PARTIAL DISCHARGE INCEPTION VOLTAGE AND BREAKDOWN VOLTAGE OF AL_2O_3 NANOCOMPOSITE AFTER THERMAL AGING

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Abstract: Nanotechnology deals with characteristics in nanometer size and/or microscopic regions on materials and functional devices. Nowadays, the nano fillers are used in different epoxy resin systems. The disadvantages of polymeric materials are ageing effects which are not understood in details so far. Penetrating water leads to complex physical and chemical interactions at the microscopic interfaces between filler and polymeric matrix. The results of the interactions at the large internal filler surfaces are an increase in the conductivity and intensification of interfacial polarizations effects. These effects can result a change of the dielectric parameters and a decrease of the electrical strength and life-span. In this study aromatic epoxy resin, and related micro filler are the same for all produced samples. Al_2O_3 was added as nano fillers. Nano fillers were mixed with the resin using ultrasound device and high speed mixer simultaneously. Then the distribution of the nano fillers was checked by means of transmission electron microscopy. The investigations were carried out under homogeneous (rogowski-rogowski) and inhomogeneous (rod-plane) electrical field. The influences of the nano fillers up to 10% by weight on the partial discharge inception voltage and breakdown voltage of the specimens after different aging periods at 130°C (15% above the glass transition temperature) were investigated and the mentioned parameters were measured at room temperature.

1 INTRODUCTION

Epoxy and epoxy based composites are preferred insulating materials for several electrical applications, especially printed circuit boards, bushings, GIS spacers, generator ground-wall insulation system and cast resin transformers. Recently, epoxy based nano dielectric systems are being increasingly investigated for their electrical properties, since the introduction of nano filler demonstrate several advantages in their properties compared with similar properties obtained for epoxy systems without nano fillers [1,2,3].
Nanotechnology is a general term covering a wide range of many fields. It deals with characteristics in nanometer size and/or microscopic regions on materials and functional devices [4]. Epoxy resin often suffers lower dielectric breakdown strength if micro-fillers are loaded. It might be possible to raise the once lowered breakdown strength, if nano fillers are added [5].
With respect to the electrical breakdown characteristics in polymer nanocomposites, several reports are available on different polymeric systems. In epoxy nanocomposites filled with TiO_2 fillers, the ac voltage endurance, short-term dc and ac dielectric strengths and impulse breakdown strengths are found to be higher as compared to micro composites [6,7]. Additionally, in another study on the electrical breakdown characteristics in SiO_2 filled epoxy systems, it is seen that although the insulation breakdown strengths in nano composites are less than that of base epoxy, they can be higher if silane coated SiO_2 nano-fillers are utilized [8]. These interesting observations in the electrical properties of epoxy nano composites are highly encouraging and they are mainly attributed to the unique properties of nano particles and the dynamics at the interfacial region [9,10].
In this study partial discharge measurement was considered as non-destructive and breakdown voltage measurement as destructive test. Then the changes were observed after three ageing periods. Such changes in the dielectric parameters of an insulation can be caused by long-time ageing phenomena or partial destructions due to electrical or thermal stresses. A relationship between these ageing phenomena and the changes in the electrical and dielectric parameters forms the physical fundamental of all dielectric diagnostic tools.
Al_2O_3, TiO_2, ZnO nano fillers are the most famous ones which would be used for researches [11]. This study reports about the influence of nano filler Al_2O_3 at different concentrations of nano filler including 0%, 1%, 3%, 5% and 10% by weight in combination with the conventional micro fillers on the electrical and dielectric properties of bisphenol-A epoxy resin. Using electron microscope observation, it was shown that the area surrounded by dispersed micro-scale silica fillers was also filled with the nano-scale fillers. Nano fillers can fill the spaces between micro fillers and as a result can protect insulation material against partial discharge (PD) generation. Furthermore, the estimate of spacing between the fillers and the filler/epoxy resin interface area shows a more densely packed structure of the filler mixture composite than the conventional filled epoxy resin. The morphological feature of the filler mixture composite improves its

insulation breakdown strength. The epoxy resin is usually filled with a large amount of micro-scale fillers, for example Quartzmehl, Wollastonit, Silica or Alumina. Using Silica or Alumina results the same low thermal expansion as aluminum or copper conductors. The conventional filled epoxy with low thermal expansion avoids exfoliating between the epoxy casting part and the conductor caused by heat-cycle. So, it is noticeable that adding a very low volume of nano fillers to epoxy resin result a better dielectric performance [12].

As partial discharges are very dangerous for solid insulation materials, nano fillers can fill the spaces in the composite and this results a better partial discharge behavior of the insulation and consequently will prolong its life [13, 14]. Nano fillers can also reduce the negative effect of the electrical treeing within the insulation material [15].

The two key parameters partial discharge inception voltage and breakdown voltage were evaluated under homogeneous and inhomogeneous electrical fields.

2 TEST CONDITION

To apply necessary thermal ageing on the specimens, the specimens were aged at 15% above glass transition temperature of the insulation material. It means, the specimens were aged at 130°C for 4000 hours in an oven. The measurements were done in three periods, furthermore, after 1000, 2000 and finally 4000 hours. To let the molecules of the specimens relax after each ageing time, the specimens were taken out from the oven and put in a sealed container at room temperature for 4 weeks.

Although the specimens were produced under vacuum, it is difficult to omit cavities during the production completely. In this condition, nano fillers can operate against PD, when the nano fillers are mixed correctly. So mixing nano fillers with the host material homogeneously is the most important point in order to reach good results.

2.1 Epoxy resin system

Indoor epoxy resin system including CY228 as resin, HY918 as hardener, DY062 as accelerator and 65% Quarz Millisil W12 as micro filler was used for all the samples.

2.2 Nano filler

Unmodified Al_2O_3 was selected as nano filler, because it is the conventional nano fillers and also economic. The specimens were produced considering maximum 10% by weight, because the final costs of the product with more than 10% by weight are uneconomic and the viscosity of the whole material after mixing goes too high and casting would be difficult consequently. The mentioned percentages by weight are valid for the end product and not only for resin or hardener.

The average particle size of the Al_2O_3 is 40 nm. The different sizes of particles influence mixing and casting process. As smaller the size of nano filler, easier is the mixing and casting.

2.3 Electrode configurations

The experiments were done under homogeneous and inhomogeneous electrical field considering two different electrode configurations. To create homogeneous field Rogowski-Rogowski and to create strong inhomogeneous field needle-plate electrode configuration were considered.

To evaluate the electrical strength of the insulation material, it is necessary to do the measurements under both mentioned fields. IEC 60455-2 discusses about this matter and a related standard recommends the determination of the breakdown voltage by brass electrodes with Rogowski profile and 1 mm distance between both electrodes. The used electrodes have a nominal diameter between 24 mm and 27 mm as shown in Figure 1.

Figure 1: Sample with Rogowski and needle electrodes configurations

0.8 mm thick and 40 mm long Tungsten pins with an angle of 30° at the end were used. The needle electrode tip radius was etched electrolytic in an etching. The final needles had a tip radius of 5 μm. 2 mm distance between needle-plate electrodes was considered. Figure 2 shows the needle before and after etching.

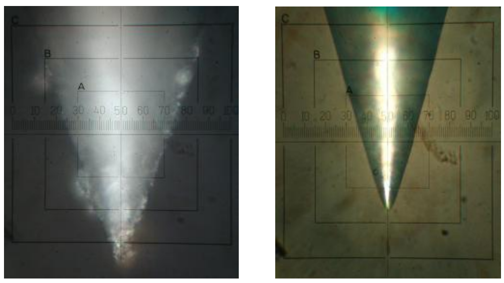


Figure 2: Needle electrode before (left) and after etching (right)

2.4 Mixing process

The nano fillers were firstly mixed with resin as the base material. After all the nano fillers were poured in the resin, both materials were mixed for 15 minutes by high speed mixer and ultra device simultaneously to make the most optimal mixing. Principally, nano fillers should be distributed homogeneously and in nano scale. Otherwise they play the role of micro fillers and will not let evaluate their influence on the based material. To prevent unwanted heating of the material, the temperature of the material was kept approximately constant by using a water cooling system.

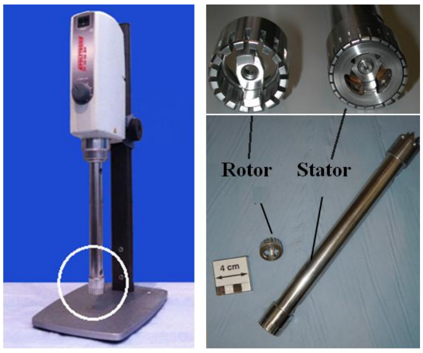
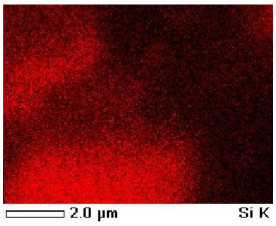
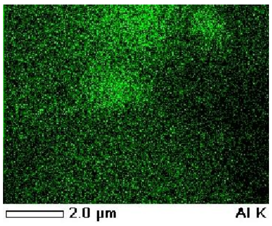


Figure 3: Mechanical mixer for dispersing nano fillers in liquid

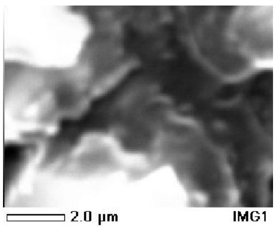
The above mentioned mixing method was used up to 5% by weight. For 10% by weight it was not possible to mix all the nano fillers with resin. So 60% of the nano fillers were mixed with resin and the rest with hardener, instead.

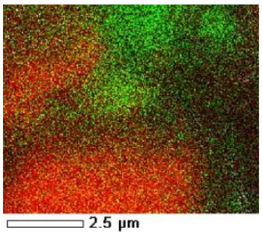


View of micro filler 65% W12

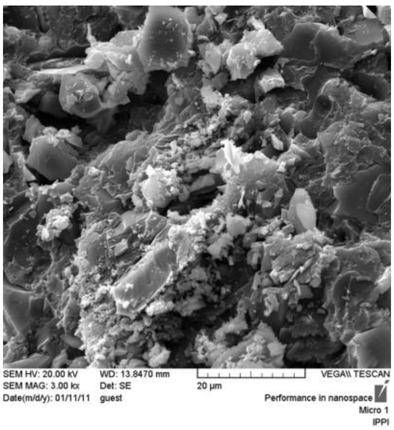


View of nano filler 5% Al_2O_3





Overlay view of 65% W12 and 5% Al_2O_3



Zoom out of the total view of 5% Al_2O_3 and 65% W12

Figure 4: TEM photos of a sample with 5% Al2O3 as nano filler and 65% W12 as micro filler

It is much better to mix the nano fillers with resin instead of hardener, because the hardener is very sensitive to humidity. So mixing nano fillers with hardener would influence the electrical and dielectric parameters of the test samples.
The materials were mixed with hardener, micro fillers and accelerator under vacuum and also the temperature was controlled in several steps for 45 minutes. Finally the mixed material was casted into the moulds with needle and Rogowski electrodes and cured for 4 hours at 80°C and 8 hours at 140°C in an oven. To reduce the internal mechanical forces, the moulds were cooled down for 6-8 hours.
To let the molecules of the samples relax sufficiently, the measurements were done 4 weeks after the curing process.
To check if the nano fillers were distributed homogeneously and in nano scale, it was validated by means of transmission electron microscopy.
The micro and nano fillers separately and also a combination of them in the epoxy resin are shown in Figure 4, so that the green points show the distribution of the Titanium atoms (TiO_2 nano fillers) and the red areas are the distribution of the Aluminium atoms (Al_2O_3 nano fillers). Using TEM technique, a separation of nano and micro fillers is possible.

3 SETUP FOR MEASUREMENTS

3.1 PD inception voltage measurement

To do PD Measurements, the PD measuring system of the company ICM Diagnostix Power Systems GmbH was used. Before each PD measurement, the system was calibrated using CAL1A calibrator with a charge of 10 pC. Then the AC voltage with the increasing rate of 0.5 kV/s was applied. While the applied voltage was increasing, the PD magnitude was monitored and as soon as a change in PD magnitude happened, this applied voltage was assumed as the PD inception voltage. PD patterns were also saved at 25% above of the PD inception voltage after 60 seconds. Figure 5 illustrates the PD measurement setup.

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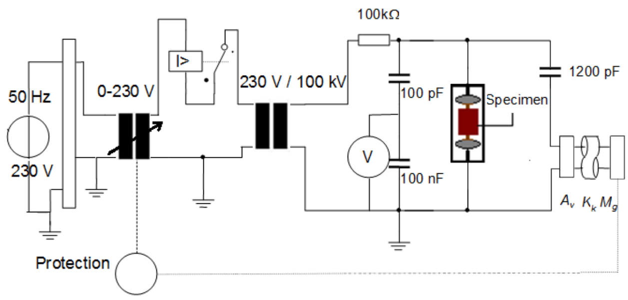

Figure 5: Schematic test setup for PD measurement

A_v: Quadrupole connection

K_k: Cable

M_g: Measuring system

3.2 Breakdown voltage measurement

In order to assess the breakdown voltage, the specimens were left for 5 minutes after each PD measurement. Then the applied voltage was increased 2kV/s up to breakdown.
Both PD inception voltage and breakdown voltage tests were done under insulation oil to avoid flashover on the surface of the test samples.

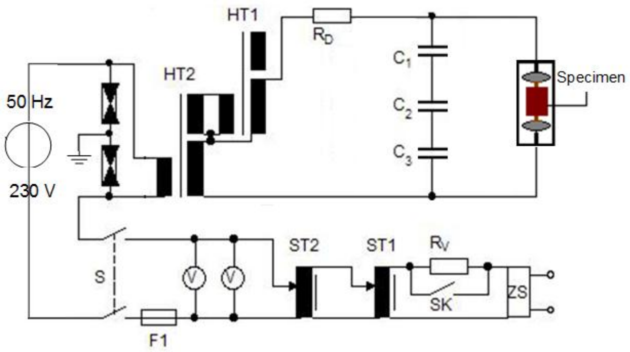

Figure 6: Schematic test setup for breakdown voltage measurement

4 MEASUREMENT RESULTS

4.1 PD inception voltage under homogeneous field

The measurements of PD inception voltage under homogeneous filed were limited up to 30 kV, because the samples were used for the determination of breakdown voltage too. As shown in Figure 7, the PD inception voltage of the samples at 0% and 1% Al_2O_3 nano filler concentration is less than the other curves. This improvement is clear up to 4000 hour aging time for the specimens with 3%, 5% and 10% nano filler. The reason can be the reduction of the cavities du the expansion of the material during aging and becoming the cavities smaller.

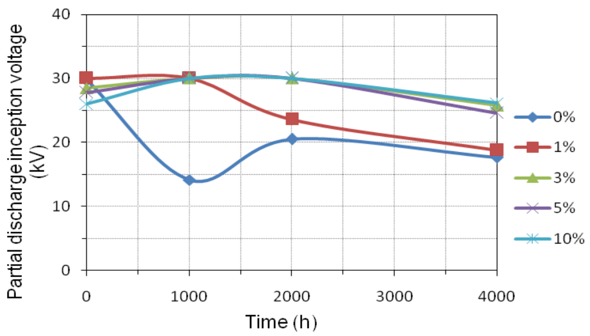

Figure 7: PD inception voltage of the specimens with Al_2O_3 under homogeneous field

At higher concentrations of nano filler, the nano fillers can be moved to the cavities and spaces during aging, especially that the aging temperature was considered 15% above T_g. In this condition, although the polymer molecular chains are trapped by the nano fillers and the material is soft approximately, the nano fillers can find their optimal place for a residence in the matrix. Another reason is better adhesion force between the electrodes and the epoxy resin, when nano fillers are used.
In general, interphases can be created using micro fillers. But considering the same volume fraction of the fillers, nano fillers have much more interphases than micro fillers as shown in Figure 8.

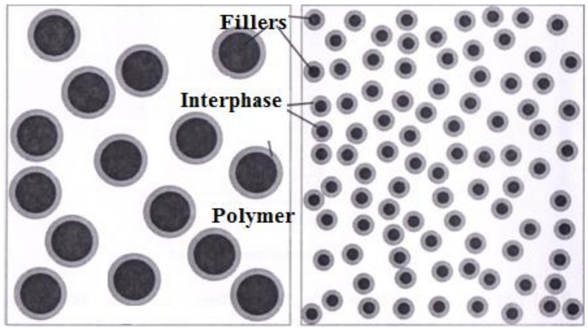

Figure 8: Inerphases of micro fillers (left) and inerphases of nano fillers (right)

4.2 PD inception voltage under inhomogeneous field

Under inhomogeneous field the results introduced in Figure 9 show that the unaged specimens have the same PD inception approximately.

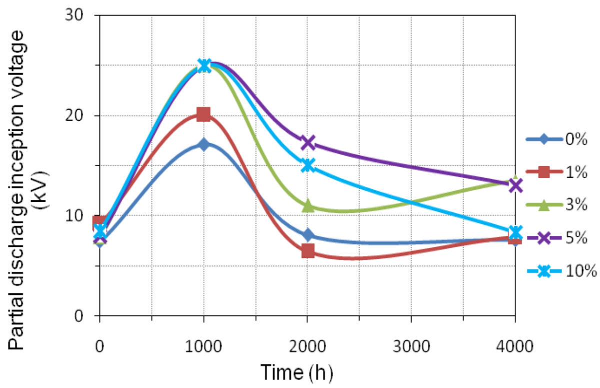

Figure 9: PD inception voltage of the specimens with Al_2O_3 under inhomogeneous field

The PD inception voltages of the curves of 3%, 5% and 10% within 1000 hours are higher than the curves of 0% and 1%. It can be also said that, the curve of 1% shows a relative increase. According to the curves, up to 1000 hours, the PD inception voltage was increased. This increase can be due to the reduction of the cavities or their sizes. On the other hand, up to 1000 hours the specimens were being post cured and dried. But after 1000 hours, the aging starts to affect the specimens stronger. Anyhow, after 1000 hours up to 4000 hours, the PD inception voltages of the specimens with nano fillers are not less than the values of the unaged specimens. As mentioned in section 4.1, the polymer molecular chains are trapped by nano fillers and there is much better interconnection

among the polymer molecules considering nano filler. This interconnection tries to keep the nano composite in better condition than composite.
Comparing the Figurer , it can be found that the optimal concentration of nano filler for this epoxy resin system is about 5% by weight.

4.3 Breakdown Voltage under Homogeneous and inhomogeneous Fields

Figure 10 shows that the unaged specimens with 1%, 3% and 10% Al_2O_3 nano filler have better electrical strengths under homogenous field. In general, it seems that the curve of 10% has better condition up to 4000 hours, so that after 4000 hours aging time, the breakdown voltage is about 25% greater than its value before aging.
Under inhomogeneous field, the curve of 3% in Figure 11 has higher values than the others.
The Thermo gravimetrical analysis (TGA) shows no difference between the residue weight of the different specimens at lower temperatures as shown in Figure 12.

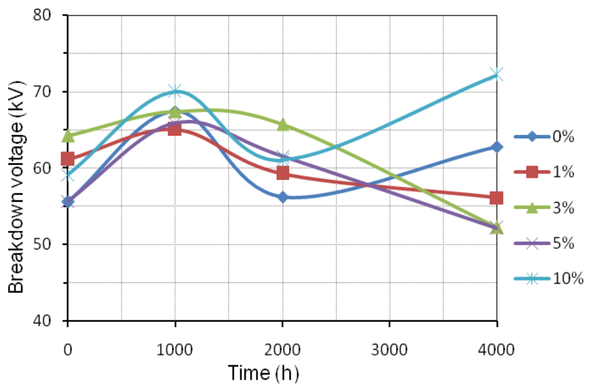

Figure 10: Breakdown voltage of the specimens with Al_2O_3 under homogeneous field

The breakdown voltage under homogeneous field and at 10% concentration was held up to 4000 hours and under inhomogeneous field at 3% concentration in a good condition.
Nano fillers did not reduce the PD inception voltage and breakdown voltage of the epoxy resin.
The optimal concentration of nano filler can be around 5%.

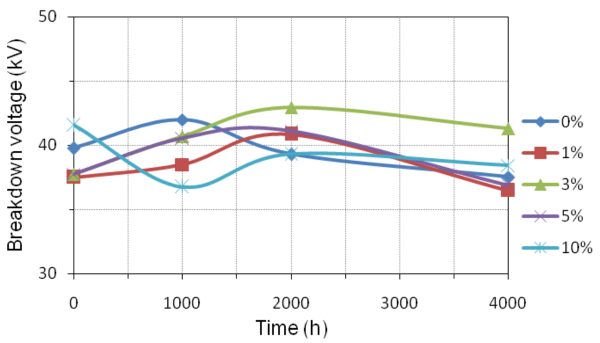

Figure 11: Breakdown voltage of the specimens with Al_2O_3 under inhomogeneous field

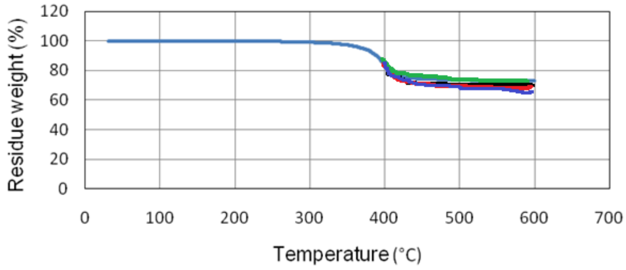

Figure 12: Thermo gravimetrical analysis (TGA)

5 CONCLUSION

The PD inception voltage can be held in normal situation up to at least 4000 hours as good as at room temperature under homogeneous field using 3%, 5% amd 10% by weight Al_2O_3 nano filler.
The PD inception voltage can be increased up to at least 4000 hours generally. The results show a considerable improvement after 4000 hours at 3% and 5% concentrations.

6 ACKNOLEDGMENT

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