Erosion Resistance of Nano-filled Silicone Rubber

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ABSTRACT

The paper presents the experimental results obtained on the erosion resistance of silicone rubber (SIR) filled with 12 nm size fumed silica (nano filler) to those filled with 5 μ m size silica filler (micro filler). The ASTM 2303 Inclined Plane Tracking and Erosion Test was used in the comparison as well as an infrared laser as the source of heat to erode the SIR samples. The erosion resistance of the SIR materials increased with increasing percentage of the fillers, and it was observed that 10% by weight of nano-filled SIR gives a performance that is similar to that obtained with 50% by weight of micro-filled SIR. The low frequency components of leakage current and the eroded mass are used to evaluate the relative erosion resistance of the composites and the third harmonic component of the leakage current shows good correlation to the measured eroded mass. The paper discusses the possible reasons for the improvement in the erosion resistance of nano-filled silicone composites.

Index Terms — Aging, silicone rubber, nanodielectrics and nanotechnology.

1 INTRODUCTION

IN the last few years, a great deal of attention has been given to the application of nanodielectrics in the field of electrical insulating materials. Nanodielectrics are a class of materials containing at least one phase at the nanometer scale [1]. It has been reported that the use of nanoparticles in the matrix of polymeric materials can greatly improve the thermal, mechanical and electrical properties of polymeric composites [2-4].

Various studies have been made comparing the performance of nano- to micro-particle filled composites. Compared to microfillers, nanofillers have been reported to mitigate more efficiently the space charge formation in polymeric materials [4-6]. It has also been reported that the use of nanofillers improves the corona resistance of polyimide films [7]. In addition, the use of nanoparticles of zinc oxide in low density polyethylene has shown a smaller decrease in the resistivity of the polymeric matrix compared to microfillers [8]. The time-to-failure of cable material due to electrical treeing in epoxy resin has been increased using nanoparticle fillers compared to microparticle fillers [10].

In general, the improved properties of nano-filled composites have been related to the structure of the nanoparticles in the polymer matrix. With nanoparticle fillers, the amount of filler is considerably smaller when compared to composites filled with microfillers where fillers as high as 50% by weight is common practice. Despite all of their reported advantages, it has only been fairly recent that researchers are trying to understand the contribution of nanofillers to the modification of dielectric properties of composite materials. ¹Nelson and others [5] have attempted to explain the difference between nano- and micro- particle filled polymers using interfacial polarization, in which the nanoparticles appear to restrict the movement of the polymer end chains. In nano-filled polyamide films, the partial discharge resistance has been attributed mainly to the formation of a stair-like structure on its surface [9]. In epoxy resins, it is thought that increasing the specific surface area contributes to improving the electrical treeing resistance [10]. The dispersion of nanoparticles in a polymeric structure might activate more submicron-defects which could result in increasing the size of the electrical tree tip damage process and hence improve the resistance of electrical treeing growth and therefore the insulation lifetime [10].

Extensive studies on the influence of fillers, their type, particle size distributions, and concentration, on the erosion resistance of SIR materials subjected to dry band arcing, has been previously reported [11-12]. However, much of the work done on SIR materials is filled with microfillers. With the growing interest in nanofillers in polymeric materials, the use of these fillers could have a strong influence on the performance of SIR materials. Nonetheless, fumed silica, which exists in nano-scale, has been extensively used for physical reinforcement of SIR composites. Because of the interest in physical reinforcement, the amount of filler has been limited to less than 6 % by weight.

In the above context, the purpose of this work is to study the effectiveness of fumed silica, with particle sizes below 12 nm, and at relatively high concentrations (up to 10% by weight) on the erosion resistance of SIR composites under conditions

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conducive to dry band arcing. In addition, the bulk and surface physiochemical characteristics, for example thermal conductivity, bonding characteristics, and chemical compositions have been carried out using TGA, SEM, and FTIR analyses.

2 MATERIALS AND METHODS 2.1 LASER TEST

The usefulness of an infrared laser as a source of heat to study erosion in filled SIR composites has been demonstrated in our earlier studies [13]. Particularly the method is easy and less cumbersome to study various combinations of different fillers in SIR. The laser technique used in this work to measure the erosion of silicone rubber samples filled with various fillers is based on the constant energy approach. In, brief, a near infrared semiconductor laser source operating at a wavelength of 808 nm was used to irradiate the silicone rubber samples, Figure 1. The samples were subjected to 5 W laser for 60 and 120 s, respectively. The output power of the laser was calibrated using a laser power meter, but the incident power was not measured. The power of the laser was adjusted offline and a shutter controlled the exposure time. The laser was then shut off and the eroded mass was measured using a microbalance.



Figure 1. Laser experimental setup.

Samples of a two-part room temperature vulcanized (RTV) SIR filled with 3%, 5%, 7% and 10% of 12 nm, hydrophobic fumed silica by weight (wt %) were prepared. For comparison, unfilled SIR has also been studied under similar test conditions. For each formulation, after weighing and mixing the proper amounts of SIR and filler, the compound was poured into a mould and degassed. Controlled heat was applied to accelerate curing. After curing, disc shaped samples of 19 mm diameter with a thickness of 9mm \pm 0.1mm were prepared for laser tests. A small amount of Fe₂O₃ (0.4% of filler weight) was added to the mixture in order to provide a light red colour to samples, favouring absorption of laser energy.

2.2 INCLINED PLANE TEST (IPT)

The test procedure followed the ASTM 2303 standard [14], with an initial voltage of 2.0 kV and a constant contaminant flow rate of 0.15 ml/min for four hours [15]. The voltage was increased at a rate of 250V/hour. At the end of the test, the samples were taken from the test bay and the eroded volume was estimated by filling the eroded volume with a soft putty of

known density and then the eroded mass was calculated. The base rubber used was the same one used in the laser experiments. The filler type/concentration used is presented in Table 1.

Table 1: Chemical composition of the tested samples in IPT test.

Filler Type and median size	Concentration of
	filler by weight (%)
12 nm fumed silica	5
12 nm fumed silica	10
5 μm ground silica	10
5 μm ground silica	30
5 µm ground silica	50

A PC based data acquisition system, composed of voltage dividers, over-voltage and over-current protection circuits, and a 100 kHz, 16-channel data acquisition card, recorded the source voltage, and voltage and current for each sample. During the test, for every second, four 60 Hz cycles of data were acquired at a sampling rate of 7580 samples per second. These four cycles were processed by the Fast Fourier Transform (FFT) technique for the leakage current (LC). Then, the LC current data were averaged and smoothed using the moving average technique [16]. The software for analyzing the data recorded by the data acquisition system was built on a LabVIEW® platform. The results presented in the following section are average values from three samples for each test case. Figure 2 shows the schematic of inclined plane setup and the data acquisition system.





2.3 INFRARED MICROSCOPY

The chemical analysis on the surface of the test samples was performed using fast Fourier-infrared spectroscopy (FTIR) in the reflectance mode (Hyperion 2000 microscope coupled to a Tensor 27 mid-infrared spectrometer, Bruker Optics). The spectra were recorded from 700 to 4000 cm⁻¹, with resolution of 2 cm⁻¹, over a square area of 150 mm x 150 mm. The final spectrum was obtained with 256 scans. Background run obtained on a mirror surface was subtracted from the spectrum of each sample.

2.4 THERMO-GRAVIMETRIC ANALYSIS (TGA)

Thermo-Gravimetric Analysis (TGA) was conducted using TA InstrumentsTM, SDT 2960. Measurements were made in a 5% oxygen and 95% helium environment with a rate of 20

^oC/minute temperature rise. The temperature was increased from 100 to 800 ^oC. The weight loss and rate of weight change in a material, as a function of temperature was recorded for both filled and unfilled silicone composites. The sensitivity of the balance is 0.1 mg with 1% accuracy.

2.5 TRANSMISSION ELECTRON MICROSCOPY

The morphology of the nanoparticle distribution in the rubber matrix was investigated using transmission electron microscopy. A thin section 80 nm thick of the SIR with 10 % fumed silica was obtained using the Leica UC6 ultracryomicrotome operating at -140 °C. a Philip 200 kV transmission electron microscope (TEM) with liquid nitrogen cooling stage was used to investigate the morphology of the nanocomposites.

3 RESULTS AND DISCUSSION

The influence of nanofillers on the erosion resistance of silicone rubber in comparison to conventionally filled rubber has been analysed using laser test results and also IPT results. In addition, to further understand the underlying mechanisms that have improved the performance of nanocomposites substantially, both the bulk and surface analyses are carried out.

3.1 WEIGHT LOSS COMPARISON

Weight loss as a measure of erosion in samples filled with nano filler exposed to laser heating is shown in Figure 3. For the range of filler concentration considered, 0 to 10 % by weight, the erosion resistance increased with increased amount of filler as expected. Also, the slope of the weight loss lines decreased as the filler concentration increased, in which it dropped from 0.0021 for unfilled SIR to 0.00013 for 10% filled SIR. Although the observations made are as expected, it is interesting to note that at as low as 10% nano filler, the weight loss vs. erosion curve is almost flat; thus confirming the expected improved behaviour at low concentrations of the nano filler.



Figure 3. Weight loss as a function of laser energy for various nano-filled silicone rubber samples.

Since conventionally filled polymers contain microfillers, it is quite important to compare the performance of both the nano- and micro- silica filled SIR. To achieve this, the standard ASTM 2303 test method was used to compare both nano- and micro- silica filled SIR. The eroded mass for all the tested samples were measured and are depicted in Figure 4, in which, it is evident that as the filler concentration increased, the eroded volume decreased. Again, the important observation to be made is in the concentration of fillers used. In the case of nano-filled SIR, the amount was only 10% of filler by weight that is required to achieve the same or better performance of the microfilled samples that contained 50% filler. On the other hand, the eroded mass of 10% microfilled SIR samples is almost one order higher compared to 10% nanofilled SIR samples.



Figure 4. Comparison between micro- nano-silica filled composites in terms of eroded mass after IPT test.

The erosion of filled SIR samples is very closely related to the third harmonic component of the leakage current (LC), which occurs during dry band arcing [16]. Hence the LC was continuously monitored during the IPT tests, and Figure 5 shows the recorded LC during the IPT for both nano- and micro- filled samples. In both cases, the third harmonic component of LC shows a good correlation with eroded mass (Figure 4). However, regardless of the degree of surface damage, the fundamental component of LC saturated at about 6 mA. This could be attributed to the controlled flow of contaminant on the sample surface in the IPT, which results from a controlled surface resistance and hence, a controlled magnitude of the fundamental component of LC.



Figure 5. Third harmonic component of leakage current during the IPT test. a, Nano-filled SIR; b, Micro-filled SIR.

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3.2 BULK MATERIAL ANALYSIS

Significantly improved performance was observed with nano-filled SIR compared to both unfilled and micro-filled SIR which can be attributed to modifications in the physical characteristics of the bulk and/or the surface of the nanocomposites. To analyse the bulk material, both the thermal conductivity and chemical bonding of filled and unfilled composites were studied.

The thermal conductivity of SIR filled with micro filler has been analysed in a greater depth earlier and it was found that the thermal conductivity improved as the concentration of silica increased in SIR [17]. In the present study, the thermal conductivity was measured for unfilled, 7% and 10% nanofilled SIR using a modified version of ASTM D 5470-95 [18]. The test was conducted in vacuum to minimize any heat loss due to conduction or convection. In addition, the thickness of the sample during the test was continuously measured and was accounted for thermal expansion. Table 2 shows the measured thermal conductivity, K along with those calculated using equation (1) [19].

$$K = \frac{K_{1}K_{2}}{K_{1}X_{2} + K_{2}X_{1}}$$
(1)

Where:

K1 = thermal conductivity of pure silicone rubber = 0.19

K2 = thermal conductivity of silica = 1.5

X1 = the fraction volume of silicone rubber

X2 = the fraction volume of silica

Table 2. Measured and Calculated Thermal Conductivity

Material	Measured T.C. (W/m.K)	Calculated T.C. (W/m.K)
Unfilled SIR	0.189	0.19 (from supplier)
SIR with nano filler (7%)	0.195	0.196
SIR with nano filler (10%)	0.213	0.199

Although the difference in the measured erosion resistance between unfilled and 10% nano-filled SIR is substantially large (Figure 3), there is no significant difference in terms of thermal conductivity for these two cases (Table 2). Thus, it can be concluded that the thermal conductivity has very little effect on the improvement in the erosion resistance of nanocomposites for the filler concentrations used.

To explore the possible improvement in chemical bonding, first the TGA analysis was conducted to compare nano-filled SIR with unfilled SIR, Figure 6. The weight loss for unfilled silicone was about 66.5%; whereas, it was only 28.5% and 22.5 for 5% and 10% nano-filled samples respectively. The weight loss for the fumed silica is also shown for the same temperature range for comparison. In addition, the first derivative of the TGA curves was used to see precisely at what temperature decomposition began for both filled and unfilled silicone rubber, Figure 7. The first peak, which is not very strong, was noticed for both the filled and unfilled silicone

rubber around 400 °C. The second peak, which appears around 500 °C and is caused by the liberation of the side chain (CH3) from the silicone backbone (Si-O-Si) [20], Figure 7, shows a significant difference in terms of the steepness that corresponds to the rate of weight loss. While the rate of weight change (%/°C) is around 0.15 for both 5% and 10% nano-filled silicone rubber, it exceeds 2.5 for unfilled silicone rubber. From this observation it can be accepted that the presence of fumed silica has improved the thermal stability of the silicone rubber matrix. Accordingly this speculation can be further supported comparing the estimated weight loss for the composites studied.



Figure 6. TGA analysis for different silicone rubber compositions.



Figure 7. Derivative of Weight loss for: (a) Both 10% and 5% nano-filled silicone rubber, (b) Unfilled silicone rubber.

The TGA results for pure SIR and pure fumed silica were used to estimate the weight loss of the 5% and 10% filled composites assuming that each component decomposed independent of the other. Since the remaining weight of the SIR with no filler was 28.5% and fumed silica did not decompose at 800 °C, Figure 4, the estimated remaining weight can be calculated using the following formula:

$$Z = \left(\frac{X * 28.5\% + Y}{X + Y}\right)$$
(2)

Where:

Z = estimated remaining weight of the composite (in %)

X= weight of the silicone rubber (in grams)

Y = weight of the filler (in grams)

By comparing the calculated value of the weight loss with that measured, Table 3, the influence on the chemical bonding between fumed silica and SIR is obtained. It is estimated that the remaining weight by adding 5% of fumed silica will be around 33% if both the filler and the SIR would decompose independent of each other. However, the actual weight remained after TGA test was 67%, which is almost the double. Similar results were noticed for 10% nanofiller as well, Table 3. The observed reduced weight loss is strong evidence that the presence of fumed silica has significantly improved the thermal stability of SIR composites. It is suggested that this behaviour is because of filler- matrix interaction resulting more stable chemical bonds.

 Table 3. Estimated and Measured Weight Loss of 5% and 10% Nano-filled

 Silicone Rubber Subjected to TGA.

Material tested	Estimated remaining weight	Measured remaining weight	Ratio of measured/estimat ed weight
5% nano filler	32.1 %	66.57%	2.07
10% nano filler	35.21 %	73.8%	2.09

Improvement in chemical bonding stability between micro filler and SIR has been reported earlier [17]. TGA analysis was also conducted for the micro-filled SIR and compared to nano-filled SIR. It is apparent from Figure 8 that there was no significant difference between nano and micro composites for the same 10% filler level in terms of TGA results. Differently, the eroded mass of the 10% nano-filled SIR was much lower than the 10% micro-filled SIR after IPT test, Figure 4. Therefore the filler size was significant parameter to improve chemical bonding stability at the surface (IPT test) of the composites, while this parameter was not so significant improving stability when thermal degradation was carried out in bulk (TGA).



Figure 8. Comparison between 10% nano and micro- filled SIR in terms of TGA analysis.

The possible agglomeration of the nano particles in the bulk of the polymeric material cannot be ignored because nanoparticles tend to agglomerate easily creating larger secondary clusters. If the size of these clusters is comparable

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to the micro particles it may explain such similarity in bulk properties (TGA performance) between the nano and microfilled SIR. A transmission electron microscopy (TEM) analysis was conducted on a microtomed section from the bulk of 10% nano-filled SIR, Figure 9. It is apparent from Figure 9 that clusters resulting from the agglomeration of primary nano particles (12 nm) are present in the submicron range. These clusters are much closer in to the size of micro filler used (5 mm) and are spread in the bulk of the material. As a result of this particle agglomeration, the interface area of nano-filled rubber will be dramatically reduced resulting in a similar level of interface for both the nano and the micro- filled SIR. This observation agrees with no significant difference in terms of bulk chemical bonding stability between the filler and the SIR.



Figure 9. TEM analysis for 10% nano-filled SIR.

3.3 SURFACE ANALYSIS

To explore other possible chemical changes on the SIR surface, FTIR microscopy before and after exposing the surface to erosion was conducted for both 10% nano-filled and unfilled SIR, Figure 10. To allow a relative comparison, the spectra were normalized using the height of the 1254 cm⁻¹ (Si-CH3 bond).

It is evident from Figure 10b that the Si-O-Si bond (1000-1100 cm⁻¹) percentage increased for damaged area compared to undamaged area for the 10% nano-filled samples. On the other hand, no such change happened for unfilled SIR, Figure 10a. Such increase could be due to increase of the silica concentration after the erosion of the polymeric material thus forming a layer of silicate. This layer might enhance the erosion resistance of SIR by providing a barrier that might protect the polymeric material from further degradation.

To further understand the difference between nano and micro-filled SIR, a detailed scanning electron microscopy (SEM) investigation was conducted on both the damaged and undamaged 10% nano- and 10% micro-filled SIR samples as shown in Figure 11. There is a significant difference between the surfaces of the damaged nano- and micro-filled SIR. While it was not possible to see the dispersion of the nano filler in the undamaged sample, Figure 11b, the fumed silica tended to agglomerate during the dry band arcing, forming a silica-like layer, Figure 11a. This layer is similar to the layer formed by pure fume silica particles, Figure 11e. The formation of this type of layer was not apparent in the micro-filled samples, as shown in Figure 11c.



Figure 10. FTIR spectra of surface undamaged and damaged SIR with laser. a, unfilled SIR; b, 10% nano-filled SIR.



Figure 11. SEM analysis for nano and micro-filled SIR. a, damaged nano-filled SIR; b, undamaged nano-filled SIR; c, damaged micro-filled SIR; d, undamaged micro-filled SIR; e, Fumed silica particles.

EDAX analysis was conducted to test the surface composition of nano and micro-filled SIR, Table 4. Both the weight percentage (wt%) and atomic weight percentage (At%) were measured.

Table 4. EDAX analysis for 10% nano and micro-filled SIR.

Sample	Si		0		С	
	Wt%	At%	Wt%	At%	Wt%	At%
Nano-filled	36.07	22.76	46.44	51.44	17.49	25.81
damaged						
Nano-filled	33.4	19.09	24.23	24.3	42.37	56.61
Undamaged						
Micro-filled	29.22	16.24	25.41	24.79	45.37	58.97
damaged						
Micro-filled	34.92	20.01	21.63	21.76	43.45	58.23
Undamaged						

A distinct difference between damaged and undamaged nano-filled SIR is found. An increase in both the silicone and oxygen concentrations was observed for the damaged SIR compared to the undamaged sample. Since both these two elements are available in fumed silica as well, it can be assumed that the fumed silica stays on the surface of nanofilled SIR. Such difference in composition is not very clear when comparing micro-filled SIR, Table 4. It can therefore be stated that the formation of silica like layer with nano-filled composites aids to resist further degradation of SIR during dry band arcing. The formation of similar protection layer on the surface of polyimide material filled with nano fillers has been reported previously and it has been mentioned that such layer is responsible of the enhanced PD resistance [9].

4 CONCLUSIONS

- Weight loss assessments using IPT test results confirm that the nano-filled SIR composites depict significant resistance to erosion compared to micro-filled SIR composites with as low as 10% of fillers, by weight, in SIR.
- No significant improvement in terms of thermal conductivity was found between nano filled and unfilled SIR. Also, TGA analysis revealed that both nano- and micro- silica filled SIR behaved in similar manner when it comes to bulk properties. More work is needed to improve dispersion of nano fillers in SIR to enhance the bulk properties and hence to attain even better erosion performance.
- As far as the surface behaviour, once the top particle free rubber erodes, the formation of silica like layer helps to resist the further degradation of SIR in nanofilled samples due to the nature of the strong bonding between nano particles with rubber. However, this surface behaviour is only a characteristic of nano filled composites by virtue of their size, distribution and interface formation.

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