Insulation Development for the Next European Dipole

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***Abstract*— The Next European Dipole (NED) Consortium is working to develop the technology of high field, dipole magnets for a future luminosity upgrade of the LHC at CERN. The proposed magnet will be a large aperture (88mm), high field (15T) superconducting dipole using Nb_3Sn Rutherford cable and a wind-and-react manufacturing scheme. The magnet scale, forces and manufacturing route present real challenges for the conductor insulation technology. This paper reports on the insulation R&D programmes carried out during the first phase of the NED project (2004 to 2007). In the R&D programmes both conventional, (glass fibre-polymeric matrix) and innovative (ceramic) insulation materials have been studied. A primary objective was to develop a working insulation specification for a dipole magnet at the NED scale. Specialized sample geometries and test methods (mechanical and electrical) have been developed to make comparison between different insulation materials and processing routes. In particular, the role of sizing on glass fibre insulation subjected to Nb_3Sn magnet processing parameters has been studied using thermo gravimetric analysis.**

***Index Terms*—Accelerator magnets, Epoxy resins, Insulation testing, Materials testing**

I. INTRODUCTION

THE Next European Dipole (NED) consortium is working to develop the technology of high field dipole magnets for a future luminosity upgrade of the LHC at CERN [1]. The proposed magnet will be a large aperture (88mm diameter), high field (15T) superconducting dipole magnet using Nb_3Sn Rutherford cable and a wind-and-react manufacturing scheme. From the start of the NED project the development of insulation technology was seen as a key factor for these magnets. Two strands of insulation development were initiated. Insulation research and development at STFC-RAL in the UK has focused on “conventional” insulation, glass fibre tape with an organic matrix. In parallel, an alternative, “innovative” inorganic insulation system is being been developed at CEA-Saclay.

A. *Insulation Specification*

An insulation specification for NED magnets was developed through the NED Insulation Development Working Group. Key specification parameters are given in Table 1. In a luminosity upgrade of the LHC the magnets and insulation will be subjected to a high fluence of secondary particles. The insulation must also exhibit good radiation resistance.

TABLE 1 NED INSULATION SPECIFICATION

GENERAL	**Comment**
Insulation thickness per cable	0.2 mm
Winding compatibility: Capable of being applied to the cable and formed into a dipole winding by a semi-automatic winding system	Minimal fraying or abrasion during winding
Compatible with Nb3Sn heat treatment cycle, 660ºC for tens of hours in argon or vacuum	Minimal degradation of basic components

MECHANICAL	**Failure stress**
Compressive stress after completion of coil fabrication – at 300 K and 4 K.	400 MPa
Shear: Short-beam shear strength at 4 K	100 MPa
Tension: Transverse tensile strength of insulation laminate at 4 K	50 MPa

ELECTRICAL	**Failure**
Breakdown voltage inter-turn tested at 2 kV	5 kV/mm

II. CONVENTIONAL INSULATION PROGRAMME

The following sections, II – VI, relate to the conventional insulation programme. The goal of the programme is to develop an industrially viable insulation system for the production of high-field accelerator magnets. To this end, we have focused on materials that are compatible with the Nb_3Sn magnet manufacturing process and which are available in industry. The work programme comprises 4 phases:

1) The identification and analysis of materials and the development of testing and characterisation techniques
2) The manufacture of standard glass-epoxy laminates to compare material variables and quantify the effect of sizing using TGA, electrical breakdown and mechanical testing
3) The manufacture of cable-stack, test pieces to study the performance of the insulation in conjunction with the cable
4) The manufacture of short model racetrack coils (420mm long by 190 mm wide) with a double pancake geometry to enable testing of the proposed conventional insulation in a real magnet. The short model coils (SMC) will be fabricated by STFC-RAL and tested at CERN.

Manuscript received August 22, 2007. This work is supported in part by the European Community-Research Infrastructure Activity under the FP6 "Structuring the European Research Area" (CARE, contract number RII3-CT-2003-506395. This work is also supported by the STFC Strategic Initiatives Programme.

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Phases 1 and 2 are largely complete: phases 3 and 4 are currently proceeding.

III. FIBRE MATERIALS

A. *General requirements*

The materials, fibres and tapes, should be available on a commercial rather than laboratory scale i.e.: in kilometric quantity. The materials need to be durable for winding and capable of withstanding the reaction heat treatment phase. The basic fibre material should not be susceptible to radio-activation e.g. the boron content of E-glass is undesirable. The insulation of Nb_3Sn magnet conductors is most commonly made with S-glass tape or a braided sleeve.

B. *Fibre sizing*

Sizing technology was identified as a key development area at the beginning of the NED programme. Glass fibres are coated with a mixture of organic materials immediately after fibre drawing. This is known as a "sizing". The sizing renders the fibre durable and compatible with subsequent weaving and bonding to organic matrix materials. Sizing formulations are proprietary and depend on the final application. The organic materials in the sizing decompose during the reaction heat treatment leaving a carbon residue which degrades the insulation electrical and mechanical properties. One approach is to remove the organic sizing prior to winding. However, this renders the tape fragile and susceptible to damage in a semi-automatic tape wrapping or coil winding process. It is important for the fibres to be protected by a sizing during magnet fabrication.

1) *High temperature sizing*

We identified a commercially available S-glass fibre cloth marketed for high temperature applications by JPS Composites [2]. This material showed promising electrical and mechanical properties under initial tests.

We have used Thermo Gravimetric Analysis (TGA) to measure the weight loss of the sizing during the reaction heat treatment. The TGA apparatus comprises a furnace, within which a small sample, typically weighing a few milligrams, is suspended. An ultra-microbalance weighs the sample and a computer logs the weight with time and temperature. The sample environment and balance was purged with argon gas.

Using the TGA apparatus we have found that the sequence of weaving and sizing (type and quantity) is critical to the electrical and mechanical properties of the final insulation. To enable weaving into a tape, more than 1% sizing by weight is required to lubricate the fibres and keep fibre damage to a minimum. We refer to this as "yarn sizing". If it is left in place, this sizing degrades during reaction and degrades the electrical and mechanical properties of the final insulation laminate. So, a conventional sizing was applied to the yarn to enable weaving of the tape. After de-sizing, a small amount of high temperature sizing was applied to the woven tape. We refer to this as "woven-resized". The resulting tape is much more robust than a desized tape and offers a much better product for cable insulation and magnet winding. After heat treatment and epoxy impregnation a laminate made by this process is light grey in colour from degradation of the small amount of sizing. After close consultation, JPS have adapted the technology to produce a thin S-glass tape (0.1 mm) with high temperature sizing.

2) *Electrical breakdown strength testing*

For electrical breakdown testing, glass fibre was heat treated at 660ºC for 24 hours. A 0.4mm thick laminate was made using four plies of glass tape and DGEBF/DETD epoxy. Electrical breakdown strength was measured to B.S.7831. TGA and electrical test results are presented in Table 2. This shows the excellent electrical properties of woven-resized tape.

TABLE 2 TGA AND ELECTRICAL BREAKDOWN RESULTS

Material	Process	Weight loss 660C %	Electrical breakdown kV/mm	Colour
S glass	Commercial sizing	1.2%	2.5 (FAIL)	Black
JPS S-glass+ HT sizing	Yarn-sizing	1.2-1.6	0.5 (FAIL)	Black
JPS S-glass+ HT sizing	Woven-resized	0.2-0.4	15 (PASS)	Light grey

IV. MATRIX MATERIALS

A. *Impregnation matrix*

In order to develop the best overall insulation and bonding system it is necessary to consider the matrix materials as well as the fibre materials. Conventionally, to bond the insulation fibre and cable into a monolithic coil of accurate dimensions, an epoxy resin matrix is used. The epoxy must have low initial viscosity and a useable life long enough to allow impregnation of the coil. A tough matrix material is desirable to reduce cracking due to cooldown and magnetic stresses and minimize energy releases which could lead to magnet quenching. The intrinsic electrical breakdown strength of organic matrices is high, but can be reduced by carbon residue from thermal decomposition of fibre sizing. We have therefore carried out a matrix evaluation programme. The standard epoxy used was a Bisphenol-F epoxy resin (e.g. D.E.R.354 from Dow) with DETD curing agent (e.g. Ethacure 100 from Albemarle) [3]. This is a low cost, low viscosity epoxy and the aromatic nature of the hardener confers good radiation stability.

B. *Alternative matrix candidates*

1) *Tetra-functional epoxies*

Evans has shown [4] that epoxies with functionalities of 3 or 4 are more radiation stable compared to conventional epoxies of lower functionality. An epoxy such as TGDM (Tetra Glycidyl Diamino Diphenyl Methane) for example Ciba MY722, has a functionality of 4. This material was included in the test programme with Ethacure 100 hardener. It is referred to as "TF epoxy" in the results.

2) *Cyanate Ester*

Cyanate ester materials, which show promise as radiation hard matrices, have been evaluated for uses in fusion magnet applications such as ITER. However, their high cost, up to ten times that of standard epoxies, has led to the testing of cyanate ester and epoxy blends. A cyanate ester with very low viscosity, EX1545 (Bryte), was sourced for this programme.

C. *Resin additives*

Two resin additives were included in the programme. A silane additive, GLYMO from Degussa, was used at 1% addition with the aim of improving glass fibre to matrix and matrix to metal bonding. Nanofillers are of interest because they offer the potential to reinforce epoxy polymers at low addition levels which would not affect viscosity and impregnation properties. These fillers interact with the epoxy at the nano scale, that is, in the free volume between polymer chains. We used a nanoclay filler, Nanomer 1.30e, from Nanocor Inc to evaluate their influence.

V. EXPERIMENTAL MECHANICAL STUDIES

A. *Sample Manufacture*

Composite panels, 150 mm square and 3 m thick, were manufactured from woven glass fibre by vacuum impregnation at 0.1mbar with well de-gassed epoxy. The standard epoxy formulation was used for most panels; Bisphenol-F epoxy resin with DETD hardener. The mixing ratio was 100:26.4 by weight. The panels were cured at a compaction stress of 1MPa in a heated press.

B. *Mechanical testing*

The composite panels were cut into samples for short beam shear and interlaminar fracture testing. Short beam shear, (SBS), testing was performed at 77K in liquid nitrogen to ASTM D2344. Interlaminar fracture to ASTM D5528 was used to measure work of fracture (WOF). Table 3 shows the results of tests made with three different types of fibre. Table 4 shows results of tests with different matrix materials.

TABLE 3 RESULTS OF LAMINATES WITH CONTROL MATRIX

Fibre type	**Sizing**	**Heat treatment**	**WOF kJ/m2**	**SBS strength MPa (77K)**
S-glass	Commercial, as-received	None	0.49	91
S-glass	De-sized	350°C in Air	0.85	97
JPS S-glass	High-temp	660°C 60h vacuum	0.67	98
JPS Quartz	High-temp	660°C 60h vacuum	0.88	64

TABLE 4 RESULTS OF S-GLASS LAMINATES MADE WITH DIFFERENT MATRIX FORMULATIONS

Epoxy	**Sizing**	**Heat treatment**	**WOF kJ/m2**	**SBS strength MPa at 77K**
Standard+ Nanoclay 1%	Commercial as-received	None	0.51	72
Standard+ Nanoclay 5%	Commercial as-received	None	0.50	101
Standard +GLYMO 1%	Desized	660°C 20h vac	0.9	83
Standard +GLYMO 1%	JPS glass + High-temp	660°C 20h vac	0.63	102
TF epoxy	Commercial as-received	None	0.36	45
CE1545	Commercial as-received	None	0.4	75

C. *Discussion*

The JPS S-glass with high temperature sizing shows better mechanical properties after heat treatment than the baseline S-glass with no heat treatment. The quartz based laminate has higher work of fracture but lower short beam shear strength than the S-glass laminates. On this basis, JPS S-glass tape with high temperature sizing was selected as the insulation material for the short model coil programme.

The laminate fabricated with nanoclay additive does not show any significant increase in work of fracture at either 1% or 5% addition level. Further characterization of this material is merited to determine if it was properly compounded into the epoxy.

The results in Table 4 suggest the effect of the GLYMO sizing additive on work of fracture and short beam shear strength is small. It is worth noting that these results are all higher than the work of fracture for the control material. Sizing additives remain of interest as they can be expected to enhance the bond strength to the copper surfaces of the cable after reaction.

Both alternative, radiation hard matrices show lower mechanical properties than the control epoxy. The results in Tables 3 and 4 give properties before any irradiation. Work needs to be carried out to determine the effect of irradiation on all these materials. One can reasonably expect the radiation hard materials to maintain their properties better than the control epoxy material under irradiation.

VI. CABLE STACK TESTING

The testing of laminates yields useful information to screen candidate materials and process changes. In a magnet, the insulation is required to bond to the cable and the fibre-to-matrix ratio of the insulation will vary strongly between the wires and interstices of the cable. It is therefore important to study the mechanical behaviour of the insulation when processed with a cable. Two types of cable stack test are under development. The cables will be wrapped with glass fibre tape, heat treated and vacuum impregnated to simulate magnet manufacturing. Samples with two cables will be fabricated and tested in short-beam-shear to quantify the bond between the matrix and copper cable. Stacks of ten cables will be used to study elastic modulus and thermal contraction properties.

VII. INNOVATIVE INSULATION DEVELOPMENT

The implementation of the Nb_3Sn remains delicate because of the great brittleness of the material after the heat treatment. The conventional insulation requires winding the coil with a desized glass tape and performing a vacuum resin impregnation after the heat treatment. The question of the weak properties of the desized tape has been addressed in the previous sections on conventional insulation development but the multi-step process is costly and raises the failure risk. As an alternative to the glass/epoxy insulation, CEA Saclay has been working on one-step “innovative” ceramic insulation deposited directly on the un-reacted superconducting cable. The aim is to obtain, after the reaction heat treatment, a coil matrix with good mechanical cohesion, while maintaining a suitable electrical insulation. The feasibility of this insulation has been demonstrated [5].

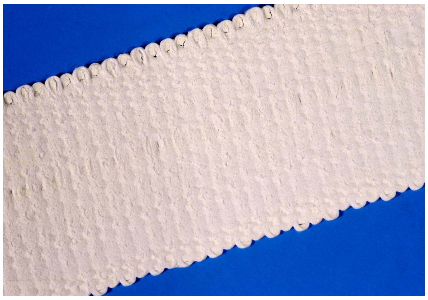

Fig. 1. Innovative insulation tape with ceramic pre-cursor

An example of tape impregnated with ceramic precursors used to wrap the conductor is shown in Fig.1. We have also shown that using this insulation in a coil manufacturing process does not affect the transport-current properties of the Nb_3Sn wires [6].

The development and test programme has continued with the mechanical characterisation of the insulation, beginning with the properties in compression. The tests have been made on stacks of ten Rutherford type, Nb_3Sn cables wrapped with innovative insulation and reacted. The tests have been carried out with an Instron screw-driven machine, the load being measured with a 150 kN cell, see Fig.2. The first results obtained after three cycles of uniaxial compression up to a stress level of 62.5 MPa at room temperature and 4.5 K are presented Fig.3 [7].

Preliminary heat transfer experiments have been performed on a stack of five insulated conductors reacted under mechanical constraint at a pressure of 10 MPa, see Fig.4. The first results show a ΔT at least five times lower than the ΔT measured during tests with polyimide insulated samples such as LHC cable insulation [8].

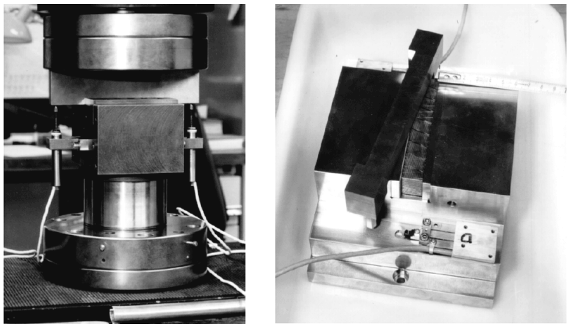

Fig.2. Measurement cell for compression test on stacks

VIII. CONCLUSIONS

A commercially available "conventional", glass fibre tape has been developed in consultation with JPS Composites Inc. which we believe is compatible with the manufacture of future Nb_3Sn, accelerator magnets by the wind-and-react process. Future work will focus on the fabrication and testing of short model coils to confirm the performance of this material in a real magnet.

The effect of radiation on insulation materials is critical to the success of magnets in the LHC upgrade. This will be studied using materials already produced for this programme.

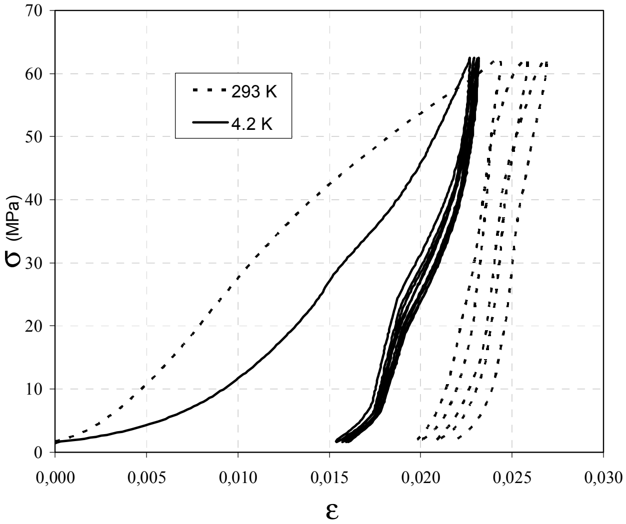

Fig.3. Stress-strain curves for compression of a 10 cable stack up to 62.5 MPa

Fig. 4 Sample for heat transfer experiment

Development of "innovative" insulation is continuing to characterize and improve the mechanical properties after heat treatment as well as to qualify the thermal behavior of the insulation. The next step will be to apply the ceramic insulation in a real magnet as part of the short model coil programme.

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