Conf-8811129

SHIELDING EFFECTIVENESS OF SUPERCONDUCTIVE PARTICLES IN PLASTICS

Thomas Pienkowski, John Kincaid
Belden Wire and Cable
Technical Research Center
Geneva, Illinios

M.T. Lanagan, R.B. Poeppel, J.T. Dusek, D. Shi, and K.C. Goretta
Argonne National Laboratory
Argonne, Illinois

CONF-8811129--1

DE89 003888

September 1988

To be presented at the Wire and Cable Symposium, November 15-17, Reno, Nevada

* Work was supported by the U.S. Department of Energy, BES Materials Sciences, under Contract W-31-109-Eng-38 and the Defense Advanced Research Projects Agency.

MASTER

SHIELDING EFFECTIVENESS OF SUPERCONDUCTIVE PARTICLES IN PLASTICS

Thomas Pienkowski, John Kincaid
Belden Wire & Cable
Technical Research Center
Geneva, Illinois

M.T. Lanagan, R.B. Poeppel, J.T. Dusek, Donglu Shi, K.C. Goretta
Argonne National Laboratory
Argonne, Illinois

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 flexibility (e.g. wire and cable) would be impractical. Therefore, this paper presents the results of a preliminary investigation into the shielding effectiveness of $YBa_2Cu_3O_{7-x}$ both as a composite and as a monolithic material. Shielding effectiveness was measured using two separate test methods. One tested the magnetic (near field) shielding and the other tested the electromagnetic (far field) shielding. No shielding was seen in the near field measurements on the composite samples, and only one heavily loaded sample showed some shielding in the far field. The monolithic samples showed a large amount of magnetic shielding.

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 would exclude magnetic interference as well as have infinite conductivity unlike copper shields. However, due to the brittleness of the ceramic high temperature superconductors, a cable shielded with this material would be impractical in most situations. An alternative might be to load a plastic with superconductive particles. This paper presents the results of a preliminary investigation into the far field electromagnetic shielding and the near field magnetic shielding of $YBa_2Cu_3O_{7-x}$ superconductors both mixed in a plastic matrix and as a pure ceramic.

Test Methods

Three different test methods were used, one testing far field shielding, one testing near field magnetic shielding, and the other testing for the presence of Meissner effect. A Superconducting Quantum Interference Device (SQUID) magnetometer was used to measure the flux expulsion or Meissner fraction.

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. 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. The National Bureau of Standards (NBS) has proposed the standardization of this fixture and it is documented in several papers.[1,2]

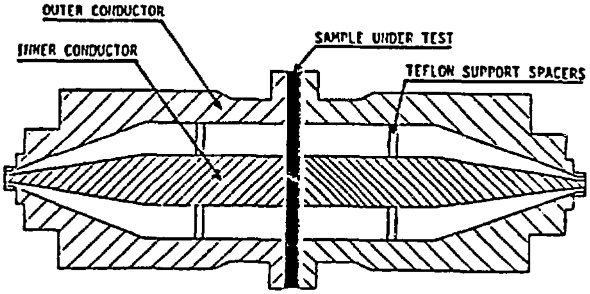

Figure 1. Flanged Coaxial Test Fixture

The NBS design for the flanged coaxial fixture was modified to allow for the liquid nitrogen cooling that is required. To allow for the boiling of the liquid nitrogen, small slots were cut through the outer conductor. This was also done in several other locations to ensure sufficient ventilation for the escaping nitrogen gas. The alternative to cutting slots in the outer conductor was to make the fixture "air tight". For safety reasons this was not attempted because of the large vapor pressure of liquid nitrogen.

The other major modification to the NBS design was to compensate for the liquid nitrogen dielectric. Liquid nitrogen has a relative dielectric constant of 1.45. To maintain a 50 ohm coaxial test fixture with this dielectric, the ratio of the inner to outer conductor was changed. It was decided to reduce the size of the inner conductor to maintain the 50 ohm impedance.

The magnetic shielding test apparatus is loosely based on the Helmholtz coil, ASTM A698 Vol. 3.04. It consists of a powered coil and a pick-up coil. The powered coil was driven by an amplifier and was swept in frequency from 10 Hz to 10 kHz. This coil produced a 22 gauss magnetic field. The pick-up coil was placed inside the powered coil and was centered for maximum output voltage. The shielding material was placed around the pick-up coil and was usually tube shaped. The ratio of the voltage generated by the shielded coil to the voltage generated by an unshielded coil is the shield effectiveness.

Processing of Samples

Composite Samples

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[3]. 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.

The YBCO particles were then 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. These samples were then pressed into .040" thick plates for testing in the flanged coaxial holder, and also rolled into tubes for the low frequency magnetic shielding test fixture.

Monolithic Tubes

Ceramic tube fabrication requires that the YBCO powder be mixed with a set of organics. A solvent provides the basic vehicle into which the oxide powder and other organics are placed. Care must be taken in selecting a solvent that is compatible with the YBCO powder and other organic constituents, and common solvents include methyl ethyl ketone, methanol, or xylene. Dispersants are utilized to deflocculate the inorganic particles in the solvent, and to assist in obtaining higher green (unfired) densities. Binders impart strength to the green body, and plasticizers allow for greater flexibility by reducing the glass transition temperature of the binder. In extrusion, the plastic mass was forced through a small aperture. Wire with radii between 0.3 and 1.5 mm have been manufactured in 12 to 20 cm lengths. In this study 6" long tubes were formed with a .190" inside diameter and a .040" wall. These tubes were used to measure shielding effectiveness in the near field test apparatus.

The heat treatment schedule for fabricated shapes is divided into three basic sections. Initially, a slow increase in temperature is required to remove organics from the green body. In this study, organics were volatilized below 350°C. During sintering a well calcined powder will have a liquid phase that has an onset temperature between 930 and 960°C[4]. The tubes were heated in oxygen to 950°C in order to sinter the powder to high density. The final step is an annealing procedure to incorporate oxygen into the YBCO lattice to form the superconducting phase. The relationship between oxygen content and phase transition to the superconducting orthorhombic phase has been studied extensively[5].

Monolithic Tapes

The tape casting process requires that the YBCO powder be mixed with a set of organics, which exactly parallels the ceramic tube fabrication. However, instead of being extruded, the tape cast samples were made by a doctor blade traversing over a glass plate to form a uniform layer. The tape was removed from the glass plate, and was very flexible in the unfired state owing to the

---

KEL-F is a Registered Trademark of 3M

organics incorporated in the cast material. The heat treatment schedule also parallels that of the tube fabrication including the final step of oxygen annealing to obtain the superconducting phase. The superconducting tapes were approximately 4" in diameter and .012" thick. These were used in the flanged coaxial test fixture for shielding measurements.

Results

Meissner Fraction

The Meissner fraction testing was performed on three composite samples and on the powder prior to mixing. The YBCO powder consistently showed a large Meissner effect. The three composites tested were the 10% and 30% samples loaded in KEL-F and the 80% sample loaded in EVA. The two KEL-F samples showed no Meissner effect. This is probably due to the lack of sensitivity in the SQUID and not due to the high processing temperature of the KEL-F.

The EVA sample which was loaded with 80% superconductive powder showed a large Meissner effect as seen in Figure 2. The horizontal scale is in °K and the vertical scale is in arbitrary units.

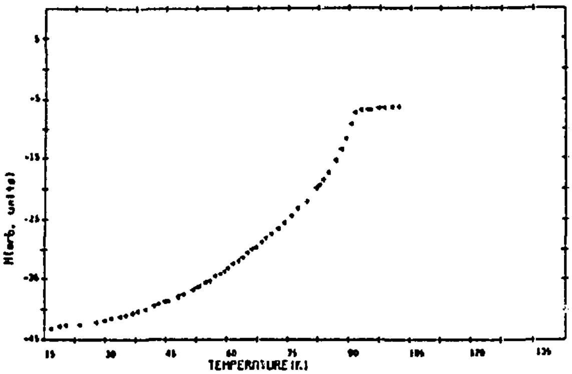

Figure 2. Meissner Fraction of 80% Loaded EVA

Magnetic Shielding Effectiveness

The magnetic shielding effectiveness testing showed no shielding from any of the composites. This might indicate that the plastics were not loaded heavily enough or that the particles were not properly aligned.

The shielding test on the monolithic superconductive tubes showed better than 60 dB of shielding in some frequency ranges. The shielding from 10 Hz to 20 kHz is shown in Figure 3. Shielding is in decibels and the horizontal frequency scale is logarithmic. Notice that the superconductor provided no shielding under 300 Hz. This shows that the field strength which causes the superconductor to become non-shielding is frequency dependent. Also shown in Figure 3 is the shielding obtained from a .048" wall copper pipe and a .006" wall steel pipe. (These two measurements were made at room temperature.) At this input power level the noise floor is around 60 dB. This implies that the shielding is actually better than 60 dB however more sensitive equipment is needed to determine it.

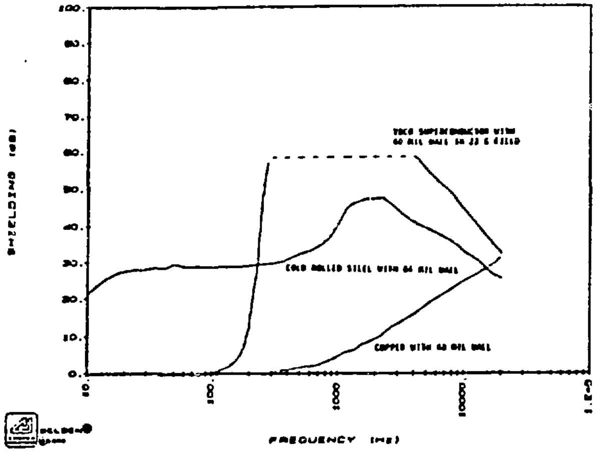

Figure 3. Magnetic Shielding Effectiveness for Copper, Cold Rolled Steel and YBCO Superconductor

As the input power level is decreased, the superconducting tube becomes superconductive at a lower frequency as seen in Figure 4. Unfortunately, as the power level is decreased, the noise floor is also decreased until only 30 dB of shielding can be seen. This, again, is not to say that this superconductive shield only provides 30 dB of shielding, it simply says the equipment is limited to 30 dB at this input power level. Figures 3 and 4 show the test results of a single tube of YBCO. Another tube has been tested and similar results were obtained.

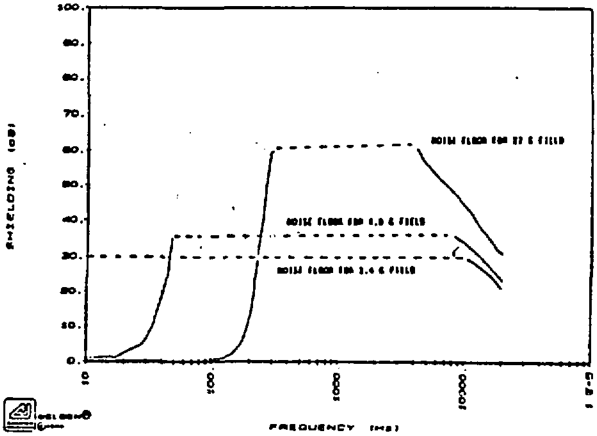

Figure 4. Minimum Magnetic Shielding of YBCO Superconductor in Different Field Strengths

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

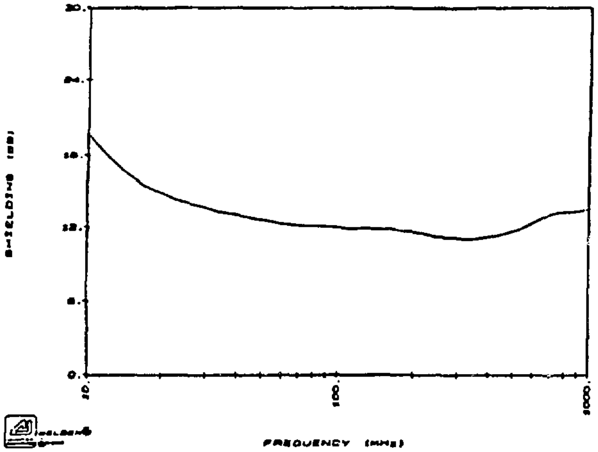

Figure 5. Shielding Effectiveness of a Sample of EVA Loaded with 70% Superconductive Powder and 9% Carbon Black

The pure YBCO tape cast sample was extremely difficult to mount in the flanged coaxial test fixture. Part of the mounting problem was due to the brittle nature of the superconductor. This problem is accentuated by the thinness of the sample. The other part of the mounting problem stems from the curved shape which the tape cast sample forms after firing. Because of this lack of "flatness" only an approximate shielding effectiveness measurement was attempted. This approximate method used a spacer ring to keep the flanges of the test fixture from crushing the sample. Unfortunately, the tape cast sample mounted in this manner showed no shielding. This result was somewhat unexpected given the relatively large amount of shielding seen by the magnetic shielding test fixture. Further work is presently being pursued to evaluate this apparent anomaly.

Conclusion

The monolithic YBCO superconductors provide a large amount of magnetic shielding. By weight or volume they provide more shielding than copper or cold rolled steel. The lack of electromagnetic shielding from the tape cast samples is as of yet unexplainable. Thicker test samples might allow direct mounting of the sample thus eliminating one source of error. There appears to be several problems with superconductive shields. One problem is the small critical magnetic field which the YBCO superconductors appear to have. Other problems include extreme brittleness and low critical temperatures. Shielding from composites with YBCO needs more development but should not be ruled out as a possible shielding material for the future.

Acknowledgements

The authors gratefully acknowledge Dave Johnson for his expertise in the manufacturing of the NBS test fixture. The work was supported by the U.S. Department of Energy, Office of Energy Storage and Distribution, and the BES-Materials Sciences, under Contract W-31-109-ENG-38.

References

1. Adams, J.W., Vanzura, E.J., Shielding Effectiveness Measurements of Plastics. National Bureau of Standards NBSIR 85-3035, 1986, January.

2. Ondrejka, A.R., Adams, J.W. Shielding Effectiveness (SE) Measurement Techniques. 1984 IEEE National Symposium on Electromagnetic Compatibility, San Antonio, Texas. 1984 April 24-26, 249-256.

3. S. Pejovnik. R.L. Porter, A.I. Kingon, T.M. Hare, and H. Palmour III, Proc. Int'l. Symp. on Sci. and Tech. of Sintering, Tokyo, Japan, Nov. 1987; Elsevier, (1988).

4. A.I. Kingon, S. Chevacharoenkul, S. Pejovnik, R. Velasquez, R.L. Porter, T.M. Hare, and H. Palmour III, Proc. Conf. on High T_c Superconductors, Chapel Hill, (1987).

5. H. Strauven, J.P. Loquet, O.B. Verbeke, and Y. Bruynseraede, Solid State Communications 65, pp. 293-296 (1988).

Biographies

Thomas Pienkowski
Belden Wire & Cable
Technical Research Center
2000 S. Batavia Avenue
Geneva, IL 60134

Thomas Pienkowski is presently working on the shielding effectiveness of superconductors and their applications. He received his MSEE from Ohio State University and his BSEE from Iowa State University. He is presently working as a Product Development Engineer with Belden's Electronic Division.

John Kincaid is Manager of Advanced Electronic Product Development at Belden's Technical Research Center in Geneva, Illinois. Mr. Kincaid is a member of the IEEE and is active in the EMC Society TC-4 Working Group on Shield Effectiveness. John Kincaid received the MSEE and BSEE degrees from the University of Oklahoma.

Dr. Michael T. Lanagan is an Assistant Ceramist at Argonne National Laboratory. His research interests include fabrication and electronic characterization of oxides. He received a BS in Ceramic Engineering at the University of Illinois at Urbana and a Ph.D. in Ceramic Science from Penn State University.

Dr. Roger B. Poeppel is a graduate of Cornell University, Ithaca, New York where he received a Bachelor of Engineering in Physics and a Ph.D. in Materials Science and Engineering. At Argonne National Laboratory, he is Manager of the Ceramics Section in the Materials and Components Technology Division. He is responsible for ceramic fabrication development and for the testing of ceramics for a wide variety of applications.

Joseph T. Dusek has worked in the field of ceramic processing and product development for nearly 50 years, the past 25 of which have been spent at Argonne National Laboratory. Prior to joining Argonne he held positions at the Armour Research Foundation and the IIT Research Institute.

Dr. Donglu Shi is a member of the Materials Science Division of Argonne National Laboratory. He received a Ph.D. in Materials Science from, and subsequently did post-graduate research at, the University of Massachusetts. He has concentrated on processing and properties high-T_c and A-15 superconductors

Dr. Kenneth C. Goretta is an Assistant Ceramist in the Materials and Components Technology Division at Argonne National Laboratory. His research has focussed on the mechanical properties and fabrication of oxide ceramics. He received a BS in Metallurgical Engineering from the University of Illinois at Urbana and Ph.D. in Metallurgical Engineering from the Illinois Institute of Technology.