EU contract number RII3CT-2003-506395 CARE Conf-05-034-NED

CRITICAL CURRENT MEASUREMENTS ON Nb_3Sn CONDUCTORS FOR THE NED PROJECT

A. den Ouden, T. Boutboul, D. Pedrini, V. Previtali, L. Quettier and G. Volpini

Abstract

The Next European Dipole (NED) project is a EU funded program aiming at the realization of a 15 T accelerator-type of Nb_3Sn dipole magnet. The objective of NED phase one is developing high performance Nb_3Sn conductor in collaboration with European industry. The envisaged Rutherford type of cable consists of about 40 superconducting strands exhibiting a non-copper critical current density of 1500 A/mm^2 at 4.2 K and 15 T. In the frame of conductor development, adequate critical current measurements in terms of accuracy and reproducibility should be ensured to qualify the ambitious strands produced. Therefore, a NED working group focuses at the establishment and implementation of appropriate and standardized procedures for sample preparation and mounting, critical current measurement, data analysis and reporting. A cross-calibration program between the institutes involved has been initiated by exchanging samples of Nb3Sn conductor from a single billet for critical current measurements, prepared at each institute. The experimental results of this program and the conclusive procedures for NED critical current measurements are presented and discussed.

Contribution to the MT19 conference 2005, Genova, Italy

Work supported by the European Community-Research Infrastructure Activity under the FP6 « Structuring the European Research Area » programme (CARE, contract number RII3-CT-2003-506395).

Critical current measurements on Nb_3Sn conductors for the NED project

A. den Ouden, T. Boutboul, D. Pedrini, V. Previtali, L. Quettier and G. Volpini

Abstract— **The Next European Dipole (NED) project is a EU funded program aiming at the realization of a 15 T accelerator-type of Nb_3Sn dipole magnet. The objective of NED phase one is developing high performance Nb_3Sn conductor in collaboration with European industry. The envisaged Rutherford type of cable consists of about 40 superconducting strands exhibiting a non-copper critical current density of 1500 A/mm^2 at 4.2 K and 15 T. In the frame of conductor development, adequate critical current measurements in terms of accuracy and reproducibility should be ensured to qualify the ambitious strands produced. Therefore, a NED working group focuses at the establishment and implementation of appropriate and standardized procedures for sample preparation and mounting, critical current measurement, data analysis and reporting. A cross-calibration program between the institutes involved has been initiated by exchanging samples of Nb_3Sn conductor from a single billet for critical current measurements, prepared at each institute. The experimental results of this program and the conclusive procedures for NED critical current measurements are presented and discussed.**

Index Terms—**Accelerator magnets, superconducting filaments wires, Niobium compounds**

TABLE I

NED NB_3SN CONDUCTOR SPECIFICATIONS

	strand	
strand diameter	1.25	mm
Cu:non Cu (λ)	$1.15 < \lambda < 1.35$	-
filament twist pitch	30	mm
geometric diameter Nb_3Sn region in filament	< 50	µm
I_c @ 4.2 K (10 µV/m criterion) at 12 T	> 1636	A
at 15 T	> 818	A
RRR after heat treatment at 675 ^{0}C	>150	-
	Rutherford cable	
number of strands	40	
mid thickness	2.275	mm
width	26.00	mm
keystone angle	0.22	
allowed I_c degradation due to cabling	10	%
I_c @ 4.2 K (10 µV/m criterion) at 12 T	58.9	kA
at 15 T	29.4	kA
RRR after heat treatment at 675 ^{0}C	> 70	

I. INTRODUCTION

The objective of NED phase one is developing high performance Nb_3Sn conductor in collaboration with European industry. The envisaged NED Nb_3Sn wires and cables are expected to generate a dipole field of 13-15 T at 4.3 K in a bore of about 88 mm [1]. Assuming a copper percentage in the wire cross section of 55 % (Cu:non Cu ~ 1.2) the wires should exhibit a non-copper J_c of about 1500 A/mm^2@15T to obtain such a field. Assuming a 2-layer cosθ reference coil design the corresponding strand and cable requirements are summarized in Table I. Industrial development of monolithic conductors exhibiting such unprecedented properties is a major ambition of the NED program and calls for reliable, reproducible and unambiguous methods for the measurement of their electromagnetic properties $I_c(B)$, $M(B)$ and $RRR(B=0)$. A Working Group on Conductor Characterization (WGCC) with representatives from CERN, CEA-Saclay, INFN-Milano, INFN-Genova and the University of Twente is charged with the definition and development of standardized and certified procedures to measure the mentioned properties of virgin, deformed and extracted strands. In this paper the focus is on critical current measurements.

A. *The concept of 'critical current' for Nb_3Sn conductors*

By convention the critical current I_c in a superconductor is the current where the longitudinal electrical field reaches the I_c criterion E_0 under accurately known (B,T,ε)-conditions. For conductors like NbTi, for which I_c is hardly sensitive to strain, above definition justifies a straightforward measurement of $I_c(B,T)$ ignoring the precise strain state. In contrast, the critical current of Nb_3Sn and most ceramic HTC conductors shows a rather strong dependency on strain, principally excluding such an I_c determination if exact knowledge of the strain state is lacking. However, $I_c(B,T,\varepsilon)$ experiments are expensive, lasting and difficult to perform and therefore unattractive as an experimental qualification procedure, especially if tenths of samples need verification [2], [3]. Besides, in tangible applications of Nb_3Sn conductors precise knowledge about the strain state throughout the manufacturing process usually lacks or is hard to predict, especially without detailed knowledge of the (σ,ε)-relation throughout the whole process.

Manuscript received September 15, 2005. This work was supported in part by the European Community-Research Infrastructure Activity under the FP6 "Structuring the European Research Area" program (CARE, contract number RII3-CT-2003-506395)

A. den Ouden is with the University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands (phone: +31534893190; fax: +31534891099; e-mail: a.denouden@utwente.nl).

D. Pedrini and G. Volpini are with INFN-LASA, 2009 Segrate ,Milano, Italy

T. Boutboul and V. Previtali are with CERN, 1211 Geneva 23, Switzerland

L. Quettier is with CEA-Saclay, 91191 Gif-sur-Yvette, France

Above implies that the concept of ‘critical current’ of a strain sensitive superconductor only holds if general agreement exists on standardized experimental circumstances for a critical current measurement. Despite the valuable recommendations from the VAMAS an ITER programs, this is unfortunately still not the case [4]-[10]

B. *Conditions affecting Nb_3Sn I_c measurement*

Table II summarizes the capabilities and experimental boundary conditions of the I_c-measurement facilities at the participating labs in the WGCC. For Nb_3Sn conductors the I_c as a function of field, temperature and strain can be fairly accurately predicted by applying phenomenological scaling relations [3]. At a constant strain and constant applied magnetic field the critical current normalized to its value at 4.20 K varies as a function of temperature nearly linearly from 15 %/K at 12 T to 25 %/K at 16 T. Assuming an uncertainty of the sample temperature of 30 mK, its influence on I_c amounts to 0.75 % maximum in this field range.

A similar linear dependency of the applied field on the critical current normalized to its value at 13 T and 4.2 K amounts to 20 %/T which varies only slightly at different normalization fields. With a typical field uncertainty at the sample of 20 mT the resulting error in I_c amounts to 0.4 %.

The strain state of the reacted Nb_3Sn in the composite sample wire is by far the most uncertain factor affecting the I_c rather severely. It is mainly determined by: 1) the tension during sample winding and wire fixation after winding, 2) the bending stress which is a function of the wire diameter and the radius of the sample holder, 3) the mechanical properties of the wire components and the sample holder during the heat treatment, 4) the temperature ramp to the reaction temperature at about 675 ^{0}C, 5) eventual sample transfer after the heat treatment to another holder and finally 6) the relative thermal contraction of the composite wire and the sample holder during heat treatment, soldering to the current leads and the cool-down to 4.2 K.

Though above factors depend also on the type of Nb_3Sn conductor, the strain response of the critical current at 12 T and 4.2 K for Nb_3Sn wire samples typically amounts to 140 %/% in terms of critical current normalized to the maximum in the I_c vs. strain curve [2], [3].

Even without knowing the final strain state of the sample a fair and sensible judgment of reproducibility and accuracy of I_c measurements on Nb_3Sn wires necessitates control of all above factors.

TABLE II

CHARACTERISTICS I_C MEASUREMENT FACILITIES

	CEA	INFN	UT
maximum current (A)	1000	1500	1200
current uncertainty (%)	< 0.1	< 0.1	< 0.1
maximum field (T)	15	13.5	15
field uncertainty at sample (%)	0.2	0.2	0.1
magnet bore (mm)	50	60	80
sample temperature (K)	1.8 – 4.2	4.2	4.2
temperature uncertainty 4.22 K (mK)	30	30	30

II. INTER-LABORATORY CROSS-CALIBRATION PROGRAM

Inspired by experiences of the VAMAS program and the successful development of standardized procedures for specifying and measuring the critical current of ITER conductors [10] the WGCC has performed a program of cross-calibration between the various test facilities with the purpose to: 1) reveal possible systematic differences of $I_c(B)$ results from samples of identical wires prepared and measured at each lab, 2) get acquainted to preparation and measurement of such large, very high I_c samples shaped as round or flat rolled wire or as strand extracted from a Rutherford cable, 3) bring forward shortcomings or limitations in existing preparation or measuring procedures and agree on a roadmap to improvements, 4) enable sample exchange between the labs 5) set up procedures for dealing with conflicting results, 6) set up agreed and approved procedures for sample mounting, measuring procedures and data analysis and 7) develop standard reporting.

A. *Stage 1:*

As a first step each lab independently prepared a series of wire samples and measured the $I_c(B)$ and n-value at 4.2 K (10 µV/m criterion, n-value determined between 5 and 50 µV/m) to the extend of their (I,B) capabilities. This first series comprised 2 samples each of a 1.06 mm LHC NbTi wire, a binary 1.26 mm Nb_3Sn wire and finally both a virgin and an extracted strand from a Rutherford cable made out of a binary 0.8 mm Nb_3Sn wire. Initially each laboratory performed heat treatment, sample preparation, measurements and report according to existing procedures. The results of this stage-1 inter-lab comparison are summarized in Table III, in which ΔI_{max} is the maximum difference in I_c between the labs and ΔI_{lab} is the maximum spread per lab for a single type of wire.

Regarding the uncertainty in temperature and field mentioned in Table II the differences in I_c of the NbTi LHC wire, referenced by very accurate measurement at CERN, are within the range of expectation. The differences for the virgin Nb_3Sn wires however are unacceptably large, most probably due to differences in sample strain as a result of differences in sample mounting and unguarded temperature evolution during the sample heat treatment. The relatively small differences for the extracted strands should be considered statistically normal but coincidentally small. Consequently a thorough comparison of sample holder lay-out, mounting and measuring procedures has been carried out.

TABLE III

RESULTS I_C MEASUREMENTS STAGE 1

	NbTi	Nb_3Sn virgin	Nb_3Sn virgin	Nb_3Sn extracted
diameter (mm)	1.06	1.26	0.84	0.84
# samples per institute	2	2	2	1
ΔI_{max} (%)	8	15	12	4
ΔI_{lab} (%)	3	7	5	-
systematic trend	lab 3 > lab 1 > lab 2			

As a result the 3 labs agreed all to employ the standard grooved TiAlV sample holder and corresponding copper rings, referred to as the ITER barrel, and alter the cryogenic insert accordingly if necessary. After mounting and screw fixation to the copper rings the sample wire remains fixed to the sample holder during the heat treatment, soldering to the copper rings and cool-down to 4.2 K. The design of the copper rings is adapted such to enable sample exchange between the labs. Furthermore, reporting will include the monitored temperatures during the sample heat treatment and thermometers will be attached to the sample holder during the I_c measurements. At this stage details of mounting procedures have been discussed but no further restrictions on sample preparation have been imposed.

B. *Stage 2:*

After the above mentioned adaptations 2 labs prepared 3 samples of the same 1.26 mm Nb_3Sn wire used in stage 1. Subsequently in stage 2-1 all labs performed an I_c measurement on all samples between 10 and 15 T at (4.22 ± 0.03) K.

Fig. 1 displays the results of these measurements arranged per lab which prepared the samples (solid markers). A first remarkable observation is the large difference between the samples prepared at each lab in the order of 15 %. This could be an indication that the sample wire exhibits inhomogeneous properties along its length, which would make this wire rather useless as reference wire. Secondly, I_c changes after subsequent thermal cycles by 3% for samples prepared at lab 2 and 6 % for samples at lab 1. Though the direction is rather random, qualitatively this behavior has also been observed by Goodrich [8] which most conceivably results from stress redistribution along the sample wire.

Above results are still non-discriminative with respect to the measuring procedures. Therefore this stage has been extended with stage 2-2, in which 3 samples were measured again by all labs, indicated by the open markers in fig. 1. The I_c of sample NED10 shows a large decrease of about 125 A, but like the other re-tested samples NED13 and NED15 the results of the 3 labs agree within 2 %. Though after 3-4 thermal cycles full stress relaxation has been established in all 3 samples, even the absolute differences between NED13 and NED15, prepared at the same lab by the same person, still amounts to 7 % which again supports the suspicion of longitudinal in-homogeneity of the sample wire. An important conclusion at this stage however is that despite some differences in the measuring procedure, the relative differences between I_c values obtained at different labs have converged to an acceptable level of ± 2%.

Above results have lead to the general conclusion that a standard sample preparation prescribed in detail must be respected to reach the highest level of relative accuracy if I_c results of samples prepared at different labs are compared. The finally agreed detailed procedures, which resemble the ITER protocol to a high degree, are summarized in Table V. The major accepted differences concern the thickness of the TiAlV barrel (related to differences in both the fixation of the copper rings to the barrel and the connection to the current leads) and voltage monitoring during the transition either at static currents (after a waiting time of about 5 seconds after each current step) or dynamically during a slow current ramp.

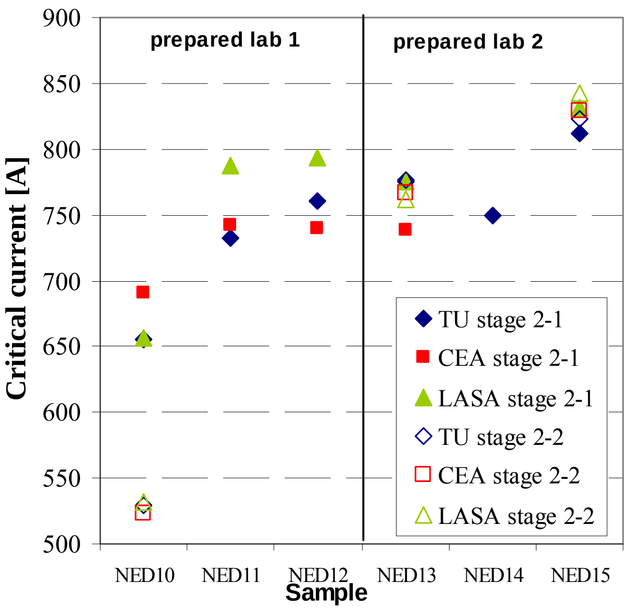

Fig. 1. Results of the I_c measurements at 12 T@4.22 K of stage 2-1 (solid markers) and stage 2-2 (open markers) on Nb_3Sn conductor samples prepared at 2 different labs. The legend indicates where the samples were measured.

C. *Stage 3:*

After obeying strictly the described sample mounting procedures from Table V, the last stage 3 of this program comprised the I_c measurement of 2 samples of the previous 1.26 mm Nb_3Sn wire, a flat rolled (unsupported, single sided reduction of 0.35 mm) version of the same wire and a state-of-the-art PIT-Nb_3Sn wire of 1 mm diameter, reaching a non-copper J_c at 12 T of about 2500 A/mm^2. Flat rolling to a compaction comparable to what is obtained in a Rutherford cable may be a viable, efficient and controlled way to predict the possible I_c degradation of the same wire in a Rutherford cable [12]. The results in Table IV show, that for the last wire the restrictions in sample preparation indeed results in an acceptable qualification window of 2 % between the labs and less than 1 % at each lab. Though a decrease in I_c of about 40 % of the flat rolled wire are observed both ΔI_{abs} max. and ΔI_{lab} max. compare quite well to the differences observed for

TABLE IV
RESULTS STAGE 3 MEASUREMENTS

	Nb_3Sn virgin	Nb_3Sn flat rolled	Nb_3Sn virgin
diameter (mm)	1.26	0.91/>1.26	1.0
Cu:non Cu	1.25	1.25	0.82
# samples per institute	2	2	2
ΔI_{abs} max. (%)	8	4	2
ΔI_{lab} max. (%)	4	3	0.6
systematic trend		none	

the virgin wire. This confirms again the longitudinal inhomogeneity of this wire but strongly supports the convergence to the required accuracy for comparing I_c results from samples prepared and measured at different labs.

TABLE V

NED SAMPLE MOUNTING AND I_C MEASUREMENT PROCEDURES

Before heat treatment	
TiAlV barrel dimensions (R_ixR_oxH) (mm)	14.2 x 32 x 32.5 (CEA,INFN) 28 x 32 x 32 (UT)
TiAlV barrel oxidized in air at ~ 250 ^{0}C	yes
wire sample cleaned	alcohol
TiAlV barrel and copper rings cleaned	alcohol or ultrasonic soap
copper rings fixed (axially and azimuthally) to barrel	stainless steel screws (CEA,INFN), punched holes (UT)
slit in copper rings	yes (CEA), no (INFN,UT)
initial wire fixation to the first copper ring	screw tensioning the wire
wire winding tension (N/mm^2)	15
final wire fixation to second copper ring	screw tensioning the wire
During heat treatment	
reaction atmosphere	argon (CEA,INFN), vacuum (UT)
ramp to T (^{0}C/h) unless otherwise prescribed	60
After heat treatment	
fixation copper rings to barrel replaced	yes (CEA,INFN), no (UT)
shunt Nb_3Sn wire transition copper-barrel	no (CEA,INFN), yes (UT)
soldering heating sequence wire to copper	ring by ring
solder	PbSn (60/40)
soldering temperature (^{0}C)	~190
cleaning after soldering copper-barrel	alcohol or ultrasonic soap
if wire movement possible, additional wire fixation (e.g. filled Stycast)	yes
curing after additional wire fixation	no (CEA,INFN), yes (UT)
gold platting of the flat contact copper rings to current leads	yes, (CEA), no (INFN), N/A (UT)
Mounting of voltage taps	
sample heating voltage tap soldering	PbSn (60/40)
voltage taps coverage from middle of barrel	3 and 5 turns (CEA) 4 turns (INFN) 3,5 and 8 turns (UT)
overall voltage tap (including copper-barrel connection)	yes
fixation voltage wires to barrel	yes (CEA), no (INFN,UT)
thermometer mounted on sample holder	yes
sample completely uncovered by any insulating materials	no (CEA), yes (INFN,UT)
Measurement procedure	
field sequence	1 T steps $B_{high} \rightarrow B_{low}$
Lorentz force	inward
I_c criterion	10 μV/m
n-determination	$5 < E < 50$ μV/m
voltage measurement	- during current ramp with 3 A/s (CEA, INFN), - at static current (UT)

III. CONCLUSION

As part of the European NED project a 3-stage iterative inter-lab comparison program between 3 European labs has been carried out with the purpose to obtain an experimental protocol ensuring reproducible critical current measurements of thick, high current Nb_3Sn conductors. Provided the sample wire is longitudinally homogeneous, this program shows that if a detailed protocol -which closely resembles the ITER protocol- on sample mounting, heat treatment and measuring procedures is respected, the inter-lab differences in I_c results of samples prepared and measured at different laboratories can be reduced to 2 % maximum, whereas the intra-lab reproducibility should be better than 1 %. Though the obtained values for the critical current lack absolute meaning due to uncertainty of the actual strain state, this relative accuracy is adequate to judge and compare the performance of industrially produced Nb_3Sn conductors.

ACKNOWLEDGEMENT

The authors gratefully acknowledge Shape Metal Innovation B.V., the Netherlands, for providing state-of-the-art PIT wire samples for this program.

REFERENCES

[1] A. Devred et al., "Status of the Next European Dipole activity of the CARE project", *IEEE Transactions on Applied Superconductivity*, vol. 15, n. 2, pp.1106-113, June 2005

[2] D.Uglietti, B. Seeber, V. Abächerli, N. Banno, R. Flükiger, "Critical current vs. Strain for LTS wires up to 21 T" *IEEE Transactions on Applied Superconductivity*, v 15, n 2, pt.3, June 2005, p 3652-5

[3] A. Godeke, "Performance boundaries in Nb_3Sn conductors", PhD.Thesis University of Twente, (2005)

[4] K. Tachikawa, K. Itoh, H. Wada, D. Gould, H. Jones, C.R. Walters, L.F. Goodrich, J.W. Ekin, S.L. Bray, "VAMAS intercomparison of critical current measurement in Nb_3Sn wires", *IEEE Transactions on Magnetics*, v 25, n 2, p 2368-74, March 1989,

[5] K. Tachikawa, "The VAMAS intercomparison of critical current measurements in superconducting Nb_3Sn wires", *Cryogenics*, v 29, n 7, p 710-15, July 1989,

[6] H. Wada, C.R. Walters, L.F. Goodrich, K. Tachikawa, "VAMAS intercomparison of critical current measurements on Nb_3Sn superconductors: a summary report", *Cryogenics*, v 34, n 11, pp 899-908, 1994

[7] Critical current measurement method for Nb_3Sn multifilamentary composite superconductors", *Cryogenics*, vol. 35, suppl. Issue, 1995

[8] L.F. Goodrich, L.T. Medina, T.C. Stauffer, "Repeatability of critical-current measurements on Nb_3Sn and NbTi wires", *IEEE Transactions on Applied Superconductivity*, vol. 7, n 2, pp. 1508-1511, June 1997

[9] K. Itoh, Y. Tanaka, K. Osamura, "Round robin test for the method of critical current measurements of Nb_3Sn composite superconductors", *Proceedings of the 16th CEC/ICMC conference*, vol. 3, prt. 3, pp.1787-1790, 1997

[10] P. Bruzzone, H.H.J. ten Kate, M. Nishi, A. Shikov, J. Minervini, M. Takayasu, "Benchmark testing of Nb_3Sn strands for the ITER model coil", *Advances in Cryogenic Engineering*, vol. 32, 1996, p. 1351

[11] L.F. Goodrich, A.N. Srivastava, "A simple and repeatable technique for measuring the critical current of Nb_3Sn wires," *Proceedings of the 7th International Workshop on Critical Currents in Superconductors*, 1994, p 609-12

[12] E. Barzi et al., "Instabilities in transport current measurements of Nb_3Sn strands", *IEEE Transactions on Applied Superconductivity*, vol. 15, n. 2, pp.3364-3367, June 2005