Influence of conductive filler loading on EMI shielding and DC volume resistivity

What is the impact of conductive filler loading on shielding effectiveness?

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Conductive fillers for plastics have been used in EMI shielding applications for many years. Initially, pure metallic powders such as nickel or copper were used exclusively; but in recent years composite powders, such as nickel-coated graphite, have become commonplace. The polymers are loaded with conductive fillers to a sufficient level to create gasket materials with the required EMI shielding performance. Shielding requirements are application-dependent, so it is up to the device designer to select suitable conductive and polymer components for gasket materials.

The electrical and mechanical properties of conductive elastomers are highly dependent on the conductive filler loading level. A previous article illustrated the relationship of conductive filler loading to volume resistivity, hardness, and tensile properties through an example of silicone elastomer loaded with nickel-clad graphite filler.[1] There is a trade-off between achieving optimal mechanical and electrical properties as they work in opposition to each other. Electrical properties are improved at the expense of mechanical properties as the loading level increases. The loading level also influences shielding effectiveness, as shielding is loosely related to volume resistivity. However, shielding effectiveness is dependent on the material form of the gasket, whereas DC volume resistivity is not.[2, 3] In designing shielding elastomers, it is useful to quantify the interrelationships of conductive filler loading, volume resistivity, and shielding effectiveness. This article examines these relationships by investigating a silicone elastomer filled with nickel-graphite conductive filler, a material commonly used for EMI shielding applications.

SHIELDING EFFECTIVENESS TESTING

Direct methods for measuring shielding effectiveness are considerably more complex as compared to DC resistance methods such as volume resistivity measurement. Shielding effectiveness measurements typically require highly trained personnel to operate equipment and rather expensive instrumentation. For that reason, volume resistivity as a DC resistance measurement is a popular method to gauge EMI shielding effectiveness indirectly. However, for high frequencies common to wireless technology, the relationships between DC volume resistivity and shielding effectiveness are not direct and can be misleading.[2] Because of this disparity, it is practical to relate volume resistivity, shielding effectiveness, and conductive filler loading over a wide loading range when designing EMI shielding gaskets.

Coaxial test fixtures have been used to measure the shielding characteristics of EMI gasket materials for many years.[2] SAE ARP 1705[4] and ASTM D 4935[5] are two commonly used standard test methods that use coaxial designs. The coaxial design requires good electrical contact between a ring-shaped sample gasket and conductors within the test unit. The measured shielding properties are influenced by surface conditions of the test unit conductors and the test sample. Consequently, the coaxial test fixture measures combined properties of the bulk sample and the electrical contact interfaces. Test gaskets that shield well as bulk material, but have poorly conducting surfaces can be expected to perform poorly in a coaxial test fixture. Thus it can be difficult to evaluate the bulk shielding properties of a material. Also, the coaxial method assumes that the impedances of the test samples are very small compared to a reference impedance of 50 ohms so accurate evaluation of test samples high in impedance can be problematic.

Although the coaxial system is proven and well-established, the authors wanted a supplementary method to measure shielding properties of planar materials that was not dependent on the electrical contact resistance between the specimen and the test fixture. Such a method would be useful in evaluating the bulk shielding properties of planar materials without the interference of surface properties or the need for electrical interfaces. The objective was to build a relatively inexpensive bench top fixture that could quickly measure shielding properties of 15-cm sized elastomer sheets without contact resistance acting as an unknown variable. The test concept was to transmit a microwave signal onto one side of a sheet sample and then to measure the signal shielding using a receiving antenna on the other side in a free-space arrangement. A similar principle is used in the room-sized MIL-STD-285 test to evaluate shielded enclosures.

In practice, a true free-space test unit would require a directional transmitting antenna spaced at least 0.5 meters from a test sample for the incident electromagnetic field at 2.45 GHz (wavelength ~12.25 cm) to be planar with polarization parallel to the material/air interface. To avoid diffraction effects, the size of the shielding material sample would need to be at least 1 meter square. The large sample size made such a test configuration impractical. A miniaturized approximation to a true free-space test would need to accommodate a much smaller sample. To accommodate samples as small as 15 cm in size, the authors designed a test fixture based on a closed metallic waveguide with a radiating aperture on one end to accommodate the sample (Figure 1). A transmitting antenna situated inside the waveguide provided a 2.45-GHz signal via a tracking generator and power amplifier. The signal radiating from the aperture through the sample, was picked up by a waveguide receiving antenna. The received signal was then delivered to the input of a spectrum analyzer. Since the test sample was coupled to the transmitting antenna via a waveguide, we named this a quasi free-space (QFS) test fixture.

EXPERIMENT

The conductive filler used in this experiment was nickel-coated graphite powder with a weight composition of 65% nickel and 35% graphite. The particle size ranged from 75 to 190 microns (0.003” to 0.0075”) with an average particle size of 120 microns (0.0047”). The true particle density and apparent density of the nickel-graphite powder was 4.3 g/cm^3 (268 lb/ft^3) and 1.39 g/cm^3 (86.8 lb/ft^3), respectively. Figure 2 shows a micrograph of the flake-shaped particles. The silicone elastomer used in this experiment was a commercially available heat-cure methylvinylpolysiloxane resin base that is commonly used in industry to produce EMI shielding gaskets. In the absence of conductive filler, the Durometer Shore A hardness of the elastomer was 30 as-cured and 46 as-post-baked and had a density of 1.1 g/cm^3 (68.7 lb/ft^3). The conductive filler was compounded with silicone resin in a two-roll mill prior to curing in an hydraulic hot press to form square 15-cm (6") sheets, 1.7 mm (0.067") thick. Following molding, each conductive rubber sheet was washed with isopropyl alcohol and was post-baked in an air-circulating oven. A total of eleven silicone elastomer sheets was prepared with the loading levels of the nickel-graphite filler varying from 42.5 to 67.5 percent filler by weight. Following post-baking, the conductive rubber sheets were measured for shielding effectiveness in the QFS test fixture. Immediately following QFS measurements, the conductive rubber sheets were cut into 1.3 x 5 cm (0.5" x 2.0") strips for volume resistivity measurement and into circular rings for coaxial shielding measurements. All volume resistivity and shielding measurements were performed within 24 hours of post-baking. The cut strips were measured for volume resis-

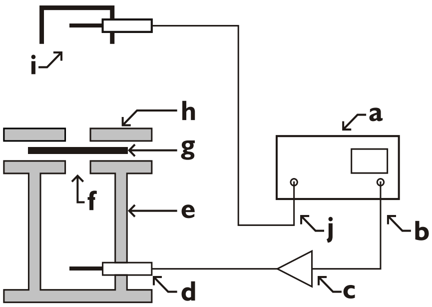

Figure 1. Quasi free-space test fixture configuration.

a = Agilent™ 8560 spectrum analyzer.
b = 2.45-GHz signal output.
c = power amplifier.
d = transmitting antenna.
e = waveguide.
f = radiating aperture.
g = sample conductive elastomer sheet.
h = sample locking plate.
i = receiving antenna with waveguide.
j = signal input.

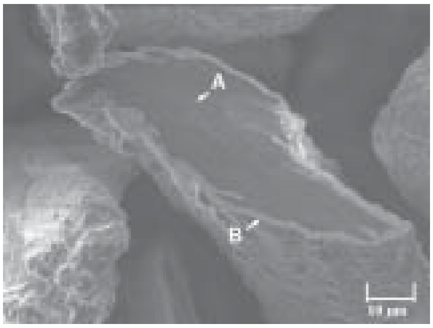

Figure 2. The nickel-graphite particles used in this work are flake-shaped and have an average particle size of 0.0047" (120 microns). A cross-sectioned particle in this scanning electron micrograph shows the graphite core “A” and the surrounding nickel cladding “B.”

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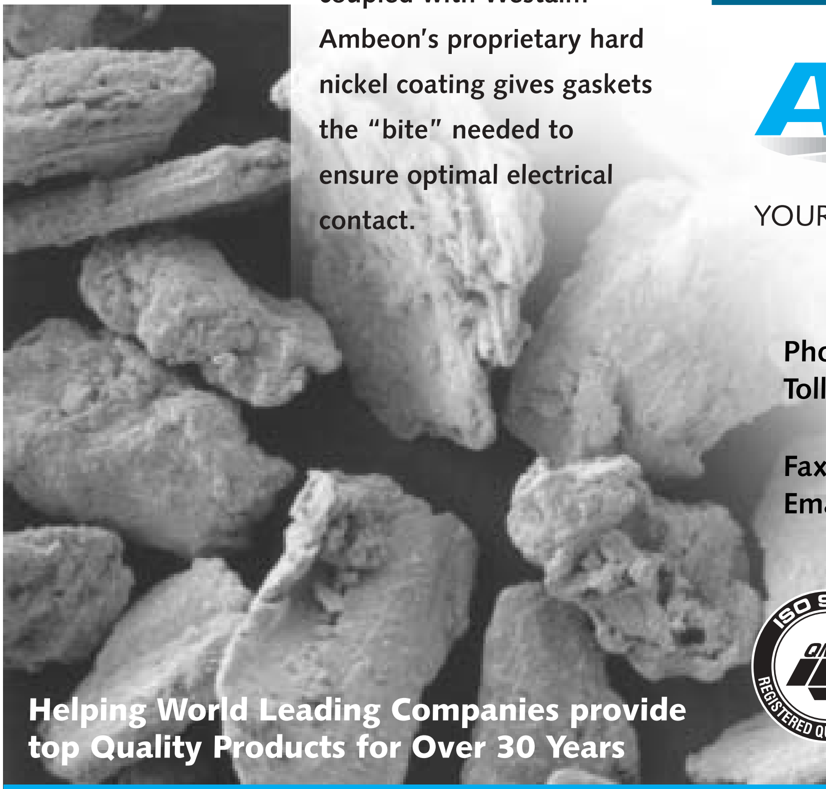

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Coaxial shielding effectiveness measurements were conducted using a Spira™ ZT-1000 test fixture in conjunction with an Agilent™ 8560-E spectrum analyzer. The operational range of the test fixture was 20 to 1000 MHz. Sample rings 6.35 cm in diameter and about 2.5 mm^2 in cross-section were cut from each of the eleven sample sheets. The rings were compressed by 20% in thickness when loaded into the test fixture. Shielding is defined as the difference, in dB, between the signal voltage at the input of the test fixture with the sample in-place, and the signal voltage at the output with the sample in-place in accordance with the SAE ARP 1705 procedure.[4] Since the resulting measured shielding value depends on the circumference of the sample ring, the values were normalized to 1 meter. The normalized shielding effectiveness was thus calculated as:

Shielding effectiveness (dB) =
Signal at input of test fixture with
sample (dBm) – signal at output of
test fixture with sample (dBm)
+ 20 log (100/πd)(dB) (1)

where
d is the median diameter of the sample in centimeters.

QFS shielding effectiveness measurements were conducted in the test fixture described in Figure 1 in conjunction with an Agilent™ 8560-E spectrum analyzer set at a fixed frequency of 2.45 GHz. Shielding is defined as the difference, in dB, of signal value at the receiving antenna of the test fixture, in dBm, with no sample in place, minus the signal value, in dBm, with the sample in place. No further corrections were applied.

RESULTS

Repeatability

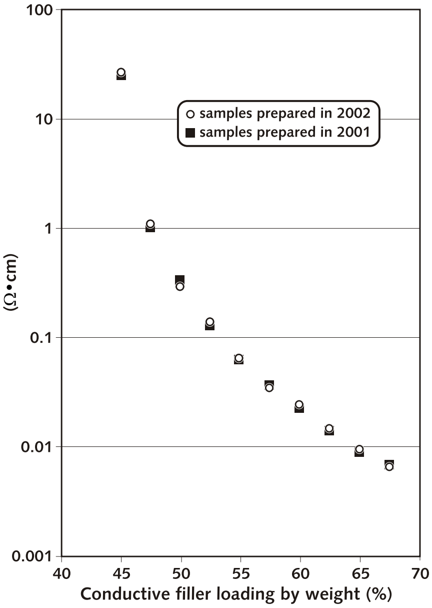

Figure 3. Comparison of volume resistivity versus loading from two identical sample sets of nickel-graphite filled silicone elastomer that were prepared and measured one year apart.

Figure 3 shows volume resistivity as a function of conductive filler loading for two elastomer sample sets that were prepared one year apart. All data points from the series are shown, and the data were not modified in any way. Volume resistivity data for samples prepared in 2001 were previously reported in ITEM 2002.[1] The volume resistivity data shown in this article were newly prepared in 2002 using the same base material lots of nickel-graphite and silicone elastomer and identical conditions of fabrication as previously reported. The two sample sets, composed of eleven samples each, were produced by different technologists. The two sample sets were highly repeatable in their measured volume resistivity values throughout the loading range. The samples loaded at the lowest level of 42.5% by weight were not conductive enough for volume resistivity measurement. The repeated results showed that control over processes of compounding, curing, and post-baking produces conductive elastomers with reproducible electrical characteristics.

Effect of Loading on Shielding Effectiveness

Shielding effectiveness versus frequency plots for the eleven samples are shown in Figure 4 for scans between 20 MHz and 1000 MHz, using the coaxial test fixture. The plots are smooth with frequency and show an overall trend of increasing shielding effectiveness with loading.

The plots begin at a filler weight loading level of 42.5% (15.9% by volume). At this lowest loading level, the shielding effectiveness of the elastomer was measured, but the material was not conductive enough to measure volume resistivity. The end of the series is

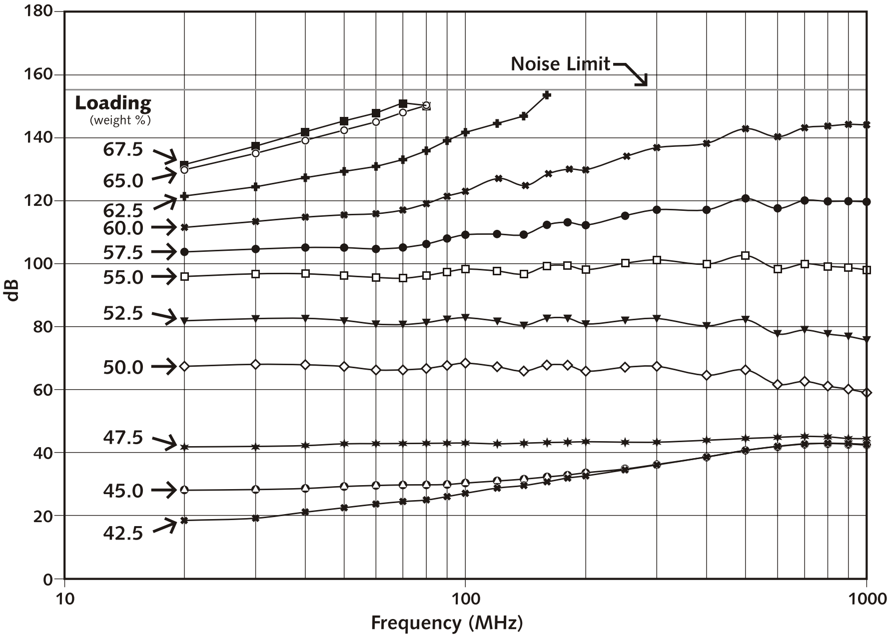

Figure 4. Frequency scans for shielding effectiveness of nickel-graphite filled silicone elastomer at different weight percent loading levels using coaxial test fixture.

marked by the highest loading level of 67.5% by weight (34.7% by volume). That highest level was the limit at which the conductive filler could be added to the silicone resin while keeping it workable to allow for molding. Different filler and elastomer types would have their own characteristics for maximum loading.

The noise limit for the instrument is 155 dB, and the higher loaded samples (62.5% and greater) meet this limit at 200 MHz and higher. Samples loaded to greater than 55% by weight show a frequency dependence on shielding effectiveness. Volume resistivity, and shielding effectiveness are shown in compilation plots as functions of conductive filler loading in Figure 5.

Loading is expressed in units of weight percent, volume percent, and parts conductive filler per hundred-weight polymer (phw). Note that the relationship between the scales of weight loading and volume loading is not linear. Equation (2) can be used to convert between weight percent and volume percent loading for any combination of filler and polymer.

$$\text{Volume loading (\%)} = wf/[wp(df/dp) + wf] * 100 \qquad (2)$$

where

wf, wp are the respective filler and polymer weights and df, dp are the respective true particle density and polymer density.

The unit of parts conductive filler per hundredweight polymer (phw) is calculated from percent weight as:

$$\text{phw} = [\text{weight \%}/(100\text{-weight\%})] \times 100 \qquad (3)$$

Plots A and B in Figure 5 show a rapid decrease in volume resistivity with increasing filler loading on linear and logarithmic scales. This rapid decrease is characteristic of the loading level at which the conductive particles begin to contact one another to form a conductive network. In this particular case, the rapid decrease (also referred to as percolation threshold) began at a loading level somewhere between 42.5 and 45.0% weight loading. The volume resistivity continued to decrease to a value of 0.007 Ω · cm at 67.5% weight loading, which marked the limit of filled silicone resin workability. For EMI shielding purposes, commercial conductive elastomer materials using nickel-graphite fillers are typically specified to be less than 0.1 Ω · cm.[6] Plots A and B in Figure 5 show that a weight loading level greater than 53% was necessary to achieve volume resistivity of less than 0.1 Ω · cm. The corresponding volume loading scale indicates greater than 22% loading by volume was necessary to attain less than 0.1 Ω · cm.

Plot C in Figure 5 shows a smooth increase in shielding with increased conductive filler loading using the QFS shielding test unit. For samples with loading levels of 57.5% by weight and greater, the shielding had attained the noise limit of 80 dB for the test fixture. Although the test fixture was effective in measuring shielding consistently and reproducibly, in this particular case, its effective range was limited to measuring samples loaded to 55% by weight and lower.

The coaxial test fixture was capable of measuring shielding effectiveness for all eleven samples across the full loading range at frequencies less than 200 MHz. Plot D in Figure 5 shows shielding plots for frequencies of 20 and 1000 MHz, as extracted from data shown in Figure 4. At high loading levels above 55%, the samples exhibited greater shielding at 1000 MHz, compared to 20 MHz. Shielding at 1000 MHz is more steeply sloped or more sensitive to loading compared to shielding at 20 MHz. The two shielding methods produced similar results with shielding steadily increasing with loading.

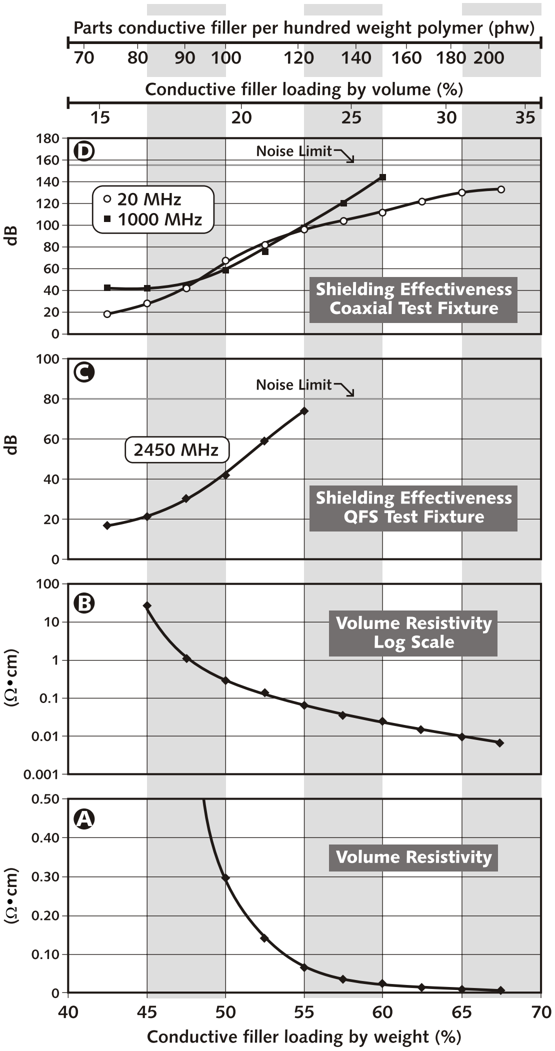

Figure 5. The relationships of loading for volume resistivity (Plots A&B), shielding effectiveness using quasi free-space test fixture (Plot C) and shielding effectiveness using coaxial test fixture (Plot D) for nickel-graphite filled silicone elastomer.

Volume Resistivity and Shielding Effectiveness

The relationship between shielding effectiveness and volume resistivity is shown in Figure 6A. The present case shows this relationship at different frequencies with a single material variable of loading level. Shielding is plotted against the log scale of volume resistivity in Figures 6A (coaxial test unit) and Figure 6B (QFS test unit). Data from both test units show a smooth drop in shielding effectiveness with increasing volume resistivity and then a sharp decrease in slope when volume resistivity is above 1 Ω · cm.

For the coaxial test unit, the de-

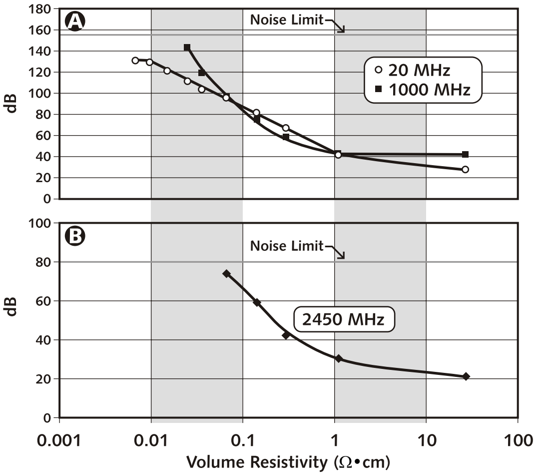

Figure 6. Relationships of shielding effectiveness with volume resistivity for nickel-graphite filled silicone elastomer using a coaxial test fixture (A) and a quasi free-space test fixture (B).

crease in shielding effectiveness is linear with the logarithmic volume resistivity scale from 0.01 Ω · cm to 1 Ω · cm at 20 MHz. This exponential relationship between shielding effectiveness and volume resistivity indicates the sensitive relationship between these two phenomena. Typically, conductive elastomers for EMI shielding applications range from 0.01 to 0.1 Ω · cm as-produced. Within that range, on the present samples, the shielding effectiveness had dropped from 130 to 85 dB because of loading. At 1000 MHz, the decay in shielding effectiveness is steeper than that for 20 MHz, showing a greater dependence on volume resistivity, or loading.

It is a common practice to decrease conductive filler loading slightly in order to increase the workability of the material or to adjust another physical property. Using the current example, if the loading were to be decreased from 60% by weight to 57.5% by weight, the volume resistivity would increase from 0.0247 to 0.0354 Ω · cm, a difference of 0.0107 Ω · cm. For the same change in loading, the shielding effectiveness (coaxial method) would decrease from 112 dB to 104 dB at 20 MHz and from 144 dB to 120 dB at 1000 MHz. Although this increase in volume resistivity is relatively small as a result of a small reduction in loading, the loss in shielding effectiveness is substantial, particularly at 1000 MHz where shielding dropped by 24 dB.

The QFS unit could measure shielding for samples with volume resistivity of 0.05 Ω · cm or greater. For EMI shielding purposes, commercial conductive elastomer materials using nickel-graphite fillers are typically specified to be less than 0.1 Ω · cm. The QFS test unit would be more practical for evaluating applications where lower levels of shielding are required.

CONCLUSIONS

The shielding effectiveness of nickel-graphite filled silicone elastomers is sensitive to changes in loading levels. The degree of sensitivity is variable and depends on frequency and on the loading level. The change in volume resistivity caused by loading is not directly associated with shielding effectiveness because it is frequency dependent. The coaxial and QFS test fixtures were comparable in indicating changes in shielding. The QFS fixture was more limited in shielding range, but demonstrated utility on low conductivity samples.

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