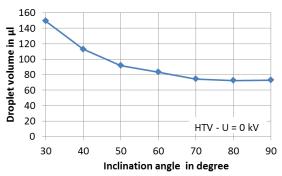


Figure 9: Droplet volume as a function of inclination angle

Even though it would be reasonable to assume, that droplets with lower volume may bridge the electrode distance to a lesser degree, reducing the intensity of micro discharges, this could not be observed in the investigated range of inclination angles (**Figure 10**). The droplet lengths remain constant and no reduction of micro discharges' intensity is expected. Therefore, the increased frequency of the discharges might be the dominating effect.

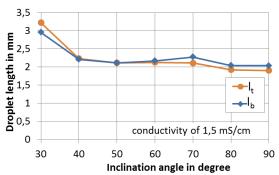


Figure 10: Top and bottom droplet lengths as a function of the inclination angle α

Further studies will be carried out to define appropriate test parameters and corresponding tolerances to evaluate and differentiate the retention of hydrophobicity of different hydrophobic polymeric insulating materials.

3.2 Hydrophobicity Transfer

For all the investigations the recommended procedure of [15] is taken as a reference method to which modified or new and different approaches have to be compared. In this method 7,5 g of SiO₂ (quartz powder SF600) and 3,5 ml of a 65%/35% by volume mixture of distilled water and isopropanol are used for the pollution layer.

The influence of the layer thickness is already investigated in the literature using the mask thickness as layer thickness or measuring the difference between the mask and the dried out pollution layer and subtracting this value from the mask thickness. But even with this correction there is some potentially relevant error, since the pollution laver as well as the underlying sample is uneven and thus the layer thickness varies randomly even on one single specimen. For that reason the height of the pollution layer at the very point of the contact angle measurement is determined using a laser-profilometer by measuring profiles of the specimen with and without pollution layer, subtracting them and placing the drops to precisely designated locations. The second reason for performing these experiments is to correlate the high scatter of the contact angle measurement values in the phase of strongly increasing angels to the scatter of the layer thickness at the point of measurement. Thus all contact angle measurements are taken exactly 22 h after the pollution layer was applied, a time after which the contact angles are already precisely measurable but are still increasing and showing significant scatter (Figure 11).

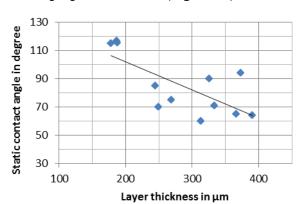


Figure 11: Static contact angle after 22 h as a function of the layer thickness on RTV-2 specimens at the point of measurement

On the one hand the results confirm that increasing layer thickness leads to lower contact angles after identical transfer time. On the other hand there is still scatter which cannot be traced to the differences in layer thickness. Taking this a step further the roughness of the pollution layer at all these measuring points are determined, as surface roughness is a well-established factor of influence on contact angles. The obtained results (not shown here)

however confirm only marginal differences in surface roughness between all measuring points (2,5 < Rz < 4) producing only a small effect on the contact angles which is not responsible for the observed scatter. For this reason further investigations concentrate on grain size distributions leading to different layer densities.

The primary requirement on the base material is its global availability. But even so SiO_2 quartz powder can undoubtedly be obtained globally; grain size as well as surface structure of the particles will differ, rendering it necessary to investigate the reproducibility of the procedure for different products. In **Figure 12** the transfer of hydrophobicity onto three different SiO_2 quartz powders with different grain size is shown.

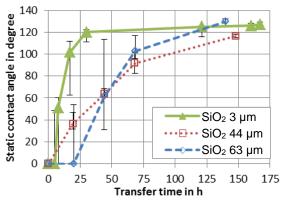


Figure 12: Hydrophobicity transfer for different SiO₂ with different grain sizes; median of 5 samples and span width; RTV-2-SIR specimens

The results show significant differences for the three types of SiO_2 . The static contact angles surpass 100° at 18 h, 68 h and 95 h respectively. On the one hand this indicates that in case of SiO_2 quartz powder for a standardised test procedure the very exact type and manufacturer has to be specified. On the other hand no clear influence of the grain size can be perceived, thus giving a hint, that maybe the surface structure of the molecules is the more prominent factor of influence.

Bearing this in mind four different kinds of Al₂O₃ powders are investigated on RTV-2-SIR, two of them chemically identical, but only different in grain size. While the Al₂O₃ with average particle size of 60 µm shows no hydrophobicity transfer at all (within 120 h) the surface on the average 180 µm particles is completely hydrophobic after only 2 h. The same applies for a fine Al₂O₃ powder of < 50 µm. The fourth material is a calcined Al₂O₃ powder having only a very poor transfer. Figure 13 shows the most promising results obtained with the fine Al₂O₃ powder. The experiment can distinguish clearly between the four investigated insulating materials even though the contact angle could not be measured precisely.

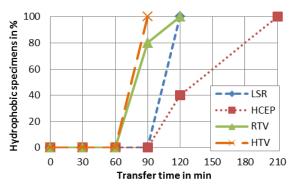


Figure 13: Transfer through fine Al₂O₃ powder for different insulating materials

When applying the drops to the already hydrophobic layer particles of the Al_2O_3 pollution covered the drop surface (**Figure 14**). Thus it is only possible to distinguish hydrophobic or not hydrophobic pollution layers.



Figure 14: Drop with Al₂O₃ fine powder on its surface

In the opinion of the author soda-lime glass beads are not only available globally but will be also easily supplied with defined grain sizes and treatment or better without any. In addition it is cheap but way more important the differences in surface structure of the particles will be most likely less than with the ground and processed SiO_2 or Al_2O_3 . Three different grain sizes of the otherwise identical glass beads are investigated (**Figure 15**).

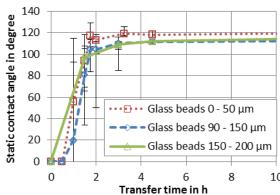


Figure 15: Hydrophobicity transfer for glass beads of different grain sizes; median of 5 samples and span width; RTV-2-SIR specimens

The results show no significant influence of the grain size rendering the glass beads at least in this respect to the ideal choice for the hydrophobicity transfer test method.

Experiments using Kaolin, Kieselgur and NaCl powder show no or only very slow (starting at 150 h) hydrophobicity transfer on the RTV-2. Thus they are disregarded as the procedure should later on be sensitive to materials with weak hydrophobicity transfer even without excessive time requirements, at least if the ranking of the materials is reproducible with the faster methods. In addition the processability of the pollution layer suspension and the measurability of the contact angles are evaluated for all base materials.

Finally the influence of the storing temperature is investigated for all base materials. One example is shown in **Figure 16**, indicating a strong influence of the temperature. With increasing temperature the mobility of the LMWs increases and the transfer through the layer is faster. The factor of influence is however different for different base materials. The glass beads for example exhibit a less severe influence than the SiO₂, giving them yet another advantage.

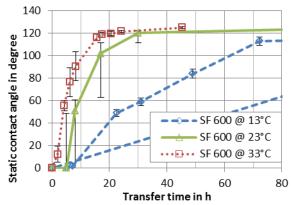


Figure 16: Influence of temperature on the transfer process; median of 5 samples and span width; RTV-2-SIR specimens

4 CONCLUSION

This paper presents parametric studies on the evaluation of dynamic hydrophobicity properties.

The retention of hydrophobicity is evaluated by the time to the temporary loss of hydrophobicity in the DDT:

- It decreases with increasing conductivity of the electrolyte.
- It also decreases with increasing inclination angles for the investigated HTV-SIR.

The transfer of hydrophobicity onto pollution layers on polymeric materials is investigated:

- Not the grain size but most likely the surface structures of the particles are responsible for extreme differences in the speed of the process.
- Out of 13 different base materials the two most promising (SiO₂ and soda-lime glass beads) are identified.

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