A Review on the Importance of Nanocomposite Processing to Enhance Electrical Insulation

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ABSTRACT

Study of the experimental literature regarding nanodielectrics indicates numerous inconsistencies in the results obtained. In many cases, the likely cause is due to a lack of quality control during nanocomposite processing. By examining examples from the literature along with an alumina/polyamideimide nanocomposite and silica/crosslinked polyethylene nanocomposite, this contribution seeks to shed light on some of the likely causes for these inconsistencies. Measurements of the dielectric breakdown strength and voltage endurance confirm that poor dispersion can lead to poor material performance and the use of quantitative techniques is highlighted. Good dispersion alone is not sufficient to achieve improved properties. The addition of nanoparticles can alter the resulting structure of the nanocomposites, can introduce water into the system resulting in cavity formation and can also result in degradation of the polymer if the processing parameters are not carefully selected. In this review, understanding the effects of nanoparticle addition requires not only characterization of relevant dielectric properties, but also careful control of the processing parameters and characterization of changes in polymer structure, particle dispersion, and water content.

Index Terms — Nanotechnology, plastics, nonhomogenous media, dielectric breakdown

1 INTRODUCTION

Nanoparticles have been shown to provide dramatic changes in space charge behavior, charge trapping and permittivity in polymer matrices [1]. These changes in the behavior of the material can manifest themselves as improvements in the dielectric properties of the composite, such as orders of magnitude improvement in the voltage endurance and improvements in the breakdown strength [2,3]. Nanoparticles can also reduce discharge erosion rates and improve thermal and mechanical properties of polymers [4,5,6].

Due to the small size of nanoparticles relative to micron sized fillers, nanoparticles have much higher interfacial area per unit volume. In order to take advantage of these effects, the nanofillers must be well dispersed. In addition to the interfacial area, the interface between the particle and the polymer is thought to have a finite thickness in which the properties of the polymer differ from the original matrix. In nanoparticle systems the total interfacial volume can be very large and may control the dielectric response of the system. At modest particle loadings, the interaction zones may begin to overlap, leading to effective percolation of the interfacial areas and dramatic effects on the properties of the nanocomposites [7].

There have been some conflicting reports in the literature regarding the effect of nanoparticles on the dielectric properties of polymer composites. Nanoparticles have been shown to both increase and decrease the breakdown strength in similar systems [3,8,9]. While variations are to be expected in different systems, the effect of processing can also be a determining factor in the final properties. For example, micron fillers typically lead to reductions in the breakdown strength, and similarly sized agglomerates of nanoparticles likely do the same [9,10]. This not only complicates efforts to determine whether a particular system is practically useful, but can also bias the conclusions drawn when investigating the mechanisms leading to changes in properties.

Therefore, it is important to carefully consider and control the processing conditions used when making nanocomposites. Unfortunately, these systems can present additional challenges when compared to microcomposites. This can include difficulty in controlling dispersion, high relative water content of the particles and changes in the polymer microstructure. In this review, the importance of processing on the properties of some nanocomposite systems will be highlighted and relevant methods of composite analysis discussed.

2 DISPERSION CHARACTERIZATION

The use of quantitative techniques for characterizing the dispersion in the composite avoids subjective judgment of the mixing degree of nanofillers. To carry out a statistical analysis of the microstructure, the general steps required are [11,12]. 1)

Taking a micrograph of the composite 2) Choosing an image sample size representative of the structure of the material 3) Identifying particles. 4) Analyzing the images and 5) Generating the statistical parameters that describe the dispersion.

For low contrast images, particles need to be selected manually, but for high contrast images the particle selection can be automated. Analysis of fracture surfaces is a common method for analyzing dispersion. However, this method is not ideal for quantification, as the exposure of particles on the surface can be biased by fracture morphology. Similarly, qualitative assessment, though perhaps useful for identifying gross changes in dispersion, also suffers from bias due to user interpretation.

TEM (transmission electron microscopy) and FIB (focused ion beam)/SEM (scanning electron microscopy) methods are useful for generating cross-sections without the bias of fracture surface morphology. FIB/SEM imaging provides a way to image nanoparticles in a two dimensional plane. A focused ion beam can be used to cut a groove in the surface of the polymer, providing a flat surface for SEM imaging. This method also helps to expose the nanoparticles by removing polymer on the surface of the particles. The FIB method of sample preparation provides the benefit of a flat sample surface which exposes the particles without introducing surface mechanical stresses seen in microtoming, which may introduce voids or particle debonding. FIB/SEM also eliminates the tedious and often time consuming sample preparation needed for TEM. Combined, the FIB/SEM method gives the benefits of TEM (flat surfaces, imaging of particle locations not possible using coated SEM samples) with the simplicity of SEM sample preparation. A comparison of the TEM imaging method and FIB/SEM imaging method is shown in Figure 1, showing that the FIB/SEM image can give comparable information to the TEM images.

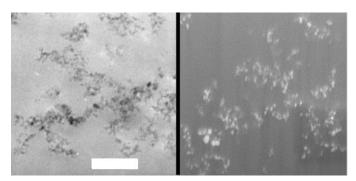


Figure 1. TEM image (left) and FIB/SEM image (right) of 5 wt % fumed silica in polyamideimide. Scale bar is 200 nm.

In this paper, quantification was performed using the quadrat method and the nearest neighbor index method (NNI). The quadrat method divides the sample into cells, counts the number of particles in each cell and reports the skewness of the particle distribution. Higher values of skewness indicate poorer mixing. The first nearest neighbor index measures the average distance between nearest neighbors and scales the result by that expected from a random distribution. Values less than 1 indicate clustering, while values greater than 1 indicate ordering. The quantification methods break down the mixing degree of the particles into two independent aspects: the dispersion (nearest neighbor index), which indicates how well individual particles are separated from one another; and the distribution (skewness), which measures the uniformity of the spacing of particles and agglomerates [13,14]. Other parameters are available for quantification including higher order neighbor indexes [13,16], Morista's index [14], the k-function [13] and Monte Carlo methods to analyze empty sites [17].

In light of the fact that there is likely an interfacial volume around the nanoparticles with altered dielectric properties [25], the absolute value of nearest neighbors is also important. When the interparticle distance becomes small enough, the majority of these interfacial volumes should overlap, leading to a shift in the response of the composite with increases in loading. In the case of agglomeration, this leads to a reduction in the effective interaction volume as well as larger continuous interfacial zones. In terms of quantifying the interparticle distance, the thickness of TEM images can influence the results of the dispersion quantification, as the depth of field is typically larger than the specimen thickness [15]. In this case, two dimensional images are preferred. A comparison of simulated SEM and TEM images of different thickness is shown in Figure 2, showing that thicker slices give a smaller apparent interparticle distance. Additionally, qualitative analysis may seem to reveal agglomerates which in fact are particles in different planes.

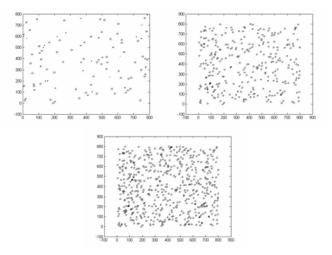


Figure 2. Simulated images of a random dispersion of 5 wt% 12 nm spherical silica nanoparticles in XLPE. Upper left: SEM image. Upper Right: 20 nm thick TEM image. Bottom: 40 nm TEM image. Images are 400 x 400 nm. Average apparent interparticle spacings are 35, 21 and 16 nm.

3 DISPERSION METHODS

Due to their small size and high surface area-to-volume ratio, nanoparticles are typically more difficult to disperse than microparticles. In order to achieve good mixing, aggressive methods are sometimes necessary. Unfortunately, this may lead to degradation of the polymer or volatilization of low molecular weight components. Due to the difficulties of dispersion and side effects aggressive mixing can have on material properties, processing of nanocomposites needs to be carefully considered.

3.1 WATER AND SURFACE TREATMENT

Many nanoparticles are hygroscopic, and the presence of water on these particles can make separating them more difficult, leading to agglomeration [18]. Drying the nanoparticles and matrix under vacuum prior to compounding is therefore a necessary step. Water uptake can also be mitigated through the attachment of hydrophobic functional groups to the surface of the nanoparticle. The chemical surface treatments replace surface hydroxyl groups and can also physically block water from getting to the surface of the particle [19]. The presence of water can have additional effects on the behavior of nanocomposites, leading to changes in the dielectric properties independent of dispersion issues [20]. Thermal analysis is useful in characterizing moisture content in nanoparticles and nanocomposites. This can be accomplished using TGA (thermogravimetric analysis) to measure water loss and DSC (differential scanning calorimetry) to measure water freezing/melting temperatures. Polymers and polymer-based composites are usually thermally stable to 200 °C, and weight loss below this temperature can be attributed to water loss (in the absence of other low molecular weight components, such as residual solvents).

Water in the system can also lead to adverse affects later in processing. Figure 3 shows the effect of improper drying of a silica/cross linked polyethylene (XLPE) composite system prior to compounding. The excess moisture present during molding, which takes place above 100 °C, leads to excessive bubbling within the sample. The silica nanoparticles make this effect more likely, as they are very hygroscopic and require extensive drying prior to compounding to remove water.



Figure 3. Silica/XLPE nanocomposite multi-recess sample with excess bubbles formed during molding.

3.2 COMPOUNDING AND PARTICLE DISPERSION

High shear force mixing has been found to be an effective way to disperse nanoparticles. Shear mixing is typically applied to polymer melts to disperse particles, for example in polyethylene nanoparticle systems. When choosing a compounding method, the elimination of agglomerates (or at least reduction of agglomerates to small sizes) needs to be considered. In many cases additional steps are necessary beyond those which would give acceptable dispersion in microcomposites. For example, in an XLPE/silica nanocomposite, it was found that pre-mixing the nanoparticles and PE pellets in a dual asymmetric centrifuge at 1800 rpm for 30 seconds improved the dispersion of the nanoparticles. A qualitative comparison of composites mixed with and without this method is shown in Figure 4.

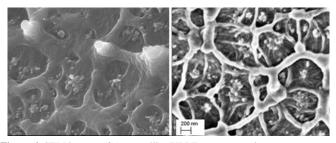


Figure 4. SEM images of 5 wt % silica/XLPE nanocomposites.

Ultrasonics have also proven useful in dispersing nanoparticles. Kurimoto et al. [21,22] used a novel technique which combined ultrasonic waves and centrifugal force to fabricate alumina/epoxy nanocomposites. The application of ultrasonics was found to be crucial for good dispersion. Similarly, Kochetov [23] combined ultrasonic processing and high shear force stirring to obtain an even dispersion of the corresponding filler in the base material.

The use of a dual asymmetric centrifuge has been found to be useful in dispersing nanoparticles in liquid resin systems. The addition of a mixing media is suggested in this case to help break apart agglomerates. For example, the effect of the addition of alumina balls during mixing on the dispersion of alumina nanoparticles in a polyamide-imide system is shown in Table 1. The dispersion of the alumina nanocomposites was quantitatively analyzed using the nearest neighbor and quadrat method. At 5 wt %, the nearest neighbor index value is closer to 1, indicating improved particle dispersion. The skewness value also drops, indicating better particle distribution. Figure 5 shows SEM images of these materials.

 Table 1. Dispersion quantification of 5 and 10 wt % alumina/PAI nanocomposites.

	No Alumina Balls (5 %)	Alumina Balls (5 %)	No Alumina Balls (10 %)	Alumina Balls (10 %)
Skewness	1.74	0.71	2.0	1.73
1st NNI	0.50	0.74	0.65	0.68

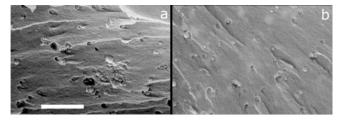


Figure 5. SEM images of 5 wt % alumina nanocomposites mixed without (left) and with (right) alumina balls. Scale bar is 1 μ m.

Even using the same mixing methods, high loadings can make dispersion more difficult. For the same alumina/PAI system, at 10 wt % there is only a slight improvement in the nearest neighbor index with the addition of alumina balls during mixing. Similarly, the skewness of the particle distribution is little improved at 10 wt % with alumina ball addition. Qualitatively, the effect of the alumina balls on dispersion at 10 wt % is shown in Figure 6. It should be noted that direct comparison of the quantitative parameters between loadings would require normalization of these parameters, which is not done here.

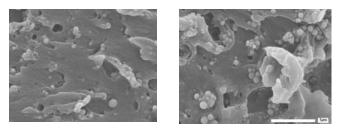


Figure 6. Comparison of 5 wt % alumina nanocomposite and 10 wt % alumina nanocomposite mixed using alumina balls. Scale bare is 1 μ m.

4 EFFECTS OF PROCESSING ON PROPERTIES

4.1 DIELECTRIC BREAKDOWN AND ENDURANCE

The effect of dispersion on the dielectric properties of the nanocomposites is highlighted through a comparison of two different dispersion states at the same loading in an alumina/PAI nanocomposite and a silica/XLPE nanocomposite.

The effect of the particle distribution and dispersion on dielectric breakdown strength is shown in Figure 7 and Figure 8. The change in the breakdown strength for the alumina nanocomposites shows a positive correlation with the nearest neighbor index and a negative correlation with the skewness. The drop in breakdown strength with the increase in skewness and decrease in nearest neighbor index shows that as the quality of mixing becomes poorer, the breakdown strength suffers. This drop in breakdown strength with agglomeration can be linked with a larger defect size in the material, similar to what would be expected for microcomposites. For the silica nanocomposite, almost no change in the nearest neighbor index is seen. However, there is a difference in breakdown strengths between the two composites and a change in the skewness. The skewness and the nearest neighbor index measure two different aspects of mixing quality, and this shows that both are necessary to characterize the composite. In the case of the silica composite, the fumed silica used forms fractal agglomerates which cannot be broken apart. The nearest neighbor index for these particles is expected to reach a maximum value of less than one, which is likely the reason there is no difference between the two composites. However, if these fractal agglomerates are not well dispersed, the breakdown strength suffers, as indicated by the skewness.

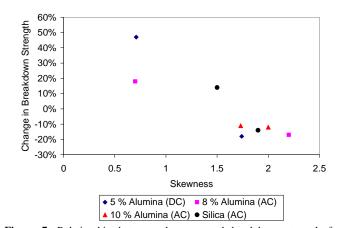


Figure 7. Relationship between skewness and breakdown strength for alumina/PAI nanocomposites and silica/XLPE nanocomposites.

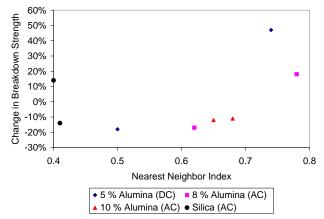


Figure 8. Relationship between nearest neighbor index and breakdown strength for alumina/PAI nanocomposites and silica/XLPE nanocomposites.

At high loadings, achieving good dispersion can become more difficult. This can manifest itself as a peak or plateau in dielectric properties, and occurs before percolation of the particles would be expected. Figure 9 shows that through 7.5 wt % for the alumina/PAI system, the breakdown strength is found to increase. At 10 wt %, where agglomerates become prevalent, the breakdown strength drops below that of the unfilled material.

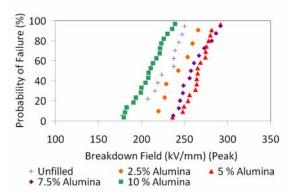


Figure 9. Weibull plot of AC breakdown strength of alumina/PAI nanocomposites (mixed using alumina balls) at 300 °C.

AC endurance testing shows that the addition of alumina

nanoparticles improves the time to breakdown when testing in air. The magnitude of the improvement is greater for 5 wt % alumina than 10 wt % alumina. The endurance life of 10 wt % alumina is improved over the unfilled material. A plot of the endurance data is shown in Figure 10. This data also shows that when the alumina nanoparticles are poorly mixed at 5 wt % the endurance life slightly reduced.

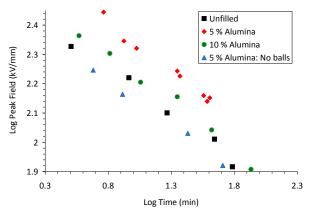


Figure 10. AC endurance in air for unfilled PAI and alumina nanocomposites.

The impact of agglomeration on breakdown strength compounds analysis of the effect of nanoparticles on the dielectric properties as presented in the literature. For example, a drop in breakdown strength at all loadings has been reported for an epoxy/ZnO nanocomposite, but the particle dispersion shows agglomerates on the order of 5 μ m [24]. A similar drop in breakdown strength for a ZnO/PE nanocomposite system is also seen, with no detail of particle dispersion [25]. However, other nanocomposite systems clearly show improvements in breakdown strength [26]. Where breakdown strength appears to be strongly linked to the dispersion, the effect is not as strong when dealing with corona resistance, and studies more consistently report improvements in PD resistance of nanocomposites [27].

As mentioned, surface functionalization can be used to help disperse nanoparticles, and can also be used to alter the dielectric properties. A silica/XLPE system serves as an example of the use of surface functionalization. Surface functionalization was applied to improve compatibility between the matrix and the polymer. Surface treatments of triethoxyvinylsilane (TES), n-(2-aminoethyl) 3-aminopropyltrimethoxysilane (AEAPS) and hexamethyldisilazane (HMDS) were used (Figure 11). Untreated fillers were found to improve the time to breakdown by two orders of magnitude, and the surface treatment, in all cases, further increased the time to breakdown [10].

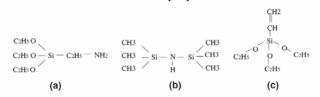


Figure 11. Schematic depiction of the surface modifiers

(a)aminopropyltrimethoxysilane (AEAPS), (b) hexamethyldisilazane (HMDS) and (c) triethoxyvinylsilane (TES) [10]

In the above example, no detectable change in the dispersion state is seen. This means that the likely cause for the change in properties is due to the interfacial effects. In addition to improvements in voltage endurance, surface functionalization has been shown to lower charge mobility and inhibit charge injection relative to untreated silica nanocomposites [28]. The addition of APTES treated nanosilica has also been found to introduce trap sites at the nanoparticle-polymer interface [29].

4.2 PERMITTIVITY

Literature reports have shown that in certain systems the real permittivity of the nanocomposite can fall well above or below that expected from volumetric mixing rules. For alumina/polyimide example, nanocomposites showed increases in permittivity above that expected due to the permittivity of the two components (with a calculated interface permittivity of 280), while an TiO2/expoy system showed the opposite effect [30]. Kurimoto et al. [21] investigated the influence of the dispersibility of nanoparticles and the effects on dielectric constant of alumina/epoxy nanocomposites. By controlling the synthesis techniques, a close link between particle dispersion and dielectric permittivity was built. A lower permittivity was found in nanocomposites compared to base resin when particles were uniformly dispersed. In these cases, interfacial effects are implicated as controlling the response. Better dispersion should lead to a larger nanoparticle-polymer interface volume, leading to a greater change in permittivity. In the PAI-alumina system shown previously, the permittivity was found to increase above that expected due to mixing rules. Of note is that between 7.5 and 10 wt %, where particle agglomeration was seen to take place, the permittivity shows a maximum, as would be expected based on agglomeration and a reduction in interfacial volume (Figure 12).

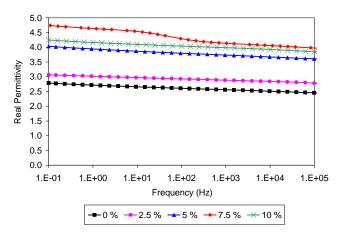


Figure 12. Real permittivity of dried alumina/PAI nanocomposites at 30 °C mixed using alumina balls.

4.3 MATRIX CHANGES

The addition of nanoparticles may also affect polymer

morphologies and thermal transitions, either through interaction of the nanoparticles with the matrix or through changes in processing parameters during compounding. The addition of fumed silica has been found to alter the crystallinity of XPLE. Figure 13 shows the crystallinity, taken from the integral of the DSC melting peak, for vinylsilane treated silica/polyethylene nanocomposites at various loadings. At loadings below 15 wt %, the crystallinity is unchanged, while at 20 and 25 wt % the crystallinity decreases. Changes in crystallinity can alter the dielectric behavior of polymers, and thus it is important to characterize these changes [31,32].

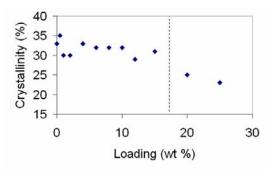


Figure 13. First heat DSC curves for vinylsilane treated silica nanocomposites. [32]

When particles with strong interactions with the matrix or high aspect ratios are added to a polymer melt during compounding, the viscosity of the melt can be significantly increased leading to higher viscosities, shear forces and heat generation during mixing. In the case of fumed silica nanoparticles in XLPE, the nanoparticles have been shown to narrow the processing window. Nanoparticles were first melt compounded with the PE. Dicumyl peroxide (DCP, BP 130 °C) was used as the crosslinking agent and added after the particles were mixed. In the case of the fumed silica filled polyethylene nanocomposites, the particles form fractal This morphology leads agglomerates. to increased particle/particle interactions relative to individual spherical particles. Due to these interactions, the viscosity increases rapidly with particle addition at modest loadings. The increase in viscosity requires an increase in processing temperature to prevent mechanical degradation of the polymer during mixing. It was found that the temperature needed to be greater than 125 °C to prevent mechanical degradation but below 130 °C to allow for proper crosslinking of the material and prevent loss of the DCP through boiling and premature degradation. One measure of incomplete crosslinking is a change in the degradation temperature of the polymer. The effects of incomplete crosslinking, as measured using TGA, are shown in Figure 14 [33]. TGA can also indicate that damage has been done to the material during compounding if the weight loss of sample starts at an abnormally early temperature [34].

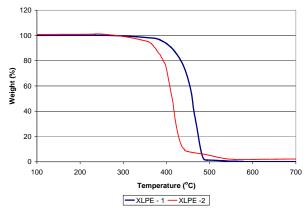


Figure 14. TGA determination of decomposition temperature of XLPE for quality control.

5 CONCLUSIONS

The addition of nanoparticles is known to change the dielectric behavior of polymers, though the response can vary based on processing parameters. If processing is not well controlled, the same system may give a widely varying response. Quantifying the dispersion of the nanoparticles allows for less bias when determining the quality of mixing. Given that the response of the nanoparticle systems is sensitive to the dispersion state, this can be important in determining where improvements in the processing procedures are necessary. If a change in processing is found to be detrimental to particle dispersion, this could lead to detrimental affects in the properties. Similarly, if the dispersion is improved, yet the breakdown or endurance properties are still detrimentally affected, this would point to other sources of failure, such as polymer degradation or moisture content. Based on the above analysis, in order to achieve repeatable properties in polymer nanocomposite systems, improve breakdown strength and draw proper conclusions, it is necessary to:

- Choose processing parameters which give well dispersed and well distributed nanoparticles.
- Carefully analyze and report nanoparticle dispersion and distribution, preferably through quantitative analysis.
- Thoroughly dry nanoparticles prior to compounding.
- Verify that the addition of nanoparticles does not lead to structural changes in the polymer matrix during processing; in cases where this does occur, these changes need to be characterized.

ACKNOWLEDGMENT

The authors would like to thank the Air Force Research Laboratory and the Electric Power Research Institute for their financial support, and the RPI Nanotechnology Center for facilities support.

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