Performance and Microstructure Variation with Maximum Heat Treatment Temperature for Recent Bi-2212 Round Wires

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***Abstract*—Bi-2212 is the only high temperature superconducting wire with the round geometry. It is multifilamentary, available in a wide range of fine filaments and twisted filament architectures and can be made into Rutherford and other cables. The properties of Bi-2212 conductors depend on powder quality, conductor fabrication and heat treatment. The heat treatment is still complex but much better understood, particularly the vital parameters of the maximum heat treatment temperature ($T_{\max}$), time-in-the-melt (t_{melt}) and the cooling rate as Bi-2212 reforms on cooling. Here we report on the performance and microstructure variation with heat treatments for more than a dozen wires made with powders produced by Engi-Mat in recent years. Wire architectures include 37 × 18, 55 × 18 and 85x18 and wire diameters range from 0.8 to 1.0 mm. $T_{\max}$ was varied between 884 and 897 °C. Wires with smaller filament diameter showed a peak J_E at the low end of $T_{\max}$ and also a J_E that was more sensitive to $T_{\max}$. J_E ($T_{\max}$) plots for all recent wires show a plateau between $T_{\max}$ of 886 and 894 °C, where J_E (4.2 K, 5 T) is 1100–1400 A/mm^2. Some wires with filament size of 13–15 μm showed a 10 °C heat treatment window ($\Delta T_{\max}$) with a plateau J_E (4.2 K, 5 T) of about 1100 A/mm^2.**

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

I. Introduction

BI-2212 is a very promising high temperature superconductor for very high field applications because of its very high critical current density (J_C) in magnetic fields over 30 T [1], [2], [3], [4], [5], [6]. Bi-2212 round wire is routinely produced on a 10 kg scale by Bruker OST, delivered in kilometer piece length, and is being used in the development of coils for accelerator and research magnets targeting fields greater than 20 T [7], [8], [9], [10], [11], [12], [13]. As reported previously [14], [15], [16], [17], J_C of Bi-2212 wire varies strongly with powder, wire architecture, fabrication, and heat treatment conditions, particularly the maximum heat treatment temperature ($T_{\max}$). Higher $T_{\max}$ in the 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 884 °C when Bi-2212 melts on heating and 872 °C when Bi-2212 begins to form on cooling [14].

Bi-2212 wire made by the powder-in-tube method is delivered in a green, unreacted state. Wire engineering critical current density (J_E) is still the major performance parameter. In order to evaluate the performance of Bi-2212 wires, we need to perform overpressure heat treatment (OPHT) for wire sections taken from the ends of the billet. Here we study more than a dozen recent Bi-2212 billets made by Bruker OST with powders produced by Engi-Mat by applying 50 bar OPHT with $T_{\max}$ varying between 884 and 897 °C. The wire architectures include 55 × 18, 85 × 18 and 37 × 18 and wire diameters vary from 0.8 to 1.0 mm.

II. Experimental Details

As listed in Table I, thirteen 2 kg and 10 kg Bi-2212 billets were fabricated by Bruker OST between 2017 and 2022 with Engi-Mat powder with a composition similar to Nexans 521 [4]. Billets 10 and 13 were drawn into three diameters, here labeled as pmm200222-08 (0.8 mm), pmm200222-09 (0.9 mm)

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TABLE I
SPECIFICATIONS OF THE BI-2212 WIRES USED IN THIS STUDY. THE FIRST TWO DIGITS IN THE WIRE ID DESIGNATE THE YEAR THE WIRE WAS FABRICATED

Billet number	Wire ID	Manufacture year	Billet size (kg)	Filament configuration	Wire dimeter (mm)	Filling factor after densification	Average equivalent filament diameter after densification[a] (μm)
1	pmm170123	2017	2	55x18	0.8	0.199	12.2
2	pmm170627	2017	10	85x18	1.0	0.195	11.1
3	pmm170725	2017	2	55x18	0.8	0.236	12.1
4	pmm180207	2018	10	55x18	0.8	0.197	11.1
5	pmm180410	2018	10	85x18	1.0	0.201	11.2
6	pmm180627	2018	10	55x18	1.0	0.217	14.5
7	pmm190118	2019	10	55x18	0.8	0.210	11.4
8	pmm190425	2019	10	85x18	1.0	0.220	11.8
9	pmm191004	2019	10	55x18	0.8	0.219	12.2
10	pmm200222-08	2020	2	55x18	0.8	0.238	12.0
10	pmm200222-09	2020	2	55x18	0.9	0.238	13.4
10	pmm200222-10	2020	2	55x18	1.0	0.238	15.0
11	pmm211105	2021	10	55x18	0.8	0.180	10.4
12	pmm220329	2022	10	55x18	0.8	0.200	11.0
13	pmm220802-08	2022	2	37x18	0.8	0.196	13.3
13	pmm220802-09	2022	2	37x18	0.9	0.196	14.9
13	pmm220802-10	2022	2	37x18	1.0	0196	16.7

[a]Average equivalent filament diameter calculated by measuring the filament area and calculating the diameter of a circle whose area equals the measured area.

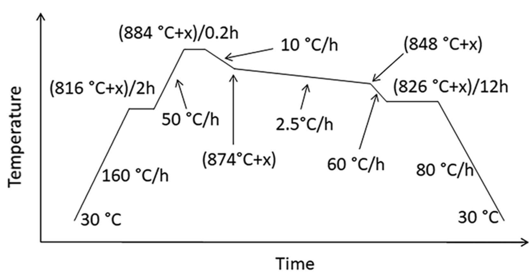

Fig. 1. Schematic heat treatment schedule for 50 bar OPHT. The maximum temperature $T_{\max}$ (= 884 + x °C) was varied from 884 °C to 897 °C. In a given heat treatment, $T_{\max}$ (= 884 + x °C) was set and all other temperatures were adjusted by the same amount of x °C, where x varies from 0.5 °C to 13 °C [17]. "h" stands for hour.

and pmm200222-10 (1.0 mm), and pmm220802-08 (0.8 mm), pmm220802-09 (0.9 mm) and pmm220802-10 (1.0 mm), respectively, for studying the effects of wire diameter and filament size on the wire performance. All of the 2 kg billets were made for Engi-Mat DOE SBIR research projects, while the 10 kg billets were for Bruker OST production wires. Wire pmm170123 was used by LBNL for a racetrack coil [10] and pmm170725 for a CCT coil [12], [13].

9 cm long samples that had both ends hermetically sealed were used for the studies. To analyze filament size, wire cross section area and filament filling factor (defined as the ratio of filament cross section area to total wire cross section area), we densified the wire samples using an overpressure pre-densification heat treatment at 830 °C for 12 hours [18]. All overpressure heat treatments were done at 50 bar total pressure with an oxygen partial pressure pO_2 of 1 bar (1 bar O_2 + 49 bar Ar). The full overpressure heat treatment (OPHT) schedule is shown in Fig. 1, which is similar to the one reported previously [17]. 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.

Critical currents of fully heat-treated wires were measured using the four-probe transport method with a 1 μV/cm criterion at 4.2 K in a magnetic field of 5 T applied perpendicular to the wire axis. The overall wire critical current density J_E was calculated using the densified whole wire cross section. Microstructures were examined with a Zeiss 1540EsB scanning electron microscope (SEM). Because J_E (H, 4.2 K) has a rather similar power law, vortex pinning behavior for many wires with nominal 521 composition, evaluation only at 5 T is convenient and representative of different Bi-2212 wires [5].

III. RESULTS

Fig. 2 shows optical images of transverse cross sections of three wires with three different filament configurations after the 830 °C pre-densification overpressure heat treatment, pmm170123 (55x18, 0.8 mm), pmm180410 (85 × 18, 1.0 mm) and pmm220802-08 (37 × 18, 0.8 mm). Wire filling factors and average filament size measured on the cross sections of the densified wire samples are listed in Table I for all the wires. The filling factor ranges from 0.180 for pmm211105 to 0.238 for pmm200222. The equivalent round filament diameter based on pre-densified wires ranges from 10.4 μm to 16.7 μm.

Fig. 3 shows J_E (4.2 K, 5 T) as a function of the maximum heat treatment temperature $T_{\max}$ for all thirteen billets. Wire pmm170123 showed the highest peak J_E of 1900 A/mm^2 at the low end of $T_{\max}$ and also the highest plateau J_E (4.2 K, 5 T) of 1400 A/mm^2 (see Fig. 3(a)). Other wires had plateau J_E (4.2 K, 5 T) in the range of 1000 to 1200 A/mm^2 and lower

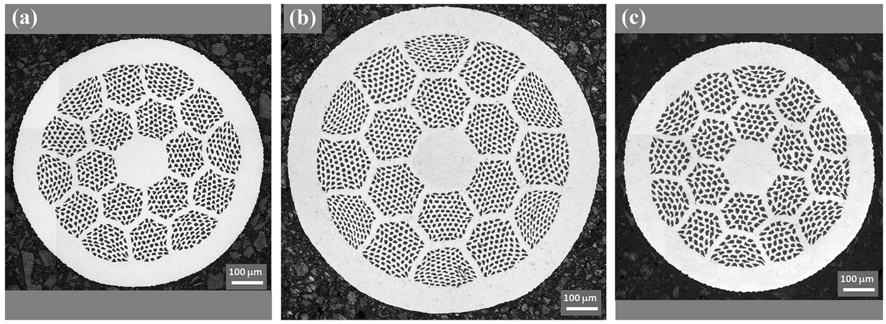

Fig. 2. Optical images of transverse cross-sections of three wires with filament configurations of (a) 55 × 18 (pmm170123), (b) 85 × 18 (pmm180410) and (c) 37 × 18 (pmm220802-08) after the 830 °C pre-densification overpressure heat treatment.

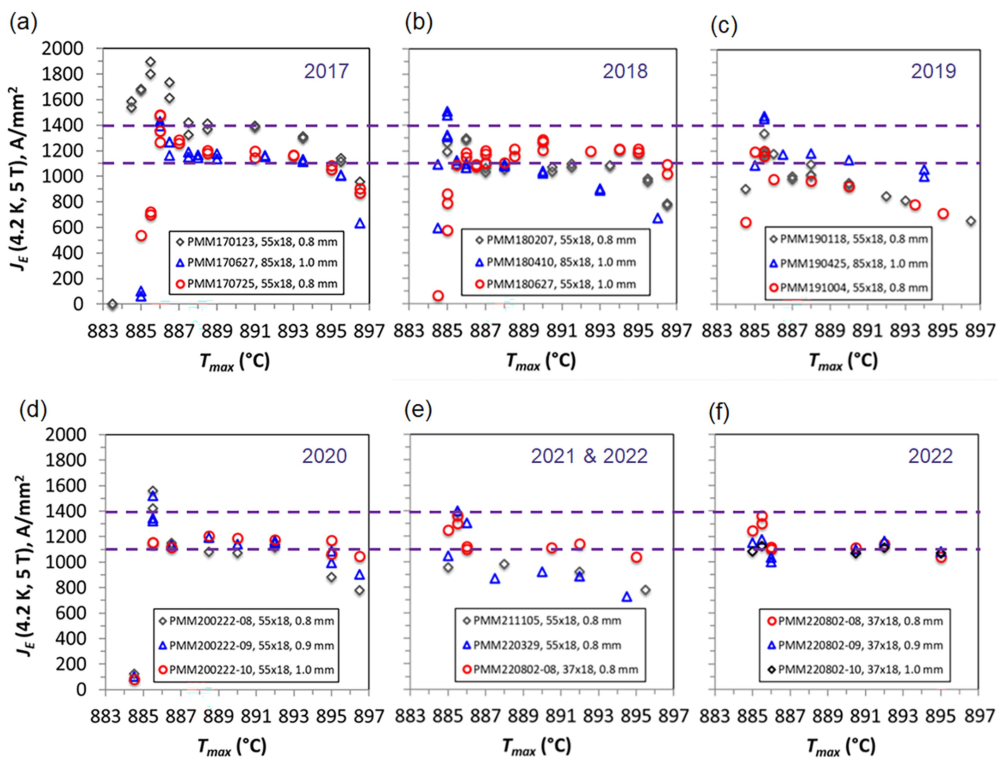

Fig. 3. J_E (4.2 K, 5 T) as a function of $T_{\max}$ for wires made from 2017 to 2022. (a) pmm170123, pmm170627, and pmm170725; (b) pmm180207, pmm180410, and pmm180627; (c) pmm190118, pmm190425, and pmm191004; (d) pmm200222-08, pmm200222-09, and pmm200222-10; (e) pmm211105, pmm220329, and pmm220802-08; (f) pmm220802-08, pmm220802-09, and pmm220802-10. The dashed lines are to guide the eye. J_E data for wires pmm170627 and pmm180410 are extracted from [17].

peak J_E values than pmm170123. Four 10 kg production billets, pmm190118, pmm191004, pmm211105 and pmm220329, showed lower J_E (4.2 K, 5 T) values (lower than 1200 A/mm^2) with increasing $T_{\max}$ than the three 2 kg research billets, pmm170123, pmm170725 and pmm200222-08, even though they have the same diameter of 0.8 mm and the same 55x18 architecture.

Fig. 4 compares SEM images of the fully heat-treated champion wire pmm170123 with $T_{\max}$ of 885.5 °C, 891 °C and 895.5 °C. Increasing $T_{\max}$ by 10 °C resulted in significant filament merging and 40% decrease in J_E. As shown in Fig. 4(a), the highest J_E sample had more uniform filaments and fewer merged filaments.

IV. Discussion

As shown in Fig. 3, all the wires showed a J_E (4.2 K, 5 T) range above 1100 A/mm^2, confirming the performance of the recent wires is reproducible. For direct comparison, J_E data of 0.8 mm wire pmm220802-08 was plotted in both Fig. 3(e) to compare the effect of wire architecture and in Fig. 3(f) to compare the effect of wire diameter. Wire pmm220802-08 with 37 × 18 architecture showed a higher and wider plateau J_E than the two 55 × 18 wires, pmm211105 and pmm220329. One of the differences between these three wires is that wire pmm220802-08 has a larger filament size (13.3 μm) because of reduced filament numbers compared to the two wires with filament sizes of 10.4 μm and 11.0 μm. This indicates that

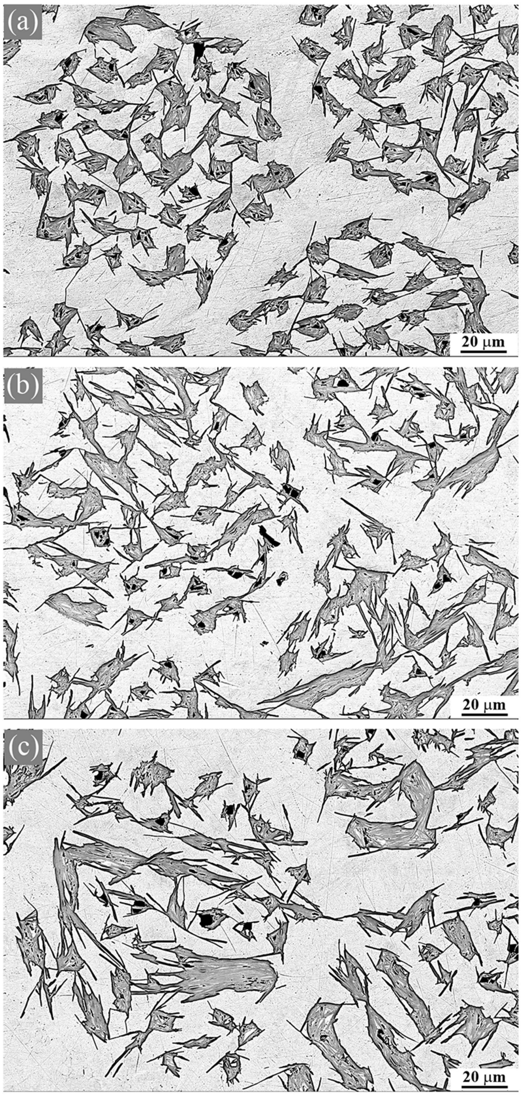

Fig. 4. Comparison of SEM images of fully heat treated pmm170123 wires with different $T_{\max}$. (a) $T_{\max} = 885.5$ °C and J_E (4.2 K, 5 T) = 1900 A/mm^2; (b) $T_{\max} = 891$ °C and J_E (4.2 K, 5 T) = 1400 A/mm^2; (c) $T_{\max} = 895.5$ °C and J_E (4.2 K, 5 T) = 1120 A/mm^2. The large black spots in the SEM images are the alkaline earth cuprate (Sr, Ca)$_{14}$Cu$_{24}$O$_x$ (14:24 AEC), or CuO.

the wider plateau J_E could result from the larger filament size (13.3 μm). Recent work by Hossain et al. [19] and by Barua et al. [20] shows that longitudinal uniformity of the filaments plays an important role in J_C. The lower J_E values in the 10 kg wires pmm211105 and pmm220329 could be caused by their filament nonuniformity, which is being analyzed.

For a given wire architecture, one can increase the filament size by stopping wire drawing at a larger diameter or increasing the filament filling factor. 55 × 18 architecture was originally designed for 0.8 mm wire used for fabricating Rutherford cables [10]. As seen in Table I, the filament size is about 15 μm for 1.0 mm wires pmm180627 and pmm200222-10. In Fig. 5, we re-plotted J_E data from Fig. 3 for wires pmm180627, pmm200222-10, and pmm220802-09. Their $J_E - T_{\max}$ plots have a J_E plateau from 885 to 895 °C without the sharp J_E peak at the low end of $T_{\max}$, further confirming that a wider J_E plateau is related to larger (∼15 μm) filament size. A wide processing window

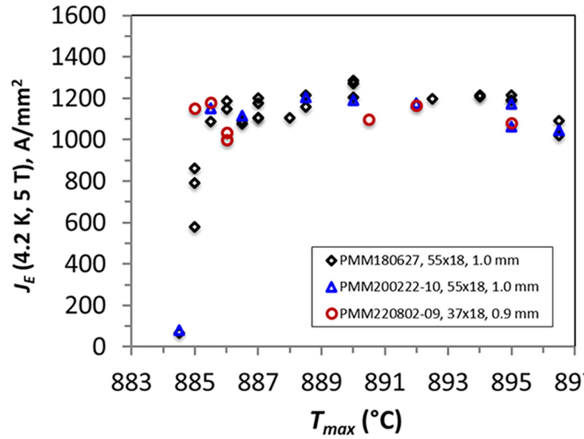

Fig. 5. J_E (4.2 K, 5 T) as a function of $T_{\max}$ for wires with very similar filament size, pmm180627 (55 × 18, 1.0 mm) with filament size of 14.5 μm, pmm200222-10 (55 × 18, 1.0 mm) with filament size of 15.0 μm, and pmm220802-09 (37 × 18, 0.9 mm) with filament size of 14.9 μm.

$\Delta T_{\max}$ (a large range of $T_{\max}$ that yields a nearly constant J_E) is desired to process large coils that have large thermal mass and significant thermal time constants that may make precise control over the desired temperature – time profiles uncertain. Thus larger filament diameters within the 10–17 μm range studied may have real benefits for magnet use.

V. Conclusion

We studied thirteen recent Bi-2212 billets by varying the maximum heat treatment temperature $T_{\max}$ during overpressure heat treatment. All the wires showed J_E (4.2 K, 5 T) values above 1100 A/mm^2, but their plateau J_E values were quite different. Wire pmm170123 showed the highest peak J_E of 1900 A/mm^2 and also the highest plateau J_E (4.2 K, 5 T) of 1400 A/mm^2. We found that wires with filament size of 10 to 12 μm showed a great sensitivity to their melting conditions. They had a distinctive peak in J_E for smaller values of $T_{\max}$ and also showed a narrow J_E plateau, whereas wires with filament size >13 μm did not show this marked peak J_E but had a wider J_E plateau of about 10 °C. Indeed data taken from 14–15 μm filament wires showed that the best $T_{\max}$ is at about 890 °C. This offers good protection against both over and under temperature within the coil. As reported previously [17], the appearance of the sharp peak in J_E at minimum $T_{\max}$ is directly related to the small filament size. The microstructures of fully heat-treated wires show that the peak J_E at the lower end of $T_{\max}$ in the $J_E - T_{\max}$ plot has more uniform filaments with fewer inter-filament connections. Although small twisted-filaments are preferred for low AC losses, wires with filament size up to 15 μm would have a wide processing window $\Delta T_{\max}$, which makes it easier to control the heat treatment of large coils.

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REFERENCES

[1] H. Miao, K. R. Marken, M. Meinesz, B. Czabaj, and S. Hong, "Development of round multifilament Bi-2212/Ag wires for high field magnet applications," *IEEE Trans. Appl. Supercond.*, vol. 15, no. 2, pp. 2554–2557, Jun. 2005.

[2] D. C. Larbalestier et al., "Isotropic round-wire multifilament cuprate superconductor for generation of magnetic fields above 30 T," *Nature Mater.*, vol. 13, pp. 375–381, 2014.

[3] Y. Huang, H. Miao, S. Hong, and J. A. Parrell, "Bi-2212 round wire development for high field applications," *IEEE Trans. Appl. Supercond.*, vol. 24, no. 3, Jun. 2014, Art. no. 6400205.

[4] J. Jiang et al., "High-performance Bi-2212 round wires made with recent powders," *IEEE Trans. Appl. Supercond.*, vol. 29, no. 5, Aug. 2019, Art. no. 6400405.

[5] M. D. Brown et al., "Prediction of the J_C(B) behavior of Bi-2212 wires at high field," *IEEE Trans. Appl. Supercond.*, vol. 29, no. 5, Aug. 2019, Art. no. 6400504.

[6] A. Angrisani Armenio et al., "Investigation of transport mechanisms induced by filament-coupling bridges-network in Bi-2212 wires," *Supercond. Sci. Technol.*, vol. 35, no. 3, 2022, Art. no. 035002.

[7] H. W. Weijers, U. P. Trociewitz, K. Marken, M. Meinesz, H. Miao, and J. Schwartz, "The generation of 25.05 T using a 5.11 T $Bi_2Sr_2CaCu_2O_x$ superconducting insert magnet," *Supercond. Sci. Technol.*, vol. 17, pp. 636–644, 2004.

[8] K. Zhang et al., "Tripled critical current in racetrack coils made of Bi-2212 Rutherford cables with overpressure processing and leakage control," *Supercond. Sci. Technol.*, vol. 31, no. 10, 2018, Art. no. 105009.

[9] L. G. Fajardo, L. Brouwer, S. Caspi, S. Gourlay, S. Prestemon, and T. Shen, "Designs and prospects of Bi-2212 canted-cosine-theta magnets to increase the magnetic field of accelerator dipoles beyond 15 T," *IEEE Trans. Appl. Supercond.*, vol. 28, no. 4, Jun. 2018, Art. no. 4008305.

[10] T. Shen et al., "Stable, predictable operation of racetrack coils made of high-temperature superconducting Bi-2212 Rutherford cable at the very high wire current density of more than 1000 A/mm2," *Sci. Rep.*, vol. 9, 2019, Art. no. 10170.

[11] T. Shen and L. Garcia Fajardo, "Superconducting accelerator magnets based on high-temperature superconducting Bi-2212 round wires," *Instruments*, vol. 4, no. 2, Jun. 2019, Art. no. 17.

[12] L. G. Fajardo et al., "First demonstration of high current canted-cosine-theta coils with Bi-2212 Rutherford cables," *Supercond. Sci. Technol.*., vol. 34, no. 2, 2021, Art. no. 024001.

[13] T. Shen et al., "Design, fabrication, and characterization of a high field high temperature superconducting Bi-2212 accelerator dipole magnet," *Phys. Rev. Accelerators Beams*, vol. 25, 2022, Art no. 122401.

[14] T. Shen, J. Jiang, F. Kametani, U. P. Trociewitz, D. C. Larbalestier, and E. E. Hellstrom, "Heat treatment control of Ag–$Bi_2Sr_2CaCu_2O_x$ multifilamentary round wire: Investigation of time in the melt," *Supercond. Sci. Technol.*, vol. 24, no. 11, 2011, Art. no. 115009.

[15] T. Shen, P. Li, and L. Ye, "Heat treatment control of Bi-2212 coils: I. Unravelling the complex dependence of the critical current density of Bi-2212 wires on heat treatment," *Cryogenics*, vol. 89, pp. 95–101, Jan. 2018.

[16] J. Jiang et al., "Effects of filament size on critical current density in overpressure processed Bi-2212 round wire," *IEEE Trans. Appl. Supercond.*, vol. 27, no. 4, 2017, Art. no. 6400104.

[17] J. Jiang et al., "Effects of wire diameter and filament size on the processing window of Bi-2212 round wire," *IEEE Trans. Appl. Supercond.*, vol. 31, no. 5, 2021, Art. no. 6400206.

[18] M. R. Matras, J. Jiang, D. C. Larbalestier, and E. E. Hellstrom, "Understanding the densification process of $Bi_2Sr_2CaCu_2O_x$ round wires with overpressure processing and its effect on critical current density," *Supercond. Sci. Technol.*, vol. 29, no. 10, 2016, Art. no. 105005.

[19] S. I. Hossain, "Understanding the role of wire architecture in determining the critic current performance of Bi-2212 round wire," Ph.D. thesis, The Florida State Univ., FL, USA, 2022.

[20] S. Barua et al., "Critical current distributions of recent Bi-2212 round wires," *IEEE Trans. Appl. Supercond.*, vol. 31, no. 5, Jan. 2021, Art. no. 6400406.