STEM-EDS of Inception Sites of Vented Trees in a HV XLPE Subsea Cable

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ABSTRACT

High voltage XLPE cable cores have been subjected to wet ageing following the recommendations in CIGRE TB 722. The electrical ageing was performed at 500 Hz and 10 kV/mm. Vented water trees were detected initiating and growing from the semi-conductive screens. The inception sites of the trees were cut by cryo-microtomy and studied using scanning transmission electron microscopy with energy dispersive X-ray spectroscopy (STEM-EDS). Inorganic impurities were detected on both sides of the smooth XLPE-semiconductive screen interface close to the inception site.

KEYWORDS

High voltage XLPE cables, Vented water trees, STEM-EDS, Inception site

INTRODUCTION

The transition from medium voltage (MV) 36 kV inter-array subsea power cables to high voltage (HV) 72.5 kV is crucial to facilitate the increase in production of renewable offshore wind energy. This voltage level is expected to further increase to 145 kV [1]. Future designs of HV subsea cables for inter-arrays also include semi-wet or wet designs where water molecules are allowed to diffuse into the insulation systems.

The primary aging mechanism of wet-design cross-linked polyethylene (XLPE) cables is water treeing. This is a slow process which over the course of several decades can eventually result in service failure [2]. The pre-requisite for water treeing is an alternating electric field and a water electrolyte. Water trees consist of strings of micro-voids filled with water and there are likely paths or channels between the voids which facilitate transport of dissolved ions. The physical and chemical changes in the polymeric material associated with water treeing are permanent, as re-wetting of dried water trees restores their appearance [2, 3]

Significant advancements have been achieved in the cleanliness of cable materials, as well as in the manufacturing and handling processes of subsea cables. In particular, development of compounds resulting in a smooth interface between the insulation and semi-conductive screens has been important to prevent increased electrical field at the XLPE-semiconductive screen interface. This is crucial, as voltage stress enhancements could also promote water treeing [2]. Vented water trees are initiated at the semi-conductive screens and are considered more critical as they do not stop growing during service. Less critical are the bow-tie water trees initiated from small contaminants within the insulation, which stop growing after some time. [5, 6]

Impurities can originate from several sources, such as the carbon black used as a filler material in the semi-conductive screens, residues from the polymerization, antioxidants, and the surrounding environment [6, 7]. It is important to determine which impurities are causing inception of vented water trees, and from this how they can be inhibited. Research performed more than 20 years ago has paid much attention to the composition of foreign particles within the treed regions and on the insulation-screen interface [8]. By internal reflection Fourier transform infrared spectroscopy (FTIR-IRS), the presence of K, Ca, Mg and Si, amongst others, has been detected in the water tree. At the tree initiation sites, Si, O and S have been observed [9]. Other techniques, including micro-particle induced X-ray emission (micro-PIXE) and ionic chromatography, have detected organic and inorganic impurities such as sulphate, oxalate and acetate in resin pellets used for the semi-conductive screens [10].

The semi-conductive screens consist of carbon black particles uniformly dispersed in a polymer matrix, typically composed of ethylene-vinyl acetate (EVA) or ethylene acrylates [11, 12]. The polar nature of the ethylene copolymers gives rise to distinctive interactions with water molecules and ionic impurities present within the matrix. The polar groups in the polymer matrix enables hydrogen bonding with water molecules, thereby facilitating diffusion. Conversely, interaction between the polymer matrix and ionic impurities can impede diffusion, leading to comparatively slower diffusion rates than for water at the same conditions [13].

The main objective of this paper is to investigate the mechanism for vented water tree initiation in high voltage subsea power cables. In particular, vented water trees growing from an apparently smooth and contamination free interface between the insulation and semi-conductive screens normally observed in high voltage cables have been studied. The analysis includes cryo-microtomy of sections close to the XLPE-semiconductive screen interface which are characterized by scanning transmission electron microscopy with energy dispersive X-ray spectroscopy (STEM-EDS).

EXPERIMENTAL

Test object/pre-conditioning/ageing

An extruded 52 kV XLPE high voltage subsea cable with a conductor cross section of 400 mm^2 was aged in the laboratory according to CIGRE TB 722 [14]. The test conditions are shown in Table 1. The cable core was soaked in salt water (3.5 wt. % NaCl) at 55 °C. The wet pre-conditioning time was more than 1000 hours to ensure a water content of at least 95 % RH across the insulation wall prior to electrical ageing [14]. The ageing of the cable was carried out at 40 °C in the same salt water solution as used during pre-conditioning. The magnitude and frequency of the electrical field were 10 kV/mm taken at the conductor screen and 500 Hz, respectively. The test duration was 3000 hours (4 months).

Table 1: Conditions during pre-conditioning and ageing of the cable.

Test sequence	Temp [°C]	Ageing stress[1] [kV/mm]	Frequency [Hz]	Time [h]
Pre-conditioning	55	NA	NA	1008
Ageing	40	10	500	3000

[1]: At conductor screen

After ageing, 1-meter lengths were randomly cut from the cable. The metallic cable conductor was removed from these lengths and the insulation system was helically spiralized with a 2 mm thickness. The helically spiralized sections were put in containers filled with de-ionized water and placed in heating chambers at 90 °C. After a minimum of 10 days the samples were removed and immersed in de-ionized water at 5 °C to supersaturate water within the water trees. The trees became milky-white and possible to detect without using chemical staining methods.

Characterization

A 10 cm section of the spiralized cable insulation was investigated using a Zeiss AXIO Scope.A1 optical microscope to locate vented water trees initiated from the semi-conductive screens. The sections were pre-cut to a known distance from the water tree using a MT 990 motorized microtome from RMC Boeckeler with a steel knife. The block face was then trimmed around the water tree. The bulk sample was cooled to –120 °C and cut into electron transparent specimens (200 nm nominal thickness) with a glass knife using the Leica EM UC6/FC6 cryo-ultramicrotome. The electron transparent specimen is illustrated in Figure 1.

The microstructure and morphology of the bulk sample after cryo-microtomy were investigated by scanning electron microscopy (SEM) using the secondary electron detector on a Hitachi S3400-N, operated at 15 kV.

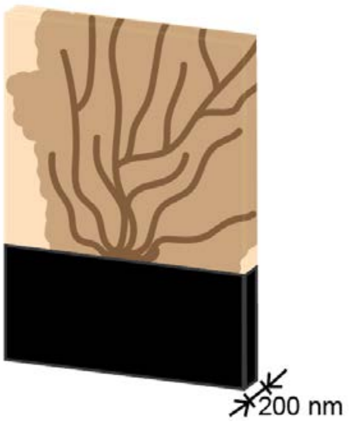

Figure 1: An illustration of the electron-transparent specimen prepared by cryo-microtomy.

The characterization was performed in low vacuum to prevent charging. This was done as an alternative to coating, which could mask the unknown contrast signatures at the inception site and its surrounding area.

Transmission electron microscopy (TEM) was performed with a double spherical aberration corrected cold FEG JEOL ARM 200FC, operated at 200 kV. Energy dispersive X-ray spectroscopy (EDS) was done with a large solid angle Centurio silicon drift detector (covering a solid angle of 0.98 sr) in scanning TEM (STEM) mode. The characterization was performed close to the inception site of a representative water tree. A double folded Cu TEM grid was used to clamp and support the TEM specimen during characterization.

RESULTS AND DISCUSSION

After the accelerated wet ageing, 10 vented water trees were detected in a 10 cm section of the insulation system. The distribution of water tree length and width for the detected trees is presented in Figure 2. The lengths range from 240 to 750 µm and there is no apparent correlation between length and width. It is likely that several water trees remain undetected because they were too small to be observed.

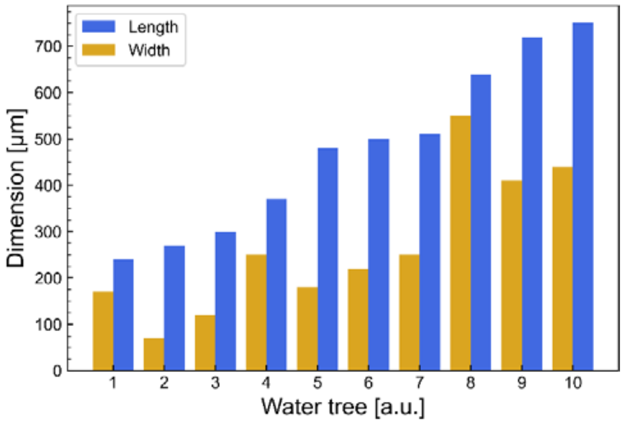

Figure 2: The maximum length and width of ten vented water trees observed in the analyzed cable section.

An optical microscopy image of a typical vented water tree is presented in Figure 3a, where the contrast is caused by supersaturation of the water tree. A smooth interface between the XLPE and semi-conductive screen was observed by low vacuum scanning electron microscopy (LV-SEM) examinations at the inception site of the water tree. No apparent defects or contaminants were observed either located on the surface of the sample/materials nor at the interface.

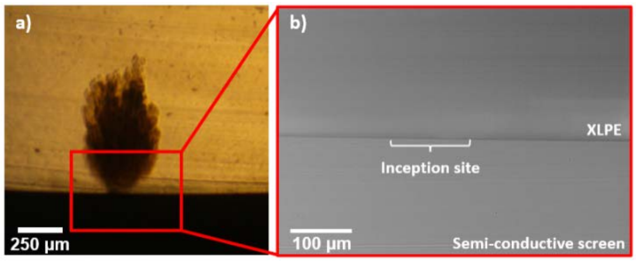

Figure 3: A vented water tree observed by (a) optical microscopy and (b) a LV-SEM micrograph of the smooth interface where the water tree was observed.

Given the absence of microstructural defects at the inception sites of the water trees, the work was focused on the chemical nature of the area close to the water tree inception site.

Analysis of vented water tree region

The specimen chosen for STEM-EDS exhibited a homogeneous microstructure across the XLPE material. However, impurity elements were detected within the water tree region approximately 10 µm from the interface between the XLPE insulation and the semiconductive screen. This is shown by the high angle annular dark field (HAADF) STEM image in Figure 4. The EDS spectrum of the analyzed area shows that O, Si, S, and Ca are present, and B is observed as a weak shoulder on the C peak. The intense C peak has been cut to better visualize the other elements.

The element maps show that regions rich in O also contain Si, indicating that impurities such as SiO_2 or organic Si-O are present in the XLPE insulation. A shell structure of Si is observed in regions with locally increased concentration of Ca at the core. Combined with the O-rich areas and a small amount of evenly distributed O it is expected that $CaSO_4$ is present. CaS is a likely impurity in regions where only S and Ca are observed. The $CaSO_4$ could be due to sulfur-containing antioxidants used in the insulation or from the carbon black. Ca may originate from the extrusion of the insulation system, where calcium stearate can be used as a lubricant [15].

Analysis of semi-conductive screen

The microstructure of the semi-conductive screen appeared homogeneous with a small concentration of Si and O throughout, with the exception of one particle observed 40 µm from the XLPE interface. A HAADF STEM EDS mapping of the particle presented in Figure 5 clearly confirms a high local concentration of Ca and Na. The anionic counterpart to these cations remains unknown but it is suggested that they exist as sodium and calcium acrylate as the resin was produced with ethylene acrylates. O and Si are evenly dispersed through the semi-conductive screen, likely due to additives introduced to the polymer matrix before extrusion.

Several of the ionic impurities related to water treeing in XLPE described in the literature have been detected in this work. However, the compounds observed here are hardly soluble in water and it is therefore challenging to see how they can migrate through the semi-conductive screens and to the insulation-screen interface. More soluble and mobile ionic impurities detected in previous work, such as K and Cl, were not observed.

Because the spiralized samples were kept in de-ionized water after wet ageing, it is plausible that the soluble and most mobile ions have diffused out of the sample. Analyzing the de-ionized water after storage could provide evidence to support this hypothesis.

Generally, ionic impurities from soluble compounds are expected to diffuse uniformly in the bulk of the polymer controlled by concentration gradients. This would result in an even distribution of impurities, and thus inception of water trees, along the XLPE interface. This is not observed, and it is therefore assumed there could exist a preferential path for diffusion of ions creating the inception site. Further, the preconditioning procedure used in [14] is based on the diffusion rate of water vapor within the polymer matrix. Impurity elements with comparatively slower diffusion

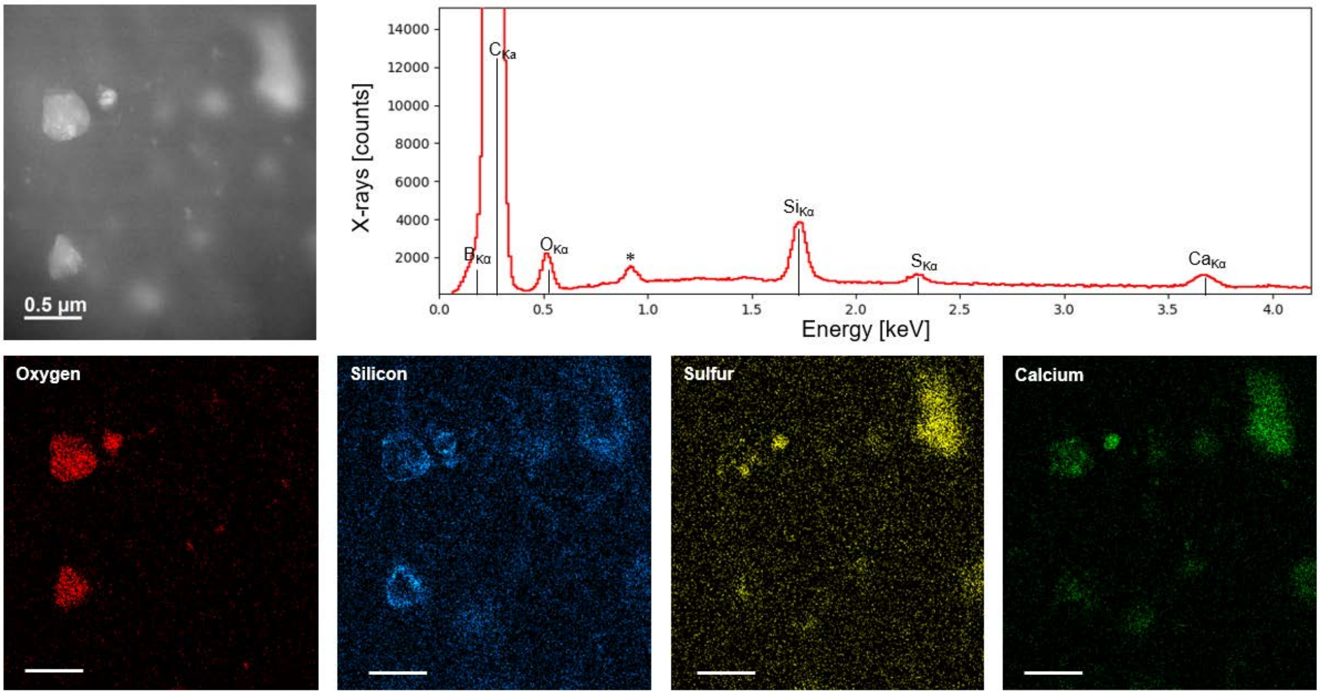

Figure 4: HAADF STEM image of the analyzed area in the insulator with the corresponding EDS spectrum and O, Si, S and Ca element maps. *Cu$_{L\alpha}$ stray signal from the TEM grid.

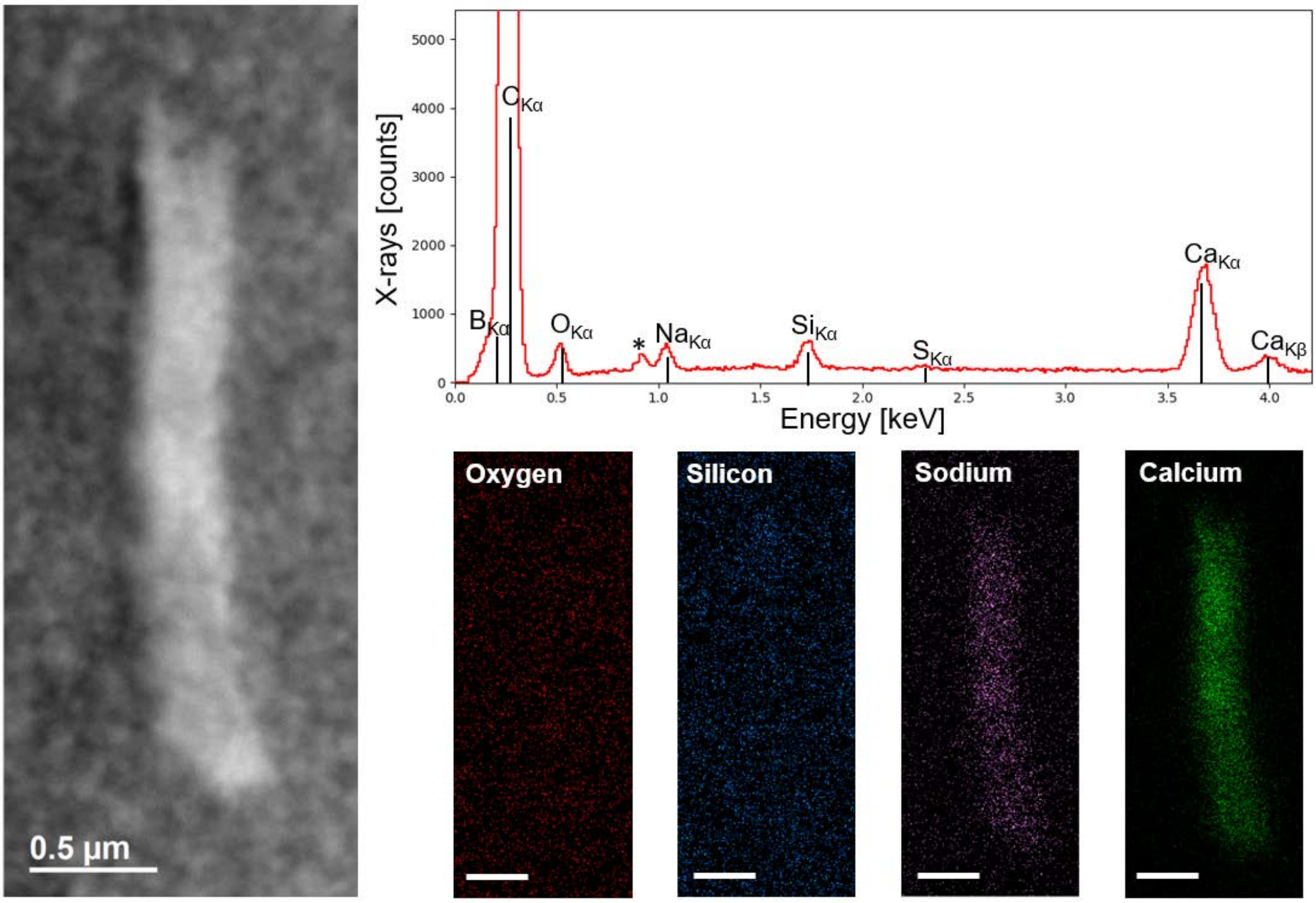

Figure 5: HAADF STEM image of the analyzed area in the semi-conductive screen with the corresponding EDS spectrum and O, Si, Na and Ca element maps (40 µm from the interface). *$Cu_{L\alpha}$ stray signal from the TEM grid.

kinetics may therefore not reach the interface during the required preconditioning. This may lead to overestimation of the insulation system's lifetime.

To establish the relation between the observed impurities and water treeing more work is needed. This will include STEM-EDS of more samples, both aged and unaged, as well as an investigation of the distribution of the impurities.

CONCLUSION

A methodology to characterize vented water trees and the area close to their inception site in the semi-conductive screens by STEM-EDS has been established, along with a procedure for sample preparation. The work clearly confirms the presence of inorganic impurities in an area close to the water tree inception site, many of which have previously been associated to the inception and growth of water trees. With further characterization, the compounds can be identified and migration of these be studied in the polymeric material of the specific system. This can provide more knowledge about the initiation of water trees and how they can be inhibited to further improve the longevity of XLPE subsea cables.

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GLOSSARY

EVA: Ethylene-vinyl acetate

HAADF: High angle annular dark field

HV: High voltage

LV-SEM: Low vacuum scanning electron microscopy

Micro-PIXE: Micro-particle induced X-ray emission

MV: Medium voltage

PE: Polyethylene

STEM-EDS: Scanning transmission electron microscopy with energy dispersive X-ray spectroscopy

TEM: Transmission electron microscopy

XLPE: Cross-linked polyethylene