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PARTIAL DISCHARGE PATTERNS AND SURFACE DETERIORATION IN VOIDS IN FILLED AND UNFILLED EPOXY.

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Abstract

Results are presented from pulse analyses on pulses from partial discharges (PD) in single spherical voids in filled and unfilled epoxy plastic related to the observed surface deterioration. The used filler types were dolomite, alumina and silica.
Long time aging tests including pulse phase/height analyses (PPA & PHA) were performed over a period of 2400 hours showing very characteristic discharge patterns for each material combination. A unique behaviour with regard to changes of pulse repetition rate and max. apparent charge could be observed for PD in alumina and silica filled epoxy.

Investigations of the void surfaces were performed by scanning electron microscopy on voids exposed to PD as well as on voids without discharges. Different kinds of surface deterioration were found and an attempt was made to establish a relationship between aging phenomena, such as void surface changes and discharge patterns.

Introduction

In this report are described results from aging tests on spherical voids in unfilled epoxy and epoxy with different fillers exposed to partial discharges for 2400 hours. Analyses have been performed on the electrical pulses from the discharges and were compared to results from surface investigations and gas analyses on voids exposed to PD.

In order to ensure a development of the discharges as 'natural' as possible, they were not triggered in any way. For the same reason, the spherical voids were made in a way that ensure a natural formed surface and a gas composition comparable to gases formed under realistic conditions.

The combination of continuous pulse analyses and surface/gas investigations with different materials made it possible to:

a) investigate aging processes in spherical voids and their dependency on the filler,
b) characterize the materials' suitability with respect to partial discharge resistivity.

Test Setup

The voids were produced by injecting clean, dry air in the filled epoxy before curing at 1 bar. The epoxy used for the tests was a standard type Bisphenol-A Diglycidylether (Ciba-Geigy CY225) cured with an acidanhydride curing agent (Ciba-Geigy HY925) containing 60% (weight) filler. The used filler types were dolomite-$CaCO_3{\cdot}MgCO_3$, alumina-Al_2O_3 and silica-SiO_2, all dried at 150°C for 24 hours. For each material combination, 5 samples with single voids of 1.2 - 1.5 mm in diameter were exposed to a 50 Hz AC Voltage, 21 kV_{rms}, for 2400 hours (1500h for unfilled epoxy). The electrode gap was 5 mm.

Pulse detection and analysis was performed with the measuring system shown in fig.1.

PD detection was based on in-line measurements of the discharge current through a 50Ω measuring impedance formed by oscilloscope and analyzer. The apparent charge is calculated by the time integral of the current pulse *i(t)* visible on the oscilloscope, and the calibration of the system is performed by relating this value to the signal read by the analyzer.

Computer controlled pulse phase and pulse height analyses were continuously performed for each sample under the aging tests.

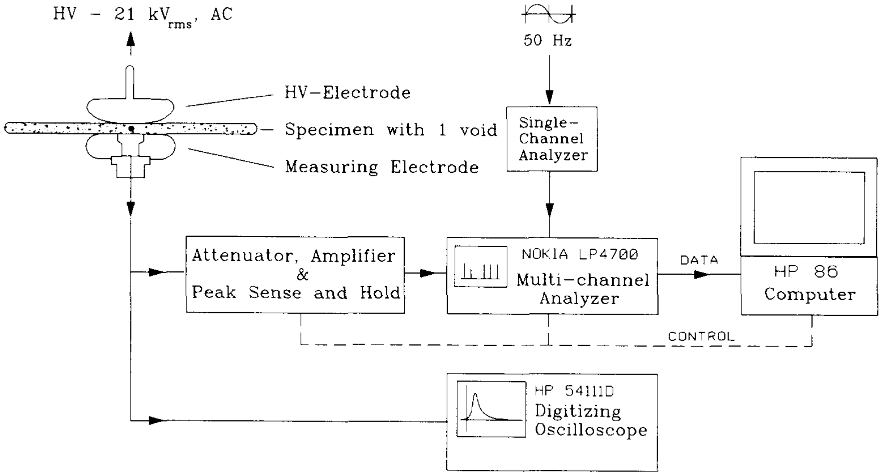

Fig.1: Measuring system

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Results

Pulse analyses

Fig.2 and fig.3 show the pulse repetition rate and the maximum detected apparent charge as the average of the results from 5 specimens for each material combination.

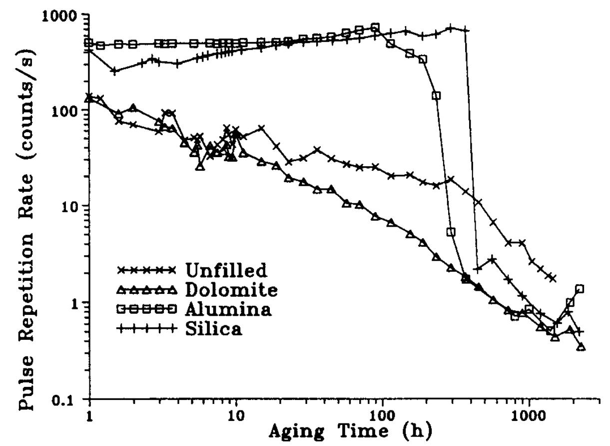

Fig.2: Pulse repetition rate as a function of aging time.

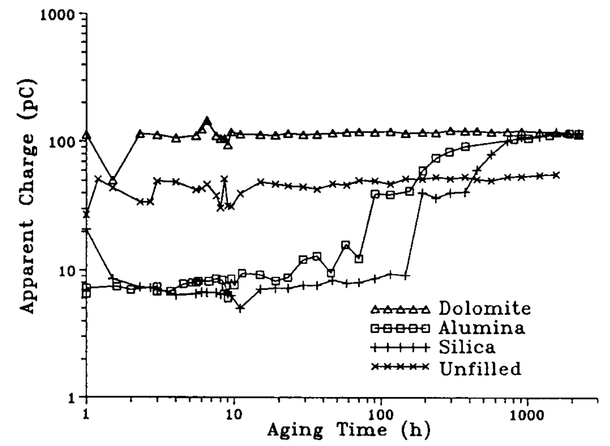

Fig.3: Maximum apparent charge as a function of aging time.

Discharges in voids in unfilled and dolomite filled epoxy showed a periodical discharge activity, which decreased continuously, while the maximum apparent charge was nearly constant. Discharges in alumina and silica filled epoxy showed a continuous and slowly increasing activity, until it after 2-300 hours within few hours dropped with a factor near 1000 to the level of the other materials and changed to periodical characteristic. The maximum apparent charge before this transition was about 20 dB below the one of unfilled and dolomite filled specimens. After the transition the maximum apparent charge of all filler specimens reached the same level. The change of the discharges' character in alumina and silica filled epoxy also resulted in a change of the charge distribution. Fig.4 shows the charge distribution after the transition.

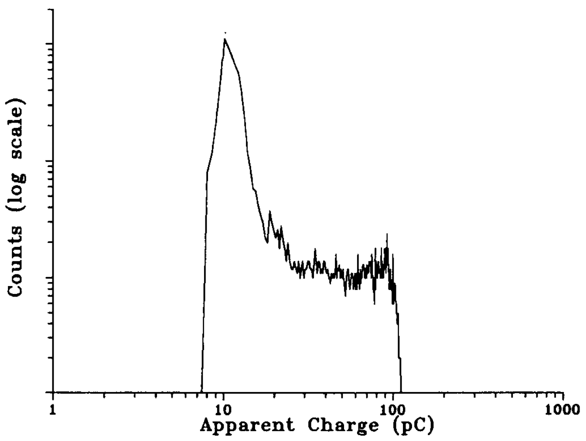

Fig.4: Typical distribution of the apparent charge for discharges in voids in unfilled and dolomite filled epoxy. Similar to the distribution for alumina and silica filled epoxy after transition to periodical activity.

Before the transition to periodical activity alumina and silica showed a charge distribution consisting only of a large number of discharges of about 10 pC.

In fig.5 and fig.6 can be seen the phase distributions of pulses from the discharges. Again, unfilled and dolomite filled epoxy showed comparable characteristics, as did alumina and silica.

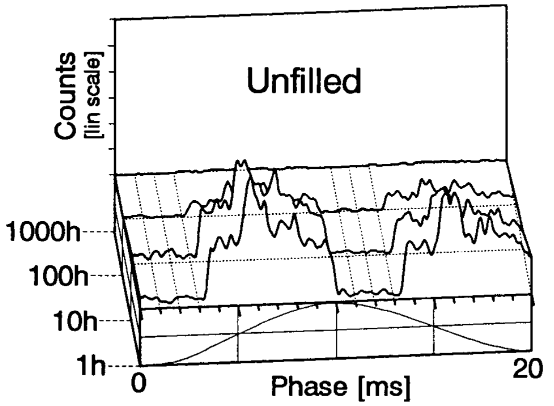

Fig.5: Phase distribution of pulses from discharges in voids in unfilled and dolomite filled epoxy as a function of aging time.

Phase analyses of discharges in the unfilled and dolomite filled specimens showed a decreasing activity, decreasing inception voltage, and a discharge pattern with only few and difficult to define pulse concentrations. The phase distributions of alumina and silica filled epoxy were characterized by very well defined pulse concentrations increasing from 5 to 9 per halfcycle before the transition to periodic activity.

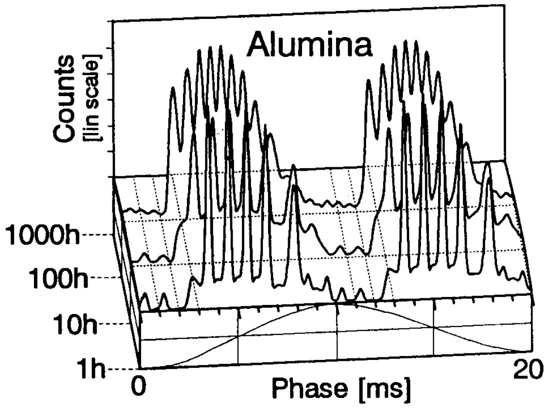

Fig.6: Phase distribution of pulses from discharges in voids in alumina filled and silica filled epoxy as a function of aging time.

Gas Analyses

After the aging tests, the voids were cleaved into two parts and the released gas was analyzed by a mass spectrometer. Apart from small amounts of CO_2, nitrogen was found to be the only gas at a pressure of about 0.1 - 0.3 bar after 2400 hours with discharges. This, compared to the pressure measured in non-exposed voids of 0.4 -0.6 bar, seems to confirm the involvement of nitrogen in the deterioration processes. The pressure in similar non-exposed voids produced after the same method and with the same epoxy has earlier been measured to 0.7 bar [1].

Surface Investigations

Surface investigations were made by scanning electron microscopy (SEM) of the voids exposed to PD and compared to the surface structures of non-exposed voids. The latter showed plain surfaces (within the resolution of the microscope) in unfilled epoxy. Dolomite filled epoxy surfaces were characterized by the highest degree of roughness and silica by the lowest. Dolomite grains were only partly covered with epoxy whereas almost all alumina and all silica particles were placed under the voids surface. For all fillers, the largest particles were facing with a flat side towards the void.

In the following can be seen some of the most typical structures on the deteriorated surfaces after being exposed to partial discharges.

Common to all materials was intense deterioration of the epoxy in the polar area (assuming the electric field in the void directed from pole to pole), whereas all filler particles seemed to be unaffected. In the equatorial area some surface changes could be seen, which could be deterioration byproducts as well as a lightly deteriorated surface (fig.7).

As can be seen in fig.8,9 & 10 some of the epoxy was removed from the surface of the filler particles under the exposure to discharges. Unfilled, dolomite and alumina filled epoxy showed an even deterioration, with the epoxy forming round shapes of less than 1 μm radius (fig.11). In silica filled epoxy the deterioration seemed to be more severe, but was limited to certain areas (fig.10). The rest of the exposed area was also affected, but only in a degree as seen in the equatorial area.

Fig.7: Surface of a spherical void (1.2mmø) in dolomite filled epoxy after 2400h of exposure to discharges. Equatorial area, when assuming the electric field in the void directed from pole to pole. (SEM picture)

Fig.8: Surface of a spherical void (1.2mmø) in dolomite filled epoxy after 2400h of exposure to discharges (SEM picture).

Fig.9: Surface of a spherical void (1.2mmø) in alumina filled epoxy after 2400h of exposure to discharges (SEM picture).

Fig.10: Surface of a spherical void (1.2mm⌀) in silica filled epoxy after 2400h of exposure to discharges (SEM picture).

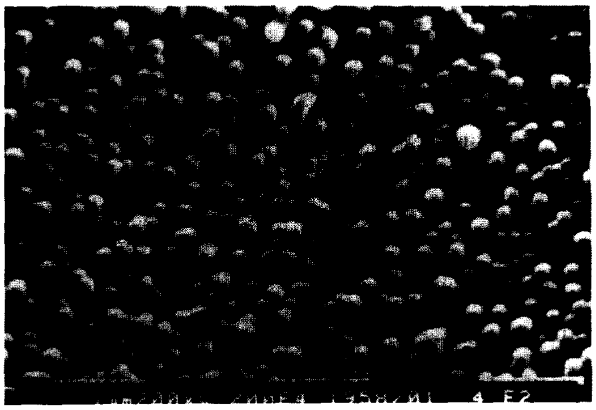

Fig.11: Surface of a spherical void (1.2mm⌀) in unfilled epoxy after 1500h of exposure to discharges (SEM picture).

Fig.12: Crystal like structure on the surface of a void in dolomite filled epoxy after exposure to discharges in 2400 hours (SEM picture).

In all of the unfilled and dolomite filled specimens some crystal-like structures were found (fig.12). The crystals could be dissolved in water but not in ethylene. The aqueous solution was non-acidic, which means that the crystals did not contain oxalic acid as earlier suggested [2]. In the same paper it was implicated that these crystals could cause a sudden high discharge activity, which we feel could possibly be due to fieldenhancements by the crystals.

Discussion

The most interesting result is the characteristic behavior of discharges in the alumina and silica filled specimens. The continuous discharge activity in the beginning and the well defined phase distribution is most likely caused by the presence of initial electrons each time the inception voltage is reached. To confirm this, the expected pulse repetition rate can be calculated from fig.6, which shows 10 pulses/cycle or 500 counts/sec which is exactly the measured pulse repetition rate for alumina in fig.2. Consequently, we must assume that the transition to the periodic activity is caused by missing initial electrons. This is confirmed by the observed change of the charge distributions: Before the transition, the narrow charge distribution indicates that only one discharge type occurs. After the transition, few discharges with a high apparent charge can be seen in fig.4. They indicate another discharge type, which earlier has been identified as streamer discharge, and occurrence of which was shown to be possibly due to overvoltages which could have been caused by missing initial electrons [3]. Irradiating of the aged voids under voltage with X-rays [4] showed a discharge activity of 450 counts/sec. That confirms the missing initial electron as the main reason for the decreasing activity and, furthermore, excludes changes of the surface resistivity as a possible reason. The decreasing inception voltage fits very well with the slightly increasing activity before the change. This may be caused by the decreasing gas pressure (measured), which according to Paschen curves will cause a lower inception voltage.

It was not possible to determine why initial electrons were present in alumina and silica filled epoxy, but not always in unfilled and dolomite filled, but there were some indications of moisture as an important factor. To prove this, it would be necessary to perform chemical analyses of the deterioration processes which was not possible with the small amounts of substance available. From the above it is most likely that the decreasing discharge activity in unfilled and dolomite filled specimens is also caused by missing initial electrons.

A consequence of the different behavior of the discharges is the different influence on the materials. As shown in [3], two very different discharge types were present in the voids, which deteriorating effect on the material is not known. Under the assumption that the deterioration is proportional to the charge separation in the void the relative exposure to discharges can roughly be estimated as follows: The charge distribution in fig.4 shows that the small number of large discharges allows for the approximation of the exposure to discharges as caused by the small 10 pC discharges only. That means that the exposure becomes proportional to the total number of discharges, and integrating the pulse repetition rate (fig.2) indicates that alumina and silica filled specimens had been exposed to discharges approximately 10 times higher than the other materials.

Compared to the similar deterioration of dolomite and alumina specimens and the more severe deterioration of silica specimens, this means that alumina seems to resist the discharges best, probably due to its highest thermal conduction (5 times higher than that of dolomite and silica). On the other hand, due to self extinction, dolomite results in the lowest exposure, a behavior that may be the most suitable for some applications.

Apart from the different degrees of deterioration there were no indications of a beginning breakdown in any of the material combinations. But it should be mentioned, that the same materials showed very different lifetimes under accelerated aging tests using the Cigré, method II, where the materials were exposed to partial discharges, some orders of magnitude higher than used in above described case [5].

An interesting detail is the equal maximum apparent charge for all filler specimens after 2400 hours aging and that the charge in the unfilled material was about half this value. The filled materials had a comparable permittivity about 2 times that of unfilled epoxy. Assuming the apparent charge from discharges in voids as being proportional to the permittivity of the surrounding material as shown in [6], it can be concluded that the aging processes in voids seem to equalize the void characteristics with respect to partial discharges for the different material combinations tested.

Conclusion

Spherical voids in unfilled epoxy and epoxy with 60% dolomite, alumina and silica filler, respectively, have been exposed for non-triggered partial discharges for 2400 hours. Analyses were made on pulses from these discharges and compared to the observed deterioration phenomena on the void surface and changes in the void gas. According to pulse repetition rate, apparent charge and the pulse analyses, the materials could be divided into two groups:

1. Unfilled and dolomite filled epoxy, which showed a periodical discharge activity, which decreased continuously, while the maximum apparent charge was nearly constant.
2. Alumina and silica filled epoxy, which showed a continuous and slowly increasing discharge activity for the first 2-300 hours after which it dropped to the same level and periodical characteristic as the other materials. The continuous activity is most likely caused by the presence of initial electrons. Under the assumption of linearity between apparent charge and deterioration, these materials were exposed to a total discharge activity about 10 times larger than unfilled and dolomite filled epoxy.

PD-aging of the voids in the different materials seems to equalize the voids electrical characteristics.

All materials showed the most intense deterioration in the voids polar area, where the filler particles had the epoxy layer removed, but otherwise were not affected by the discharges. The deterioration seemed to be most severe in silica filled epoxy.

Based on the above results it seems that the optimum filler with respect to partial discharge resistance should be a filler with high thermal conductivity, a gas composition not releasing initial electrons and without forming fieldenhancing structures on the surface.

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