The Effect of Filament Diameter on J_e in High Filament Count Bi2212/Ag Round Wire

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***Abstract*—In this study, we investigated the effect of filament size on J_e for high filament count Bi2212 round wire conductors partial melt processed with T_{max} between 882 and 890°C in pure oxygen. The filament size that produced maximum J_e was between 12 and 15 μm depending on conductor design and T_{max}. When the filament size was below 12 μm the filaments were unstable during partial melt processing leading to a break-up in the structure of the filament array and low J_e. This effect is attributed the formation of interconnects between filaments which leads to coarsening driven by a reduction in interfacial surface energy.**

***Index Terms*—Bi2212/Ag round wire, high-temperature superconductors, superconducting filaments and wires.**

I. Introduction

FILAMENT size is a major design consideration in the manufacture of high current capacity Bi2212 round multifilamentary wire. Several studies have reported on the effects of filament size on critical current capacity including our previous work on single restack conductor designs [1]–[7]. The general trend reported in these studies is for the critical current to increase as the wire diameter and hence filament diameter decreases. However, there is no definitive filament size that maximizes conductor performance, and some of the results regarding optimum filament size are contradictory. It appears that factors such as heat treatment, filament count, Ag/SC ratio, and wire diameter may affect the optimum filament diameter.

Motowidlo *et al.* [1]–[3] investigated filament size effects on J_c in 37 and 300 filament single restack designs. They also produced several double restack designs. One was a 37 × 7 configuration consisting of 259 filaments using seven of the 37 filament wires as subelements, while another was a 61 × 7 arrangement. The wires were drawn to sizes ranging 0.37 mm to 1.02 mm. All the wires had different ceramic fill factors: 35–40% for the 37 filament wire, 25% for the 300 filament wire, 25–35% for the 259 filament wire, and 17% for the 427 filament wire. The 37 filament wire was drawn to produce wire with filament diameters of 30, 60 and 100 μm. The 259 filament wire was used to produce wires with 11 and 16 μm filaments. At 0.81 mm the 300 filaments wire had an average filament diameter of 20 μm, while it was 15 μm in the 427 filament double restack wire. The wire were given a modified partial melt solidification treatment in pure oxygen atmosphere using a step cooling sequence from a peak temperature of 885°C. An inverse linear relationship was found between filament diameter and J_c over the entire range of filament sizes investigated, and all the conductors showed similar behavior. The highest J_c was achieved with the 37 × 7 double restack with a filament diameter of 11 μm. These results were attributed the effect of increased Ag/ceramic interface in promoting greater c-axis alignment as filament size decreased with wire diameter.

Hasegawa *et al.* [4], made double restack conductors of 61 × 7, 91 × 7, 127 × 7 163 × 7 and 37 × 19 configurations. The ceramic fill for all the wires was nearly identical at approximately 25%. The wires were drawn to sizes ranging from 0.8 to 1.3 mm. These conductors had filaments sizes at a 1 mm wire diameter of 20, 14, 12, 10 and 14 μm respectively. At the largest wire size of 1.3 mm, the 163 × 7 wire had the highest J_c. As wire diameter was reduced down to 0.9 mm, the J_c increased for all the wires. However, below 0.9 mm, the J_c of all the wires decreased except for the two wires, 61 × 7 and 91 × 7, which had the largest filaments. At 0.8 mm the 91 × 7 wire produced the highest J_c, and the largest drop in J_c occurred with the 163 × 7 wire, which had the smallest filaments. These effects were attributed to differences in the aspect ratio of the filaments between the various wires and the quality of the filaments. It is also interesting to note that although both the 37 × 19 and 91 × 7 wires had similar size filaments, the J_c of the 37 × 19 wire decreased at 0.8 mm while that of the 91 × 7 wire increased.

Kim *et al.* [5], made two low filament count wires using a 55 × 7 design, and two high filament count wires with 85 × 7 and a 121 × 7 configurations. The low filament count wires had ceramic fills of 31% and 21%, while the high filament count wires had ceramic fills of approximately 33%. The wires were drawn to sizes between 0.82 mm and 1.35 mm diameter. For the low fill factor, low filament count wire, J_e increased only slightly with decreasing wire diameter. The peak J_e was produced at 0.82 mm and filament size of 19 μm, while the two high count wires, exhibited peak J_e at 0.95 mm. The highest J_e was recorded for the 121 × 7 wire at 0.95 mm diameter and corresponding filament size of 18 μm.

Based on an optimized 85 × 7 design having 15 μm filaments at 0.8 mm diameter, Miao *et al.* [6] produced a series of conductors having 85 filaments in the first restack and 19, 37 and 127

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TABLE I
CONDUCTOR DESIGN PARAMETERS

Conductor	Wire Dia., mm	No. Filaments	Ceramic Fill , %	s/d	Filament Diameter, μm
11B	0.47	931	55	0.25	10
11B	0.65	931	55	0.25	15
11B	0.79	931	55	0.25	20
14B	0.65	889	35	0.41	14
14B	0.72	889	35	0.41	15
14B	0.79	889	35	0.41	17
15C1	0.79	2443	25	0.34	8
15C2	0.79	1393	22	0.32	10
15C3	0.79	973	22	0.37	12
15C1	1.0	2433	25	0.30	10
15C2	1.0	1393	22	0.31	12.5
15C3	1.0	973	23	0.37	15

subelements in the second restack to make wires with 85 × 19, 85 × 37 and 85 × 127 filaments arrangements. At a wire diameter of 1.5 mm the filament sizes were 17 μm, 11 μm, and 4 μm respectively. The wires were partial-melt processed at a peak temperature between 885°C and 895°C in flowing oxygen. They found that J_e increased as filament size decreased for the smaller diameter wires with smaller filaments. When tested in fields of 10 to 16 T, the wire having 17 μm filaments produced the highest J_e. However, lower J_e's were recorded for a 85 × 7 design with 19 μm filaments, while the wires with 11 μm and 4 μm filaments had even lower J_e's. They concluded that lower J_e's with the smaller filament wires were due to sausaging and non-uniformity caused by having too many filaments.

Previously we reported on several single restack designs having filament sizes ranging from 18 to 27 μm at a wire diameter of 0.79 mm [7]. We found that the J_c exhibited an inverse relationship with filament size. The maximum J_e at 4.2 K and self-field was 716 A/mm^2. In related work, Liu and Schwartz [8] showed that J_e could be increased from 750 A/mm^2 to 1450 A/mm^2 at 4.2 K and self-field when the wire diameter was reduced from 0.79 mm to 0.41 mm, which corresponds to a reduction in filament size from 19 μm to 10 μm [8].

This work is part of our effort to develop Bi2212 wire for MRI magnets, and has focused on high filament count single restack designs as a way to more economically manufacture conductor. Due to billet size limitations and other processing factors, our single restack wire can be most economically manufactured with a filament diameter between 18 and 20 μm for a wire diameter of 0.79 mm. This filament size is larger than what has been reported produces maximum J_e. Although it may be possible to enhance J_e by a reduction in filament size, it is necessary to determine the degree of performance increase possible considering the additional processing costs and difficulties required to make the conductor.

II. Methods and Materials

In this study we investigated the effect of filament size using two single stack conductor designs and three double restack conductors having a similar design but with different filament sizes and filaments counts. Although our focus is on single restack designs, a double restack with high filament counts is a more convenient way to produce test conductors with small filament diameters in our target wire diameters of 0.79 to 1.0 mm.

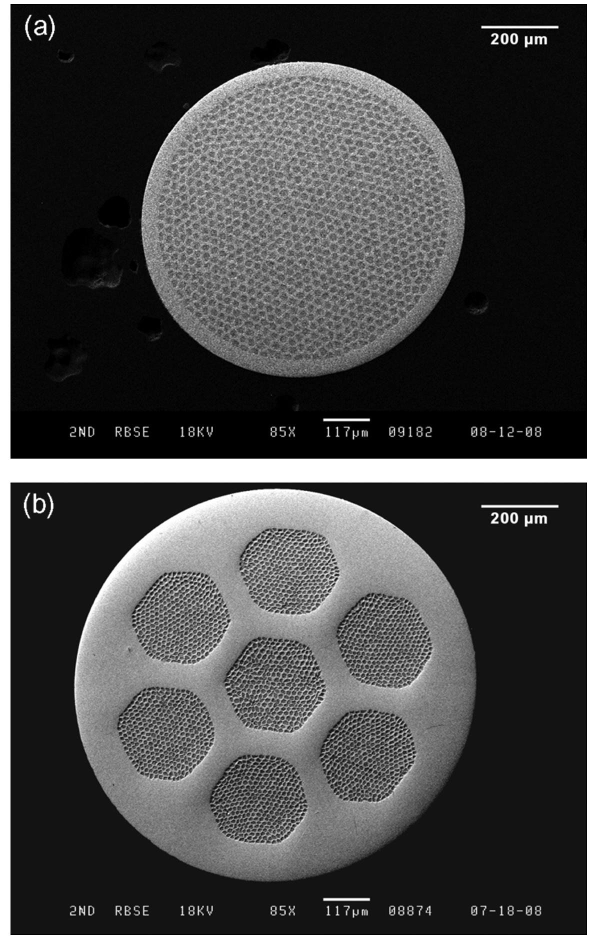

Fig. 1. Transverse cross sections on unreacted wire showing filament arrangement. (a) single restack wire 14B at 0.79 mm diameter with 889 filaments. (b) double restack wire 15C1 at 1.0 mm diameter with 349 × 7 filament configuration.

All the conductors were made by standard PIT processing. Bi2212 granular precursor powder was obtained from Nexans Superconductors. The composition expressed as a cation ratio was 2.19 Bi, 1.95 Sr, 0.88 Ca, and 1.98 Cu. Monofilaments were made by filling pure silver tubes with powder and cold drawing to the restack size. The monofilaments were cut to length and restacked in a second silver tube. The two single restack conductors, 11B and 14B contained 931 and 889 filaments respectively. For the double restack conductors, the first restack billets contained 349, 199 and 139 filaments. These billets were drawn to a predetermined size of a hexagonal cross section and seven subelements of each were restacked in separate Ag-Mg alloy tubes, after which these billets were drawn to diameters 1.0 mm and 0.79 mm producing wires having average filament diameters between 7.8 and 15 μm. The single restack 11B was drawn to 0.79 mm, 0.65 mm and 0.47 mm, producing wires having average filament diameters between 10 and 20 μm. The 14B conductor was drawn to 0.79 mm, 0.72 mm and 0.65 mm, producing wires having average filament diameters between 14 and 17 μm. The sizes of the primary tubes and bundling tubes were proportioned to achieve a desired ceramic fill factor and s/d. Final filament size was controlled by the restack diameter of the monofilament wire. Details for each conductor are given Table I, and representative transverse cross sections are shown in Fig. 1.

10 cm length samples were cut from each wire and heat treated in pure flowing oxygen using the following partial melt solidification cycle:

$$\begin{aligned}
&\text{Ramp to } 820^\circ\text{C at } 120^\circ\text{C/HR}\\
&\text{Hold at } 820^\circ\text{C for 6 HR}\\
&\text{Ramp to } \mathrm{T_{max}} \text{ at } 12^\circ\text{C/HR}\\
&\text{Hold at } \mathrm{T_{max}} \text{ for 0.33 HR}\\
&\text{Cool to } 878^\circ\text{C at } 60^\circ\text{C/HR}\\
&\text{Cool to } 830^\circ\text{C at } 2^\circ\text{C/HR}\\
&\text{Hold at } 830^\circ\text{C for 24 HR}\\
&\text{Furnace} - \text{cool to room temperature}
\end{aligned}$$

$\mathrm{T_{max}}$ is the peak temperature reached during the partial melt cycle. Since $\mathrm{J_e}$ is strongly dependent on $\mathrm{T_{max}}$, samples were evaluated at $\mathrm{T_{max}}$ of 882, 884, 886 and 888°C. Previous testing with this Bi2212 powder produced maximum $\mathrm{J_e}$ at $\mathrm{T_{max}}$ between 886 and 888°C [8].

$\mathrm{I_c}$ measurements were made at 4.2 K and self-field using a standard 4 point resistance arrangement to measure V-I on 4 cm samples with a voltage tap spacing of 1 cm and an $\mathrm{I_c}$ criterion of 1 $\mu\mathrm{V/cm}$. $\mathrm{J_e}$ was calculated by dividing $\mathrm{I_c}$ by the total cross sectional area of the wire. Measurements of filament diameter, fill factor, and s/d ratio were performed on polished cross sections of unreacted wire using SEM photomicrographs and digital image analysis.

III. Results

Fig. 2 shows the effect of wire diameter and $\mathrm{T_{max}}$ on $\mathrm{J_e}$ for the single restacks wires. When the wires were partial melt treated with $\mathrm{T_{max}}$ of 886°C or 888°C, a peak in $\mathrm{J_e}$ was obtained with the 0.65 mm wire. This wire diameter corresponds to a filament size of 15 μm for 11B and 14 μm for 14B. When $\mathrm{T_{max}}$ is above or below the 886 to 888°C range, $\mathrm{J_e}$ decreases with a reduction in wire diameter. These trends are similar to those observed in other studies [4], [5], [9]–[11].

The $\mathrm{J_e}$ results for the double restack wires are shown in Figs. 3 and 4. There is a broad maximum in $\mathrm{J_e}$ for $\mathrm{T_{max}}$ between 884 and 886°C. This range is two degrees less than for the single restack wires. With 0.79 mm wires, peak $\mathrm{J_e}$ occurs at a $\mathrm{T_{max}}$ of 884°C, while peak $\mathrm{J_e}$ occurs at $\mathrm{T_{max}}$ of 886°C for the 1.0 mm wires. As shown in Fig. 4, $\mathrm{J_e}$ decreases as the filament size decreases, and the rate of falloff was less for the 1.0 mm compared to the 0.79 mm wire.

The ceramic fill in the double restack wires is much lower than in either of the single restack wires. To compare the filament size effect between the various wires, $\mathrm{I_c}$ values were converted to $\mathrm{J_c}$ by dividing $\mathrm{I_c}$ by the cross sectional area of ceramic in the wire prior to heat treating. The results are shown in Fig. 5. Peak $\mathrm{J_c}$ corresponds to filament sizes between 12 and 15 μm. The results for the double restack wires and the single restack wire 11B exhibit similar behavior. However, for 14B, $\mathrm{J_c}$ increases much more rapidly as filament size is reduced over the range of 17 to 14 μm. The maximum $\mathrm{J_c}$ for wire 14B is for 14 μm filaments, which is the smallest size tested. These results suggest that there is an optimum filament size that depends on

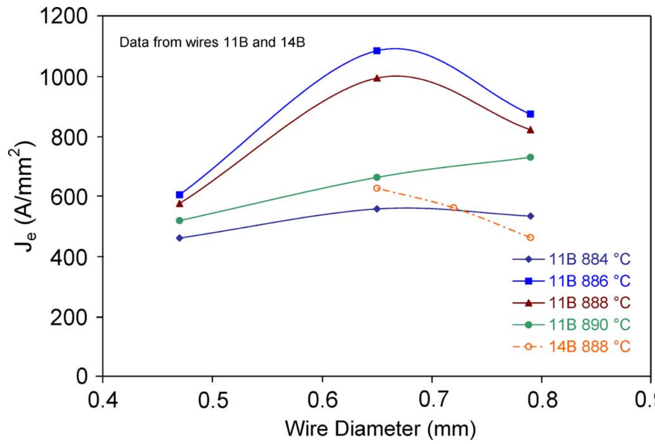

Fig. 2. The effect of wire diameter on $\mathrm{J_e}$ for single restack wires 11B and 14B.

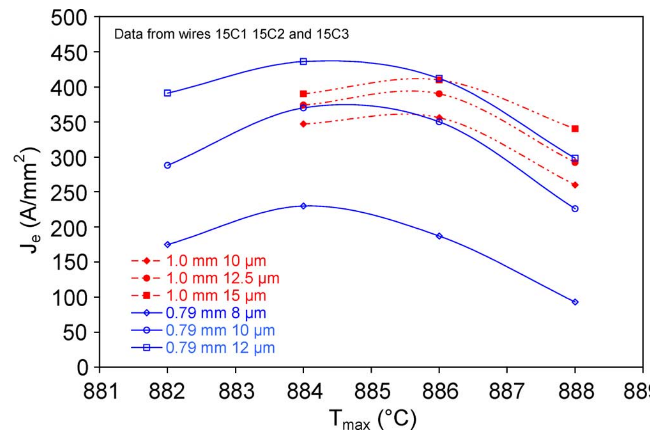

Fig. 3. The effects of $\mathrm{T_{max}}$ on $\mathrm{J_e}$ for the double restack wires at 1.0 mm and 0.79 mm diameters. Peak for $\mathrm{J_e}$ shifts to higher $\mathrm{T_{max}}$ with larger diameter wire.

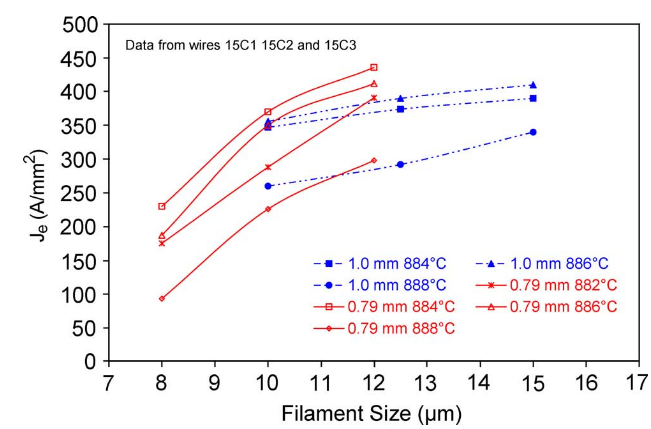

Fig. 4. Correlation between filament size for the double restack wires partial melt treated at $\mathrm{T_{max}}$ between 882°C and 888°C. The falloff in $\mathrm{J_e}$ with smaller filament size is greater for the 0.79 mm diameter wires.

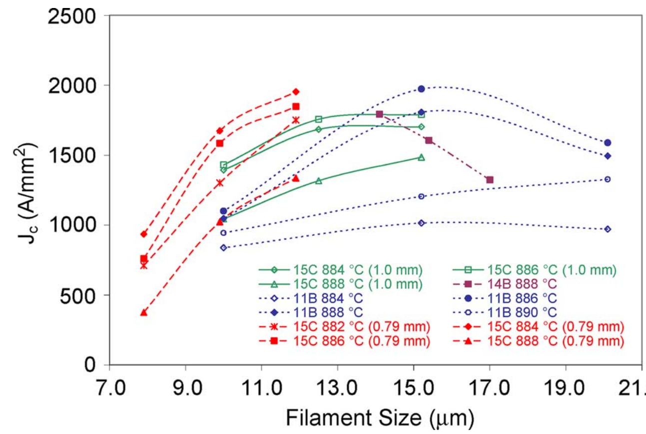

Fig. 5. The effect of filament size and T_{max} on J_c for single and double restack partial melt processed wires. Peak J_c occurs within the range of 12 μm to 15 μm depending on wire size and T_{max}.

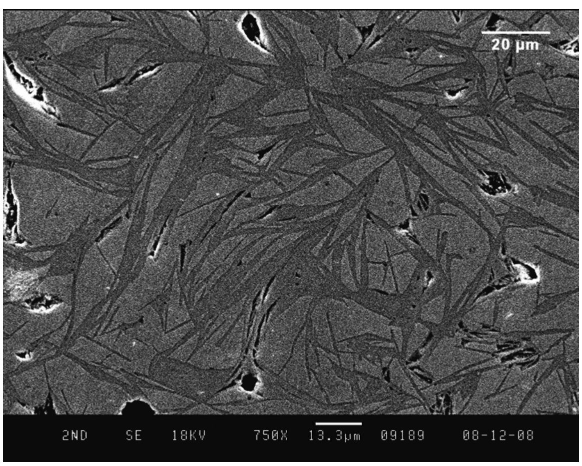

Fig. 6. Transverse cross-section of filament structure in 11B at 0.47 mm partial melt treated at $T_{max} = 888°C$.

conductor geometry and that T_{max} must be adjusted for wire diameter and filament size to maximum J_c.

The microstructures of the wires were examined to correlate the filament structure with J_c. Several prominent characteristic are evident at level of the microstructure revealed by the SEM. In the wires with filaments less than 15 μm, the partial melt heat treatment causes the filament array to morph into a highly interconnected structure consisting of large coalesced filaments bridged by thin ribbons of 2212 forming an irregular interconnected structure. The degree of filament coalescence is greatest in the small diameter wires with small filaments processed at high T_{max}.

Fig. 6 shows the filament structure of 11B at 0.47 mm after T_{max} of 888°C. The filaments have coalesced to form several large regions at the center of the wire, and what appear to be interconnected tentacles of 2212. This type of structure is associated with low J_c. From Fig. 5, the J_c for the 11B wire at 0.47 mm is less than half that of the peak J_e value for the 0.65 mm wire.

The microstructures of the double restack wires exhibited filament bridging when T_{max} was greater than 884°C. The degree of bridging became more pronounced as filament size decreased

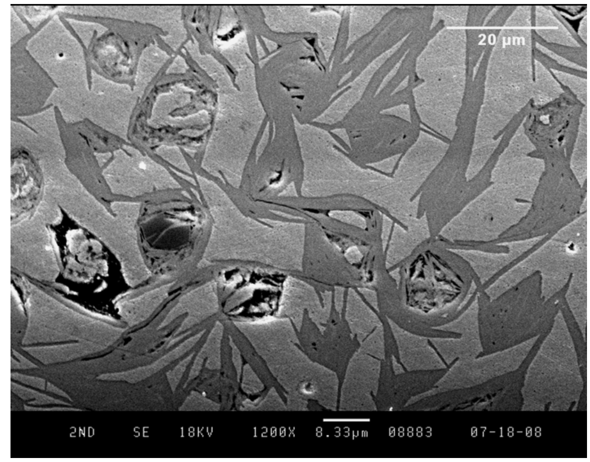

Fig. 7. Transverse cross-section of filaments in 15C3 after partial melt treatment at $T_{max} = 888°C$. Original filament size is 15 μm.

and T_{max} increased. Fig. 7 is the structure of 15C3 at 1.0 mm wire diameter after partial melt at T_{max} of 888°C. The original filament diameter was 15 μm. The filaments are highly branched and all the filaments are interconnected. Thus it appears that, even at 15 μm, filaments are beginning to coalesced structure in the double restack wires.

The filaments in single restack 0.65 mm and 0.79 mm 14B wires appear to be more stable during partial melt processing. Fig. 8 shows transverse cross sections from 0.65 mm wires of 11B and 14B reacted at a T_{max} of 888°C. The filament structure in 14B is still largely intact, although there are some whiskers and filament bridging. The lower amount of filament bridging in 14B is most likely due to the larger spacing between the filaments, and this may the reason that this wire has lower J_c compared to 11B.

A longitudinal cross section of 14B is shown in Fig. 9. There are several characteristic features of these wires evident in Fig. 9. These include interconnects of 2212 grains that form bridges between the filaments. There are also regions where 2212 has pooled and formed non-aligned grains, and some areas of granular 2212. There is some 2201 within the 2212 structure, and there are AEC phases, voids, and pinched-off filaments. Within the filaments can be seen regions where the 2212 grains have formed at an angle to the filament axis and completely span the filament. These features, which are present in all the conductors to some extent, greatly restrict the flow of super currents and lower J_c.

IV. Discussion

To produce high J_e wires, it is generally considered necessary for the wire to have uniform filaments that have the a–b planes of their 2212 grains aligned along the axis of the filaments. Grain alignment occurs during solidification in partial melt processing. The improvement in J_e with smaller filaments is due to better alignment of the 2212 grains as a result of higher interfacial surface area.

There are two explanations for the filament size effect in promoting better grain alignment. The first is based on observations made from tapes, where it was found that there is good grain alignment next to the silver substrate [12]–[15]. This theory is

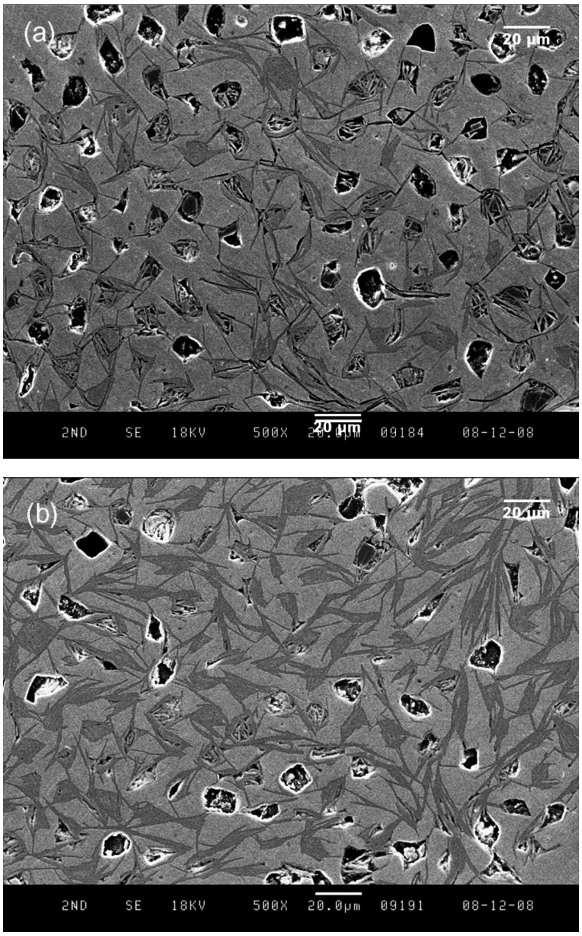

Fig. 8. Microstructures of single restack wires at 0.65 mm diameter after partial at $\mathrm{T\,max} = 888°\mathrm{C}$. (a) wire 14B, $\mathrm{s/d} = 0.35$. (b) wire 11B, $\mathrm{s/d} = 0.25$. Note more extensive bridging in 11B.

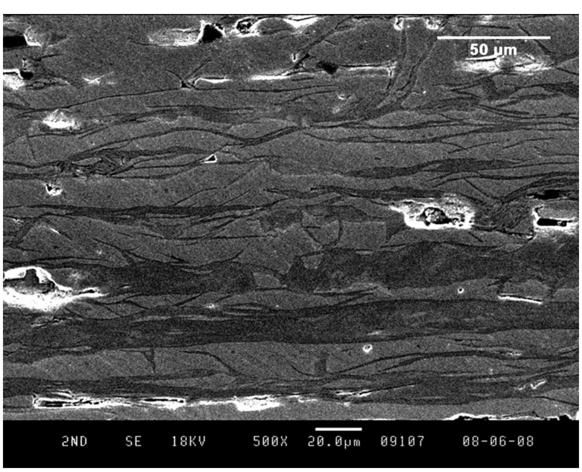

Fig. 9. Longitudinal cross section from wire 14B at 0.65 mm diameter partial melt treated at $\mathrm{T\,max} = 888°\mathrm{C}$. Note formation of interconnects between filaments, and the presences of void and non superconducting phases within the filaments.

based on the concept of oriented nucleation, with silver as a site that preferentially nucleates a–b planes of 2212 grains. It is assumed that by increasing the interfacial surface area, more nucleation sites will be produced thus resulting in a high degree of alignment and high J_e.

The second theory is based on oriented growth, and the fact that 2212 grains grow faster in the a–b directions than they do parallel to the c-axis [16], [17]. In this theory, 2212 grains nucleate and grow randomly in the melt, but only those grains that are favorably oriented with respect to the filament axis will be able to grow appreciably. The growth of unfavorably oriented grains will be stopped by impingement with the silver matrix or grains growing parallel to the silver interface. Neither theory, however, explains why there should be a limit to the filament size effect.

Both of these theories predict that smaller filaments should produced conductors with higher J_e. However, the filaments in real conductors contain defects such as non-superconducting phases, voids and non-aligned 2212 grains. When the size of these defects approaches the filament diameter, current flow will be affected. Unless the current can be shunted around these defects, J_e will be low regardless of filament size. This could be the reason that some amount of filament bridging is usually observed in the microstructures of high J_e conductors.

Since the filament structure has a large effect on J_e, it is important to understand how the filaments are affected by conductor design and partial melt processing. Although the composition of the 2212 precursor, and oxygen concentration in the heat treating atmosphere are known to have an effect on filament structure after partial melt treating [18]–[21], in this study, the composition of the Bi2212 precursor was the same for all the wires and pure oxygen was used during the partial melt heat treatment. Thus the observed J_e values were due to differences in the filament structures that resulted from the different wire designs and T_{max} during partial melt processing.

During melt processing the 2212 precursor powder melts incongruently producing a liquid and non superconducting phases. At T_{max} between 882 and 890°C the primary phases formed in pure oxygen are 1:1 and 14:24 AEC [22], [23]. These phases react with liquid during slow cooling to form 2212 grains. T_{max} affects the phase assemblage, size of the solid phases, and amount of liquid phase in the filaments.

When the 2212 precursor melts, it dissolves some of the silver matrix surrounding the filaments. The liquid penetrates into the silver forming whiskers. The whiskers continue to grow during the slow cooling growth portion of the cycle to form interconnects. Higher T_{max} and longer times promote the formations and growth of interconnects.

Whiskers and interconnects also deplete 2212 from the filament causing voids, discontinuities and leaving isolated and unreacted non-superconducting phases in the filament. Since smaller filaments have more surface area, they also have a proportionately greater number of whiskers in relation to the filament volume compared to larger filaments.

If an interconnect forms between two filaments, the larger filament will grow at the expense of the smaller filament leading to filament coalescence and the formation of an irregular filament structure. As the process continues, interconnects thicken and the overall structure coarsens driven by a reduction in interfacial surface energy. Small diameter filaments have a greater driving force for bridging and coalescence, and wires containing small filaments more readily coarsen compared to those containing large filaments. Thus small diameter filaments are inherently less stable.

Although not directly evaluated in this study, increasing s/d appears to improve filament stability at smaller filament sizes. The spacing between filaments in a wire decreases as the wire is drawn to smaller diameters. This reduces the path length of interconnects and increases the rate of filament coalescence. It may be possible to produce wire with small filaments if the s/d is increased. However, if the ceramic fill is reduced to achieve higher s/d, the J_e of the conductor will be decreased.

The conductors produced for this study exhibit a characteristic response to partial melt processing. Wires with filaments greater than 15 μm diameter are relatively stable and can be processed at a higher T_{max}, although overall J_e is lower. Between 12 and 15 μm, J_e is maximized, but the range of T_{max} is smaller. Filaments less than 12 μm are not stable within the range of T_{max} high enough to produce partial melting.

V. Summary and Conclusions

The high filament count conductors produced for this study had maximum J_e when the filament size was in the range of 12 to 15 μm. As filament size was reduced below 15 μm, the filament structure became more unstable during partial melt processing, and all of the wires with filaments less than 12 μm exhibited excessive bridging and coalescence. All of the wires with small diameter filaments exhibited instability. Although the uniformity of the filaments may have some affect on the filaments morphology that develops during partial melt processing, the phenomenon of filament bridging and coalescence appears to be a fundamental effect associated with the interfacial surface energy between the silver and Bi2212 oxide.

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