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Serial Block-Face Scanning Electron Microscopy for Three-Dimensional Imaging of Electrical Trees

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Abstract—Electrical trees are defects that can grow in polymeric insulation and cause power equipment failure. They have been imaged mainly using two-dimensional approaches. Here we present the use of Serial Block-Face Scanning Electron Microscopy (SBFSEM) for three-dimensional (3-D) model reconstruction of electrical trees. SBFSEM is an automated process of serial sectioning combined with block-face imaging of the sample, inside a chamber of a low-vacuum SEM. Two polymeric samples with a bush and branch-type electrical trees were examined using SBFSEM. 3-D replicas of sections of the tree were created and tree characteristics quantified. Parameters such as diameter and number of tree channels, tree volume and proportion of volume degraded were obtained and compared, indicating that electrical trees can be classified through these parameters.

Keywords—electrical tree, imaging, three-dimensional, 3-D, SEM, SBFSEM.

I. INTRODUCTION

Electrical trees are the main mechanism of ageing of polymeric high voltage insulation, a precursor to failure of electrical power equipment. Imaging of laboratory grown electrical trees has been one important method of studying and understanding better the development of electrical trees [1, 2]. Imaging techniques that have been used are mainly optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). These techniques are able to resolve the structure of the tree in a two-dimensional perspective. Other but less common used techniques include ultrasound and nuclear magnetic resonance, which can detect areas of treeing, but not structural details of the tree.

Electrical tree growth is three-dimensional (3-D), therefore, a 3-D analysis provide a more accurate representation of the tree structure. We have shown that using X-ray computed tomography (XCT) 3-D replicas of electrical trees can be reconstructed [1, 3]. Through virtual slicing of the replica the tree can be internally inspected and its entire structure analysed. In this paper, we present the alternative of using Serial Block-Face SEM (SBFSEM) to generate 3-D models of electrical trees.

SEM, low-vacuum SEM and SBFSEM

SEM is a well established technique for imaging the surface of materials for identification of morphology/topography. In SEM a beam of electrons is scanned across the surface of the specimen. The incident beam interacts with the surface of the sample and signals are emitted as a result of these interactions; these emitted signals are collected to form an image. The main information is carried by the 'secondary electrons', which are produced by inelastic collisions of incident primary electrons with the sample [4]. These low energy secondary electrons provide information about the topography or surface structure of the sample, since they are ejected from the orbitals of the specimen atoms, within shallow depth (< 2 nm) of the material surface [5].

Traditionally, SEM is used to image conductive surfaces under high vacuum conditions. Alternatively, when imaging wet and insulating materials, the so-called 'environmental' (or 'low-vacuum') SEM can be used [4]. In contrast with conventional SEM, low-vacuum SEM can maintain a gaseous environment around the sample and insulators do not need to be coated with a conductive layer, with only a small loss in image quality [4]. The capability of imaging non-conductive surfaces has opened the possibility of using SBFSEM to reconstruct 3-D structures. In SBFSEM, the 3-D set of data is acquired by automated serial sectioning combined with block-face imaging of the sample, inside a chamber of a low-vacuum SEM [6].

Imaging electrical trees using SEM

SEM has been used previously for exploring electrical trees. The specimen is cut with a microtome typically along a tree branch for exploration of its interior surface [7-10]. Alternatively, cross-sections of the tree can be cut to obtain images of tree channels [11]. However, we are not aware that SEM has been used to reconstruct 3-D models of electrical trees. In the following section, the use of SBFSEM is presented for 3-D imaging of electrical trees in polymeric insulation.

II. METHODOLOGY

Two samples previously explored using X-ray computed tomography [1] were examined using SBFSEM. A Gatan 3View® system was used to perform the automated sectioning process. The 3View® was housed within an FEI Quanta FEG 250 instrument, which has a field emission gun and a variable pressure chamber. The microscope was operated at low vacuum using water vapor to reduce problems with sample

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charging. In this manner, the sample is microtomed within the SEM, providing sequential surface images through the material. Result of this procedure was a stack of images containing cross-sections of the tree. The images were then processed and segmented, which is the process through which the feature is virtually created, via labeling voxels (volumetric pixel).

A. Sample 1: Bush-type electrical tree

An electrical tree was created in a point-to-plane geometry as described in [3] and as can be seen in [12], where the results of 3-D reconstruction using XCT can be found. For the SBFSEM, the needle was removed and the sample microtomed to be prepared for the process. For the scan, the accelerating voltage was set to 10 kV and the pressure 120 Pa. The automated slicing-imaging process was performed starting from the tree tip to the initiation point of the tree (where the needle tip was originally located), with a cut thickness of 0.2 μm between slices. The dimensions of each image (slice) were 4000 px by 4000 px with a pixel size of 200 nm. However, due to technical problems related to sections been drawn into the final aperture and distorting the beam, parts of the data were corrupted and the information of those sections was lost.

B. Sample 2: Branch-type electrical tree

The sample was prepared employing a similar procedure as for Sample 1. Details about sample preparation and feature creation are described in [1], and results showing the previous 3-D reconstruction using XCT can be seen in [12]. The operating conditions for this scan were set to an accelerating voltage of 5 kV and a pressure of 87 Pa. These operating conditions are slightly different from Sample 1, adjusted to obtain the best images in this scan. The same procedure of cutting from the tree tips towards the initiation point of the tree was followed for this sample, however, a cut thickness of 150 nm was deployed. The dimensions of the images acquired were 2500 px by 2500 px, with a pixel size of 147 nm. The tree in Sample 2 was smaller than the tree in Sample 1, therefore, the scanning area was set smaller accordingly. Also, the cut thickness was chosen similar in value than the pixel size, which is normally desired for model reconstruction. Similar technical problems as with Sample 1 were experienced in this case, resulting in data loss of a portion of the data. As a result, the 3-D set of data was divided in two parts and therefore, the tree could not be entirely reconstructed.

III. RESULTS AND DISCUSSION

A. Sample 1: Bush-type electrical tree

Two separated portions of the electrical tree were successfully imaged. The first part consisted in 1273 cross-sections of the tree, equivalent to around 255 μm from the tree tips down to the tree. The second part was composed of 1567 slices (around 313 μm), separated by 34 μm from the first part. This second part did not include the initiation region of the tree, because the data loss previously explained. Before image segmentation, the original set of images was cropped preserving the region of interest, normalised for pixel intensity and filtered. For the first part, the filters applied were Smoothing-Edge Preserving and Smoothing Non-Local Means

available in Avizo processing image software (VSG, Burlington MA). The second part of data was filtered using the Gaussian Blur 3D filter in ImageJ software and the Smoothing Edge Preserving filter in Avizo. As an example, one post-process slice, located 156.8 μm away from the bottom slice, is shown in Figure 1 (left), whereas an animation showing all the slices in first and second part are available to view in [12]. The 3-D model was completed using Avizo software. The results of the models of the two sections of Sample 1 are shown in Figure 2, and a virtual animation of them can be seen in [12].

Electrical tree characteristics were quantified for both reconstructed parts. The results are shown in Table 1. The mean diameter of the tree channels was calculated using the local thickness plugin in ImageJ software. Tree volume and the convex hull volume were calculated using Avizo. Convex hull is the smallest convex polyhedral surface that contains all the points of the feature [1]. Thus, the proportion of tree volume degraded was calculated as the ratio between volume and convex hull volume of the tree.

Figure 1. Processed SEM images. Left: Slice from second part of Sample 1. Right: Slice from second part of Sample 2. Scale bars: 30 μm .

Additionally, the quantification of tree characteristics was carried out along the cross-sections of the tree. The number of electrical tree channels from the bottom slice of the second part (closer to the initiation point of the tree) to the top slice of the first part (tree tips) is shown in Figure 3 (top). The channel area and 2-D convex hull area in each slice are also shown in Figure 3 (bottom). All of these quantities were calculated using Matlab 2012a. From the proportion of area degraded (ratio between the channel area), a trend is evident, though irregularities are seen in the number of channels after the gap.

B. Sample 2: Branch-type electrical tree

The data collected comprised of two sections separated by approximately 22 μm . Section one consisted in 134 μm (892 slices) from the tree tip. The second section was 33 μm thick, corresponding to 221 slices. The images were cropped leaving only the region of interest (where the channels were present),

Table 1. Quantification of electrical trees.

	Sample 1		Sample 2	
	Part 1	Part 2	Part 1	Part 2
Number of slices	1273	1567	892	221
Mean diameter tree channels [μm]	3.3	4.5	1.3	1.9
Standard deviation [μm]	1.5	2.6	0.5	0.9
Tree volume [μm^3]	4.51×10^5	3.94×10^6	3.07×10^3	2.56×10^3
Tree convex hull volume [μm^3]	7.41×10^7	1.09×10^8	7.18×10^5	1.27×10^5
Proportion of volume degraded ⁽¹⁾	0.6%	3.6%	0.4%	2.0%

⁽¹⁾ Ratio between volume and convex hull volume

Figure 2. Sample 1 - First part (top) and second part (bottom) of the 3-D rendering using SS-SEM. Scale bar: 100 μm .

and then filtered using the Smoothing Non-Local Means filter in Avizo. One slice of the second section (located 19.8 μm away from the bottom slice) is shown in Figure 1 (right), while all the slices from both parts of the data can be seen in an animation in [12].

The electrical tree feature (composed of the two separated parts) was segmented using Avizo, for further analysis and volume rendering. The 3-D model created is shown in Figure 4 and animations of axial rotation can be viewed in [12].

Similar quantification procedure as for Sample 1 was followed for Sample 2, and it is also summarised in Table 1. Again, additional electrical tree characteristics were calculated along cross-sectional slices of the tree, aligned perpendicularly to the axis of where the needle was. The number of electrical tree channels as a function of the distance from the bottom slice (near to the initiation point) is shown in Figure 5 (top). The channel area and convex hull area for each slice are depicted in Figure 5 (bottom), whereas the proportion of area degraded is represented at the top (dashed line) of the figure. The scattering of the data in the figure is mainly attributed to fewer features to count and the issue of disconnected channels in the model created as is shown in Figure 4. It is not clear why some features seemed to finish and later appeared again in the slicing-imaging exercise, yielding to the disconnected branches.

Some technical problems were encountered during the experiment. Sample charging was experienced in spite of the water environment of the low vacuum SEM. The situation was solved by normalising the pixel intensity of the images afterwards. In addition, some images presented scratches

Figure 3. Cross-section analysis of Sample 1. Top: Continuous line, number of electrical tree channels. Dashed line, proportion of area degraded. Bottom: Continuous line, total cross-sectional area covered by tree channels. Dashed line, convex hull cross-sectional area of tree channels. Blue vertical line corresponds to the slice in Figure 1 (left), at 156.8 μm .

produced by the diamond knife in the slicing procedure, which was manually corrected in the image segmentation.

Figure 4. Sample 2 - First part (top) and second part (bottom) of the 3-D rendering using SS-SEM. Scale bars: 10 μm .

Figure 5. Cross-section analysis of Sample 2. Top: Continuous line, number of electrical tree channels; dashed line, proportion of area degraded. Bottom: Continuous line, total cross-sectional area covered by tree channels; dashed line, convex hull cross-sectional area of tree channels. Blue vertical line corresponds to the slice in Figure 1 (right), at 19.8 μm .

SEM images of Sample 2 were composed of black and white features, which was not the case of Sample 1, where only white features were observed (see Figure 1). This may be attributed to the nature of the electrical tree channel: white colour for non-conductive channels and black for more conductive channels.

Samples 1 and 2 were made of different polymers, stressed and then scanned under different conditions. Considering both parts of the two samples, a noticeable difference in the diameter of tree channels is observed (Sample 1: 4.4 μm , Sample 2: 1.5 μm), which might be related to the difference of polymers and that Sample 1 was under stress for much longer period of time. These values corresponded to the values obtained using XCT [1], and were in the order of the normally reported. The proportion of volume degraded in Sample 1 was 2.4%, which is greater than the 0.7% obtained for Sample 2, as expected when comparing a bush-type against a branch-type electrical tree. Local properties at a given cross-section can be compared too. Arbitrarily considering the slice that is 100 μm from the bottom slice, the number of tree channels in Sample 1 was 525 while in Sample 2 was 15. Moreover, the area covered by the channels was 15000 μm^2 compared to 37 μm^2 in Sample 2.

IV. CONCLUSIONS

Virtual replicas of electrical trees have been created using SBFSEM. Through the model, tree characteristics, such as the diameter of tree channels, tree volume and net density of damage, amongst others have been quantified. A bush tree and

branch tree have been characterised by 'density of damage' (5.2% and 1.4%, respectively) and 'channels per cross-sectional area' (1230 channels/ mm^2 and 30 channels/ mm^2 , respectively), values calculated from the slices located 100 μm away from the bottom slice.

The technique provides good spatial resolution of around 0.2 μm . SBFSEM allows any insulating material to be scanned without the need of metallic coating of the surface. However, the automatic imaging-slicing process has limitations. When the process is set for thousands of slices, it was experienced that slices were drawn into the final aperture, distorting the beam resulting in loss of sections of the image.

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