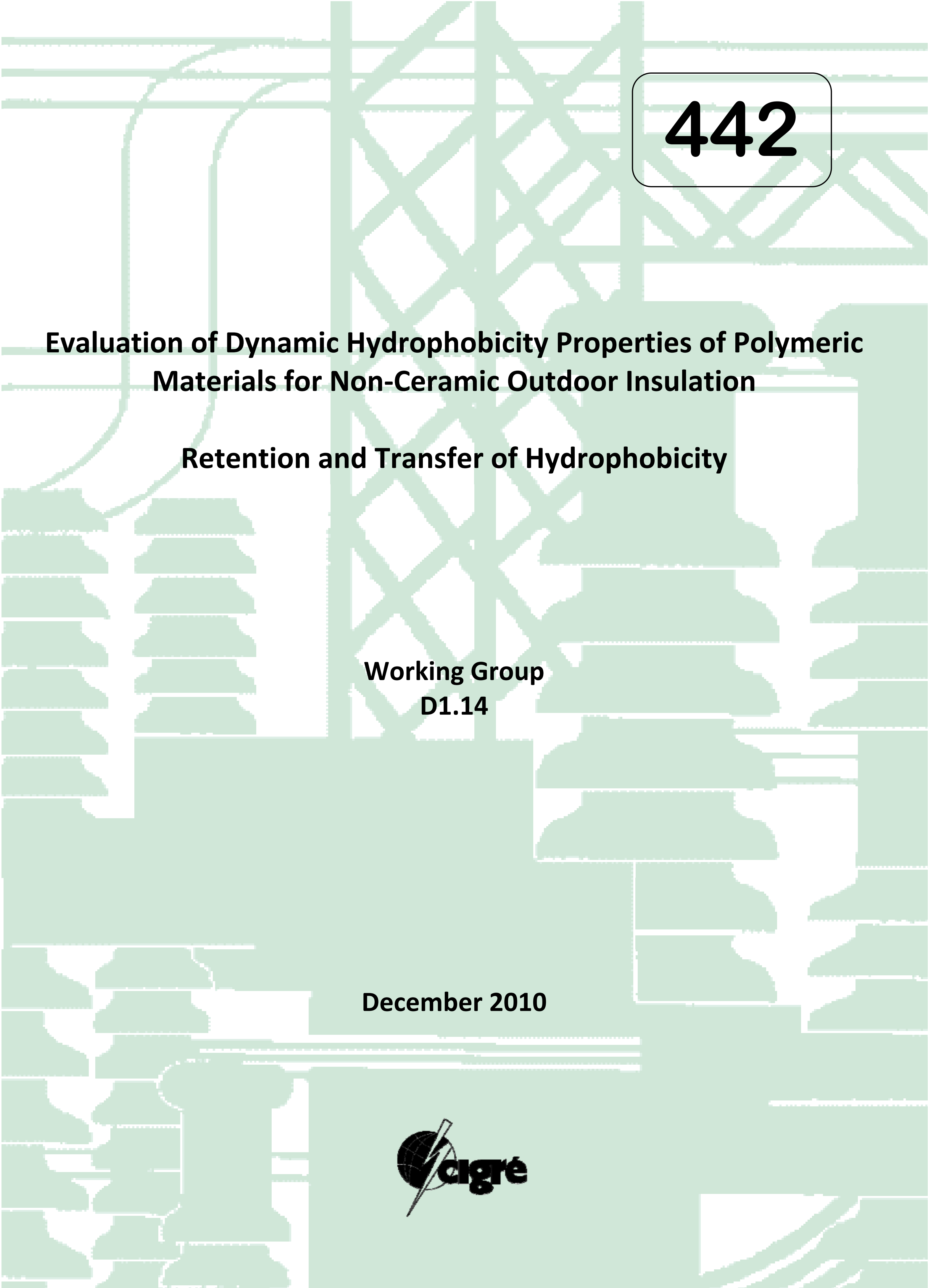

442

Evaluation of Dynamic Hydrophobicity Properties of Polymeric Materials for Non-Ceramic Outdoor Insulation

Retention and Transfer of Hydrophobicity

Working Group
D1.14

December 2010

Evaluation of dynamic hydrophobicity properties of polymeric materials for non-ceramic outdoor insulation – Retention and transfer of hydrophobicity

“Material properties for non-ceramic outdoor insulation”

Working Group D1.14

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TABLE OF CONTENTS

EXECUTIVE SUMMARY

In previous work of CIGRE Working Group D1.14, the need to find suitable test methods and evaluation criteria for the dynamic hydrophobicity properties of insulating materials (CIGRE TB No. 255 “Material properties for a non-ceramic outdoor insulation – state of the art” August 2004), was identified. The working group concludes that the dynamic hydrophobicity properties of insulating materials can be covered by two tests: the first, to evaluate the retention of the hydrophobicity, and the second, to evaluate the hydrophobicity transfer.

This report describes test methods and evaluation criteria for the dynamic hydrophobicity properties of insulating materials. For both tests relevant test methods and evaluation criteria have been developed.

- The retention of hydrophobicity is understood to be the resistance of polymeric insulating materials to loss of hydrophobicity due to pollution initiated surface discharges. The test is based on the so called Dynamic Drop Test, where material samples are subject to simultaneous stress of voltage and drops of NaCl solution that are running down the surface of a tilted sample. The retention of hydrophobicity is evaluated as the time elapsed for the stressed area to change from hydrophobic to hydrophilic state.
- The hydrophobicity transfer is understood to be the transfer of hydrophobicity from the bulk of the sample material onto artificial pollution layers on the surface. The test utilizes artificial pollution layers on the material sample which are applied as slurry. The hydrophobicity transfer is evaluated as the time needed for the applied pollution layer to change from hydrophilic to hydrophobic state under given conditions of storage.

The reproducibility of the test methods has been verified by round robin testing.

No minimum requirements and/or classification of material have been introduced because it was felt that more experience is needed with the application of these tests.

1 Introduction

Under wet and polluted conditions, high voltage insulation with hydrophobic external surfaces show significantly higher flashover voltages than the same insulation with hydrophilic surfaces [1]. Under normal operating conditions such insulation with hydrophobic surfaces show relatively low leakage currents so that critical damages of the insulating material like deep erosion or tracking due to partial surface discharges induced by conductive flow under simultaneous voltage stress can be avoided [2]. Thus, hydrophobicity of external surfaces is considered to be an important factor for the pollution performance of high voltage insulations.

Polymeric materials used for high voltage outdoor insulation may offer long lasting and stable hydrophobicity even under polluted conditions. Hence, the evaluation of hydrophobicity and its dynamic properties like retention, transfer and recovery, are of importance for the choice of materials. It would be helpful for the development of materials and products as well as for quality conformance testing if the dynamic hydrophobicity properties of different insulating materials can be compared and ranked.

In contrast to other material properties no standardized test procedure is available for the evaluation of the properties of materials with respect to hydrophobicity. Hence, it was the purpose of the study to qualify existing test procedures or, if necessary, to develop new test procedures that give reproducible results.

2 Degree of hydrophobicity versus dynamic properties of hydrophobicity

According to IEC 62073 [3] the words hydrophobic and hydrophilic describe two extreme levels of wettability of a surface by water. A surface is called hydrophobic if it has low surface tension and is water-repellent. The opposite is a hydrophilic surface that has high surface tension and thus is wetted by water in form of a film.

If water is sprayed on hydrophobic surfaces distinct water droplets are formed. If water is sprayed on hydrophilic surfaces water spreads on the surface.

The degree of hydrophobicity, i.e. the wettability of external surfaces of insulators can be evaluated by three methods (IEC 62073 [3]):

- Method A: Determination of the contact angle (static or dynamic contact angles)
- Method B: Determination of the surface tension by using different fluids
- Method C: Spray method according to STRI-Guide 92/1 [4]

The methods differ with respect to the necessary equipment, the measuring uncertainty, the required size of the measuring area and its applicability. Method A and B are usually applied in laboratories. Method C is suitable for laboratory measurements and onsite measurement on insulations as well.

Contact angles can be defined on the surfaces of insulating materials. On ideal surfaces of solid materials the interfacial tensions γ_{GL} (Gas-Liquid), γ_{GS} (Gas-Solid) and γ_{SL} (Solid-Liquid) result in an equilibrium angle θ according to the Young equation $\gamma_{GL} \cos \theta = \gamma_{GS} - \gamma_{SL}$ (Figure 1) [5].

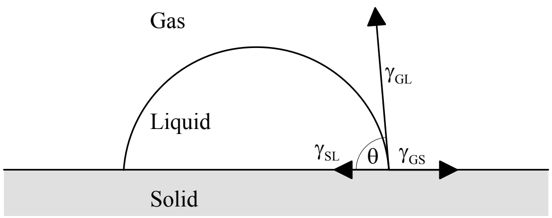

Figure 1: Equilibrium contact angle θ

On real surfaces of solid materials, the so called static contact angle θ_s appears. Further, at a drop rolling down an inclined surface the dynamic contact angles i.e. the advancing angle θ_a and the receding angle θ_r can be defined (Figure 2) [4]. It is possible to measure the dynamic contact angles on horizontal surfaces by increasing or decreasing the drop volume, respectively [5]. The relation of the contact angles is given as $\theta_r \leq \theta_s \leq \theta_a$.

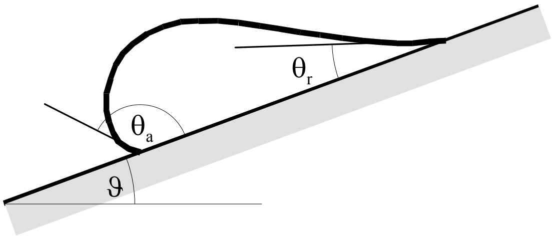

Figure 2: Advancing (θ_a) and receding (θ_r) contact angles on an inclined surface

The contact angle measurement can be used to evaluate the degree of hydrophobicity on clean and polluted surfaces of insulating materials. Further, the degree of hydrophobicity can also be evaluated by measuring partial discharge activity or leakage current of material samples or insulations under electric stress [6], [7]. It has been verified that from the various static and dynamic contact angles the receding contact angle corresponds best to the electrical performance of wetted surfaces [7].

Beside the degree of hydrophobicity, the dynamic hydrophobicity properties of insulating materials are of interest (**Figure 3**) [8], [9].

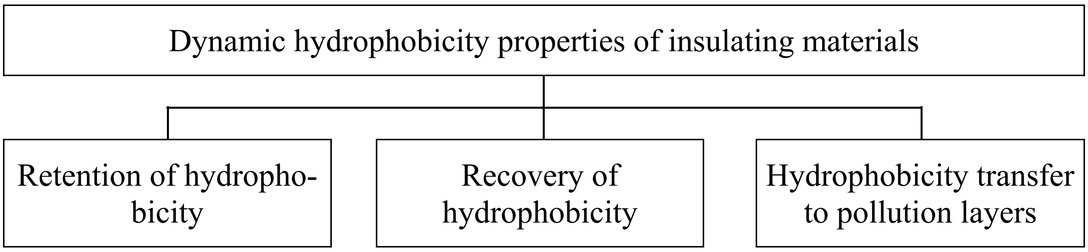

Figure 3 Dynamic hydrophobicity properties of insulating materials (according to [8])

No standardized test procedures exist for the evaluation of the properties of materials with respect to hydrophobicity. Hence, it is necessary to qualify existing test procedures and, if necessary, develop new test procedures which give reproducible results in the evaluation of the properties of materials with respect to hydrophobicity. Such tests for the evaluation of the dynamic hydrophobicity properties would be applicable to housing and structural materials if the material is associated with intrinsic dynamic hydrophobic properties [9], [10]. The test is considered to be applicable also to coating materials.

3 Retention of hydrophobicity

Experience shows that the hydrophobicity of insulating materials can reduce under stresses like water immersion, exposure to UV-radiation or intense electrical discharges [8], [11]. Various test methods for the evaluation of the “loss of the hydrophobicity” give information about the retention of the hydrophobicity under such stresses. Due to the lack of standardised test methods and criteria for the evaluation of the resistance to loss of hydrophobicity, various test methods are in use [9].

3.1 Loss of hydrophobicity due to corona

A needle-plane arrangement has been used to evaluate the loss and recovery of the hydrophobicity of hydrophobic cycloaliphatic epoxy [12]. The needle electrode is positioned in a distance of 10 mm above a test specimen which is positioned on a grounded plane. 18 kV ac (r.m.s) voltage is applied for 150 min.

A multiple needle arrangement, has been used for studying the mechanism of hydrophobicity loss and recovery on polydimethylsiloxane (PDMS) materials [13]. This corona electrode is equipped with 31 needles with tips having a radius of 56 µm (Figure 4). The treated sample was placed on circular glass plates on the grounded plane electrode at a distance of 40 mm from the needle tips. The applied voltage was up to 20 kV ac (r.m.s). The electrodes are mounted in a desiccator to provide a controlled atmosphere.

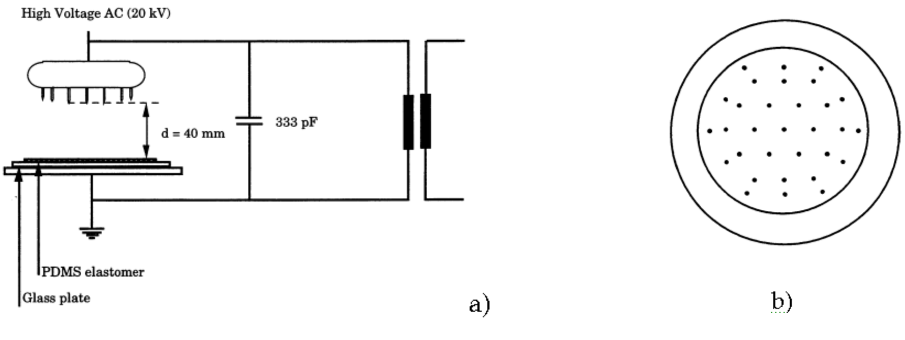

Figure 4: Multiple needle arrangement used by Hillborg [13] for corona treatment of silicone rubber (a) and positioning of the needles as seen from below (b).

3.2 Loss of hydrophobicity due to sustained wetting

Sustained wetting of silicone elastomers may lead to a loss of hydrophobicity. When samples of silicone elastomers are stored in water (distilled water at room temperature) a loss of hydrophobicity can be observed within a few hundred hours [8], [14]. The effect is explained by the reorientation of polymer chains near the surface. Remarkably the effect of water storage on the hydrophobicity is much more pronounced on the receding contact angle than on the static or advancing contact angle.

3.3 Loss of hydrophobicity due to UV-radiation

UV-radiation may influence the degree of hydrophobicity of polymeric materials. In EN 828 [15] it is proposed to determine the influence of UV-radiation on the wettability of polymeric materials by using contact angle measurement. The measurement according to the “dynamic advancing drop method” is recommended.

3.4 Loss of hydrophobicity due to pollution initiated surface discharges

From a practical point of view, the most important scenario for loss of hydrophobicity is the simultaneous stress of the surface of polymeric insulations by voltage and pollution layer. The pollution initiated surface discharges occurring under such conditions may cause erosion and eventually tracking of the housing or structural material of the insulation. Various test methods have been developed to test the external surface of insulations or the surface of material samples with respect to their resistance to loss of hydrophobicity due to pollution and simultaneous electric stress.

3.4.1 Evaluation of different test procedures

For the purpose of fundamental studies special test procedures with water condensation have been developed [16], [17]. By using fog chamber and rotating wheel dip tests the loss of hydrophobicity can be achieved according to the relevant test parameters, i.e. specific electric stress and conductivity of the electrolyte. The mechanism causing loss of hydrophobicity is the development of partial discharges in connection with water drop corona [8], [17], [18]. The criterion for the loss of hydrophobicity is the characteristic change of the leakage current [6], [8], [11], [16].

In fog chamber tests, the samples are subjected to salt fog and simultaneous electrical stress (Figure 5). By intention the samples should have super smooth surfaces. The report about development of a test technique to assess a polymer’s long term ability to suppress leakage currents under high voltage and low conductivity salt fog conditions by CIGRE WG 15.04 concluded that the test results were very sensitive to variations of the fog conditions and surface defects of the samples. The testing of several rods made from silicone rubber (SIR) with small surface defects such as roughness and small cracks demonstrated that the initiation and development of leakage current on SIR rods are strongly related to the surface quality of the SIR samples. Therefore, it is recommended that the test samples should have a similar surface quality as the actual insulators produced with this material [19].

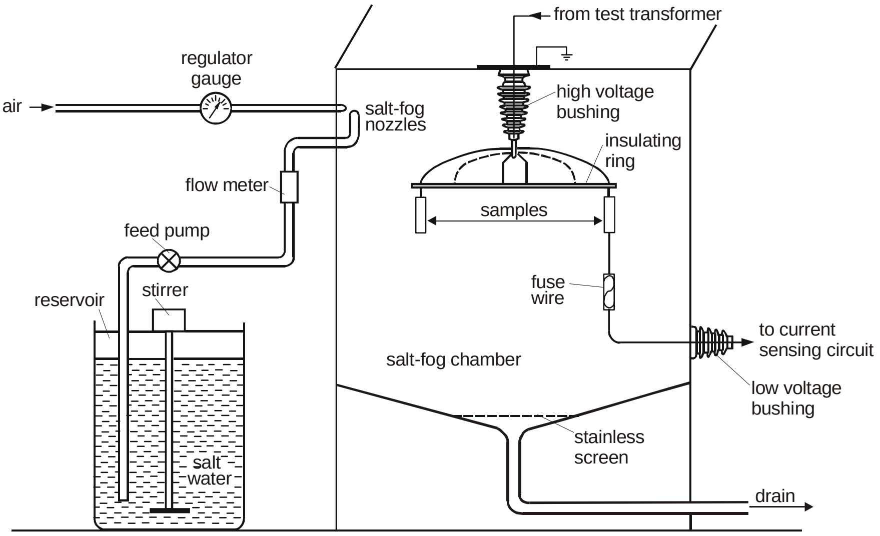

Figure 5: Test setup (schematic) for the performance of salt fog test according to [19]

In the Modified Rotating Wheel Dip Test (MWDT), cylindrically shaped test specimens of about 30 mm diameter and 100 mm in length are used. 4 specimens are mounted on a wheel, which moves 90 degrees after each 60 s (Figure 6). In contrast to wheel tests for other purposes, i.e. tracking and erosion testing, the Modified Rotating Wheel Dip Test uses different stress levels and a different evaluation criterion. For the Modified Rotating Wheel Dip Test, NaCl-solution with a conductivity of 1,5 mS/cm is proposed for silicone elastomers and the recommended specific electrical stress is 0,6 kV/cm [8], [9].

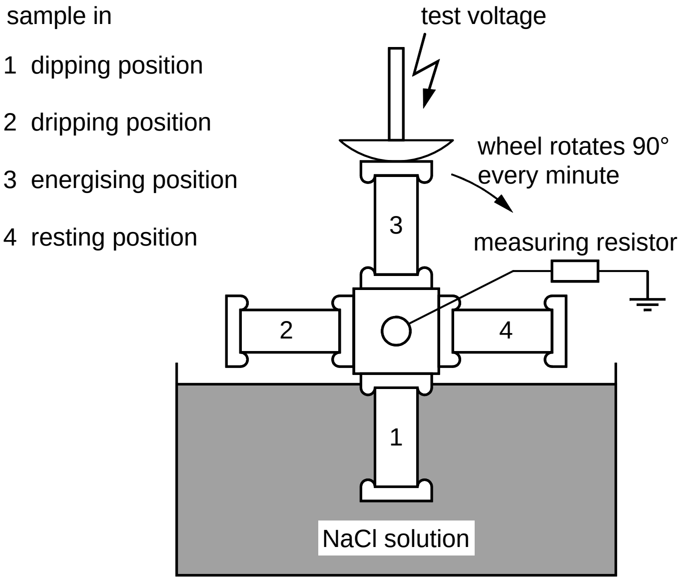

Figure 6: Test setup for rotating wheel dip test (schematic)

Both salt fog and Modified Rotating Wheel Dip Test show relatively large scatter of results and suffer from the difficulty to produce cylindrical test samples with smooth surfaces [8], [20]. Further, the reproducibility has not been proven yet [21].

With both salt-fog and rotating wheel dip test it can be observed that electrical discharges occur at the ground electrode when rolling drops contact the electrode. The hydrophobicity is reduced with the number of drop-initiated discharges. The number of water drops rolling down depends on various factors and is subject to significant variation. This explains the large statistical scatter observed in the test results [9]. In order to get reproducible results the number and frequency of water drop initiated discharges must be controlled.

3.4.2 Dynamic Drop Test

As an alternative to the Modified Rotating Wheel Dip Test described above, the Dynamic Drop Test was developed to evaluate the resistance of hydrophobicity against simultaneous stress by drops and voltage [22]. The main aim of this test was to allow flat plate material samples, to get results in short time and to reduce their scatter by controlling the roll-off frequency of water drops causing partial surface discharges at the electrodes [20].

On the basis of experience with the available test methods the Dynamic Drop Test has been considered the most promising test method to achieve reproducible results for testing resistance to loss of hydrophobicity due to conductive flow and simultaneous electric stress.

The test setup of the dynamic drop test consists of flat material samples, tilted by 60 degrees to the horizontal and having two electrodes separated by 50 mm (Figure 7). An example of the test setup is shown in (Figure 8). The flow of the electrolyte can be generated by a peristaltic pump.

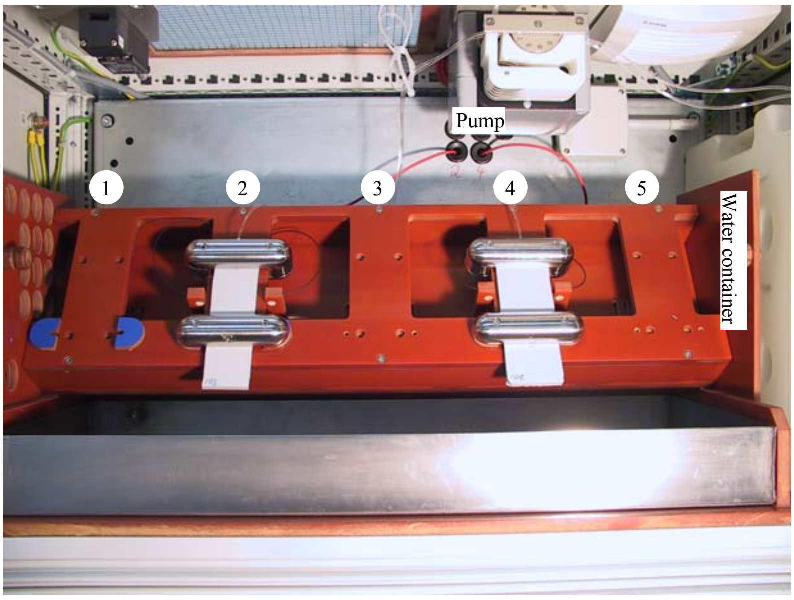

Figure 7: Example of electrode arrangement for Dynamic Drop Test

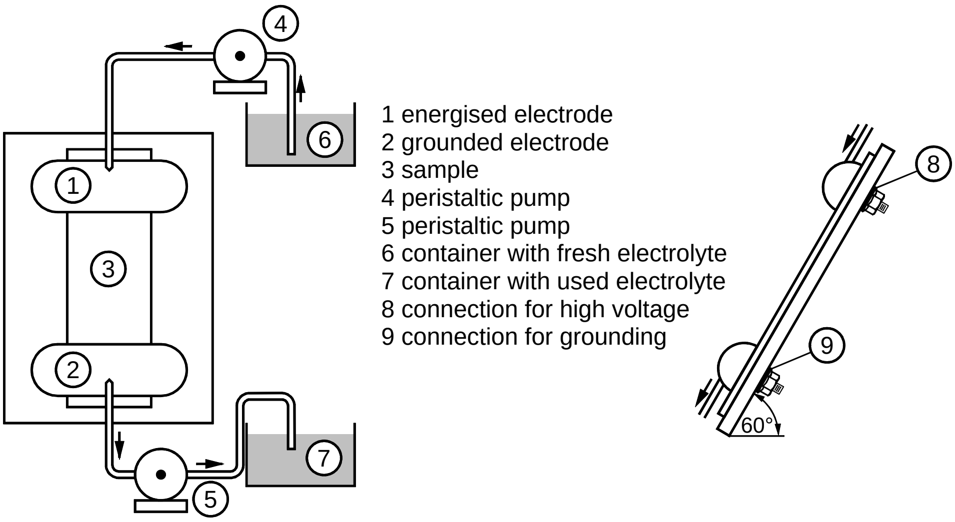

Figure 8: Test setup for dynamic drop test (schematic)

Depending on the flow rate, drops roll off the surface with a certain frequency. In the initial phase the drops may roll off in different paths. There is also a significant number of mini drops remaining on the roll-off paths on the sample. Therefore, it is recommended to precede testing by a calibration period without voltage. During the calibration period the roll off process is stabilizing so that the drops roll off in the same path and the number of mini drops left behind is more or less constant. At the end of the calibration period which may last up to five minutes the ac test voltage shall be switched on. Given a constant flow rate the drop frequency is increasing with increasing voltage due to electrostatic forces which act on the drop at the upper electrode [20].

Due to micro discharges caused by electrolytic flow along the surface and the simultaneous voltage stress, the hydrophobicity is partially lost (Figure 9 a und b). At a later phase the electrolyte may form a continuous path between the electrodes (Figure 9 c) until eventually complete loss of hydrophobicity occurs and the electrolyte forms a continuous path (Figure 9 d).

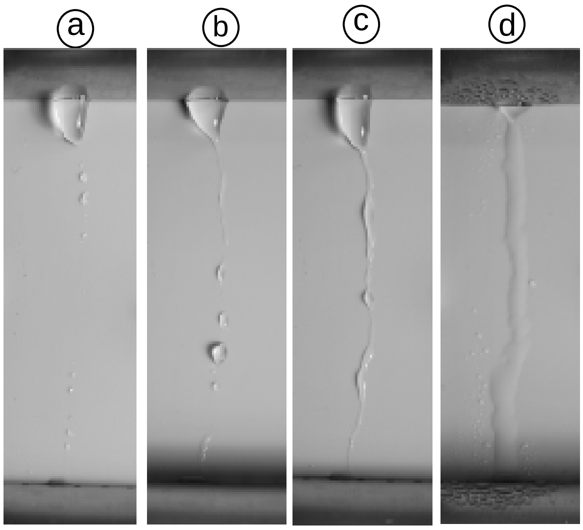

Figure 9: Photos of different stages during testing [20]

The different phases which can be observed visually (Figure 9) can also be diagnosed by the development of the leakage current (Figure 10). During phase 1 and phase 2 while individual drops roll off the surface the current is nearly purely capacitive and amounts to a few µA. The occurrence of a liquid path for a short moment leads to sudden peaks in the leakage current. Since the material under test is still hydrophobic the liquid path is interrupted until the next drop rolls off (phase 3). When hydrophobicity is lost partially along the whole drop path a continuous path of electrolyte is formed and the leakage current is nearly continuous with values from few hundred µA to few mA. The occurrence of such currents indicates the loss of hydrophobicity (phase 4) and is used as a criterion to determine the failure time [20].

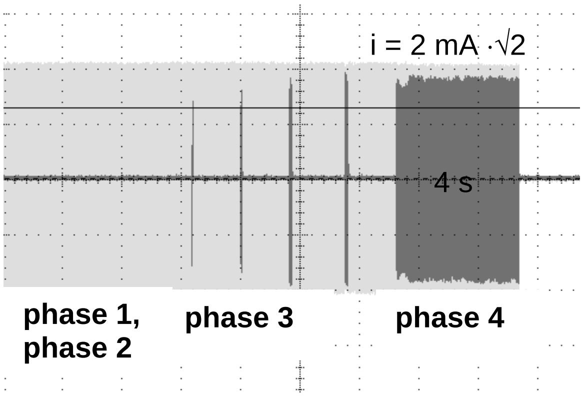

Figure 10: Development of leakage current close to the moment when hydrophobicity is lost, i.e. the failure criterion is reached in the Dynamic Drop Test [20]

Like in similar test procedures, the time until loss of hydrophobicity decreases with increasing test voltage as well as with increasing conductivity of the electrolyte.

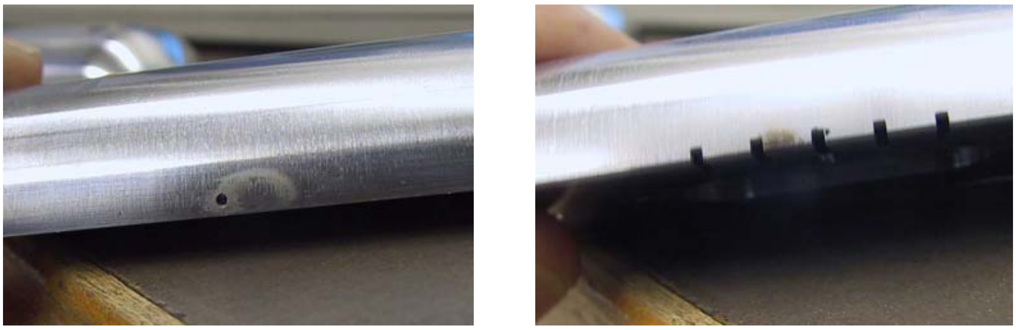

Figure 11: Corrosion of high voltage electrode (left) and ground voltage electrode (right)

In the initial phase of round robin testing using this test method it turned out that corrosion of the electrodes was the main reason for unexpectedly high scatter as well as for lack of reproducibility. Under the influence of electric stress and the presence of electrolyte the electrodes may corrode although they are made from stainless steel and even had been passivated chemically to avoid corrosion (Figure 11). Corrosion of electrodes was observed when the current is not shut off after hydrophobicity has been lost, e.g. when the current criterion is set too high. When such signs of corrosion occur, the drops need to get a larger volume until they start to roll off. In comparison to drops with smaller volume, large drops elongate and may momentarily bridge the electrode distance chosen for the test. If this phenomenon occurs, partial discharges become visible and the sample may fail within a few minutes. The failure time is significantly reduced with increasing drop volume while the corrosion becomes more severe (Figure 12).

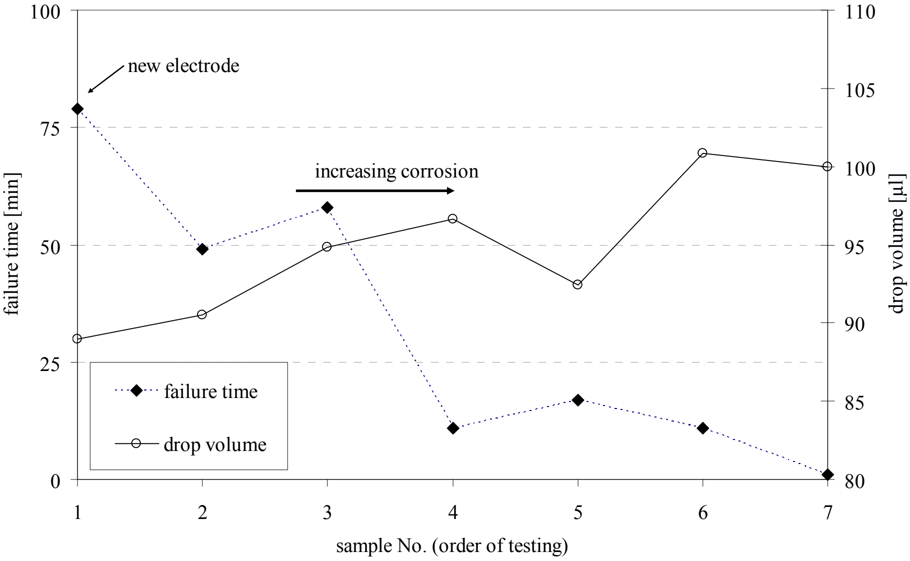

Figure 12: Reduction of failure time and increase of drop volume with increasing corrosion of the electrode with a RTV silicone rubber tested at 5 kV [20]

The relation between time to failure and the drop volume was investigated at two voltage levels (Figure 13). It turned out that the effect was more pronounced with increasing test voltage.

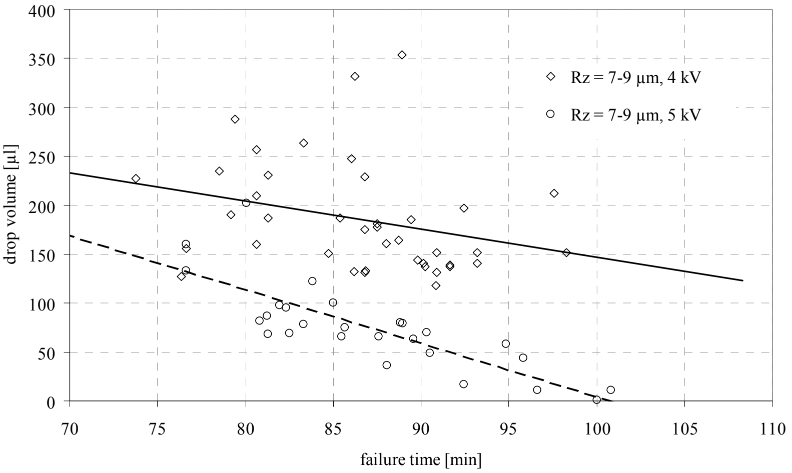

Figure 13: Relation between failure time and drop size measured with silicone elastomer (RTV) having surface roughness of 7 to 9 µm

To avoid any adverse effects by corrosion of electrodes it is necessary to use materials with high corrosion resistance. If signs of corrosion occur these should be wiped off by polishing with suitable detergents. Polishing should result in a surface roughness $R_Z = 5$ µm of the electrodes.

Another important factor is the surface roughness of the material sample. Different degrees of surface roughness may influence the roll-off performance of the electrolyte and the resistance to loss of hydrophobicity. In general, it can be stated that the failure time decreases with increasing surface roughness (Figure 14). It should be noted that the test conditions used for the test shown in Figure 14 were similar, but not exactly the same as those used for the Round Robin Test performed later. Hence, the results may not be comparable to those gained during Round Robin Testing as shown below.

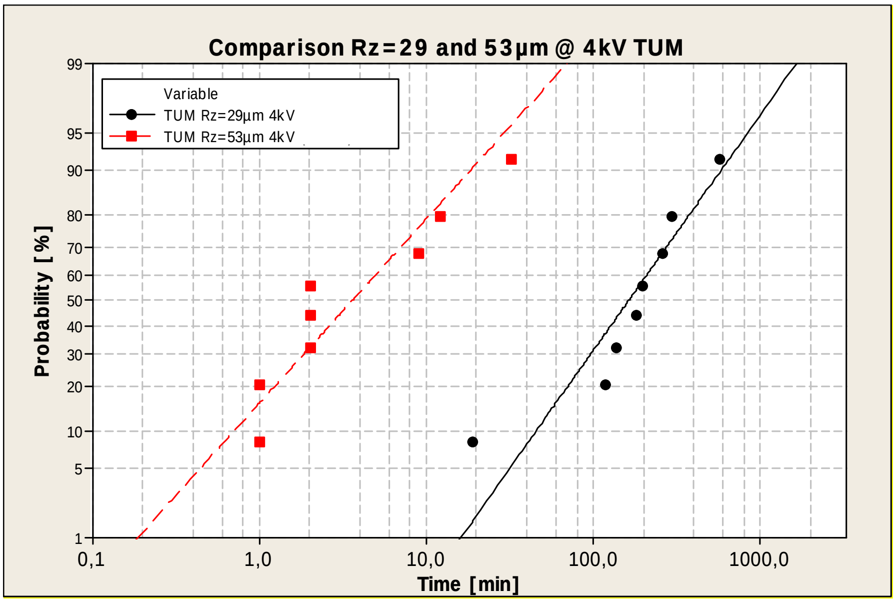

Figure 14: Influence of surface roughness on failure time measured in laboratory of TUM

As a result of the extensive pretesting, suitable parameters could be found for the Dynamic Drop Test. The test parameters are specifically suitable for silicone elastomers (see Table 1) [20].

Table 1: Test parameters for Dynamic Drop Test

Test parameter	Value
distance between electrodes	50 mm
test voltage (50/60 Hz)	3 - 6 kV
conductivity of electrolyte	1,5 ± 0,2 mS/cm
flow rate	1,0 ± 0,2 ml/min
drop frequency	12 ± 1 min^{-1}
failure criterion	I = 2 ± 0,5 mA (rms) for 4 ± 0,5 s

3.4.3 Round robin testing using the Dynamic Drop Test

In order to check the reproducibility of the Dynamic Drop Test a round robin test has been performed. Laboratories and responsible persons participating in the round robin testing:

- Prof. Dr. Josef Kindersberger, (Project Coordinator)
  Technische Universität München (TUM)
  Dept. of Electrical Engineering and Information Technology
  Theresienstrasse 90, 80333 München, GERMANY
- Prof. Dr. Roland Bärsch
  University of Applied Science Zittau/Görlitz (Zittau)
  Theodor-Körner-Allee 16, 02763 Zittau, GERMANY
- Prof. Dr. Masayuki Nagao
  Dept. of Electrical and Electronic Engineering

Toyohashi University of Technology (TUT)
Tempaku, Toyohashi, 441-8580, JAPAN

- Prof. Dr. Masahisa Otsubo
  Dept. of Electrical and Electronic Engineering, Faculty of Engineering,
  University of Miyazaki
  1-1 Gakuenkinbanadai-Nishi, Miyazaki, 889-192, JAPAN
- Prof. Dr. Takaie Matsumoto
  Dept. of Electrical and Electronic Engineering
  Shizuoka University
  3-5-1 Johoku, Naka-ku, Shizuoka, 432-8561, JAPAN

Based on the results of the above mentioned pretesting the test procedure and the test parameters had been chosen. For details see Appendix A. The Round Robin Testing (RRT) was performed with RTV silicone rubber. The test voltage was 4 kV and 5 kV, respectively.

To quantify the effect of roughness also in the RRT it was intended to produce samples with a surface roughness $R_Z = 5$ µm und $R_Z = 10$ µm. The samples finally had a surface roughness $R_Z = 7$ µm and $R_Z = 9$ µm. These samples had been tested with an electrolyte having a conductivity of 1.5 mS/cm. No significant difference between the samples having a roughness of $R_Z = 7$ µm and $R_Z = 9$ µm could be found. Therefore, the samples are considered to belong to the same one population and the results are treated accordingly.

The statistical evaluation showed that the distribution of the results fit best the normal distribution function. Accordingly all results are given in plots scaled according to the normal distribution function including the confidence internal for 95% probability. The results using samples with roughness $R_Z = 9$ µm and voltage stress of 4 kV and 5 kV are shown for the laboratories participating in the RRT (Figure 15 to Figure 19).

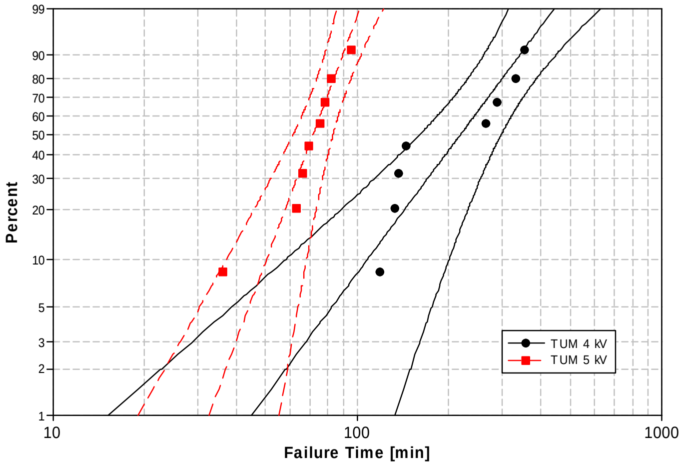

Figure 15: Influence of the test voltage on failure time measured in laboratory of TU München (TUM)

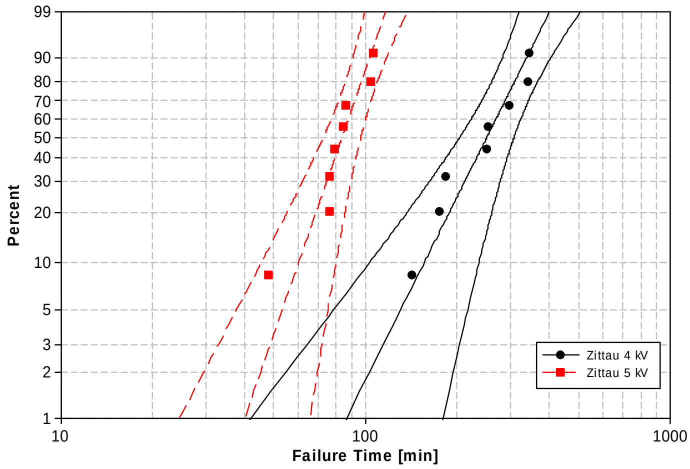

Figure 16: Influence of the test voltage on failure time measured in laboratory of University of Applied Science Zittau/Görlitz (Zittau)

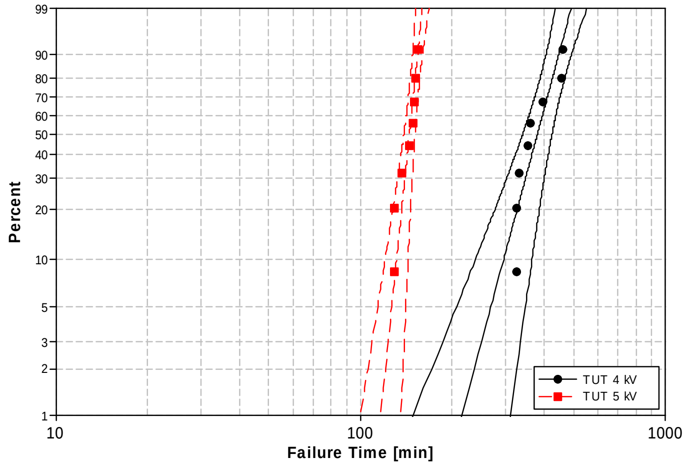

Figure 17: Influence of the test voltage on failure time measured in laboratory of Toyohashi University of Technology (TUT)

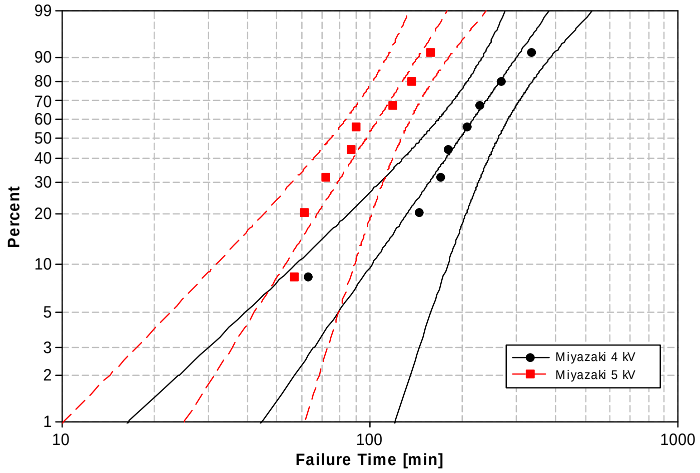

Figure 18: Influence of the test voltage on failure time measured in laboratory of University of Miyazaki (Miyazaki)

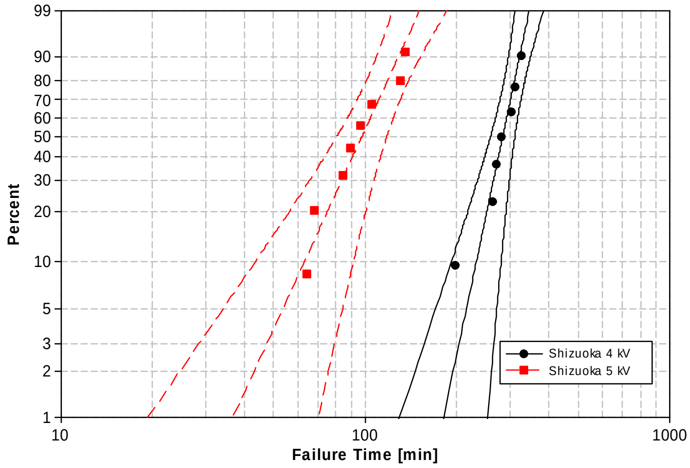

Figure 19: Influence of the test voltage on failure time measured in laboratory of Shizuoka University (Shizuoka)

The results from all participating laboratories are shown in Figure 20 for an applied voltage of 5 kV.

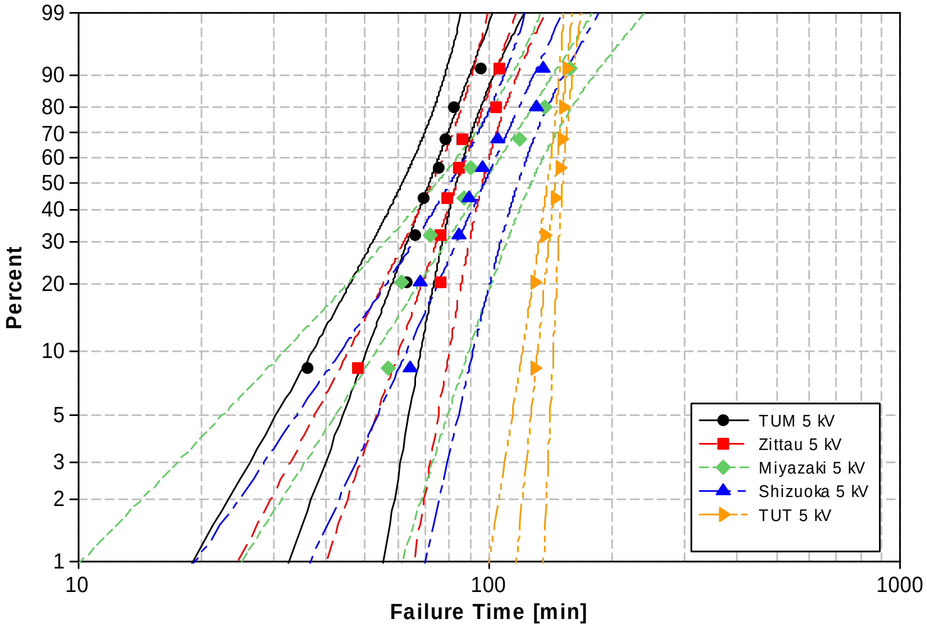

Figure 20: Failure time in Dynamic Drop Test with 5 kV from all participating laboratories – distribution functions and 95% confidence intervals

The comparison of the 50% quantiles of the distribution function including the relevant 95% confidence intervals is shown in Figure 21. It is obvious that there is no significant difference between the results from laboratory in Munich, Zittau, Miyasaki and Shizuoka. Only the results from Toyohashi University (TUT) show a longer failure time. This is valid for 4 kV and 5 kV (Figure 21).

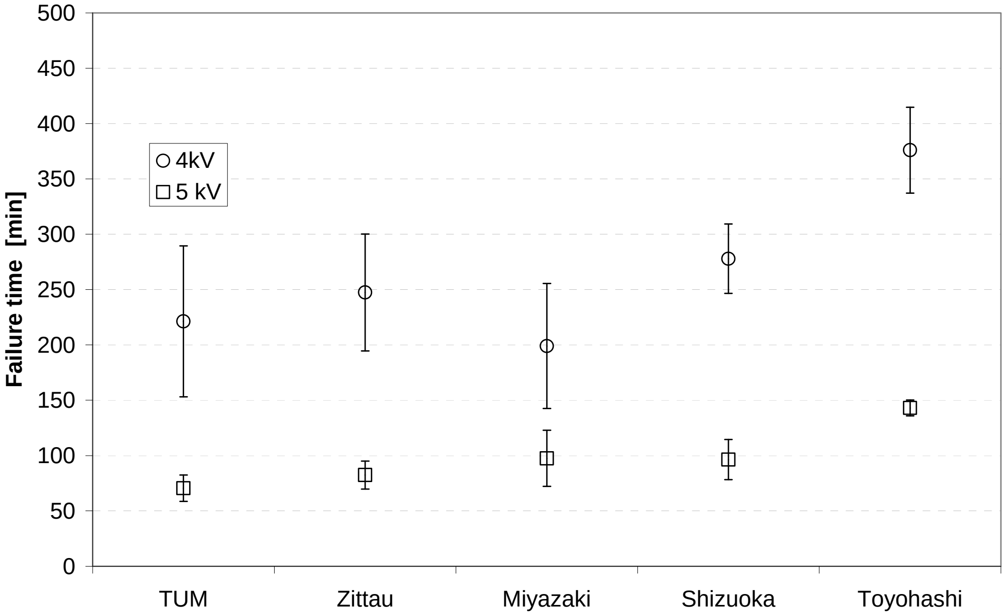

Figure 21: Failure time in Dynamic Drop Test with 4 kV and 5 kV – 50% quantiles and 95% confidence interval of the distribution function

Although it could not be clarified why one of five laboratories showed significantly higher failure times, it can be concluded that the test procedure applied for the round robin test (Appendix A) leads to reproducible results.

3.4.4 Recovery of hydrophobicity tested by Dynamic Drop Test

As already pointed out in a previous report any comparative evaluation method for recovery of hydrophobicity should start at the same low level of the hydrophobicity [9]. To reach the same low level of hydrophobicity would require different pre-stressing of different materials. Consequently, a material which shows good and stable hydrophobicity would become pre-stressed much more intensively to destroy the hydrophobicity than a weak material. Hence, it would be doubtful whether a comparison of the ability to hydrophobicity recovery is reasonable under such conditions.

Besides this, the dominating mechanism leading to recovery of hydrophobicity is the migration of low molecular weight components. This mechanism is evaluated with the hydrophobicity transfer test (see section 4).

Nevertheless, the Dynamic Drop Test has also been used to check whether it is suitable to test for the recovery of hydrophobicity. For this purpose, samples were tested until they lost their hydrophobicity. After a certain recovery time the samples were tested again. Two different recovery times, i.e. 24 h and 168 h had been applied. Despite this difference the results are displayed showing the failure time after recovery tested with 4 kV (Figure 22) and tested with 5 kV (Figure 23). It seems that the Dynamic Drop Test has the potential to give reproducible results for the property of recovery of hydrophobicity. Attention has to be paid to the details of the test. It is mandatory that the position of the electrode with respect to the sample and in particular with respect to the wetted path in the recovery test must be the same as in the initial test.

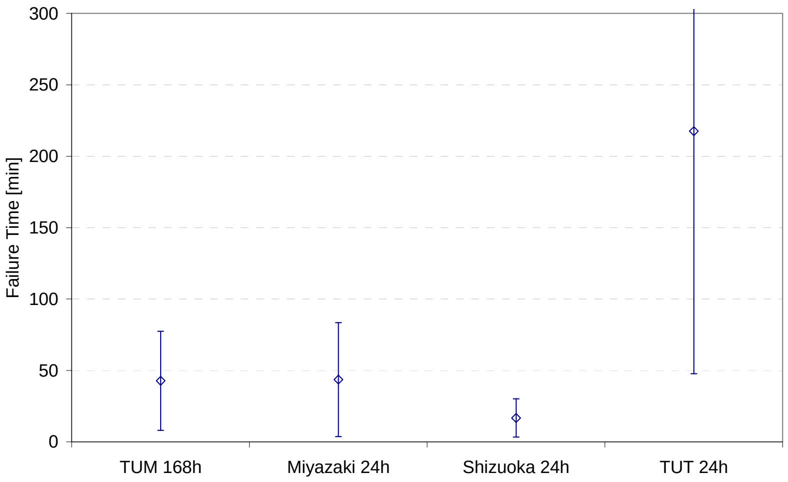

Figure 22: Failure time in Dynamic Drop Test with 4 kV after recovery for 168 h and 24 h – 50% quantiles and 95% confidence interval of the distribution function

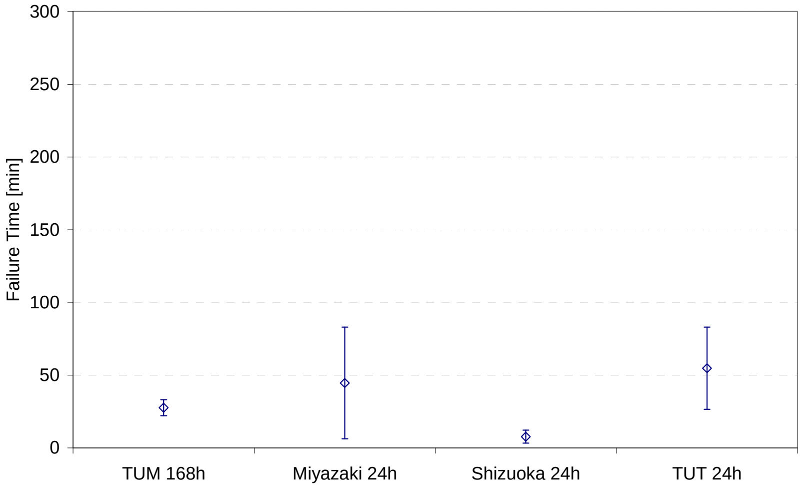

Figure 23: Failure time in Dynamic Drop Test with 5 kV after recovery for 168 h and 24 h – 50% quantiles and 95% confidence interval of the distribution function

3.4.5 Conclusion of Round Robin Testing with the Dynamic Drop Test

The retention of hydrophobicity, i.e. the resistance to loss of hydrophobicity due to surface discharges has been evaluated by using the Dynamic Drop Test (according to Appendix A).
By round robin testing it was confirmed that the Dynamic Drop Test leads to reproducible results.

No minimum requirements and/or classification of material have been introduced because it was felt that more experience is needed with the application of these tests.

4 Transfer of hydrophobicity

Insulating materials with intrinsic dynamic hydrophobic properties like silicone elastomers are able to transfer hydrophobic properties on pollution layers. The hydrophobicity transfer is due to the diffusion of low molecular weight components (LMWs) from the bulk material into the pollution layer even if the pollution layer is hydrophilic [23], [24], [25]. There is a need to evaluate the ability of materials with respect to transfer hydrophobic properties onto pollution layers by reproducible test methods. The test method shall be applicable for bulk material as well as for (RTV-)coatings.

4.1 Application of artificial pollution layer

Various test methods are available in the literature using different types of materials for the pollution layer as well as applying different methods for application of these layers [26]. Pollution layers can be produced by mixing slurry based on different silica powders, kaolin or kieselgur and a liquid. The liquid can be tap water, distilled water or alcohol and can contain a wetting agent or even salts like NaCl which is used to produce hygroscopic pollution layers [8]. The layer thickness can be adjusted by covering the samples with a glass plate at a defined distance [9]. It is also possible to dip the samples into the slurry [18], [23] but the dipping method limits the layer thickness to very thin layers of about 0.05 to 0.1 mm [23], [27]. For pollution testing a pollution layer is produced on glass and porcelain insulators by spraying slurry onto the samples [28]. A combination of spraying and dipping was investigated in [29], which also resulted in rather thin pollution layers. Another method to apply artificial pollution layers is to disperse a powdery substance (e.g. silica powder (SiO_2), kaolin, kieselgur etc.) onto the surface of the insulating materials. The requested layer thickness can be adjusted by wiping off the excess powder with a suitable tool [30] or by weighing [31], [32]. The resulting pollution layer is not firmly fixed and the samples have to be treated very carefully.

The results obtained with all of the methods mentioned above cannot be compared to each other due to the different layer materials and layer thicknesses. The use of different materials leads to significantly different results [32].

To make the test reproducible it is mandatory to apply artificial pollution layers with defined thickness having a smooth surface without any defects. The solid material used for the pollution layer should be available all over the world, should be produced in an industrial process (not a natural material from the soil) and should have no surface treatment that is difficult to control and check. Based on these requirements untreated silica powder with a medium grain size of 3 µm (SF600) was chosen. Attention has to be paid to possible surface treatment of silica powder, e. g. by silanes. Although silane treated silica powders offer an improved applicability onto the specimens it is recommended to use untreated silica powder for investigations on the hydrophobicity transfer because the fraction and distribution of silanes in a batch of silica powder may vary significantly and thus may influence the results.

Material samples of the dimensions 50 mm x 50 mm were used for the test. A thickness of 6 mm was chosen as it avoids the influence of unwanted thickness variations [23]. Prior to testing the specimens were first cleaned with isopropanol and then water. After that they were stored at room temperature for at least 24 hours [26]. When the test is performed with coatings, glass plates proved to be useful as substrates. Attention has to be paid to the thickness of the coating.

The artificial layer material is applied in form of slurry. To control the thickness of the applied slurry the sample the specimens were covered with adhesive foil in such a matter that a window of the dimensions (LxW) 30 mm x 30 mm was obtained (Figure 24). The thickness of the adhesive foil defines the thickness of the pollution layer. By putting several foils on top of each other different layer thicknesses can be achieved.

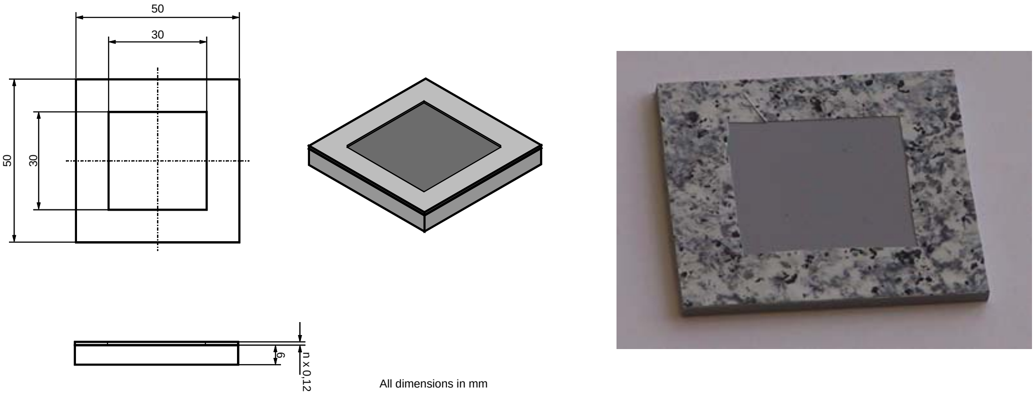

Figure 24: Specimen with adhesive foil (acc. to [26], [33])

The use of untreated silica powder and distilled water to produce pollution layers may lead to an uneven pollution layer and to cracks when the slurry dries out. This is especially true for thicker layers (Figure 25).

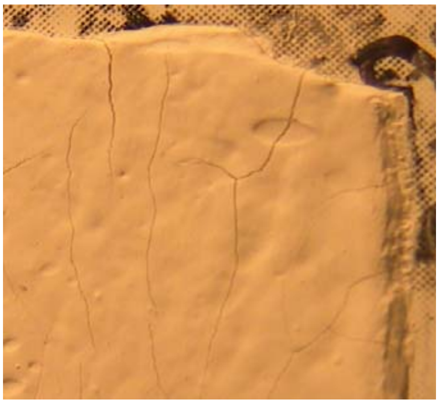

Figure 25: Pollution layer with cracks, layer thickness 0.6 mm (acc. to [26])

A slurry made of 7.5 mg silica powder (untreated i.e. not silanised, medium grain size 3 µm) and 3.5 ml of a mixture of water and isopropanol (65 vol.-% water, 35 vol.-% isopropanol) results in a smooth and plane pollution layer surface. Details on the influence of surface treatment of the powder, the grain size and the pollution layer thickness are given in [26].

The temperature in the laboratory during the whole test procedure is kept constant at 23±2°C.

4.2 Evaluation of the hydrophobicity transfer

The evaluation of the hydrophobicity transfer can be performed by monitoring the development of the degree of hydrophobicity during the transfer process. The degree of hydrophobicity can be determined in the same way as discussed in section 2. Further, it is possible to evaluate the leakage current of a pollution layer as a function of hydrophobicity transfer [8], [36] or by using a drop-roll-of method like proposed in [31]. For practical purposes the contact angle method was used. The static contact angle was chosen although the dynamic contact angles, especially the receding angles exhibit a higher degree of correlation with the electrical properties of the insulating material surface than the static contact angle [7]. Evaluating the transfer of the hydrophobicity with the presented procedure it was found that the receding angle needed more time to reach its initial value than the static and advancing angles [8]. However, for an evaluation and comparison of different insulating materials with regard to the hydrophobicity transfer property the static contact angle is sufficient [33].

Static contact angles were measured after certain time intervals after the slurry was applied. The volume of the drops was 5 µl. For each point in time only one drop per specimen is applied in the centre area of the specimen (Figure 26). The drops shall be distributed in this centre area i.e. no measurements shall take place on the exact same location on the sample.

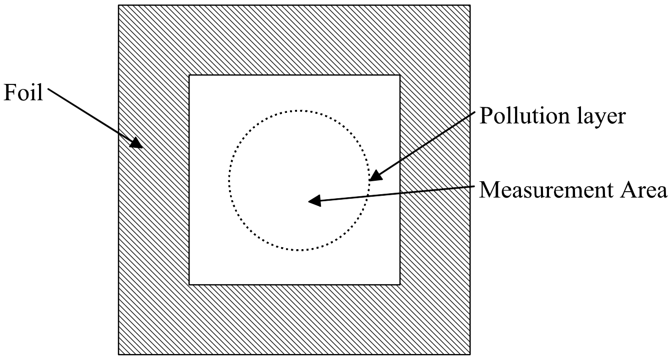

Figure 26: Area for the drop application on the sample

4.3 Storage of the Specimens

The results of the first Round Robin Tests (RRT) indicated a possible influence of the humidity during the storage. Therefore, it was decided to store the specimens at a defined relative humidity. To keep the necessary equipment simple and to avoid possible draught the specimens were stored under controlled humidity in desiccators over aqueous solutions (Figure 27) according to [34].

Tests at TU München were undertaken with the following relative humidities (RH.):

- 33% RH. with a magnesium chloride solution
- 53% RH. with a magnesium nitrate solution
- 75% RH. with a sodium chloride solution

5 specimens were put into the desiccator right after the application of the slurry. The specimens were taken out of the desiccators for the determination of the degree of hydrophobicity and then put back.

Figure 27: Desiccator for the storage of the polluted specimens over aqueous solutions

4.3.1 Pollution layer thickness

A very important parameter for any procedure to evaluate the hydrophobicity transfer property is the thickness of the artificial pollution layer. As a matter of fact the thickness of the pollution layer after drying out of the liquid components of the slurry is significantly less than the thickness of the applied slurry. To investigate the thickness and the homogeneity of the dried layer (in contrast to the thickness of the adhesive foils) a laser profilometer was used (Figure 28).

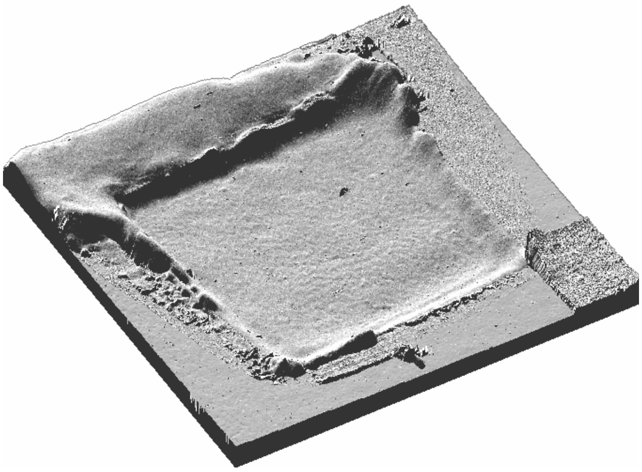

Figure 28: Scanning laser profilometer picture of a specimen with artificial pollution layer (heights are overdone) [33]

To investigate the homogeneity as well as the thickness of the artificial pollution layer, profiles with a length of 40 mm were measured in the middle of the specimens in the direction of the main axis (Figure 29).

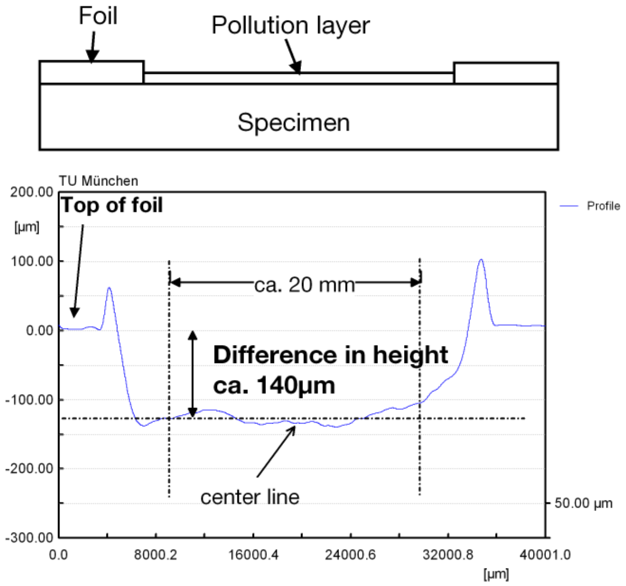

Figure 29: Profile of a specimen with pollution layer, foil thickness 0.36 mm ([5], [33])

It can be seen that the thickness of the artificial pollution layer deviates only by a few µm from the center line in the area which is unaffected by fringe effects. Therefore, the dry pollution layer can be considered to be even and homogeneous [33]. To avoid an influence on the measurement of the contact angles the drops are exclusively place in the area of a approximately 10 mm radius around the center of the pollution layer.

The systematic evaluation of the pollution layer thickness was done with

- 51 RTV specimens, stored in PE containers without humidity control
- 75 RTV specimens, stored in desiccators at controlled humidity
- 55 LSR specimens, stored in desiccators at controlled humidity

In total a number of 181 specimens were available for the measurement of the layer thickness. The thickness of the foils was 0.36 mm (3 foils á 0.12 mm). To investigate a possible influence of the kind of polymeric material or the storage condition on the pollution layer the 2t-test with 5% error probability was used to check the results of the test series. The test shows that the samples are derived from one population. An influence of the kind of material or the storage conditions on the pollution layer thickness can be excluded. The measurements confirm the earlier published measurements [5] and result in a mean value of 0.162 mm for the thickness and a standard deviation σ of 0.023 mm. Figure 30 shows the cumulative frequency based on a normal distribution of the measurement results [33].

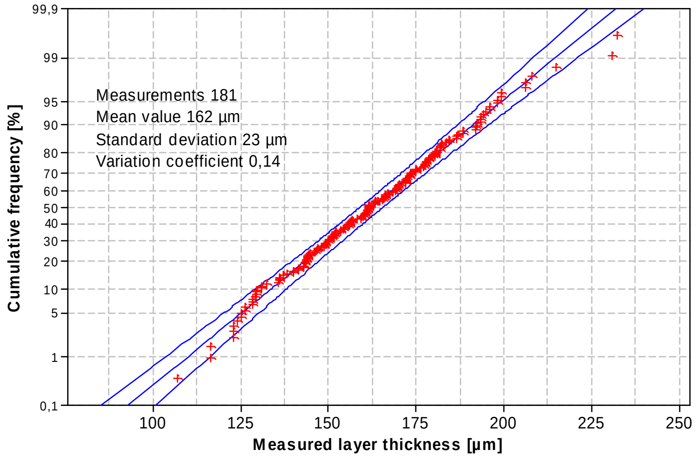

Figure 30: Cumulative frequency function with 95% confidence interval of the measured artificial pollution layer thickness [33]

For practical reasons the thickness which is mentioned in connection with hydrophobicity transfer test is the thickness of the applied slurry, i.e. the thickness of the foils unless otherwise stated.

4.3.2 Influence of humidity during storage on the hydrophobicity transfer

The first measurements at controlled humidity were undertaken at 53% RH. as this is close to the standard atmosphere of 50% RH. recommended in [35]. In several test series it was confirmed that the results of the measurements at a controlled humidity of 53% are repeatable (Figure 31) [33].

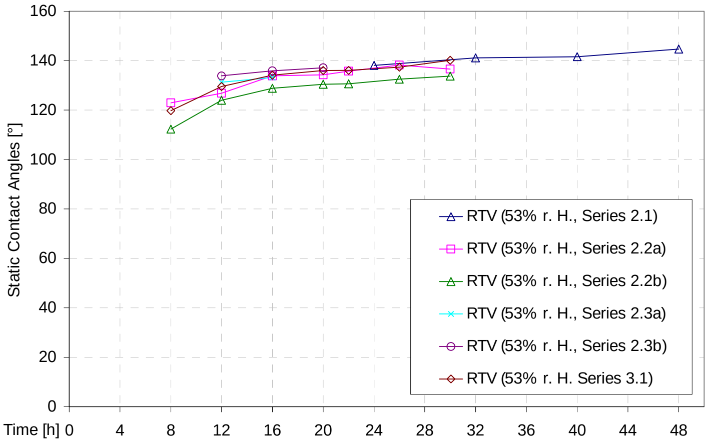

Figure 31: Hydrophobicity transfer at 53% RH. in desiccators, material RTV, 6 independent measurement series

In previous test series samples were stored in containers made from polyethylene (PE) with small holes in the lid. The humidity in the PE container was not controlled and the relative humidity in the laboratory varied from 35% to 65%. Compared to the samples stored under controlled conditions in the desiccator, the hydrophobicity transfer is slower for the samples stored in the PE container under uncontrolled humidity (Figure 32). The specimens which were stored in PE containers with small holes in the lid (uncontrolled humidity) exhibit a noticeable hydrophobicity transfer ($\theta_S > 100°$) at about 24 hours after application (Figure 32). On the other hand the specimens stored in desiccators already show a noticeable hydrophobicity transfer after 8 hours (Figure 31).

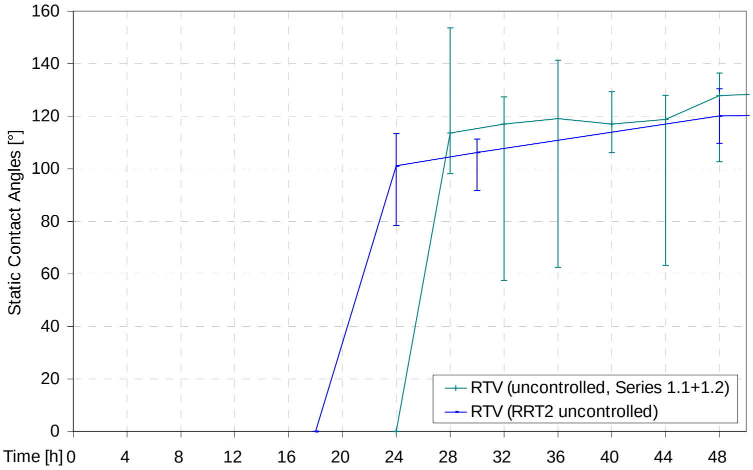

Figure 32: Hydrophobicity transfer by RTV samples stored at uncontrolled relative humidity in PE containers; results from 2 independent test series

The reason for this phenomenon can be found in the different drying times. Figure 33 shows the relative humidity in the desiccators and in the PE containers with perforated lids. The time to reach the desired relative humidity of 53% in the desiccators after putting the specimens into them is about 2 hours. In PE containers it takes about 18 hours to reach the ambient humidity of the laboratory. This time corresponds to the time for which the hydrophobicity transfer is delayed in the PE containers [33].

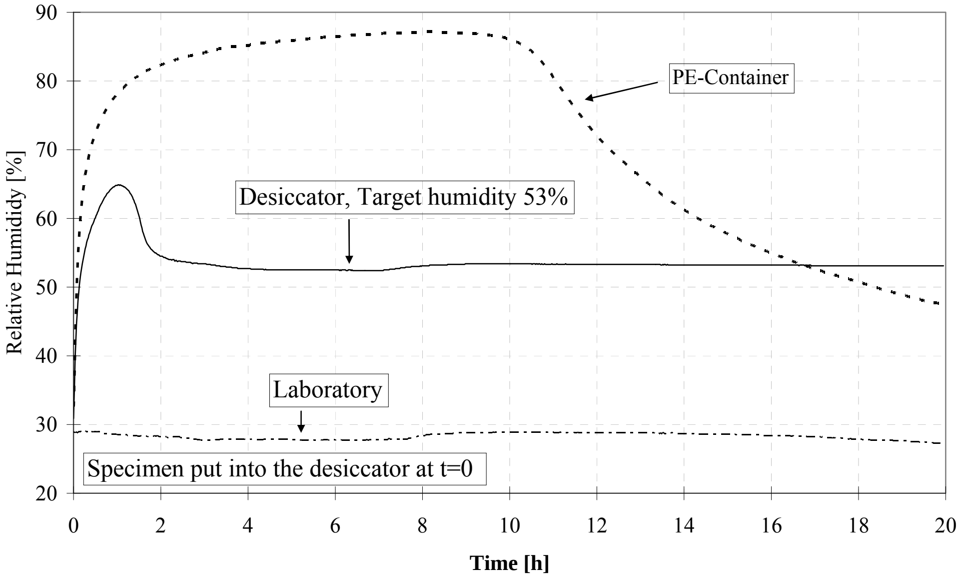

Figure 33: Relative humidity for the storage of 5 polluted RTV-specimens each in desiccators (target humidity 53%) and PE containers

In order to quantify the influence of the different but defined humidity on the hydrophobicity transfer several test series at 33%, 53% and 75% RH. were performed. The transfers at 33% and 53% RH. do not differ significantly while at 75% RH. the transfer is significantly delayed (Figure 34) [33].

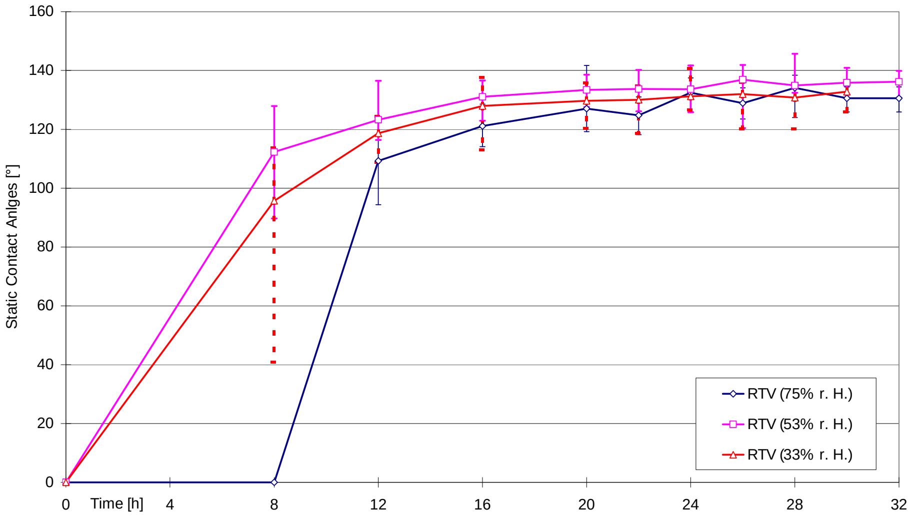

Figure 34: Hydrophobicity transfer at 33%, 53% and 75% RH., material RTV

An explanation for the difference in hydrophobicity transfer can be found in the different time needed to reach the target humidity in the desiccator after 5 specimens were put into them right after application of the slurry (Figure 35). Within about 2 hours the relative humidity in the desiccators reaches the target values of 33% and 53%, respectively. In the desiccator with a target value of the relative humidity of 75% it takes about 4 hours [33]. During this time the pollution layer has still a high water content which slows down the hydrophobicity transfer [8].

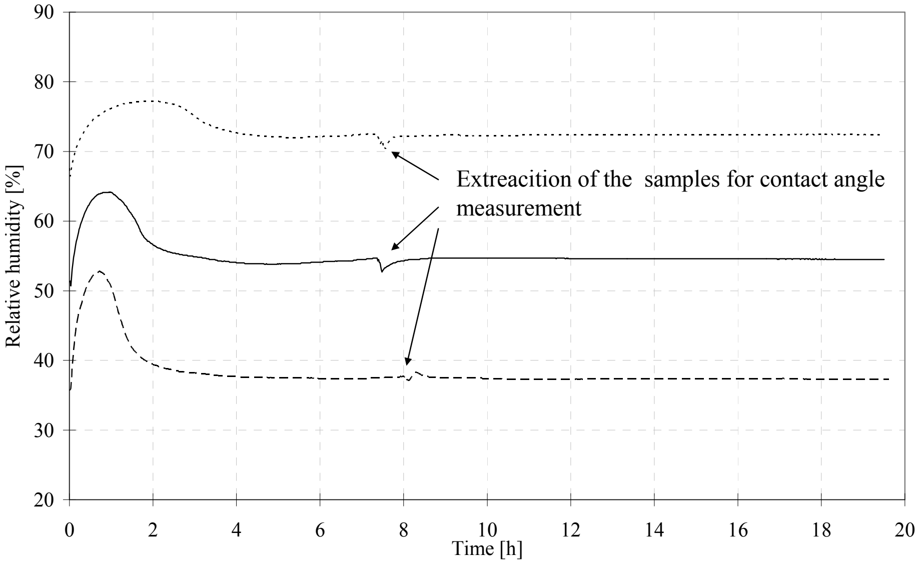

Figure 35: Relative humidity in desiccators with 5 specimen and different target humidities

4.4 Results of Round Robin Testing

In order to check the reproducibility of the test with respect to evaluate a materials ability to transfer hydrophobicity on artificial pollution layers a round robin test has been performed.

All tests within the final Round Robin Testing were carried out according to the test procedure as described in Appendix B.

Laboratories and responsible persons participating in the round robin testing:

- Prof. Dr. Josef Kindersberger, (Project Coordinator)
  Technische Universität München (TUM)
  Dept. of Electrical Engineering and Information Technology
  Theresienstrasse 90, 80333 München, GERMANY
- Prof. Dr. Roland Bärsch,
  University of Applied Science Zittau/Görlitz (HTWS)
  Theodor-Körner-Allee 16, 02763 Zittau, GERMANY
- Dr. Xavier Kornmann, ABB Schweiz AG
  Corporate Research, CHCRC.V2
  Im Segelhof 1, CH-5405 Baden, SWITZERLAND
- Prof. Dr. Masayuki Nagao
  Dept. of Electrical and Electronic Engineering
  Toyohashi University of Technology (TUT)
  Tempaku, Toyohashi, 441-8580 JAPAN
- Dr. Hiroya Homma
  Electric Power Engineering Research Laboratory
  Central Research Institute of Electric Power Industry (CRIEPI)
  2-6-1 Nagasaka, Yokosuka, Kanagawa, 240-0196 JAPAN
- Prof. Dr. Seisuke Nishimura
  Dept. of Electrical and Electronic Engineering,
  Graduate School of Engineering,
  Yokohama National University.
  79-5 Tokiwadai, Hodogaya-ku, Yokohama, 240-8501, JAPAN
- Prof. Dr. Masahisa Otsubo
  Dept. of Electrical and Electronic Engineering, Faculty of Engineering,
  University of Miyazaki
  1-1 Gakuenkinbanadai-Nishi, Miyazaki, 889-192, JAPAN
- Mr. Itsuki Umeda
  NGK High Voltage Laboratory (Polymer Engineering), Power Business Group
  NGK Insulators, LTD.
  1 Maegata-cho, Handa, 475-0825, JAPAN

The investigated insulting material was a HTV-Silicone rubber. The results from the different laboratories are shown in Figure 36 to Figure 46.

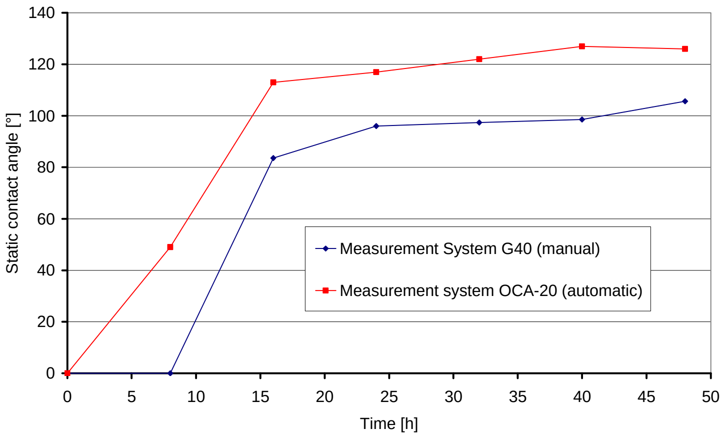

Figure 36: Transfer of hydrophobicity - results from Zittau; comparison manual and automatic contact angle measurement

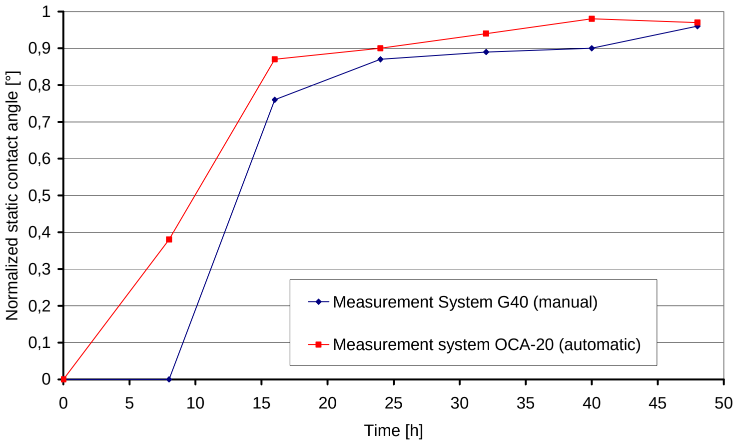

Figure 37: Transfer of hydrophobicity – results from Zittau – normalized values; manual and automatic contact angle measurement

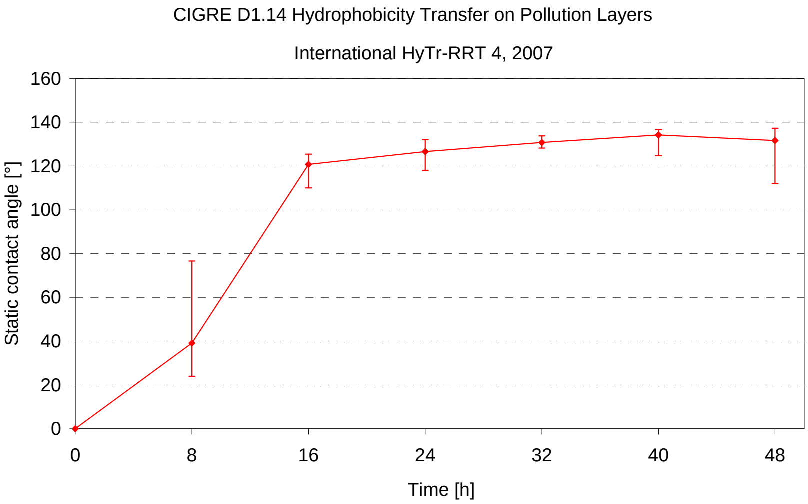

Figure 38: Transfer of hydrophobicity – results from TUM

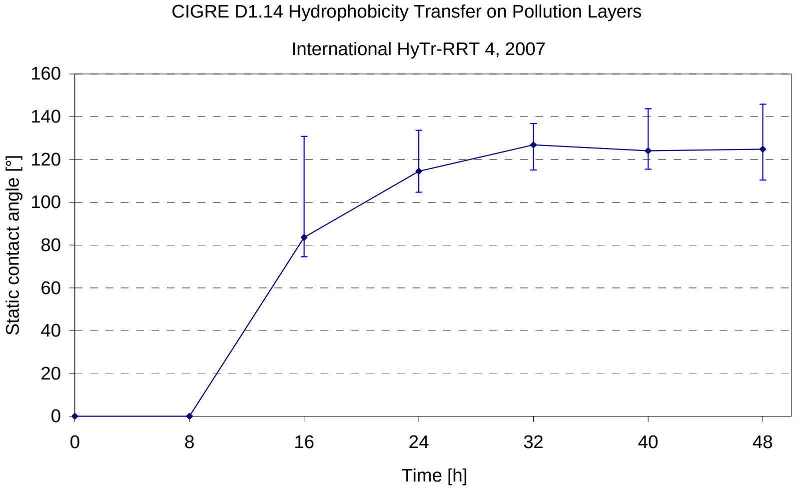

Figure 39: Transfer of hydrophobicity – results from ABB

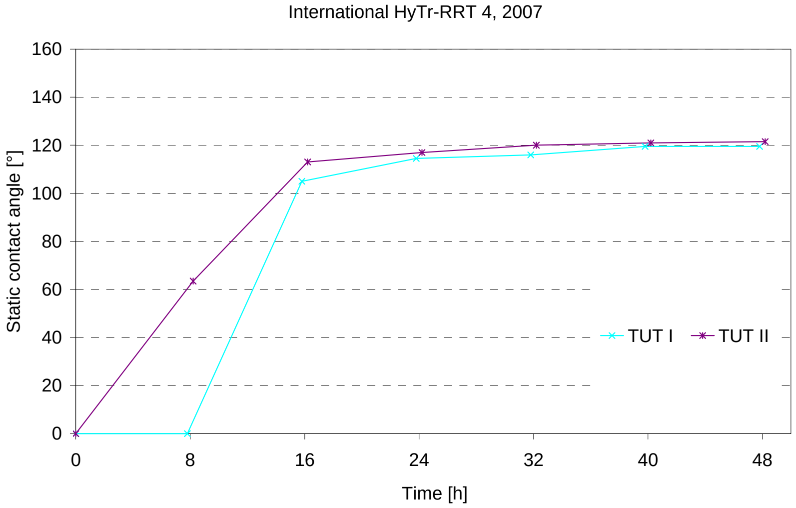

Figure 40: Transfer of Hydrophobicity – results from TUT

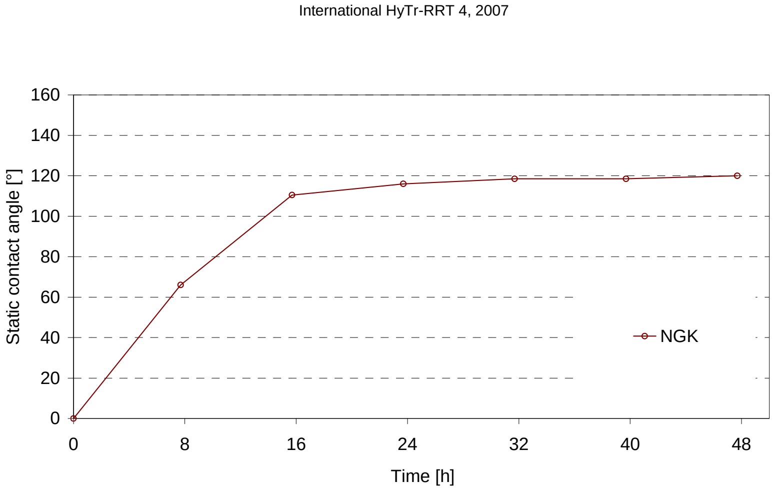

Figure 41: Transfer of hydrophobicity – results from NGK

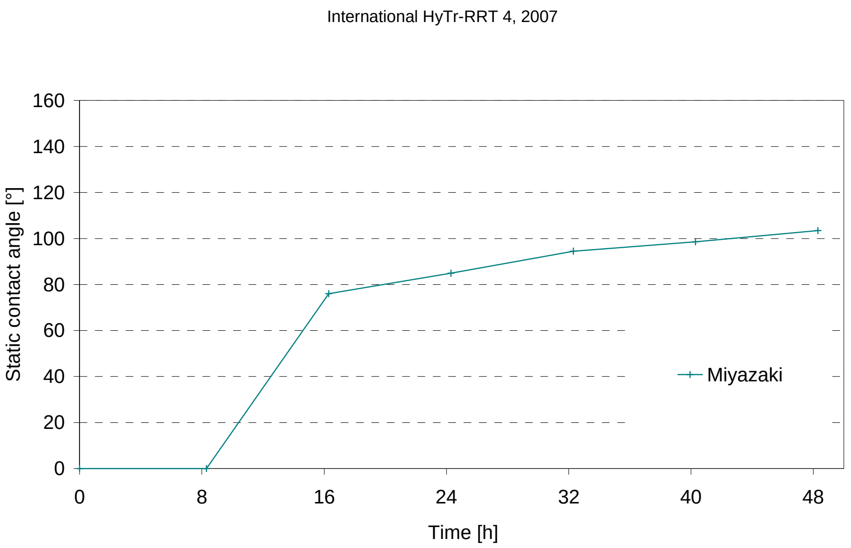

Figure 42: Transfer of hydrophobicity – results from Miyazaki

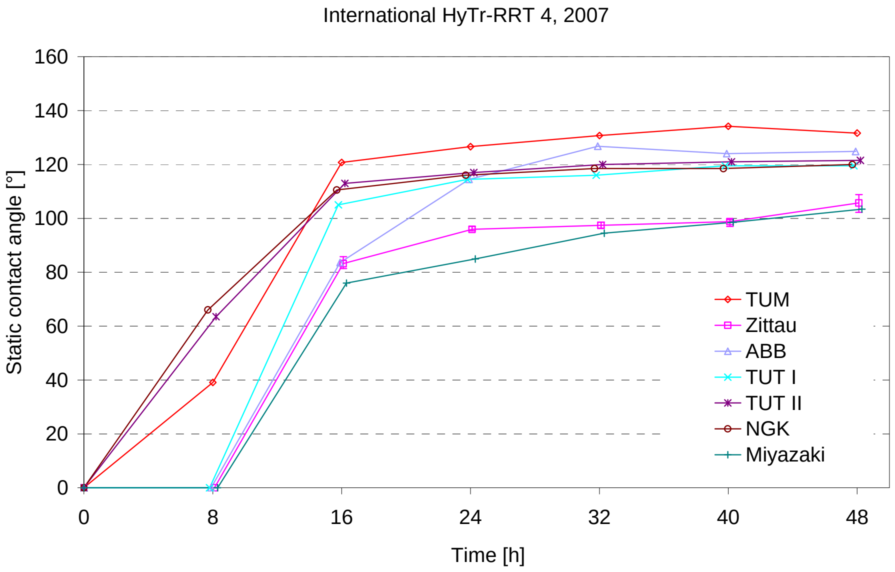

Figure 43: Results from all participating laboratories – absolute values

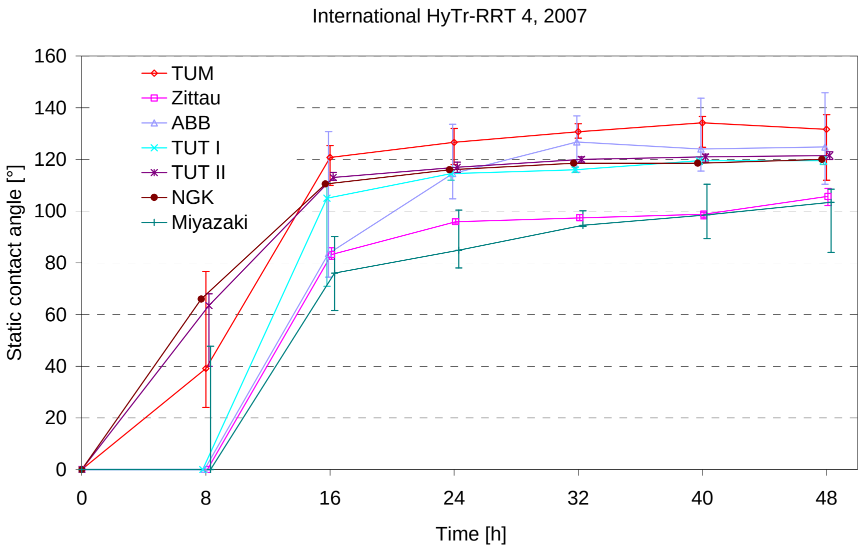

Figure 44: Results from all participating laboratories – absolute values including spread

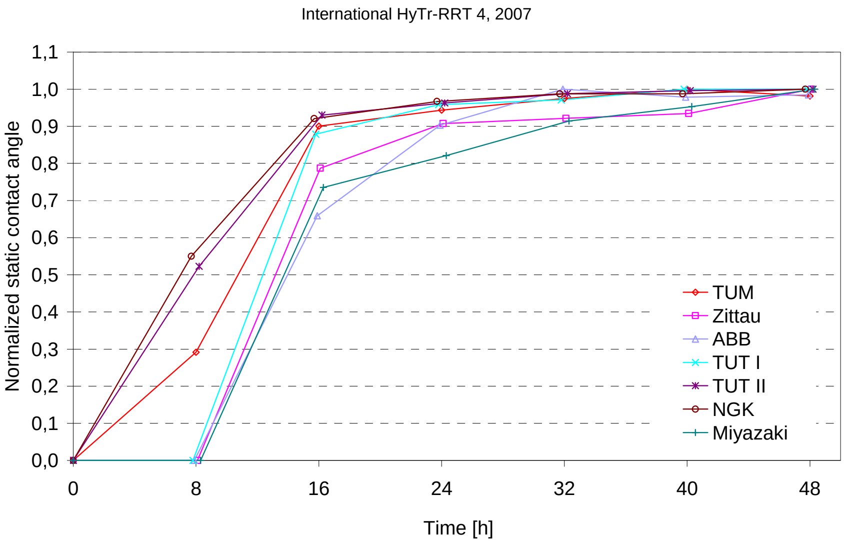

Figure 45: Results from all participating laboratories –values normalized to the maximum value

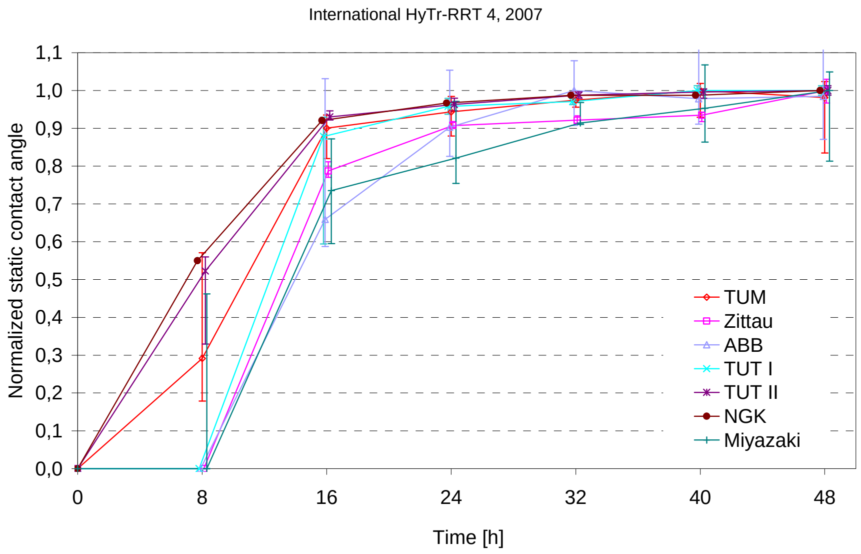

Figure 46: Results from all participating laboratories – normalized to the maximum value including spread

4.5 Conclusion of Round Robin Testing with Hydrophobicity Transfer Test

The results of the 4th Round Robin Test to evaluate a materials ability to transfer hydrophobicity on artificial pollution layers confirmed that the applied test procedure (for details see Appendix B) leads to reproducible results.

During several Round Robin Tests it turned out that a major factor of influence is the storage conditions of the samples after applying the pollution layer in form of a slurry. Storage of the specimens at controlled climate conditions, i. e. 53% RH. in desiccators over aqueous solutions is recommended.

As a side effect it appears that the absolute static contact angle seems to depend on the type of measuring equipment. There may be a tendency that manual measurement leads to smaller static contact angles. As a consequence the kind of measurement shall be noted in the test report.

No minimum requirements and/or classification of material have been introduced because it was felt that more experience is needed with the application of these tests.

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Appendix A:

Evaluation of the retention of hydrophobicity of polymeric insulating materials using the Dynamic Drop Test

The following test is designed to evaluate the retention of hydrophobicity of polymeric insulating materials for outdoor insulation at AC voltages greater than 1 kV

1 Introduction, scope and object

Hydrophobicity of polymeric insulating materials is a surface property which suppresses critical leakage currents which may occur when an electrolytic pollution layer is stressed by high voltage as given e.g. for polymeric insulators in outdoor application. When hydrophobicity has a sufficient retention pollution related flashovers or deterioration of insulating material like tracking or digging erosion may be avoided. The retention of hydrophobicity depends mainly on the insulating material (kind of polymer, kind and concentration of functional fillers and additives), the surface structure (e.g. roughness), the electrical stress and the kind and property of stress by pollution layer.

The aim of the test is to evaluate the retention of hydrophobicity of solid polymeric insulating materials under accelerated and calibrated test conditions so that different insulating materials can be easily compared with respect to this property.
According to the state of the art two different criteria for stability can be regarded:

Criterion for retention of hydrophobicity:
Time until the development of at least 1 continuously wetted path between the electrodes (partial loss of hydrophobicity). The formation of continuously wetted path can be determined by measuring the leakage current. The insulating materials can be evaluated according to

- Criterion A1: Time to formation of a continuously wetted path or
- Criterion A2: Test voltage which does not lead to the formation of a continuously wetted path within 6 h. (withstand voltage).

According to the above mentioned evaluation criteria the insulating materials may be classified in so-called hydrophobicity-stability-classes (HS-classes, e.g. HS-A1-XXXs or HS-A2-XXkV, respectively).

From practical considerations, an evaluation according to criterion A1 is more suitable.

Recovery test:
Certain insulating materials (e.g. silicone elastomers) show a recovery of hydrophobicity after a sufficient period without electrical and pollution stress (recovery of hydrophobicity). Insulating materials may be evaluated by performing a recovery test.

Insulating materials may later be classified in so-called hydrophobicity recovery classes.

The following description of the dynamic drop test refers to the evaluation of the stability of hydrophobicity according to **criterion A1**.

2 Test samples and test set-up

2.1 Samples and sample preparation

Insulating materials in plates of 120 mm x 50 mm (length x width) are used as samples. The preferred thickness of the sample is 6 mm. Other dimensions of the sample size shall be recorded in the test report. Preferably, 8 samples are to be used for every test voltage.
The samples shall be produced with tools which allow producing a defined and reproducible surface structure. The roughness Rz (mean value of roughness) of the samples in the relevant area of testing shall be Rz = 9 µm. Deviations shall be recorded in the test report.

2.2 Sample preparation:

- The samples shall be cleaned with isopropanol and distilled water and using a clean cotton cloth (not fibrous).
- Before start of the test the samples shall be stored under room conditions, i.e. (23 ± 2)°C, 40 to 60% relative humidity, for a minimum duration of 24 h.
- The sample surface which shall be tested shall not be touched or damaged, e.g. during mounting.

2.3 Electrodes

- The dimensions of the high voltage and the earth electrode are shown in Figure 47 and Figure 48.
- The electrodes shall be made of stainless steel and shall have a polished surface. The roughness of the polished electrodes shall be approximately Rz = 5 µm.
  Note: The use of a chlorine containing solution requires certain stainless steels containing molybdenum (e.g. W1.4401 [EN 10088]). The electrodes may be passivated to remove impurities from the surface (see Appendix A1).
- Before each test the electrodes shall be cleaned with isopropanol and a smooth and clean cotton cloth. Corrosion spots shall be removed with a mild abrasive method

2.4 Test set-up

The test set-up shall consist of the following components:

- The electrodes and samples shall be mounted on the inclined back plate so that the electrode distance across the sample is 50 mm.
- A plate of insulating material (sample) which is inclined by 60° against the horizontal axis. The electrodes shall be made from stainless steel and shall be mounted onto the sample. The electrodes have to be mounted in a way that there is neither a gap between the electrodes and the samples nor an indention of the electrodes into the samples due to too much force. The electrodes shall be connected to the high voltage from behind in order to avoid field distortion. No visible discharges shall be present in the vicinity of the samples.
- A container for the electrolyte, a pump with adjustable flow rate and tube (feeder) to the high voltage electrodes as well as drainage for the electrolyte from the grounded electrodes.
- Device to monitor the running off of drops (drop frequency) installed at least at one sample.
- Adjustable high voltage source (50 Hz/60 Hz AC voltage) with including measuring device and series impedance (series resistance) in the connecting line to the samples.
- Device to monitor the leakage current of each sample with over current trip out.

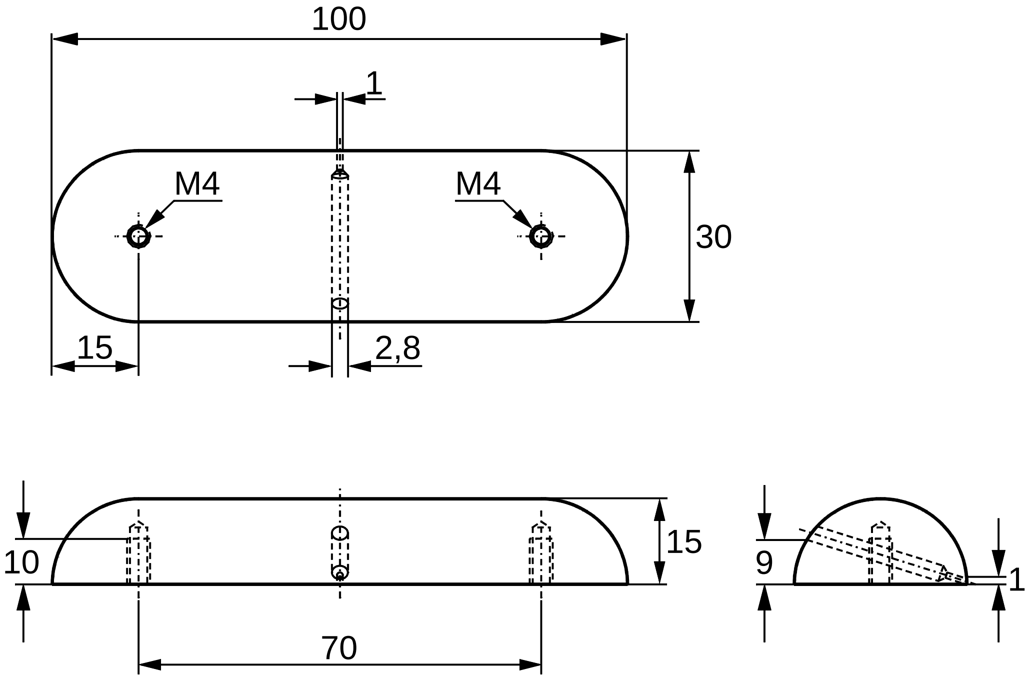


Figure 47: High voltage electrode (made from stainless steel)

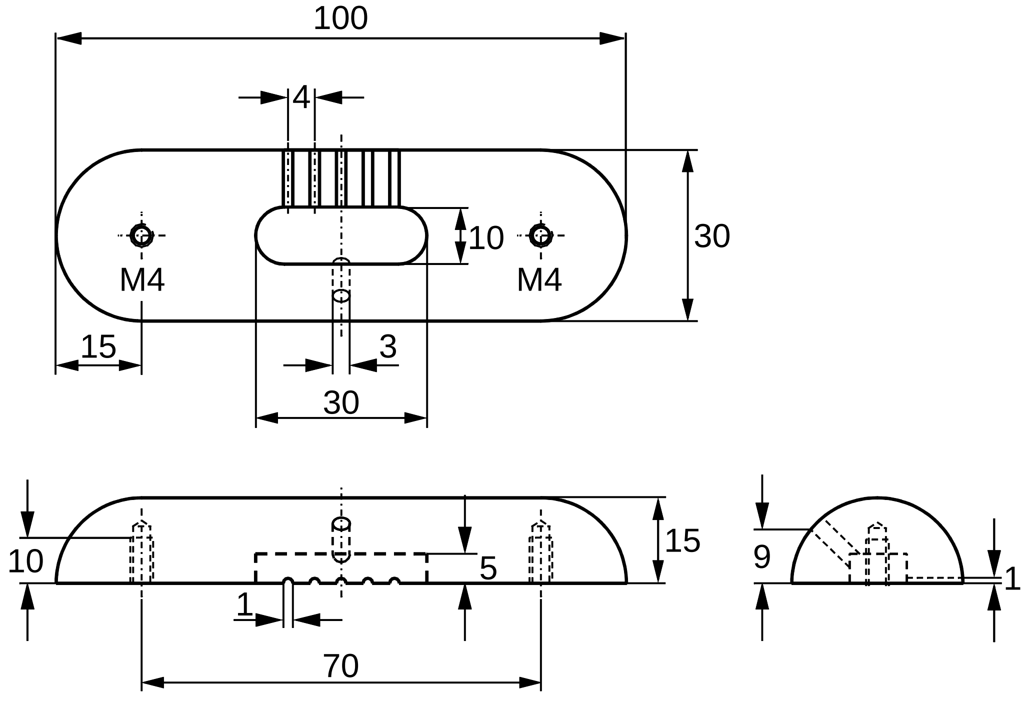


Figure 48: Earth electrode (made from stainless steel)

3 Test procedure

3.1 Test voltage

The preferred AC test voltage is 4 kV ± 0,1 kV (U_{rms} or $\hat{U}/\sqrt{2}$) for the evaluation according to criterion A1.

To gather information tests with different voltages, e.g. 4 kV ± 0,1 kV and 5 kV ± 0,1 kV may be performed.

The voltage source shall have sufficient stiffness. At a total current of 100 mA the voltage shall be not drop more than 5%.

3.2 Test liquid

The contaminant consists of deionised or distilled water with NaCl. The contaminant shall have an electrical conductivity of 1.5 mS/cm ± 0.2 mS/cm at (23 ± 1)°C. The temperature of the test liquid shall be (23 ± 3)°C. Caution shall be paid to possible biological contamination of the electrolyte. During extended periods of testing the electrolyte shall not be used for more than 1 week. At least once a week the container and the tubes shall be cleaned and disinfected with isopropanol.

To ensure a high degree of reproducibility it is recommended to use new tubes for each sample.

Note: It is known that in peristaltic pumps even at constant pump speed the flow rate decreases with time due to aging of the tube.

3.3 Flow rate and calibration the drop frequency

- The flow rate per sample shall be continuous and adjustable in the range from at least 1,0 ± 0,2 ml/minute. A peristaltic pump is recommended.
- After the samples have been mounted the drop frequency shall be adjusted without voltage application to 12±1 drops per minute. The drop frequency achieved after adjustment, but before switching on the voltage, shall be noted in the test report for every sample. Attention has to be paid to the proper flow of the electrolyte through the channels of the earth electrode.

Note: If the flow rate deviates from 1.0 ± 0.2 ml/minute at the desired drop frequency of 12±1 drops per minute the test setup (tubes etc.) has to be checked.

The adjustment period is 5 minutes. The test period shall be started immediately after the 5 minute adjustment period. The time when the test voltage is switched on is considered the start of test, i.e. then the testing time starts to count. After the start of the test the drop frequency shall be measured again during a period of at least 2 minutes and noted in the report.

Note 1: The drop frequency may change during the total period of a test. Despite this change the flow rate shall be kept constant.

Note 2: The roll off of the drops during the adjustment period may have an influence on the test duration. To avoid any adverse effect on reproducibility the adjustment period has been fixed to 5 minutes.

Note 3: At a given flow rate the drop frequency is influenced by the electric field. Usually the drop frequency increases when the voltage is turned on. Despite this increase the flow rate shall be kept constant according to the adjustment of drop frequency before the voltage is applied.

3.4 Leakage current

At the beginning of the test usually all drops roll off separately and the leakage current is in the range of a few µA (period 1). With increasing time residual small drops may be observed on the insulating surface. The residual drops lead to a discontinuous roll off frequency and the residual drops may partly merge (period 2 and 3). During this period more or less spontaneous peaks of the leakage current in the range up to mA may be monitored. Further, electrical discharges may be visible on the insulating surface. With increasing time a continuously wetted path may develop which leads to continuous leakage current in the range of several mA (period 4, criterion for end of test).

The over current device shall be adjusted to a current trip out level of 2± 0.5 mA (rms) with a delay time of 4± 0.5 s. A sample which reaches the trip out level for more than the specified delay time shall be considered to have failed the test.

If the high voltage circuit is not switched off automatically, it has to be switched off manually in order to avoid stress by partial arcs which may influence the evaluation of the recovery of hydrophobicity.

If the sample shall be used for a subsequent recovery test the voltage shall be shut off and the liquid flow shall be stopped after the sample has failed.

Note 1: Stress by partial arc and permanent humidity may influence the recovery of hydrophobicity.

Note 2: For insulating materials others than silicone rubbers shorter delay times of the over current device may be applicable.

3.5 Test procedure

The test shall be executed in the following sequence:

a) Preparation and conditioning of sample and electrodes
b) Mounting of the samples in the test arrangement
c) Switching on the pump
d) Adjustment of the drop frequency (5 min)
e) Switching on the test voltage and switching on the leakage current measuring instrument (over current delay relay)
f) End of the test when over current is reached

For the recovery test samples shall be used which have already failed during the original dynamic drop test. The recovery test shall be made 24 hours after the original test. During the rest time the samples shall be stored in a dust free environment. The samples shall not be touched or bent. To make sure that the property of hydrophobicity recovery is tested the stress in the recovery test shall be at the same area as during the original test. That means the path where the drop rolls off shall be the same during the recovery test and the original test.

If a recovery test is required the test procedure shall start with b).

4 Test report

The test report shall include the following information:

- Type and designation of material under test
- Details of the samples, i.e. method of production; dimensions; surface structure (roughness); cleaning procedure; pre-conditioning
- Orientation of samples with respect to the electrodes (e.g. tool direction)
- Time until the failure of the samples with the specified test voltage (criterion A1)
- Surface roughness of the electrodes
- The rest time in case of recovery test

Appendix A1:

Guidance for treatment of test electrodes processed from stainless steel.

General:
Stainless steel is sensitive with regard to rust impurity caused by surface metal contamination in contact with different metals and alloys. Therefore the following guide lines are brought to attention in order to minimize possible surface contamination.
These guide lines are recommendations only and provide instructions in case test electrodes present surfaces rust and residues and help to check possible root cause of surface contaminations and their prevention.

- **Machine processing**
  All cutting, milling and sawing tools have to be determined for stainless steel alloys only. Other tools would contaminate the surface with residues leading to corrosion later.

- **Polishing**
  Polishing machines and their devices need to be reserved to stainless steel applications only

- **Scouring**
  Despite of the former carefully observed machine process using the correct tools it is recommended to eliminate any possible contamination deposits by scoring the stainless metal working parts. For this process several chemical materials are available e.g. Antox 71E or Antox 3d (Oakite Europe GmbH). The chemistry consists of a paste, which is wiped onto the surface of the working part and left there for 60 to 120 minutes. Afterwards the working parts should be neutralised by chemistry like Antox NP.

- **Inertness**
  All the processes clean the surface of the working parts thoroughly from contamination and residues created during the machine processing.
  Working parts later exposed to chemical fluids might show again surface stains and rusty areas. An inerting process helps preventing this result.
  Stainless steel surfaces become inert against chemical attack, when the surface will be enriched with oxygen (passivation). E. g. heavy oxidizing acids like 20% nitric acid will migrate oxygen into the working part surface and seal the working part against further chemical attack. In the industry the chemical - e.g. Antox 90E (Oakite Europe GmbH) – provides an inert surface of stainless steel working parts.
  The chemicals referenced here are trade names and might be available under different descriptions and from other traders.

- **Attention:**
  This process and handling are very critical with respect to safety & health and environment control regulations during the working process. Therefore special care has to be taken in operation with these chemicals. Working instructions and advises in the safety data sheets are mandatory.
  Instructions and safety data sheets are available on the internet.

- **Remaining advises**
  During the installation of the working parts into the applications skin contact should be avoided. Sweat and grease could affect the surface again. It is recommended to use latex gloves.
  Further the installation components like bolts, washers, springs and nuts should made from the same alloy quality as the working parts.

Appendix B:

Test for the evaluation of hydrophobicity transfer property of polymeric insulating materials

1 Guidance on measurement of contact angles (acc. to IEC 62073)

The measurement of the static contact angle θ_s shall be performed by applying a water droplet of a certain volume on the horizontal surface of a test specimen by using a pipette or a syringe with a scale. The volume of the drops shall be approximately 5 µl.
If needed, the measurement of the dynamic contact angles is proposed to be performed on a horizontal plane. By adding water to the droplet the advancing angle θ_a can be measured. After that the receding contact angle θ_r can be measured by withdrawing water from the droplet.
It is recommended to keep the capillary pipette of the syringe immersed in the droplet during the entire measurement in order to avoid vibrations and distortions of the droplet that otherwise may affect the result.

2 Test Procedure to evaluate the hydrophobicity transfer property

2.1 Specimens

a) Bulk material

The specimens are plain plates with the dimensions (L×W) 50 mm × 50 mm. The thickness is 6 mm. The samples are produced with a foil between material and mould to avoid the use of mould release agent.

b) Coatings

Coatings of 0.5 mm thickness are applied on glass substrates by pouring.

2.2 Test Procedure

Measurement is performed with 10 samples.

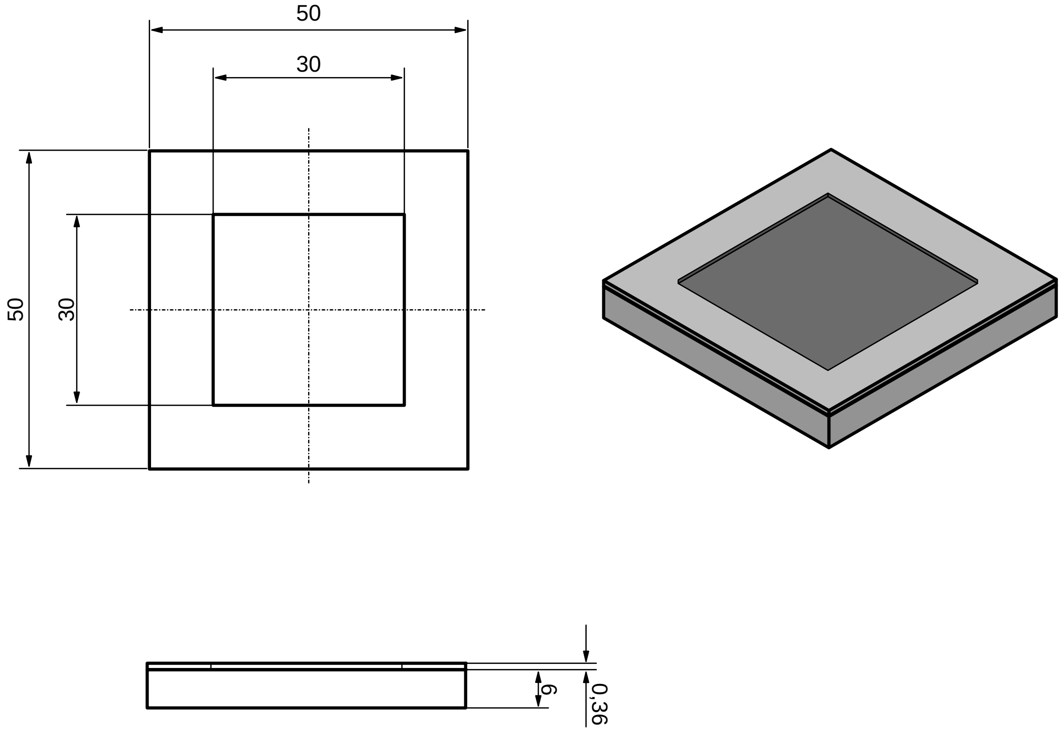
In order to achieve a clean and smooth surface the specimens are first cleaned with isopropyl alcohol and then with deionized water. After that the specimens are dried for at least 24 hours under normal laboratory conditions.

Note: Samples of different material should be stored separately from each other in order to avoid contamination.

The method is testing the intrinsic properties of hydrophobicity and effects of materials on the surface shall be excluded by proper pre-treatment of the materials. For example, if a mould release agent is used to produce the samples, the layer containing the agent must be completely removed for example by mechanical treatment.

The specimens shall be covered with adhesive foil in a matter that a window of the dimensions (L×W) 30 mm × 30 mm is obtained (Figure 49). The thickness of the adhesive foils defines the latter thickness of the pollution layer. A thickness of 0.36 mm shall be used.

Note: If for example the thickness of a single foil is 0.12 mm a thickness of 0.36 mm can be achieved by putting three foils on top of each other.



All dimensions in mm

Figure 49 – Specimen with adhesive foil

Inside the area marked by the window of foil the specimens are then coated by applying a slurry. The slurry is made of 7.5 g silica powder (untreated i.e. not silanised, medium grain size 3 µm) and 3.5 ml of a mixture of water and isopropanol (65 vol.-% water, 35 vol.-% isopropanol) and is homogenized by stirring. A plastic stick is used to wipe off the excess slurry. This results in a smooth and even surface (Figure 50).

Note 1: Slurry stirred for approximately 2 minutes is usually sufficient to avoid clots.

Note 2: As isopropanol tends to evaporate the slurry shall be used shortly after the preparation.

Note 3: The shape and material of the stick may influence the thickness of pollution layer.
To evaluate possible effects it is suggested that details of the stick (i.e. shape and material) are recorded in the test report.

Note 4: After drying of the slurry the thickness of the remaining layer will be less than the thickness of the foils, i.e. 0.36 mm. The results of this test refer to the nominal thickness of the slurry applied to the sample which is the thickness of the foils.

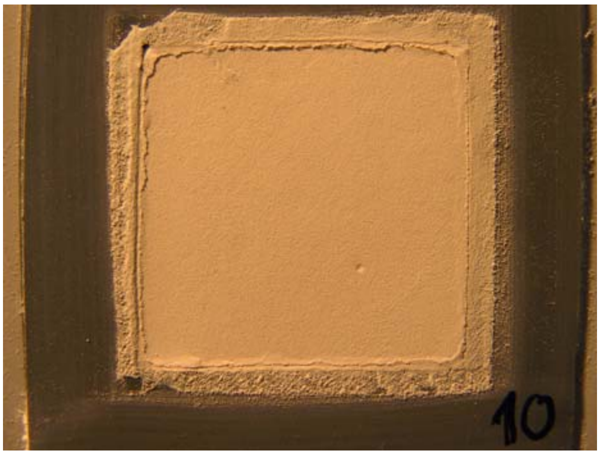


Figure 50 – Specimen with pollution layer

After the application of the slurry the samples shall be stored in desiccators under controlled humidity of 53% relative humidity and at a temperature of (23±2)°C (e.g. by using a saturated solution with magnesium nitrate, Figure 51). The desiccators shall meet the requirements given in ISO 483. The humidity and the temperature in the desiccators shall be recorded. Samples to be tested at different times shall be stored in a separate desiccator. Alternatively each sample can be stored in a separate desiccator. The relative humidity in the desiccators has to be monitored and recorded.

Note 1: The desiccator shall not be overloaded with samples so that the target relative humidity is reached within relatively short time, e.g. within less than about 2 hours.

Note 2: Attention shall be given to the period of time between the application of the slurry on the samples and putting the samples into the desiccator as uncontrolled drying of the applied slurry may occur.

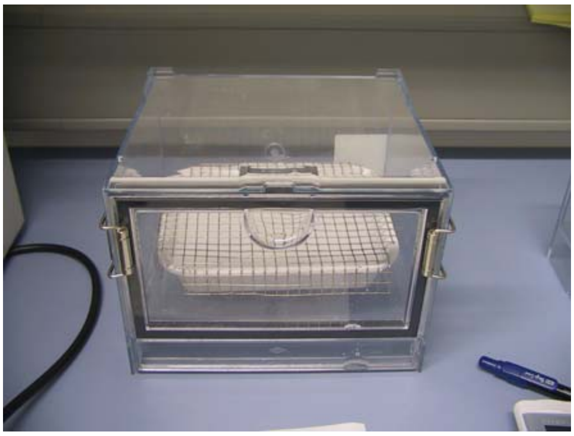


Figure 51 – Desiccator with saline solution

The time when the pollution layer is applied is the start time (0 hours).

The contact angle shall be measured in different time intervals after the pollution layer has been applied.

For more detailed information measurement of the static contact angles of the pollution layer at the following time intervals is suggested: 8, 16, 24, 32, 40, and 48 hours. In order to obtain all of these measurements two test-series with 5 samples each are necessary. Samples of each series can be stored in 1 desiccator. Alternatively the first test-series is started in the morning, the second one in the afternoon. The (5) samples of one test series can be stored in one desiccator. Table 1 gives an overview of the test-series:

Test-Series	Measurement times				
1	0	8	24	32	48
2	0	16	24	40	48

For the duration of the measurements the samples are taken out of the desiccators and put back in afterwards. The measurement of the contact angle shall be finished within 1 minute after the application of the drop to the surface. If a longer time is required the actual time needed for the measurement shall be documented in the test report.

If the drop is sucked in the pollution layer within 3 minutes after its application the contact angle shall be set to zero whatever the measured contact angle is.

For each selected time interval 1 drop has to be applied on the pollution layer of each of 5 samples. The volume of each drop shall be in the range of 5 to10 µl. The drops shall be placed in the centre area of the sample (Figure 52). This may alleviate the effects of an unequal pollution layer thickness. The drops shall be distributed in this centre area i.e. no measurements shall take place on the exact same location on the sample.

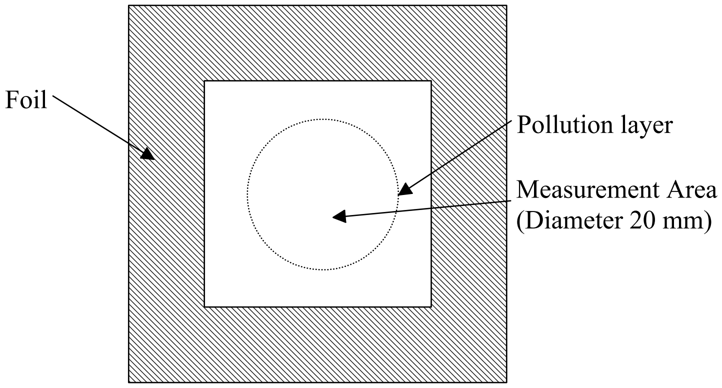


Figure 52 - Area for the drop application

Note 1: By using manual measurement devices one contact angle per drop is usually measured. It is also possible to measure at the same time the contact angles on both sides of each drop, e.g. by using computer aided measurement devices.

Note 2: As the application of drops may cause small deformation of the surface structure the application of drops at the same spot shall be avoided. Therefore, the drops shall be placed randomly within the designated areas.

Note 3: If samples on which drops have been placed are put back to the desiccator the drops can be removed from the surface, to avoid bringing additional humidity into the desiccator.

The median value of the readings is the contact angle which should be taken into account.

Note: If three (3) or more drops out of five (5) are sucked into the layer, the resulting contact angle is 0°.

2.3 Evaluation criteria

The ability of hydrophobicity transfer is evaluated according the time which is needed to transfer hydrophobic properties onto artificial pollution layers under the conditions given by the test.

No classification of material has been introduced yet because it was felt that more experience is needed with the application of these tests.

3 Test Report

The test report shall contain:

- Static contact angles for each drop applied
- Median contact angle for all sample at the times of measurement
- Drop volume
- Temperature in laboratory and in desiccator [°C]
- Relative humidity in laboratory and in desiccator [%]

* * * * *

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