

## CARBON NANOTUBE RF ABSORBING MATERIALS\*

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### Abstract

The use of carbon nanotubes (CNT's) in an alumina ceramic matrix is experimentally studied. The weight percentage of multi-walled CNT's is varied from 0.5%-10% and the complex permittivity and permeability measured. The RF absorption is strong and consistent for a CNT weight percentage in the range 1%-2.5% in the frequency band 1-40 GHz. The permittivity is observed to increase dramatically with increasing CNT weight percentage as observed elsewhere and in accord with theoretical treatments. The DC conductivity of the CNT-alumina nanocomposite is also sufficient, even at cryogenic temperatures, to drain static charge in particle accelerator environments.

### INTRODUCTION

The choices available for broadband RF absorbing materials compatible with particle accelerator beamline environments are limited, such as for use in higher order mode (HOM) loads. Ferrites and ceramics that have the vacuum, radiation, and particulate compatibility tend to have strong RF absorption over only about a decade of frequency once above 1 GHz. Further, their properties often change considerably when going from room temperature to cryogenic temperatures, as is the case for applications in superconducting RF cryomodules.

Mixing carbon nanotubes (CNT's) into a bulk matrix has recently shown promise for providing a broader band of RF absorption [1-3]. These materials have shown much flatter absorption in the measured frequency bands than traditional RF absorbing materials. Most of these studies have used resins as the matrix, which are unlikely to be vacuum, radiation, or particulate compatible in accelerator applications. A few studies, however, have used alumina and silica as the matrix for the CNT's [4-7], which are common materials in accelerator environments. The silica material has likewise shown encouraging flat RF absorption in the frequency range 8-12 GHz [7]. Presented here are the initial measurements of the RF absorption properties of alumina composites in the broad frequency range 1-40 GHz. A few cryogenic tests of resin materials will also be discussed.

### SAMPLE PREPARATION AND RF MEASUREMENT METHOD

The multi-wall CNT's (MWCNT) nanocomposites were fabricated by first mixing and homogenizing alumina and MWCNT powders. Then, fully dense ceramics were obtained by performing spark-plasma sintering (SPS) in a Sumitomo 1050 SPS apparatus. The nanocomposite samples from the SPS apparatus were typically cylinders 1cm-2cm in diameter and 1cm tall.

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The microwave measurements require the ceramic shapes to be thin "washers" for coaxial measurements in the 1-12.4 GHz frequency range and thin rectangular slabs for waveguide measurements in the Ku (12.4-18 GHz), K (18-26.5 GHz), and Ka (26.5-40 GHz) frequency bands. These shapes were machined from the sintered cylinders using various diamond-embedded grinding tools. A photograph of three such samples is shown in Fig. 1.

The RF measurements utilize the TRL method where the "Line" reference is empty for the calibration sequence of the Agilent E8363B network analyzer. The nanocomposite is then placed inside the Line reference and the measured complex scattering parameters S11, S21, S22, and S12 are used to calculate the permittivity and permeability of the inserted sample [8]. Photographs of the Ka band waveguide and Line reference are shown in Fig. 2.

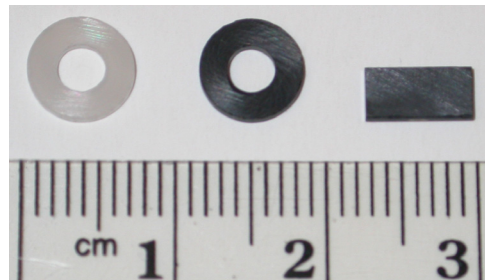


Figure 1: Photograph of alumina-MWCNT nanocomposite samples for RF measurements. The light washer on the left is a control sample with zero MWCNT content.

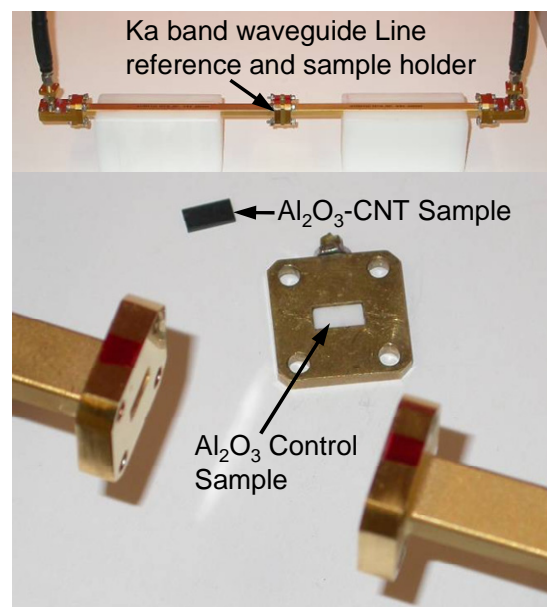


Figure 2: Photographs of the Ka band waveguide and Line reference for the 26.5-40 GHz measurements.

### MEASUREMENT RESULTS

The RF properties of alumina nanocomposites with various weight percentage MWCNT's were first measured in the frequency range 1-12.4 GHz to get an indication of permittivity dependence on MWCNT percentage. From these results a MWCNT weight percentage was selected for RF measurements over the full 1-40 GHz frequency range. The bulk DC conductivity at 293K and 77K was also measured for several samples.

#### RF Measurements 1-12.4 GHz

The real and imaginary parts of permittivity of alumina nanocomposite samples with various weight percentage MWCNT's are shown in Figs. 3 and 4, and shown in Fig. 5 is the loss tangent. There is a dramatic increase in permittivity with increasing MWCNT weight percentage. For the lower MWCNT weight percentages, both the real and imaginary parts of permittivity are relatively flat with frequency. These results are consistent with those seen in studies with resin nanocomposites [1-3]. The measurements in Figs. 3 and 4 have error bars of about  $\pm 25\%$  due to small deviations of the sample shape from the ideal which would completely fill the coaxial waveguide. Since there are no magnetic materials in these samples, the measured permeability shows the expected result of the real part  $\mu'/\mu_o \cong 1$  and the imaginary part  $\mu''/\mu_o \cong 0$ .

The dramatic increase in permittivity with increasing MWCNT percentage is related to the theory of large polarizability enhancement in materials containing small metallic particles [9]. This theory has been verified experimentally [10-12] and advanced to describe inclusions with high aspect ratio [13], as is the case for CNT's. For the high aspect ratio shapes, dramatic polarization effects can occur above the percolation threshold [13].

#### RF Measurements 1-40 GHz

From the measurements in Figs. 3 and 4, a promising broadband RF absorber would have a MWCNT weight percentage in alumina in the range 1%-2%. The results for 1% MWCNT samples across the full frequency range of 1-40 GHz are shown in Figs. 6, 7, and 8. The discontinuities in permittivity at the boundaries of frequency bands are mostly indicative of the measurement error. The pieces for the 1-12 GHz and 26.5-40 GHz bands were machined from one sample, and the pieces for the 12-18 GHz and 18-26.5 GHz bands were machined from a second sample. There is also likely a systematic error slope of permittivity within the frequency bands. This is possibly due to variations of the sample shape from the ideal, which would have a greater effect on measurement error with increasing frequency as the shape perturbation becomes a larger fraction of an RF wavelength. Nevertheless, the 1% MWCNT weight percentage in alumina shows better broadband absorption than any other presently available HOM load RF absorber material. Within a band, though, there do exist materials that have the desirable property of  $\sqrt{\mu_r/\epsilon_r} \approx 1$ .

#### 08 Ancillary systems

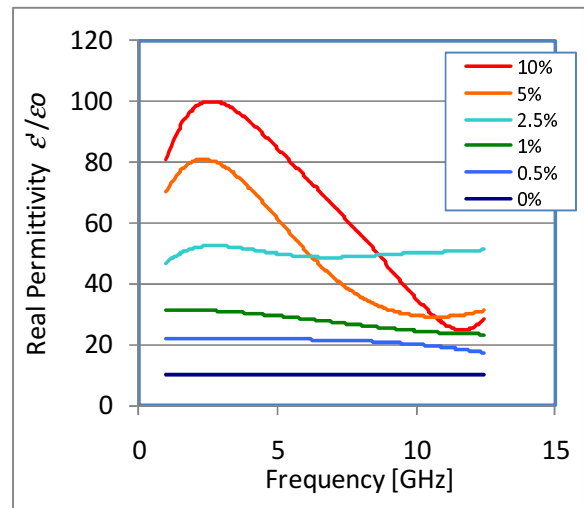


Figure 3: Real part of permittivity for alumina nanocomposites with varying weight percentage of MWCNT's.

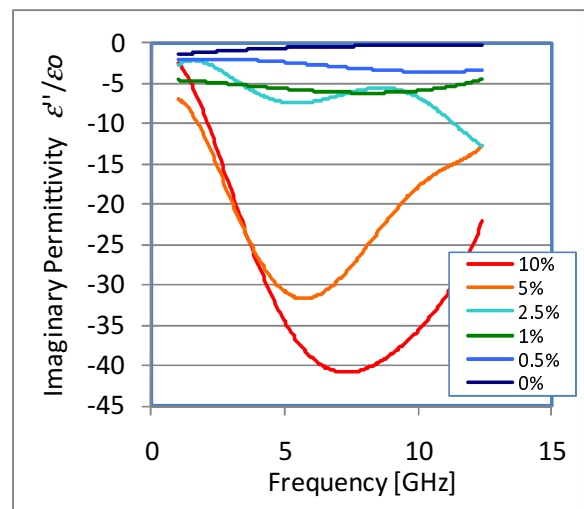


Figure 4: Imaginary part of permittivity for alumina nanocomposites with varying weight percentage of MWCNT's.

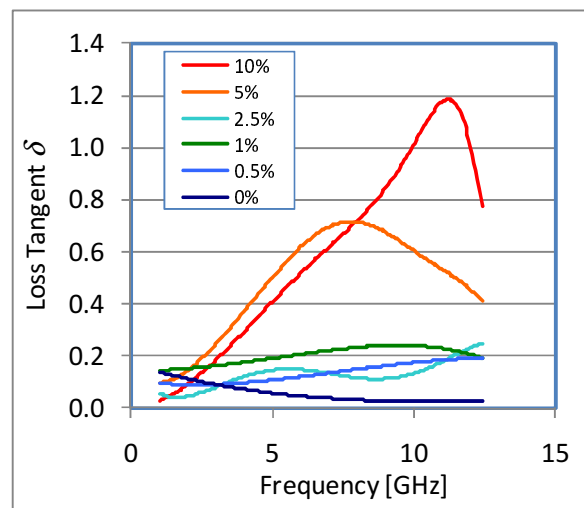


Figure 5: Loss tangent for alumina nanocomposites with varying weight percentage of MWCNT's.

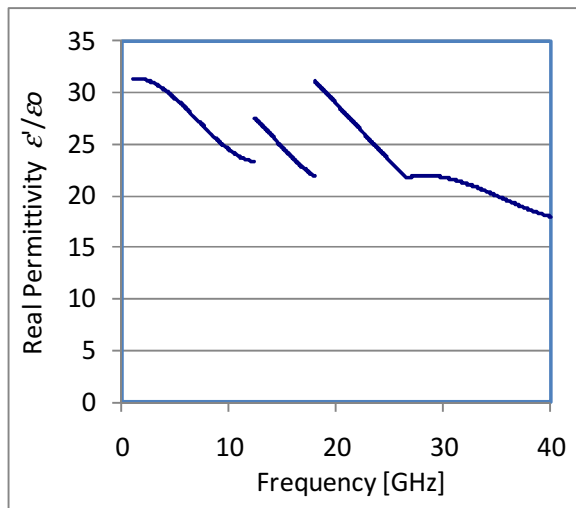


Figure 6: Real part of permittivity in the 1-40GHz range for alumina nanocomposites with 1% weight MWCNT's.

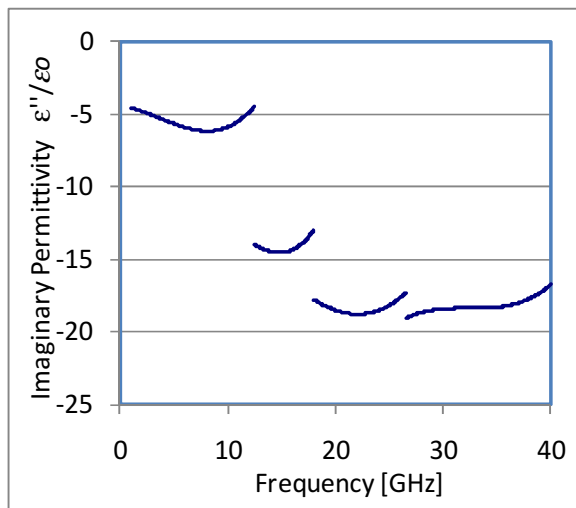


Figure 7: Imaginary part of permittivity in the 1-40GHz range for alumina nanocomposites with 1% weight MWCNT's.

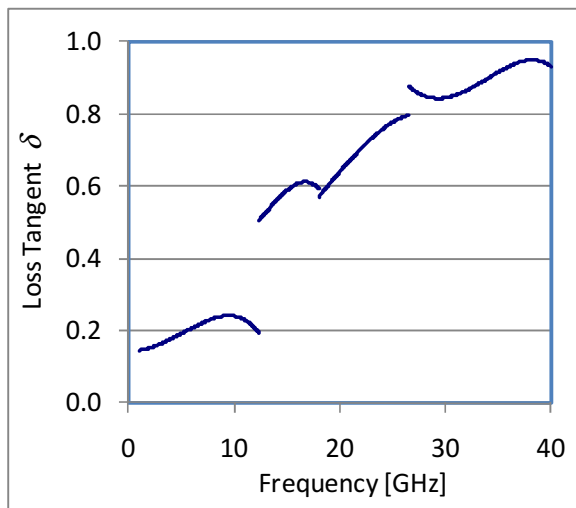


Figure 8: Loss tangent in the 1-40GHz range for alumina nanocomposites with 1% weight MWCNT's.

### DC Conductivity

The DC electrical conductivity of an RF absorber in an accelerator beamline environment must be sufficient to drain static charge that may be deposited by stray particles or generated by x-rays, possibly even at cryogenic temperatures. The dramatic increase in permittivity with increasing MWCNT percentage seen in Fig.3 is indicative of the CNT's being near or above the percolation threshold. A benefit of being near the percolation threshold is that the bulk DC conductivity of the nanocomposite also becomes satisfactory for draining static charge, as statistically significant conduction paths are established by CNT linkages. The time scale for static charge to decay, assuming the case with the longest conduction path where all of the charge is deposited onto the surface of an absorber, is approximately the simple RC time constant

$$RC = \frac{\rho d}{A} \cdot \frac{\epsilon A}{d} = \rho \epsilon \text{ [sec]} \quad (1)$$

where  $\rho$  is the material resistivity [ $\Omega \cdot m$ ],  $\epsilon$  is the dielectric constant [F/m],  $A$  is the area onto which charge is deposited, and  $d$  is the thickness of absorber material to a metallic substrate at ground potential.

The DC electrical conductivity was measured at room temperature and 77K using the alumina nanocomposite samples prepared for the RF measurements. The results for the 1% and 2.5% MWCNT samples are shown in Fig. 9, where the conductivity is acceptable and has minor change when going to cryogenic temperatures. The time scales for static charge to decay at 77K for the 2.5% and 1% MWCNT concentrations are about 0.4 ns and 50 ns, respectively. For the 0.5% MWCNT sample (not plotted in Fig. 9) the conductivity at 293K is  $\sigma = 4.5 \times 10^{-11} (\Omega \cdot m)^{-1}$ , and drops below the instrument resolution when cooled to 77K. This indicates that the 0.5% MWCNT concentration is below the percolation threshold, and has a room temperature static charge decay time scale of about 4 sec. The room temperature static charge decay time is marginal for the 0.5% sample, and likely unacceptable at 77K.

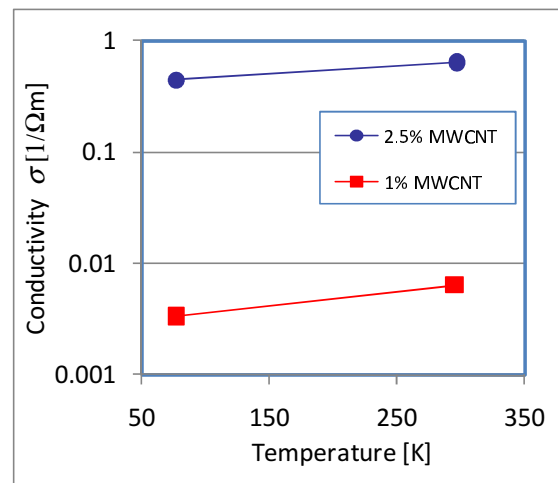


Figure 9: DC electrical conductivity of alumina-MWCNT nanocomposites near the percolation threshold.

## CRYOGENIC RESINS

In the interest of developing a very low cost HOM load utilizing a resin-based RF absorber, as opposed to the robust ceramics discussed in the previous sections, the cryogenic properties of a few candidate resins were explored. The vacuum-compatible epoxy Torr-Seal<sup>®</sup> was cured in a 3mm deep, 30mm x 50mm wide "pond" cut into a copper plate. Upon slowly cooling to 77K, the epoxy fractured significantly. Next, the vacuum sealant Vacleal<sup>®</sup> II was deposited into an identical pond and cured at 80C for 24 hours. This resin shrinks considerably with evaporation of volatiles upon curing, and fractured during curing in this excessively thick application. Vacleal<sup>®</sup> II is intended to be used as a thin film and was found to survive curing and a 77K cooling cycle as a thin film. Unfortunately, the desired RF absorber application will likely require a relatively thick matrix rather than a film. Lastly, the low thermal expansion epoxy Emerson & Cuming Stycast<sup>®</sup> 2850 FT was tested in a copper pond. The Stycast<sup>®</sup> survived curing and a 77K cooling cycle very well, but has a very high vacuum outgassing rate. The outgassing rate would be unacceptable in most particle accelerator environments, especially in close proximity to SRF cavities. Perhaps a thin overcoat could be used to seal the Stycast<sup>®</sup>, but flaking and DC conductivity issues would have to be rigorously tested.

## SUMMARY

The RF properties of alumina ceramic nanocomposites with a weight percentage of MWCNT of 1%-2.5% show promise as a robust broadband RF absorbing material as measured in the 1-40 GHz frequency range. The permittivity is observed to increase dramatically with increasing CNT weight percentage as observed elsewhere and in accord with theoretical treatments. The DC conductivity of these materials is also sufficient to drain static charge in particle accelerator environments at cryogenic temperatures. Cryogenic tests of alternate resin-based materials show that the resins are unlikely to satisfy the requirements of an RF absorber in particle accelerator environments.

Follow-on development of the alumina ceramic nanocomposites will be required to test the RF properties at cryogenic temperatures, to produce larger ceramics for use in an HOM load, and possibly to add a magnetic loss component. The test results to date, nevertheless, show that these materials are good candidates to satisfy all of the stringent requirements of an HOM load RF absorber material in particle accelerator environments.

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