Processing long-length Ag-sheathed $Bi_2Sr_2CaCu_2O_x$ wires for high critical current density: internal gases and creep of Ag at high temperatures

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Abstract

High engineering current density J_E of >500 A/mm^2 at 20 T and 4.2 K can be regularly achieved in the Ag-sheathed multifilamentary $Bi_2Sr_2CaCu_2O_x$ (Bi-2212) round wire when the sample length is several centimeters. However, J_E(20 T) in Bi-2212 wires of several meters length, as well as longer pieces wound in coils, rarely exceeds 200 A/mm^2. Moreover, long-length Bi-2212 wires often exhibit signs of leakage after melt processing that are rarely found in short, open-ended samples. Expanding on work by Malagoli *et al*. [1,2], combined with detecting gas releases during heat treating the state-of-the-art powder-in-tube (PIT) Bi-2212 wire using mass spectroscopy and modeling gas transport in Bi-2212 wire, we explicitly show that J_E degradation with length is a direct consequence of Bi-2212 de-densification due to wire swelling produced by high internal gas pressures at elevated temperatures. We examined the wire expansion at critical stages of the melt processing of as-drawn PIT wires and the wires that received a degassing treatment or a cold-densification treatment before melt processing, further showing that wire swells because under the influence of internal gas pressure, silver creeps and ruptures, and that the creep rupture of silver sheath naturally leads to the leakage of Bi-2212 liquid. Identifying the mechanisms of J_E degradation and leakage led us to conclude that it is feasible to develop a long-length, leak-free Bi-2212 conductor that carries a J_E of >500 A/mm^2 for very high field superconducting magnet technology through a combination of reducing or removing the source of internal gases with techniques such as applying an external gas pressure during heat treatment to inhibit de-densification of Bi-2212 or to even induce compressive Ag creep to further densify Bi-2212.

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1. Introduction

An important index of high-field superconducting magnet technology is the total current carried by a superconducting strand divided by its total cross-section area, or engineering current density J_E. High J_E in magnetic fields of >20 T was demonstrated in $Bi_2Sr_2CaCu_2O_x$ (Bi-2212) multilayer, dip-coated tapes [3] and multifilamentary, powder-in-tube (PIT) tapes [4] processed by a melt processing approach [5], and then similar success was later reproduced in PIT multifilamentary Bi-2212 round wires [6-9]. Thus, melt-processed Bi-2212 wires are being considered for future very high field magnet technology, beyond the ~20 T limit of Nb_3Sn magnet technology. As an example of the state of the art, J_E reached 480 A/mm^2 at 4.2 K and 20 T in 2005 in a 1 m barrel sample made by Oxford Superconducting Technology [6,7], and this benchmark was recently pushed further to approximately 540 A/mm^2 at 4.2 K and 20 T in short pieces of ~10 cm wires [10]. A J_E of 500 A/mm^2 is generally considered as sufficient for a new high-field magnet technology.

Despite of these successes in obtaining high J_E in short-pieces of Bi-2212, melt-processing of long-length Bi-2212 conductor often yields J_E much less than expected [11-13]. J_E of coils has rarely exceeded 200 A/mm^2 at 4.2 K and 20 T [11-13]. Moreover, melt-processed Bi-2212 coils have often suffered from leakage, showing clear discolorations where Bi-2212 liquid leaks through the encasing metal (Ag or Ag alloy) at high temperatures and reacts with surrounding materials such as insulation [11,14]. The leakage degrades J_E as well as mechanical properties of superconductor composite [14-18]. These problems seriously limit magnet applications of Bi-2212 but ways to eliminate the leakage and to improve the J_E in coils have yet to be found because little has been known as regards to what causes leakage and J_E degradation in long-length Bi-2212.

In the case of Bi-2212 tape conductor, one of major causes of low J_E in long-length Bi-2212 was suggested to be that the high internal gas pressures at high temperatures [19-23]. Internal gases may include residual air and humidity present during as-drawn wires, gases released as a result of contaminants adsorbed on powder surfaces [24] and solid remnants in powder gasifying upon heating. Contaminants and gas impurities may include hydrates, hydroxides, carbonates [25], nitrates, and residues such as wire drawing lubricants (hydrocarbons); they transform to gases (H_2O, CO_2, or NO_2) upon heating. The consequence is that tapes with high carbon contents were observed to swell over long length, crack, or even bulge locally [23]. Gases can presumably diffuse along the conductor axis and out the ends when conductors are relatively short and are not sealed at the ends, but they would be trapped inside when conductors are relatively long or when their ends are sealed, creating high internal gas pressure and causing the conductor to deform irreversibly when heated at ambient pressure. The argument gives a plausible, qualitative explanation for J_E degradation in long-length tapes, but details about contamination amounts, gas pressures, at what temperatures gases are being released are lacking.

For PIT round wires later fabricated from modern low-carbon powder (<200 ppm by weight, normalized to the weight of oxide filaments), the effect of trapped gas is more subtle and not yet clearly defined. For an Ag/AgCu sheathed multifilamentary Bi-2212 wire, Kuroda et al. [26] examined J_E variation in short 3 cm open-ended wire, long 1 to 3 m open-ended wires, and a 10 m long coil. They found that: (1) J_E in the 10 m long coil is 35% of that in the 3 cm, open-ended wire; (2) J_E in the 1.5 m open-ended wire

strongly varied along the length, with the middle section having the lowest J_E and the end 20-30 cm section having J_E comparable to that of the short, open-ended 3 cm samples. Malagoli *et al.* [1] verified these findings in a more recently made Bi-2212 wire with a Ag-0.2wt.% Mg external sheath. Malagoli *et al.* [1] also made an important observation: the 1 m wire sample swelled along the entire length despite the ends being open during melt processing, and the greatest wire expansion occurred at the central section. Given the importance of removing porosity in PIT Bi-2212 for obtaining high J_E revealed recently [10,27-32], the observation made by Malagoli et al. [1,2] naturally led to the conclusion that J_E degradation in long-length Ag-sheathed wire is a direct consequence of the de-densification of Bi-2212 due to the wire swelling produced by the internal gas pressure.

While it has been tentatively established that internal gases are causing wire swelling in Ag sheath and J_E degradation in long-length Bi-2212 wires, a number of important questions remain. The most crucial one concerns how silver deforms at high temperatures and at what temperatures the wire swells most significantly, since such understanding may shed lights on what gases are primarily causing wire to expand or may even suggest whether possible remedies exist. Second, it is important to establish to what extent the low J_E in long length wire is due to residual gas in green wires, and to what extent it is controlled by gases being released into wires during heat treatment, including O_2, H_2O, and CO_2, as such understanding will help assess whether an optimization in conductor design (such as using stronger sheath materials), or a modification in wire manufacturing step will reduce or eliminate the J_E degradation. Third, it is important to establish a quantitative relationship between the rate of wire swelling and the gas impurity levels because superconductor industry will need a clear idea of what levels of solid impurities can be tolerated for wires with different designs. Fourth, it is also important to have a quantitative description of gas transport in Bi-2212 wires so the question as to what accounts to be a long-length sample can be properly answered and the J_E dependence on the conductor length could perhaps be predicted. We have addressed some of these issues by observing wire swelling for a group of representative state-of-the-art PIT multifilamentary round wires of Bi-2212 at critical stages of melt processing. Important and new information about the mechanism of wire swelling, leakage, and J_E degradation has been obtained. We will also present a model of gas transport in Bi-2212 wires. The results of our studies are useful for developing Bi-2212 magnets that will consistently realize the full potential of this high field material for >22 T high field magnet applications.

2. Experimental design

The multifilamentary superconductor round wire selected for this study were manufactured by Oxford Superconducting Technology at New Jersey using the PIT technique by drawing a pure Ag tube with Bi-2212 precursor powder, then using a double restack to form several bundles. The final wire, 0.8 mm in diameter, contains hundreds of oxide filaments (37x18 filaments, ~20 μm in diameter) embedded in a matrix of pure silver, which is again encased in a stronger precipitation-hardening Ag-Mg sheath. The superconductor/Ag/AgMg ratios are 0.25/0.50/0.25. The filaments in as-drawn wires have an oxide packing density of ~74%. The wire was fabricated from a highly homogeneous, melt-cast precursor powder with a composition of $Bi_{2.17}Sr_{1.94}Ca_{0.90}Cu_{1.98}O_x$ developed by Nexans SuperConductors GmbH. Nexans indicated that the powder contained 30 ppmw carbon and 70 ppmw hydrogen. Such a 37x18,

0.8 mm wire has been demonstrated to carry a J_E of 800 A/mm^2 at 4.2 K and 5 T (an extrapolation will gives a J_E of >540 A/mm^2 at 20 T) [10] and has been investigated for making Rutherford cable for high-field magnets such as >16 T dipole.

Three key experiments were designed and performed. First set of experiments, including two tests, were designed to quickly check whether internal gases are causing J_E degradations. First we melt processed 8 cm and 1.2-1.4 m wires with their ends sealed and compared their J_E and microstructure with those with their ends open during heat treatment. Sealing the ends of short-length wires before melt processing prevents gases from escaping and allows the behaviors of long-length wires to be studied. The heat treatment schedule for melt processing was to heat from room temperature to 820 °C at 160 °C/h, hold at 820 °C for 2 hours, heat again from 820 °C to 889 °C at 48 °C/h, hold at 889 °C for 0.2 hour, cool to 881 °C at 10 °C/h, further cool to 835 °C at 2.5 °C/h, hold at 835 °C for 48 hour, and then cool to room temperature. The processing was performed in 1 bar flowing O_2.

Critical current I_c of fully reacted 8 cm wires were determined through the standard four point methods using an electric criterion of 1 μV/cm in a background field of up to 14 T. The I_c variation along 1.2-1.4 m wire was examined using a method adapted from those established for testing critical current density of brittle Nb_3Sn conductors [33]. The 1.2-1.4 m long Bi-2212 strand was wound onto a grooved cylinder machined from a 96% alumina and then reacted. After the heat treatment, the spiral sample was transferred to a tubular barrel made of Ti-6Al-4V alloy and cooled down to 4.2 K for critical current measurement. Critical current along the length was determined by six pairs of voltage taps each monitoring current-voltage characteristics of 10 cm conductor at 4.2 K and fields up to 11 T at Brookhaven National Lab and to 14 T at Fermi National Accelerator Lab (Fermilab).

Second, gas release from Bi-2212 at high temperatures was monitored by analyzing the composition of gases in a vacuum tube furnace using a quadrupole residual gas analyzer while heating 5 cm Bi-2212 wires (with ends open) at 180 °C/h from 25 °C to 900 °C.

The second set of experiments we performed, including two tests, was central to this study. First, we observed the wire swelling and leakage patterns of wires through melt processing for 8 cm wires with ends sealed and compared to those with ends open. Second, we determined the rate of wire swelling at critical stages of melt processing for wires receiving various thermo-mechanical treatments. The wire swelling rate was measured by the rate at which wire diameters increased. Wire diameters were measured using a laser micrometer with a measurement resolution of 0.3 μm. The micrometer measures the diameters along eight axes by rotating the wire in steps of 22.5 ^{o}; an average wire diameter and a standard deviation are then recorded.

The third set of experiments was performed to gain more insights into the question of whether the wire expansion and leakage can be eliminated by reducing gases in the wires. We gave 8 cm and 1.4 m wires a degassing treatment at 450 °C in vacuum for 9 to 48 hours before melt processing (during vacuum annealing, sample ends were open to allow gases to escape.) The samples were then melt processed with their ends open or sealed, after which their diameter and J_E along length were determined and analyzed, and compared to those samples that received no such treatments.

3. Results

3.1. Impact of sealing wire ends

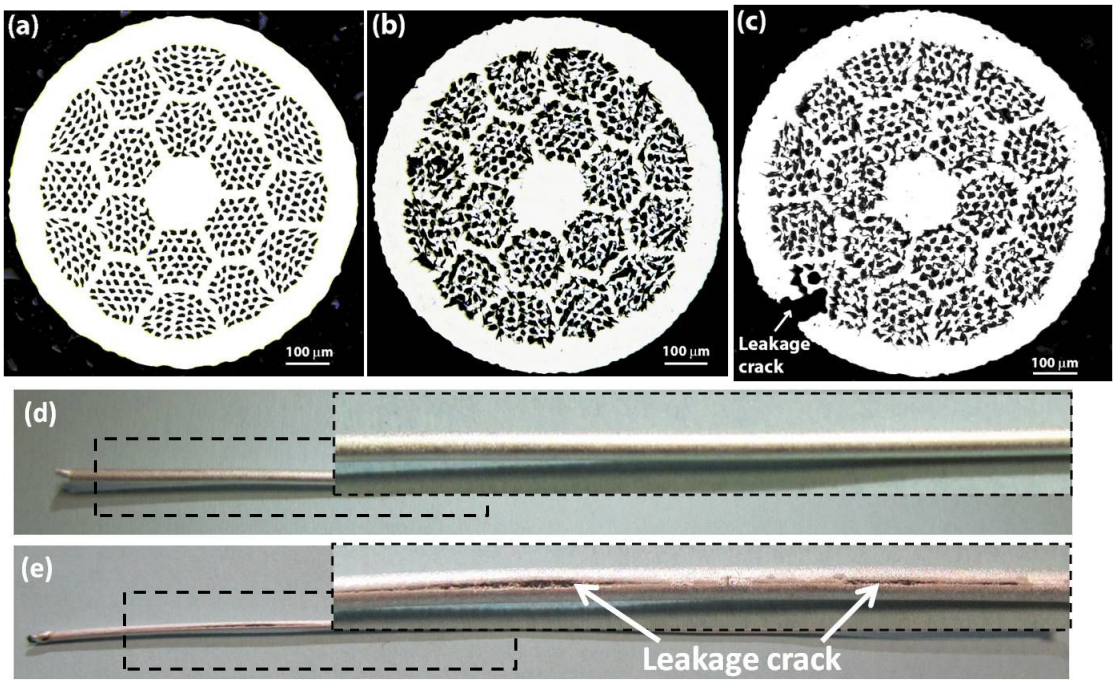

Figure 1: Representative micrographs of transverse cross-sections of (a) a unreacted Ag-sheathed multifilamentary Bi-2212 round wire, (b) an 8 cm long Bi-2212 wire after being melt processed with its ends open, (c) an 8 cm long Bi-2212 wire after being melt processed with its ends sealed. Photographs of sample (b) and (c) are shown in (d) and (e) respectively. The graph (e) shows longitudinal cracks nearly parallel to the wire axis.

Figure 1 shows micrographs of a representative PIT Bi-2212 round wire before and after melt processing. Heat treating with ends sealed caused wire to swell: Diameter for sample (a) and (b) is 0.803 mm whereas the diameter for wire (c) increased to 0.830 mm. Such wire swelling is accompanied by significant critical current degradation: Wire (b) carried an I_c of 180 A at 4.2 K and 5 T whereas I_c of wire (c) scattered from 70 to 150 A. The sealed-ends sample leaked, showing longitudinal leakage cracks like the one in Figure 1.

Figure 2 shows the wire diameter as well as critical current I_c along a 1.2-1.4 m wire. For open-ends sample, wire swelling is the most pronounced in the middle section of the wire (by 1.5%) and decreased towards the ends (by 0.6%); the parabolic pattern of wire swelling was matched by a parabolic distribution of critical current density, which is the lowest in the middle section of the open-ends wire. No leakage was found in 1.2-1.4 m open-ends samples. Sealing the ends of the 1.2-1.4 m long wire caused it to swell more (by >2% in the center) and leak, and further reduced the wire J_E.

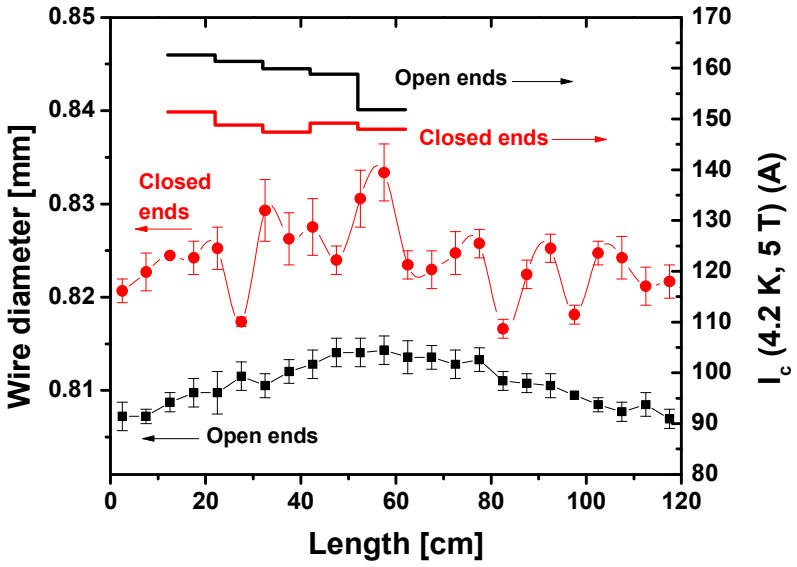

Figure 2: Variation of critical current I_c(4.2 K, 5 T) and wire diameter in 1.2-1.4 m long Bi-2212 wires melt processed with ends open or ends sealed. The wire diameter of sealed-ends sample show large error bars because wire cross-section became irregular as it leaked. The samples for diameter measurement were 1.2 m long whereas the samples for I_c measurement were 1.4 m long. Only half of the 1.4 m samples were measured for I_c due to the expected symmetry.

3.2. Gas release by Ag-Bi-2212

Figure 3 shows the gas partial pressures in the vacu um furnace during heating 20 pieces of 5 cm long Bi-2212 wires at 180 °C /h to 900 °C. Major gases detected included CO_2, CO/N_2, H_2O, H_2, and O_2. A sudden increase of O_2 signal at 860-870 °C, an indication of the melting of Bi-2212 powder, was accompanied with an increase in CO_2 signal. On further increasing the temperature to 900 °C, partial pressures of CO_2, H_2, H_2O, and N_2/CO fell whereas the partial pressure of O_2 persisted at high levels.

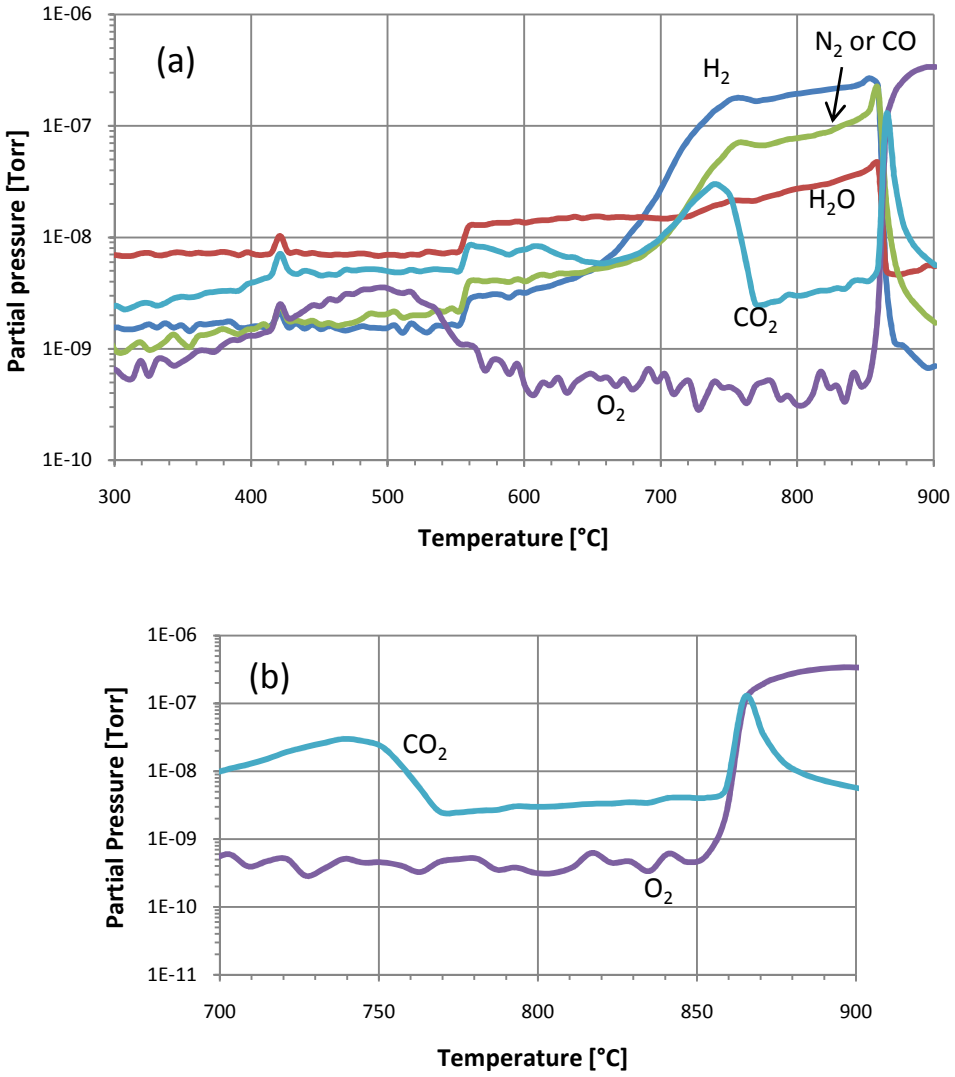

Figure 3: The gas partial pressure in a vacuum furnace detected by a residual gas analyzer. Inside the furnace were Bi-2212 round wires being heated at 180 °C/h to 900 °C. About 20 pieces of wire each 5 cm long with open ends were used for these measurements.

3.3. Rates of wire swelling and leakage at high-temperatures

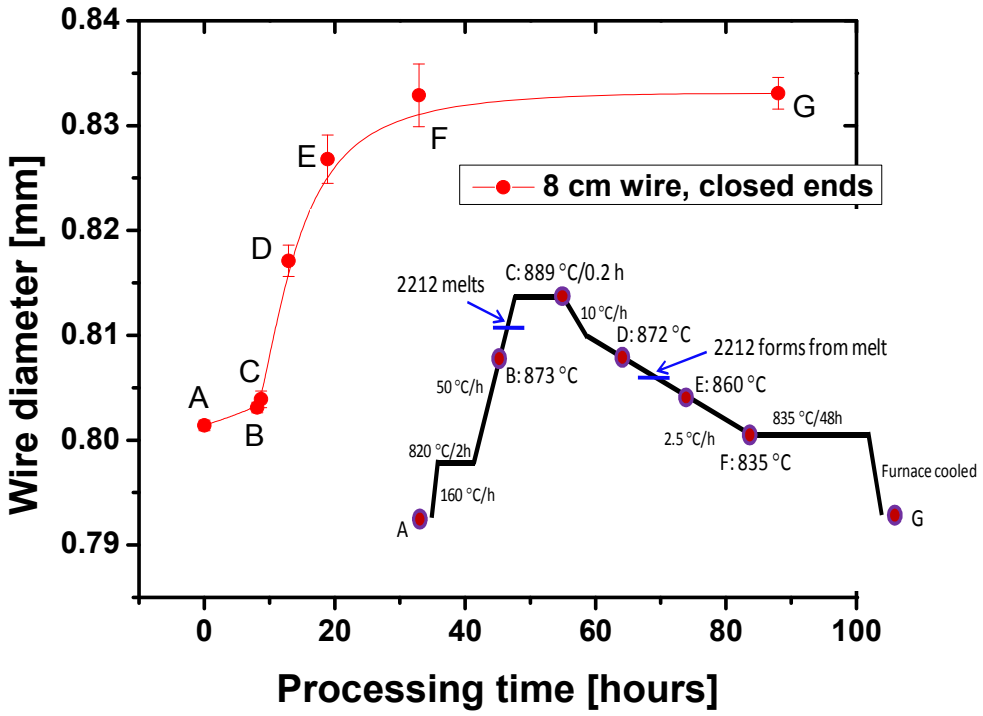

Figure 4: Progression of wire diameters of Bi-2212 along the melt processing. Wires were heat treated following the processing schedule to specific points and cooled down, after which their diameters were measured. The inset shows the melt processing used and critical processing points where the wire swelling was evaluated.

Figure 4 presents the progression of wire diameters of Bi-2212 along the melt processing. The diameter of closed-ends sample increased over the entire processing, whereas wire diameter of open-ends sample remained relatively unchanged. Wire swelling in closed-ends samples accelerated after Bi-2212 melts around 880 °C on heating, peaked at 889 °C in the melt, decreased on cooling, and nearly stopped before 48 hour annealing at 835 °C. The leakage cracks were first observed in sample E.

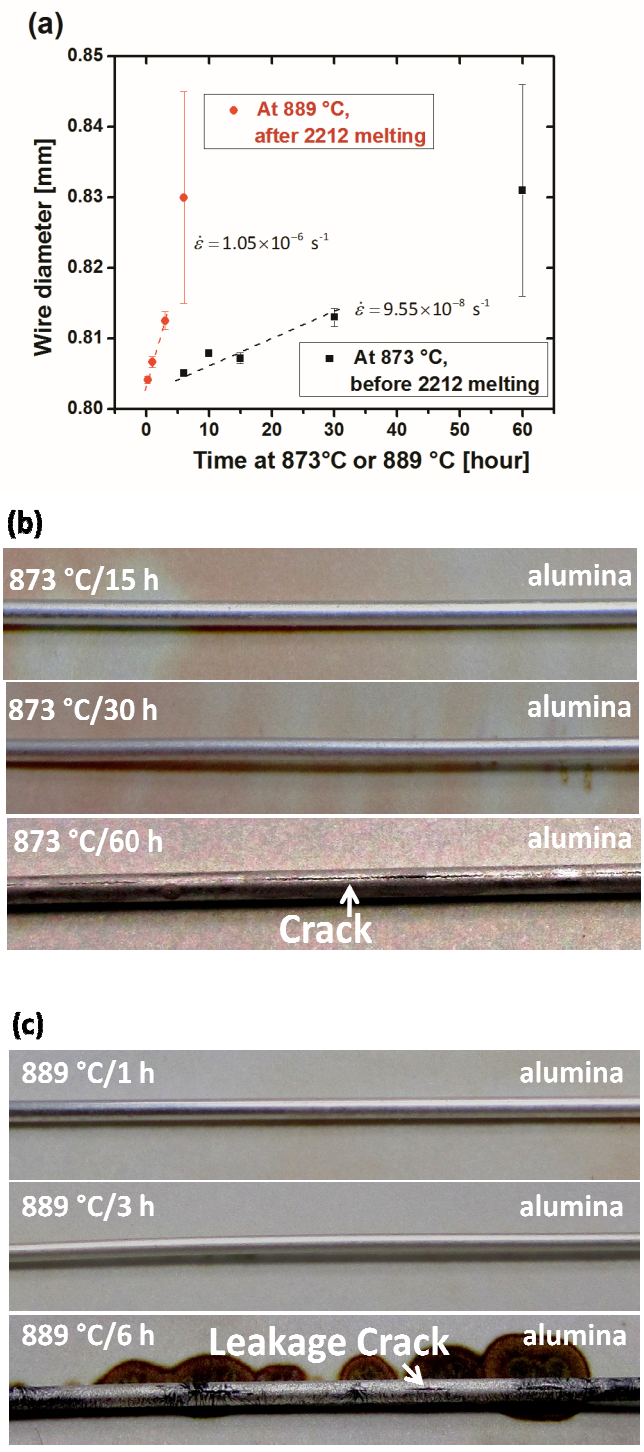

Figure 5: The rate of wire swelling at 873 °C (before melting), and 889 °C (after melting): (a) Wire swelling for 8 cm Bi-2212 sealed-ends wires at 873 °C and 889 °C. Wires were heated from room temperature to 820 °C at 160 °C/h, annealed at 820 °C for 2 hours, and then heated at 50 °C/h to 873 °C or 889 °C, respectively. Wires were then held either at 873 °C for up to 60 hours or at 889 °C for up to 6 hours and furnace cooled; sample diameters were then measured. (b) Photographs of Bi-2212 sealed-ends samples cooled after being held at 873 °C for 15 hours, 30 hours, and 60 hours. (c) Photographs of Bi-2212 sealed-ends samples after being held at 889 °C for 1 hour, 3 hours, and 6 hours. In the case of (b) and (c), samples were photographed with the alumina paper on top of which samples laid during reaction.

Figure 5 presents the rates of wire swelling at two critical points of melting processing, at 873 °C before Bi-2212 melting and at 889 °C after Bi-2212 melting. Wire gradually swelled at both 873 °C and 889 °C. The wire diameter went up approximately at a rate of 0.955×10^{-8} s^{-1} at 873 °C but the rate increased by a factor of eleven to 1.05×10^{-6} s^{-1} at 889 °C. Wire held at 873 °C for 60 hours cracked (but didn't leaked because the Bi-2212 filaments have not yet been melted.) whereas at 889 °C wire cracked and leaked for only 6 hours. Wires were reacted on top of a 99.9% alumina paper. In the case of 889 °C – 6 hours, a clear reaction occur between leaked Bi-2212 liquid and alumina, whereas for 873 °C – 60 hours, wire cracks but there was no sign of such reactions.

3.4. Impact of degassing treatments

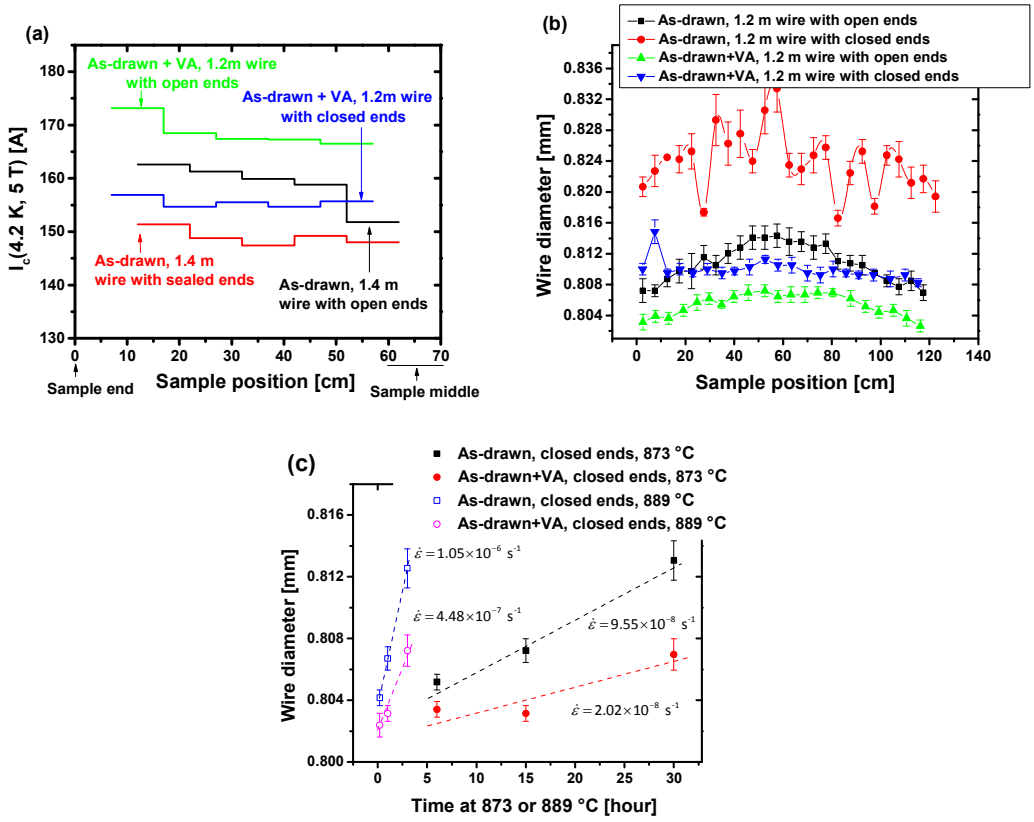

Figure 6: (a) The variation of I_c in 1.2-1.4 m long wires that are melt processed after receiving a vacuum annealing at 450 °C for 9-48 hours, as compared to that of samples melt processed without a vacuum annealing. During vacuum annealing, the ends of all samples were open. (b) The wire diameter along the same group of samples. (c) The wire swelling at 873 °C or 889 °C for wires that received a vacuum annealing at 450 °C for 9 hours, as compared to that of samples that received no such treatments. VA=vacuum annealing.

Figure 6 shows that giving 1.2-1.4 m wires a vacuum annealing at 450 °C for 9 hours before melt processing raised wire I_c, while reducing the degree of wire swelling. Figure 6c also indicates that with vacuum annealing at 450 °C for 9 hours, the rate of wire swelling decreased to 2.02×10^{-8} s^{-1} at 873 °C and to 4.48×10^{-7} s^{-1} at 889 °C

4. Discussion

We determined J_E and high-temperature expansion in a representative state-of-art Ag-sheathed PIT multifilamentary Bi-2212 round wire heat treated with open-ends and sealed-ends, and we found: (1) The J_E of closed-ended samples was severely degraded; in addition, the closed-ended sample swelled, cracked, and leaked; (2) the mass spectroscopy experiment indicated many gases, including CO_2 and H_2O, were being generated upon heating Bi-2212 wires, and (3) J_E in 1.2 m open-ended sample was not only degraded, but also strongly varied along the wire length, showing a parabolic pattern similar to that found by Kuroda *et al*. [12] and Malagoli *et al*. [1]. (4) Applying a degassing treatment to Bi-2212 reduced the wire swelling and improved the I_c/J_E uniformity in 1.2 m long sample; moreover, such a vacuum annealing treatment allows 1.2 m sample to reach >95% of the short-sample I_c. All of these findings are consistent with the underlying hypothesis that internal gases causes wire swelling and J_E degradation.

Discussion of our experiments will proceeds in three phases. First, we will associate the J_E degradation of melt-processed long-length Bi-2212 wires to a decrease in the density of Bi-2212 filaments caused by wire swelling. Second, we will proceed in a more quantitative vein to understand how wire swelling occurs at high-temperatures under the influence of internal gas pressure by silver creeping and will conclud that the leakage is a natural consequence of the creep-rupture of Ag-alloy external sheath. Simple expressions will be given to describe the stress-sensitive nature of the creep-rupture behavior and time constants for gas diffusion in Bi-2212 wires. We will conclude with a discussion of the implications of our results for conductor development and high-field magnet engineering of Bi-2212.

4.1. J_E degradation of long length wires caused by de-densification of Bi-2212 filaments

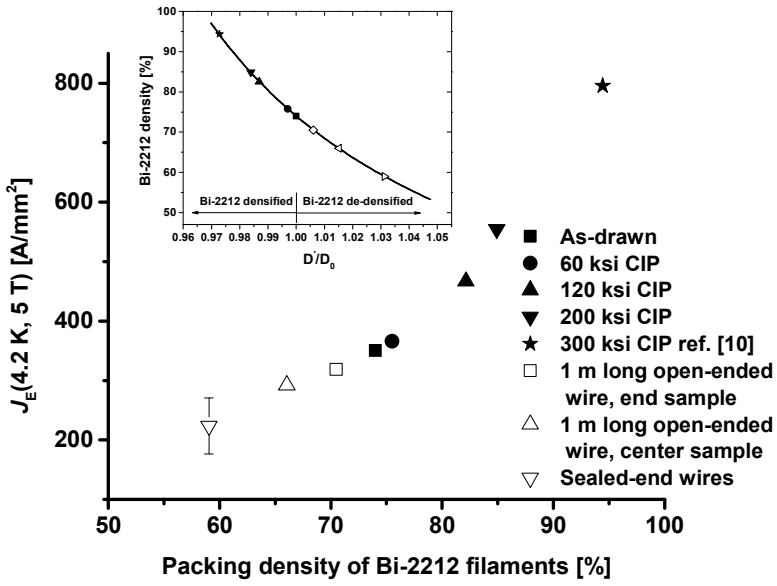

Figure 7: Changes in diameter and density of Bi-2212 filaments induced by cold densifications or wire swelling in a multifilamentary PIT round wire of Bi-2212 and their influence on its J_E(4.2 K, 5 T). D_0, the original diameter of the as-drawn wire, is 0.802 mm, whereas D' is the new diameter of the wire after being subjected to various treatments. The density of Bi-2212 was calculated assuming incompressibility of Ag and AgMg and the packing density is 74% in as-drawn wires, other 26% of whose filament volumes are occupied by gases. CIP=cold isostatic processing, which was applied to as-drawn wires before melt processing. The as-drawn sample and CIP samples are 8-10 cm short samples melt processed with their open ends.

A direct consequence of the swelling of Ag-Bi-2212 composite is that the density of Bi-2212 filament will decrease. Figure 7 presents the calculated oxide filament density and I_c(4.2 K, 5 T) of representative long-length Bi-2212 samples that experienced various amount of de-densification, together with those of samples that received cold-isostatic pressing (CIP) powder compaction treatments. Figure 7 shows a strong proportionality between oxide filament density and J_E in polycrystalline, melt processed Bi-2212 wires. A 1.2% decrease in the wire diameter under cold-isostatic pressing at 120 ksi increased the density of Bi-2212 filament from 74% to 82% , and as a result, its I_c (open-ended 8 cm sample) increased by 29%. For 1 m long open-ended sample and closed-ended samples, the oxide density decreased to be less than 59%, inducing degradation of J_E by 55% (as compared to the as-drawn, open-ends 8 cm sample). J_E degradation in long-length wires is therefore due to the de-densification of Bi-2212 filaments.

The metallurgical interpretation of Figure 7 is that the key to extending high critical current density in short-length Bi-2212 samples to long-length wires is to eliminate the de-densification caused by wire swelling. The basis of porosity playing a major role in controlling connectivity and J_c in Bi-2212 wires was given by the quench experiments combined with microscopy [27-31], which snapshot critical stages of melt processing and which revealed that upon melting, porosity in Bi-2212 filaments agglomerates into

gas bubbles that are large and long enough to divide filaments into segments [28,29,31]. Though reshaped and redistributed at the latter stage of melt processing, these pores remain to block current flow in fully processed wires. The importance of reducing porosity for achieving high J_E found by quench experiments was further reinforced by Jiang et al. [10], who increased the Bi-2212 filament density of a multifilamentary PIT wire from 74% to 92% using CIP and doubled its J_E(4.2 K, 5 T) from 400 A/mm^2 to 800 A/mm^2. The accompanied quench studies and in-situ high-energy X-ray tomography studies of CIPed sample [32] revealed that CIPping significantly reduced the volume fraction as well as the size of gas bubbles in the melt.

4.2. The creep and rupture of silver sheath at high-temperatures as a result of Ag-Bi-2212 being subjected to high internal gas pressure

In this section, we will discuss how high internal gas pressure can lead to swelling and rupture of silver sheath at high temperatures. Similar to a cylinder metal tube subjected to high internal pressure at high temperatures, the Ag/Bi-2212 wire would yield or creep under the influence of high internal gas pressure nearing the melting temperature of silver (920 °C in O_2) for a long duration time. Figure 5 shows that the Bi-2212 wires swell and then rupture, following a typical metal creep behavior that include three stages of plastic deformation: After a short primary creep stage, it went through a long period of steady-state creep, end with a negligible tertiary creep stage, and rupture. The temperature and stress dependence of the steady-state creep rate $d\varepsilon / dt = \dot{\varepsilon}$ can be described by a semi-empirical relation:

$$\dot{\varepsilon} = A \cdot \sigma^n \cdot \mathrm{e}^{-\frac{Q}{R \cdot T}}$$

Where A is a constant, n the stress exponent, Q the activation energy, R the gas constant, and T the absolute temperature. The creep behaviors of pure Ag and dispersion strengthened AgMg under a simple tension or compression stress have been investigated by Leverant et al. [34], Nieh et al. [35], Goretta et al. [36,37]. Our discussion concerns the deformation of Ag and AgMg at >800 °C. The most relevant data was provided by Leverant et al. [34]. Their data show that the stress components are 4.34 for pure silver and 3.05 for Ag + 0.2 at. % MgO while the activation energies are 177.7 and 186.0 kJ/mol for pure silver and Ag + 0.2 at.% MgO respectively.

Here we summarize the steady-state creep rates determined for several representative samples in Table 1 (assuming that samples were in the steady-state creep state). Table 2 also indicated that the creep strain rate at 889 °C in the as-drawn wire is 1.05 x 10^{-6} s^{-1}, which decreased to 4.48 x 10^{-7} s^{-1} in the wire that received a vacuum degassing treatment and which increased to 1.98 x 10^{-6} s^{-1} in the wire CIPped at 125 ksi before melt processing. Such changes in creep rates can be readily explained by changes in internal gas pressures. According to [34], an axial stress of 2.8 MPa is needed to produce a creep rate of 10^{-6} s^{-1} at 889 °C under the simple tension test in pure Ag.

Table 1: Creep rates in wires that received different thermo-mechanical treatments at high-temperatures. Sample 1 is an 8 cm open-ended, as-drawn wire whereas sample 2 is an 8 cm closed-ended, as-drawn wire. Sample 3 is an 8 cm closed-ended, as-drawn wire that was given a vacuum annealing at 450 °C for 9 hour before being melt processed. Sample 4 is 8 cm closed-ended wire that was given a cold-isostatic pressing treatment at 125 ksi before being melt processed.

Sample	**Circumferential creep strain rates (s^{-1})**		**Leakage in fully processed wires?**
	873 °C, before Melting	889 °C, in the melt	
1	Negligible	Negligible	No
2	9.5×10^{-8}	1.05×10^{-6}	Yes
3	2.02×10^{-8}	4.48×10^{-7}	No
4	2.53×10^{-7}	1.98×10^{-6}	Yes

Long-time creep in metals and their alloys ends with inter-crystalline cracking and fracture substantially transverse to the direction of the principal stress. For our Bi-2212 round wire samples in which circumferential stress and radial stress are significant, long cracks parallel to the wire axis were observed (figure 1 and figure 5). We therefore conclude that the leakage of Bi-2212 is a natural consequence of the creep rupture of Ag sheath.

The creep-rupture strains were determined to be 12.57% for pure Ag and 1.31% for Ag + 0.1 at % Mg [35,37] at 400 °C and under an uni-axial stress of 35 MPa. This report showed that our wire had a rupture circumferential strain of 2.5-3.0%, indicated that this wire is limited by the ductility of AgMg. With the steady-state creep rate as large as 10^{-6} s^{-1} for Bi-2212 wires at 889 °C, the time to rupture is small, about 5.6 hours. This prediction is quantitatively consistent with what was observed in figure 5, which showed wires leaked when held at 889 °C for 6 hours.

Many mathematical models have been developed to predict the stress state in metallic cylinder vessels subjected to internal pressures and/or external pressures at high temperatures and methods have also been developed to relate its circumferential strain rate to that given by a simple tension creep test. However, deducing the internal gas pressure from the creep rates in Table 1 is difficult because predicting the creep rate in multifilamentary round wire Bi-2212 is substantially more difficult due to the multifilament nature and the composite material structure.

4.3. Internal gases and their pressure in closed-end Bi-2212 wires at high-temperatures

Understanding that the fundamental cause of J_E degradation and leakage is the internal gas pressure, we may explore the nature of such internal gases. The first question to ask is to what extent the low J_E in long-length wires is due to the residual gas in green wires. Our results implied that the residual gas unlikely plays a major role for two reasons. First, the residual gas analyzer clearly saw many gases were being released during heat treating as-drawn wires (figure 3). Second, if the residual gas is responsible for the Ag creep, the creep rate can't increase by a factor of 10 from 9.5 x 10^{-8} s^{-1} to 1.05 x 10^{-6} s^{-1} when increasing temperature from 873 °C to 889 °C. Such significant increase in the creep strain rate could be explained by the release of CO_2 during the melting of Bi-2212 as observed by Figure 3. CO_2 release at the melting of Bi-2212 powder may indicate that a major source of carbon gas is $SrCO_3$.

To roughly estimate the internal gas pressures in sealed-ends Bi-2212 PIT wires in the melt, we assume: (1) after Bi-2212 melts, 100% of C & H impurities are converted into gases; (2) the solubility of CO_2 and H_2O in Bi-2212 liquid is negligible. As measurements indicated that W1 contained 134 ppmw hydrogen and 186 ppmw carbon, the internal gas pressure P in the as-drawn PIT Bi-2212 wire (oxide packing density=74%; gas in unreacted wires is air) is 16.2 MPa, with $P(H_2O)$=12.5 MPa, $P(CO_2)$=2.90 MPa, and P(air)=0.4 MPa. Such high gas pressure is certainly strong enough to induce a creep rate in an order of 10^{-6} s^{-1} in AgMg alloy, according to [34] [36,37]. This represents a ball-park analysis of the high-end pressure that wires could experience and we should note that at other processing regions, the internal gas pressure can be much lower. However, predicting such gas pressure can hardly be very accurate in the melt state or in other processing regions because of many complexities involved with melt processing a material like Bi-2212 during which gas phases may continue to react with other phases in the system.

4.4. Gas diffusion along wire axis in long-length Bi-2212 wires

For wires heat treated with their ends open, some fraction of gas can diffuse out through their ends. We will discuss how such gas diffusion leads to the J_E degradation with increasing length and the strong variation of J_E in a 1-2 m long wire as demonstrated by Figure 2 and Figure 6.

The gas transport in Ag-2212 composite conductor at high-temperatures is driven by gradients in gas molecule concentrations and amplified by gradients in internal gas pressure. We will first treat it as a gas diffusion problem without considering pressure effects, as for practical purposes, we do not need precise values of time constants of gas transport, and an appreciation of the orders of magnitude is sufficient.

The gas diffusion in a multifilamentary around wire of Bi-2212 along the filaments can be described by a one-dimensional diffusion equation:

$$\frac{\partial C}{\partial t} = \frac{\alpha^2}{L^2}\frac{\partial^2 C}{\partial x^2} \qquad 0 < x = \frac{x'}{L} < 1 \quad 0<t<\infty$$

$$\text{Boundary conditions} \begin{cases} C(0,t) = 0 \\ C(1,t) = 0 \end{cases}$$

$$\text{Initial condition} \quad C(x,0) = C_0$$

where *C* is the concentration of gas molecules, $\alpha^2 = D_e$ is the effective gas diffusion coefficient, *L* is the length of the conductor being heat treated, *x'* is the location coordinates, and x is dimensionless coordinates. The model here applies to the gases whose diffusivity through Ag matrix is negligible; the diffusion of oxygen through Ag will be separately discussed in next section.

Such partial-differential equation system can be solved analytically using the method of separation of variables. The solution is [38]:

$$C(x,t) = \sum_0^\infty \{A_{2n+1} \cdot e^{-[\frac{(2n+1)\cdot\pi\cdot\alpha}{L}]^2\cdot t} \cdot \sin[(2n+1)\cdot\pi\cdot x]\}$$

$$\text{With } A_{2n+1} = \frac{4C_0}{(2n+1)\cdot\pi}$$

In long time, the terms further out in the series get small very fast due to the factor $e^{-[\frac{(2n+1)\cdot\pi\cdot\alpha}{L}]^2\cdot t}$. Hence, for long time periods, the solution is approximately equal to the first term [38]:

$$C(x,t) \cong A_1 \cdot e^{-(\frac{\pi\alpha}{L})^2\cdot t} \cdot \sin(\pi x)$$

$$\text{With } A_1 = \frac{4C_0}{\pi}$$

The time constant for gas diffusion is approximately as

$$t^* = \frac{L^2}{\pi^2\cdot\alpha^2}$$

As we stated at the beginning, this diffusion process is accelerated by pressure gradients we have not yet taken into account so this t^* is rather a conservative estimation for how quick gas transport can occur in Bi-2212.

Gas transport through a porous media like Bi-2212 powder mainly occurs by molecular diffusion and/or advection through the pores. Several empirical and semi-empirical perdition models can be used to estimate D_e. One of such models was proposed by Millington and Quirk (M-Q-model) [39,40]:

$$D_e = \theta_a \cdot T_a \cdot D_a^0$$

$$\text{With } T_a = \frac{\theta_a^{7/3}}{n^2} = n^{1/3}\cdot(1-S_r)^{7/3}$$

Where D_a^0 is the free (undisturbed) diffusion coefficient in air ($D_a^0 = 1.8x10^{-5}$ m^2 / s for oxygen at 22 °C, $D_a^0 = 1.6x10^{-5}$ m^2 / s for CO_2 in air at 22 °C), θ_a is equivalent porosity, T_a is the gas-phase tortuosity, n is the porosity in the wire, and S_r is the degree of saturation.

When 2212 is in powder state, S_r=0,

$$D_e = n^{4/3} \cdot D_a^0$$

In as-drawn wire, n=0.25. Using a $D_a^0 = 1.8x10^{-5}$ m^2 / s, the above equation gives $t^* = 9.9$ h for a 1 m long conductor at room temperature.

The M-Q model doesn't take into account gas diffusion through the liquid phase, so that for S_r=1 (saturated medium), it leads to an effective diffusion coefficient of zero (D_e=0), which is an unrealistic value but accurate enough for the case of gas diffusion through Bi-2212 liquid at high-temperatures, because a compound's diffusion coefficient is ~10^4 as great in air then in liquid.

$$t^*(\text{when Bi-2212 is in liquid state}) \cong 10^4 \cdot t^*(\text{when Bi-2212 is in powder state})$$

Approximating temperature dependence of diffusion coefficient as $D \propto T^{3/2}$ [Chapman–Enskog theory],

$$t^*(\text{in Bi-2212 liquid, } 890^\circ\text{C}) \cong 13600 \text{ h} \quad \text{for 1 m conductor}$$

It is far greater than the time in the melt t_{melt} for typical Bi-2212 melt processing, which is often less than 5 h so gas diffusion through liquid to two ends of wires can be omitted. This conclusion is verified by the residual gas analyzer data in the figure 3, which showed that the signals of CO/N_2, H_2, and CO_2 diminished after Bi-2212 melted.

Table 2 presented the estimated time constants t* for Bi-2212 wires in 0.1 m, 1.2 m, and 100 m. One use of such data is to predict whether leakage will occur in a particular sample length. Note that significant CO_2 released during the melting of Bi-2212 powder has only <10 minutes to diffuse out. Therefore, samples of >25 cm in length will trap some fractions of CO_2 so they are essentially no-longer a short-length sample. Also, note that the melt processing we used allows samples to stay in the powder state above 800 °C for more than 3.5 hours, so in 1.2 m long or shorter open-ended sample, the gas generated before 820 °C can diffuse out. Further increasing wire length to >3 m will trap nearly all gases, producing a gas pressure comparable to that of the sealed-end sample. Therefore, leakage will begin to appear even in 3 m long open-end sample. All of the above predications on leakage were confirmed in representative as-drawn PIT wires.

Table 2: Estimated time constants for gas diffusion in Bi-2212 wires of 8 cm, 1 m, and 100 m in length

Sample description	**t* (hour) (800 °C)**	**Maximum P_{total} Predicted (MPa)**	**Leakage occurrence**
8 cm, open-end sample	0	0.4	No
8 cm, closed-end sample	-	16.2	Yes
1.2 m, open-end sample	2.12	>2.90 but <16.2	No
100 m, open-end sample	14700	16.2	Yes

4.5. Oxygen diffusion through Ag matrix

It is worth discussing oxygen diffusion in Bi-2212 wires separately because it would diffuse through Ag quickly and also because there is big misconception in the literature (see for example [22]) that deformation of Ag matrix is dominantly caused by O_2 release.

When melted, Bi-2212 powder releases oxygen. From the thermal gravimetric data given by Kanai et al. [22] we can roughly estimated that the amount of oxygen released is >11800 ppmw. Release of such large amount of oxygen into wire void space in a small time period of Bi-2212 melting has been argued to cause sudden increase in internal gas pressure, bulging Ag and causing "effluence of Bi-2212 liquid out of wire" [22]. Here we calculate the time constant for oxygen diffusion through Ag and argue that this is not the case.

The diffusivity and solubility of oxygen in solid silver follow the relations [41]:

$$D(\text{Oxygen in Ag}) = 4.9\times10^{-7}\cdot e^{-\frac{11{,}600}{R\cdot T}}\ \text{m}^2/\text{s}$$

$$N(\text{Oxygen in Ag}) = 7.2\cdot e^{-\frac{11{,}500}{R\cdot T}}\ \text{at. percentage}$$

At 880 °C, D=1.5 x 10^{-7} m^2/s, N=2.17 at. %. The time constant for radial oxygen diffusion through Ag matrix an Ag-Bi-2212 composite wire in diameter of 0.8-1.2 mm at 880 °C is 0.12-0.24 s so O2 can readily diffuse through Ag matrix, even when Bi-2212 is in melt state as seen in figure 3.

When processed in 100% O_2, Bi-2212 melting occurs predominantly in 880-886 °C. The melting event in our experiment (heating rate=0.8 °C/min) took $\approx$7.5 minutes, which should prevent pressure jump due to oxygen release.

4.6. Leak-free, long-length Bi-2212 wires that carry J_E of >500 A/mm^2 at 4.2 K and 20-30 T: feasibility and measures

The mechanisms of J_E degradation and leakage identified above led us to conclude that it is feasible to develop a long-length, leak-free Bi-2212 conductor that carries a J_E of >500 A/mm^2 for very high field

superconducting magnet technology through a combination of two strategies. First, further conductor development should reduce or remove the source of internal gases through developing powders with lower gas impurity, adopting careful atmosphere control during wire processing, or using degassing treatments similar to that used in this study. Soon after this study, one wire manufacturer (OST) made significant progress on producing wires with low gas impurity contents, delivering a R&D batch of Bi-2212 to Fermilab with 104 ppmw carbon and 30 ppmw hydrogen.

The understanding also suggests a second measure that might be very useful: applying an external gas pressure higher than 1 bar during heat treatment to not only inhibit de-densification of Bi-2212 but also to densify Bi-2212 to >95% of its ideal density through inducing compressive Ag creep. Such methods were tried in 1990s [42,43] and recently a form of such methods (1 bar oxygen + *x* bar inert gases, *x* can be as high as 100) was used by Jiang et al. [44] to raise the J_E(4.2 K, 5 T) of a sealed-end 0.8 mm Bi-2212 wire to 1000 A/mm^2.

4.7. Some final comments

The above discussion and calculation help establish that the deformation behaviors of PIT Bi-2212 wires at high-temperatures can be described by creep of silver sheath produced by high internal gas pressure, and help illustrate many aspects of the issue of J_E degradation in long-length Bi-2212. Such analysis is useful for guiding Bi-2212 conductor development, but one has to bear in mind that the stress in silver sheath is a complex function of wire architecture, gas impurities levels, heat treatment history, and porosity levels in Bi-2212 filaments when applying similar calculations to their wires for prediction of creep rates, leakage occurrence, and J_E levels.

5. Conclusion

We have presented experiments and calculations that explicitly identified the internal gas pressure as the root cause of low J_E as well as leakage in long-length Bi-2212 wire. We conclude that the J_E degradation in long-length Ag-sheathed Bi-2212 wire is a direct consequence of de-densification of Bi-2212 filaments due to wire swelling produced by high internal gas pressures at elevated temperatures, confirming findings in [2]. We further demonstrated that the expansion of Ag-sheathed Bi-2212 PIT wires can be well understood in terms of silver creep at high temperatures. The leakage follows the creep-rupture of Ag-sheath and it is the final manifestation of damages caused by internal gas pressure. Silver creep was due to strong hoop and radial stresses in silver matrix produced by high internal gas pressures.

Our work presents a conceptual framework to understand many puzzling experimental results such as the strong variation of J_E in 1 m long sample and to predict if, when in the processing, and how a leakage would occur in a wire. The results of this study also have important consequences to the problem of attaining high critical current densities in long length Ag-sheathed multifilamentary conductors. Our data suggest that, by developing low gas impurity conductor and by controlling the silver creep to obtain high oxide filament density, it is feasible to develop long-length Bi-2212 wire that carries a J_E(4.2 K, 20 T)

of >500 A/mm^2, which is 2.5 times higher than those being obtained presently in Bi-2212 coils; in addition, the conductor will be leak-free.

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References

1 A. Malagoli, F. Kametani, J. Jiang, U. P. Trociewitz, E. E. Hellstrom, and D. C. Larbalestier, Evidence for long range movement of Bi-2212 within the filament bundle on melting and its significant effect on J_c. *Supercond. Sci. and Technol.* **24**, 075016 (2011).

2 A. Malagoli, P. J. Lee, A. Ghosh, C. Scheuerlein, M. D. Michiel, J. Jiang, U. P. Trociewitz, E. E. Hellstrom, and D. C. Larbalestier, Evidence of length-depedent wire expansion, filament dedensification and consequent degradation of critical current density in Ag-sheathed Bi-2212 wires, to be submitted.

3 H. Kitaguchi, H. Kumakura, K. Togano, H. Miao, T. Hasegawa, and T. Koizumi, $Bi_2Sr_2CaCu_2$ O_x/Ag multilayer tapes with J_c(4.2 K, 10 T) of 500,000 A/cm^2 by using PAIR process. *IEEE Trans. on Appl. Supercon.,* **9**, 1794 (1999).

4 K. Marken, W. Dai, L. Cowey, S. Ting, and S. Hong, Progress in BSCCO-2212/silver composite tape conductors. *IEEE Trans. on Appl. Supercon.,* **7**, 2211 (1997).

5 K. Heine, J. Tenbrink, and M. Thoner, High field critical current densities in $Bi_2Sr_2CaCu_2O_{8+x}$/Ag wires. *Applied Physics Letters* **55**, 2441 (1989).

6 K. R. Marken, H. Miao, M. Meinesz, B. Czabaj, and S. Hong, BSCCO-2212 conductor development at Oxford Superconducting Technology. *IEEE Trans. on Appl. Supercon.* **13**, 3335 (2003).

7 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. on Appl. Supercon.* **15**, 2554 (2005).

8 W. T. Nachtrab, C. V. Renaud, T. Wong, X. T. Liu, T. M. Shen, U. P. Trociewitz, and J. Schwartz, Development of High Superconductor Fraction $Bi_2Sr_2CaCu_2O_x$/Ag Wire for MRI. *IEEE Trans. on Appl. Supercon.* **18**, 1184 (2008).

9 T. Shen, X. Liu, U. P. Trociewitz, and J. Schwartz, Electromechanical Behavior of $Bi_2Sr_2CaCu_2O_x$ Conductor Using a Split Melt Process for React-Wind-Sinter Magnet Fabrication. *IEEE Trans. on Appl. Supercon.* **18**, 520, (2008).

10 J. Jiang, W. L. Starch, M. Hannion, F. Kametani, U. P. Trociewitz, E. E. Hellstrom, and D. C. Larbalestier, Doubled critical current density in Bi-2212 round wires by reduction of the residual bubble density. *Supercond. Sci. and Technol.* **24**, 082001 (2011).

11 H. W. Weijers, Q. Y. Hu, Y. S. Hascicek, A. Godeke, Y. Vouchkov, E. Celik, J. Schwartz, K. Marken, , W. Dai, and J. Parrell Development of 3 T class Bi-2212 insert coils for high field NMR. *IEEE Trans. on Appl. Supercon.* **9**, 563 (1999).

12 T. Kuroda, Y. Tanaka, K. Togano, Y. Suga, T. Sakamoto, Y. Abe, K. Miura, T. Yanagiya, and M. Ishizuka, Critical current densities of AgCu-sheathed Bi-2212 multifilamentary round wires in long length. *Physica C* **357-360, Part 2**, 1098 (2001).

13 C. M. Friend, M. Hanping, Y. Huang, Z. Melhem, F. Domptail, M. Meinesz, H. Seung, E. A. Young, Y. Yang. The Development of High Field Magnets Utilizing Bi-2212 Wind & React Insert Coils. *IEEE Trans. on Appl. Supercon.* **20**, 583 (2010).

14 J. Schwartz, T. Effio, X. Liu, Q. V. Le, A. L. Mbaruku, H. J. Schneider-Muntau, T. Shen, H. Song, U. P. Trociewitz, X. Wang, H. W. Weijers, High Field Superconducting Solenoids Via High Temperature Superconductors. *IEEE Trans. on Appl. Supercon.* **18**, 70 (2008).

15 A. Godeke, D. Cheng, D. R. Dietderich, C. D. English, H. Felice, C. R. Hannaford, S. O. Prestemon, G. Sabbi, R. M. Scanlan, Y. Hikichi, J. Nishioka, T. Hasegawa, Development of Wind-and-React Bi-2212 Accelerator Magnet Technology. *IEEE Trans. on Appl. Supercon.* **18**, 516, (2008).

16 A. Godeke, D. W. Cheng, D. R. Dietderich, C. R. Hannaford, S. O. Prestemon, G. Sabbi, X. R. Wang, Y. Hikichi, J. Nishioka, and T. Hasegawa, Progress in Wind-and-React Bi-2212 Accelerator Magnet Technology. *IEEE Trans. on Appl. Supercon.* **19**, 2228, (2009).

17 M. Dalban-Canassy, D. A. Myers, U. P. Trociewitz, J. Jiang, E. E. Hellstrom, Y. Viouchkov, and D. C. Larbalestier, A study of the local variation of the critical current in Ag-alloy clad, round wire $Bi_2Sr_2CaCu_2O_{8+x}$ multi-layer solenoids. *Supercond. Sci. and Technol.* **25**, 115015 (2012).

18 N. Cheggour, X. F. Lu, T. G. Holesinger, T. C. Stauffer, J. Jiang, and L. F. Goodrich, Reversible effect of strain on transport critical current in $Bi_2Sr_2CaCu_2O8_x$ superconducting wires: a modified descriptive strain model. *Supercond. Sci. and Technol.* **25**, 015001 (2012).

19 T. Haugan, S. Chen, S. Patel, F. Wong, P. Bush, D. T. Shaw, Pinhole defects in Ag sheath of PIT Bi-2212 tapes. *Cryogenics* **35**, 853 (1995).

20 T. Haugan, M. Pitsakis, J. Ye, F. Wong, S. Patel, D. T. Shaw, Fabrication of 90-m length $Bi_2Sr_2CaCu_2O_{8+x}$ Ag-sheathed superconducting tapes. *Appl. Supercond.* **3**, 85 (1995).

21 E. E. Hellstrom, W. Zhang, Formation and prevention of bubbles when melt processing Ag-sheathed $Bi_2Sr_2CaCu_2O_x$ (2212) conductors. *Supercond. Sci. and Technol.* **8**, 317 (1995).

22 T. Kanai, T. Kamo, Control of oxygen release from Bi-2212 phase in a partial melt process. *Supercond. Sci. and Technol.* **6**, 510 (1993).

23 S. Patel, T. Haugan, D. T. Shaw, Deformation of silver-sheathed oxide superconducting tapes by trapped gases. *Cryogenics* **34**, 1031 (1994).

24 Q. B. Hao, C. S. Li, S. N. Zhang, L.F. Bai, X. B. Ma, J. Q. Feng, Modified heat-treatment process of precursor powders for Bi-2212 tapes preparation. *Physics Procedia* **27**, 268 (2012).

25 W. Zhang, E. E. Hellstrom, The influence of carbon on melt processing Ag sheathed Bi2Sr2CaCu2O8 tape. *Physica C* **234**, 137 (1994)

26 T. Kuroda, T. Nakane, H. Kumakura, Y. Suga, K. Miura, The influence of carbon on the critical current densities of melt processed AgCu-sheathed Bi-2212 wires. *Physica C* **412-414, Part 2**, 1167 (2004)

27 T. Shen, *Processing, microstructure, and critical current density of Silver-sheathed $Bi_2Sr_2CaCu_2O_x$ multifilamentary round wire* Ph.D. thesis, Florida State University, (2010).

28 T. Shen, J. Jiang, F. Kametani, U. P. Trociewitz, D. C. Larbalestier, and E. E. Hellstrom, Heat treatment control of Ag-$B_2Sr_2CaCu_2O_x$ multifilamentary round wire: investigation of time in the melt. *Supercond. Sci. and Technol.* **24**, 115009 (2011).

29 T. Shen, J. Jiang, F. Kametani, U. P. Trociewitz, Larbalestier, D. C., Schwartz, J. & Hellstrom, E. E. Filament to filament bridging and its influence on developing high critical current density in multifilamentary $Bi_2Sr_2CaCu_2O_x$ round wires. *Supercond. Sci. and Technol.* **23**, 025009 (2010).

30 T. Shen, J. Jiang, A. Yamamoto, U. Trociewitz, J. Schwartz, E. Hellstrom, D. Larbalestier, Development of high critical current density in multifilamentary round-wire $Bi_2Sr_2CaCu_2O_x$ by strong overdoping. *Appl.Phys. Lett.* **95**, 152516 (2009).

31 F. Kametani, T. Shen, J. Jiang, C. Scheuerlein, A. Malagoli, M. D. Michiel, Y. Huang, H. Miao, J. A. Parrell, E. E. Hellstrom, and Larbalestier, D. C. Bubble formation within filaments of melt-processed Bi2212 wires and its strongly negative effect on the critical current density. *Supercond. Sci. and Technol.* **24**, 075009 (2011).

32 C. Scheuerlein, M. D. Michiel, M. Scheel, J. Jiang, F. Kametani, A. Malagoli, E. E. Hellstrom, and D. C. Larbalestier, Void and phase evolution during the processing of Bi-2212 superconducting wires monitored by combined fast synchrotron micro-tomography and x-ray diffraction. *Supercond. Sci. and Technol.* **24**, 115004 (2011).

33 R. Soika, L. Cooley, A. Ghosh, A. Werner, in *AIP Conf. Proc.* **711**, 67 (2004)

34 G. R. Leverant, F. V. Lenel, G. S. Ansell, The high-temperature steady-state creep of pure silver and internally oxidized silver-magnesium alloys. *ASM Trans. Q.* **59**, 890 (1966).

35 T. G. Nieh, W. D. Nix, A study of intergranular cavity growth in Ag + 0.1% MgO at elevated temperatures. *Acta. Metall. Mater.* **27**, 1097-1106 (1979).

36 K. C. Goretta, W. E. Delaney, J. L. Routbort, J. Wolfenstine, W. Zhang, E. E. Hellstrom, Creep of silver at 900 °C. *Supercond. Sci. and Technol.* **9**, 422 (1996).

37 K. C. Goretta, J. L. Routbort, R. L. Thayer, J. P. Carroll, J. Wolfenstine, J. Kessler, J. Schwartz, Deformation of Ag/1.2 at.% Mg. *Physica C* **265**, 201 (1996).

38 S. J. Farlow, *Partial differential equations for scientists and engineers*. (Dover, 1993).

39 M. Aachib, M. Aubertin, M. Mbonimpa, in *CGS-IAH Conference* (Niagara Falls, 2002).

40 R. J. Millington, Gas Diffusion in Porous Media. *Science* **130**, 100 (1959).

41 T. Ramanarayanan, R. Rapp, The diffusivity and solubility of oxygen in liquid tin and solid silver and the diffusivity. *Metall. Mater. Trans. B* **3**, 3239 (1972).

42 T. Kanai, N. Inoue, Development of gas pressure melting (GPM) method for Ag-sheathed Bi-2212 wires. *J. of Mater. Sci.* **30**, 3200 (1995).

43 J. L. Reeves, M. Polak, W. Zhang, E. E. Hellstrom, S. E. Babcock, D. C. Larbalestier, N. Inoue, N., and M. Okada, Overpressure processing of Ag-sheathed Bi-2212 tapes. *IEEE Trans. on Appl. Supercon.* **7**, 1541 (1997)

44 J. Jiang, E. E. Hellstrom, and Larbalestier, D. C. private communication (2012).