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MEASURING THE SHIELDING EFFECTIVENESS OF SUPERCONDUCTIVE COMPOSITES

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Abstract

The ability to cool superconductors with liquid nitrogen instead of liquid helium has opened the door to a wide range of research. The well known Meissner effect, which states superconductors are perfectly diamagnetic, suggests shielding applications. One of the drawbacks to the new ceramic superconductors is the brittleness of the finished material. Because of this drawback any application which required any flexibility would be impractical. Therefore, this paper presents the results of a preliminary investigation into the measuring of shielding effectiveness of $YBa_2Cu_3O_{7-x}$ both as a composite and as a monolithic material. A flanged coaxial test fixture was selected to measure the shielding. The composite samples showed little or no shielding. The monolithic sample showed substantially less shielding than predicted. Possible explanations for this lack of shielding are discussed.

Introduction

The recent advent of higher critical temperatures in superconductors has made it practical to investigate them for a wide range of applications. One application, suggested by the Meissner effect, is to use superconductors as a means of shielding. The Meissner effect simply states that a magnetic field is excluded from a superconductor. Thus, a monolithic superconductive shield should exclude magnetic interference as well as have infinite conductivity unlike copper shields. However, due to the brittleness of the ceramic high temperature superconductors, most applications would be impractical. An alternative might be to load a plastic with superconductive particles, thus providing the flexibility required. As with the investigation of most new materials, superconductive composites pose a new set of measurement problems. This paper presents the results of an investigation into measuring shielding effectiveness of superconductive composites, as well as monolithic samples.

There are many well established techniques for measuring the shielding effectiveness of different materials [1, 2, 3]. Unfortunately none of the techniques address the problems associated with superconductive shields. The most difficult problem is to keep the sample below it's critical temperature, without altering the electrical characteristics of the test. The test method selected was the split coaxial line. With relatively few modifications, which will be discussed later, it was adapted for superconductive samples.

Test Methods

A flanged coaxial holder design was selected for measuring electromagnetic shielding, and like many shielding effectiveness test methods, it is based on an insertion loss type measurement. Figure 1 shows a diagram of the fixture.

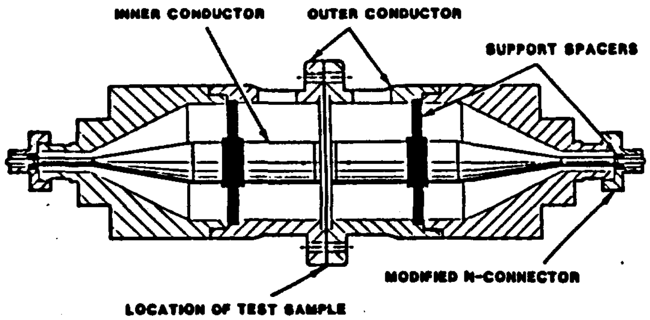

Figure 1. Flanged Coaxial Test Fixture.

A flanged coaxial fixture relies on displacement currents as opposed to conduction current. The disadvantage of this fixture is the extra measurement steps which must be taken to compensate for the perturbation of the transmission line caused by the insertion of the sample. This measurement method is usually used between 10 MHz and 1 GHz. Below 10 MHz the perturbation in the transmission lines are too great to be easily compensated and above 1 GHz the wave mode starts to change. The National Institute of Standards and Technology (formerly National Bureau of Standards, NBS) has proposed the standardization of this fixture.[1]

Design Considerations

Several modifications of the original NBS design were required to accommodate immersing the test fixture in liquid nitrogen. The fixture could have remained essentially unchanged if complete sealing to prevent entry of liquid nitrogen could be ensured. However, since the sample itself would form part of the seal, it was decided that this would not be feasible. The other possibility was to allow free access of the nitrogen to all interior sections of the fixture. Accordingly slots were made in the outer conductor and holes drilled in the

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inner conductor to allow free passage of the nitrogen. The slots were made small enough so that electrically the fixture was unchanged, yet large enough so that the gaseous nitrogen could escape.

Liquid nitrogen has a relative dielectric constant of about 1.45 which necessitated making the center conductor smaller to maintain the nominal 50 ohm impedance. Teflon™ was selected for the center conductor supports because its electrical and physical properties were suitable and readily available for low temperatures. The dimensions of the supports were then calculated to maintain a constant capacitance for each cross section of the fixture.

Clearance between the center conductor and the supports is required to accommodate the difference in expansion as the fixture cools to it's working temperature. The supports were expected to shrink away from the outer conductor but this was not considered to be a problem. During the machining of the test fixture the conical inner conductor sections were purposefully left oversize. The fixture was then tuned using a time domain reflectometer (TDR). The TDR reflections showed large discontinuities at the support cross sections before lowering into the liquid nitrogen. As the fixture cooled these reflections disappeared and the impedance differences in the conical sections were revealed. The geometries of these sections were adjusted until the impedance differences were less than about one ohm. The end connectors caused impedance mismatches which appeared to be caused by nitrogen not flowing past the Teflon end support buttons. Additional holes in the connectors eliminated this problem.

Processing of Samples

Powder Synthesis

The fabrication process begins with a well characterized $YBa_2Cu_3O_{7-x}$ (herein designated YBCO) powder. Powder synthesis may be carried out by a variety of methods, and the mixed oxide route was chosen in this study. Starting materials of $BaCO_3$, Y_2O_3, and CuO were mixed in stoichiometric amounts and pressed into disks for calcination (reaction at high temperatures). In this study, the calcination temperatures were between 900 and 950°C for durations of 16 to 48 hours, and the disks were crushed after firing. Powder calcination was a complex process due to low melting eutectics and residual $BaCO_3$, and several heat treatments were necessary to obtain the proper YBCO phase.[4] The procedure was repeated to produce a phase pure powder as judged by x-ray powder diffraction patterns. The final particle size and morphology will affect all of the subsequent processing operations. Milling operations were carried out to achieve the desired particle size and distribution. In this study,
the calcined powder was milled to a median particle size in the 4-7 μm range.

Plate Processing

Plates, 5 inch square, have been made by pressing powder at 4000 psi, and great care was taken to achieve uniform powder packing. Powder was added to an acrylic/stearic acid solution, and the mixture was dried to a hardened mass. Granules were made by grinding and sieving and were produced so as to facilitate material flow in the die.

The processed plates were heat treated by slowly increasing the temperature in the furnace to 240°C so as to effect a slow binder burnout. This allowed the organic binder to be volatilized without cracking the sample. The furnace was again ramped in temperature to 900°C to densify the plate. Finally, the plate was O_2 annealed at 435°C to incorporate sufficient O_2 into the lattice to render the orthorhombic superconducting phase.

Superconductivity was verified by levitating a magnet, and the sample was subsequently machined to fit the measurement apparatus.

Composite Samples

The YBCO particles were mixed with two different plastics, Ethylene-Vinyl Acetate (EVA) and Polychorotrifluordethylene (KEL-F). The KEL-F™ was selected for its low glass transition temperature and the EVA was selected because of its low processing temperature. Each plastic was loaded with superconductive particles on a plastics mill. The KEL-F processed at 450°F and the EVA at room temperature. However, due to the shearing stresses built up in the EVA during the milling, its temperature reached 150°F. The KEL-F samples were loaded to 10%, and 30% by weight and the EVA samples were loaded to 10%, 30% and 80% by weight. A fourth EVA sample was loaded with 70% YBCO and 9% carbon black. The addition of the carbon black increased the press temperature to 300°F. All samples were pressed into .040" thick plates for testing in the flanged coaxial holder.

Electromagnetic Shielding Effectiveness

The electromagnetic shielding measurements showed no shielding from any of the composite samples except the sample loaded with 70% superconductor and 9% carbon black. This sample's shielding effectiveness is shown in Figure 2.

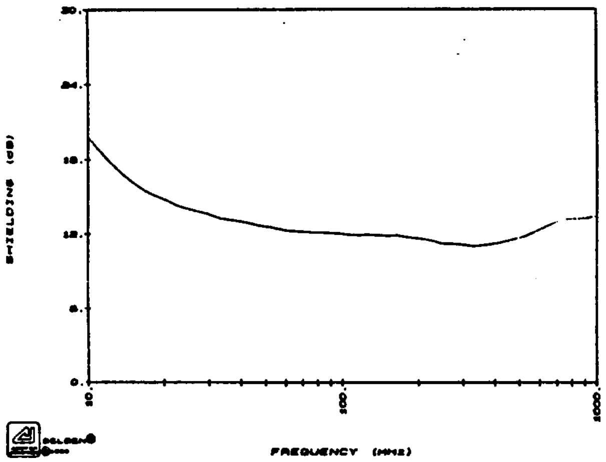

Figure 2. Shielding Effectiveness of a Sample of EVA Loaded with 70% Superconductive Powder and 9% Carbon Black

This meager 12 dB of shielding was first wrongfully attributed to the carbon black. An additional sample was loaded exclusively with carbon black in the same percent loading. This sample's shielding effectiveness was measured in liquid nitrogen and only 1 dB of shielding was seen. A large change in

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KEL-F is a Registered Trademark of 3M

shielding was also seen when the 70% superconductive and 9% carbon black sample was cooled past its critical temperature. This also indicates superconductive shielding.

The plate of pure YBCO was extremely difficult to mount in the coaxial test fixture. Part of the mounting problem was due to the brittle nature of the superconductor. The other part of the mounting problem stems from the curved shape which the sample forms after firing. The curved sample was flattened through a grinding process, which helped the mounting problem. However, a spacer was still used to prevent crushing the sample. The shielding measured is shown in Figure 3.

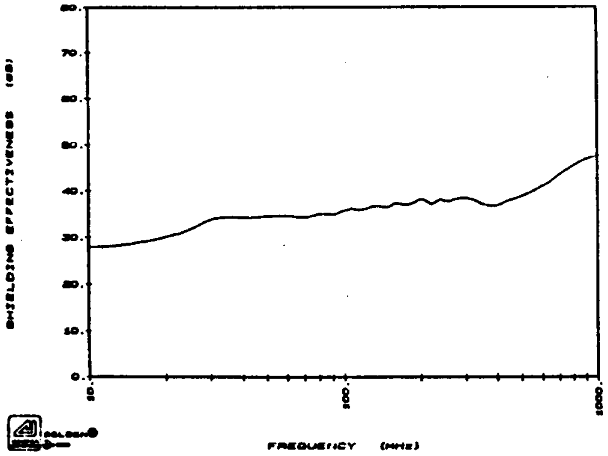

Figure 3. Shielding Effectiveness of Monolithic Sample of YBCO.

The shielding in Figure 3 was less than predicted. This might be attributed to several things.
Great care was taken to achieve uniform powder packing when the plate was pressed. However, any non-uniformity in the finished crystalline structure would manifest itself as a micro or macroscopic crack. These cracks have the potential to "leak" electromagnetic energy. Several cracks were observed in the pure YBCO plate used.

Another possible cause for the low shielding measurement is a high surface impedance of the bulk YBCO ceramic. One study has shown that the bulk ceramic had a higher surface resistance at 150 GHz than the thin film, and both samples had significantly higher surface resistances than theoretically predicted.[5] The discrepancy between experimental results and theory was attributed to surface inhomogeneities and second phases. It is suggested that these extrinsic effects may also play a role in the far-field shielding effectiveness of YBCO.

The difficulty in measuring the shielding in materials with a high surface impedance is most easily seen in the equation derived in reference 2; as follows:

$$\mathrm{S.E.}_{dB} = 20\ \mathrm{LOG}\ 1 + \frac{Zo}{2(Z_L + Z_C + Z_D)}$$

Where Z_o and Z_L are the characteristic impedance of the test fixture and the impedance of the test sample, respectively. Z_C and Z_D are the surface impedances between the fixture and the sample. It is easily seen from the above equation that if Z_C and Z_D are large they would dominate the measurement. It is expected that the Z_L term would be small for a superconductor.

Conclusion

There appears to be several problems associated with measuring the shielding of high T_c superconductors. The most obvious difficulty is their extreme brittleness and low critical temperature. The results from the flanged coaxial test fixture indicate that a method which is not dependent on surface impedance might give better results.[6] The lack of shielding seen in the composite samples needs to be investigated further. However, they should not be ruled out for possible shielding materials in the future.

Acknowledgements

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