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ABSTRACT

The insulation systems the most commonly used for Nb_3Sn coils fabricated by the "wind and react" technique are made up of a mineral-fiber support positioned around the conductor, completed by a vacuum impregnation with resin. In the case of accelerator magnet coils, the support is positioned prior to coil winding and is subjected to the heat treatment required to the reaction of the Nb_3Sn compound reaction. Furthermore, the vacuum impregnation is performed on the whole coil after heat treatment. We have carried out an experimental program to evaluate 3 types of supports: (1) E- or S2-glass fiber tapes, (2) mixed wraps made up of mica-glass foils and E- or S2-glass fiber tapes, and (3) quartz fiber tapes. We report here on tensile strength and dielectric strength measurements performed on samples representative of accelerator magnet coil insulation and we compare the performances of the various supports before and after heat treatment, at room temperature and in helium environment down to 4.2 K.

INTRODUCTION

The highest field reached on a dipole magnet wound from NbTi cables is 10.53 T at 1.77 K, well above the present (1999) specification of 8.33 T for the dipole magnets of the Large Hadron Collider.[1] In 1995, a short dipole magnet model, wound from Nb_3Sn cables, and referred to as MSUT, achieved 11 T on its first quench at 4.4 K.[2] In 1997, another short dipole magnet model, also wound from Nb_3Sn cables, and referred to as D20, reached, after some training, a record field of 13.5 T at 1.8 K.[3] These encouraging results show that the Nb_3Sn technology may open viably the 10-to-15-T range to accelerator magnets.

Nb_3Sn is an intermetallic compound belonging to the A15 crystallographic family whose critical temperature and upper critical field far exceed those of NbTi.[4] However, like all members of the A15 family, the Nb_3Sn compound is very brittle and its superconducting properties are strain dependent.[5] This implies that, once the Nb_3Sn compound is reacted, everything must be done to minimize its deformation.

In practice, several processes have been developed to produce round wires of small diameters containing precursors to the formation of a Nb_3Sn multifilamentary composite. The Nb_3Sn filaments are reacted by subjecting the wire at final size to a heat treatment at a temperature between 650 and 700 °C for 2 to 10 days in vacuum or with circulation of inert gas. For accelerator magnet coils, which are wound from Rutherford-type cables and which require small winding radii,[6] the heat treatment is usually applied on the whole coil after winding completion. This technique is referred to as "wind and react".

The "wind and react" technique minimizes the deformation applied to the reacted Nb_3Sn filaments but complicates the realization of the turn-to-turn electric insulation. Such insulation is required to prevent the occurrence of turn-to-turn shorts during a quench. The insulation system is usually made up of a mineral fiber support (also referred to as reinforcement) positioned around the conductor to ensure a proper spacing between coil turns. It is completed by a vacuum impregnation with epoxy resin, conferring a rigid shape to the coil assembly and enhancing dielectric strength.

In the case of accelerator magnet coils, the reinforcement is positioned around the conductor prior to coil winding and the vacuum impregnation is realized on the whole coil after heat treatment. It follows that the mineral fibers of the support must be able to sustain the heat treatment without significant degradation. Also, all organic materials, such as sizing or finish, must be removed from the fibers to prevent the formation of carbon compounds that lower the dielectric strength. The sizing removal is performed by carbonization in air prior to conductor insulation. This de-sizing adds to the difficulty of manufacturing Nb_3Sn coils for the fibers then become fragile and are easy to tear off by friction.[7]

The ideal support for Nb_3Sn coil insulation would be a fiber tape wrapped helically around the conductor with a 50% overlap. In order to maximize the overall current density in the magnet coil, the tape thickness should be as small as possible, typically, of the order of 50 to 60 μm, resulting in an overall turn-to-turn insulation thickness of less than 250 μm.

The cheapest and most readily available mineral fibers are E-glass fibers. However, E-glass has a recrystallization temperature of the order of 660 °C,[8] and can be expected to degrade significantly during heat treatment. S2-glass has a recrystallization temperature of the order of 780 °C,[8] but is produced in much smaller quantities and cannot be found in fiber tape form of the desired thickness. To circumvent this difficulty, the conductor of magnet model D20 was insulated with a S2-glass sleeve pulled along its entire length,[3] while the conductor of magnet model MSUT relied on a butt-wrap of S2-glass fiber tape completed, on one side of the conductor, by a mica-glass foil designed to ensure the continuity of the electrical insulation.[2] Neither of these systems can be easily industrialized.

Another option is the use of quartz fibers. Quartz fibers have a good behavior at high temperatures (they stay in the vitreous state for temperatures up to 1050 °C) and can be found in small enough diameters to allow the weaving of thin tapes.[9] Nevertheless, before using quartz fiber tapes in a magnet, it is required to check their mechanical strength and to compare their behavior with that of other supports.

In this paper we present the results of tensile strength and dielectric strength measurements carried out on samples representative of various insulation systems for Nb_3Sn accelerator magnet coils. The measurements were carried out at room temperature and in helium environment down to 4.2 K and we compare the performances of the various supports before and after heat treatment.

SAMPLES PREPARATION

Three types of reinforcements have been investigated: (1) E- and S2-glass fiber tapes wrapped with a 50% overlap (1 layer, 2 thicknesses), (2) E- and S2-glass fiber tapes butt-wrapped (1 layer, 1 thickness) with one mica-glass foil for every two layers (as for the insulation used in model magnet MSUT) and (3) two types of quartz fiber tapes wrapped with a 50% overlap (1 layer, 2 thicknesses). Selected parameters of the various tapes and of the mica-glass foils used in this study are summarized in Table 1.

Table 1. Selected parameters of the mineral fiber tapes and of the mica-glass foils used as reinforcements.

Support Type	Fiber Tex (g/km)	Thickness (mm)	Width (mm)	Number of Fibers per cm Warp	Number of Fibers per cm Woof	Specific Weight (g/m^2)
E-Glass	34	0.13	25	22	10	363
S2-Glass	34	0.10	15	15	13	106
Standard Quartz	17	0.07	15	21	15	64
COC Quartz	17	0.07	15	21	15	64
Mica-Glass	–	0.105	as required	–	–	130

The S2-glass fiber tape is expected to be the most robust. However, its thickness is of the order of 100 μm, resulting, when wrapped with a 50% overlap, in a turn-to-turn insulation of the order of 400 μm. This turn-to-turn thickness can be reduced by using a butt-wrap, but it is then necessary to insert, for every two layers, a 100-μm-thick mica-glass foil to ensure the continuity of the electrical insulation. This results in an overall insulation thickness of 300 μm, decreasing to 280 μm under pressure.[2] The turn-to-turn thickness can be further reduced by using quartz fiber tapes, which, when wrapped with a 50% overlap, yield an overall insulation thickness of 240 μm under pressure. The E-glass fiber tape is thicker than the S2-glass fiber tape and is only used as a reference.

As already indicated, two types of quartz fiber tapes have been investigated: (1) a tape woven from standard fibers and (2) a tape woven from chromium-oxide coated (COC) fibers. The chromium oxide, which unlike the organic sizing, can sustain high temperatures, is supposed to reduce friction and, thereby, to improve the mechanical behavior of the tape. However, during the custom manufacturing of the present tape, the rubbing of the COC fibers against the metallic comb of the weaving loom resulted in a corrosion of the comb's teeth which caused numerous breakages. Consequently, the COC quartz fiber tape used in our samples presented a lot of defects, which may have seriously degraded its performance. The comb is now being treated to eliminate the corrosion problem and a new batch of COC quartz fiber tape will be produced for further evaluation.

To prepare the samples, the tapes were wrapped with the desired patterns around 90-mm-wide, 590-mm-long, and 10-mm-thick mandrels coated with Teflon. The mandrels were then inserted into a mold for vacuum impregnation. The mold design allowed for four mandrels to be simultaneously impregnated: two for tensile test samples and two for dielectric test samples. The main difference in the samples preparation resides in the number of layer wraps: the samples for the dielectric tests include only one elementary wrapping pattern representative of the turn-to-turn insulation, while the samples for the tensile tests include several elementary patterns, so as to achieve an overall thickness of the order of 3 mm.

Prior to wrapping, the sizing was removed from the E- and S2-glass fiber tapes and from the mica-glass foils by heating them up at 350 °C in air overnight. The quartz fiber tapes were heat-treated at 700 °C in air for 10 to 15 minutes by the manufacturer, but the de-sizing process appeared to be incomplete. Upon reception at Saclay, they were heat-treated again at 350 °C in air overnight. After de-sizing, half of the tapes were used as is, while the other half was subjected to a heat treatment representative of the heat treatment needed to react the Nb_3Sn compound: up-ramp at 15 °C per hour to 660 °C, followed by a plateau at 660 °C for 120 hours, with a circulation of pure argon.

The epoxy resin used for the vacuum impregnation was a diglycidylether of bisphenol A, mixed with cresylglycidylether as reactive diluent and phtalic and hexahydrophtalic anhydrides as curing agents. The applied curing cycle was 12 hours at 95 °C followed by 48 hours at 110 °C. This epoxy resin has been studied elsewhere,[10] and its measured density at room temperature is 1.221 g/cm^3.

Table 2. Samples description.

System Reinforcement	Denomination	Elementary Wrapping Pattern[a]	Fiber Content (vol%)
E-glass fiber tape	EE	2 layers of E-glass fiber tape wrapped with a 50% overlap	39
S2-glass fiber tape	SS	2 layers of S2-glass fiber tape wrapped with a 50% overlap	41
E-glass fiber tape + Mica-glass foil	EM	2 layers of E-glass fiber tape butt-wrapped with 1 mica-glass foil	
S2-glass fiber tape + Mica-glass foil	SM	2 layers of S2-glass tape butt-wrapped with 1 mica-glass foil	
Standard quartz fiber tape	QS	2 layers of standard quartz fiber tape wrapped with a 50% overlap	31
Chrome-oxide-coated quartz fiber tape	QH	2 layers of COC quartz fiber tape wrapped with a 50% overlap	30

[a] Representative of the turn-to-turn insulation in a magnet coil.

Upon completion of the vacuum impregnation, the composite plates were detached from the Teflon-coated mandrels and were machined to obtain samples of the desired shapes. The plates reinforced by glass or quartz fiber tapes alone appeared to have been well impregnated, while the plates containing mica-glass foils exhibited a tendency to delaminate. These delaminations are likely due to a bad impregnation of the resin into the mica layer, whose bonding capacity may have been reduced by the presence of residues of the silicon binder holding the mica-glass together.

Table 2 summarizes the constitution of the various samples. The last column provides the fiber volumetric fraction measured on tensile test samples.

Most of the samples cut out from the composite plates with mica-glass are brittle and tend to delaminate.

TENSILE BEHAVIOR

Experimental Procedure

Tensile tests were carried out with screw-driven machines, at room temperature and in pool boiling helium at 4.2 K. Samples with a dogbone shape were cut from the composite plates described above. They had a cross-sectional area of 3x12 mm^2 and a radius of 27 mm between the heads and the useful area. They were held with bolted friction grips at both ends. For all tests, the crosshead displacement speed was set to 0.5 mm/min. The displacement was measured with a regular extensometer over a gauge length of 12.5 mm and the load was measured with a 150 kN or a 300 kN cell. All samples were loaded to rupture.

Measurement Results

The Young's modulus and the ultimate tensile stress have been estimated for each type of insulation system by taking an average over two samples. Before reviewing the data, let us stress that we are not trying to compare the properties of the different fibers, but that, for each system, we want to investigate the effects of temperature, of loading direction (warp or woof), and of the heat treatment at 660 °C. (Note that the data from the mixed systems with mica-glass are not presented here because all samples exhibited signs of delamination.)

For all tested materials, the behavior at 4.2 K differs significantly from the behavior at room temperature. In particular, and as illustrated in Fig. 1, cooldown results in a sizeable improvement of the mechanical properties.

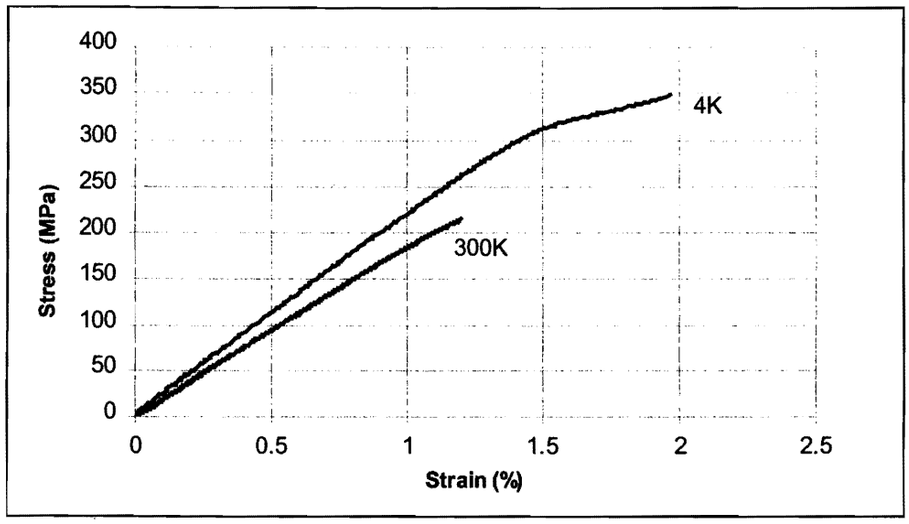

Figure 1 : Effect of temperature on the tensile behavior of standard quartz reinforced composites

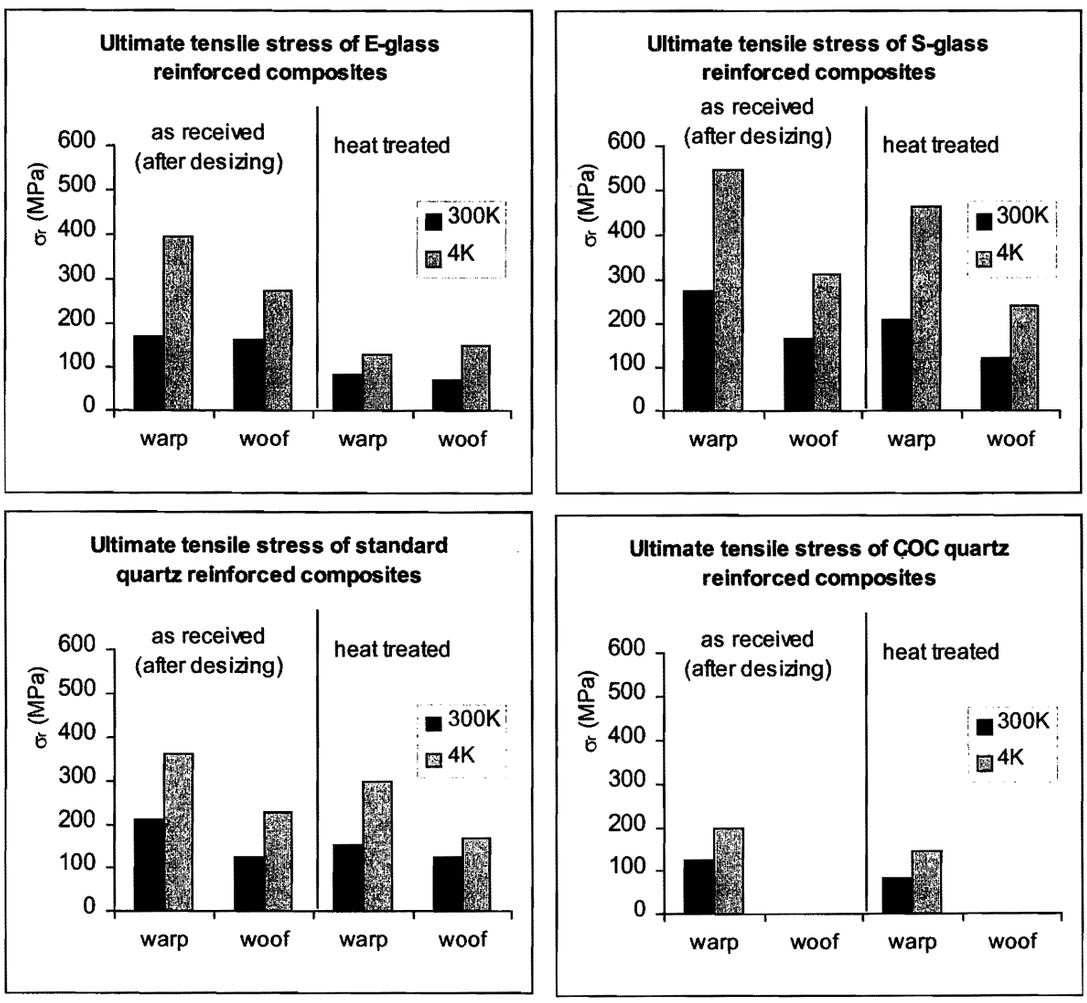

Figure 2 : Effect of load direction, cooling and heat treatment on the ultimate tensile stress of the different insulation systems. (Note that the fiber volumetric fraction is different for each system and lies between 30 and 40%).

It appears also that the shape of the stress-strain curve at 4.2 K is different from the shape of the room-temperature curve. All room-temperature curves increase quasi-linearly up to the rupture while most of the 4.2-K curves exhibit a marked slope change. This slope change can be attributed to the failure of the epoxy resin matrix. It occurs for a tensile strain between 0.7 and 1.4%. It is likely that, below this strain, the matrix and the fiber reinforcement both contribute to the mechanical strength, while, beyond this strain, the mechanical strength is solely provided by the fiber reinforcement.

From curves such as those in Fig. 1, the Young's modulus of the various systems is estimated between 15 and 25 GPa at room temperature. It can reach 33 GPa at 4.2 K. Within the measurement errors, no clear effects of the loading direction and of the heat treatment at 660 °C can be seen on the Young's modulus.

The role played by the loading direction and the heat treatment is more readily brought into light by the ultimate tensile stress data summarized in Fig. 2.

Regarding the effects of loading direction, the maximum tensile strength corresponds almost systematically to the warp direction.[11] Note, however, that for the samples taken along the woof direction, the fracture face is always located between turns of the wrapping. This fact is observed both at room temperature and at cryogenic temperature.

Regarding the effects of heat treatment, it leads, for all composites, to a significant decrease of the strength to rupture. As could be expected from the low recrystallization temperature, the maximum decrease is obtained for the samples reinforced with E-glass fiber tapes. For the samples reinforced with S2-glass fiber tapes and standard quartz fiber tapes, the decrease in the ultimate tensile stress is between 15 and 25%, while it reaches 35% for the samples reinforced with COC quartz fiber tapes. The low values measured for the COC quartz fibers may be due to the corrosion problems encountered during tape weaving. The tests with and without heat treatment will be duplicated on the new COC quartz fiber tape that is in production. Let us finally point out that, for all insulation systems, the effect of heat treatment is of the same order of magnitude at room temperature and at 4.2 K.

In overall, the results of these mechanical measurements are rather encouraging. When looking for a trade-off between tensile properties and small turn-to-turn insulation thickness, quartz-reinforced polymers appear to have a good potential for our applications.

DIELECTRIC TESTS

The turn-to-turn insulation must withstand voltages of the order of 1 kV/mm to be acceptable for magnet design. In order to evaluate and compare the dielectric properties of the different insulation systems described above, two series of tests were performed: (1) a series in helium environment at different temperatures, so as to verify that the insulation satisfies the aforementioned criterion, and (2) a series in oil at room temperature, so as to determine the breakdown voltage. In both cases, the samples were cut out from composite plates representative of the turn-to-turn insulation in a magnet coil. Samples' thickness varied from 0.24 to 0.45 mm.

The first series of tests were carried out at CEA/Saclay. The samples were cut out in the shape of disks, with a 19-mm diameter. The disks were tested by pairs, with a cathode on the top and a cathode on the bottom, and a common anode in between. The electrodes had an 8-mm diameter and were connected to a voltage generator which could supply up to 5.8 kV (corresponding to 13 to 25 kV/mm). During the tests, the samples were held under a transverse pressure of 70 MPa, reproducing the operating conditions of the turn-to-turn insulation in a magnet coil. The tests were carried out in liquid helium at 4.2 K and in gaseous helium at 4.2 K, 100 K and 300 K to simulate a temperature rise consecutive to a quench.

A total of four to five samples were tested per type of insulation system. All samples passed the 1 kV/mm criterion at all temperatures. All samples were also able to sustain the maximum voltage of 5.8 kV at 4.2 K in liquid and gaseous helium. One failure was observed at 100 K on one sample relying on heat-treated, type-E glass fiber tape. This failure occurred at the edge of a layer wrap for a voltage of about 4.7 kV (corresponding to

Table 3. Breakdown voltages of various samples of insulation systems measured in oil at room temperature.

Denomination	Heat Treatment at 660 °C	Sample Thickness (mm)	Breakdown Voltage (kV)	Breakdown Voltage per Unit Length (kV/mm)
EE	No	0.40	12.5±1.5	31
EE	Yes	0.44	15.8±1.1	36
SS	No	0.38	14.5±1.0	38
SS	Yes	0.35	16.0±0.5	45
EM	No	0.35	16.0±1.0	46
EM	Yes	0.40	16.2±1.0	41
SM	No	0.30	15.5±0.5	52
SM	Yes	0.24	17.2±0.5	73
QS	No	0.25	12.6±0.5	51
QS	Yes	0.24	13.2±1.1	55
QH	No	0.25	13.6±0.9	54
QH	Yes	0.26	13.2±0.8	51

10.9 kV/mm, hence well above 1 kV/mm). It may have been caused by residues left over from the organic thread used in the weaving process to mark the tape edges. For all other samples at 100 K, the tests were limited by the bypass voltage in helium.

The second series of tests were carried out by the Magnet and Superconductor Application (MSA) unit of Alstom Energie S.A.,[12] according to the French standard defining the methods of test for electric strength of solid insulating materials at power frequencies.[13] The samples were taken from composite plates similar to the ones used to cut out the disks and were immersed in oil at room temperature. The breakdown voltages measured during these tests are summarized in Table 3. The quoted values are averaged over 5 tests. The first column refers to the denominations defined in Table 2, while the second column specifies if the fiber tapes and the mica-glass foils have been or not heat-treated at 660 °C prior to wrapping.

The data of Table 3 show that the insulation systems with the poorest voltage breakdown per unit length are those relying on E-glass fiber tapes (31 to 36 kV/mm). The introduction of the mica-glass appears to raise significantly the dielectric strength of both types of glass fiber tape reinforcements. Some of the best results are nevertheless obtained for the insulation systems relying on quartz fiber tapes, which always exceed 50 kV/mm.

CONCLUSION

We have investigated in detail the tensile behavior and the dielectric strength of various systems that can be considered to insulate Nb_3Sn particle accelerator magnet coils manufactured by the "wind and react" technique. The systems relying on quartz fiber tapes wrapped with a 50% overlap exhibit some promising results which encourage us to pursue their development. It remains, however, that the manipulation of thin, de-sized, mineral fiber tapes is very delicate and that more work has to be done to improve the robustness of the conductor wrap prior to vacuum impregnation.

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