Determination of the Effect of Al2O3 Nano Fillers on the Loss Factor and Insulation Resistance of Conventional Indoor Epoxy Resin

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Abstract
Nano fillers have been found in recent years in several areas of technology and can cause electrical, mechanical and chemical improvements. Adding small amount of nano fillers to epoxy resins can influence the mentioned properties. There are different types of nanocomposites and insulating material is composed of host and guest material. In this study, a conventional indoor epoxy resin as host material and related micro filler are the same for all produced specimens. Al2O3 and nano fillers were considered as guest material. Different percentages of the nano fillers were mixed with epoxy resin using ultrasound device and high speed mixer simultaneously. Regarding to the importance of a homogeneous distribution of the nano fillers in the epoxy resin, an even distribution of the nano fillers was validated by means of transmission electron microscopy.

Considering guard ring, the measurements of loss factor, permittivity and capacity were carried out at 200 V over the frequencies of 1 kHz, 50 Hz and 1 mHz. In addition, specific volume resistivity of the specimens was measured and evaluated.

To study the dependency of the measured parameters on temperature, the effect of temperature was investigated from 23°C till 230°C.

1. Introduction
To achieve a compact and reliable design of electrical equipment for the present day requirements, there is an urgent need for better and smart insulating materials and in this respect, the reported enhancements in dielectric properties obtained for polymer nano composites seems to be very encouraging [1].

There are many reasons to emphasize the importance of polymeric nano composite exploration, such as excellent mechanical properties, high thermal conductivity, good partial discharge (PD) and tracking resistance and so on. Due to those good properties and performances, polymeric nano composite dielectrics are being studied in detail all over the world. There are many papers reporting plenty of dielectric properties on polymer nano composite and researchers do believe its potential application in electrical and electronic engineering [2].

The effect of filler size on the dielectric property of the polymer composites has not been understood fully. It is with the advent of nanotechnology leading to the availability and commercialization of nano particles that polymer nano composite technology started to gain momentum. Polymer nano composites have been found to exhibit enhanced physical, thermal and mechanical properties, compared to the traditional polymer materials and with at low nano filler concentrations (1-10%) [3-5].

2. Test conditions
Although the samples were produced under vacuum, it is difficult to omit cavities during the production completely. In this condition, nano particles 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.

3. Used materials
3.1. Host material
Indoor epoxy resin system including CY228 as resin, HY918 as hardener, DY062 as accelerator and 65% Quarz Millisil W12 as micro filler were used as the host material for all the specimens. The glass transition temperature of the epoxy resin is about 115°C.

3.2. Nano filler
Unmodified Al2O3 was selected as nano filler, because this is the conventional nano filler and also economic. The samples were produced considering maximum 10 % pbw, because the final costs of the product with more than 10 % pbw are uneconomic and the viscosity of the whole material after the mixing is too high and casting would be difficult consequently.

Fig.1. Al2O3 nano fillers (spherical, 40nm)

The mentioned percentages by weight are valid for the end product and not only for resin or hardener.
The average size of the spherical Al₂O₃ fillers is 40 nm. The different sizes of fillers influence mixing and casting process. As smaller the size of nano filler is, as easier the mixing and casting.

3.3. Electrode configuration
For the measurements, three electrodes were considered according to IEC 60250 and VDE 0303 part 30. In addition to what the mentioned standards recommend, the measuring and protecting electrodes were constructed separately, so that both can be placed fully optimized on the specimen independently.

3.4. Mixing nano with resin
The nano fillers were firstly mixed with resin as the base material. After all the nano particles 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 particles 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. The above mentioned mixing behaviour was used till 5 % pbw. For 10 % pbw 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.

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 controlled temperature in several steps for 45 minutes. Finally the mixed material was casted into the molds 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 forms were cooled down for 8 hours.

To let the molecules of the samples relax, the measurements were done 4 weeks after the curing process. If the nano fillers were distributed homogeneously and in nano scale, a proper sample of each material was prepared and an even distribution of the nano fillers was validated by means of transmission electron microscopy.

The results show that the nano fillers were well distributed in the whole volume. REM technique a separation of nano and micro filler is possible. As explained, it is obvious that the spaces between micro fillers are mostly filled by nano fillers.

Fig. 2 shows the micro and nano fillers separately and also a combination of them in the host material, so that the green points show the distribution of nano particles and the red areas the distribution of micro fillers. Du to

4. Setup for Measurements
4.1. Setup for loss factor measurement
According to IEC 60250, loss factor was measured using an insulation plate with 2 mm thickness and considering 200 V, 50 Hz in a shielded measurement system with an Omicron measuring device named DIRANA. To make the measurement time shorter, loss factor was measured based on both FDS and PDC. At higher frequencies the FDS method was used and at lower frequencies, the PDC method was used in order to use the time optimally.
4.2. Setup for specific volume resistance measurement

This measurement was done according to VDE 0303 part 30 considering current-voltage measurement. The applied DC voltage was 2000 V. One Minute after applying the DC voltage on the specimen, the current flowing through the specimens was measured.

5. Results

5.1. Loss factor measurement

The measurement was renewed every 30°C temperature increasing up to 230°C. To reach the desired temperature all over the specimen, each specimen was pre-heated for one hour before each measurement.

Fig. 3 up to Fig. 7 show the loss factor of the specimens under different temperatures and three frequencies. Comparing all 5 figures, it can be found that nano filler is responsible for decreasing of the loss factor of the specimens with 5% Al2O3.

Fig. 6 shows that adding 5% Al2O3 causes a considerable improvement of loss factor up to high temperatures.

It was found that the value of dielectric loss peak of the micro composite is higher than that of the nano composite. This indicates that the interface trap energy level of nano composite is deeper than that of the micro composite, which results in less space charges participating in conduction and depolarization. Therefore, the value of dielectric loss peak of the nano composite is lower than that of the micro composite [2].

The curve of 1 mHz in Fig. 6 shows a relative constant value of loss factor up to 170°C, while the other curves of 1 mHz show a sudden increasing at the temperatures between about 50°C and 80°C.

Taking a look at all 5 curves of 50 Hz, it is clear that the first peak points have been shifted to higher temperatures. Especially with 5% concentration, so that at around 115°C (Tg), the loss factor is about 0.2%.

Fig. 4. Loss factor of the specimens with 1% pbw Al2O3 nano filler

In addition at about 185°C or about 75°C above Tg, the value of the loss factor is still 2%, which is two times smaller than its nominal value considering no nano filler. So the maximum value of the loss factor would probably to be happened at temperatures of higher than 230°C.

Considering 5 curves of 1 kHz, the values of the loss factor show, that adding 5% Al2O3 reduces the loss factors considerably in comparison with the other materials and the maximum value of the loss factor was shifted to much higher temperatures than 230°C.

Fig. 5. Loss factor of the specimens with 3% pbw Al2O3 nano filler
5.2. Insulation resistance measurement

The measurements were done from 23°C up to 200°C. After each 30°C temperature increasing up to 200°C the measurement was renewed. To reach the desired temperature all over the specimen, the specimens were pre-heated for one hour before each measurement.

Fig. 8 shows an increase of specific resistance of the specimens with 5 % pbw nano filler. So, the reduction of the loss factor with 5 % is the consequence of the increasing insulation resistance. The specific resistance is approximately constant from room temperature up to about 140 °C with 5 % pbw, which is about 25°C above Tg. Above 140°C, the resistance reduces, but the curve of 5 % is still above the other ones.

6. Conclusions

- Adding 5 % Al2O3 to the introduced host material causes a considerable improvement in loss factor

7. Acknowledgments

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8. References


