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<<http://dx.doi.org/10.1109/CEIDP.2000.885246>>



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**Original citation:** Dissado, L. A., Fothergill, J., Seeley, A., Stevens, G. C., Markey, L., Laurent, C., Teyssedre, G., Nilsson, U. H., Platbrood, G. & Montanari, G. C. (2000). Characterizing HV XLPE cables by electrical, chemical and microstructural measurements on cable peeling: Effects of surface roughness, thermal treatment and peeling location. *Electrical Insulation and Dielectric Phenomena*, 2000, Annual Report, 1, 136 - 140. doi: 10.1109/CEIDP.2000.885246  
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# Characterizing HV XLPE cables by electrical, chemical and microstructural measurements on cable peeling: effects of surface roughness, thermal treatment and peeling location

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## Abstract

Characterization of the electrical, chemical, and microstructural properties of high voltage cables was the first step of the European project "ARTEMIS", which has the aim of investigating degradation processes and constructing aging models for the diagnosis of cross-linked polyethylene (XLPE) cables. Cables produced by two different manufacturers were subjected to a large number of electrical, micro-structural, and chemical characterizations, using cable peelings, instead of lengths of whole cables, as specimens for the measurements. Here the effect of surface deformation and roughness due to peeling and the relevance and significance of thermal pre-treatment prior to electrical and other measurements is discussed. Special emphasis is put on space charge, conduction current and luminescence measurements. We also consider the dependence of these properties on the spatial position of the specimen within the cable. It is shown that even though the two faces of the cable peel specimens have different roughness, the low-field electrical properties seem quite insensitive to surface roughness, while significant differences are detectable at high fields. Thermal pre-treatment is required to stabilize the insulating material to enable us to obtain reproducible results and reliable inter-comparisons throughout the whole project. The spatial position of the specimens along the cable radius can also have a non-negligible influence on the measured properties, due to differential microstructure and chemical composition.

## Introduction

The two HV cables investigated were insulated by XLPE produced using extra-clean materials and processes. The insulation thickness was 14 mm. The project started with the initial characterization of cable insulation, in order to constitute the reference for electrical, micro-structural, and chemical properties needed to single out aging markers on the basis of the property evolution with aging time. The first problems that were faced were the choice of the specimens to be used and the kind of pre-treatment necessary in order to have reproducible results. The choice made for the specimens posed a very delicate problem. Most of the measurements could not be carried out (or would have been much more expensive) on full-size cables. The use of small-size model cables does not seem to solve the problem, since they could not be realized with the same manufacturing conditions as full-size cables, and thus could be unrepresentative of the real cable behavior. For the same reason, use of insulation plaques by compres-

sion-molding was discarded. Therefore, the basic characterization of the cable insulation was undertaken using films peeled from cables, having an average width of 8 mm and a thickness of 150  $\mu\text{m}$ . Only a few measurements (particularly of space charges) were performed on full-size cable samples. Due to the cutting tool and procedure, the surfaces of the films were not well smoothed (as for press-molded specimens), and one of the faces was considerably rougher than the other. Thus, extensive investigations of the effect of the surface quality on the electrical properties were carried out and are summarized in this paper. The effect of the radial position of the specimen in the insulation on the electrical properties, is also evaluated. The decision to subject specimens to a thermal treatment before any test contributing to the initial characterization was a compromise. It is clear that cross-linking by-products will not completely leave the cable during aging and thus could contribute to degradation processes. On the other hand, it was necessary to set up a description of the unaged material which would not change with storage time and would be repeatable and reproducible for each specimen taken from different lengths of the same cable (at a given distance from the inner or outer semiconductor screen). By-product concentration was shown to vary as a function of time and insulation radius [1]. The choice of the thermal treatment procedure and the variation of electrical properties as function of treatment are discussed in the following. For the purpose of this paper data taken from different cables are reported, without referring to the relevant manufacturer, since the results for the different cables involved in the project did not in general vary significantly.

## Experimental procedures

The peelings were cut from the cables using a lathe equipped with a specially designed knife to get optimum surface smoothness. From a pre-screening study of the field distribution it was decided to focus the investigation on the region from 2 to 4 mm along the radius, starting from the inner semiconductor. This also fits in with the subsequent determination of the distribution of morphological characteristics and of polar species arising from the migration of low molecular weight products from the semicon and of by-products of the cross-linking reaction. Before any tests were carried out to establish the basic electrical characterization of unaged insulation, specimens were thermally treated as described in the following section. The electrical properties described here are those associated with space charge, charging-

discharging current, and electroluminescence (EL) measurements.

Space charge and charging-discharging current measurements were performed at different values of constant DC fields for times ranging between 3000 s and 10000 s for space charge and about 24 h for charging current measurements. After poling, specimens were grounded (depolarization, volt-off). Space charge measurements were performed at normal atmospheric pressure, while charging-discharging current measurements were carried out under pressure of 3 bar, in nitrogen gas. Both measurements were carried out at different values of constant temperature, ranging between 20 and 80°C. In most of the space charge measurements the electrodes of the PEA system, i.e. semicon and aluminum, were applied directly to the specimens. However, PEA measurements were also performed with gold-coated electrodes (obtained by cold sputtering). The same electrodes (but with an additional guard ring) were used for all the charging-discharging current measurements [2].

Electroluminescence (EL) refers here to light emitted intrinsically by the material under the influence of an electric field (excluding gaseous discharges). It is an important phenomenon to monitor as its onset might define a threshold for space charge formation, and possibly also for chemical degradation [3]. EL tests were performed at room temperature, in a vacuum of  $10^{-7}$  mb, on specimens with semi-transparent gold-coated electrodes. In these tests, the voltage (AC and DC) was increased in steps of a few hundred volts (depending on the experiment) each step lasting 300 s. Detection of the EL on each step was performed by means of a photomultiplier working in photon counting mode, and the emission spectrum obtained using a monochromator coupled with a liquid-nitrogen cooled CCD camera. Two other forms of luminescence were analyzed to identify the chemicals responsible for EL and also their excitation mechanisms. One of these was excited using a non-electrical stimulation (photoluminescence), the other was stimulated in experimental conditions suitable to excite luminescence upon charge recombination of trapped carriers (charge-recombination induced luminescence, thermally stimulated or not).

## Results and Discussion

### *General observations on peel quality*

The two surfaces of the specimens taken from peeled rolls of cables have different surface roughness. Measurements using an atomic force microscope showed that the RMS roughness on the shiny face was typically 150nm, whilst that on the other face was 350 nm. The pictures reported in Fig. 1 show the two faces of the curled film obtained from peeling. As can be seen, one face is considerably smoother than the other (this face corresponds to the concave surface of the film). This description does not change, neither qualitatively nor

quantitatively, as function of the insulation radius where the peel is taken.

In TEM observations of permanganically etched peels, surface deformation features (mainly ridges in the cutting direction) are observed. In Figure 2, the micrograph on the left shows the morphology of the undeformed material. It consists of lamellar domains of small size. The second micrograph taken on a peeled sample shows a deformation layer which has been partially etched away during the TEM preparation process. The peeling process therefore influences the surface microstructure of the material. The deformation layer could be removed by increasing the etching time by 20%. We estimate that the deformation layer is approximately 5 to 10  $\mu\text{m}$  thick. Lamellar micro domains with some V-shaped lamellae were revealed in the underlying microstructure. Micro-Raman spectroscopy reveals differences between the shiny and mat faces. By normalizing the spectra to the  $\text{CH}_2$  twist near  $1300\text{ cm}^{-1}$ , a significant increase (shiny/mat) is observed in the intensity of some bands, all of  $A_{1g}$  and/or  $B_{1g}$  symmetry. Given that the relative orientation between the exciting laser polarization and the sample axis remains constant, this indicates that the shiny face is more oriented than the mat one. A study of the influence of the orientation angle of the peelings with respect to the incoming laser polarization showed significant variations of the  $1440\text{ cm}^{-1}$   $\text{CH}_2$  bend,  $1415\text{ cm}^{-1}$  orthorhombic  $\text{CH}_2$  bend and  $1130\text{ cm}^{-1}$  C-C stretch, all of which are sensitive to the orientation of polymer chains. By using ATR-FTIR, it is possible to measure the surface amorphous fraction [4,5], and therefore examine the difference of crystallinity between the two faces. Results show that the surface amorphous content is slightly higher (by 1-2%) on the shiny surface than on the mat one. We expect this to be a result of the peeling process. We can also obtain crystallinity values from Raman spectra [6] and in a more usual way, from DSC measurements. Both methods show that the crystallinity of the peeled films is a few % higher (on average) than the crystallinity of the cable material itself. The peeling process therefore affects the surface crystallinity and chain orientation of the peel samples.

### *Thermal treatment*

The thermal treatment was carried out at a temperature of 50°C, which is below any significant crystallite melting, as revealed by differential scanning calorimetry (DSC) measurements. Measurement of the concentration of cross-linking by-products by FTIR and HPLC revealed that they were below the detection level of these techniques after two days at 50°C and ambient pressure: see Table 1. Thus, the time for thermal treatment was fixed at 48 h.

Substance / time	0 h	5 h	24 h
Cumyl alcohol	13350	2420	0
Acetophenone	3700	88	0
$\alpha$ -methylstyrene	116	0	0
Cumene	0	0	0

Table 1: Concentration (in weight-%) of cross-linking by-products as function of treatment time (50°C, ambient pressure) by HPLC. Also confirmed by FTIR.

The photoluminescence properties of XLPE materials are partly due to the by-products of the cross-linking reaction. This was confirmed by the evolution of the luminescence yield vs. thermal treatment. However, strong evidence for the persistence of spectral features associated with by-products, or derivatives chemically linked to the polymer, is found in the analyses of specimens treated for 48 h. This can be explained by the higher sensitivity of the luminescence technique as compared to those that have been used to quantify the residual by-product concentrations in the samples. Further out-gassing of the XLPE peelings does not induce a marked change in the photoluminescence spectrum, which indicates that the luminescent centers are probably linked chemically to the polymer. The thermal treatment adopted in the project, however, remains valid as regards the stability of the sample properties with respect to time. Measurements of space charge, charging current, electric strength and EL clearly emphasize the importance of the thermal treatment. Without treatment the above property values varied from specimen to specimen depending on the time elapsed between peeling and measurement (even when the specimens were kept in aluminum envelopes and/or under mild vacuum).

Space charge profiles of treated and untreated specimens are shown in Figure 3 at a poling field of 90 kV/mm. As can be seen, thermal treatment strongly reduces the amount of accumulated charge. Extension of the treatment to 96 hours does not, however, produce any further change. A longer treatment time is therefore unnecessary as very good reproducibility of data is reached after 48-hours. Figures 4 (a) and (b) show the EL vs. voltage characteristics for treated specimens submitted to AC and DC voltage, respectively. For AC stress, the characteristic has been recorded in a spectral window suitable to reject spurious light emission as described elsewhere [7]. Each data point on the graphs represents the averaged EL measured at each voltage step. Three tests were performed under AC, and it can be seen that there is a good reproducibility of the results both regarding the onset field above which EL is detected and the light intensity. Under DC, there is also a good definition of the EL onset field, and different tests give a good reproducibility of the data points. Several difficulties were noticed regarding the testing of untreated specimen. In the worst cases, i.e. in fresh samples containing a high concentration of by-products, strong instabilities of the EL

level were detected for a constant applied field, making it impossible to obtain a typical characteristic. For samples that have been exposed to the normal atmosphere for some days after peeling, it was possible to record the EL vs. voltage characteristic, but both the EL onset field and light magnitude were dependent on the time the sample had spent in the atmosphere. The thermal treatment was therefore a key procedure to stabilize the material properties.

#### *Specimen roughness*

The PEA technique was used in order to investigate the effect of the surface on electrical property measurements. Four different test configurations (able to take into account all the combinations of specimen surface & cell electrode) were considered and tests were performed at different field levels, voltage polarity and, with and without gold-coated electrodes. On the whole, only small differences between the different configurations were found at low fields (i.e. up to 30 to 40 kV/mm DC). Space charge profiles obtained at high field (e.g. 100 kV/mm), however did show some differences. Un-coated specimens in the configuration C i.e. with the smooth face in front of the upper, positive electrode were found to contain larger amounts of charge than was found in other configurations. Gold coated specimens in both configuration A (rough face in front of the upper positive electrode) and C increased the charge accumulated. These were found to be general results. Charging-discharging current measurements are even more sensitive to the test configuration than space charge measurements, as shown by Fig. 5 which compares the charging current versus time curves at 30kV/mm for configurations A and C. The latter gives rise to the larger current values. Being the worst case, particularly for un-coated specimens, configuration C was thus taken as the base reference case for all the electrical property measurements carried out for insulation characterization.

EL imaging was performed in order to check if the surface roughness influences the spatial distribution of the light emitted by the sample, through possible effects of field enhancement along the stripes of the surface. This was carried out by measuring the light distribution in the sample plane, i.e. the optical axis is perpendicular to the sample plane. Although the EL excited under AC voltage exhibited some localization around specific areas, there was no correlation between the emitting zones and the macroscopic roughness features of the surface. The non-homogeneous distribution of EL in the sample plane does not appear characteristic of the XLPE peeling. It is a general characteristic of EL excited by AC voltage on metallized polymer films.

On the basis of these results, it can be argued that the specimen surface roughness affects electrical properties only at medium to high fields, while it should not play a significant role at low fields, i.e. those close to the design stress. This supports the choice of peeled films for

insulation characterization and subsequent aging investigation.

#### Radial variation

Morphology as function of insulation thickness, i.e. of the distance from the inner semiconductor screen, is shown by the different DSC curves shown in Fig.6. The crystallinity is relatively constant ( $\sim 40\%$ ) across the cable but decreases near the inner semiconductor (at 2 mm a 2.3% and at 30 micron a 25% decrease). It is possible for the manufacturing process to influence the thermal history and its radial profile due to the thermal gradient. In the present case, however, the thermal history peak is situated at  $74.81^\circ\text{C}$  and no variation of this temperature has been observed as a function of the radial position of the peeling. An extensive spectroscopic mapping study of the distribution of chemical species along the insulation radius is reported elsewhere [1]. Here we summarize these results: Firstly, short chain acrylate species have been found to migrate from the semi-conductive screen co-polymeric material and can be observed in large concentration (up to  $45\text{mg}/\text{cm}^2$ ) close to the screens. In contrast, cross-linking by-products (acetophenone and cumyl alcohol) were found in larger concentration ( $\sim 8\text{mg}/\text{cm}^2$ ) in the bulk of the insulation. These by-products were hardly detectable by FTIR after thermal treatment. On the basis of these non-negligible changes of morphology and chemistry, some differences in the electrical property behavior was expected. Actually, space charge measurements showed a qualitative behavior similar to that mentioned above regarding the effect of surface roughness, that is, significant differences between space charge profiles and dynamics were detected only at quite high fields. As an example, Figure 7 reports the space charge profiles at  $90\text{ kV}/\text{mm}$  for gold-coated specimens taken from close to the inner and the outer semiconductor screens. The profile obtained in the external part of the cable reveals a larger charge than in the peels cut close to the inner semiconductor screen.

#### Conclusions

On the basis of the results presented here, it seems that the electrical characterization of the insulation of high-voltage cables and, thus, the investigation of thermo-electrical degradation can be carried out using films peeled from cables. Some constraints may, however, still affect the validity of peeled-film results as representative of the insulation materials. So while a thermal treatment is needed to stabilize the specimen and allow reproducible behavior over the timescale of the electrical property measurements, this may affect the degree to which the behavior of peeled specimens relates to that of the cable. The surface quality does not, however, play a noticeable role especially at fields close to those used for cable design and accelerated life tests (i.e. from  $10$  to  $30\text{ kV}/\text{mm}$  AC) but it will have an influence at higher fields. Given that the purpose of the project is the inves-

tigation of diagnostic properties and the comprehension of the degradation mechanisms, the use of peeled films with appropriate conditioning seems the best compromise between practicality, economic constraints and relevant representation of the cable.

#### Acknowledgements

This work was carried out under the ARTEMIS program EU Contract Number BRPR-CT98-0724. The authors thank the ARTEMIS publications committee for permission to publish.

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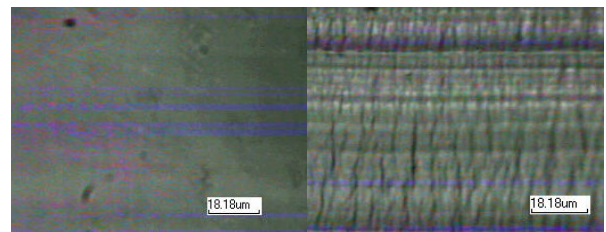


Fig. 1: Pictures of the two faces of the specimens used for electrical property measurements. Left: smooth face, corresponding to the concave surface of the curled film obtained from peeling, Right: rough face.

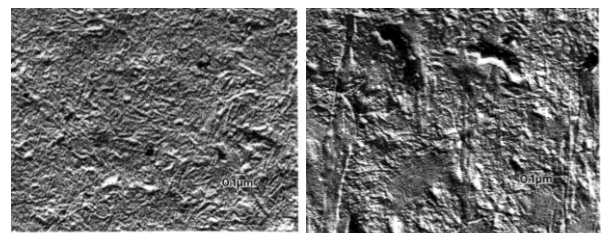


Fig. 2: TEM micrographs of permanganically etched cable material, undeformed (left), peels (right).

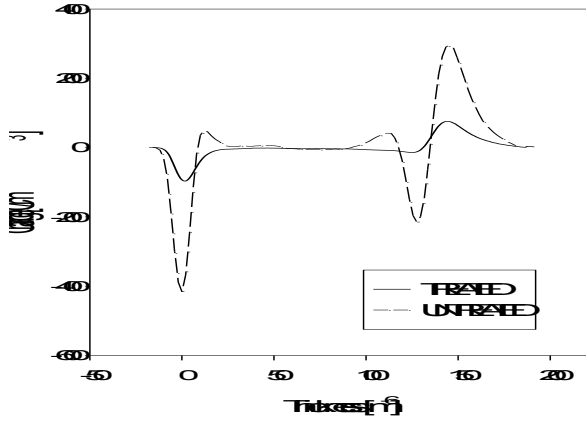


Fig. 3: Examples of space charge profiles at a poling field of 90 kV/mm for untreated (A) and treated (B) specimens (after 10000 s of poling), 20°C.

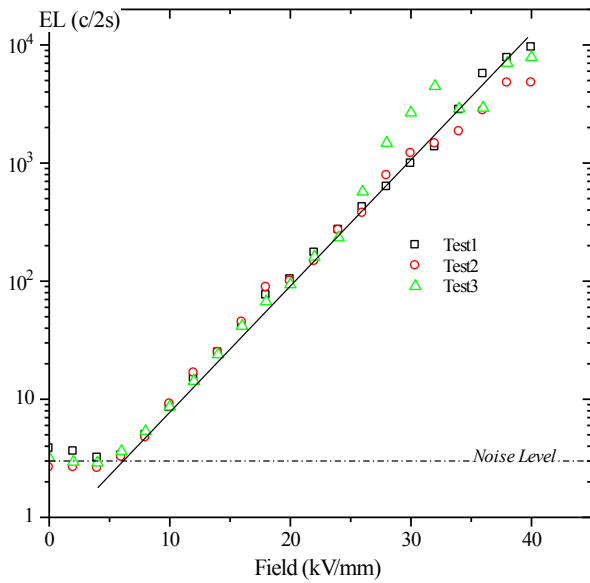


Fig. 4(a) EL vs. AC voltage for treated specimen

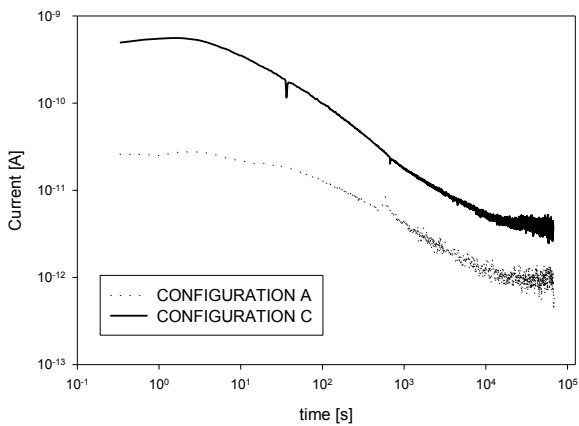


Fig. 5: Charging current as a function of time at 30kV/mm for configurations A and C

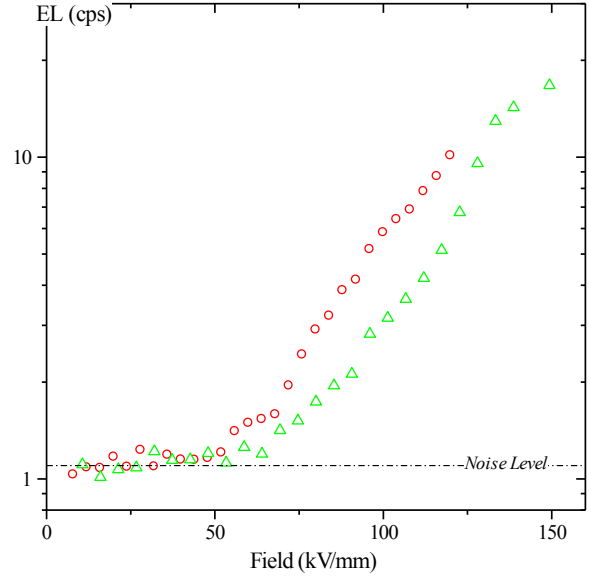


Fig 4(b) EL vs. DC voltage for treated specimen

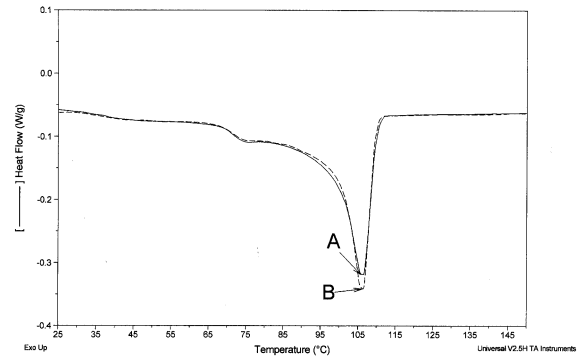


Fig. 6 DSC graphs for specimens taken from peels close (2 mm) to the inner semiconductor screen (A) and to the outer semiconductor screen (B)

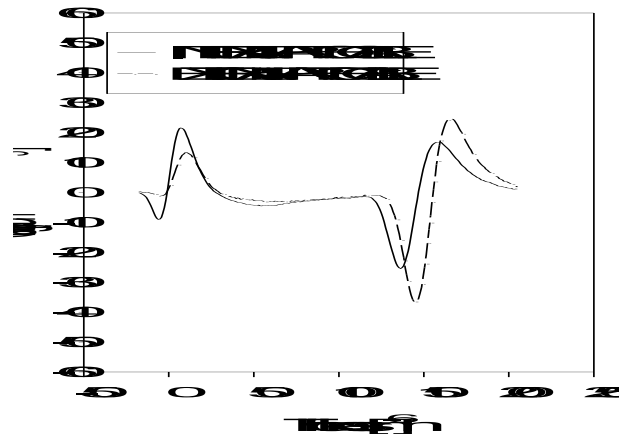


Fig.7: Space charge profiles at 90 kV/mm for gold-coated specimens taken from close to the inner (A) and the outer (B) semiconductor screen