In-situ observation of electrical tree evolution in epoxy dielectrics with internal cracks

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Abstract
Gas-filled internal crack might appear in thermostet materials like epoxy resin during the equipment manufacturing, which would become a vulnerable local region to initiate the electrical tree, thus prone to cause insulation failure. The withstand voltage test was carried on epoxy samples with artificial cracks based on a rod-plane electrode arrangement. Simultaneously, surface state variation and tree evolution with crack were observed by an optical microscope in conjunction with a charge-coupled device camera. The changes in morphology and chemical status of the crack surface were characterized by scanning electron microscopy, laser Raman spectrometer and energy dispersive spectrometer, respectively. It was found that the erosion and tree started from the borderline of crack under a relatively low electric field strength; however, the area near the electrode had relatively little damage. The breaking of epoxy molecular chains coarsens the crack surface and further forms deep channels on a micro-level, which is the forerunner of the electrical tree inception. Based on these, the initiation mechanisms of the crack-induced electrical tree and the reasons for the erosion near the borderline have been revealed. This study provides a train thought for the polymer degradation opening up into the initial tree channel during tree evolution processes.

1  INTRODUCTION

Epoxy resin has the advantages of excellent mechanical and electrical strength as well as good chemical stability, which is widely used in electronics and power equipment [1], such as electronic encapsulation [2], saturable reactors [3], cable terminals, insulators used in gas insulation system and epoxy-gas composite insulation bushings [4, 5]. However, due to the harsh operating environment, treeing degradation is prone to occur in the insulation component of AC high-voltage equipment and it is the beginning of insulation breakdown. Once the tree bridges through the high-voltage electrode and the ground electrode, it will lead to insulation failure before long [6], which will cause severe damage to the power equipment.

Therefore, numerous studies have been proposed on the investigation of the treeing processes for the characteristics and the mechanisms because the electrical trees were found [7]. Tree structures and external conditions during the growth process are the two most commonly used objects in exploring the mechanisms of electrical tree evolution. Applied voltage, temperature and other terms would affect the shape of the tree and its initiation voltage, growth rate, fractal dimension and so on. The characteristics of electrical trees could influence by lots of different factors [8], including the effects of varying operation temperatures [9], various voltage components (e.g. AC and DC, repetitive pulse, superimposed harmonic voltage) [10, 11] and frequency [12] have been reported. In addition, the process of tree growth would be accompanied by many physical and chemical phenomena. Over the years, based on previous studies, many scholars have been devoted to achieving the self-healing of electrical trees by combining the nature of electrical trees and advanced materials technology [13, 14]. The most recent breakthrough has used the electroluminescence phenomenon during the treeing process and the microcapsules to recover the insulation strength in epoxy [14]. Thus, the fundamental study of the characteristics and
the mechanism of electrical tree initiation in different conditions is greatly significant.

Internal air-filled defect is the major factors resulting in insulation deterioration in practical engineering. Electrical trees are often caused by such defects under long-term operation and lead to power equipment failure eventually. Previous studies have focused on the electrical tree behaviour and the breakdown performance of polymer dielectrics with artificial defects [12, 15], in which different defect types and frequency have been systematically considered. The differences in electrical tree characteristics between the undamaged sample and the sample with needle-generated air gap have been studied by comparing the initiation voltage, tree shape and tree length [16]. In recent years, Rowland and co-workers investigated the influence of electrode separation on electrical tree growth [17, 18]. The air gaps were fabricated between a needle and epoxy resin, and an AC stress of 50 Hz was applied to induce the development of the growth of electrical trees artificially. In particular, the partial discharge property accordingly was systematically studied. Unlike soft polymers such as cross-linked polyethylene, epoxy resin is prone to be affected by mechanical stress from the production and manufacture processes. Defects like internal cracks and delamination may occur in such kinds of polymeric materials due to various stresses during operation [19], which will become a potential risk for the safety and stability operation of power equipment.

Besides, no matter whether there exist macroscopic defects, the electrical tree seems to initiate from the interface between two different materials. Therefore, the change and state evolution around the interface is worthy of being paid attention to. Moreover, how chemical bonds are broken and how the degradation opens up into the initial tree channel are still unknown [20]. The cognition of the whole process from interface state evolution to tree initiation is a way to understand the inception of the electrical tree. However, there is a lack of systematic research on the interfacial state during tree evolution and few studies ever concerned the effect of the air gap on the treecing process so far.

The interface state and the tree evolution in polymer dielectric with artificial air-filled crack were studied. Surface state variation on the interface during the whole treecing process was recorded by an optical microscope and charge-coupled device (CCD) camera. With the help of a scanning electron microscope (SEM), the micro-morphology on the interface before tree initiation was investigated for the first time. Raman spectrum and energy dispersive spectrometer were utilized to detect the molecular structure and element composition and analyse the chemical properties on the crack surface. Given that the erosion and the tree initiates at the borderline of crack, the process and the mechanism will be discussed in detail. The results can contribute to further explanation of insulation failure in long-term operation and give a probable research method of tree initiate under a relatively low electric field.

2 | EXPERIMENTAL PROCEDURE

2.1 | Sample and experimental apparatus

The Bisphenol A epoxy resin (CT5531Cl) and the carboxylic anhydride curing agent (HT903Cl) are supplied by Huntsman. After well mixing at a speed of 500 r/min at 120°C, the mixture is degassed for 10 min. Then the liquid cured under a specific temperature process in the sample mould. In order to obtain cracks that have similar actual width, the cured epoxy samples were treated by directional mechanical disruption with the following steps: (i) Fix the sample on a horizontal platform and (ii) Adjust a hammer with a conical arrow to a certain height and then let it fall freely towards the sample. The energy of directional mechanical disruption was deliberately controlled by the gravitational potential energy of the hammer falling. Each sample was treated by directional mechanical disruption only once to ensure that only one crack will generate in each sample.

Figure 1a gives an overview of the crack with the epoxy sample from two different views. After mechanical treatment, the shape of the crack is a regular sector whose wavy stripes can obviously be seen in the circumference direction. It is due to the propagation of mechanical waves during mechanical treatment, which makes the cracks in the sample approximate to those in the insulation equipment. The maximum width of the crack is ∼22.0 μm, as shown by the SEM image in Figure 1b, which located at the cross-section image of the sample.

The rod-plate electrode system is applied to research the development process of the electrical tree with crack. The rod electrode with a diameter of 1.25 and ∼0.05 mm fillet is 10 mm from the ground electrode, inserting into the epoxy sample with a dimension of 10 × 60 × 15 mm. Figure 2 depicts the schematic of the experimental sample and arrangement. Copper foil is placed on the bottom of the sample as the ground electrode, and cracks opening is sealed by room temperature vulcanization (RTV) silicone rubber whose colour is white. When sealing, the opening of the crack was oriented in the same direction of gravity to prevent RTV from entering the crack. After sealing, it is checked that the crack remains a certain degree of transparency to confirm that there is no RTV silicone rubber inside the crack. The experiment is carried out in a silicone oil bath at a constant temperature of 20°C. 20 kV, 50 Hz AC voltage is applied to the rod electrode. When the sample nearly breaks down, or severe damage is observed, the experiment stops. In this study, an online image monitoring system consisting of a stereomicroscope (PX59-T) and a CCD camera (TOUPCAM U3CMOS18000KPA) was used to record the evolution of surface state during the tree initiation period.

2.2 | Characterizations of surface physical and chemical properties

The morphology of erosion on the crack surface was observed by SEM (KEYENCE, VE-9800S). Synchronously, relative compositions of elements, chemical state, and structures before
and after the experiment were used to characterize the degradation of the crack surface by using energy dispersive spectrometer (EDS) (Aztec, X-maxN50mm2/TESCAN MAIA3 MLH) and Laser Raman Spectrometer (Horiby, LabRAM HR Evolution).

3 | RESULTS

3.1 | Process of surface erosion and tree initiation with crack

According to the severity of macro damage during the whole test, the changes of crack surface state could be divided into three stages shown in Figure 3-5, with the characteristics of slight damage, brown ablation and carbonization respectively. All images were taken under the light reflection mode.

From the observation of stage I, the main feature is slight damage at the borderline of the crack, as is shown in the schematic drawing of Figure 3. In Figure 3b, after loading 20 kV AC voltage for a relatively short period, about 90 min, slight damage independently appears at the fillet of the rod electrode and the borderline of the crack. The damaged region turns to milky white, accompanied by a decrease in transparency. As time goes by gradually, the damage expands to the surrounding area, and the colour becomes evident in Figure 3e. Finally, slight damages have extended entirely across the crack surface in Figure 3d. It seems like ‘bridging’ as they link up into a single stretch.

Surface erosion moves into stage II at about 300 min, while brown ablation appears. Figure 4a shows the whole process in stage II. Figure 4b is the enlarged views of the brown ablation in Figure 4a. As is shown in Figure 4a (1), the colour of the ablation zone near the borderline turns from white to transparent brown. Continuous channels appear at this time. It is noteworthy that the channels initiate at a certain distance from the borderline and extend toward the rod electrode subsequently, as the reverse ablation in Figure 4b (2).

The extension trend towards rod electrode, instead of the borderline of crack, has similarities to the reverse tree in epoxy. Carbonization occurs at brown channels in 781 min, continuous diffusing towards the periphery, as is shown in Figure 4b (3). It can be seen that a small area near the borderline does not develop into a brown ablation but turns to char directly. The changes in borderline and brown ablation zone seem to develop independently in location but to be a connection in time. It can be considered that the micro-electrical trees initiate on the borderline, and the carbonization begins here at the moment.

In stage III, carbonization extends and electrical trees grow rapidly. Electrical trees that are similar in length and shape could be observed in epoxy shortly after stage III begins, given in Figure 5b (1). However, the region near the rod electrode only changes to brown ablation with a tiny branch, while more severe carbonization does not generate nearby. Besides, the state and the range remain unchanged at the location marked in Figure 5a (2). Figure 5b (2) gives an overview that the electrical trees grow outward from the crack. Several electrical trees break through the borderline of the crack here and expand. After the extension
**FIGURE 3** Micrograph on crack surface of stage I

**FIGURE 4** Micrograph on crack surface of stage II

**FIGURE 5** Micrograph on crack surface of stage III
of the carbonization, two approximate parallel branch trees grow outward, with distinct trunk and short branches, given in Figure 5a (3). Since then, these two trees have been developing from high voltage to the grounded electrode.

3.2 | Development of carbonization

Stage III almost accounts for about half the time of the whole process, and the development of carbonization is very rapid. Colour recognition algorithm based on Hue Saturation Value (HSV) colour space can effectively identify the borderline of the carbonized area by setting a reasonable threshold. The proportion of carbonization can reflect the development of surface erosion in the last stage is in the whole image with time. Figure 6 gives the proportion of carbonized area to total image area (S1/S2) over time after HSV colour identification analysis.

As shown in Figure 6, at the beginning of stage III, the carbonized area grows at a high speed as the time varies. It can be seen that before the 21st hour, the proportion of the carbonized area increases linearly with time. However, when the electrical tree is approaching the ground electrode, the development speed of the carbonization slows down significantly. It is analogous to the phenomenon of electrical tree hysteresis. Moreover, after the carbonization propagates to a certain extent, the carbonization area almost has no change, which appears after the 23rd hour.

3.3 | Micro-morphology

By using SEM, the typical characteristics of the crack surface are investigated. What we focus on is the evolution of surface state under the microscopic. The SEM images of the surface erosion process are shown in Figure 7. It is evident that long-term operation will cause severe damage to the crack surface. With time, the depression and the delamination appear, and eventually, visible gullies emerge. Some regions develop from pores and capillary ablation to severe spalling.

In Figure 7a, the crack surface becomes rough. Crisscross shallow gullies occur on the crack surface, and the surface looks relatively continuous and smooth. Corroded protrusions and depressions get worse and worse as time goes on. Directional shallow channel emerges then, and there seems to be a continuing progressive trend. The uneven surface may become
defects that would distort the electric field distribution. Macroscopically, this zone of the crack loses lustre gradually.

Figure 7b shows that the shallow channels expand and go deeper as the erosion develops. It can be seen from Figure 7b that capillary cracks, pores, and delamination are apparent around the edge of the channels. It could be observed that the surface near the channels suffers from extensive damaging. It becomes a vicious circle that the coarse surface distorts the electric field more seriously, which would accelerate the damage of polymer and make the ablation gradually deeper. At this time, the epoxy is further decomposed, accompanied by the change of surface colour.

The depth of the channel increases significantly and the trees initiate in Figure 7c. The wall of deep gullies is severely damaged and much more distinct. The channels have not only developed along the crack surface but also expend toward epoxy resin solid. Electrical trees initiate now. In this case, the carbonization zone has become a semi-conductive or conductive region because of the decomposition of epoxy, which makes a sharp increase of damage.

3.4 Chemical components on the crack surface

The chemical components of the crack surface were analysed by Laser Raman Spectrometer and EDS for studying the degradation of epoxy. It should be noted that the spectra named original in Figure 8 and Figure 9 represent the result of untested samples, and stage I and stage II reflect the spectra of slight damage and brown ablation in Figures 3 and 4, respectively.

Raman spectroscopy is an effective means to characterize the chemical nature of the polymer and electrical tree structure [21–23]. Here the degradation process of the crack surface in different stages was analysed by Raman spectra and shown in Figure 8. Note that the characteristic peaks are basically in the same position, but the intensity of the fluorescent background is gradually increasing, by comparing with the curves A. No carbon is generated, indicating that the crack surface is still non-conductive until the stage III though it is aged to some extent [22].

To further analyse the chemical change during the test, Table 1 gives the Raman spectral peak assignment of epoxy resin. The characteristic peaks at 821, 1112 and 1188 cm⁻¹ are weakened, which correspond to C–H wagging, C–H bending and C–H scissoring. Therefore, the decomposition of C–H is one of the main chemical changes in the early stages. In the different stages, the characteristic peaks at 640 cm⁻¹ (corresponds mono-substituted benzene), 1580 and 1610 cm⁻¹ (correspond C=C stretching of benzene ring) are gradually weakening, showing that parts of benzene ring are broken. Figure 8 further shows that a slight change of CH₃ scissoring at the peak of 1440 and 1464 cm⁻¹, C–O stretching at the peak of 1232 cm⁻¹, indicating that CH₃ bond and C–O bond also destroy in a certain extent.

For EDS analysis, the characteristic peaks of C element and O element are at about 0.27 and 0.52 keV, respectively, as the

\[ \text{Peak/cm}^{-1} \quad \text{Assignment} \]

| Peak/cm⁻¹ | Assignment                     |
|-----------|--------------------------------|
| 640       | Mono-substituted benzene       |
| 821       | \( \omega \) C–H               |
| 1112      | \( \delta \) C–C, \( \beta \) C–H |
| 1188      | \( \delta \) C–H               |
| 1232      | \( \nu \) C–O                 |
| 1440      | \( \delta_m \) CH₃             |
| 1464      | \( \nu \) C=C of benzene ring  |
| 1580      | \( \nu \) C=C of benzene ring  |

Note: \( \omega \)—wagging, \( \delta \)—scissoring, \( \nu \)—stretching, \( \beta \)—bending in plane.
evident peaks, C K and O K showed in Figure 9. It can be seen that the elementary composition of the crack surface is almost the same in different stages. Nevertheless, with the development of erosion, significant changes in C and O elements appear. From original to stage I, the content of the C element has a little change while O is significantly enhanced, indicating that the oxidation has occurred in the polymer. The main reason is that a small amount of air is sealed in the sample. During the long-term high-voltage test, the oxygen in the air reacts with the epoxy. With the increase of test time, the content of the C element increases accordingly. It means that the O element is removing from solid during the further decomposition and the carbonization of epoxy would begin soon.

4 | DISCUSSION

4.1 The process of electrical tree initiation with crack

A series of chemical reactions have generated in the epoxy resin, which changes the physical characteristics of the crack surface. Combined with the observation and test results, the destruction process of the crack surface could be described in detail. Ascribe to the charge injection and extraction before the tree initiation, the energy can be transferred to the polymer, resulting in the fracture of the band [27, 28]. The changes in the chemical structure during this process can be depicted in Figure 10a. The broken of molecular chains is accompanied by the changes in surface state, which is slowly corroded and roughened. Thus, the Raman spectra superimpose on the fluorescent background and reveal that the peak of C–H, CH3, benzene ring weaken. The surface state changes act on the electric field distribution and dielectric properties, leading to the local field distortion and the future degradation of the polymer matrix in turn. Thereby, ablation appears and expends with brown channels on the crack surface (Figure 10b). When the chemical degradation accumulates to a certain extent, carbon is attached to at the crack surface and stack on the channel wall, formatting the conductive region [29] as shown in Figure 10c. Owing to this, a vicious circle that leads to continuous carbonization in the surrounding polymer is formed. In this case, the development of erosion can expand along the crack surface towards the high voltage electrode, or break through the borderline into the epoxy bulk. As a result, the electrical trees initiate to develop in terms of physical manifestation at the borderline of the crack in Figure 5a (1).

\[ f t | G_n - G_{th} | = C_i \]  

\[ G_n = A \exp(-B\phi^2E^{-1}) \]  

\[ G_{th} = A \exp(-B\phi^2E_0^{-1}) \]  

where \( f \) is the power frequency, \( A \) and \( B \) are constant, \( \phi \) and \( E_0 \) are related to the electrode and polymer respectively. According to Equations (1) and (2), higher electric field can transfer more energy to the polymer with the same operation time, indicating that it is more likely to destroy the polymer. Thus the phenomena marked in Figure 5a (2) can be describe as follow:

![Figure 10](image-url)
**FIGURE 11** Electric field line distribution and field components at different locations

Electric field strength on the marked area is weaker than that near the electrode fillet. The electric force is not enough to drive a large number of charges moving in the epoxy matrix. As a result, near the electrode fillet brown erosion appears while the marked area has little damage.

However, it was found that the area near the rod electrode under maximum field strength is just slightly eroded in the test, even maintained a certain degree of transparency. While the slight damage (Figure 3b), brown ablation (Figure 4a (1)), carbonization (Figure 5a (1)) start in the area near the borderline of the crack under a relatively low field and it has more severe damage in the long-term operation.

To explain this, Figure 11 gives a schematic diagram of the electric field component varying from the rod electrode to the borderline of the crack. Due to the size of the crack opening is tiny relative to the size of the rod electrode, the electric field line is almost along with the interface between crack and epoxy. By comparing Regions 1 and 2 shown in Figure 11, it reflects that the closer to the borderline of the crack, the higher the normal field strength is. In order to look into the electric field distribution of the sample with crack, a finite element simulation model was established and calculated by COMSOL. Figure 12 gives partial electric field distribution along the path in Figure 11, and the inset of Figure 12 shows the enlarged figure near the borderline. Ideally, we assume that the crack surface is smooth. It can be seen that the electrical field in Region 1 is almost tangent component, while in Region 2, it changes to normal component sharply. It indicated that although the electrical field strength near the electrode is high, the tangential electric field hardly drives charge injection and extraction in the polymer in this case. While on the borderline of the crack, driven by the normal electric field the surface was slowly and continuously corroded and carbonized.

On the other hand, a charge from the electrode can get more energy when it arrives the borderline because along the field direction the mean free path between collisions is high in the crack [10]. It means that the impact between charge and polymer has more probability to cause ionization and enhance discharge near the borderline. Such phenomena have high enough energy so that it is more likely to damage the crack surface. In addition, when observing the samples without being conducted with withstand voltage test, all of them have existed wavy stripes near the borderline, as is shown in Figure 13. The convexity of wavy stripe can distort local field distribution and also enhance the collision driven by the electric force. As a result, the charge would have a higher probability to destroy the epoxy, thus severe damage occurs.

**FIGURE 12** Electric field distribution along the path of the sample

**FIGURE 13** Scanning electron microscope image of sectional view near the borderline of crack

5 | CONCLUSIONS

The internal cracks were introduced into samples of epoxy resin to investigate its influence on electrical tree evolution. The physical and chemical changes of the internal crack surface during the treeing process were analysed for the first time. The following conclusions can be drawn:

i. A feature revealed for the first time by treeing with crack is that the erosion (including slight damage, brown ablation and carbonization) and the electrical tree are partial to initiate at the low-electric-field region rather than near the
rod electrodes. It is related to a combined action of electric field strength, field direction, charge and surface state.

ii. Carbonization is a demarcation line that only when it appears the electrical trees would initiate at the borderline of crack. After appearing, it keeps extending at a constant rate reversely to the rod electrode and slows down until the insulation is close to failure but will never extend to the rod electrode.

iii. Treecing in crack makes it easy to obtain the micro-morphology and chemical components during the whole process of electrical tree evolution. On a micro-level, the surface-coarsening transforms into deep gullies on the interface is the precursor of the degradation that opens up into the initial tree. In this process, chemical bonds (e.g. CH, CH3, benzene ring) are broken and oxidized. Eventually, carbon shows up and results in the tree branches.

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