TECHNICAL NOTE

Development of a short sample test facility for evaluating superconducting wires

M. R. Singh, D. G. Kulkarni, V. C. Sahni, G. Ravikumar and K. L. Patel*

In this paper we describe a short sample test facility we have set up at Bhabha Atomic Research Centre (BARC). This facility has been used to measure critical currents of NbTi/Cu composite superconducting wires by recording V versus I data at 4.2 K. It offers sample current as large as 1500 A and a transverse magnetic field up to 7.4 T. A power law, $V \sim I^{n(H)}$ is fitted to the resistive transition region to estimate the exponent n, which is a measure of the uniformity of superconducting filaments in composite wires. It is observed that inadequate thermal stabilization of sample wire results in thermal runaway, which limits the V–I data to $\sim 2\,\mu V$. This in turn affects the reliability of estimated filament uniformity. To mitigate this problem, we have used a sample holder made of OFHC-Cu which enhances thermal stabilization of the sample. With this sample holder, the results of measurements carried out on wires developed by the Atomic Fuel Division, BARC show a high filament uniformity (n ~ 58).

THE property of type-II hard superconductors to carry large currents in high magnetic fields with practically zero resistance provides a practical way to make extremely stable as well as powerful magnets, which not only saves energy, but also delivers high magnetic fields, far beyond those obtainable by the ordinary electromagnets because of the saturation of iron core. Indeed, most of the present-day magnets, whether for high-energy particle accelerators, or for magnetic confinement fusion devices like Tokomak, or for other applications like high-field NMR or MRI are built using one or the other type-II hard superconductors. High gradient magnetic ore separation to either segregate feebly paramagnetic particles or to improve clay quality (for pottery industries) or for mineral (ore) beneficiation, and the superconducting energy storage systems (SMES) are some of the other areas of large-scale applications that have emerged.

The most widely used material for generating magnetic fields up to ~ 8 T at 4.2 K is Nb–Ti alloy system, while Nb_3Sn, V_3Ga, etc. are deployed for higher fields. Nb–Ti is a type-II superconductor. While processing this material into actual conductor, one deliberately introduces large number of defects to pin the vortices, so that there is a consequent increase in the irreversible magnetization and hence an enhancement in the current-carrying capacity, which is proportional to this irreversibility. Such materials with high magnetic irreversibility are referred to as hard type-II superconductors. The critical temperature (T_c) is ~ 9.3 K and upper critical field (H_{c2}) at 4.2 K can be up to 11 T depending upon the composition of Nb–Ti alloy[1]. Due to its high ductility and strength, it has been the workhorse for numerous applications. The Ti content ranges from 46 to 50% by weight (62 to 66 atomic%)[2].

Bhabha Atomic Research Centre (BARC) has had a long-standing programme to develop Nb–Ti-based superconducting wires. This programme started in a modest way for building small laboratory magnets. But its scope has grown in recent times, when applications such as superconducting cyclotron were identified, involving currents ~1 kA or more.

For actual use in a magnet, the superconductor material is generally processed in the form of multi-filamentary strands embedded in a Cu matrix. The reason for choosing this type of arrangement for the filaments is as follows. Since all high-field magnet applications utilize the superconductor in the 'mixed state', one must prevent the movement of vortices, which can otherwise lead to a quench of superconducting state essentially because of flux jumps. Flux jumps are linked to the fact that all superconductors are bad conductors of heat and very poor conductors of electricity in their normal state. This means that if flux movement starts, it takes over a superconductor before heat is dissipated away to the cryogen bath and thermal runaway occurs. Dictated by these considerations, superconductors are thermally stabilized by making them in the form of fine filaments embedded in oxygen-free high conductivity (OFHC) copper. In the event of the initiation of a flux jump, the heat generated

The authors are in the Technical Physics and Prototype Engineering Division, Bhabha Atomic Research Centre, Mumbai 400 085, India.
*For correspondence. (e-mail: mrsingh@apsara.barc.ernet.in)

is carried away through copper. Thus copper provides dynamical thermal stability as also cryostatic stability besides acting as a shunt to share current. The amount of Cu required generally depends on the nature of the application. Usually, the Cu volume ranges from 55 to 70% (of wire volume)[2]. The actual Nb–Ti/Cu composite conductor is fabricated generally through extrusion process which helps in the smooth reduction of filament diameter. It should be mentioned that to minimize flux-jumps, the diameter of the filament should be kept below a certain limit. This limit depends on the material properties like specific heat, critical current density, thermal conductivity and the magnetic field. For Nb–Ti a 60 μm diameter filament will be thermally stable at 5 T magnetic field[1].

The magnetization in the superconducting filaments can set up persistent currents within the filaments. In the case of superconducting magnets used in accelerators, involving multiple coils in complex geometries, this (persistent current) effect can give rise to unwanted magnetic multi-pole contributions. The magnitude of polluting multi-poles becomes relatively significant at lower magnetic fields[3]. Since the magnetization is proportional to critical current density (J_c) and diameter of filament, the need for purity of magnetic field, particularly in high-energy accelerators, further restricts the upper limit on diameter of these filaments. For most of the magnets in use, the number of filaments in a wire ranges from 50 to 5000, and filament diameter from 5 to 50 μm (ref. 2). Finally, to minimize eddy currents and the ac coupling between filaments via Cu-matrix during the magnetic field sweep, the multi-filamentary superconducting wire is generally twisted with a pitch varying from 5 to 30 times the wire diameter[4] depending on the field sweep rate.

The manufacturing process for the superconducting wire containing a large number of filaments entails many iterative steps of billet forming and extrusion, thereby reducing the filament diameter and increasing the number of filaments in the wire[2]. However, this process often introduces non-uniformity (sausaging) in the diameter of individual filaments along the direction of drawing. This results in the broadening of the resistive transition as seen in voltage–current (V–I) characteristics. The uniformity of filament-diameters along the length of the wire, which is one of the key characteristics of its high quality, is estimated by fitting an empirical power law $V \sim I^{n(H)}$ to the resistive transition region[5]. Generally, higher the n value the better is the wire and smaller is the sausaging effect.

The finished wire, which may be in kilometres in length, has to be characterized for its critical current, I_c and n value. To characterize long pieces of superconducting wires is a difficult task. Generally one takes recourse to evaluating some small segments (typically ~ 1 m in length) of the wire. At the development stage of the wire, these segments would have to be taken at random. Subsequently these could be taken from the ends. Indeed, I_c and n are established by short sample electrical transport measuring technique, where V–I characteristics are recorded for a small length of sample cut from the finished wire. The I_c, which is a function of transverse magnetic field, is customarily defined as transport current causing a voltage drop of 0.1 μV/cm along the wire at 4.2 K (ref. 4) and at a specified magnetic field. For good quality wires, the estimated value of the exponent, called quality index, n should be ≥ 30 (ref. 5). The parameters I_c and n constitute the most important inputs to the magnet designers besides providing necessary feedback for optimization of material processing itself.

In the following we describe development of a short sample test (SST) facility, which has been used for the characterization of multi-filamentary high-current superconducting Nb–Ti/Cu composite wire, developed at BARC, motivated in part by the upcoming programme to build a superconducting cyclotron at VECC, Kolkata. The reliable estimation of n warrants recording V–I curve up to a voltage drop, well beyond the criterion of 0.1 μV/cm. But due to thermal runaway, the recording of V–I characteristic for a high-current superconducting wire is often limited to just about ~ 2 μV/cm. In our initial set-up, when thermal runaway was acute, measurements could only be made up to voltage level < 1.0 μV/cm. However, we have subsequently suitably modified our sample holder to provide enough thermal stabilization, which has facilitated the recording of V–I curve for reliable estimation of n. Details of our set-up are described below.

The short sample test facility (Figure 1) has been developed to characterize superconducting wire up to a transport current of 1500 A at liquid helium bath tempe-

Figure 1. Photograph of the short sample test set-up. The high current power supply is seen on the right hand side, the electronic units are in the rack on the left hand side, while the cryostat is housed inside the box at the centre.

rature. The measurement of transport critical current is performed in the presence of a transverse magnetic field. For this we have designed and fabricated a superconducting solenoid in our laboratory, using commercially available Nb–Ti/Cu multi-filamentary wire. The bore diameter of the solenoid is 55 mm and it provides field of up to a maximum of 7.4 T at 4.2 K. The field-homogeneity achieved is 10^{-3} in 2-cm diameter spherical volume at the centre of the solenoid. For I–V measurements, a test piece of wire is wound helically in a groove carved on a sample holder whose diameter is at least 20 times the wire diameter[4]. The helically-wound sample uses a reasonable length of wire (~ 50 cm). Compared to other sample geometries[6], it is more suitable for multiple voltage taps. There is negligible deviation from the condition of transverse magnetic field, as the plane of the helically-wound sample is more than 87° with respect to the axis of the solenoid.

Some of the initial measurements were done using a stainless steel (SS) mandrel[5,6] for sample mounting. The ASTM standards and International Committee for Future Accelerators (ICFA)[4], however, specified G-10 material for sample holder. We tried that option also. However, we have observed that due to thermal instabilities, the use of G-10 limits I_c to only a few hundred amperes, whereas in our test facility we were required to carry out measurement of $I_c \geq 1400$ A.

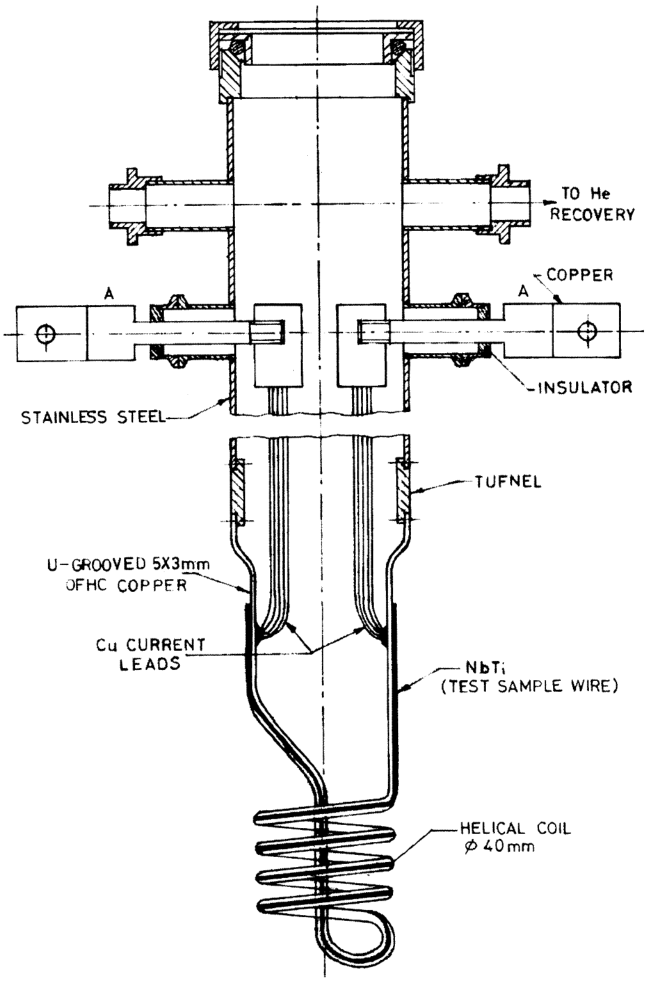

Figure 2. Schematic diagram of sample insert. Positions of voltage probes are not shown.

Our decision to build a facility using a solenoid with a bore of ~ 55 mm was dictated by the following considerations. The upcoming superconducting cyclotron at VECC, needs a conductor capable of carrying 1030 A at 4.2 K and 5.5 T, and is closely following the design of a similar machine, in which to augment the cryogenic stability of the final conductor, the effective Cu : SC ratio is enhanced by soldering the superconductor wire (of diameter ~ 1.4 mm) onto a U-grooved Cu channel made of OFHC copper with an outer cross-section ~ 5 mm × 3 mm. In fact, for the finished conductor, Cu : SC ratio is 20 : 1. Therefore, we decided to use the same Cu channel for making our sample holder. This channel has been wound in the form of a helical coil of diameter 40 mm. The sample to be tested is soldered into the U-shaped groove of copper spiral, which provides increased thermal stability. Given the size (~ 40 mm) of the sample coil diameter, the solenoid bore, in which the sample coil is to be inserted, is chosen to be 55 mm so that the sample insert shown in Figure 2, can get easily accommodated.

Figure 1 shows a photograph of the facility which consists of a high current power supply to pass currents up to 1500 A into the short sample test piece, the cryogenic assembly carrying the test sample, a 100 A constant current source for superconducting magnet and an electronic measuring unit. The sample current source is a constant current power supply made by M/S Hindustan Rectifiers Ltd, Mumbai. It has a provision to limit maximum output voltage to either 3 or 10 V at maximum current. The output current from this supply can be ramped up/down remotely using a PC and DAC card in any convenient step. Copper bus-bar pairs, which is used

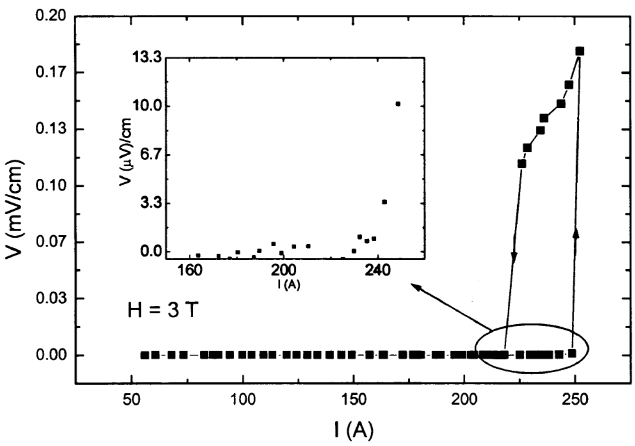

Figure 3. V–I curve for Vacuumschmelz wire showing hysteresis due to thermal runaway during the increasing current leg, when voltage across the sample jumps to mV range. The wire remains in the normal state, unless current is reduced substantially.

for transporting the current near the cryostat, are fixed along the wall as seen in Figure 1. A water-cooled standard resistance is put in series between one copper bus-bar and the sample insert to measure sample current. Thereafter, flexible heavy current links (Ag-coated braided Cu wire) are used to couple current through the points AA (Figure 2), which are close to the upper end of cryogenic sample insert. Transport of such large (~ 1.5 kA) current to the sample immersed in liquid helium bath poses severe problems of heat load and consequent boil-off of cryogen. The conductor for this purpose consists of a bunch of enamelled Cu-strands of suitable gauge connected in parallel at the ends. We have made a pair of such conductors (marked in Figure 2 as current leads) which runs through the sample insert, whereby the enthalpy of evaporating He is utilized to keep these current conductors cool. This is essential to minimize boil-off of cryogen as well as to extend the measurement time. These leads are soldered onto the sample. Voltage taps are soldered to the sample at points appropriately away from the current contacts so that the effect of 'current transfer length' is excluded from the measurements. A nano-voltmeter is used to record voltage drop across the sample. A separate micro-voltmeter is connected across a standard resistance for measuring the sample current. These instruments are interfaced to a PC using IEEE-488 bus. This PC also controls a separate constant current power supply for setting the magnetic field in the superconducting solenoid.

For standardizing the SST facility, we recorded *V–I* data of a superconducting wire procured from M/S Vacuumschmelz, Germany, for which I_c–H property was already known as part of the specifications. The Vacuumschmelz wire diameter is 0.55 mm and it contains 45 filaments with overall Cu : SC ratio of 1.8 : 1. The sample holder used for this sample was a SS mandrel. The *V–I*

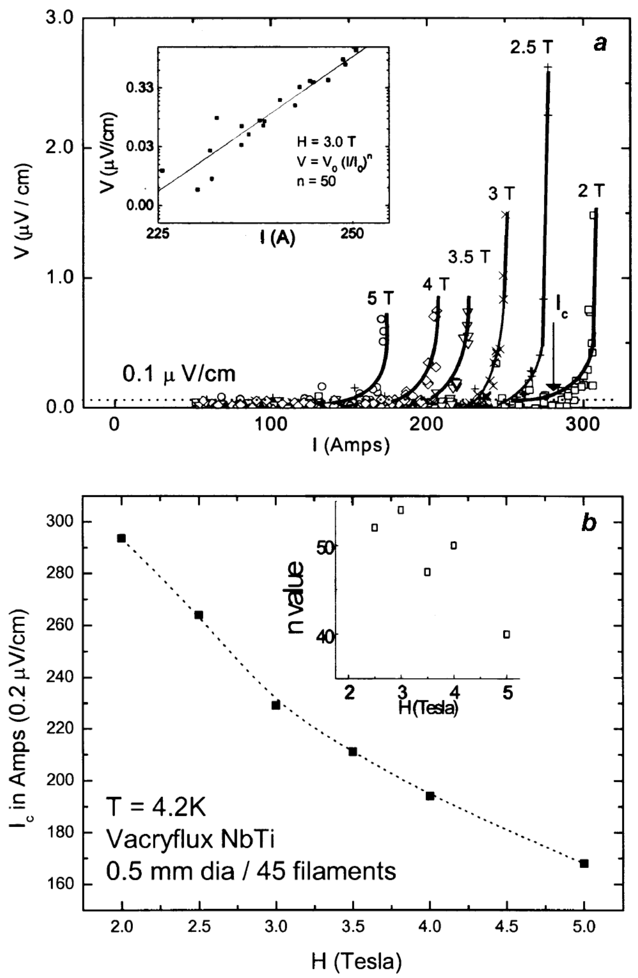

Figure 4. ***a***, *V–I* characteristics of Vacuumschmelz wire (0.5 mm dia.) as a function of magnetic field at 4.2 K. (*Inset*) Fitting of power law using log–log plot, in the resistive transition region of *V–I* data for 3 T (estimated $n = 50$); ***b***, Critical current (I_c) for Vacuumschmelz wire as a function of transverse magnetic field. (*Inset*) Value of n at different fields for the same wire.

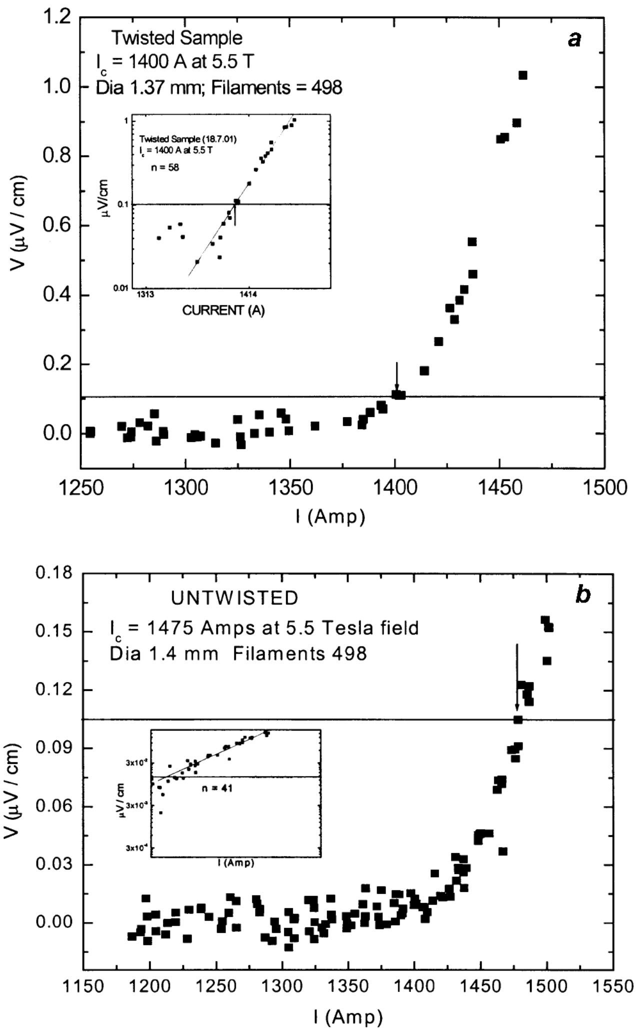

Figure 5. *V–I* characteristics of wire developed at BARC at 4.2 K and 5.5 T. ***a***, Twisted sample: $I_c = 1400$ A; ***b***, Untwisted sample: $I_c = 1475$ A. *Insets* show the log–log plots of the resistive transition regions to estimate the respective n values.

data were recorded for both current increasing and decreasing cycles. The hysteresis (Figure 3), which is seen when the sample current is being decreased, is due to thermal runaway at ~ 2–3 μV/cm during the ramping-up of sample current. In Figure 4 *a* we have presented comprehensive data taken on this wire and at 3 T, $n = 50$ has been estimated from the slope of *V–I* data on a log–log plot (inset of Figure 4 *a*). Figure 4 *b* and its inset show the variation of I_c and n with magnetic field respectively.

After qualifying this facility on the sample of Vacuumschmelz, it has also been used to characterize various superconducting wires being developed at BARC. However, for wires having $I_c > 600$ A, due to thermal runaway at ≤ 1 μV/cm, n could not be estimated reliably using SS mandrel. Therefore, we switched to a sample holder made out of OFHC-Cu with a U-channel. The sample holder has been subsequently modified to have two Cu U-channels wound parallel in helical shape, to facilitate mounting two samples simultaneously. Though the samples are connected in series, the direction of current flow in one sample is opposite to that in the second sample. Thus, the effect of self-field due to large transport current in the sample is minimized in this arrangement. The capability of the set-up can be gauged by the results on two samples at 5.5 T presented in Figure 5 *a* and 5 *b*. Both samples are from wires of diameter 1.37 mm, consisting of 492 filaments and are made of Nb–Ti/Cu, but given different types of heat treatment. The filament diameter is ~ 40 μm and overall Cu : SC ratio is 1.2 : 1. While the twist pitch is 12.4 mm for one sample, the other one is untwisted. For the twisted sample (Figure 5 *a*), the I_c value was found to be 1400 A at 4.2 K and 5.5 T, while for the untwisted sample (Figure 5 *b*) it was 1475 A. The value of n is obtained from the slope of log V versus log *I* plot and the estimated value at 5.5 T is 58 and ~ 41 respectively for the two samples. The n value for the untwisted wire is approximate, as its accurate estimation will require sample current larger than 1500 A. It is clear from these results that our set-up can reasonably well characterize superconductor wires for these I_c values. The estimated n values of the wire developed at BARC compare well with those of imported wires.

To conclude, we have presented the salient features of superconducting wires used for high-current applications, and reported on the development of an automated SST facility for measuring electrical transport characteristics of such wires up to ~ 1.5 kA at liquid helium temperature and subjected to a maximum transverse magnetic field of 7.4 T. This is a unique facility in India, and should stimulate development of superconducting wires in the country.

1. Wilson, M. N., in *Superconducting Magnets*, Clarendon Press, Oxford, 1983, p. 2.
2. Lee, P. J., *Abridged Metallurgy of Ductile Alloy Superconductors*, Wiley Encyclopedia of Electrical and Electronics Engineering, Wiley, New York, 1999, vol. 21, pp 75–87.
3. Brianti, G., in Proceedings of the Workshop on Superconducting Magnets and Cryogenics (International Committee for Future Accelerators), Brookhaven National Laboratory, Brookhaven, BNL52006, 1986, pp. 3–6.
4. Barzi, E., *Short Sample Measuring Techniques*, Technical Division TD-98-014, Fermilab, 11 February 1998.
5. Warnes, W. H. and Larbalestier, D. C., *Cryogenics*, 1986, **26**, 643–653.
6. Goodrich, L. F. and Fickett, F. R., *ibid*, 1982, **22,** 225–241.

ACKNOWLEDGEMENTS. We would like to acknowledge the invaluable support provided by many past and present colleagues from our Division and Atomic Fuel Division for this activity, particularly A. N. Garud, H. N. Karandhikar, M. K. Malik, P. K. Mishra, T. V. C. Rao and V. K. Handu.

Received 23 November 2001; revised accepted 23 April 2002