Magnetization Measurements of High- J_c Nb₃Sn Strands

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Index Terms—Filament Size, Flux Jumps, Magnetization, Nb₃Sn, Scaling Parameters

I. INTRODUCTION

IN THE framework of the LHC luminosity upgrade [1]-[2], the Nb₃Sn superconductor has been chosen as the conductor for the new generation of high field (>10 T) accelerator magnets. At present Nb₃Sn wires are produced in large quantities in the world because of their massive use in the construction of the ITER Tokamak [3]. Differently from the ITER Nb₃Sn wires, the conductor envisaged for future accelerator magnets has a critical current density J_c that is pushed to the limit of the present technology. The accelerator-type Nb₃Sn wires have a critical current density (at 4.2 K and 12 T) that ranges from 2500 to 3000 A/mm² depending on the wire configuration and the heat treatment used for the strand reaction. The high J_c , which is more than two times larger than the one of ITER type wires, is obtained at the expense of a large effective filament size D_{eff} of the superconductor. In these wires each sub-element acts as a single filament: in Powder In Tube (PIT) wires the sub-element is constituted of a Nb tube filled with Nb-Sn powder, and during the reaction the tin diffuses transforming the Nb tube into a Nb₃Sn one; in Internal Tin (IT) Restacked Rod Process (RRP[®]) wires the tiny Nb filaments (few microns in diameter) contained into the sub-element merge together during the reaction. The high J_c

and the large D_{eff} cause a huge magnetization within the conductor that might compromise the magnet field quality [4]-[8]. Indeed such a large magnetization not only implies a specific design of the magnets to compensate for the field contribution of the persistent currents, but also generates flux-jumps [4]-[8]. These magnetic instabilities produce an erratic behavior of the conductor magnetization, which can suddenly change in a millisecond time scale. If the flux jumps are sufficiently big, as it was recently observed during the test of the HQ magnet [9] at CERN, they can significantly affect the field quality in the magnet bore and because of the erratic behavior this effect cannot be easily compensated.

In order to characterize the magnetic behavior of high J_c wires, a campaign of magnetization measurements was launched at CERN. PIT strands by Bruker-EAS and RRP[®] strands by Oxford Superconducting Technology (OST) were measured between 0 T and 10.5 T at different temperatures (ranging from 1.9 K to 14.5 K). The samples, based on strands with different sub-elements dimensions (35 to 80 μ m), were measured with a Vibrating Sample Magnetometer (VSM). The experimental data were analyzed in order to: 1) calculate the effective filament size and the optimal parameters for the pinning force scaling law; 2) define the field-temperature region where there are flux jumps. In this paper the experimental results and the analysis are reported and discussed.

II. MAGNETIZATION MEASUREMENTS

The magnetic moment of 5-mm-long straight samples was measured by sweeping the applied field (perpendicular to the strand axis) in a Vibrating Sample Magnetometer at CERN. From the magnetic moment, the sample magnetization (magnetic moment per unit volume of the strand) was calculated by using the measured weight of the non reacted sample and the measured density of the considered strand. The measurements were carried out in a helium gas flow and the temperature was measured and regulated by three thermometers, one of which was mounted directly on the sample holder. The samples were high J_c Nb₃Sn wires of the type which is presently used in the development of next generation accelerator magnets. Two types of conductor were characterized: the PIT and the RRP[®] strands. For the RRP[®] conductor, strands with different sub-element dimensions and doping materials (Tantalum and Titanium) were measured. Ti-doping was recently introduced in the RRP[®] wires because it seems to significantly improve their behavior under axial strain. Table I summarizes the main strands properties. The identification code of each sample corresponds to the original billet number of the considered wire. Besides the main strand layout characteristics (strand diameter, type of strand and of the doping material, number of the sub-elements, copper

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TABLE I
STRAND PROPERTIES

ID (Billet #)	Diameter ^I [mm]	Type	Doping	Number of Sub-elements	Cu to non-Cu	Sub-elm. Size ^{II} [μm]	Heat Treatment	RRR	J_c (4.3 K-12 T) [A/mm ²]	B_{c2} (4.3 K) [T]	D_{eff} [μm]
7419	0.803	RRP	Ta	54/61	0.92	79	695 °C / 17 h ^{III a}	300	2995	25.09	83
13126	0.704			108/127	1.25	45	665 °C / 50 h ^{III b}	45	2838	23.15	54
802	0.701			114	1.21	44	640 °C / 120 h ^{III c}	150	2616	23.69	45
904	1.004	PIT		192	1.24	48	650 °C / 90 h ^{III c}	235	2385	22.89	44
10044	0.704	RRP	Ti	198/217	0.84	37	695 °C / 8 h ^{III a}	69	2449	23.88	41
11500	0.704			192/217	1.18	34	695 °C / 8 h ^{III a}	23	2203	23.03	42
11976	0.803			108/127	1.02	54	665 °C / 50 h ^{III b}	90	2658	24.56	59
14393	1.004			132/169	1.25	58	650 °C / 50 h ^{III b}	200 ^{IV}	2773	23.93	64

^I Measured before heat treatment

^{II} Estimated from the strand diameter, the number of sub-elements and the copper to non-copper (by assuming round sub-elements)

^{III} Intermediate temperature plateaus: a) 210 °C / 72 h, 400 °C / 72 h; b) 210 °C / 48 h, 400 °C / 72 h; c) 620 °C / 120 h

^{IV} Measured by OST

to non-copper ratio, estimated sub-element size), the table shows the heat treatment cycle and the electrical properties of the reacted wire (the Residual Resistivity Ratio, RRR , and the critical current density). If not explicitly reported, the measurements were carried out at CERN. The critical current density was measured using transport current measurements of wires mounted on a typical Ti-6Al-4V ITER barrel. Finally the table reports the strand upper critical field B_{c2} (at 4.3 K) and its effective filament size; these values were estimated by using the magnetization measurements and the procedure described later in the paper.

Each sample was measured at different temperatures: 1.9 K, 4.3 K, 6.5 K, 8.5 K, 10.5 K, 12.5 K and 14.5 K. At each temperature, the test consisted of two parts: 1) initially the magnetic moment was measured by continually sweeping the applied field at 0.2 T/min within ± 4 T and taking data every second, then 2) the measurement was carried out at fixed applied fields from 0 T to 10.5 T (or the maximum B_{c2} for a certain temperature) with steps of 0.25 T while increasing and decreasing the field (0 T $\rightarrow$ 10.5 T $\rightarrow$ 0 T). In this second part of the test, the measurement started at 0 T with the sample negatively magnetized from the previous continuous measurement. The applied field was then increased to 10.5 T by alternating 0.5 T/min field ramps with 10-s-long field plateau. During each plateau, 10 magnetization measurements were performed. The same measurements were done while decreasing the field from 10.5 T to 0 T. Fig. 1 shows the results obtained using this test procedure for the sample 10044 measured at 10.5 K.

A. Flux jumps analysis

The first part of the test (continually sweeping the applied field) was carried out in order to investigate the presence and the amplitude of flux jumps as a function of the temperature for the different samples.

Flux jumps were observed at 1.9 K in all samples, while at temperatures equal or higher than 6.5 K no jumps occurred. At 4.3 K flux jumps appeared in all Ta-doped wires whereas for the Ti-doped strands flux jumps occurred only in the two samples with lower RRR (10044 and 11500). Fig. 2 shows the

flux jumps in the two Ta-doped samples with the largest and the smallest effective filament size. It is important to notice that at 1.9 K: 1) the flux jumps start at higher fields and are significantly smaller than those at 4.3 K; 2) there is a field region around 0 T (about ± 3 T) where flux jumps occur continuously before reaching the full penetration state of the superconducting sub-elements. In this field region, because of the continuous demagnetization caused by the flux jumps, the magnetization at 1.9 K is generally lower than at 4.3 K. This phenomenon is particularly evident for the Ti-doped samples that did not experience flux jumps at 4.3 K, see Fig. 3.

It is also important to notice that the largest flux jump at 1.9 K is generally happening at the extremities (highest field) of the unstable region where the sample is fully penetrated by the field. By reducing the field at lower and lower values, the flux jump amplitude decreases (as the field penetration: the magnetization value is practically constant while J_c is significantly increasing) and in some cases the jumps are hardly visible (Fig. 2 b and Fig. 3).

Fig. 1. Magnetization as a function of applied field at 10.5 K for the 10044 sample. The line represents the measurement carried out while ramping the field at 0.2 T/min whereas the marks the data taken with a fixed field.

Fig. 2. Magnetization as a function of applied field for the 7419 sample (D_{eff} 83 μ m, plot on the left) and for the 10044 sample (D_{eff} 41 μ m, plot on the right).

Fig. 3. Magnetization as a function of applied field for the 11976 Ti-Doped sample (no flux jumps at 4.3 K).

Table II summarizes the characteristics of the flux jumps observed in all the samples. The first column shows the amplitude of the hysteresis loop ΔM at 3 T and 1.9 K for each sample; this value was chosen as parameter to quantify the magnetization of the sample in the field region where flux jumps do not take place. The next columns present the

TABLE II
FLUX JUMPS CHARACTERISTICS

ID	ΔM	Max Field Flux Jump		Flux Jump Amplitude	
	1.9 K, 3 T [mT]	1.9 K [T]	4.3 K [T]	1.9 K [mT]	4.3 K [mT]
7419	468	2.7	0.9	113	397
13126	309	2.7	0.61	26	129
802	230	2.63	0.81	8	52
904	173	2.18	0.28	14	91
10044	206	1.53	0.46	15	192
11500	196	1.81	0.26	4	30
11976	319	2.56	-	23	0
14393	323	1.74	-	54	0

maximum field at which flux jumps were recorded and their maximum amplitude. From this table and from Figs. 4 and 5, one can notice that the maximum flux jump amplitude at 1.9 K is correlated to the magnetization of the sample: the larger the magnetization the larger the flux jump amplitude. This relationship is particularly clear when plotting the magnetization of each sample just before it experiences the largest flux jump and the amplitude of this flux jump (Fig. 5).

B. Calculation of fitting parameters and of the D_{eff}

The magnetization measurements in the stable field region (and with the sample fully magnetized) were then analyzed to derive the pinning force (F_p) curve using the following relationship:

$$\Delta M \cdot B = \frac{4}{3\pi} \frac{1}{1 + \lambda} D_{eff} F_p(T, b) = C(T) b^p (1 - b)^q \quad (1)$$

where ΔM is the amplitude of the hysteresis loop at a certain applied field B , λ is the Cu to non-Cu ratio, F_p is equal to J_c times B , b is the reduced field equal to $B/B_{c2}(T)$. The magnetization data ($\Delta M \cdot B$) were used to calculate the optimal

Fig. 4. Magnetization as a function of applied field at 1.9 K. In the legend the samples are ordered by increasing magnetization.

Fig. 5. Relationship between the maximum flux jump amplitude of each sample at 1.9 K and its magnetization (at 1.9 K); the triangles and the squares respectively represent: 1) the sample magnetization just before the largest flux jump occurred; 2) the amplitude of the sample hysteresis loop ΔM at 3 T.

$C(T)$, p , q and B_{c2} parameters of the pinning force scaling law [10]-[11]. Initially, the p , q parameters were calculated together with $B_{c2}(10.5 \text{ K})$ and $C(10.5 \text{ K})$ by fitting the 10.5 K data; while for the remaining datasets only $B_{c2}(T)$ and $C(T)$ were used as parameters to fit the data (p and q were kept constant and equal to the values found from the 10.5 K dataset). Fig. 6 shows the pinning force curve and the fit obtained using the procedure described above. The 10.5 K data set was selected for calculating the p and q parameters because it was the one at the lower temperature covering the all range of reduced field from 0 to 0.8. In general the data with a reduced field above 0.8 were excluded because they might be strongly influenced by a small part of the superconductor with a different B_{c2} that has a negligible effect at lower fields. This procedure worked very well for the RRP[®] wire while it was not appropriate for the PIT strands that showed a shape of the pinning force curve depending on the temperature. Because of this anomaly, for the PIT conductor the p and q parameters were calculated using the 4.3 K data set instead of the 10.5 K one. That allowed having a good fit in

Fig. 6. Normalized pinning force curve derived from the magnetization for the 10044 sample.

the most interesting temperature region (1.9 - 4.3 K) at the expenses of a not optimal fit at higher temperatures. At these larger temperatures, since for the PIT it was not possible to properly fit all the data without changing the p and q parameters, the only fitted data were those with normalized fields larger than 0.2.

Once calculated, the $B_{c2}(T)$ and $C(T)$ values were fitted (see Fig. 7) using the following laws [10]-[11]:

$$B_{c2}(T) = B_{c20} (1 - t^{1.52}) \quad (2)$$

$$C(T) = C_0 B_{c2}(T) (1 - t^2) \quad (3)$$

where B_{c20} is the upper critical field of the Nb_3Sn at 0 K, t is the reduced temperature equal to T/T_{c0} , and T_{c0} is the critical temperature at 0 T. All the parameters calculated in this study refer to the case where the Nb_3Sn strain is not zero. Indeed, in our magnetization samples the Nb_3Sn experiences the strain caused by the differential thermal contraction of the different materials constituting the strand. In the $B_{c2}(T)$ fit, B_{c20} and T_{c0} act as parameters while for the $C(T)$ fit only C_0 is a parameter (for the T_{c0} the same value calculated from (2) is used). Table III summarizes all the calculated parameters.

Finally the strand effective filament size D_{eff} was estimated by fitting the critical current data at 4.3 K (from the transport current measurements) using the following equation (derived

TABLE III
PINNING FORCE FITTING PARAMETERS

ID	C_0 [kA/m]	B_{c20} [T]	T_{c0} K	p	q
7419	140.8	29.11	15.57	0.38	1.85
13126	87.2	27.32	15.57	0.4	1.85
802	70.6	27.4	16.2	0.49	1.99
904	65.9	26.94	16.32	0.49	1.97
10044	68.4	27.49	15.4	0.43	1.87
11500	54.1	26.77	15.54	0.45	1.82
11976	91.2	28.71	15.53	0.43	1.87
14393	96.3	28.19	15.63	0.44	1.85

Fig. 7. Fit of the $B_{c2}(T)$ and $C(T)$ data derived from the magnetization measurements for the 10044 sample.

from (1) :

$$J_c(4.3 \text{ K}) = \frac{1}{\frac{4}{3\pi} \frac{1}{1+\lambda} D_{eff}} \frac{C(4.3 \text{ K})}{B} b^p (1-b)^q \quad (4)$$

where D_{eff} was the only fitting parameter; for $C(4.3 \text{ K})$, $B_{c2}(4.3 \text{ K})$, p and q , the values derived from the magnetization measurements were used. Fig. 8 shows an example of this fitting procedure.

III. DISCUSSION

The peculiar instability behavior of high J_c Nb₃Sn strands at 1.9 K observed in the magnetization measurements, was hypothesized in previous publications [12]-[14] to explain the limited effect (with respect to the 4.3 K case) of the magnetization in the premature quench currents at low field and 1.9 K. It was noticed that while at 4.3 K the quench current in the low field region (0-3 T) was significantly reduced when the sample was magnetized (V-H measurements), that was not the case at 1.9 K. This behavior was explained by assuming that the Nb₃Sn was so unstable at 1.9 K that the magnetization (and the energy associated to it) in the low field region was even lower than the one at 4.3 K. The magnetization measurements performed confirm this interpretation and the fact that magnetization instability is not a major issue for premature quenches of high field accelerator magnets at 1.9 K. This behavior it is also very interesting for the magnet field quality. The reduced flux jump amplitude at 1.9 K has a much smaller impact on the field quality with respect to the 4.3 K case. Indeed a Nb₃Sn HQ magnet developed by the LARP collaboration and recently tested at CERN [9] showed that while the field quality was largely jeopardized by the flux-jumps at 4.3 K, the magnetic measurements could hardly detect the jumps at 1.9 K. The HQ magnet used 0.8 mm 54/61 and 108/127 RRP[®] strands with relatively large D_{eff} ($\sim 80 \mu\text{m}$ and $60 \mu\text{m}$ respectively). Furthermore the reduced magnetization at 1.9 K (with respect

to 4.3 K) might explain the unexpected behavior of the sextupole field observed in the 11 T Nb₃Sn demonstrator dipole for LHC upgrades that was recently tested at FNAL [15]. In the current interval from 1 to 2 kA, by reducing the temperature from 4.5 K to 1.9 K, the hysteresis loop width decreased 3 % while simulations predicted approximately 7% increase because of the higher J_c [15].

At a certain temperature, the maximum amplitude of the flux-jumps is proportional to the magnetization of the conductor, hence strands with smaller sub-elements have smaller flux jumps (for a certain critical current density), which means: 1) a reduced possibility of premature magnet quenches caused by low field instabilities at 4.3 K; 2) reduced field quality issues related to flux-jumps.

In the analysis of the pinning force curve derived from the magnetization measurements, it was found that while for the RRP[®] strand one set of p and q parameters was sufficient for accurately describe the conductor behavior, the PIT strands showed a shape of the pinning force curve depending on the temperature.

The calculation of the effective filament size showed that the PIT strand has a D_{eff} very close to the geometrical diameter (see Table I). On the other hand the RRP[®] D_{eff} is about 10 % larger than the geometrical one. This difference is most likely due to the different geometry of the sub-elements: circular for the PIT and hexagonal for the RRP[®].

IV. CONCLUSIONS

Eight different high J_c Nb₃Sn wires, which are relevant for the CERN high field magnet program, were measured between 0 T and 10.5 T at different temperatures (ranging from 1.9 K to 14.5 K) in a Vibrating Sample Magnetometer. The experimental data were analyzed in order to: 1) define the field-temperature region where there are flux jumps; 2) calculate the effective filament size and the optimal parameters for the pinning force scaling law that can be used by magnet designers to determine the critical surface of their conductor. The measurements showed that at 4.3 K the flux jumps have larger amplitude and they are fewer than at 1.9 K. At 1.9 K there is a field region around 0 T where the magnetization is even lower than at 4.3 K because the sample is so unstable that flux jumps occur before the sub-elements reach the full penetration state. This mechanism is consistent with the transport current measurements, and indicates that the magnetization instability is not a major issue for premature quenches of high field Nb₃Sn accelerator magnets at 1.9 K. Furthermore, this conductor behavior is in agreement with recent magnetic measurements carried out at CERN on a LARP Nb₃Sn HQ magnet [9] and at FNAL on a 11 T Nb₃Sn demonstrator dipole for LHC upgrades [15]

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Fig. 8. Fitting of the critical current data at 4.3 K for the 10044 sample by using (4) and the parameters derived from the magnetization measurements. The effective filament size of the strand is the only fitting parameter.

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