# Effects of Wire Diameter and Filament Size on the Processing Window of Bi-2212 Round Wire

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*(Invited Paper)*

***Abstract*—High engineering critical current density ($J_E$ of 1300 A/mm$^2$ at 4.2 K and 15 T) in Bi-2212 round wire has been achieved through a partial melt, overpressure heat treatment process. $J_E$ varies strongly with processing conditions, particularly the maximum heat treatment temperature ($T_{\max}$). Increasing $T_{\max}$ results in longer time in the melt (defined as the time between when Bi-2212 melts on heating and when Bi-2212 begins to form on cooling), more bridging between the filaments, lower $J_E$, and higher ac losses. A wide processing window with a large range of $T_{\max}$ that has a nearly constant $J_E$ is desired for processing large coils with large thermal mass and significant thermal time constants that may make precise control over the desired temperature – time profiles uncertain. Accordingly, we wanted to explore broadening the $T_{\max}$ window by controlling the Bi-2212 powder melting or wire architecture design. Here we report on studies of the performance variation with $T_{\max}$ for two production wires with a filling factor of about 20% and 85 × 18 filaments where filament size was varied by changing the wire diameter, a process which also shortens the distance between filaments. We found that wires with smaller filament diameter (9 to 11 $\mu$m) showed a peak $J_E$ at the low end of $T_{\max}$ and also a $J_E$ that was more sensitive to $T_{\max}$. A $J_E$ – $T_{\max}$ plot showed a plateau $J_E$(4.2 K, 5 T) of ∼1100 A/mm$^2$ between $T_{\max}$ of 886 and 894 °C for 1.0 and 1.2 mm wires, where $J_E$ is less sensitive to the wire diameter and $T_{\max}$. This $J_E$ plateau range is a preferred processing window for achieving high $J_E$ in coils.**

***Index Terms*—Superconductor, Bi-2212 wire, critical current density, superconducting magnet.**

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## I. Introduction

Bi-2212 round wire is a very promising conductor for very high field applications because of its very high critical current density ($J_C$) in fields of 20 – 40 T [1]–[7]. Bi-2212 wire is made by the powder-in-tube technique and Bi-2212 magnets are being developed using a wind and react technology [8]–[16]. To achieve high $J_C$ and high whole wire $J_E$, Bi-2212 powder must go through a partial melt heat treatment. Tight control of the heat treatment, especially of the maximum temperature ($T_{\max}$), is critical to the performance of Bi-2212 wire [17]–[23]. The effects of $T_{\max}$ on $J_C$ were studied previously with 1 bar [21], [22] and 50 bar heat treatments [22], [23]. Higher $T_{\max}$ in a standard heat treatment schedule causes Bi-2212 to be in the melt state for a longer time, which is called time in the melt $t_{melt}$, (defined as the time between when 2212 melts on heating (884 °C) and when Bi-2212 begins to form on cooling (872 °C)), and decreases $J_C$. [6], [21]–[23]. To achieve the highest $J_C$, we need to minimize $t_{melt}$ by heat treating in a narrow $T_{\max}$ window, but it is not easy to achieve a uniform temperature – time profile *T(t)* in a large coil of large thermal mass. Some believe that this narrow $T_{\max}$ window ($\Delta T_{\max}$) will be a constraint for large-scale applications like long particle accelerator magnets several meters long. An important issue to address is whether we can achieve a uniform $J_C$ in a wide range of $T_{\max}$ (or in an extended $t_{melt}$) by controlling the Bi-2212 powder melting or by proper design of the wire architecture. We previously studied the effect of filament size on $J_C$ in a Bi-2212 wire with 121 × 18 filaments made from Nexans powder and found that $J_C$ to be independent of filament size over the range from 9.3 $\mu$m to 13.8 $\mu$m, although wire with larger filaments did show higher *n* values [24], indicating more uniform current flow in the larger filament wire. Here we investigate how varying $T_{\max}$ affects wire performance for two recent 10 kg Bi-2212 billets made with Engi-Mat powder and an 85 × 18 filament architecture and wire diameters ranging from 0.8 to 1.2 mm.

## II. Experimental Details

As listed in Table I, two 10 kg Bi-2212 billets (billet numbers pmm170627 and pmm180410) were fabricated by Bruker OST from Engi-Mat powder lots LXB86 and LXB116 with a

TABLE I
SPECIFICATIONS OF THE BI-2212 WIRES USED IN THIS STUDY

| Wire ID | Wire dimeter (mm) | Filament configuration | Filling factor after densification | Average filament diameter after densification (µm) | Average minimum distance between filaments (µm) |
|---|---|---|---|---|---|
| pmm170627-10 | 1.0 | 85×18 | 0.195 | 11.1 | 3.80 |
| pmm170627-12 | 1.2 | 85×18 | 0.195 | 13.5 | 4.85 |
| pmm180410-08 | 0.8 | 85×18 | 0.201 | 9.0 | 3.17 |
| pmm180410-10 | 1.0 | 85×18 | 0.201 | 11.2 | 3.88 |
| pmm180410-12 | 1.2 | 85×18 | 0.201 | 13.5 | 4.73 |

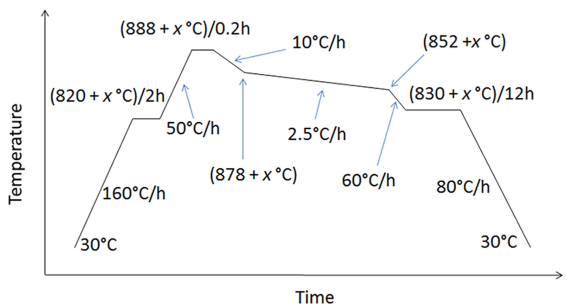


Fig. 1. Schematic heat treatment schedule for 50 bar OP-HT. The maximum temperature $T_{\max}$ ($= 888 + x$ °C) was varied from 883.5 °C to 896.5 °C. In a given heat treatment, $T_{\max}$ ($= 888 + x$ °C) was set and all other temperatures were adjusted by the same amount of $+ x$ °C, where $x$ varies from $-3.5$ °C to $+ 8.5$ °C. "h" stands for hour.

composition similar to Nexans 521 [6]. Wire pmm170627 was drawn to diameters of 1.2 and 1.0 mm (here labelled as pmm170627-12 and pmm170627-10, respectively), and pmm180410 to diameters of 1.2, 1.0, and 0.8 mm (pmm180410-12, pmm180410-10, and pmm180410-08, respectively) for studying the effects of wire diameter and filament size on the wire performance.

9 cm long samples that had both ends hermetically sealed were used for the studies. To analyze filament size, minimum distance between the filaments, and wire cross section area, we densified the powder using an overpressure pre-densification heat treatment at 830 °C for 12 hours [25]. All overpressure heat treatments were done at 50 bar total pressure with an oxygen partial pressure $pO_2$ of 1 bar. The complete overpressure heat treatment (OP-HT) schedule is shown in Fig. 1. It is similar to that reported previously [6]. In order to simulate coil heat treatments where temperature gradients may exist across the coil, all the temperature set points were adjusted by the same amount as $T_{\max}$ was changed, as indicated by the $+ x$ in the temperature – time plot of Fig. 1.

Transverse cross-sections of pre-densified and fully-processed wires were mounted in conductive epoxy and dry polished using a series of SiC papers with decreasing grit sizes. Final polishing was conducted with a suspension of 50 nm alumina in methanol using an automatic vibratory polisher (Buehler Vibromet). The cross section areas of the wire and Bi-2212 filaments after the powder densification were measured with an Olympus BX41M-LED light microscope. Microstructures were examined with a Zeiss 1540EsB scanning electron microscope (SEM). The SEM images were taken to highlight one of the six inner sub-elements which have a hexagonal shape.

Critical currents of fully-processed wires were measured using the four-probe transport method with a 1 $\mu$V/cm criterion at 4.2 K in a magnetic field of 5 T applied perpendicular to the wire axis. Our previous study showed that a power-law fit $J_C \propto B^{-\alpha}$, where $\alpha = 0.280$, works well at 4.2 K over the field range of at least 3 – 30 T [7], so that we can calculate $J_C$(4.2 K, B) based on the measured values of $J_C$(4.2 K, 5 T). The overall wire critical current density $J_E$ was calculated using the densified whole wire cross section. $J_C$ values reported here used the densified cross-section of the filaments after the 830 °C/12 h densification as the area. Magnetization measurements were performed in an Oxford vibrating sample magnetometer (VSM) at 20 K in a swept field from $-2$ T to 14 T. The VSM samples were cut to 5 mm in length and oriented with their axis orthogonal to the field direction.

## III. RESULTS

Fig. 2 shows SEM images of transverse cross sections of 1.0 and 1.2 mm wires of pmm170627 after the 830 °C powder densification overpressure heat treatment. Wire filling factors (defined as the ratio of Bi-2212 powder cross section area to the total wire cross section area), filament size and minimum distance between filaments measured on the cross sections of the densified wire samples are listed in Table I for both wires pmm170627 and pmm180410. The minimum distance between filaments is defined as the shortest length from one filament to its neighbor filaments. Drawing wire pmm170627 from 1.2 mm to 1.0 mm diameter reduced the average filament size from 13.5 to 11.1 $\mu$m (18% reduction), and also reduced the average minimum distance between the filaments from 4.85 $\mu$m to 3.80 $\mu$m (22% decrease).

Fig. 3 shows $J_E$(4.2 K, 5 T) as a function of the maximum heat treatment temperature $T_{\max}$ for 1.0 and 1.2 mm pmm170627 wires. Both the 1.0 and 1.2 mm wires showed a $J_E$(4.2 K, 5 T) plateau of about 1100 A/mm$^2$ over an 8 °C range in $T_{\max}$ from 886 °C to 984 °C. The 1.0 mm wire also showed a peak $J_E$(4.2 K, 5 T) value of 1400 A/mm$^2$ at the lower end of $T_{\max}$. This $J_E$ peak was not observed for the 1.2 mm pmm170627 wire.

Fig. 4 shows $J_E$(4.2 K, 5 T) and the $n$ value as a function of $T_{\max}$ for 0.8, 1.0 and 1.2 mm pmm180410 wires. The $J_E$ plateau region is about 1000 A/mm$^2$ over a 5 °C range in $T_{\max}$ from 885 °C to 890 °C. ($\Delta T_{\max}$ for the plateau for pmm180410 is 5 °C, compared to 8 °C for pmm170627). $J_E$ decreased with increasing $T_{\max}$ above 890 °C especially for the 0.8 mm wire, whose $J_E$ decreased faster than that of 1.0 and 1.2 mm wires. Fig. 4(b) shows that the $n$ value is larger for larger diameter wire, suggesting a more uniform supercurrent flow in the larger wire.

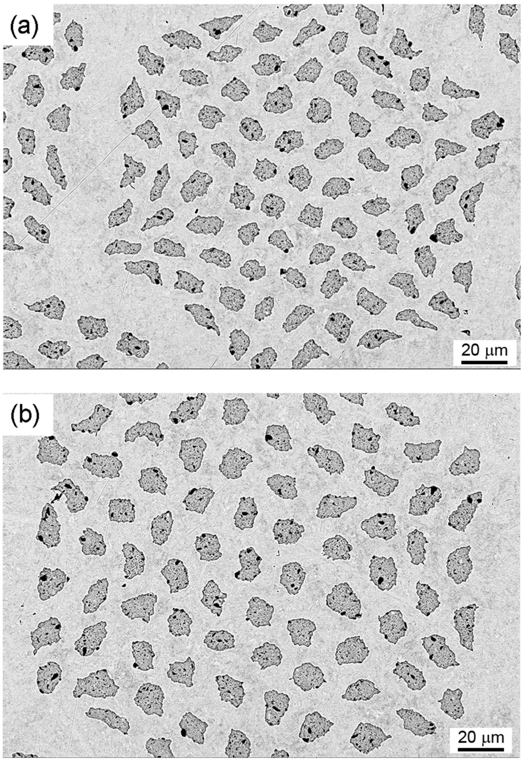


Fig. 2. SEM images of transverse cross sections of pmm170627 wires densified at 830 °C/12 h and 50 bar with diameters (a) 1.0 mm and (b) 1.2 mm. The black spots in the filaments are alkaline earth cuprate (Sr, Ca)$_{14}$Cu$_{24}$O$_x$ (14:24 AEC), or CuO.

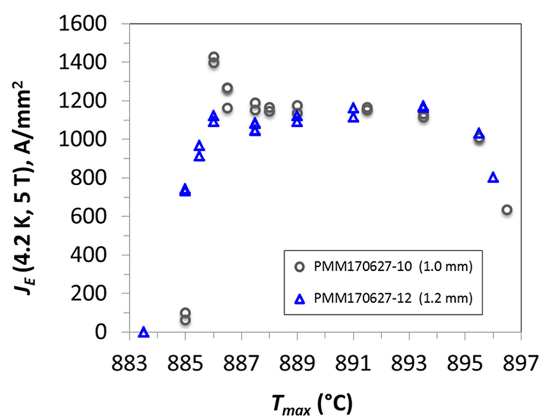


Fig. 3. $J_E$(4.2 K, 5 T) as a function of $T_{\max}$ for wire pmm170627 with diameters of 1.0 and 1.2 mm.

Fig. 5 compares SEM images of the fully heat treated 0.8, 1.0 and 1.2 mm pmm180410 wires with $T_{\max}$ of 885 °C and 896 °C. As shown in Fig. 4(a), wires heat treated with $T_{\max}$ of 896 °C have much lower $J_E$ than wires with $T_{\max}$ of 885 °C. Comparing the images in Fig. 2 and Fig. 5, the wires heat treated with $T_{\max}$ of 885 °C almost kept their original filament structure, whereas the filament structure significantly changed in the wires heat treated with $T_{\max}$ of 896 °C. The wires in Fig. 5 with $T_{\max}$ of 896 °C, showed clusters of bonded filaments. In the 0.8 mm wire (see Fig. 5(b)) the filaments had merged into a few large clusters of bonded filaments, and there were very few filaments that retained their original shape. In contrast, the 1.2 mm wire

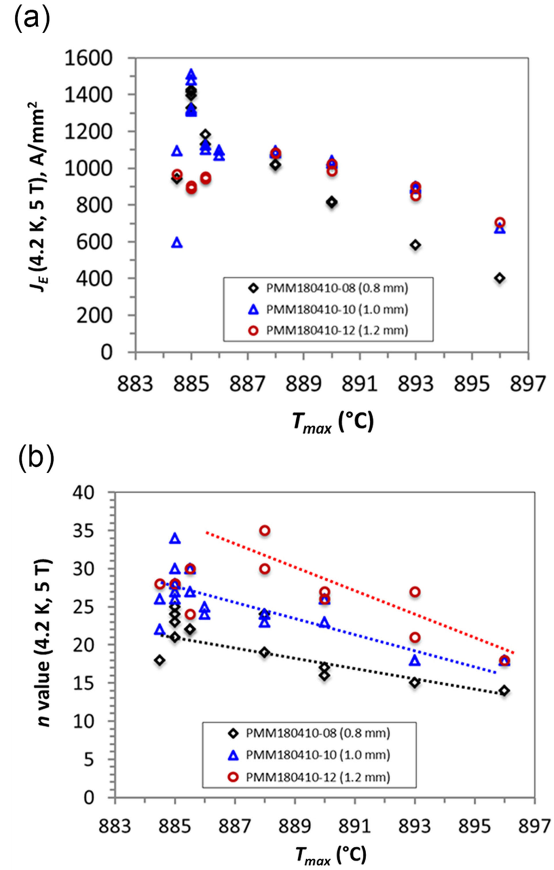


Fig. 4. (a) $J_E$(4.2 K, 5 T) – $T_{\max}$ and (b) $n$ value – $T_{\max}$ for wire pmm180410 with diameters of 0.8, 1.0 and 1.2 mm. The dashed lines in (b) are to guide the eye.

(see Fig. 5f) had fewer and smaller clusters of bonded filaments and more filaments retained their initial shape than in the 0.8 mm wire.

## IV. Discussion

As shown in Figs. 3 and 4, $J_E$ is quite sensitive to $T_{\max}$, especially for the smaller diameter 0.8 and 1.0 mm wires. Previous studies of 1 bar and 50 bar heat treatments suggested that $t_{melt}$ rather than $T_{\max}$ is the critical variable controlling $J_C$ because it is possible to have the same $t_{melt}$ for different $T_{\max}$ by changing the heating rate to the maximum temperature and the cooling rate from the maximum temperature of the heat treatment [21]–[23]. They found that by controlling $t_{melt}$ $J_C$ was relatively insensitive to $T_{\max}$ over a temperature range from 887 to 897 °C. For the heat treatments used in this study (shown in Fig. 1), $T_{\max}$ of 885 and 890 °C correspond to $t_{melt}$ of 2.4 and 4.5 hours, respectively. We replotted the data from Fig. 3 in Fig. 6 by converting $T_{\max}$ to $t_{melt}$. Except for the very sharp initial peak in $J_E$ at short $t_{melt}$ found only in the 0.8 and 1.0 mm wires, Fig. 6 shows that $J_E$ is fairly insensitive to $t_{melt}$ after this peak but then decays when $t_{melt}$ exceeds about 4 hours.

In this study, the Bi-2212 filament size was reduced by drawing the wires to smaller diameters but Table I shows that smaller diameter wires have shorter average inter-filament distances too. For the pmm180410 wire, Fig. 6 shows that $J_E$ of the 0.8 mm

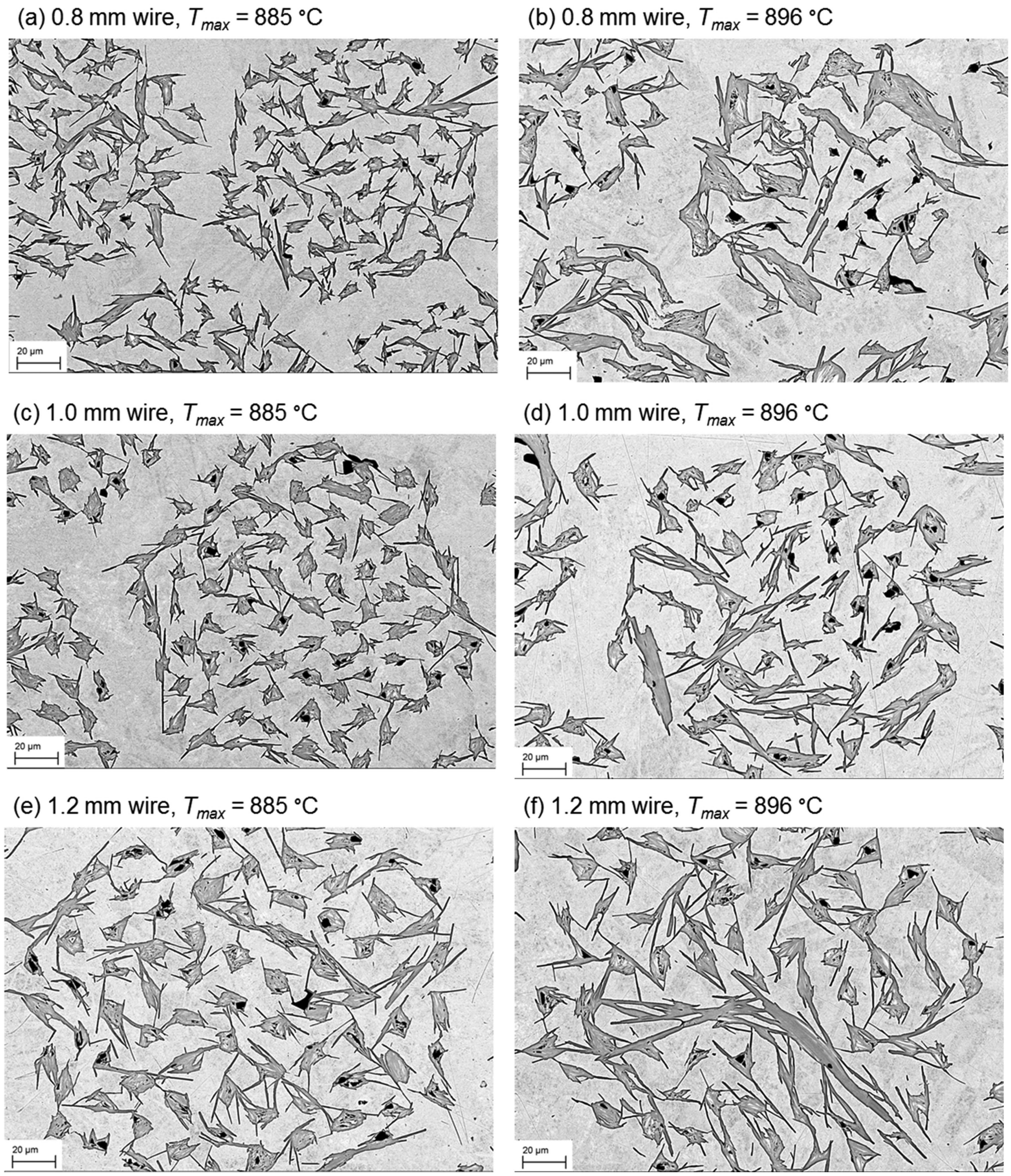


Fig. 5. Comparison of SEM images of fully heat treated pmm180410 wires with $T_{\max} = 885$ °C and 896 °C. (a) 0.8 mm with $T_{\max} = 885$ °C and $J_E$(4.2 K, 5 T) = 1426 A/mm$^2$; (b) 0.8 mm with $T_{\max} = 896$ °C and $J_E$(4.2 K, 5 T) = 402 A/mm$^2$; (c) 1.0 mm with $T_{\max} = 885$ °C and $J_E$(4.2 K, 5 T) = 1512 A/mm$^2$, (d) 1.0 mm with $T_{\max} = 896$ °C and $J_E$(4.2 K, 5 T) = 672 A/mm$^2$; (e) 1.2 mm with $T_{\max} = 885$ °C and $J_E$(4.2 K, 5 T) = 902 A/mm$^2$, and (f) 1.2 mm with $T_{\max} = 896$ °C and $J_E$(4.2 K, 5 T) = 706 A/mm$^2$. The large black spots in the SEM images are the alkaline earth cuprate $(Sr, Ca)_{14}Cu_{24}O_x$ (14:24 AEC), or CuO.

wire decreases more rapidly than the 1.0 mm and 1.2 mm wires when $t_{melt}$ is longer than 4.5 hours. Shen *et al.* [21] suggested that (1) molten Bi-2212 can dissolve Ag grain boundaries, penetrating from one filament to another when the distance is short, a process that allows filaments to merge (bond) together altering the filament architecture; (2) and possibly Cu loss from 2212 filaments. As shown in Figs. 5(b), 5(d), and 5f, filament bonding is quite common for wires heat treated with $T_{\max}$ of 896 °C (corresponding to $t_{melt}$ of 7.1 hours), especially in the 0.8 mm wire (Fig. 5(b)), where filaments completely lost their original shape. As discussed previously [26], we believe this filament bonding that occurs during the long $t_{melt}$, degrades $J_E$, but the larger diameter wires (1.0 and 1.2 mm) performed better than 0.8 mm wire when $t_{melt}$ is longer than about 4 hours. To better understand the influence of $t_{melt}$, we fast-cooled Bi-2212 from the melt state. Samples of wire pmm180410 were heated to 888 °C, held at 888 °C for 4 hours, and then furnace cooled. Fig. 7 shows SEM images of the fast-from-the-melt cooled wires. Fig. 7 shows that 27 of 85 filaments merged in the 0.8 mm wire while only 3 of 85 filaments merged in the 1.2 mm wire. The much more frequent filament merging in the 0.8 mm wire correlates directly to the much shorter distance between filaments that makes the dissolution/diffusion/precipitation process that merges filaments more prevalent.

Fig. 8 shows the Kramer function as a function of applied field for fully heat treated 1.0 mm pmm180410 wire with $T_{\max} = 885$, 893 and 896 °C, corresponding to $t_{melt}$ of 2.4, 5.9, and 7.1 hours, respectively. Although the $J_E$ value varies by a factor of two,

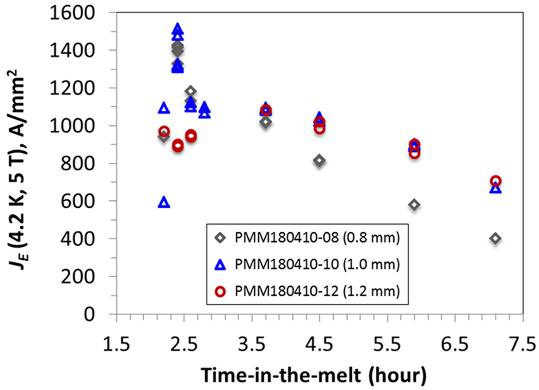


Fig. 6. $J_E$(4.2 K, 5 T) as a function of $t_{melt}$ for 0.8, 1.0 and 1.2 mm wires of pmm180410.

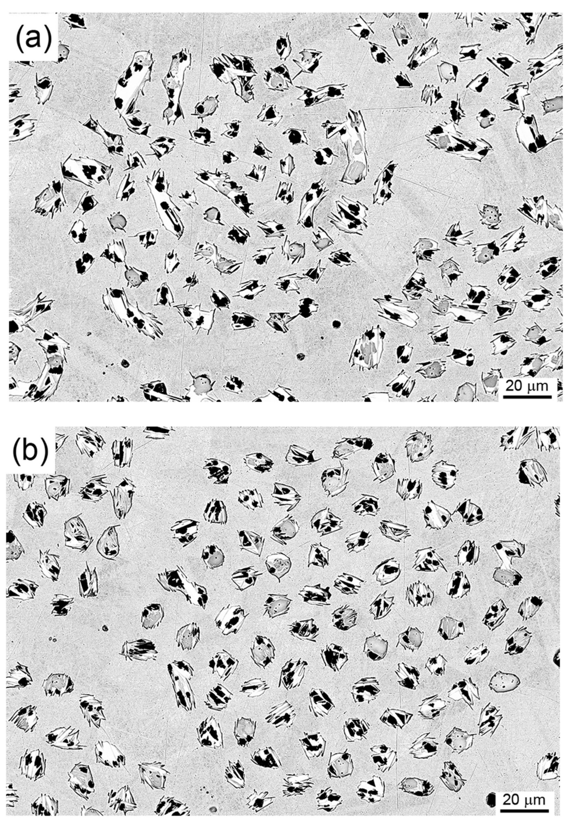


Fig. 7. SEM images of (a) 0.8 mm and (b) 1.2 mm pmm180410 wires after holding at 888 °C for 4 hours and fast-cooled.

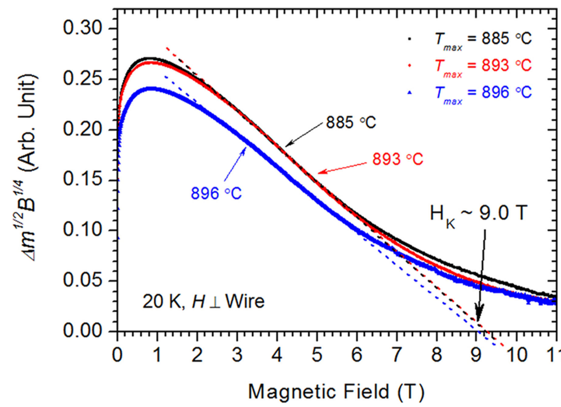


Fig. 8. Kramer function $\Delta m^{1/2}B^{1/4}$ measured at 20 K as a function of applied field for fully heat treated 1.0 mm pmm180410 wire with $T_{\max} = 885$, 893 and 896 °C . $J_E$(4.2 K, 5 T) of these samples are 1480, 901 and 672 A/mm$^2$, respectively. The dashed lines are drawn by fitting the linear sections of the Kramer plots, and the irreversibility ($H_K$) is defined by the linear extrapolations to the field axis.

the $H_K$(20 K) values for all the three samples are about 9 T, indicating that extending $t_{melt}$ to 7 hours does not reduce the flux pinning of the wire and the decrease in $J_C$ with increasing $t_{melt}$ is caused by reduced connectivity from filament merging. This is consistent with the reduced $n$ values shown in Fig. 4(b).

Fig. 3 shows a $J_E$(4.2 K, 5 T) plateau of $\sim$1100 A/mm$^2$ for wire pmm170627 between 886 and 894 °C, where $J_E$ is independent of $T_{\max}$ and wire diameter, which is consistent with a previous study on a wire with 121 $\times$ 18 filaments and diameters between 1.0 and 1.5 mm [24]. Having a $J_E$ plateau range is a preferred processing window for coil heat treatment.

Both Figs. 3 and 4(a) show a $J_E$ peak at the lower end of $T_{\max}$ for 0.8 and 1.0 mm wires. The $J_E$ peak occurred over a narrow $T_{\max}$ range. Matsumoto *et al.* observed a similar $J_C$ peak in their 1 bar heat treatment studies in a wire with 127 $\times$ 7 filaments and diameter of 1.0 mm [27]. These very high $J_C$ values at the peak remain interesting as a target for yet further improvement of $J_C$ and for better understanding the properties of Bi-2212 wire, even if, for now, such a short $t_{melt}$ seems infeasible in a large Bi-2212 coil. Luckily, the $J_E$ values found in the plateau region of the $J_E$ – $t_{melt}$ plot is still interesting for high-field magnets. It seems the $J_E$ peak is related to the small filament size in the smaller wires since this peak is absent for 1.2 mm wire. Previous work by Kametani *et al.* [28] and Oloye *et al.* [26] strongly suggested that the geometrical confinement of the narrow filament space is vital in order to develop the best quasi-biaxial texture of Bi-2212 grains because the narrow filament cavity is favored for better Bi-2212 grain alignment. The melting of Bi-2212 powder during the heat treatment is not instantaneous, and it involves Ag dissolving in the melt [29]. Quench studies have shown that the liquid contains about 4 at.% Ag [30]. Smaller filament size could mean a shorter time needed for Ag to dissolve in the melt and for Bi-2212 powder to melt because of the reduced distance from the Ag-filament interface to the filament center, and more uniform melting at the lower end of $T_{\max}$ range (shorter $t_{melt}$) in Figs. 3 and 4. We plan further quench studies to better understand this process. The main characteristic of the microstructure in the peak $J_E$ samples shown in Figs. 5(a) and (c) is that they show the least filament merging, which is consistent with our previous observation that reducing filament merging is beneficial for strong texture formation because having discrete filament cross sections most effectively aligns the Bi-2212 grain growth direction along the filament direction. As we discussed above, $J_E$ of the smallest filament wire ($\sim$9 $\mu$m for 0.8 mm wire) is more sensitive to $t_{melt}$ because its shorter distance between the filaments makes filament bonding easier, so that the peak $J_E$ could be a result of the interaction between the small filament size and the short distance between the filaments.

## V. Conclusion

We studied two 85 × 18 filament Bi-2212 wires with 0.8, 1.0 and 1.2 mm diameters. We varied the maximum heat treatment temperature $T_{\max}$ during overpressure heat treatment. We found that the 0.8 and 1.0 mm wires with filament sizes of 9 to 11 $\mu$m showed a peak $J_E$ at the lower end of $T_{\max}$ with a $J_E$ that was more sensitive to $T_{\max}$ than the larger wire. The $J_E$ peak was absent for 1.2 mm wires with a filament size of $\sim$13.5 $\mu$m. Samples with the highest $J_E$ values showed the least filament merging, further confirming that reducing filament merging is effective for forming strong Bi-2212 texture and high $J_E$. $J_E$ – $T_{\max}$ plots of wire pmm170627 showed a plateau $J_E$(4.2 K, 5 T) of $\sim$1100 A/mm$^2$ between $T_{\max}$ values of 886 and 894 °C, where $J_E$ is less sensitive to the wire diameter and $T_{\max}$. The $J_E$ plateau shown in Fig. 4 for 1.0 and 1.2 mm pmm180410 is about 5 °C, which is narrower than that for pmm170627. We are not sure what causes this performance difference, and it could be related to the precursor powder difference or the filament quality.

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