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Transport properties of single MgB_2 filaments extracted from multifilamentary conductors

E.A.Young, Q. Zhang, Y. Yang

***Abstract*—Using an electrochemical bath copper and nickel metals and alloys are removed in multifilamentary MgB_2 conductors to leave single MgB_2 filaments surrounded by a Nb sheath. Starting from a 0.55 mm 36 filament diameter conductor, the etched filaments are 60 microns in diameter. Seven wires were characterized by transport critical current measurement, and one investigated further by resistivity when progressively etched. Their radial location in the original multifilamentary wire structure is then used to explore if the critical current is sensitive to the deformation process. The average critical current of the outer layer of filaments is 35% higher than the inner layers. The average filament critical current was 32 A which is within 10 % of the 36 filament wire critical current per filament when adjusted for self field.**

***Index Terms*— Critical current density, Electrochemical processes, multifilamentary superconductors, high temperature superconductors.**

I. Introduction

Multifilamentary high temperature superconductors are a composite of ceramic superconductor and at least one metal. The ceramic superconductor is polycrystalline and in the case of the superconductor Magnesium Diboride, MgB_2 contains cracks, pores and non-superconducting phases. The sheath metals are chosen for a variety of reasons including mechanical strength, barrier layer, thermal stabilization but their properties can differ from the expected due to reactions with their composite neighbor or the surface properties. To characterize these properties as a composite is the ultimate test but to understand how to improve them it can be necessary to separate out the constituent part properties.

The MgB_2 manufacturing process is either powder in tube, PIT, or continuous tube forming and filling (CTFF). In either case the wires are drawn down to their final size, and in this process the pre-reaction density of each filament is determined. Within the conductor a different location of a filament will result in a different mechanical stress, so filaments towards the centre have undergone a different mechanical deformation process to those near the outside.

The relationship between the figure of merit for practical superconductors, the critical current density, *Jc* and the microscopic materials properties is an area of continuous investigation. How to relate the microscopic barriers to the current path such as porosity or oxide barriers to the critical current has become increasingly important in MgB_2. Researchers have used the temperature dependent resistivity as a benchmark for the effective current path in bulk and monocore wires. More recently magnetic *Jc* or 3D tomography percolation models and parametrical indicators such as tortuosity have been used on multifilamentary conductors, [1-4]. The variations in density for in-situ wire are significantly more than for ex-situ and there is clear evidence for a more percolative current path. What has not been achieved to date is to electrically measure individual filaments from a multifilamentary conductor, nor to compare filaments within a conductor.

The mechanical measurement of individual filaments from a multifilamentary conductor has already been undertaken, but not the electrical [5]. The limiting condition is that with filaments 10’s of microns in diameter, the superconductor alone may not be mechanically strong enough to withstand handling and so in this paper individual filaments are measured with a small amount of metal retained to support the filament, which is then gradually removed. The 36 filament MgB_2 conductor manufactured by Hyper Tech analyzed in this paper is doped with 2% C, and has a metal sheath composed of copper, niobium and monel alloy.

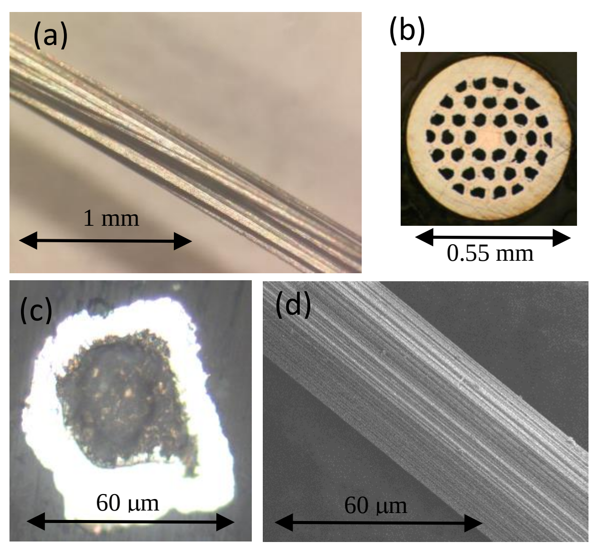

Fig. 1. (a) 36 $Nb/Mg(B_{0.98}C_{0.02})_2$ filaments after removing the monel and copper. (b) The cross section of the Hyper Tech wire showing the 36 $Nb/Mg(B_{0.98}C_{0.02})_2$ filaments the monel and the copper. (c) A cross section of a single $Nb/Mg(B_{0.98}C_{0.02})_2$ filament. (d) A secondary electron image of the outer surface of a $Nb/Mg(B_{0.98}C_{0.02})_2$ filament.

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II. EXPERIMENTAL

The removal of the monel outer and copper inner network seen in the cross section in Fig.1. can be done in a variety of ways but the difficulty is then to make electrical contact to the niobium which forms a stable oxide layer. The current density at the contact of a single filament is higher than the 36 filament wire and early attempts failed due to heating from the contact due to contact resistances of the order of 100 $n\Omega cm^2$ or higher. The method described below reduced the contact resistance to the order of 0.1 $n\Omega cm^2$. It both inhibits formation of Nb oxides by being undertaken in an Argon environment with careful control of the cell current to prevent anodization, and removes any oxides that may have formed despite precautions.

A. Etch and Plate

The monel outer sheath and copper inner were removed electrochemically with a solution of 120 g/l Cu_2SO_4 5% vol H_2SO_4 at a maximum current of 2 mA/mm^2 (anode) which on average required 0.3 V. Individual filaments (RHS Fig. 1) were cut from the filament bundle (Fig. 1(a)) and attached to a copper electrode with silver paint.

The Nb/Mg($B_{0.98}C_{0.02}$)$_2$ filament was further etched for 5 minutes in an acid solution of NH_4F:HCl:H_2O_2:H_3PO_4 in a ratio 2:2:4:1 where 1 part is 30 g/l to remove any Nb oxides.

Etched filaments were immediately copper plated in a solution of 60 g/l Cu_2SO_4 12% vol H_2SO_4 plus small additives of HCl and Thiorea for between 10 and 60 minutes at a maximum current of 0.3 mA/mm^2 which on average required 0.03 V, (Fig.2).

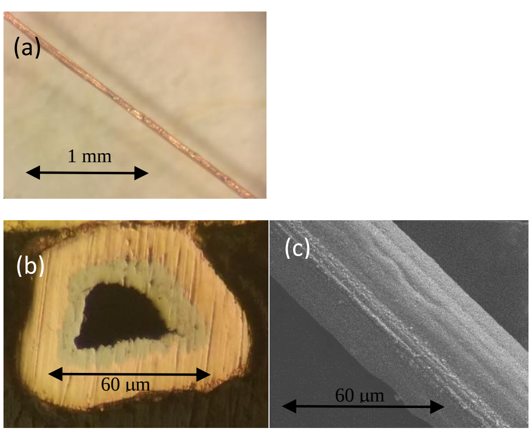

Fig. 2. Optical images of Copper plated Nb/ Mg($B_{0.98}C_{0.02}$)$_2$ filaments (a) laterally and (b) transverse cross section. Scanning electron image laterally (c).

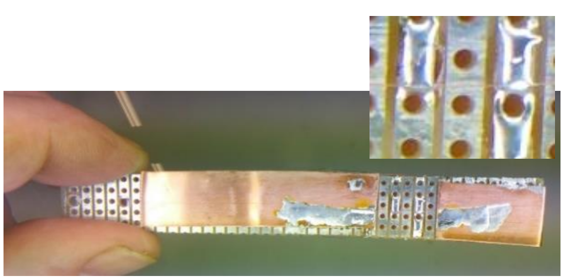

Fig. 3. Copper plated Nb/ Mg($B_{0.98}C_{0.02}$)$_2$ filament mounted on copper current terminals for critical current measurement. Inset shows voltage taps separated by 1.5 mm in higher magnification.

B. Critical Current Measurement

Copper plated Nb/Mg($B_{0.98}C_{0.02}$)$_2$ filaments were cut to length and individually mounted on sample holders and joints soldered with 60/40 lead tin (Fig. 3.). For 6 samples the current joints were 15 mm long to reduce the current contact resistance, with 1.5 mm between voltage taps. A 7th sample was made with 15 mm between voltage taps to investigate continuity of critical current with increasing length.

The mounted filaments were immersed vertically in liquid helium at 1 bar. Current steps of 100 ms duration were applied in 1 A increments until a voltage increase or a quench occurred. Voltage data was logged at 9 kHz. Where no quench occurred the current was reduced and applied for up to 5 s, before increasing the current level again.

The measured filaments were then potted and cross sections polished for identifying which layer the filament came from in the 36 filament wire.

C. Resistivity Characterisation

The remaining length of one of the individual copper plated Nb/Mg($B_{0.98}C_{0.02}$)$_2$ filaments was mounted to a 4 terminal circuit board, and the contacts soldered with 60/40 lead tin. The board was mounted on the resistivity puck of a Quantum Design Physical Property Measurement System and measured for temperature dependent resistivity with both 4–wire (sample resistivity only) and 3-wire measurement (sample and 1 joint resistivity) at fields up to 9 T.

The 4 terminal circuit board was removed and all but the middle section of filament coated in etch resistant varnish. An electrochemical copper etch process was then applied before the circuit board was returned to PPMS for resistivity measurement. The room temperature resistivity of the wire was monitored between etched processes to assess when all the copper was removed. The etching/measurement process was undertaken two more times using the Nb oxide etch.

III. RESULTS

A. Critical Current

The critical current value was taken as the first onset of voltage or the quench of the wire. In one sample, one current contact resistance was of the order of 100 $n\Omega cm^2$ and the voltage increase leading to quench occurred at this contact. As the Ic was low the filament was likely to be at a Temperature greater than 4.2 K, and so the result is not included. The contact resistance of the other 6 samples were of the order of 0.1 $n\Omega cm^2$. The voltage measured in these 6 samples would increase from the lowest voltage resolution of ~1 μV to more than 10 μV in 1 A. Subsequently the filament would quench to above 50 mV in ~ 200 μs. After warming samples had a visible burnt section ~0.1 mm long was visible either halfway between the voltage contacts or halfway between the current and voltage contacts.

Fig. 4. shows cross sections of the measured filaments with the measured critical current values and the cross sectional area of the Mg($B_{0.98}C_{0.02}$)$_2$ core. The error in area was established by

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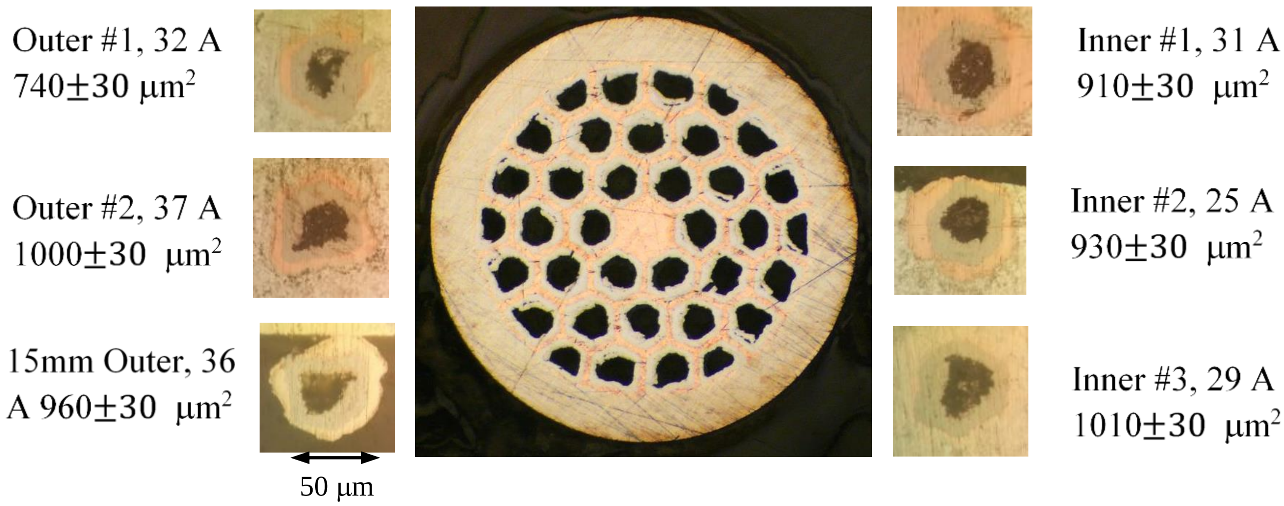

Fig. 4. Optical images of Copper plated Nb/ $Mg(B_{0.98}C_{0.02})_2$ filaments measured for critical current, with central image the 36 filament wire before etching. Included are the critical current values and the cross sectional area of the MgB_2.

measuring the range in area in multiple cross sections of the same filament. A fingerprinting identification was made comparing the measured filaments to the 36 filament wire cross section in order to identify which layer they belonged to. The shape of the outer 18 filaments is distinct to the inner two layers seen by the 5 faces of the Nb as opposed to 6. The inner 6 filaments are harder to distinguish from the middle 12 filaments so remain grouped as the inner 18 filaments.

B. Resistivity

The field dependent resistivity for the outer 2 filament is plotted in Fig. 5. The 4 wire measurements were used to obtain a Bc_2 of 24.7 T. The 3 filament measurements are distinct to the 4 wire measurements due to the additional resistance in the current contact which remains below the *Tc* of the $Mg(B_{0.98}C_{0.02})_2$. In zero field the contact resistance drops to the minimum resolution of 10 μΩ's at ~6 K, showing the superconducting transition of Nb at 9 K and the PbSn solder at 7 K.

The temperature dependent resistivity for the progressively etched outer 2 filament is shown in Fig. 6. Plotted alongside are the modelled resistance of a parallel circuit of niobium using the area taken from the cross section of 2000 μm² (dashed) and 1600 μm² (dash dot) using, $1/R = 1/R_{Nb} + 1/R_{MgB2}$ with $R_{MgB2} = \infty$.

IV. DISCUSSION

The very high critical current density precluded a stable 1 μV/cm measurement of critical current or the full *IV* characteristic. There are few measurements of MgB_2 wires in self field at 4.2 K because of the challenge of a stable measurement due to heating from the current contacts. However given the rapid rise in voltage within 1 A away from the current contacts, the critical current is accurate to ± 0.5 A.

The area of the MgB_2 core in 5 of the filaments are similar between 910 and 1000 μm^2 but in one is significantly lower at 740 μm^2. Of the 3 outer filaments two have the highest *Ic*'s of

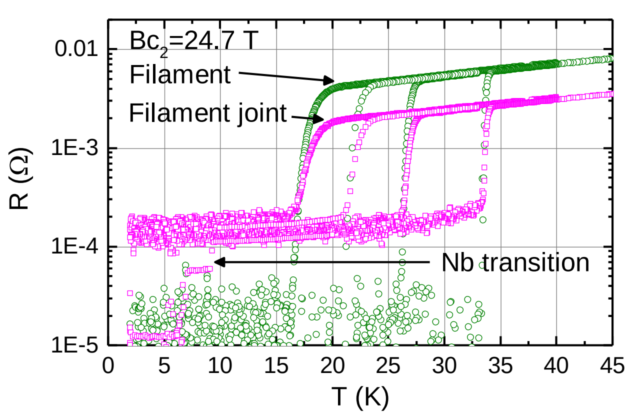

Fig. 5. Resistivity in fields of 0 T, 3 T, 6 T, and 9 T of the outer #2 copper plated Nb/ $Mg(B_{0.98}C_{0.02})_2$ filament. The 3 wire measurement includes the resistance of the current injection and hence the SnPb 60/40 solder and Nb transitions in 0 T.

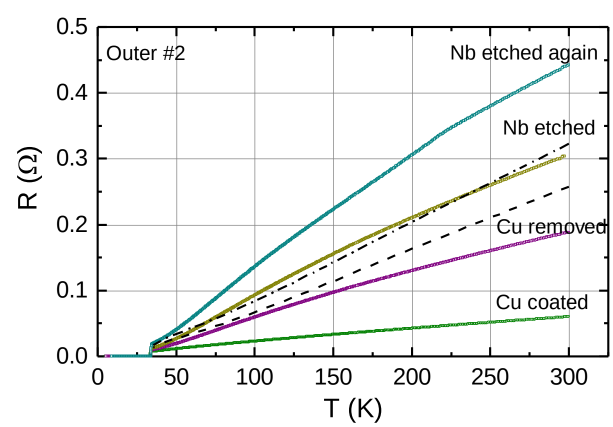

Fig. 6. Temperature dependent resistivity for a progressively etched copper plated Nb/ $Mg(B_{0.98}C_{0.02})_2$ outer 2 filament. The dashed line is the modelled resistance of the Nb only when the copper has been removed, and the dash dot line is the modelled resistance of the Nb only after Nb etching. Inset is the low temperature detail.

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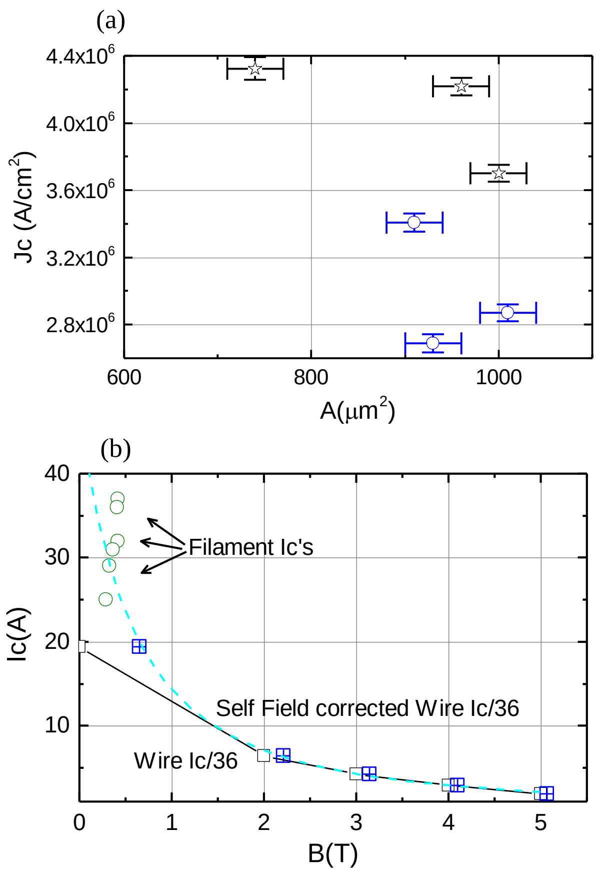

Fig. 7. (a) The critical current density of copper plated Nb/ $Mg(B_{0.98}C_{0.02})_2$ filaments as a function of area for the outer filaments (stars) and inner filaments (circles) as a function of area. (b) the filament *Ic* against self-field compared with the 36 filament wire critical current per filament against applied field and also against applied field plus self-field.

36 A and 37 A with an average of 35 A. For the 3 inner filaments both the average *Ic* and *Jc* is lower than the outer filaments at 28 A.

As one of the outer filaments has a significantly smaller cross section if we consider *Jc*, the outer average is 35% higher than the inner. The highest filament *Jc* is 160% of the lowest.

The *Ic* of the filaments is compared to the *Ic* per filament of the 36 filament wire in Fig. 7 as a function of field, (wire *Ic* values extrapolated from [6]).

The maximum self-field of a single filament can be as high as 0.5 T. The self field was calculated using a first order round wire calculation of $B = \mu_0 I/2\pi r$, where r is derived from the root of the filament area. For the 36 filament wire the maximum self-field is even higher at $\mu_0(36I)/2\pi(14r)$, as current is increasing faster than the radius. The extrapolated data with this first order self-field adjustment is also plotted and correlates to an average filament *Ic* of 30 A.

The gauge length (between voltage) taps of the filament tests were 1.5 mm (5 off) and 15 mm (1 off), which is comparable to the 36 filament wire gauge length of 20 mm.

The resistivity data was intended to be used to extract the $Mg(B_{0.98}C_{0.02})_2$ core resistivity and subsequently find the effective cross section and residual resistivity. The data however cannot be modelled as a pure Nb sheath with a $Mg(B_{0.98}C_{0.02})_2$ core. After removing the copper, but before etching the Nb (Fig. 6) the resistance of the filament looks plausible when compared to the model of Nb only, as it is higher, hence the $Mg(B_{0.98}C_{0.02})_2$ core is contributing to lowering the resistance. However after etching some of the Nb and making the same comparison the filament resistance is higher than the model. There could be two complications. One is the possibility of Nb alloys in the inner layers, causing an increasing sheath resistivity with etching, [8]. The other is differential thermal contraction of the Nb and $Mg(B_{0.98}C_{0.02})_2$ core affecting the current sharing as a function of temperature, by either altering any contact resistance between the Nb and $Mg(B_{0.98}C_{0.02})_2$ core, or within the $Mg(B_{0.98}C_{0.02})_2$ core itself. There Nb resistivity, is also measured in the radial direction in the 3 wire measurement in Fig. 5. The resistivity calculated for current passing radially in a tube with a length of 1.5 mm and inner and outer diameters of 40 μm and 60 μm using $\rho = 2\pi l R/ln(b/a)$ with R=40 μΩ is 1 μΩm. This is a larger resistivity than measured as both Nb and MgB_2 so there must be a contribution from contact resistance between the solder and MgB_2. The value as a contact resistance (40 μΩ over 1.5 mm) is also significantly larger than the contact resistance measured in the critical current measurement (0.1 μΩ over 15 mm). Contact resistance should scale with area so one explanation is there are regions of low contact resistance between the solder and MgB_2 on a length scale greater than 1.5 mm. The copper plating may still have oxides in regions leading to a reduced cross section of contact.

V. CONCLUSION

6 filaments were extracted from a 36 filament conductor and individually tested for critical current performance. The average *Ic* was 32 A, which is close to that of the extrapolated *Ic* per filament for the 36 filament conductor when adjusted for self field. There was a variation of ± 30 % in the filament *Ic* values with the outer filaments measuring on average 35 A, and the inner 28 A. The critical current values are 35% higher in the outer filaments than the inner. The results have implications for optimum conductor architecture and mechanical deformation as they point towards a higher critical current density in filaments that have undergone more mechanical strain. Resistivity measurements with an increasingly etched filament could not be modelled as a parallel circuit of pure Nb and $Mg(B_{0.98}C_{0.02})_2$. Neither could the radial resistance of the Nb and copper plating be treated as a constant.

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