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Fast Track Communication

Critical current of dense Bi-2212 round wires as a function of axial strain

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Abstract

The critical current (I_c) of dense Bi₂Sr₂CaCu₂O_{8+x} (Bi-2212) round wires with Ag-alloy matrices was measured as a function of axial applied strain (ϵ_a), to determine whether the $I_c(\epsilon_a)$ behavior is improved in comparison to wires with a large (30–50%) void fraction in the Bi-2212 filaments. Wires were reacted at approximately 890 °C under a 1% O₂ in Ar gas mixture at 100 bar pressure to densify the Bi-2212 fraction during the partial melt reaction. After measurement of the I_c at 4.2 K and 5 T at Florida State University, wire sections were sent to Lawrence Berkeley National Laboratory where $I_c(\epsilon_a)$ was measured at 4.2 K at 5 and 15 T using a U-shaped bending spring. We found that $I_c(\epsilon_a)$ has a 0.3% wide linear reversible strain range. An unexpected result is that the width of this reversible range seems comparable to that found for porous samples reacted at only 1 bar, apparently negating an earlier plausible supposition that fuller density samples would be more resilient.

Keywords: Bi-2212, overpressure, strain, wire

1. Introduction

It has recently been shown [1] that overpressure (OP) reaction of Bi-2212 wires enables densification of the Bi-2212 fraction. OP reaction eliminates the approximately 30% void fraction that is required for wire drawing, and also opposes the internal pressure generated in the wire during the reaction, which originates from expansion of trapped gas, evolution of oxygen, and the presence of contaminants such as carbon and hydrogen that react with oxygen [2]. In reactions without OP, agglomerated 'bubbles' form that block the transport current in the Bi-2212 filaments [3]. Densification yields current densities that surpass the 600 A mm⁻² level at 4.2 K and 20 T that is required [4] for high-field accelerator magnets. This result has amplified interest in Bi-2212 for magnets for high energy physics, as well as for nuclear magnetic resonance and high-field user facilities.

High current densities in high fields translate to high Lorentz loads, and the next step towards implementation of Bi-2212 is therefore to determine the allowable stress and strain levels, since these will drive the mechanical configuration of magnets. One tool to map out the electromechanical properties is a measurement of $I_c(\epsilon_a)$. When observing the available literature data on non-densified Bi-2212, a generic behavior emerges that is summarized in figure 1, using an empirical model that was proposed in 1996 [5] combined with recent $I_c(\epsilon_a)$ results on round Bi-2212 wires [6]. Parallels are drawn to the axial strain sensitivity of (dense) Bi₂Sr₂Ca₂Cu₃O_x (Bi-2223), which exhibits identical behavior under strain [7].

The $I_c(\epsilon_a)$ behavior can be divided into three regions: (1) a central strain range with a linear, reversible $I_c(\epsilon_a)$ dependence; (2) an irreversible reduction on the compressive side of the reversible range; (3) a steeper irreversible reduction on the tensile side of the reversible range. The present consensus is that the linear reversible dependence is an intrinsic dependence that has its origin in the linear rise in the critical temperature (T_c) with pressure for both the *a* and *b* crystal



Figure 1. A model of literature results on non-densified Bi-2212 wires and tapes, after Ten Haken *et al* [5] using slopes suggested by Cheggour *et al* [6].

orientations [8–10]. Cycling of axial strain on the linear reversible strain range up to 10 000 times showed that this range was not perfectly reversible for early Bi-2212 tapes, but that the I_c reduction saturated at about 200 cycles after a few percent irreversible loss in I_c [11]. Recent high resolution $I_c(\epsilon_a)$ results on modern wires measured on a Cu-2wt%Be Walters spring [6], suggest that the linear range is fully reversible, with a 2.5% reduction in I_c per percent of applied axial strain. Outside the reversible range, the I_c is irreversibly reduced, and the 25–40%/% reduction on the compressive side is about one order of magnitude smaller than the 150–300%/% reduction on the tensile side [6].

Loss of proportionality outside the linear reversible strain range between lattice strain and externally applied strain in early x-ray diffraction studies on Bi-2212 [12] and in recent quantum beam diffraction studies on Bi-2223 [7], suggested crack formation to be the cause for the irreversible reduction of I_c , which was confirmed by microscopy on Bi-2212 [6] and by magneto-optical imaging on Bi-2223 [13]. If the applied strain is reversed after cracks have formed, a new linear reversible strain range is observed that is wider but located at a lower I_c level, while having the same slope as the original reversible range [5, 6]. This wider strain margin at the cost of an irreversibly reduced I_c is likely due to initial failure in weaker regions of the highly irregular Bi-2212 filament structure, after which the current is carried by mechanically stronger, still intact Bi-2212 fractions.

The pre-compression after cool-down to 4.2 K is determined by the larger thermal contraction of the surroundings, such as the Ag/Ag-alloy matrix and the material of a strain rig, compared to the Bi-2212 fractions. It was experimentally found that the pre-compression of Bi-2212 wires that are





Figure 2. Cross-sections of 0.8 mm diameter wire from OST billet PMM100610 (W13, left), and from Showa billet B3005 (right), after reaction in 100 bar overpressure.

soldered to a Cu-2wt%Be substrate matches the strain state of free wires within 0.01% [14].

2. Experiment

In the experiments by Cheggour *et al* [6], crack formation after strain application was observed mainly in Bi-2212 regions containing porosity, whereas no apparent cracks were observed in dense Bi-2212 regions. This suggests that crack formation should be reduced or postponed (i.e. yielding a wider linear reversible strain range) if the wires contain no porosity. To test this hypothesis, we measured $I_c(\epsilon_a)$ on dense Bi-2212 wires. We investigated four Bi-2212 wires manufactured by Oxford Superconducting Technology (OST), and two wires manufactured by Showa. Important differences between our experiment and that of Cheggour *et al* [6] are, respectively, a smaller voltage tap length of about 5 mm versus 80 mm, the use of 5 cm straight samples versus about 50 cm long spiraled samples, and the use of Ti-6Al-4 V compared to Cu-2wt%Be substrate material.

Nominally 80 mm long wire sections with sealed ends were reacted at Florida State University (FSU) using an appropriate OST heat treatment [15] in a 100 bar, 1 at% O_2 in Ar gas mixture. Two representative scanning electron microscopy (SEM) cross-sections of the densified wires are shown in figure 2. The Bi-2212 fractions in both wires are highly dense. Black regions, likely to be alkaline-Earth cuprates, are visible in both wires. The light grey regions in the filaments of the Showa wire cross-section are likely $Bi_2Sr_2CuO_x$ (Bi-2201) impurities [16] resulting from the heat treatment used, which was optimized for OST wire rather than for Showa wire. The I_c of 50 mm long sections was measured at FSU at 4.2 K and 5 T, after which the samples were shipped to Lawrence Berkeley National Laboratory (LBNL) for $I_{c}(\epsilon_{a})$ measurements. At LBNL, 45 mm sections were soldered to a Ti-6Al-4 V U-shaped bending spring [17] using eutectic Pb-Sn solder, and the I_c at zero applied strain was measured at 4.2 K and 5 T, to check for potential handling damage. The $I_{\rm c}$ at 15 T was then measured as a function of compressive or tensile axial strain. When a lower I_c was detected, the sample was unloaded to a smaller applied strain to determine whether the I_c reduction was irreversible. $I_c(\epsilon_a)$ for one OST wire was



Figure 3. Normalized critical current as a function of axial applied strain at 4.2 K and 15 T using a Ti-6Al-4 V U-shaped bending spring. Sample W7-B17 was measured at 5 T.

measured at 5 T to find out whether the observed behavior depends on the magnetic field amplitude. The samples were returned to FSU for microscopic analysis after strain testing.

3. Results

An overview of the samples and I_c and *n*-values at an electric field criterion of $E_c = 10^{-4}$ V m⁻¹ is given in table 1. The I_c and *n*-values were determined by fitting at least a one decade electric field range around E_c . It is seen that the 5 T I_c values are reduced by 1.4–3.4% for the OST wires, and by 30.6 and 6.8% for the Showa wires, the first of which was clearly damaged by handling. The *n*-values measured at LBNL are typically 25% lower than at FSU, which is mainly attributed to the reduced resistance of the parallel current path as a result of the solder and holder, and the *n*-value of the damaged Showa wire B3005-C1 is reduced by 50%.

A summary of the axial strain results is given in figure 3. It was found that the linear reversible strain range for the OST W7 wire set is at least 0.3% wide, and that the compressive side measured for the OST W13 wire behaves similarly. The OST W13 wire was not measured in tension, and reversibility was not checked at every strain step due to time constraints. This limited the resolution by which the irreversibility strain



Figure 4. Typical cracks as detected in etched sections of W13 after compression (left), and W7 after tension (right).

was determined. The W7-B17 sample, which was measured at 5 T in compression, has a similar dependence as sample W7-B16 that was measured at 15 T. The Showa B3005 wire set has a linear reversible strain range that, within the limited resolution of the determination of the irreversibility onset, appears to be 0.4-0.5% wide. When the strain was reduced after a reduction in I_c was observed, it was found that the I_c remained constant, indicating a new linear reversible strain range at a lower I_c level, except for the I_c reduction at -0.2%strain in the compressive measurement of wire W7-B17. The irreversible I_c reduction on the compressive side is about 50% per % strain, whereas on the tensile side roughly 400%/% is found. The irreversible reduction is therefore similar to or slightly larger, then for the not-densified wires in figure 1 that showed 25-40% and 150-300% reductions for the compressive and tensile sides, respectively [6].

Longitudinal cross-sections were made after completion of the strain experiment, and figure 4 shows typical cracks that were observed after application of compressive and tensile strains. Finally, we performed Vickers hardness tests on the external alloyed sheaths of each wire and found 115 HV and 123 HV for the OST and Showa alloys, respectively.

4. Discussion

In order to make a direct comparison of our results, which were measured on a Ti-6Al-4 V bending spring, to the results on similar, but not-densified OST wires that were measured on a Cu-2wt%Be Walters spring by Cheggour et al [6], an adjustment of the strain axis has to be made to compensate for the difference in thermal contraction of the two holder materials. The solder that was used to mount the wires in both experiments was eutectic Pb-Sn solder, which solidifies at 456 K [18]. An overview of the thermal contraction of the relevant materials from 456 K down to 4.2 K, is given in table 2 [18]. The thermal contraction of the Bi-2212/Ag-alloy composite wire cannot accurately be calculated due to the anisotropy in the coefficients of thermal expansion in the Bi-2212 fraction (and most likely also in its mechanical properties), as well as the yield in the Ag(-alloy) matrix that will likely occur. We therefore estimate the contraction of the free wire from 456 K down to 4.2 K using the rule of mixtures, assuming a 25% Bi-2212 fraction and that the Bi-2212 is axially aligned in the a-b direction. It is found that our estimate of the thermal contraction of a free wire over this

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Table 1	. Sample identification.	, critical current	, and <i>n</i> -values at E _a	$h = 10^{-4} \text{ V m}^{-1}$	¹ and no applied axial strain.
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						$I_{\rm c}$ at FSU		<i>I</i> _c at LBNL			
					Bi-2212	$\mu_0 H =$	5 T	5 T		15	Г
Sample	Manuf.	Billet number	Matrix	Stack	Fraction	<i>I</i> _c [A]	n	<i>I</i> _c [A]	n	<i>I</i> _c [A]	п
W13-97	OST	PMM100610	Ag-0.2wt%Mg	37 × 18	25%	422	18	416	14	303	11
W7-A11	OST	PMM091221-1	Ag-0.2wt%Mg	37 × 18	25%	497	19	484	15	360	16
W7-B16	OST	PMM091221-1	Ag-0.2wt%Mg	37 × 18	25%	410	18	396	12	288	8
W7-B17	OST	PMM091221-1	Ag-0.2wt%Mg	37 × 18	25%	412	19	401	15	_	_
B3005-C1 ^a	Showa	B3005	Ag-Mg-Sb	61 × 7	33%	300	13	208	6	163	7
B3005-B99 ^a	Showa	B3005	Ag-Mg-Sb	61 × 7	33%	307	14	286	11	222	12

Showa wires were reacted using a heat treatment that is optimized for OST powder compositions.

Table 2. Thermal contraction from 456 to 4.2 K [18].

Material	$\Delta L/L$ [%]
Ti-6Al-4 V	-0.30
Cu-2wt%Be	-0.61
Ві-2212 а-ь	-0.29
Bi-2212 c	-0.55
Ag	-0.71
Wire $a-b$ (calculated)	-0.60

temperature range matches that of Cu-2wt%Be within 0.01%, which is in agreement with the experimental literature result [14]. This means that the pre-compression in the Bi-2212 fraction when using a Cu-2wt%Be holder will closely match that of the free wire, provided that the Ag/Ag-alloy matrix does not yield. The Ti-6Al-4 V holder, however, places the Bi-2212 into +0.31% tension when compared to the free wire, as is shown in figure 3. The slope of the reversible strain range is, within our limited resolution, comparable to the -2.5%/% that is reported in the literature. The tension in the Bi-2212 generated during cooldown on the Ti-6Al-4 V holder can thus partly explain the lower 5 T I_c values at LBNL, since the measurements at FSU were performed on wires that were not soldered to a substrate. With this pre-strain adjustment, the width of the linear reversible strain range, as well as its location on the strain axis, match the results for not-densified OST wires [6] within the limited resolution at which the irreversibility onset can be determined in our experiments. This result, if it is confirmed by higher resolution measurements, is contrary to the expectation that dense wires would be more resilient to crack formation, especially on the compression side where the probability for buckling should be small due to the very limited presence of voids in these highly dense wires. At the same time, the absolute value of I_c for the dense wires is roughly a factor three higher than for the notdensified wires that were reacted with open ends (which allows for an undefined amount of contaminant gas to escape at the ends while creating internal pressure and porosity in the central wire sections [19]).

The 5 T result on sample W7-B17 supports the assumption that $I_c(\epsilon_a)$ is largely crack dominated, suggesting that more cost effective measurements in self-field and at elevated temperatures, e.g. at 65 K, can be used to analyze a

conductors' onset of crack formation. Measurements at 65 K will likely show a substantially larger slope in the reversible strain range, due to the much lower value of the irreversibility field $(H_{irr}(T))$. If the behavior in the linear reversible strain range is determined by $H_{irr}(T)$ as a function of strain, then a larger slope can also be expected on the linear reversible strain range at 15 T compared to 5 T, but this cannot be detected with the limited resolution of our experiment.

A more pronounced irreversible reduction of I_c outside the reversible strain range in the densified wires might be expected, due to the absence of voids that might inhibit crack propagation. Such a larger reduction is only partially supported by our data. Specifically with respect to the compressive side, no indications of buckling were observed inside the Bi-2212 filaments (figure 4), in contrast to the only partially dense wires [6], suggesting at least improved support of the Bi-2212.

Though the $I_c(\epsilon)$ results on the Showa wires in figure 3 could suggest a wider reversible strain range for these wires, perhaps due to the irreversibly reduced I_c (see figure 1), the data are clearly compromised by handling damage to the wires and are therefore considered unsuited for conclusions.

5. Conclusion

We arrive at the surprising conclusion that the linear reversible strain range width of 0.3% strain in porous wires appears not improved upon by densification of the Bi-2212. The observed strain margin before crack formation in dense Bi-2212 is similar to what has recently been observed in dense Bi-2223 [7]. Explanations for the Showa wires are inconclusive, due to handling damage to the wires. Improved support through reinforcement of the matrix in close proximity to the Bi-2212 filaments could perhaps increase the strain margin before crack formation occurs, but this has to be investigated further. Failing that, although a 0.3% strain margin for a magnet conductor is not large, it is predictable, and a magnet design that accurately takes into consideration differences in thermal contraction will enable the selection of a desired strain state within the linear reversible strain range, as is supported by the strain scaling for the different holder materials.

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References

- [1] Larbalestier D C et al 2014 Nat. Mater. 13 375-81
- [2] Shen T, Ghosh A, Cooley L and Jiang J 2013 J. Appl. Phys. 113 213901
- [3] Kametani F et al 2011 Supercond. Sci. Technol. 24 075009
- [4] Bottura L and Godeke A 2012 *Rev. Accel. Sci. Technol.* 5 25–50
- [5] ten Haken B, Godeke A, Schuver H J and ten Kate H H J 1996 IEEE Trans. Magn. 32 2720–3
- [6] Cheggour N, Lu X F, Holesinger T G, Stauffer T C, Jiang J and Goodrich L F 2012 Supercond. Sci. Technol. 25 015001



- [7] Osamura K, Machiya S, Hampshire D P, Tsuchiya Y, Shobu T, Kajiwara K, Osabe G, Yamazaki K, Yamada Y and Fujikami J 2014 Supercond. Sci. Technol. 27 085005
- [8] Meingast C, Junod A and Walker E 1996 Physica C 272 106-14
- [9] van der Laan D C, Douglas J F, Clickner C C, Stauffer T C, Goodrich L F and van Eck H J N 2011 Supercond. Sci. Technol. 24 032001
- [10] Lu X F, Goodrich L F, van der Laan D C, Splett J D, Cheggour N, Holesinger T G and Baca F J 2012 IEEE Trans. Appl. Supercond. 22 8400307
- [11] ten Haken B, Beuink A and ten Kate H H J 1997 IEEE Trans. Appl. Supercond. 7 2034–7
- [12] ten Haken B and ten Kate H H J 1996 Physica C 270 21-24
- [13] van der Laan D C, Schwartz J, ten Haken B, Dhallé M and van Eck H J N 2008 Phys. Rev. B 77 104514
- [14] Sugano M, Itoh K and Kiyoshi T 2006 IEEE Trans. Appl. Supercond. 16 1039–42
- [15] Jiang J, Starch W L, Hannion M, Kametani F, Trociewitz U P, Hellstrom E E and Larbalestier D C 2011 Supercond. Sci. Technol. 24 082001
- [16] Naderi G and Schwartz J 2014 Appl. Phys. Lett. 104 152602
- [17] ten Haken B, Godeke A and ten Kate H H J 1999 J. Appl. Phys.
 85 3247–53
- [18] Ekin J W 2006 Experimental Techniques for Low-Temperature Measurements (New York: Oxford University Press)
- [19] Malagoli A, Kametani F, Jiang J, Trociewitz U P, Hellstrom E E and Larbalestier D C 2011 Supercond. Sci. Technol. 24 075016