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Understanding the densification process of $Bi_2Sr_2CaCu_2O_x$ round wires with overpressure processing and its effect on critical current density

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Abstract

Overpressure (OP) processing increases the critical current density (J_C) of $Bi_2Sr_2CaCu_2O_x$ (2212) round wires by shrinking the surrounding Ag matrix around the 2212 filaments, driving them close to full density and greatly increasing the 2212 grain connectivity. Indeed densification is vital for attaining the highest J_C. Here, we investigate the time and temperature dependence of the wire densification. We find that the wire diameter decreases by 3.8 ± 0.3 % after full heat treatment at 50 atm and 100 atm OP. At 50 atm OP pressure, the filaments start densifying above 700 °C and reach a 3.30 ± 0.07 % smaller diameter after 2 h at 820 °C, which is below the melting point of 2212 powder. The densification is homogeneous and does not change the filament shape before melting. The growth of non-superconducting phases is observed at 820 °C, suggesting that time should be minimized at high temperature prior to melting the 2212 powder. Study of an open-ended 2.2 m long wire sample shows that full densification and the high OP J_C (J_C varies by about 3.1 times over the 2.2 m long wire) is reached about 1 m from the open ends, thus showing that coil-length wires can be protected from leaky seals by adding at least 1 m of sacrificial wire at each end.

I. INTRODUCTION

$Bi_2Sr_2CaCu_2O_x$ (2212) round wire, made by the powder-in-tube (PIT) process, is the only high temperature superconductor (HTS) that can be made into multifilamentary round wire [1]–[4]. An important current-limiting mechanism in 2212 – bubbles - is generated by the low (~60%) powder density in the filaments of as-delivered wires and the behavior, during melting, of trapped gas in this void space. During heating in 1 atm flowing oxygen, the trapped gas expands, provoking outward creep of the Ag sheath and expanding the filament cavities. When the powder melts, the well-distributed porosity agglomerates into large, filament-size, gas bubbles [5]. Outward creep of the Ag sheath leads not just to filament expansion but also, in the worst case, to cracks and leaks of liquid 2212 through the Ag sheath [6]–[9] during the melt step of the heat treatment (HT). Larbalestier *et al.* showed that overpressure (OP) processing at 100 atm of short samples with well-sealed ends prevented gas expansion, densified the wire, and increased the engineering current density (J_E) by about a factor of 6 compared to wires processed at 1 atm with closed ends (from 146 A/mm^2 to 911 A/mm^2 at 5 T, 4.2 K at 1 and 100 atm respectively) [10]. The closed end samples processed at 1 atm showed severe diameter increase and sheath rupture. The transformative

outcome of OP is that 2212 can now provide the J_E needed to generate fields well above 20 T.

The OP process strongly decreases the volume of the gas-filled space in the filaments that corresponds to the difference in volume between the as-manufactured filament cavity and the 2212 powder. Densifying the filaments with OP also decreases the wire diameter. The root cause of the increase in J_C with OP is reducing the empty (i.e., gas filled) filament volume and consequently increasing the 2212 density in the filaments. The purpose of this paper is to provide an explicit study of the 2212 wire densification before melting the 2212 and also to explore the consequences imperfect seals have on densification under OP conditions, which is particularly important for coils where many hundreds of meters of wire may be at risk of having degraded J_C if the end seals are not perfect.

II. EXPERIMENTAL DETAILS

All experiments were performed with powder-in-tube 2212/Ag wires made from Nexans powder with standard 521 composition ($Bi_{2.17}Sr_{1.74}Ca_{0.89}Cu_{2.00}O_x$) by Oxford Superconducting Technology (OST) [11]. This study was done with double stack 0.8 mm diameter wires with an 18x37 architecture (18 bundles of 37 filaments) and a 1.3 mm diameter wire with an 18x121 architecture (18 bundles of 121 filaments). The outer sheath of the wires is made of Ag-0.2wt%Mg alloy and the inside matrix is made of pure Ag. Short samples were 8 cm long with fully-sealed ends made by wrapping a short piece of pure Ag wire around each end of the 2212 wire and then melting it to create the Ag seal. OP heat treatments were done with Ar/O_2 mixtures with total pressures up to 100 atm but always with constant oxygen partial pressure of 1 atm.

Wire diameters were measured before and after OP heat treatments with an iNexiv Nikon VMA 2520 light microscope set up with the light source located above the wire. The wire diameter is measured automatically through a program that selects both wire edges using the high contrast between the dark wire and the bright background. The measurement is repeated every 0.25 mm along a 2 cm section in the middle of the wire to avoid any artifacts introduced by the sealing process. The measurements are then averaged to obtain the wire diameter and its standard deviation.

For SEM cross-section imaging, samples were put into conductive epoxy resin and polished manually with 320 to 1200 grit SiC papers, and then finished for about 12 h using a Buehler Vibromet 2 vibratory polisher with a 0.05 μm Al_2O_3 powder mixed with ethanol. For filament imaging in a Zeiss 1540 XB SEM scanning electron microscope (SEM), wires were dipped into a hydrogen peroxide, ammonium hydroxide and methanol solution in a 1:1:1 volume ratio, for up to 2 h to etch the Ag. Wires were then carefully rinsed with pure methanol for cleaning.

Critical current (I_C) was measured on 4.5 cm long sample sections using the four probe technique with a 1 μV/cm criterion at 4.2 K and 5 T, a standard benchmark for our I_C testing of 2212. Voltage taps were set 1.75 cm from each end and were 1 cm apart.

Densification of 2212 round wires, reported in section III.A, was studied as a function of time and temperature below the melting temperature of 2212 to understand the domain in which the Ag matrix of the wire can creep and permit densification. To study the temperature dependence, samples with sealed ends were heat treated according to Figure 1a at 50 atm for 2 h at different maximum temperatures (T_{max}) with ramp up rate of 160 °C/h, which corresponds with the heating rate in the standard heat treatment Figure 2a [11]. The pressure was first set at 10 atm at room temperature and then increased to 50 atm when T_{max} was reached to establish conditions such that there was no increase in pressure during heating to T_{max}. We chose to spend 2 h at T_{max} because it corresponds to the first plateau of the standard (but empirically developed) melt heat treatment in which the temperature is held at 820 °C for 2 h, originally as an aid to allow gas to escape from open-ended wires. OP pressures of 50 atm and 100 atm were explored. To study the time dependence of the densification process, sealed samples were heat treated at 50 atm, as shown in Figure 1b, at 820 °C with ramp up at 160 °C/h. The time spent at 820 °C (t_{max}) was varied from 2 min to 48 h. The pressure was set at 50 atm from room temperature.

Densification of a 2.2 m long wire with open ends is reported in section III.B. For this experiment we used a 1.302 ± 0.001 mm diameter wire made by OST with a 121x18 filament architecture. Before HT, the two ends were polished with 800 grit SiC paper to make sure that all the filaments were as open as possible. The sample was bent into a race track shape with bend diameters of 15 mm to keep most of the wire as straight as possible, to make it easier to measure the wire diameter. The 2.2 m wire was heat treated at 100 atm following the standard heat treatment but with a 2 h soak at 831 °C instead of the standard 48 h (this time was reduced to shorten the time of the experiment), as shown in Figure 2. To provide a reference for J_C and wire diameter, short samples were fully heat treated following the standard heat treatment at 100 atm OP [11].

III. RESULTS

A. OP densification of filaments as a function of T_{max} and time done at temperatures below the melting point of 2212

Figure 3 shows the evolution of the wire diameter as a function of T_{max}. The average as-drawn wire diameter (0.803 mm ± 0.001 mm) is shown at T_{max} = 25 °C as the baseline. The diameter of wires after a full standard heat treatment at 50 and 100 atm OP with T_{max} = 888 °C is also shown. Up to T_{max} = 611 °C the wire diameter after heat treatment is similar to the as-drawn wire diameter. At T_{max} = 722 °C, the wire had started to densify with a diameter decrease of 0.6 ± 0.2 % to a diameter of 0.798 ± 0.001 mm indicating that measurable densification starts between 611 °C and 722 °C. At T_{max} = 830 °C, which is below the melting temperature of 2212 ($T_{melt} \cong$ 884 °C), the decrease in diameter was 3.3 ± 0.4 % to a diameter of 0.777 ± 0.003 mm. The full heat treatment at 50 atm and 100 atm OP with T_{max} = 888 °C decreased the wire diameter by 3.5 ± 0.2 % to a wire diameter of 0.775 ± 0.002 mm at 50 atm, and 3.8 ± 0.3 % to a wire diameter of 0.772 ± 0.002 mm at 100 atm. Samples processed at 50 and 100 atm show similar densification behavior. Analysis of wire transverse cross-sections showed that the wires are still round and show no significant change in roundness.

The time dependence of the densification process at 820 °C is shown in Figure 4. As a reference, the average wire diameter before heat treatment is shown (0.802 mm ± 0.001 mm). After 2 min at 820 °C, the wire diameter decreased by 2.68 ± 0.15 % to 0.781 ± 0.001 mm and after 2 h the diameter had decreased by 3.30 ± 0.07 % to 0.776 ± 0.001 mm, which is consistent with Figure 3. The maximum densification at 820 °C occurs within 6 h with a decrease in diameter of 3.69 ± 0.11 % to 0.773 ± 0.002 mm. A longer time spent at 820 °C did not lead to further densification.

Figure 5 compares filaments after etching the Ag away from as-drawn wires and wires heat treated at 820 °C for 2 h and 12 h at 50 atm OP. The filaments are very similar in shape in all three cases, showing that the densification occurs homogeneously, as would be expected from the isotropic external pressure field provided by the gas. Figure 5b and c show large grains within the filaments of the heat treated samples. After 2 h at 820 °C, the large grains are numerous and up to 10 μm long, and after 12 h at 820 °C, the grains are even larger, up to 20 μm long, but less numerous. Energy dispersive X-ray analysis revealed that the large grains are $Bi_9(Sr,Ca)_{16}O_x$ (copper-free phase (CF)) and $Bi_2Sr_2CuO_{6+x}$ (2201), which are non-superconducting secondary phases. However, no large 2212 grains are visible, and the 2212 still has its original powder morphology.

Figure 6 compares the filament cross sections of an as-drawn wire, a wire heat treated at 820 °C for 12 h at 100 atm OP, and a wire fully processed at 100 atm OP. Wire densified at 820 °C for 12 h shows a microstructure very similar to as-drawn wire, i.e. filaments that are fully independent of each other, but much denser. The secondary phases described above (Figure 5) are also present in the wire heat treated at 820 °C for 12 h at 100 atm and there are dark spots visible in Figure 6a, corresponding to alkaline earth cuprate phase, that are not observed in Figure 5c. Microstructures similar to Figure 5b are observed after densification at 820 °C for 2 h at 100 atm OP. Fully-processed wires have a very complex microstructure due to melting of the powder and growth of new 2212 grains on cooling, some of which connect across the Ag matrix between the filaments.

B. Wire densification as a function of distance from the open ends

Figure 7a shows that the diameter of the 2.2 m wire decreases smoothly, reaching a stable value, corresponding to the diameter of a fully-sealed short sample, about 1 m from either end. Both ends show a slight increase in diameter in the first 20-30 cm (maximum expansion of 0.56 ± 0.08 % to a diameter of 1.310 ± 0.001 mm), while the center of the wire shrinks by 4.10 ± 0.23 % to a diameter of 1.249 ± 0.004 mm. Figure 7b shows J_C and n-values at 4.2 K and 5 T of samples along the length of the wire. J_C is calculated as the ratio of I_C to the densified filament cross section area (2212 + voids) of the wire after the typical 3.2 % reduction in diameter to 1.261 mm after heat treatment at 820 °C for 12 h at 50 atm. The measured fill fraction of the wire is 0.254 after densification at 820 °C for 12 h at 50 atm. The J_C curve follows the inverse trend of the diameter curve with the lowest J_C (1364.3 ± 3.2 A/mm^2) at the open ends of the wire and the highest J_C (4303.8 ± 21.8 A/mm^2) at the center. For comparison the fully heat treated short wires showed an average diameter decrease of 4.44 ± 0.21 % to 1.245 ± 0.003 mm and J_C of 4416.6 ± 107.3 A/mm^2 [4.2 K, 5 T]. The two samples located at 165 cm and 192 cm show very low J_C and also very low n-values

(respectively 4.4 and 2.9), leading us to believe that they were damaged by handling. The n-values of all samples are relatively constant at 21.7 ± 2.4. The two low n-value samples at 165 and 192 cm were not included in the calculation. Figure 8 shows J_C of the sections of this open-ended 2.2 m long wire is a linear function of the measured diameter. The data do not include the two samples at 165 and 192 cm. J_C is about 3.1 times higher in the middle of the wire compared to the open ends, so it is clearly important to take account of the possibility to severely degrade J_C if the wire ends are poorly sealed. The data point in Figure 8 showing a low J_C of 2824.6 ± 12.0 A/mm^2 (4.2 K, 5 T) at a decrease in diameter of 3.14 % is at 138 cm in Figure 7b and the sample shows an abnormally high n-value of 33.5. This may indicate a forced quench during the I-V test suggesting that J_C for the sample is actually larger than shown in Figure 8.

IV. DISCUSSION

A. Reduction of internal porosity in 2212 round wire

Densification starts between 611 °C and 722 °C and is almost complete after 2 h at 830 °C, well below the melting point of the powder. There may be a distinct advantage of densifying the filaments before the 2212 powder melts, which is that the densification occurs homogeneously throughout the whole wire cross section without changing the filament shape. This is a consequence of the powder and voids being uniformly distributed in the as-drawn filaments before melting, which may not be the case if densification occurred after melting when the voids have agglomerated into large, filament-size bubbles. Our experiments also indicate that 50 atm OP pressure is high enough to densify the 0.8 and 1.3 mm diameter wires throughout their entire cross section and that the 2212 powder density is uniform across the entire cross section. Examination of the OP densification process suggests that these results will extrapolate well to full-size coils made with several hundred meters to km of conductor. Indeed, wires extracted from small coils made with tens of meters of wire show full densification and high J_C.

Figure 4 shows that a sample began to densify in just 2 min after reaching 820 °C with 50 atm OP during the entire heat up (Figure 1b), indicating that densification occurs very rapidly. However, this is a bit misleading, as densification occurred in this sample during the time the sample was being heated up at 160 °C/h, during the 2 min it was at 820 °C, and during the furnace-cooling to about 700 °C, which was at a rate of about 120 °C/h. This suggests that the wire has attained the maximum densification it can achieve at 820 °C while heating to 820 °C at 160 °C/h.

The secondary phases observed at 820 °C after 2 h show that 2212 decomposes on heating to grow second phases. It is unclear if the second phases were already present as nuclei in the as-drawn filaments or if they nucleate on heating. However the fact that the grains of the secondary phase increase in size and reduce in number with increasing time at 820 °C suggests that 2212 decomposes into 2201 and CF phases on heating, which is consistent with [12] that shows 2212 decomposes on heating at P_{O2} higher than 1 atm. This suggests that P_{O2} in the wire during our OP might be slightly higher than 1 atm, which is set by the total OP pressure and the fraction of O_2 in the Ar/O_2 mixture. Spending a long time at high temperature before melting the 2212 could therefore be detrimental for J_C due to the

increasing amount of secondary, non-superconducting phases in the filaments that might not be consumed as 2212 grows from the melt on cooling. The growth of 2201 in wires heated in air has been observed to start at around 200 °C and is maximum around 500 °C [12] showing that growth of secondary phases is slow and continuous with increasing temperature and time. Reducing or eliminating the 2 h annealing time at 820 °C in the standard heat treatment should reduce the amount and size of secondary solid phases in the melt and possibly improve I_C. As noted above, densification is already high on reaching 820 °C at 160 °C/h at 50 atm OP, but a small amount of additional densification will occur as the wire is heated at 50 °C/h from 820 °C up to the melting point of 2212, and possibly during melting and cooling.

B. Densification of open-ended wires

The densification of 2212 wires during OP is controlled by the quality of the sealing at each conductor end because only a full hermetic seal allows isostatic pressure to be applied to the whole wire length. Our state-of-the-art method for wire sealing today is very efficient and few samples show compromised densification. However in the manufacture of a coil involving hundreds of meters of conductor, we need to be certain that the entire coil is densified. Figure 7a shows that a 2.2 m long wire with the worst case scenario of both ends being fully open, reaches the usual high OP densification and high J_C about 1 m in from the open end. This is an important result for magnet manufacture because it indicates that the entire wire length can be densified during the OP heat treatment even with a poor end seal by adding at least 1 m of extra conductor to each end. These sacrificial ends can be removed after the heat treatment is complete.

The results in Figure 8 from the 2.2 m open-end wire show a linear correlation between J_C and decreased wire diameter with 100 atm OP processing. The cause of the enhanced J_C is the reduced porosity within the filaments and consequent enhancement of the filament connectivity. Reducing the wire diameter by about 4 % enhances the critical current density by about 3.1 times compared to the ends of the open-ended wire that did not densify. This shows the huge J_C penalty for only partially densifying the filaments. The linear relation in Figure 8 suggests that it may be possible to predict J_C after full OP processing by measuring the diameter of the densified wire.

In the experiments with the 2.2 m long wire we decreased the length of the annealing time at 831°C during cooling from the standard 48 h to just 2 h to reduce the length of time for the experiment. Initially we did not plan to measure J_C on theses samples, so we were not concerned with shortening the 831°C step; however, we subsequently decided to measure J_C. We found that J_C for just 2 h at 831°C was as high as for the full 48 h at 831 °C during cooling. Shen [13] showed that 36-48 h were needed at 831°C when processing in 1 atm flowing oxygen after 2212 growth for high J_C for a different wire made with a composition slightly off the Nexans standard 521 composition. We have subsequently completely removed the 831°C annealing step in the full OP heat treatment with no degradation in J_C showing that the annealing step at 831°C can be eliminated with OP, which significantly shortens the standard HT time and is very beneficial for coil processing.

SUMMARY

We have studied the time and temperature dependence of wire densification with OP at 50 and 100 atm and the position dependence of I_C in from the open ends of a 2.2 m long wire. We found that densification is almost fully completed before melting the 2212 powder resulting in a homogeneous densification of the filaments while maintaining their initial shape. The wire greatly densifies during heating at 160 °C/h from 700 °C to 820 °C. In the case of open-ended wires, we observed that full densification occurred 1 m in from the open ends during full heat treatment at 50 atm OP. This shows that at least 1 m of sacrificial wire should be added to each end of the coil so that if the seals are poorly made, the critical portion of the coil will still have proper densification.

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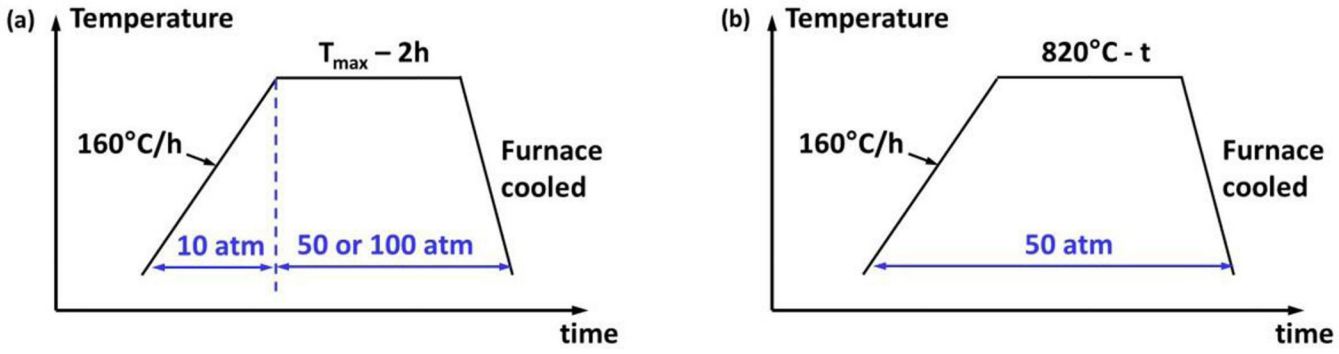

Figure 1.

Schematic of the heat treatment schedule used to study the (a) temperature and (b) time dependence of wire densification. Schedule (a) was set to maintain the pressure below 50 atm on heating. We then modified the furnace system, which allowed us to control the pressure on heating more accurately and use 50 atm from room temperature without overshooting 50 atm during heating.

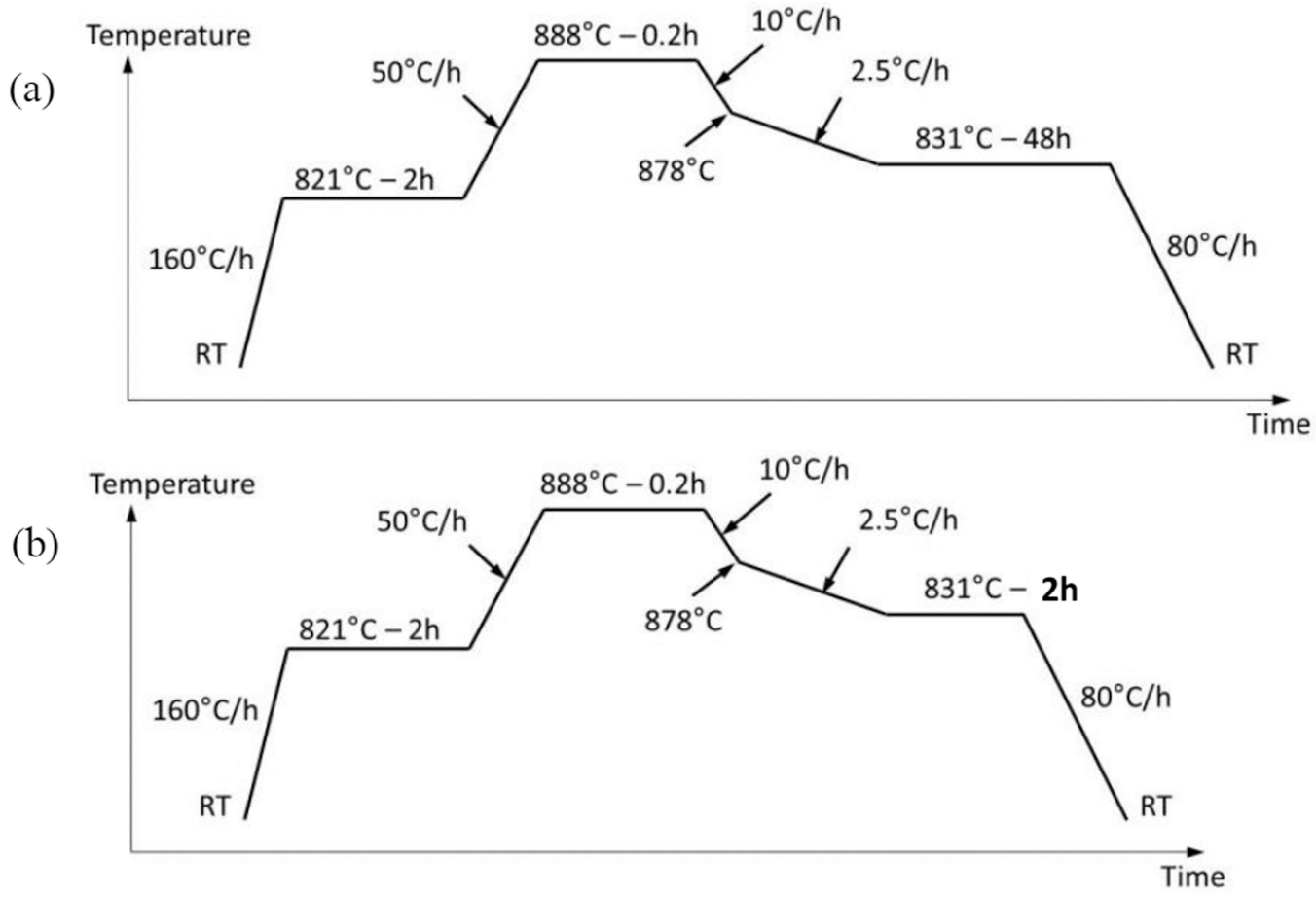

Figure 2.

Schematic of (a) the standard OP heat treatment [11] and (b) the shortened OP heat treatment used to study the densification of a 2.2 m long 2212 wire at 100 atm OP with both wire ends fully open. The 48 h plateau at 831 °C was shortened to 2 h. The OP pressure was set at 100 atm the entire time of the OP heat treatment.

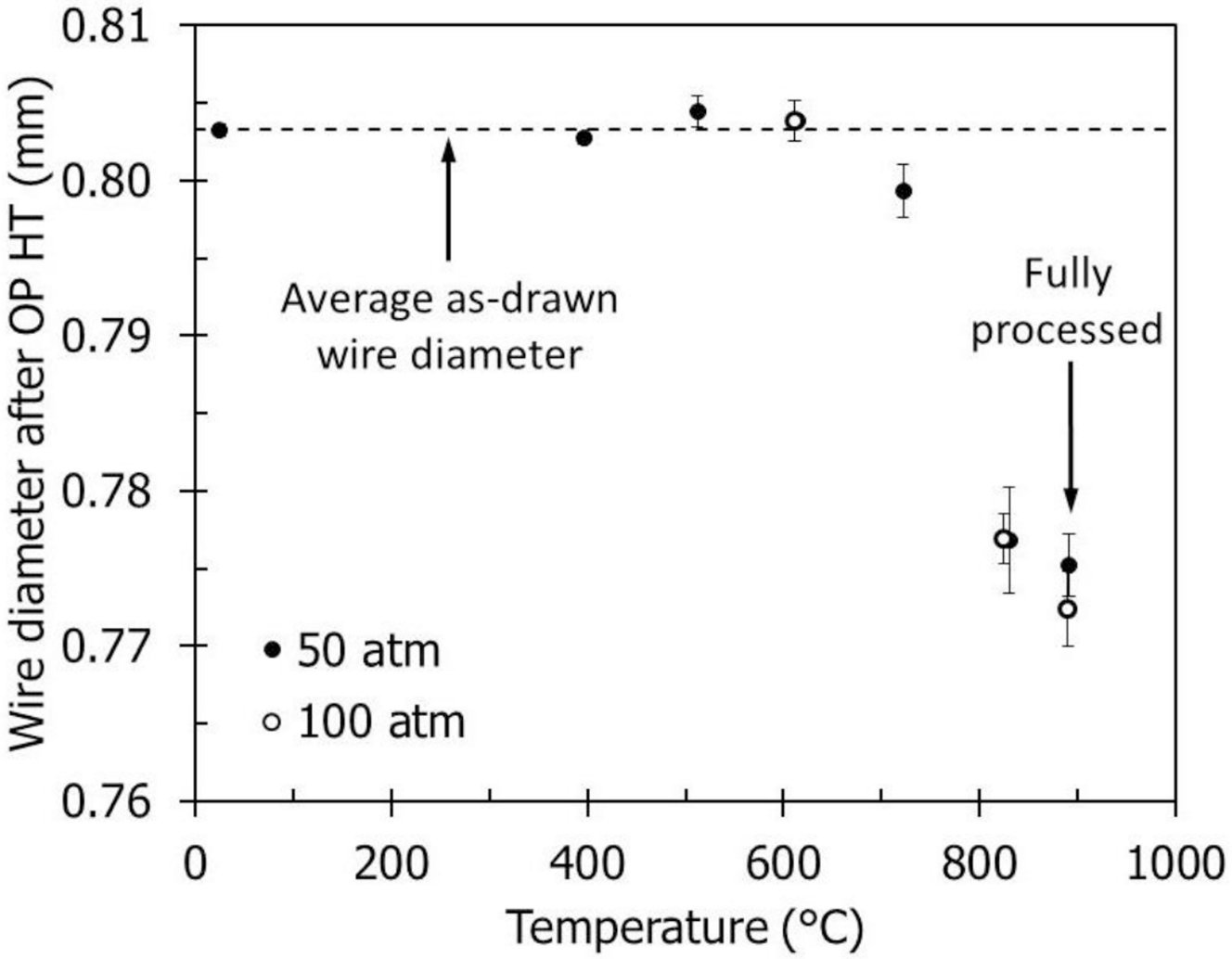

Figure 3.
Wire diameter after heat treatment at T_{max} for 2 h using the HT in Figure 1a. The fully processed wires have been heat treated with the standard heat treatment in Figure 2a at 50 and 100 atm OP.

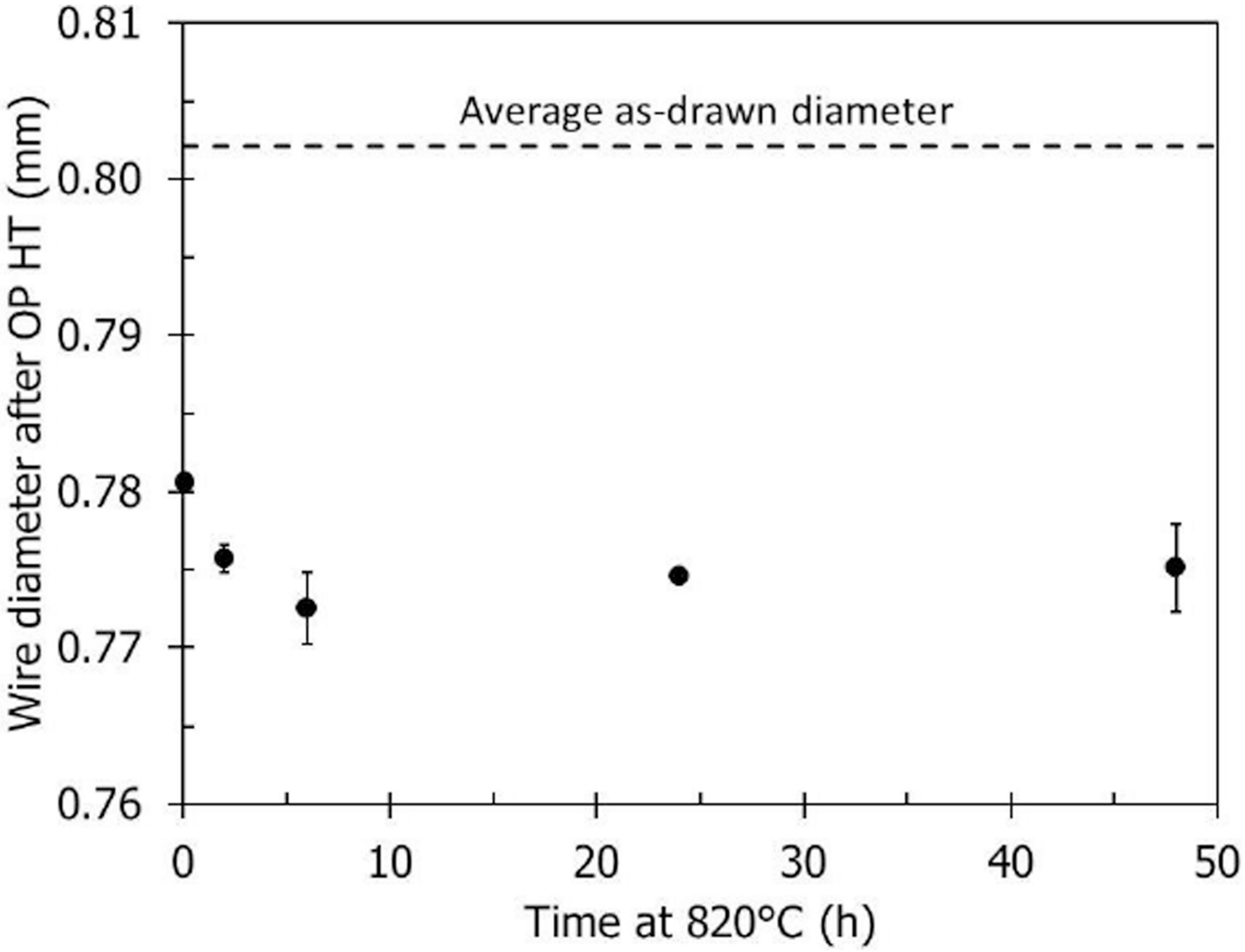

Figure 4.
Wire diameter after heat treatment (Figure 1b) at 50 atm OP as a function of time spent at 820 °C.

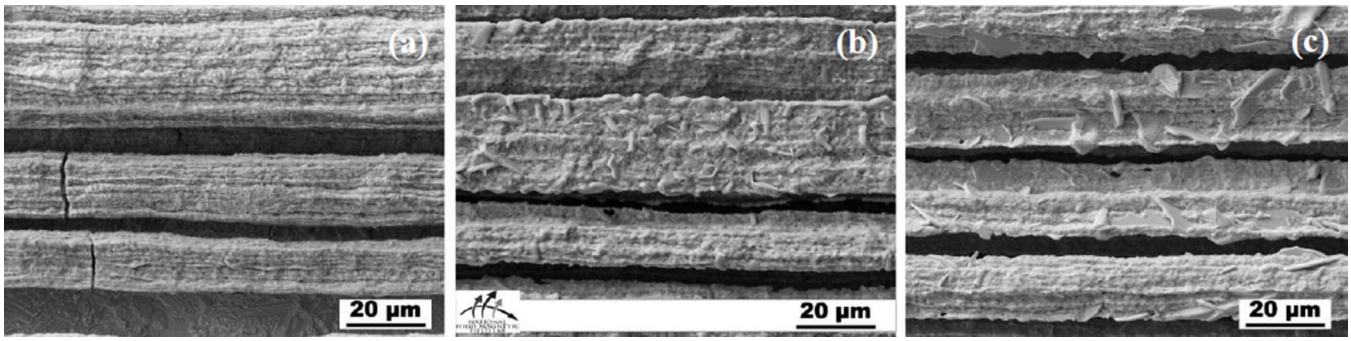

Figure 5.

SEM images of filaments from 0.8 mm diameter (a) as-drawn wire, and wire after 100 atm OP at 820 °C for (b) 2 h and (c) 12 h. The cracks visible in the as-drawn filaments in (a) occurred during etching.

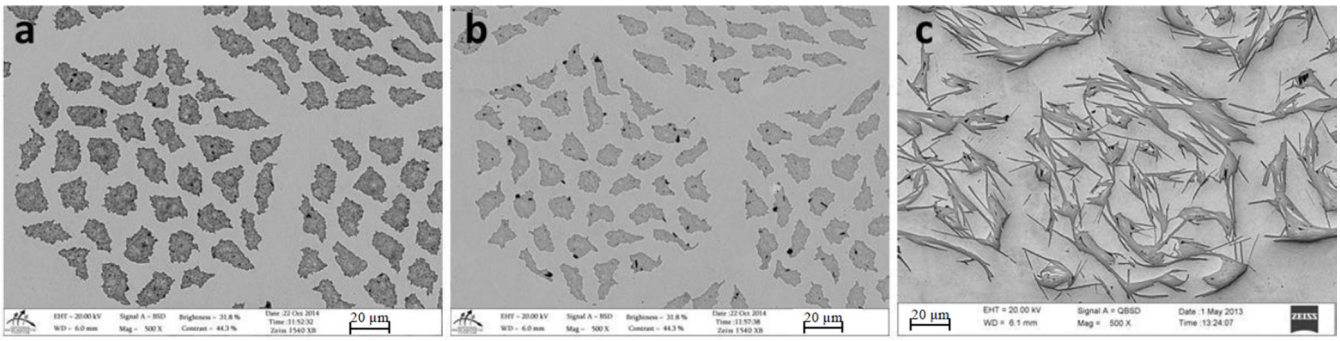

Figure 6.

SEM backscattered electron images of cross sections of 0.8 mm diameter (a) as-drawn wire, (b) wire after 12 h at 820 °C at 100 atm OP, and (c) wire after standard heat treatment at 100 atm OP.

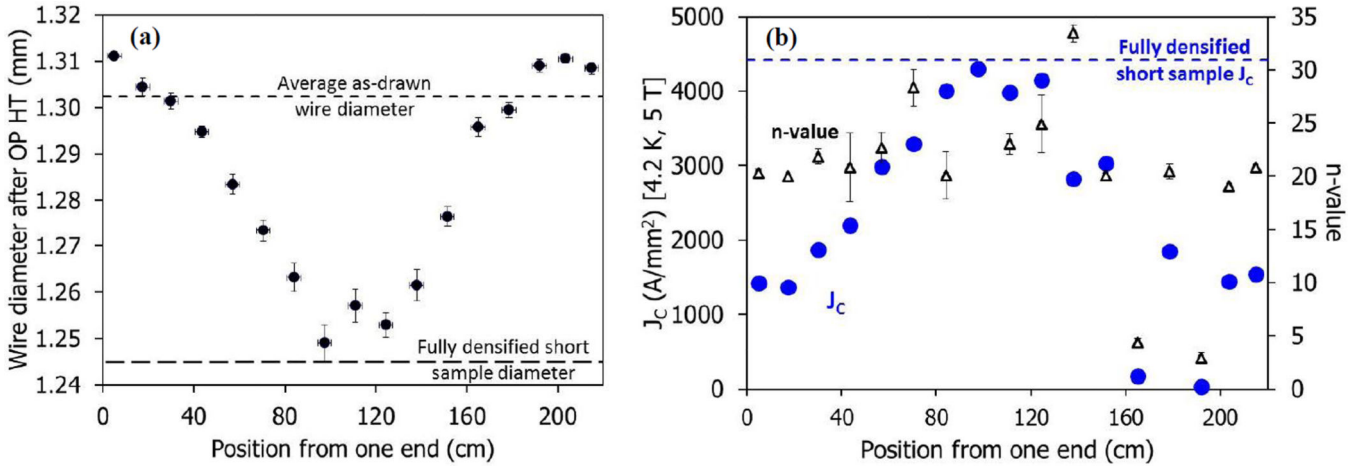

Figure 7.

(a) Diameter, and (b) J_C and n-value at 4.2 K and 5 T of a 2.2 m long wire after full heat treatment at 100 atm with both ends fully open. The diameter of the as-drawn wire and the diameter of the fully densified short wire that had both ends closed are shown for reference. J_C is calculated as the ratio of I_C to the densified cross section area of the filaments (2212 + voids) measured after 820 °C for 12 h at 50 atm.

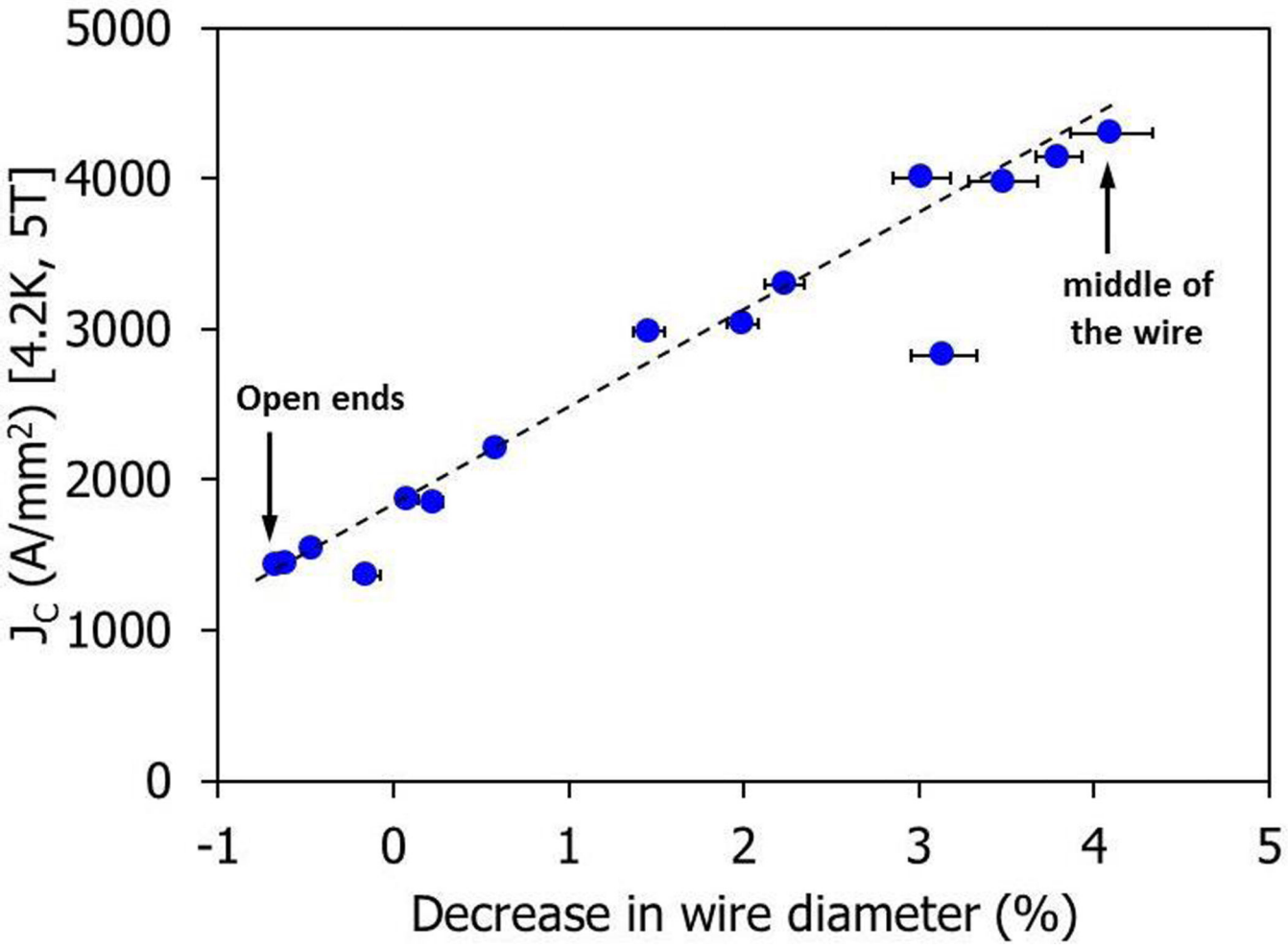

Figure 8.

J_C at 4.2 K and 5 T (from Fig. 7b) as a function of the decrease in wire diameter after full heat treatment at 100 atm with both ends fully open for a 2.2 m long wire. The dashed line is a guide for the eyes.