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VACUUM IMPREGNATION WITH EPOXY OF

LARGE SUPERCONDUCTING MAGNET STRUCTURES*

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ABSTRACT

The Lawrence Berkeley Laboratory (LBL) has been developing a new generation of superconducting magnets which have the helium cooling system as an integral part of the magnet structure. The LBL technique calls for large sections of the magnet structure to be vacuum impregnated with epoxy. The epoxy was chosen for its impregnation properties. Epoxies which have good impregnation characteristics are often subject to cracking when they are cooled to cryogenic temperatures. The cracking of such an epoxy can be controlled by: (1) minimizing the amount of epoxy in the structure; (2) reducing the size of unfilled epoxy spaces; and (3) keeping the epoxy in compression.

The technique for using the epoxy is often more important than the formulation of the epoxy. The LBL vacuum impregnation and curing technique is described. Experimental measurements on small samples of coil sections are presented in the paper. Practical experience with large vacuum impregnation superconducting coils (up to two meters in diameter) is also discussed.

INTRODUCTION

The Lawrence Berkeley Laboratory has been building a series of thin solenoid magnets for use in high energy physics experiments.^{1,2,3} The LBL thin magnet concept integrates a superconducting coil, a shorted secondary and a tubular cooling system into a single coil package. The magnet coil is vacuum impregnated with a low viscosity epoxy which, after casting and curing, forms an integrated crack resistant package.

This paper talks about the engineering realities of potting large superconducting magnet structures. The epoxy LBL has chosen was selected for its vacuum impregnation properties rather than its thermal contraction properties. The LBL experience suggests that the fabrication technique could be more important to the success of large coils impregnated with epoxy than the formulation of the epoxy itself. The apparent success that we have had building solenoids up to two meters in diameter (no training up to the coil critical current) shows that this view may have merit. We describe the LBL epoxy formulation, the technique for using the epoxy, and the LBL epoxy casting and curing method in large coils.

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THE LBL EPOXY FORMULATION

The epoxy formulation used by LBL was developed about 10 years ago to vacuum impregnate large room temperature magnet coils. The successful vacuum impregnation of large coils not only requires the proper epoxy but it also requires proper casting and curing technique. The general properties of the epoxy used by LBL are:

1. Low viscosity (~ 500 centipoise at 25°C).
2. Long pot life (~ 4 hours at 50°C).
3. Good wetting power.
4. A reasonable cure schedule and temperature (24 hours with a maximum temperature at 80°C).
5. Good crack resistance. (This applies at room temperature.)
6. Good vacuum properties. (It has been checked down to 5×10^{-6} torr.)

There are a number of formulations which yield the properties listed above. The formulation used for the LBL test coil epoxy is:

1. 50 parts by weight of unmodified low-viscosity resin (EPON 826),
2. 50 parts by weight of polyglycol diepoxide resin (Dow Chemical Company DER-736),
3. Silicone antifoam fluid - about 1 drop per kilogram of mixture (Dow Chemical Company DC-200), and
4. 28 parts by weight aromatic - amine hardener (TONOX, Haugatuc Chemical Division of Uniroyal).

Other formulations are suggested in Reference 4. The formulation given here is the only actually used in our test coils. The formulation is important, but mixing technique is just as important. The procedure used by LBL is as follows:

1. Mix the two resins together (the EPON 826 and DER-736) and heat to about 45°C . Deaerate prior to Step 3.
2. Meet the TONOX slowly at 80°C . When the TONOX is melted, strain it through a cheese cloth strainer and cool down to about 45°C .
3. Pour the TONOX mixture into the resin mixture slowly while stirring with a mechanical stirrer which stirs in as little air as possible. Stir at a temperature of $40-45^{\circ}\text{C}$. Continue stirring until the mixture is completely mixed.

Once the epoxy has been mixed it is deaerated at a temperature of $40-45^{\circ}\text{C}$. Deaeration of the mixture is accomplished by pumping on the mixture until large bubbles cease to form. Deaerate down to a pressure of $200\mu\text{m}$ or lower. In order to have the maximum pot life while pouring, much of the

deaeration should occur before mixing the resin and the TONOX. The pot life of the resulting mixture will be about 4 hours (at 50°C). At 45°C the pot life can be extended to about 6 hours. Note that the pot life varies according to ambient temperature and the size of the batch. For more information see Reference 4.

SMALL SAMPLE TESTS

LBL has not, in general, gone through the extensive cryogenic epoxy studies other laboratories have.^{5,6,7} However, measurements of cracking resistance and total thermal contraction coefficients have been made at LBL on a number of samples of filled and unfilled epoxy as well as other plastics and composite materials. Two groups of measurements were made during the last three years, and they are in agreement with measurements made at other laboratories. The LBL measurements include small sections of superconducting coil. The LBL cracking and thermal contraction measurements were all done at liquid nitrogen temperature. (These yield enough information in order to make reasonable engineering decisions.)

Table 1 shows the results of various measurements made at LBL of thermal contraction and cracking resistance. The materials used in the thin superconducting solenoid magnets are included in the Table. The crack resistance of unfilled epoxy sections is often rather poor. Experience indicates that when these epoxy sections are filled the crack resistance improves greatly.

VACUUM IMPREGNATION OF LARGE SUPERCONDUCTING COILS

The three high current density LBL test superconducting solenoids were built using the epoxy vacuum impregnated epoxy described previously. These magnets use a copper based niobium titanium superconductor which operates at current densities as high as $1.24 \times 10^9 \text{ Am}^{-2}$. These coils are intrinsically stable. Therefore, relatively little energy deposited locally is required to drive them normal (about $1-4 \times 10^4 \text{ Jm}^{-3}$ depending on current density and magnet induction).⁹ Since epoxy cracking can release energy locally it is important that a good epoxy technique be used in these coils.

The epoxy chosen for impregnation of the large superconducting test coils was chosen for its impregnation properties. Impregnation of conventional coils has been done in the LBL shops for over a decade.

The LBL epoxy formulation appears to crack at cryogenic temperatures if large epoxy volumes are unfilled. Care has been taken to avoid cracking within the epoxy. (In theory, the coil assembly is designed for rapid cool down. One should be able to cool the coil in liquid nitrogen and have the coil assembly survive.) Cracking of the epoxy within the coil can be controlled by the following procedure:

TABLE 1 TOTAL THERMAL CONTRACTION FROM 295°K TO 77°K AND THE CRACK RESISTANCE FOR VARIOUS MATERIALS WHICH ARE USED IN THIN SUPERCONDUCTING MAGNETS

Material	Total Thermal Contraction Coefficient 295°K to 77°K	Crack Resistance
Copper *	2.9×10^{-3}	good
Aluminum *	3.8×10^{-3}	good
Sn 62, A 2, Pb 36 Solder	5.8×10^{-3}	good
Epoxy EPON-820, 50 parts versamid-140, 50 parts unfilled	12.0×10^{-3}	poor
Epoxy EPON-826, 50 parts ** DER-736, 50 parts TONOX, 28 parts, unfilled	8.8×10^{-3}	moderate
NEMA-G10 rod along the length	$2.3-2.7 \times 10^{-3}$	good
NEMA-G10 Sheet along the grain	$1.7-2.2 \times 10^{-3}$	good
⊥ to the grain	$7.0-7.8 \times 10^{-3}$	good
ESCAR superconducting coil section along the conductor	2.5×10^{-3}	good
⊥ to the conductor	2.7×10^{-3}	good
TPC superconducting coil section ** along the conductor	2.4×10^{-3}	good
⊥ to the conductor	3.6×10^{-3}	good

* Standard Values

** 1978 measurements, all other values were measured in 1975 by D. Hunt, see Reference 8.

1. The epoxy should be kept in compression. The coil is pretensioned in order to minimize the amount of tensile stress the epoxy sees. (This technique is analogous to the technique used to make prestressed concrete.
2. The amount of epoxy within the structure should be minimized. This reduces the time needed to introduce the epoxy and under vacuum and it minimizes the potential for cracking.
3. The unfilled spaces within the epoxy should be minimized, this reduces the size of potential cracks. (Thus, the energy released due to cracking is minimized.)
4. Voids and bubbles should be eliminated as much as possible.
5. Sharp corners within the mold and on surfaces exposed to the resin should be eliminated.

The basic structure of a magnet coil is shown in Figure 1. This Figure shows a cross section of the one-meter diameter coils. The two-meter diameter coil has a slightly different but similar structure. The spaces in the coil were either filled with glass cloth or with dacron cord. The superconducting wire was wound on the coil form under considerable prestress (about $2 \times 10^8 \text{ Nm}^{-2}$). This prestress guarantees that the relative thermal contraction between the coil and aluminum bore tube is matched. In addition, the coil has enough prestress so that a minimum amount of tensile stress is put into the epoxy.¹⁰

The cooling tube, which is wound outside of the magnet coil, will carry two phase helium to cool the coil. During potting it is used as a means for heating the coil while the epoxy is poured and cured. (This second use for the cooling tube is extremely important.) The thick, nearly pure aluminum bore tube also distributes the heat to the epoxy during pouring and curing. As a result, the epoxy temperature within the coil can be controlled to about 1°C .

The aluminum bore tube is made vacuum tight so that it can be used as part of the vacuum impregnation mold. (A vacuum tight mold is essential.) The outer shell of the vacuum mold consists of sheet steel which is wrapped and clamped around the coil. The vacuum tightness of the vessel is insured through the use of a generous amount of dux-seal, RTV (silicone rubber) and good clamping of the steel onto the coil cooling tube. The vacuum space within the coil was pumped to about 10^{-5} torr while the cooling coil was heated by warm water at a temperature of about 80°C . Once the mold assembly has been leak checked with a mass spectrometer, it remained on the pump for 24 hours at 80°C to outgas water and other contaminants. Then the temperature was dropped to around 45°C . The finished mold assembly for the two-meter diameter test coil is shown in Figure 2.

Once the coil mold was made vacuum tight, pumped down and outgassed, it was backfilled with dry nitrogen to a pressure of about 2 torr. The temperature of the epoxy, which had been mixed and deaerated, was carefully controlled over a temperature range of 45 to 50°C. (The epoxy pot life at 45°C is about 6 hours; at 50°C the pot life goes down to 4 hours.) The coil mold was temperature controlled by flowing hot water through the cooling tube. The water used to maintain the epoxy pot and mold temperature was heated by a cam controlled temperature controller which regulates the water temperature during deaerating, potting, and curing.

The epoxy flow within the coil is extremely important. The LBL epoxy will flow through channels as small as 25µm but the flow rate is slow. Care must be taken to ensure good epoxy flow without large sections of unfilled epoxy. In the LBL solenoid, the bond between the coil and the aluminum bore tube is very important. The bore tube was sand blasted and treated to insure a good bond. The mold on the outside was treated with mold release so that separation could be achieved. In general, it has been found that multiple entry ports for the epoxy results in a much faster pour. The A coil (a one-meter diameter test coil) took 7.5 hours to fill through one port. The B coil (also a one-meter diameter solenoid) took about 1.5 hours to fill because 4 ports were used. The C coil (a two-meter diameter solenoid) took about 7 hours to fill through multiple ports. The C coil has longer distances for the epoxy to flow; it had almost three times the epoxy mass; and it had more than batch poured into the mold. Therefore, the fill time was longer.

The cam controlled temperature controller sets the cure cycle for the epoxy. Once the epoxy is introduced, the temperature is held for about 12 hours, then the temperature is slowly raised to 60°C (this takes about 3 hours). The cure at 60°C takes 8 hours. Then the temperature is raised to 80°C. (This takes about 2 hours.) The magnet and the mold is maintained at 80°C for at least 8 hours. The outer molding shell can be removed after the magnet temperature has dropped to room temperature. The pour and cure temperature schedule for the A magnet (one of the one-meter diameter solenoids) is shown in Figure 3. (Note the epoxy was not held at 50°C for 12 hours before starting the cure.)

After potting and curing, the steel potting shell was broken away. Rough edges on the coil surface were buffed in preparation for a surface glaze. The surface glaze consists of 60 parts by weight of low viscosity resin (EPON 826) and 40 parts by weight of polyamide resin (versamid 140). The surface glaze was applied with a brush and almost wiped dry to form a layer as thin as possible. (This epoxy is prone to cracking but it is well away from the superconductor.) The glaze epoxy was cured at room temperature or under heat lamps until it was hard and dry to the touch. Figure 4 shows the finished C coil solenoid magnet.

The A coil trained due to sudden coil motion which was caused by the breaking of the epoxy bond between the coil and the bore tube.¹¹ There was no evidence of training due to energy release caused by epoxy cracking or fractures within the niobium titanium. The three magnets operated at high current densities and at high stored energies. It is felt that the bad epoxy bond in coil A was due to epoxy which had begun to set before it was poured. (The part of the coil which was poured last was the part which failed.)

The A magnet was cooled down 6 times; the B magnet was cooled down 5 times; and the C magnet was cooled down 4 times. All three magnets show evidence of cracking on the surface. The cracks go into the layer of glass just below the surface. There was no degradation of performance with subsequent cooldowns. Once the magnet achieved its critical current there was little subsequent training. Tests on coil B and coil C showed no degradation of the bond between the coil and the bore tube.

CONCLUDING REMARKS

The epoxy impregnation technique used by LBL appears to work despite rather poor cryogenic properties of the basic epoxy formulation. It appears that technique is more important than epoxy formulation in determining the performance of large potted superconducting solenoid coils. It should be noted that dipole or quadrupole magnet, may not perform as well as our solenoids because the force distribution within the coil is different. The key to using epoxy in large high current density solenoids is minimizing the epoxy mass, filling the epoxy, and prestressing the coil so that the epoxy is kept in compression.

As a result of the LBL test coil experiments, the Laboratory has started construction of a 2.16 meter superconducting thin solenoid which is 3.3 meters long. The current density in the superconducting matrix will be about $7 \times 10^8 \text{ Am}^{-2}$; the magnet stored energy will be $10.9 \times 10^6 \text{ J}$. The design current is set at 70 percent of the superconductor critical current along the load line. The magnet is expected to contain over 70 kg of epoxy resin.

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TESTS OF THE SUPERCONDUCTING COILS

The tests of the three superconducting solenoid magnets show that the epoxy large vacuum impregnated epoxy technique is viable in large solenoid magnets. The three coils do not train like many coils which have been built elsewhere. Table 2 compares the three large LBL solenoids.

TABLE 2 THE CHARACTERISTICS OF THE THREE LBL LARGE VACUUM IMPREGNATED SUPERCONDUCTING SOLENOID MAGNETS

	COIL A	COIL B	COIL C
Magnet Diameter (m)	1.035	1.035	2.004
Magnet Length (m)	0.464	0.461	0.697
Conductor Prestress (Nm^{-2})	1.6×10^8	2.1×10^8	2.3×10^8
Epoxy Mass (kg)	9.2	8.5	25
Epoxy Potting Temperature ($^{\circ}\text{C}$)	50	50	47
Epoxy Pour Time (hr)	7.5	1.5	7.0
Magnet Critical Current (A)	804	1036	1440
Maximum Current Achieved (A)	804	972*	1440
Maximum Superconductor Matrix Current Density Achieved (Am^{-2})	1.044×10^9	1.237×10^9	0.814×10^9
Maximum Magnet Stored Energy (J)	0.255×10^6	0.374×10^6	1.921×10^6
Number of Training Quenches	6	0	0
Current for First Training	597	***	***

* This magnet did not train. 972A is the limit of the power supply.

** There was no training observed.

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FIGURE CAPTIONS

- FIGURE 1 A Cross Section of the One-Meter Diameter Test Solenoid Magnet Vacuum Impregnated Coil Package
- FIGURE 2 The Two-Meter Diameter Test Solenoid Vacuum Impregnation Shroud Showing the Multiple Pouring Posts and Vacuum Pumping Port
- FIGURE 3 The Cure Cycle (Temperature VS Time) for the A and B One-Meter Diameter Test Solenoids
- FIGURE 4 The Finished Two-Meter Diameter Test Solenoid (Coil C)

Fig. 1

XBL 757-3453

CBB 774-3638

Fig. 2

CBB 776-6029

Fig. 3

XBL 786-9171

Fig. 4

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