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# Effect of sheath material and reaction overpressure on Ag protrusions into the TiO<sub>2</sub> insulation coating of Bi-2212 round wire

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**Abstract.** In order to develop a high current density in coils, Bi-2212 wires must be electrically discrete in tight winding packs. It is vital to use an insulating layer that is thin, fulfils the dielectric requirements, and can survive the heat treatment whose maximum temperature reaches 890 °C in oxygen. A thin (20-30 μm) ceramic coating could be better as the insulating layer compared to alumino-silicate braided fiber insulation, which is about 150 μm thick and reacts with the Ag sheathed Bi-2212 wire during heat treatment. At present, TiO<sub>2</sub> seems to be the most viable ceramic material for such a thin insulation because it is chemically compatible with Ag and Bi-2212 and its sintering temperature is lower than the maximum temperature used for the Bi-2212 heat treatment. However, recent tests of a large Bi-2212 coil insulated only with TiO<sub>2</sub> showed severe electrical shorting between the wires after over pressure heat treatment (OPHT). The origin of the shorting was frequent silver protrusions into the porous TiO<sub>2</sub> layer that electrically connected adjacent Bi-2212 wires. To understand the mechanism of this unexpected behaviour, we investigated the effect of sheath material and hydrostatic pressure on Ag protrusions. We found that Ag protrusions occur only when TiO<sub>2</sub>-insulated Ag-0.2%Mg sheathed wire (Ag(Mg) wire) undergoes OPHT at 50 bar. No Ag protrusions were observed when the TiO<sub>2</sub>-insulated Ag(Mg) wire was processed at 1 bar. The TiO<sub>2</sub>-insulated wires sheathed with pure Ag that underwent 50 bar OPHT were also free from Ag protrusions. A key finding is that the Ag protrusions from the Ag(Mg) sheath actually contain no MgO, suggesting that local depletion of MgO facilitates local, heterogeneous deformation of the sheath under hydrostatic overpressure. Our study also suggests that predensifying the Ag(Mg) wire before insulating it with TiO<sub>2</sub> and doing the final OPHT can potentially limit Ag protrusions.

## 1. Introduction

Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>x</sub> (Bi-2212) is the only high temperature superconductor available in a round wire form that has a very high critical current density ( $J_c$  is around  $10^5$  A cm<sup>-2</sup> up to 45 T) and irreversibility field (>100 T) at 4.2 K [1,2]. This round wire geometry is preferred by magnet designers and builders as it can be easily twisted and cabled into desired shapes. Thus Bi-2212 round wire is an attractive candidate for very high field applications that are beyond the range achievable by using Nb<sub>3</sub>Sn and Nb-Ti



technology [3-6]. In order to attain high  $J_c$ , the Bi-2212 round wire undergoes an overpressure heat treatment (OPHT) at 50 bar with a 1 bar oxygen partial pressure ( $PO_2$ ) that prevents the formation of large gas bubbles that significantly degrade  $J_c$  in 1 bar processing [4, 7]. High superconducting intergrain connectivity is developed by the highly-aligned grains resulting from the strong quasi-biaxial texture that forms in a narrow filament cavity in the round wire architecture [8]. Bi-2212 wires are made with the Bi-2212 powder in contact with pure Ag because of the good chemical compatibility between them. But, pure Ag is weak (yield strength 45 MPa at 4.2 K after OPHT), so the wire is strengthened by using a stronger outer sheath that is usually a dispersion strengthened Ag-0.2 wt% Mg (hereafter Ag(Mg)) [9].

One of the essential components of any superconducting magnet is electrical insulation, which prevents electrical shorting while charging the magnet to the operating current or discharging during a quench. For Bi-2212, the insulation is applied to the as-drawn wire and it must survive being wound into a coil, as well as the heat treatment, which has a maximum temperature around 890 °C in an oxidizing environment. Also, it has to be chemically compatible with the Ag(Mg) sheath and with Bi-2212, and it has to have sufficient dielectric strength so that the thin layer of insulation provides adequate electrical standoff within the coil winding pack while maintaining a high conductor packing factor. The alumino-silicate braid Bruker-Oxford Superconducting Technology has used to insulate Bi-2212 wires is quite thick (about 150  $\mu\text{m}$ ) and reduces the winding current density in coils. This insulation material also absorbs Ag from the sheath and Cu from the Bi-2212 core during the heat treatment, which reduces  $J_c$  by 15-20% compared to bare wire [10]. Considerable efforts have been made over the years to develop an effective insulation layer for Bi-2212 that is thin, chemically compatible, has good adhesive properties, and is viable for long length coating [11-15].  $TiO_2$  insulation developed by nGimat LLC and by Kandel *et al.* appeared to be a successful solution for insulating Bi-2212 [16, 17]. The thin (10-30  $\mu\text{m}$ )  $TiO_2$  coating has sufficient scrape resistance in both as-deposited and heat treated condition, is chemically compatible with Bi-2212 and Ag(Mg), and has high breakdown voltage and dielectric strength [17-19].

However, a test coil at the National High Magnetic Field Laboratory (NHMFL) that used Ag(Mg) Bi-2212 wire insulated with  $TiO_2$  suffered from severe electrical shorting after OPHT. Post-mortem microstructural studies revealed that severe Ag protrusion through the insulation layer caused the shorting (figure 1). Here we report on a detailed investigation to determine what causes this Ag protrusion. We used Bi-2212 wires with both Ag(Mg) and pure Ag sheaths and carried out heat treatments at 50 bar (OPHT) and at 1 bar total pressure. Our results suggest that the combination of a Ag(Mg) sheath that is coated with  $TiO_2$ , and the overpressure that is applied during the OPHT causes the Ag protrusions. We also found that we can potentially limit Ag protrusions into  $TiO_2$  by predensifying the Ag(Mg) wire before coating it with  $TiO_2$  and then winding the coil and doing the final OPHT [20].

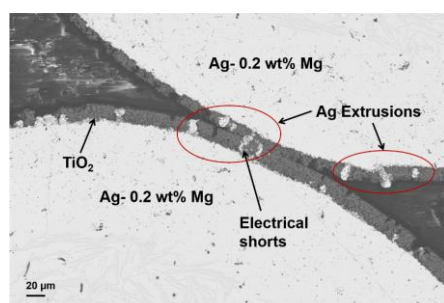
## 2. Experimental procedure

All the wires used in this study were fabricated by Bruker-Oxford Superconducting Technology (Bruker-OST) using the powder in tube (PIT) method with Bi-2212 powder in a composition of  $Bi_{2.17}Sr_{1.74}Ca_{0.89}Cu_{2.00}O_x$  produced by Nexans. The wire architecture was a double restack with the Bi-2212 powder in contact with pure Ag. The outer sheath was either Ag-0.2wt%Mg (referred to as Ag(Mg) wire) or pure Ag to study the effect of different sheath materials. The diameter of the as-drawn Ag(Mg) wire and pure Ag sheathed wire without insulation were 1 mm and 1.3 mm respectively. The as-drawn wires were dip coated with the  $TiO_2$  slurry at the NHMFL, followed by a burn out heat treatment at 450 °C in 1 bar flowing  $O_2$  to remove the organics from insulation layer [18]. A systematic variation in coating thickness from the dip coating process was observed along the wire circumference. The thinnest region was around 13  $\mu\text{m}$  thick, which continuously increased to 23  $\mu\text{m}$  thickness at the opposite side of the wire. Careful observation of transverse cross-sections revealed that the coating thickness was the same (between 13 and 23  $\mu\text{m}$ ) after 1 bar and 50 bar heat treatment.

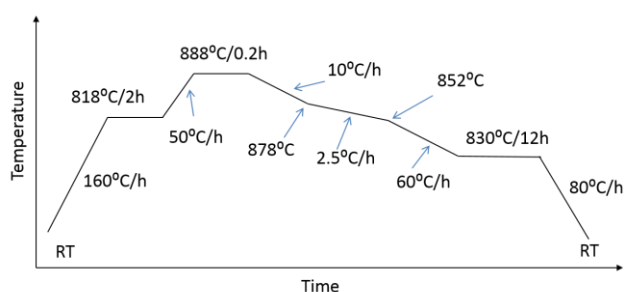
The wires were then heat treated in a flowing gas system following the heat treatment procedure shown in figure 2 [21]. Heat treatments were carried out at both 1 bar and 50 bar (OPHT) total pressure.

The 1 bar heat treatments were done in pure O<sub>2</sub> and the OPHT were done in a Ar-2%O<sub>2</sub> gas mixture at 50 bar total pressure, both of which maintained PO<sub>2</sub> = 1 bar during the heat treatment. 8 cm long samples were used because earlier observations showed that Ag protrusions also occurred in these short samples. For the 1 bar heat treatment, the wire ends were kept open to allow gas (typically CO<sub>2</sub> and H<sub>2</sub>O) that was present in the as-drawn wire to escape out the ends, thus avoiding swelling and degradation of the wire. In contrast, both ends of the wires that underwent OPHT were hermetically sealed with a Ag-bead using a torch.

Cross-sections of the heat treated wires were dry polished using SiC papers to 800 grit, followed by final polishing in an automatic vibratory polisher (Buehler Vibromet 2) using a suspension of 50 nm alumina powder in methanol. A Zeiss 1540EsB scanning electron microscope (SEM) was used to observe the microstructure. We prepared thin lamellae for STEM observations using a focused ion beam (FIB) in the Zeiss 1540 EsB. A JEOL ARM200cF was used to perform high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) imaging.



**Figure 1.** Transverse cross section from the shorted coil. The circled regions show Ag protrusions (white) in the TiO<sub>2</sub> insulation. Some of the protrusions shown in the middle of the figure connect adjacent Bi-2212 wires, causing the shorting.



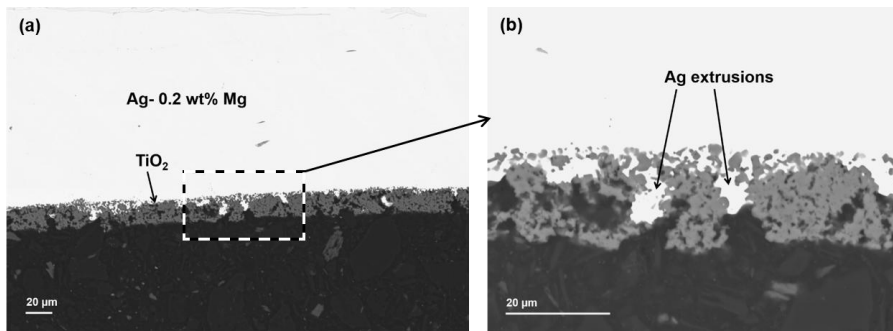
**Figure 2.** Heat treatment schedule for Bi-2212. This heating schedule was used for the 50 bar OPHT and for the 1 bar heat treatment.

### 3. Results

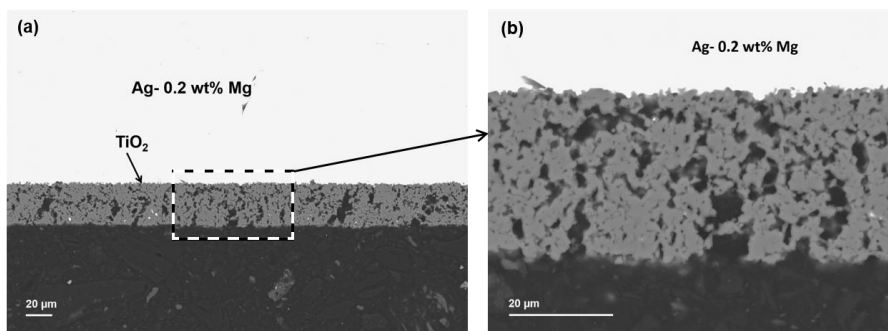
#### 3.1. Effect of external pressure

To investigate the effect that external pressure during the heat treatment has on Ag protrusion into TiO<sub>2</sub>, samples of Ag(Mg) wire were heat treated at 1 and 50 bar total pressure. As seen in the longitudinal cross-section in figure 3, the Ag(Mg) wire that underwent OPHT has frequent Ag protrusions into the TiO<sub>2</sub>, but there are fewer protrusions than in the shorted coil shown in figure 1. We observed a longitudinal section of the interface between the sheath and the insulation that was about 4.5 cm long, and found that the Ag protrusions were randomly distributed along the entire length. The Ag protrusions were irregular in shape and size, and most of them did not penetrate the entire thickness of the TiO<sub>2</sub> layer. Previous studies in our lab showed that cracks and porosity formed in the insulation layer during the sintering of TiO<sub>2</sub> particles [17, 18]. Thus, the Ag protrusions did not crack the insulation, rather they grew into the pre-existing gaps and pores in the TiO<sub>2</sub> insulation.

No Ag protrusions were observed in the Ag(Mg) wire heat treated at 1 bar (figure 4), even though cracks and porosity in the TiO<sub>2</sub> layer were present to the same extent as observed in the OPHT sample (figure 3). The interface between the sheath and insulation was well defined.



**Figure 3.** (a) SEM image of the interface between the Ag(Mg) sheath and the TiO<sub>2</sub> insulation in a Ag(Mg) wire after 50 bar OPHT (longitudinal cross section). (b) Higher magnification image of a region in (a).



**Figure 4.** (a) SEM image of the interface between the Ag(Mg) sheath and the TiO<sub>2</sub> insulation in a Ag(Mg) wire after 1 bar processing (longitudinal cross section). (b) Higher magnification image of a region in (a). Note: The thickness of the TiO<sub>2</sub> layer is greater than 23 μm in this longitudinal cross section because the section is not at the exact middle of the wire.

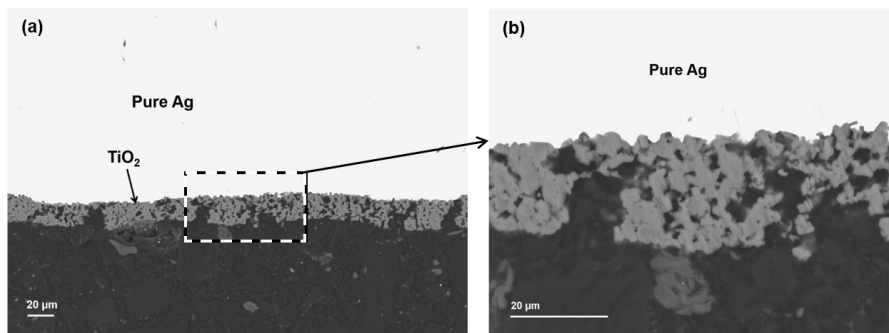
### 3.2. Effect of sheath material

We also applied OPHT on wire with a pure Ag sheath to understand what role the composition of the sheath plays in Ag protrusion. Figure 5 shows there were no Ag protrusions and that there was a well-defined interface between the pure Ag sheath and the TiO<sub>2</sub> insulation layer. This was identical to the behaviour of Ag(Mg) wire heat treated at 1 bar (figure 4).

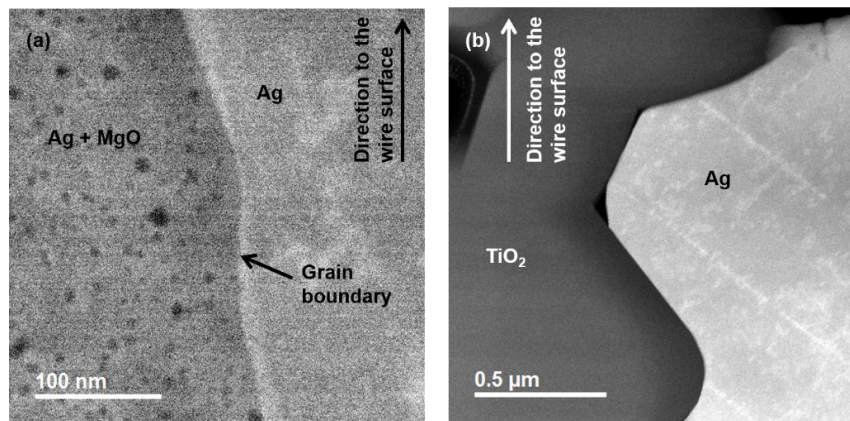
### 3.3. HAADF-STEM images

The HAADF-STEM image of figure 6(a) shows the distribution of MgO in two adjacent grains in the Ag-Mg sheath at a depth of ~8 μm from the surface of the bare wire (this wire was not coated with TiO<sub>2</sub> insulation) in a OPHTed Ag(Mg) wire. It is apparent from the figure that the density and distribution of MgO varies significantly between the two grains, one of them being devoid of MgO while the other had a uniform distribution of spherical MgO precipitates. In general, we found that the density of MgO particles is significantly lower near the wire surface than in the middle of the Ag(Mg) sheath. Figure 6(b) shows the HAADF-STEM image of a Ag protrusion in the TiO<sub>2</sub>. Interestingly, the Ag protrusion appeared to be completely devoid of MgO precipitates. A sharp interface was observed between the Ag and TiO<sub>2</sub>, which indicates no chemical reaction occurred between Ag and TiO<sub>2</sub>, confirming that they are chemically compatible.





**Figure 5.** (a) SEM image of the interface between the Ag sheath and the TiO<sub>2</sub> insulation in a wire with a pure Ag sheath after OPHT (longitudinal cross section). (b) Higher magnification image of a region in (a).



**Figure 6.** HAADF-STEM images of (a) grains within the Ag(Mg) alloy sheath. The darker round shaped particles are MgO. This sample was OPHTed without a TiO<sub>2</sub> coating. (b) A Ag protrusion in the TiO<sub>2</sub> insulation layer. The brighter contrast inside the Ag is due to defects introduced by the Ga ion beam during sample preparation by using focused ion beam. This samples was OPHTed with a TiO<sub>2</sub> coating.

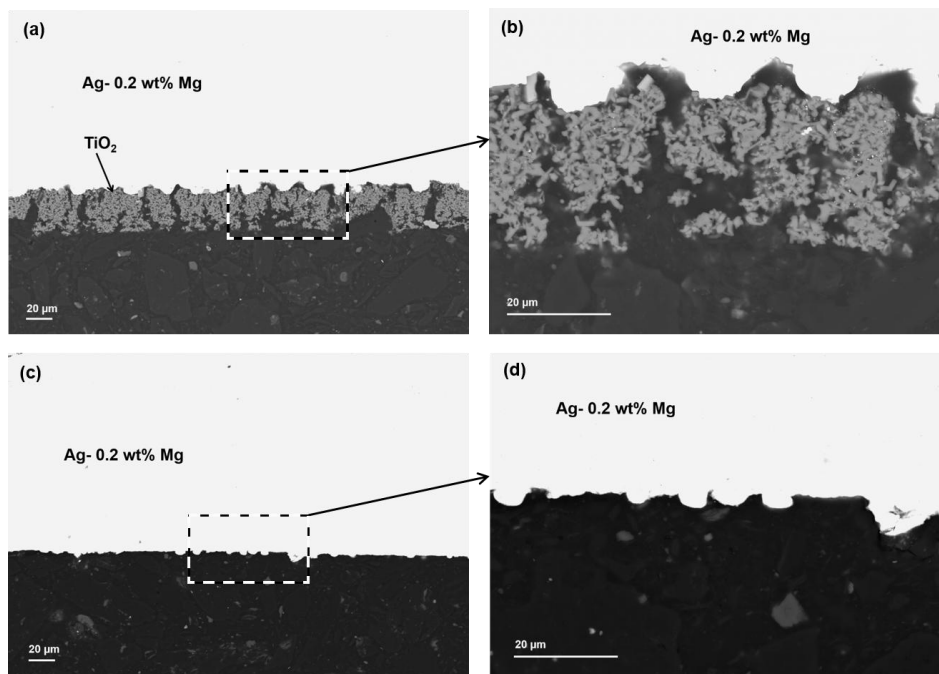
### 3.4. Effect of predensification

To understand if densifying the wire before the application of insulation can prevent Ag protrusion into TiO<sub>2</sub>, a small coil with Ag(Mg) wire was heated to 820 °C for 2 h at 50 bar (1 bar PO<sub>2</sub>) with a heating and cooling rate of 160 °C/h following the procedure in [19]. The coil was then unwound, the TiO<sub>2</sub> insulation coating was applied, followed by rewinding, organic burn out, and OPHT at 50 bar. As figures 7 (a, b) show, no Ag protrusions were observed in this predensified wire. However, small, smooth, regularly-spaced, undulations on the surface of the Ag(Mg) sheath were observed. We have also checked the microstructure of Ag(Mg) wires without insulation that had been predensified separately and did not undergo OPHT (figure 7 (c, d)). They showed undulations of similar shape but smaller size and shorter periodicity.

## 4. Discussion

The Ag(Mg) sheath is used for Bi-2212 wires, because, when the alloy is oxidized, MgO precipitates form in the Ag that strengthen the Ag sheath. It was expected that the MgO particles in the heat-treated wires would be The Ag(Mg) sheath is used for Bi-2212 wires, because, when the alloy is oxidized, MgO

precipitates form in the Ag that strengthen the Ag sheath. It was expected that the MgO particles in the heat-treated distributed uniformly; however, we found a non-uniform MgO particle distribution in the Ag(Mg) after the OPHT. In particular, the MgO density is significantly reduced and nonuniform near the wire surface. This nonuniform distribution of MgO likely causes a local variation in mechanical properties between different regions in the sheath. We expect the regions with higher MgO content to be stronger and harder than regions with lower MgO content. Our hypothesis is that, while the external pressure provided by the isostatic gas pressure during OPHT is isotropic, the regions with different MgO content respond differently to this applied pressure, causing MgO-free Ag to be squeezed out of the matrix and into the insulation. We expect that the MgO-poor, weaker regions in the Ag(Mg) form the protrusions, which is based on the lack of MgO precipitates in the Ag protrusions shown in figure 6(b). At this point, we do not know what causes this variation in MgO distribution. The wire with the pure Ag sheath that was OPHTed had no MgO, so we expect the mechanical properties of the sheath to be uniform, so there was no Ag protrusion.



**Figure 7.** (a, b) SEM image of the interface between the Ag(Mg) sheath and the TiO<sub>2</sub> insulation in a sample that was predensified, coated with TiO<sub>2</sub>, and then OPHTed. (c, d) The surface of the Ag(Mg) sheath in a sample that was only predensified and was not coated with TiO<sub>2</sub> (all images are showing longitudinal cross section).

In 1 bar processed Ag(Mg) wire, there is no external pressure gradient to cause Ag to protrude. We confirmed the need for a pressure gradient to form Ag protrusions by doing OPHT on a Ag(Mg) wire with open ends at 50 bar. With open ends, a pressure gradient could not form between the outside and inside of the wire. As with the 1 bar processed Ag(Mg) wire, no Ag protrusions formed even with 50 bar total pressure when the wire ends were open..

Matras *et al.* showed that 50 bar OPHT reduces the diameter of Bi-2212 round wires by around 4% [22]. They found that about 80% of this diameter reduction occurs while holding for 2 h at 820 °C, which is below the melting point of Bi-2212, during the standard heat treatment. Figures 7 (a, b) showed no Ag protrusions in the sample that was predensified at 820 °C before being insulated with TiO<sub>2</sub>, and then given a full OPHT. The quasiperiodic undulations on the surface of this initially predensified wire (figures 7 (a, b)) appeared underneath the insulation layer. In addition, the predensified Ag(Mg) wire

without insulation shows similarly shaped (although smaller size) undulations (figure 7 (c, d)). These observations suggest that the surface undulations were formed during the predensification step where the majority of densification takes place. HAADF-STEM analysis of these undulations revealed that they too contain almost no MgO precipitates.

Based on our observations, we propose that the following conditions are needed to form Ag protrusions: (1) a sheath made from Ag(Mg) (2) the wire has to be coated with TiO<sub>2</sub> insulation before the majority of the densification due to overpressure takes place. Our results suggest that it may be possible to limit Ag protrusions by predensifying the Ag(Mg) wire before applying the TiO<sub>2</sub> insulation, followed by full OPHT.

During the review process of this paper, one of the reviewers suggested that as the wire diameter decreased in diameter by 4%, the TiO<sub>2</sub> layer had to move in the circumferential direction to adhere to the shrinking wire, and this TiO<sub>2</sub> motion scraped up nonuniformities on the surface of the Ag that became the Ag protrusions in the TiO<sub>2</sub>. However, the question is whether there is enough movement of the TiO<sub>2</sub> layer to cause Ag protrusions the size that we see in our wire. We have designed an experiment to check this hypothesis.

## 5. Conclusions

We studied the formation of Ag protrusions through the TiO<sub>2</sub> insulation layer in Bi-2212 round wires using two different sheath materials and under various heat treatment conditions. Our results suggest Ag protrusions occur only in Ag-0.2 wt% Mg sheathed wires during heat treatment under a substantial overpressure. HAADF-STEM observations showed non-uniform distribution of MgO in the Ag(Mg) sheath near the surface of the wire, which potentially leads to local variations in mechanical properties in different regions of the sheath causing Ag to protrude during OPHT. The results of our study suggests that predensifying Ag(Mg) wire below the Bi-2212 melting temperature before applying the insulation and then doing a full OPHT may be an effective way to limit Ag protrusions into the TiO<sub>2</sub> insulation layer.

## Acknowledgement:

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