# Insulation Systems for Nb<sub>3</sub>Sn Accelerator Magnet Coils Manufactured by the *Wind & React* Technique

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<sup>1</sup>Abstract—Superconducting dipole and quadrupole magnets for particle accelerators and storage rings usually rely on saddle-shaped coils wound from Rutherford-type cables. When using Nb<sub>3</sub>Sn, the small radii of curvature of coil ends impose that the winding be performed with un-reacted conductors and that the heat treatment needed for Nb<sub>3</sub>Sn compound formation be applied on the whole coil upon winding completion. This results in very stringent requirements on the cable insulation system, which must be able to sustain the high-temperature cycle while retaining its mechanical strength and avoiding the formation of deleterious carbon compounds. Furthermore, it is also desirable to integrate the insulation system as a means of conferring a rigid shape to the manufactured coils, in order to ease subsequent handling and assembly and to protect the brittle material. We review here how various accelerator magnet R&D programs around the world are presently dealing with these issues.

*Index Terms*—Accelerator Magnets, Cable Insulation, Nb<sub>3</sub>Sn, Wind & React.

#### I. INTRODUCTION

**M**<sup>OST</sup> accelerator magnets built until now rely on similar design concepts that were pioneered in the late 1970's for the Tevatron at Fermilab [1], [2]. The magnetic field is produced by saddle-shaped coils, which, in their long straight sections, approximate  $\cos\theta$  or  $\cos2\theta$  conductor distributions, and which are wound from flat, two-layer, Rutherford-type cables made up of NbTi multifilamentary composite strands.

For Tevatron, HERA, UNK, most SSC and early LHC magnets, the cable insulation relies on one or two layers of polyimide tape, wrapped helically with a 50% overlap, completed by an outer layer of resin-impregnated glass fiber tape, also wrapped helically but with a small gap. The resin is of a thermo-setting type and requires heat to increase cross link and cure into a rigid bonding agent. The curing is realized after winding completion in a mold of very accurate dimensions to control coil geometry and Young's modulus. The curing temperature ranges from 135 to 160 °C.

RHIC and present LHC magnets use a so-called *all-polyimide* insulation, where the outer glass fiber wrap is replaced by another layer of polyimide film with a polyimide adhesive on its surface [3]. Here too, the coils are cured upon

winding completion in a very accurate mold. The adhesive softening temperature ranges from 180 to 225 °C.

The highest field reached on a  $\cos\theta$  dipole magnet relying on NbTi cables is 10.53 T at 1.77 K, well above the present specification of 8.33 T for LHC [4]. Nevertheless, the LHC dipole magnet R&D program seems to indicate that the limit for this kind of magnets is likely to be between 9 and 10 T. Going beyond this limit requires a technological jump, which involves necessarily a change of superconductor, and may require also a calling into question of the  $\cos\theta$  design.

The only other superconducting material that is readily available at (small) industrial scale is Nb<sub>3</sub>Sn, thanks, in particular, to the Engineering Design Activities for the ITER program [5]. Unlike NbTi, which is a ductile, solid-solution alloy, easy to co-process with copper, Nb<sub>3</sub>Sn is an intermetallic compound with a rather well defined stoichiometry. In such compound, the valence electrons are not free to move, but are shared between neighboring atoms to create covalent bonds. The covalent bonds increase hardness, but render Nb<sub>3</sub>Sn very brittle and strain sensitive [6]. As a result, Nb<sub>3</sub>Sn multifilamentary composite wires cannot be processed in the same way as NbTi wires, and once the Nb<sub>3</sub>Sn compound is formed, everything must be done to minimize deformations.

In practice, Nb<sub>3</sub>Sn conductors are processed from uncompounded precursors, and, upon achievement of the desired shape and size, the final conductor, or even the coil wound from this conductor, is subjected to a special treatment for Nb<sub>3</sub>Sn formation. Most industrial processes rely on the fact that when a bronze/niobium composite is heated to a temperature of about 700 °C, Sn atoms selectively diffuse from the bronze into the niobium, and react with Nb atoms to precipitate Nb<sub>3</sub>Sn. This solid state diffusion process is slow and a full reaction of the Nb bulk can require several hundreds of hours. Hence, the special treatment applied to the final conductor is usually a heat treatment at temperatures between 650 and 700 °C for a duration of 2 to 10 days, performed in a vacuum or with a flow of inert gas (pure argon or nitrogen) to prevent oxidation of stabilizing copper.

The timing of the required heat treatment depends on conductor and winding configurations. If the bending radii encountered during winding are large enough to limit the strain induced in Nb<sub>3</sub>Sn to a level that does not degrade significantly the superconductor critical current density, the

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Fig. 1. Winding of a saddle-shaped, Wind & React Nb<sub>3</sub>Sn coil for a  $\cos\theta$  dipole magnet model at Twente University [7].

treatment can be applied to the conductor prior to winding. Such technique is referred to as *React & Wind*. Conversely, if coil winding requires small bending radii, as is usually the case for saddle-shape coils (see Figure 1), the heat treatment must be applied to the whole coil, upon winding completion. This technique is referred to as *Wind & React*.

The conductor insulation for a React & Wind coil, can be wrapped around the conductor after heat treatment, and can be similar to the one used for NbTi magnets. However, the insulation scheme of Wind & React coils cannot be the same, for anything wrapped around the conductor prior to winding, must be able to sustain the high temperature cycle, which excludes the use of polyimide tapes.

In spite of the difficulties related to the brittleness and strain sensitivity of Nb<sub>3</sub>Sn, a number of quite successful Nb<sub>3</sub>Sn dipole magnet models have been developed over the last decade. In the Summer of 1995, a  $\cos\theta$  dipole magnet built at Twente University and cold tested at CERN reached 11.03 T on its first quench at 4.4 K [8]. In March 1997, another  $\cos\theta$  dipole magnet built and cold tested at LBNL was trained up to 13.5 T at 1.8 K [9]. More recently, a racetrack-type dipole magnet model, also built and cold tested at LBNL, was trained to a record field of 14.7 T at 4.2 K [10]. The coils of these magnets were all produced by the Wind & React technique. These encouraging results seem to indicate that Nb<sub>3</sub>Sn technology could open the 10-to-15 T range for accelerator magnet application.

Before deciding if Nb<sub>3</sub>Sn technology is mature enough either to upgrade an existing accelerator or to proceed with a larger machine such as the proposed Very Large Hadron Collider (VLHC) [11], a significant R&D effort has still to be carried out to demonstrate the feasibility and reliability of Nb<sub>3</sub>Sn magnets and to lower their costs. Indeed, the present cost of Nb<sub>3</sub>Sn magnets far exceed that of NbTi magnets. This comes from the fact that Nb<sub>3</sub>Sn wires are at least 4 to 5 times more expensive than NbTi wires. It is also due to the fact that, in its present form, the Wind & React technique requires a number of delicate manufacturing steps that are not suitable for industrial production. In recent years, several laboratories around the world are focusing their efforts into developing more production-oriented magnet designs.

These efforts, carried out mainly in the USA, follow two different but complementary paths: (1) one is to optimize coil configuration to switch to React & Wind and/or to allow a better management of Lorentz stresses during energization, and (2) the other is to improve the insulation scheme to simplify manufacturing process and reduce failure risks.

After recalling the conventional insulation scheme the most widely used for Wind & React accelerator magnet coils, we review the problems and limitations of this scheme, and we describe some of the innovations that are in the work and which could foster the technological jump sought after by magnet developers.

#### II. CONVENTIONAL INSULATION

## A. Main Features

The insulation of Nb<sub>3</sub>Sn Wind & React coils is usually realized in two steps: (1) at first, the cable is wrapped with a mineral fiber cloth, prior to winding and heat treatment, and (2) upon winding and heat treatment completion, the fragile and loose coil is transferred into a mold of very accurate dimension and is vacuum-impregnated with epoxy resin.

The fiber cloth has three main roles: (1) it provides conductor spacing and facilitates resin penetration, (2) it helps controlling conductor positioning as required by field quality reasons, and (3) it limits crack propagation in resin.

The epoxy resin has a least two functions: (1) it greatly enhances the turn-to-turn breakdown voltage (to values in excess of 10 kV/mm in liquid helium at 4.2 K and in gaseous helium at 4.2 K, 100 K and 300 K [12]), and (2) it confers a rigid shape to the coil, thereby facilitating subsequent handling and assembly. Some authors also argue that it can limit the occurrence, upon transverse loading, of stress concentrations at the crossovers between strands of the twolayer Rutherford-type cable [13].

## B. Fiber Wrap

The main consideration regarding the choice of fiber wrap are: (1) ability to withstand high temperature cycle, (2) effective thickness, which must be small to maximize overall current density in magnet coil, (3) easiness of wrapping, and (4) ability to ensure a physical continuity along conductor to limit the risks of turn-to-turn shorts.

The relationship between insulation thickness and overall current density can be readily understood by considering the case of LHC arc quadrupole magnets. Here, the NbTi cable mid-thickness is 1.48 mm, while the polyimide insulation thickness is 0.11 mm per conductor broad face (under 80 MPa). It follows that the overall current density over the insulated cable is at least 14% lower than the current density

over the bare cable. A doubling of the insulation thickness leads to a 23% reduction, which, when reaching this level, can be regarded as a waste of costly superconductor.

The ideal solution would be a thin tape, woven from cheap glass fibers, and wrapped helically around the cable with a 50% overlap. Glass fibers are made up of small filaments (a few microns in diameter) assembled into a yarn. The filaments are processed from a silica-based mixture, that is melted and drawn at high speed. The initial mixture includes melting agents (such as CaO and MgO), and stabilizers chosen to improve selected properties (such as  $Al_2O_3$  for mechanical strength and  $B_2O_3$  for dielectric strength).

The cheapest and most commonly used grade of glass is *E* glass, which comprises ~55 wt% of SiO<sub>2</sub>, a large concentration of melting agents (20 to 25 wt%), ~15 wt% of Al<sub>2</sub>O<sub>3</sub> and some B<sub>2</sub>O<sub>3</sub>. However, E glass has a recrystallization temperature of ~650 °C which is too low for our application.

The next grade is *S* or *S2* glass in the USA and *R* glass in Europe. S/S2 and R glasses have a higher concentration in SiO<sub>2</sub> (in excess of 60 wt%) and in Al<sub>2</sub>O<sub>3</sub> (between 20 and 25 wt%), but less melting agents (below 15 wt%), and no or very little B<sub>2</sub>O<sub>3</sub>. Their recrystallisation temperatures are above 750 °C and they are well suited for our application. The problem, however, is that, to the author's knowledge, the minimum thickness of commercially available S or R glass fiber tapes is ~0.1 mm. For a wrapping with a 50% overlap, this yields ~0.2 mm per conductor broad face, which is excessive for most accelerator magnet designs.

Several solutions are presently used to circumvent this difficulty. LBNL relies on a slightly oversized sleeve, woven from S2 glass fibers, which is slid over the cable prior to winding. The sleeve is provided by Fiber Innovations [14], and yields an effective thickness of 4.2 mils (~107  $\mu$ m) per conductor face (under 1 to 2 kpsi or 7 to 14 MPa) [15]. The maximum cable length that can be insulated in such a way is ~250 m. For longer lengths and/or to achieve a tighter dimensional accuracy, the sleeve would have to be woven directly on the conductor.

Twente University relies on a mixed system, made up of two components [7], [16]: (1) a 0.1-mm-thick mica-glass sheet, folded on three conductor sides (one broad face and the two small edges), which ensures physical continuity, and (2) a ~0.1-mm-thick S2 glass fiber tape, provided by Hiltex [17], which is butt-wrapped on top of the mica-glass. Sheet and tape are positioned manually, on-line with winding, and, under nominal pre-compression, yield an effective turn-toturn insulation thickness of 0.28 mm (0.14 mm per conductor face). It is worth mentioning that, after heat treatment, the mica-glass tends to delaminate.

An alternative to S or R glass fibers is quartz fibers, which are made from almost pure silica (concentration in excess of 99.5 wt%), have a recrystallisation temperature above 1200 °C, and can be found in small diameters (down to 17 g/m or tex). Over the last couple of years, DAPNIA/STCM at CEA/Saclay has developed, in collaboration with industry, a 15-mm-wide, 60-µm-thick quartz fiber tape, that seems promising, but has yet to be tried in a real magnet [12], [18]. The weaving, which requires old wooden looms to prevent breakages, is performed by STB Tiss Tech [19] using Quartzel<sup>TM</sup> fibers [20].



Fig. 2. Example of R glass fiber tape heat-treated at 660 °C for 240 hours in a vacuum without removing sizing. The tape turned out grayish (left hand side) and carbon residues were revealed by dipping it partially into epoxy resin (right hand side) [21].

## C. Sizing/De-Sizing/Re-Sizing

One of the main issues concerning the use of mineral fiber tapes or sleeves with un-reacted Nb<sub>3</sub>Sn conductors is the fact the filaments forming the yarns are usually sized with organic compounds. During the heat treatment at ~700 °C in a vacuum or with a small flow of inert gas, the organic compounds undergo a graphitization-like decomposition, which, as illustrated in Fig. 2, leaves deleterious, electrically-conducting carbon residues. Hence, all these compounds must be eliminated from the cloth prior to heat treatment.

The sizing is a surface treatment which has at least three main functions: (1) to protect the filaments against abrasion, (2) to lubricate and reduce filament-to-filament and yarn-to-contact-surface friction, and (3) to provide anti-static properties. It is indispensable for weaving (either the yarns or the cloth), and ensures the mechanical integrity of the woven product. When the fiber cloth is used as a support for resin impregnation, the sizing can also include specific agents to help fiber wetting by the resin. The sizing composi-tion is usually secret and depends on the manufacturer.

There are two techniques to remove sizing: (1) by chemical dissolving or (2) by carbonization in air. The first technique may be the most practical, but it presents at least two disadvantages: (1) it requires a detailed knowledge of the chemical composition of the sizing and of its solvents, which, as already mentioned, most manufacturers are unwilling to disclose, and (2) there is no way of ensuring the uniformity and completeness of the process.

The second technique is more radical and is more likely to eliminate all traces of organic compounds. However, it has one serious disadvantage: as confirmed by Thermal Gravimetric Analyses (TGA), the burnout (in air) of sizing agents takes place between 200 and 250 °C for E and S glass fiber tapes, and around 300 °C for Quartzel<sup>TM</sup> fiber tapes [22]. These temperatures are too high to be applied in presence of the Cu-stabilized Nb<sub>3</sub>Sn wires, and imposes that the *de-sizing* step be carried out prior to conductor wrapping. Most laboratories perform the de-sizing treatment in house, at temperatures between 300 and 350 °C for a few hours.

As an illustration, Fig. 3 presents SEM views of Quartzel<sup>TM</sup> filaments before and after a de-sizing heat

treatment at 350 °C for 4 hours in air. The de-sized filament not believed to be a problem for the insulation of the short exhibits no visible degradation, and even after a subsequent heat treatment at 660 °C for 15 hours, measurements by X-ray diffraction over a 4-hour period show no evidence of recrystallisation [22].



Fig. 3. SEM views of Quartzel<sup>TM</sup> filaments: (a) as received and (b) after a de-sizing heat treatment at 350 °C for 4 hours in air [22].

Once the sizing agents have been removed, the glass or quartz filaments are not protected against abrasion anymore, and the cloths become fragile and easy to tear off by This complicates most manufacturing friction [23]. operations (such as conductor wrapping, coil winding, and coil handling in and out of heat treatment retort), and renders insulation damage almost unavoidable. In terms of industrial production, the weakness of de-sized tapes or sleeves is one of the most critical issues and creates a high failure risk. Note, however, that one may recover from some of the damages at the time of vacuum impregnation.

One way of tackling this difficulty is as follows: once the manufacturer's sizing has been removed, it can be replaced by a homemade and more volatile one that partially restores lubricant properties. This procedure is applied at LBNL to help sliding of sleeve over cable. First, the as-received sleeve is de-sized by carbonization in air, and then, it is *re-sized* by running it through a palmitic acid solution. The palmitic acid is a so-called fatty acid, of formula, CH<sub>3</sub>(CH<sub>2</sub>)<sub>14</sub>CO<sub>2</sub>H, characterized by an oily nature. It has a melting point of 61-64 °C and a boiling point of ~350 °C, and it is soluble in hexane. Most of the re-sizing is eliminated during the hightemperature ramp of the final heat treatment, but even with a flow of argon, there always remain some traces and the coil does not come out perfectly white [14]. These residues are

model magnets that are presently being built.



Fig. 4. Stress-strain curves measured at room temperature on conductor stacks representative of accelerator magnet coils: (a) Nb<sub>3</sub>Sn cables wrapped with quartz fiber tapes and vacuum-impregnated with epoxy resin [33], and (b) NbTi cables with all-polyimide insulation [31].

One last issue regarding sizing is the fact that it also provides water-resisting properties. There are ample evidences in the literature that water adsorption on glass fiber results in degradations of mechanical properties [24], and that a great care should be taken in storing de-sized materials. It is worth mentioning, however, that the experience at Twente University is quite the opposite: after de-sizing, the S2 glass tape is left exposed to un-controlled atmosphere for several weeks and this seems to reinforce its properties [7].

### D. Resin Impregnation

Vacuum-impregnation of superconducting magnet coils is a well-known technique and there are several commercially available epoxy resins that are well suited to this application [25]-[28]. European laboratories usually rely on Ciba-Geigy products [29], while several US laboratories presently use CTD 101K, provided by CTD [30].

The design of the vacuum-impregnation mold is instrumental in determining coil geometry and size. Given the field quality requirements and the accuracy with which the conductors need to be positioned, the mold cavity must be carefully dimensioned and very tightly toleranced.

The tolerancing is all the more crucial when impregnated coils are significantly stiffer than all-polyimide coils [31]-[33]. This is illustrated in Fig. 4, which shows a comparison stress-strain of curves measured for transverse loading/unloading cycles performed at room temperature on a stack of NbTi cables wrapped with polyimide tapes and a stack of Nb<sub>3</sub>Sn cables wrapped with quartz fiber tapes and vacuum-impregnated with epoxy resin. The NbTi cable stack is representative of LHC arc quadrupole magnet coils [31], while the Nb<sub>3</sub>Sn cable stack is representative of the coils of a quadrupole magnet presently under development at CEA/Saclay [18], [33]. The two cables are made to the same geometrical specifications, and the nominal insulation thickness per conductor broad face is 0.11 mm for the NbTi cable and 0.12 mm for the Nb<sub>3</sub>Sn cable.

The two sets of curves exhibit similar features (a first loading clearly distinct from subsequent ones, hystereses between loading and unloading branches of stabilized cycles, and non-linearities), but those for the impregnated stack are quite a bit steeper. As a result, the effective Young's modulus estimated from the slopes of the quasi-linear loading branches of stabilized cycles is 33 GPa at room temperature and 45 GPa at 4.2 K for the Nb<sub>3</sub>Sn stack [33], while it is between 6 and 7 GPa at room temperature and between 11 and 12 GPa at 4.2 K for the NbTi stack [31]. Note that the Young's modulii reported in Ref. [32] for impregnated Nb<sub>3</sub>Sn stacks are in the same range (~40 GPa), but that, somewhat surprisingly, no apparent increase in stiffness was observed with decrease in temperature.

For completeness, let us note that the integrated thermal shrinkage coefficient from room temperature to 4.2 K is between 3.5 and 3.9 mm/m for impregnated Nb<sub>3</sub>Sn stacks [32]-[33], and ~5.0 mm/m for all-polyimide NbTi stacks [31].

## **III.** INNOVATIVE SCHEMES

As we have seen, the conventional insulation for Nb<sub>3</sub>Sn Wind & React coils has at least three shortcomings: (1) the fragility of de-sized tapes, (2) the looseness and lack of protection of brittle Nb<sub>3</sub>Sn coil upon heat treatment completion, and (3) the necessity of performing a vacuum impregnation, which adds to the cost and raises failure risk. To the authors' knowledge, at least two R&D programs are being carried out to try to solve these problems and develop manufacturing procedures that are more production oriented: (1) one at CTD, with technical support from Fermilab and LBNL, and (2) one at DAPNIA/STCM in collaboration with Institut Européen des Membranes of Montpellier, France (CNRS UMR 5635) [34].

CTD is developing two families of innovative insulating materials for Wind & React processing [35], [36]. The first family is a ceramic/organic hybrid insulation, which relies on two main components: (1) an inorganic binder, designated as CTD 1002x, applied prior to heat treatment, and (2) an organic resin, such as CTD 101K, impregnated into the coil after heat treatment. The epoxy-impregnated system is referred to as CTD 1102x and is commercially available in the USA. The second family is an all-ceramic insulation, where, as the name implies, no organic is added at the end of heat treatment. This material is in the very early stages of development and is not available commercially. In June 2001, CTD was awarded a two-year grant from the US Department of Energy to continue its development work. The main research tasks are now to increase the strength of the all-ceramic material through matrix optimization.

The Fermilab high-field dipole magnet program presently relies on a CTD 1102x insulation scheme [32], [37]. The manufacturing procedure first calls for a wrapping with a 50% overlap of a 0.125-mm-thick, de-sized ceramic fiber tape around un-reacted Nb<sub>3</sub>Sn cable. It is followed by

the application of CTD 1002x binder on the wrapped cable, which improves insulation strength and reduces damages upon subsequent bending. The next step is coil winding, with further binder application on coil turns, followed by a short curing heat treatment at 150 °C for 30 minutes. The curing results in a bonding of coil turns and provides a rigid shape to the coil, which becomes easier to handle. The coils are then assembled in their final configuration and are heat treated together at high temperature. Upon heat treatment completion



Fig. 5.  $Nb_3Sn$  cable stack insulated with ceramic/organic hybrid insulation CTD 1102x [36].

the coil assembly is vacuum-impregnated with CTD 101K epoxy resin. Figure 5 shows a photograph of a Nb<sub>3</sub>Sn cable stack manufactured according to this procedure.

As pointed out, this innovative insulation scheme offers at least three advantages: (1) partial restoration of mechanical strength after binding application, (2) easiness of cured coil handling, and (3) possibility of assembling cured coils together prior to heat treatment. However, in its actual form, it presents two disadvantages: (1) the insulation thickness is in excess of 0.2 mm per conductor broad face, and (2) the need for a vacuum impregnation has not been eliminated.

The main goal of the program underway at DAPNIA/STCM is to develop a thin, pre-impregnated fiber tape that meets two series of requirements: (1) flexibility and strength to allow conductor wrapping and coil winding according to conventional techniques, and (2) ability, during the heat treatment required for Nb<sub>3</sub>Sn compound formation, to transform the loose coil into a rigid body, while maintaining proper conductor positioning and suitable turn-to-turn electrical insulation.

To illustrate the progress being made, Fig. 6 shows an example of bending test performed on a wrapped cable, while Fig. 7 presents a stack of insulated conductors after heat treatment. The cable wrap appears to maintain its mechanical integrity upon bending, while the conductors of the stack are bonded together. These results demonstrate the feasibility of this innovative insulation scheme, and the program is now aimed at implementing it in a real coil.

#### IV. CONCLUSION

Over the last few years, a lot of effort has been put into increasing the performances and lowering the production costs of Nb<sub>3</sub>Sn wires. In parallel, several 10-to-15-T dipole magnet models have been successfully built and cold tested, thereby opening a new field range to accelerator magnet application. The next step is to simplify manufacturing procedures in order to enable mass-production in industry. For Wind & React Nb<sub>3</sub>Sn coils, one of the most critical issue remains cable insulation, but several innovative schemes are presently under development, which may provide technically-viable and cost-effective solutions to this problem.



Fig. 6. Bending test on un-reacted Nb<sub>3</sub>Sn cable wrapped with innovative preimpregnated fiber tape at CEA/Saclay.

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Fig. 7. Stack of Nb<sub>3</sub>Sn cables insulated with CEA/Saclay innovative preimpregnated fiber tape after heat treatment.

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