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# Reduction of internal porosity in $Bi_2Sr_2CaCu_2O_x$ round wires and its effect on current transport

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***Abstract*— $Bi_2Sr_2CaCu_2O_x$ is the only cuprate superconductor that can be made into multi-filament round wires. Bi-2212 wires are made using the powder in tube process. The main obstacle to high engineering critical current density ($J_E$) is the low packing density (25 %) of the 2212 powder within the filament which leads to gas-filled filament size bubbles acting like barrier against current transport. We reduced this internal porosity by applying an external over-pressure (OP) during the heat treatment to compensate the internal gas expansion and densify the filaments up to 98 % at an OP of 50 atm. The densification occurs at 821°C within 10 min. With an OP of 100 atm, $J_E$ increased to 917 A/mm$^2$ at 4.2 K, 5 T, compared to a $J_E$ around 200 A/mm$^2$ at 4.2 K, 5 T when long samples are heat treated in 1 atm. Bi-2212 is now a good candidate for magnet application above 20 T.**

## I. Introduction

The high-temperature superconductor $Bi_2Sr_2CaCu_2O_x$ (Bi-2212) is one of the most promising material for high field magnet application above 20 T. The main advantage of Bi-2212 is its ability to be processed into round wires using the powder in tube (PIT) method, which is the ideal shape for magnet winding and magnetic isotropy [1], [2]. The PIT process used to make Bi-2212 wires generates a powder packing density of only about 70 %. The other 30 % is void space filled with gas trapped during the PIT process. During the heat treatment at high temperature Bi-2212 turns into liquid and the gas agglomerates into filament-size bubbles that expand with increasing temperature. Most of the time gas expansion leads to external cracks and leaks. Multiple studies showed that these filament-size gas bubbles were the most important factor limiting current transport [3], [4]. A way to significantly reduce the size of the bubbles and prevent leakage is to apply an external over-pressure (OP) onto the wire during the heat treatment. Compared to already known techniques like cold isostatic pressing (CIP), swaging and hot isostatic pressing (HIP) [5], [6], OP processing maintains the high pressure during the entire heat treatment and ensure a continuous renewing of the needed oxygen by allowing continuous gas flow through the system during the heat treatment. In this paper we study the densification of Bi-2212 wires and its effect on current transport.

## II. Experimental details

The Bi-2212 wire selected for this study was made by Oxford Superconducting Technology (OST). It has a diameter of 0.8 mm with an architecture of 37 filaments in 18 bundles (37x18) as shown in Fig. **1**. 8 cm long samples were cut and their ends were sealed with pure silver to trap the internal gas.

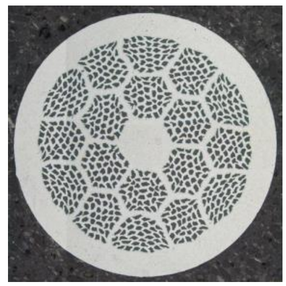

Fig. 1. Cross section of an 0.8 mm in diameter Bi-2212 round wire made by OST.

This precaution is necessary to be able to apply the external pressure and also to reproduce in short lengths of wire the same behavior than coil length of wire.

In the first part, densification of wires without melting Bi-2212 has been studied. Samples were heat treated up to 821°C following a simple ramp up – soak – ramp down sequence with an OP of 50 atm. The time spent at 821°C was varied from 0 min (direct cooling) up to 48 h. The heating and cooling rate were set at 160°C.h$^{-1}$. In the second part, the effect of wire densification on $J_E$ has been studied. Samples were fully processed using a standard heat treatment [7] up to about 890°C with different OP pressures varying from 5 to 100 atm. In both sets of studies, the partial pressure of oxygen was kept at 1 atm during the entire heat treatment to limit the amount of non-superconducting secondary phases that formed [8] ($(Sr,Ca)_{14}Cu_{24}O_x$ (AEC), $Bi_2Sr_2CuO_x$ (2201), and $Bi_9(Sr,Ca)_{16}O_x$ (CF)).

Wire diameter was measured before and after heat treatment using an iNEXIV Nikon VMA 2520 optical microscope by projection of the wire shadow. The density measurements were performed by mass and cross section analysis. The cross section images were taken with an optical microscope and analyzed with *Photoshop* software. Finally, critical current measurements were done using the four probe method with a 1 μV.cm$^{-1}$ criterion at 4.2 K in a magnetic field of 5 T.

## III. Results and discussion

### *A. Densification at 821°C*

Bi-2212 powder melts around 880°C, so at 821°C it is in the solid state. Fig. 2 shows filament cross sections from wires held at 821°C for 0 min, 10 min and 48 h. A reference sample with no sealed ends and that was held at 821°C for 48 h is shown for comparison. It appears that the sample with no soak at 821°C (0 min) shows the same cross section as the sample without sealed ends that was held at 821°C at 48 h. The calculated filament density for these two types of wires was about 85 % of the density of Bi-2212 phase (6.6 g/cm$^3$), showing only slight densification. However, the wires held

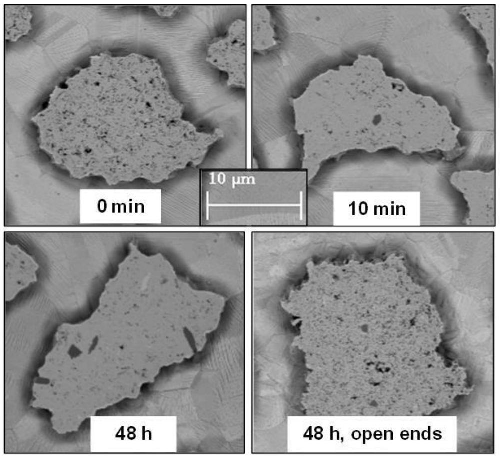


Fig. 2. Cross section of Bi-2212 filaments from sealed wires heat treated at 821°C for 0 min, 10 min, 48 h and 48 h with open ends. The OP was set at 50 atm with 1 atm of $O_2$.

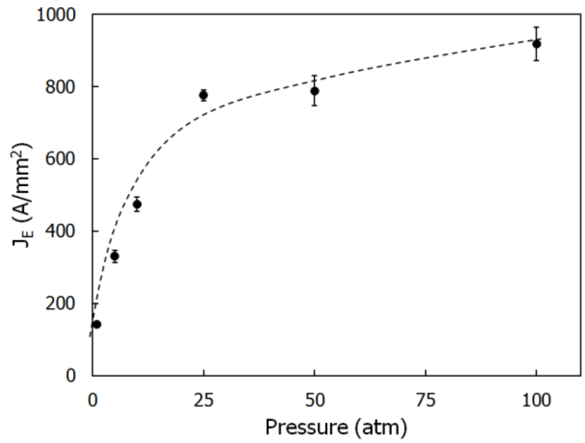


Fig. 3. Evolution of the engineering current density as a function of the OP pressure applied during the heat treatment. The partial pressure of oxygen is kept constant at 1 atm the entire heat treatment.

between 10 min and 48 h at 821°C with sealed ends show good densification that confirmed by our calculation with a filament density of 98±1 %. The first main conclusion of this portion of the study is that OP processing can densify Bi-2212 even when it is a powder. It means that trapped gas (e.g. $CO_2$ and $H_2O$) is still evenly distributed along the filament and therefore the densification happens uniformly along the filaments allowing the filaments to keep their original shape even after melting of Bi-2212. Indeed if densification could only occur after the Bi-2212 melted, it would occur only at the bubble locations generating a sausage shape. In addition the densification occurs very quickly, within the first 10 minutes at 821°C. By pushing our study further, we showed that the longer the time spent at 821°C the larger the grains of secondary phase within the filaments grew. Therefore it is crucial to limit the time at 821°C, which is possible since Bi-2212 wire densifies in 10 min with a pressure of 50 atm.

### B. *Effect of filament densification on $J_E$*

Samples have been fully processed using the standard heat treatment schedule with different OP pressures to understand the impact of the filament density on $J_E$. Fig. 3 shows the evolution of $J_E$ as a function of the applied OP pressure during the heat treatment. As a reference, short samples heat treated at 1 atm with sealed ends show a $J_E$ of 142 A/mm$^2$ at 4.2 K, 5 T. It appears that $J_E$ increases with increasing pressure. Between 1 atm and 25 atm, $J_E$ increases rapidly from 142 A/mm$^2$ to 775.4±14.7 A/mm$^2$ and then slowly increases up to 917.9±45.6 A/mm$^2$ at 100 atm. The highest value obtained with this type wire is 980 A/mm$^2$. Therefore $J_E$ of Bi-2212 round wires increased by a factor of 6 in sealed short samples by applying 100 atm OP which significantly reduce the size of the bubbles. However the gas is still present in the filaments under high pressure. The record value ever reached by OP processing on Bi-2212 round wire is 1089 A/mm$^2$ [4.2 K, 5 T] and was reached with another OST wire made with the same architecture and from a different batch of the same powder from Nexans SuperConductor (Germany). It seems that 100 atm OP is close to the maximum pressure needed to densify filaments for this specific wire architecture. The interesting point to highlight about OP processing is the fact that the pressure is relatively low compared to the high pressure used in HIP units (around 14 katm). $J_E$ of 917.9±45.6 A/mm$^2$ obtained for Bi-2212 round wires with 100 atm OP at 4.2 K, 5 T represents a $J_E$ of 611 A/mm$^2$ at 4.2 K, 20 T. This makes Bi-2212 round wires a candidate, along with $YBa_2Cu_3O_x$ (YBCO) tapes for high field magnet application above 20 T.

## IV. Conclusion

Bi-2212 round wire transport current properties were significantly increased up to $J_E$ of 917.9±45.6 A/mm2 by using over-pressure processing at 100 atm. It has been shown that the densification needed to increase $J_E$ occurs quickly at 821°C where Bi-2212 is still in the solid state. The main current transport limiting factor – bubbles – are removed by OP processing. Those results were confirmed on 1 m long sealed samples, giving proof of the possible application of OP on large coils, which makes Bi-2212 the perfect candidate for the next generation of high field magnets. The National High Magnetic Field Laboratory increased its OP capabilities by acquiring two 100 atm furnaces. One is a three-zone furnace with a 4.5 cm inner diameter for small coils and short samples. The second is a large scale 100 atm furnace designed to process coils up to 17 cm in diameter and 50 cm tall.

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