Fabrication of 90-mm Nb_3Sn Quadrupole Coil Impregnated With MATRIMID –Bismaleimide Based Material

R. Bossert, S. Krave, G. Ambrosio, N. Andreev, E. Barzi, G. Chlachidze, V.V. Kashikhin, M. Lamm, A. Nobrega, I. Novitski, A.V. Zlobin

Abstract—Fermilab is developing Nb_3Sn superconducting magnets for present and future accelerators, including quadrupoles and dipoles, to support luminosity upgrades for the Large Hadron Collider. Insulation is a primary element of the coil design, essential for maintaining electrical, mechanical and thermal performance. The traditional fabrication process involves reaction of the coils at high temperature and impregnation with epoxy to restore the electrical and mechanical properties of the insulation. The traditional epoxy offers adequate structural and electrical properties, but has low radiation strength, limiting the lifetime of magnets operating in severe radiation environments. This paper presents the results of a study in which the traditional epoxy was replaced with Matrimid® 5292 as a coil impregnation material. Test stacks of cable were fabricated and impregnated with epoxy and Matrimid. Electrical, structural and thermal properties were measured and compared. A 90 mm bore, 1-meter long Nb_3Sn quadrupole coil made of RRP 54/61 strand was fabricated, impregnated with Matrimid for testing in a single coil structure (quadrupole magnetic mirror) at 4.2 K and 1.9 K. The coil was instrumented with voltage taps, heaters, and strain gauges to monitor mechanical and thermal properties and quench performance. Test results will be compared to the results for similar coils impregnated with epoxy.

***Index Terms*— Nb3Sn Quadrupole, magnetic mirror, Matrimid.**

I. Introduction

HIGH gradient Nb_3Sn superconducting dipole and quadrupole magnets are being developed at Fermilab in support of future luminosity upgrades for the Large Hadron Collider (LHC) [1] within the framework of the US LHC Accelerator Research Program (LARP) [2]. A key element in the performance of accelerator magnets is the insulation performance. The traditional coil manufacturing process includes a reaction phase at high temperature followed by an epoxy impregnation of the coil to provide the desired electrical, mechanical, and thermal properties. Traditional magnets are impregnated with an epoxy based system [3] that offers good electrical, mechanical, and thermal properties, but is not adequate for use in the high radiation environments of next generation magnets. In this paper, the use of Matrimid 5292, a bismaleimide based material, is investigated for suitability for potting of superconducting Nb_3Sn magnets and ability to be integrated into current manufacturing processes.

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II. Comparison of Matrimid to CTD-101K

Matrimid 5292 is a hot melt bismaleimide solution, which presents a number of challenges that need to be overcome before it can viably be used for potting a magnet when compared to the standard CTD-101K. Primarily, working temperature and cure temperatures are significantly higher when using Matrimid with respect to the CTD-101K. In addition, Matrimid shows tradeoffs between low viscosity and long pot life.

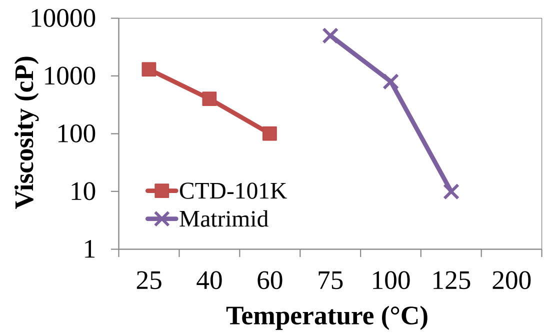

Fig. 1. Viscosity data provided by CTD and Huntsman.

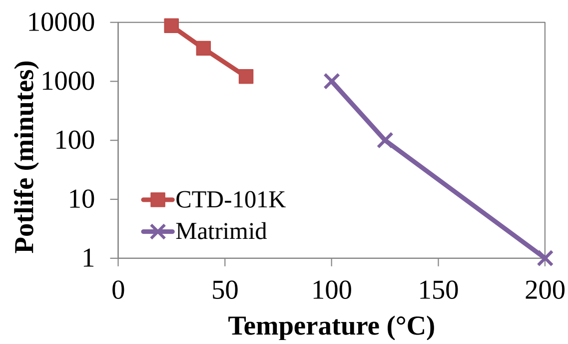

Fig 2. Pot life data provided by CTD and Huntsman.

The rapid rise in viscosity of Matrimid with reduced temperatures (Fig. 1) poses some challenges in terms of the impregnation setup. At conventional potting temperatures, Matrimid can freeze and cease to flow completely. To avoid this case, higher potting temperatures must be used, and all exterior lines must be temperature controlled to avoid freezing and premature curing. The higher temperatures also significantly reduce potlife (Fig 2.).

However, Matrimid does offer a number of benefits. In addition to belonging to a radiation resistant family of materials, it has been shown to have a slightly higher thermal conductivity and higher dielectric strength [4,5]. Mechanical properties have also been confirmed to be similar to CTD-101K [5].

III. Test Stacks

A series of stacks of ten cables are being prepared for mechanical, electrical and thermal comparisons of CTD101K to Matrimid at room temperature and 4.5 K, to confirm and expand data from previous measurements [5]. Only one electrical measurement has been made to date, comparing hipot data of one stack of TQ cable made with Matrimid to a stack using CTD101K. Individual cables in both stacks were surrounded with identical S2 glass sleeves of 125 µm thickness and were 125 mm long. Further stacks using TQ as well as cable from other Nb_3Sn models are being prepared for testing.

Initial results from electrical testing show that the Matrimid stacks exhibit almost no leakage at up to 100 MPa and 500 V ($\leq$1 nA/in^2) and small leakage at 150 MPa and 500 V ($\leq$15 nA/in^2). These results compare well with CTD-101K, stacks, which showed no measureable leakage at pressures to 150 MPa and 500 V. Pairs of cable interfaces were also tested in the Matrimid stack to dielectric breakdown. At pressures of up to 100 MPa, breakdown voltages were above 1.4 kV, with breakdown occurring at 360 V at 150 MPa and 280 V at 200 MPa.

IV. Small Model Impregnation

A. Small Model Test Setup

Matrimid has previously been shown to produce a good quality impregnation in short 150 mm long stacks of cable [5]. Nevertheless, given the viscosity-pot life tradeoff that Matrimid presents, the decision was made to create a small experiment to test the quality of impregnation in a 1 m sample before potting an entire coil. To show the suitability of impregnation using Matrimid to that of CTD-101K, a test was devised using an impregnation fixture from a previous experiment. The test sample was an unreacted 6-stack of TQ cable 1 m long, compressed to nominal thickness with a goal of fully saturating the length of the stack. For impregnation, the sample fixture was heated to process temperature under vacuum. The degassed potting compound was then allowed to enter the test fixture and saturate the cable. After saturation, the sample was returned to atmospheric pressure and cured in place.

For the processing of Matrimid, transfer lines to the fixture were heated to prevent the material from freezing. Heat was applied by using temperature controlled heat tape and maintained at 125 °C. For impregnation with CTD, the fixture was located in a 6 m reaction oven for temperature control and curing. A trial with Matrimid in this same configuration was also made, but the material froze in the transfer lines within a few cm of the reservoir. With availability constraints on the oven, a different approach was used for additional tests. The fixture was then wrapped with high temperature heat tape and insulated. Transfer lines were heated outside of the oven. Fixture temperature was controlled by a PID controller and held to ±1 °C. One sample using CTD was created, as well as two using Matrimid. The two Matrimid samples used different cure schedules, the first curing for 6 hours at 250 °C and the second for 10 hours at 200 °C. The fixture was sealed with silicone RTV and pumped down to ~60 milliTorr. Matrimid flowed completely through a 1 m section of cable on both trials and showed that it would be suitable to pot a short model coil.

After completion of the samples, the impregnation and cure times and temperatures were chosen for the 1-meter coil. A summary of the values compared to those used for previous coils made with CTD101K are contained in Table 1.

TABLE I
Impregnation Details for 1 meter coil

Detail	Matrimid	CTD101K
Impregnation Temperature	125 °C	60 °C
Impregnation Time	15 min	240 min
Curing Temperature	200 °C	125 °C
Curing Time	10 hours	16 hours
Viscosity at Impreg Temp	10 cP	100 cP
Pot Life at Impreg Temp	90 min	1200 min

B. Internal Splices

Nb_3Sn coils include internal splices to NbTi power leads, one each for the inner and outer layers. Since Matrimid requires higher curing temperatures than CTD101K, a test was made to ensure that the splices would not melt or be otherwise damaged during the curing process.

The sample cured at 200 °C contained a pair of splices, one with the Fermilab traditional Pb70/Sn30 solder and the other with lead-free Sn96/Ag4 solder used in the LHC. The Sn/Ag solder becomes liquid near 230°C while the Pb/Sn solder enters a “slurry” state near 200 °C.

C. Small Model Results

After impregnation, the 1 m samples were sectioned. Sections were polished and viewed on a microscope to compare potting quality (Fig 3.). Voids are shown in white. Samples are also compared to a section of TQ coil from a previous magnet for additional validation. The Matrimid and CTD-101K samples had similar results, with Matrimid showing better wetting of the cable. The TQ coil section was superior to either of the samples, presumably because the TQ coil was reacted, while the potting samples were not.

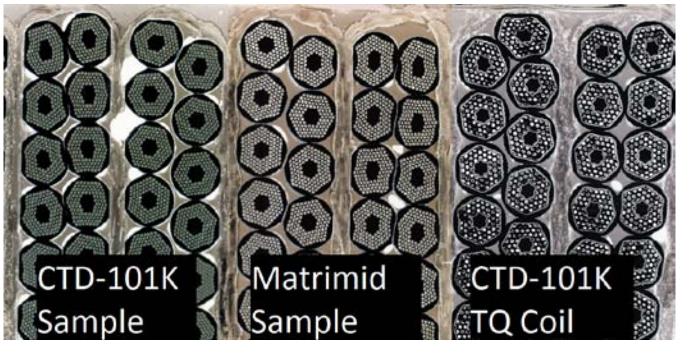

Fig. 3. Comparison of potting samples.

The wetting behavior is probably related to the cable surface finish, since the unreacted cable is not identical to fully reacted coils. Both cure temperatures using the Matrimid showed good performance in terms of wetting (200 °C shown).

The two splices contained in the 200 °C sample were inspected. The Pb70/Sn30 solder exhibited better fill internal to the cable, but showed some signs that it had softened during cure. The Sn96/Ag4 solder remained unchanged. Nevertheless, due to the superior internal fill, the Pb/Sn material was used for the splice in the 1 m coil.

V. TQ Coil Impregnation

After proving the suitability of using Matrimid to pot a coil 1 m in length, a short model coil of TQ style was chosen for the initial attempt [6-8]. The impregnation setup for potting the TQ coil was designed to be as similar to the short model impregnation setup as possible. The impregnation tooling was placed in a 1 m oven with vacuum and heated transfer lines exiting the oven. Impregnation fixture vacuum sealing was accomplished by O-ring seals along the length and RTV on the end plates. Power leads were saturated with solder at the fixture exit and sealed using RTV as shown in Fig 4. Vacuum and transfer lines were 3/8 in soft copper tubing. The fixture and transfer lines were preheated to the potting temperature of 125°C overnight under 60 milliTorr vacuum to drive off volatiles. Fixture temperature was monitored using the oven's PLC.

Fig. 4. Coil impregnation fixture in oven.

Impregnation took place the following day with a potting time of 15 minutes and an additional 1 hour rest at atmospheric pressure before ramp to cure temperature. Impregnation direction was from return end to lead end. The Matrimid was allowed to harden in the external transfer line on exit from the oven as additional application of vacuum could lead to evaporation of Matrimid and formation of voids in the coil. The fill reservoir and transfer lines remained fluid to allow voids to collapse and fill under atmospheric pressure.

After curing, the coil was allowed to cool to room temperature before removal from the oven and demolding. Fig. 5 shows that the OD of the coil had an excellent impregnation with a smooth, consistent finish. The coil was contained in the impregnation tooling with the aperture side facing up, resulting in some small voids located near the aperture on the lead end, shown in Fig. 6.

To reduce voids in future coils, it may be beneficial to flow a larger volume of material through the coil to draw out any trapped gasses that may be in the tooling. A small additional volume for trapped gasses to accumulate might also help.

VI. Coil Preparation and Magnet Assembly

After demolding, the coil was measured in a free state on a coordinate measuring machine (CMM). Measurements show the coil to be larger than nominal by approximately 100 µm per side azimuthally and 50 um radially [9], similar to past TQ coils impregnated in the same tooling with CTD101K. Coil dimensions were then used to determine the shim system for magnet assembly. Some minor inconsistencies were noticed along the coil midplane surfaces as a result of the use of segmented tooling for the mold cavity.

The coil was instrumented with two strain gage stations located on the inner surface of the titanium pole of the inner layer, each containing a compensated full bridge reading pole strain in the azimuthal direction and a compensated full bridge reading pole strain the longitudinal direction. Each station also contained four uncompensated gages bonded directly to the coil reading strain at the midplane and near the pole.

The coil was assembled into a "magnetic mirror" structure, a device used to test an individual coil at cryogenic temperatures. Fig. 7 shows the mirror structure cross section, consisting of the coil, an iron "mirror", which replaces the three missing quadrupole coils, an iron yoke and a stainless steel shell. The assembly is bolted together as shown. Details of the mirror design and assembly procedure were previously reported in [10-12].

Fig 5. Coil after impregnation with Matrimid 5292.

Fig 6. Inner surface of coil showing small voids.

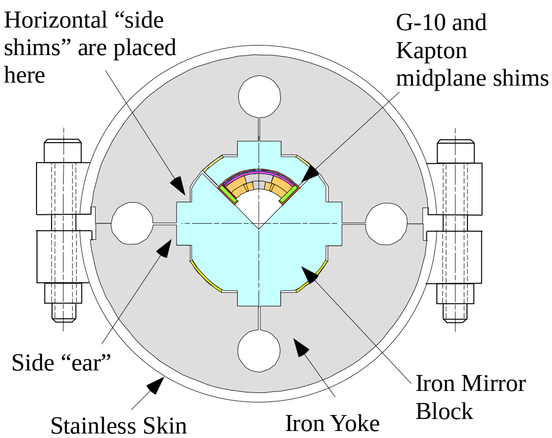

Fig. 7. TQ magnetic mirror structure.

The coil is insulated from ground by Kapton® sheets. The Kapton thickness is adjusted to achieve the desired preload at room temperature. The measured coil size is used to determine the amount of mid-plane shim, based on previous experience and finite element analysis. Shims are also placed onto the horizontal surface of the side "ears" shown in Fig. 7.

The side shims are used to adjust the preload during pressing and control the preload during cool-down.

The structure is placed into a hydraulic press and the pressure is increased until the desired preload is achieved (verified by the strain gauges). Then the press is released, a specified amount of shim is removed, and the pressure is reapplied until the strain gauges reach the same value as they had before the shims were removed. The bolts are then tightened, leaving an open area above the ears into which the structure can contract during cool-down, increasing the preload.

The coil preload was done using four separate pressings. After an initial pressing with a "side shim" of 650 µm, shims were removed between pressings in increments of 75 or 50 µm, until the final desired preload was achieved with a side shim of 525 µm. The "cool-down" gap of 175 µm was then added by reducing the amount of side shims to 350 µm. The final pressing was then completed and the bolts were tightened to close the structure. Strain gauge readings during this process are shown in Fig. 8 for TQM05. The final azimuthal coil pre-stress of TQM05 at room temperature was 144 MPa. As in all TQ mirrors, a load of 5 kN was applied to each end through 50 mm thick stainless steel end plates.

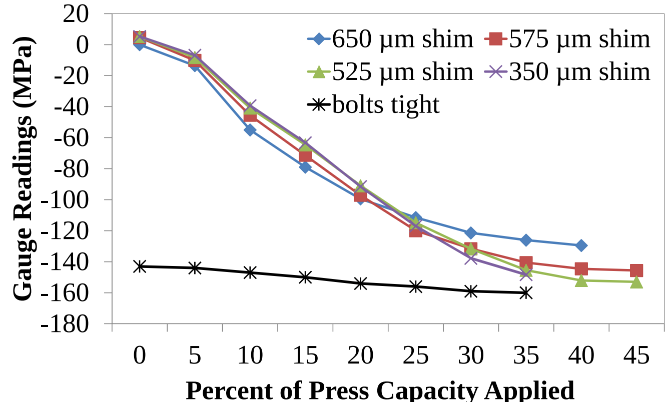

Fig. 8. TQM05 azimuthal preloads during pressing. Horizontal axis denotes % of press capacity, where full press capacity is 900,000 kG.

Assembly of the mirror has been completed, and the magnet is ready for testing at cryogenic temperatures.

VII. Next Steps

TQM05 will be tested at 4.5K and 1.9K in October of 2012. Quench performance, as well as mechanical and thermal properties will be analyzed and compared to the performance of past coils potted with CTD101K.

Some coils of the HQ style [13] with 120 mm bore will be impregnated with Matrimid. One short (1 meter) coil (HQ18) will be potted and sectioned to observe conductor placement and quality of impregnation in a fully reacted coil. Further HQ coils, both short and long (4 meter) may be impregnated with Matrimid and tested, as well as at least one 11T dipole coil [14]. Due to the short pot life of the Matrimid, extending the procedure used for 1-meter coils to lengths of 4 meters and more will present challenges.

The test stack program will be completed, with mechanical, thermal and electrical testing of stacks at room temperature and 4.5K using cables of various types, including TQ, HQ, 150 mm bore quadrupole (MQXF) and 11T dipole cable. Radiation testing of samples of Matrimid will be completed and compared to other candidate materials [15].

VIII. Conclusion

A 1-meter Nb_3Sn coil was successfully impregnated with Matrimid, a bismaleimide based material that has a radiation resistance significantly higher than materials used currently for Nb_3Sn R&D coils. The coil will be tested in a mirror structure, and further coils will be manufactured. If successful, this work will demonstrate that Matrimid qualifies as a potting material for future Nb_3Sn accelerators such as the LHC upgrade.

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