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# Are Nano-composites really better DC Insulators? A study using silica nanoparticles in XLPE

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## ABSTRACT

A DC step test and a number of DC life endurance tests have been performed at a temperature of 20°C on 200 µm thick samples of a commercial (AC grade) XLPE and its nanocomposite which is intended for DC use. It was found that the breakdown strength of the nano-composite on the DC step test was considerably larger than that of the unfilled XLPE. However, the endurance tests showed that the difference between the characteristic lifetime of the nano-composite and its unfilled base polymer reduced as the applied field was reduced and the lifetimes became essentially the same when the applied field was 130 kV/mm, which is the lowest for which we have data at present. The life line was analyzed as an inverse power law and gave median life exponents of  $n = 10.76$  for the nano-composite and  $n = 13.58$  for the XLPE. These values are consistent with estimates from step tests on the same materials that have been recently published, and imply that the nano-composite will perform worse than the AC grade XLPE as a DC insulator if the inverse power law continues to be obeyed down to service fields.

Index Terms — XLPE, nano-XLPE, DC aging parameter, inverse power law

## 1 INTRODUCTION

A recent paper [1] presented a new method for analyzing the data of step-up voltage tests in order to determine the parameters of the assumed inverse power law (IPL) life ( $t_b$ ) expression:

$$t_b V^n = A \quad (1)$$

with  $A$  being a constant. Account was taken of both the dwell times and the ramp-up times and the value of  $n$  was obtained as the optimum from step tests with three different dwell times, rather in the way that it can be found from continuous ramps with different ramp rates [2]. The technique was applied in the Xi'an laboratory to DC tests of commercial samples of XLPE and a nano-composite of the same base XLPE intended for DC application, where it was found that the nano-composite had the bigger breakdown strengths, but surprisingly its IPL exponent  $n$  was lower for all three temperatures used. Since this clearly implies that the lifetime of the nano-composite will be less than the XLPE in endurance tests below some value of applied voltage (field), it was essential that the result be validated using endurance tests of longer duration than the step-tests.

Accordingly, we have carried out at Leicester a DC step-test similar to those reported in [1] together with DC endurance tests at three different voltages on 200 µm disc samples made from exactly the same materials as used in [1], together with a DC step-test similar to those reported in [1]. The latter test was partly carried out to demonstrate that the quality of the samples made in the two laboratories was comparable in the sense of yielding similar breakdown strengths. The higher breakdown strength of the nano-silica-XLPE vis-à-vis unfilled XLPE obtained in [1] was also verified, and together with the DC endurance tests values for the life exponent  $n$  were obtained that confirmed the lower value estimated for the nanosilica-XLPE vis-à-vis the unfilled XLPE reported in [1].

The results presented here for  $T = 20^\circ\text{C}$  therefore confirm the differences in breakdown strength and life exponent ( $n$ ) value between XLPE and nano-silica-XLPE deduced in [1]. Furthermore a value of field is found in which the life endurance of both materials cannot be distinguished. Although the results presented here are limited to nano-silica-XLPE with a given filler content and particle size, our aim is to initiate a wider discussion in the dielectric society and outline the necessity of

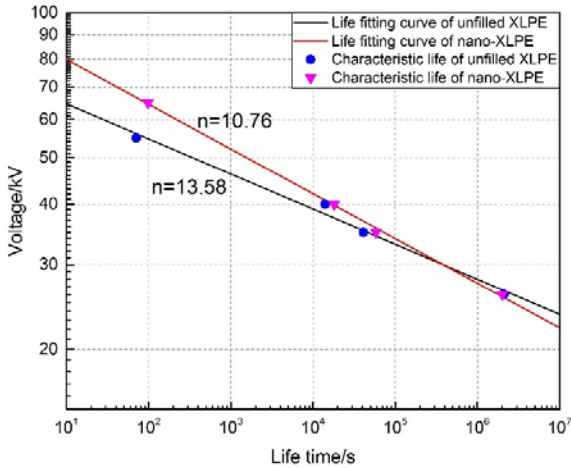
long-term low-voltage tests to verify the performance of nano-filled materials at service conditions.

## 2 EXPERIMENTAL DETAIL

Thin disc-shaped samples were made from pellets of a crosslinkable XLPE and nano-XLPE supplied by a commercial company. The XLPE was WANMA MACROMOLECULE 101B. The nanocomposites have the same antioxidant and crosslinking agent as the XLPE, but include a dispersion of 1wt% SiO<sub>2</sub> nano-filler surface treated for compatibility with XLPE with a size range between 50 nm and 100 nm. The pellets were placed in a mould and heated, first to 120°C for 20 min at 2 MPa pressure to melt the pellets and then to 180°C at 15 MPa to crosslink the polymer. The discs were water cooled to room temperature in 10 min. The thickness of each resulting disc was measured and found to be 200 μm ±5%. Before use the samples were degassed at 80°C for 48 hours. Step tests were carried out with the samples immersed in silicone oil using a protocol in which the first step was to a voltage of 20 kV with subsequent steps of 5 kV. The step-up time was 3 s and the dwell time on each step was 57 s. The time to failure was noted as well as the failure voltage. The sample diameter was 70 mm and the upper (brass) electrode diameter 25 mm; the number of samples tested was eleven for the XLPE and ten for the nano-XLPE. The endurance tests were also carried out in silicone oil at voltages of 40 kV, 35 kV, and 26 kV, with between eight and eleven samples per test.

## 3 RESULTS

Figure 1 shows the lifelines for the characteristic lifetime of both materials determined by fitting the measured times-to-failure to a Weibull distribution.



**Figure 1.** Voltage dependence of the characteristic DC lifetimes of XLPE and nano-filled XLPE

The corresponding Weibull parameters and their 95% confidence intervals (CI) are given in Table 1. The first row in Table 1 for both XLPE and nano-silica-XLPE (the Weibull parameter values for 55 kV and 65 kV respectively) refers to

the Weibull distribution obtained from the step-tests. The life tests at 26 kV had to be suspended after five nano-XLPE and four XLPE samples (out of eight) had failed. The characteristic lifetimes were evaluated by treating the data as a singly censored set [3], therefore the Weibull parameters at 26 kV have not been included in Table 1.

**Table 1.** Characteristic Weibull parameters obtained at each breakdown voltage level and their 95% confidence intervals (CI). Beta values for 55 and 65kV are reported for the step tests.

Samples	Breakdown Voltage (kV)	$\alpha$ (10 <sup>3</sup> s)	95% CI of $\alpha$ (10 <sup>3</sup> s)	$\beta$	95% CI of $\beta$
Unfilled-XLPE	55	0.473	0.437 0.511	8.14	5.30 12.50
	40	14.0	8.82 22.3	1.35	0.82 2.14
	35	41.2	19.9 85.2	0.96	0.69 1.54
Nano-XLPE	65	0.587	0.552 0.625	10.5	6.56 16.83
	40	18.1	10.5 31.3	1.35	0.82 2.22
	35	58.4	37.9 89.9	1.60	0.98 2.63

The plot also includes data from the step-test. Since this was only carried out for steps with a single dwell time an effective lifetime at the characteristic breakdown field has had to be estimated. This time has a lower bound given by the time before failure on the final step and an upper bound of the total time on the test up to failure. The two values together with the characteristic lives at 40 kV and 35 kV give a maximum and minimum value of exponent  $n$ . In this way, it was found that for unfilled XLPE at  $T = 20$  °C  $n$  lies between 10.6 and 16. Similarly it was found that for nanosilica-XLPE  $n$  lay between 8.7 and 12.3. The corresponding values in [1] are XLPE:  $n = 13$ ; nano-silica-XLPE:  $n = 8.8$ . A median value for  $n$  can be obtained by estimating the effective lifetime,  $t_{eff}$ , for the breakdown voltage  $V_{final}$  with allowance for the effect of all the previous voltage steps in the step-up test. In the spirit of the analysis of [1], equation (2) which is derived from equation (1) for a sequence of voltages,  $V_k$ , each of 60s duration is rewritten as equation (3) for  $t_{eff}$ ,

$$t_{eff}(V_{final})^n = t_{final}(V_{final})^n + 60 \sum_{k=1}^{k=final-1} V_k^n \quad (2)$$

$$t_{eff} = 60 \times \sum_{k=1}^{k=final-1} \left( \frac{V_k}{V_{final}} \right)^n + t_{final} \quad (3)$$

In this equation,  $t_{eff}$  is the effective life time under the step-up test, voltage,  $V_k$  is the voltage of the  $k$ th step,  $V_{final}$  is the breakdown voltage,  $n$  is the life index,  $t_{final}$  is the endurance time at  $V_{final}$ . Equation (3) is utilized in the following way. The range of values of  $n$  between the upper and lower bounds was divided into 10<sup>3</sup> equal steps and each value used to calculate an effective lifetime at  $V_{final}$  from (3). This was used together with the characteristic lifetimes at 40 kV and 35 kV to determine a value for  $n$ . Iteration allowed a value for  $t_{eff}$  to be obtained that gave a value of  $n$  that was consistent with that assumed in equation (3) to calculate  $t_{eff}$ . The technique of

mixing step testing with life testing in this way is useful for establishing the likely voltage levels required for life testing. Since the same (IPL) law is used for both, the results should be compatible. Further ageing tests are underway and will be reported in a full paper in due course.

## 4 DISCUSSION

In common with much previous work [4, 5] and particularly [1] on the same materials we find that the characteristic breakdown strength of the nano-silica -XLPE is greater than that of the base XLPE. However we also confirm the unique result of [1] that the life index  $n$  of the DC IPL life expression is smaller for the nano-silica -XLPE than for base XLPE for the same materials. Using both step and life tests at 20°C we find  $n = 13.6$  for XLPE and 10.8 for nano-silica -XLPE. These values are consistent with those of  $n = 13$  (XLPE) and  $n = 8.8$  (nano-silica -XLPE) obtained in [1] solely from an analysis of step-tests, particularly in the case of the base XLPE. Such values imply that at some value of the applied voltage the characteristic life of the nano-silica -XLPE should be the same as that of the XLPE. This we have observed to occur at a voltage of 26 kV (field =130 kV/mm). The further implication is that at lower fields, such as would occur in many service conditions, the lifetime of the nano-silica -XLPE will be shorter than that of the XLPE. This is clearly of importance for the putative application of nano-silica -filled XLPE as a DC insulator. Of course, we recognize that different ageing mechanisms may come into play at lower fields leading to deviations from the IPL behavior. It is possible that, at lower fields and longer times the ageing behavior of the two materials are becoming similar; longer tests will be required to characterize the behavior accurately. The results illustrate that the use of short-term tests should be treated with caution, especially where new materials are being introduced.

There are a few further points to be considered. In the first instance it could be argued that the XLPE and nano-silica -XLPE have similar characteristic lives in the life tests because of the ingress of silicone oil. While this cannot be disproved until samples are weighed regularly during the life test, it should be noted that the values of  $n$  that we obtain are close to those found on the step-tests in [1] whose maximum duration (600s at 20 °C for a 1 min dwell time) can be regarded as too short to allow significant oil penetration. We have also progressively measured the dielectric response of both XLPE and nano-silica -XLPE during the test at 40 kV and find that there is nothing that can be attributed to oil ingress bringing the two materials to similarity. We will report this in detail in a full paper once longer-term ageing tests are completed.

The implication that reducing the field below 130 kV/mm (voltage 26 kV) would result in a nano-silica -XLPE whose characteristic life would be shorter than that of XLPE relies on the assumption that the IPL continues to be obeyed to low fields or voltages, which is not necessarily the case [6] although it is often a good approximation [2]. Further work in hand on longer

term tests will go some way to resolving this issue. It will also help resolve the issue of silicone oil ingress since in that case it would be expected that both materials should have the same lifetimes at any field. It is interesting to note that the present results indicate that using the present XLPE cables for DC power transmission may not be a problem, although again further work is needed. What is very clear from the present work is that selecting nano-composites for DC applications based on their high DC breakdown strengths is not justified. Long term ageing tests are urgently required. The step-test analysis of [1] can be used only if their evaluation of the IPL and its exponent  $n$  is validated.

## 5. CONCLUSIONS

The present work confirms that the DC IPL life index  $n$  is smaller for 1wt% nano-silica - XLPE than for XLPE. As a result a field has been found for which the nano-silica -XLPE and XLPE have the same characteristic lifetimes, even though the breakdown strength of the nano-silica -XLPE is bigger than that of XLPE on a short term step-test. These results imply that long term tests are required to determine whether or not nano-composites will perform better than the base polymer as DC insulating materials.

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