LOSSY BEAM PIPE HOM LOAD CERAMICS WITH DC CONDUCTIVITY*

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Abstract

Ceramic materials used in beam pipes of superconducting RF accelerators as absorbers have the problem of charging due to the electron cloud and secondary electron emission. A novel solution is in the application of conductive nanoparticles to the lossy ceramic. The lossy ceramic is pre-processed to provide for pores that will accept the conductive nanoparticles and then coated with a thin film to prevent the nanoparticles from entering the environment. The same process was also done with sub-micron carbon particles. Measurements of surface conductivity with and without a vacuum compatible sealant are reported on, along with microwave measurements.

BACKGROUND

Carbon coated ceramic rods have been used in helix traveling-wave tubes (TWTs) for many years as attenuators and severs. The ceramic materials used as support rods for the helix were typically AL300 alumina, beryllium oxide (BeO), or anisotropic boron nitride (APBN). APBN had good thermal conductivity and the process of pyrolytically depositing carbon from the cracking of methane gas provided good adherence to the APBN. The process of varying the density of the carbon along the length of the APBN was a bit of an art and the process of measuring the results varied from (1) the resistance measurement of two carefully spaced knife edge probes to, (2) a carefully designed waveguide measurement technique. Innovations to the standard processes included the use of lossy resonators in the form of meander lines deposited on the APBN rods between the helix turns. This created frequency sensitive microwave loss and with careful design choices provided a frequency variable sever [1].

With the improved processing of nanoparticles we proposed to use nickel and copper nanoparticles infused into specially fabricated ceramics (pre-forms) to determine their effectiveness for controlling the conductivity of beam-line ceramics. In addition, experiments were also performed to determine the effectiveness of sub-micron sized carbon particles in these ceramics as well.

Prior work determined the porosity one could achieve with the process of carbon loading the ceramic mixture and firing to create a preform [2]. The preforms were then loaded with Ni and Cu nano-particles and carbon sub-micron particles. Based on the percentage of carbon that was added to the ceramic mixture and the temperature of sintering, the density preform could be varied from 87% to 99.8% as shown in Table 1.

The importance of this process is that any lossy ceramic base, mixed with carbon sub-micron particles and sintered in air, can be used as a preform for the later infusion of materials to create specific values of loss tangents and surface conductivity.

<table>
<thead>
<tr>
<th>Weight of Carbon (%)</th>
<th>Sintering Temperature</th>
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<tbody>
<tr>
<td></td>
<td>1007C</td>
</tr>
<tr>
<td>0.22%</td>
<td>100%</td>
</tr>
<tr>
<td>0.52%</td>
<td>84.75%</td>
</tr>
<tr>
<td>1.50%</td>
<td>84.90%</td>
</tr>
</tbody>
</table>

SURFACE RESISTIVITY WITH A SEALED SURFACE

After the initial firing of the ceramic and carbon mixture, the sintered ceramic has many pores open to the surfaces. It is these pores that are then filled with particles of various types. This is unlike the processing of helix support rods, where the carbon from cracked methane forms micron-sized flakes and is simply allowed to float and adhere to the APBN in the vacuum-processing chamber.

The measure of open pore volume in ceramics is accomplished by simply placing the ceramic preform into water and through capillary action allowing the pores to fill up. The weight gain of the preform in grams is then the cubic centimetres of total volume available for nanoparticles and/or sub-micron particles to reside. The individual pore sizes in the preform are generally the size of the carbon particles that were used to create them with depths on the order of 5 mm from the surface.

It was determined that using nano-particles of copper and nickel were potentially hazardous to the environment. This resulted in the need to seal the surface containing the nano-particles. For this purpose, the application of VacSeal® [3] was used and the change in surface resistivity was measured. The results are shown in Figure 1.

From the data, a thin film of VacSeal® increased the surface resistivity by about an order of magnitude. The uncoated preform infused with nickel nano-particles measured from $10^6$ to $10^7$ ohms/sq. depending on the concentration of the nano-particles. After the application of VacSeal® and allowing it to dry for 24 hours in air, the surface resistivity was again measured. Those results indicated the surface resistivity had increased to $10^7$ to $10^8$ ohms/sq.
Figure 1: Surface resistivity measured before and after the application of VacSeal® to Ni nanoparticles in the ceramic preform.

**RF MEASUREMENTS OF NANO-PARTICLES**

Porous lossy ceramic preforms 1 cm diameter by 1 cm long with a measured dielectric constant of about 5 were then infused with nano-particles, dried and coated with VacSeal®. The low dielectric constant is partially due to the 90% porosity figure for the preforms used in these experiments. In Figure 2 the microwave cavity measurements at 1.5 GHz are shown for preforms loaded with nano-particles.

Figure 2: Loss Tangent measured at 1.5 GHz for Ni and Cu nano-particles sealed into ceramic preforms.

From these measurements the loss tangent of the porous ceramic with nano-particles could only be varied in the range of .008 to .012. An additional process that may increase the measured loss tangent is a reduction firing of the nano-particles. This method was proposed, and could be implemented, by the suppliers of the nano-particles: Applied Nanotech.

Figure 3: Surface resistivity as a function of the percentage by weight of carbon in water to form the slurry.

**SUB-MICRON CARBON IN THE CERAMIC**

This section discusses the results of loading the porous ceramic with micron sized particles rather than the nano sized particles discussed previously.

**Figure 3**

Surface Resistivity of sub-micron carbon in ceramic

**Figure 4**

Loss Tangent measured at 1.5 GHz for Carbon sub-micron particles in ceramic preforms.

To create loss in the ceramic, the carbon particles were suspended in water and dispersed by ultrasonic agitation. Into this slurry the ceramic preforms were immersed for 5-10 minutes, sufficient time for the slurry to be drawn into the open surface pores by capillary action and lodge the sub-micron particles in place. The samples were then air dried for 24-48 hours and the surface resistivity measured. The surface of the ceramic was then cleaned of loose carbon so that only the filled pores were part of the measurements. An example of the preforms used is shown in Figure 5. The RF measurements were made in the same fixture as the nano-particle measurements.

Figure 3 shows the measured surface resistivity as a function of the ratio of the weight of carbon particles to water mixture; Figure 4 the loss tangent. The ceramic preform was measured to be about 92% dense.

For the purpose of these experiments, the same batch of carbon sub-micron particles was used in the making of the lossy ceramic as was used in the creation of the porous
ceramic. The sub-micron particles are roughly two to three orders of magnitude larger than the nano-particles used previously. Thus, the pore size (created during the ceramic sintering process) and the lossy particle size are pretty much equivalent.

DISCUSSION

The carbon nanoparticles in the same class of ceramic preforms produced loss tangents of up to 3 times that of the Ni and Cu nano-particles. In addition, the spread of loss tangent values seemed more controllable with carbon concentrations. From these measurements at 1.5 GHz the loss tangent of the porous ceramic with nano-particles could only be varied in the range of .008 to .012. While the loss tangent with carbon sub-micron particles could be varied from .011 to .027. (An additional process that may increase the loss tangent of the nano-particles was the previously mentioned reduction firing proposed by Applied Nanotech.)

The handling of nano-particles required sealing the ceramic with a coating that increased the surface resistivity. With carbon sub-micron particles it may be possible to work with surfaces that do not need this additional coating. As discussed in the background section of this paper, pyrolytic carbon was deposited on various types of ceramics in helix support rods in the microwave tube industry. These helix TWTs performed for many thousands of hours without degradation of the lossy carbon films and were not coated with a sealant.

Figure 5 shows the dielectric wedge used to measure the broadband characteristics of the lossy ceramic in JLAB studies at cryogenic temperatures. Also, pictured is the cylindrical ceramic preform used in the 1.5 GHz cavity measurements. The surface discoloration is the residue from the slurry mixture of carbon and water and is one of the issues to be examined in further studies: should a grinding operation be performed after the infusion of particles into the ceramic preform?

FURTHER STUDIES

In the Phase II proposal it was proposed to add an additional firing cycle to the nano-particle sourced loss to reduce any oxides that may have formed on the nano-particles (a rather common occurrence in practice). Because of potential nano-particle hazards, the supplier (Applied Nanotech) would perform the reducing firing process on the lossy ceramics.

Since the nano size of the particles would create handling problems at the labs, it seems possible that sub-micron to micron size carbon particles offer a good solution. The distribution of the size of the pores depends on the distribution of the size of the carbon used to create them. In this study sub-micron particles were used. Larger size distributions are available and should be examined in the same manner.

Further experimentation with different ceramic materials, differing pore size, and air versus other atmospheres firing techniques should be performed to determine the limitations of this method.

CONCLUSION

Use of nano-particles for loading porous ceramics presented some handling problems. Sealing the surface with VacSeal® solved the handling issues but created an increase of the surface resistivity by about an order of magnitude. Carbon sub-micron sized particles, initially used to create the porosity in the ceramic, proved to be as good if not better than nano-particles in creating surface resistivity. The loss tangent and surface resistivity measurements of carbon infused ceramics offered a wide range of operating conditions. The measured loss tangents of carbon particles were up to three times greater than the nano-particles infused into the same porosity ceramics. Lifetimes of carbon coating on ceramics in a vacuum are well documented in the stability of helix support rods in helix TWTs.

ACKNOWLEDGEMENTS

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REFERENCES

