

# Nanostructural Characterization of Low Resistance Joints Using Ag Pastes for $\text{GdBa}_2\text{Cu}_3\text{O}_{7-x}$ Coated Conductors

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**Abstract.**  $\text{GdBa}_2\text{Cu}_3\text{O}_{7-x}$  coated conductors were splice jointed by a face-to-face manner using a paste containing nano-sized Ag particles under a pressure of about 50 MPa at 150 °C for 1 hr. The low electrical resistance of 6 nΩ at the joint was attained. Nanostructural characterizations of the starting Ag paste and the jointed region of the coated conductors were carried out using scanning electron microscopy and transmission electron microscopy. The size of the Ag particles in the starting pastes were confirmed to be a few tens of nanometers in diameter. The size of Ag particles became larger during the jointing process. Both the surfaces of the stabilizing Ag layers were partially bonded by the Ag particles. No oxides or other elements were detected in the region of the bonding parts.

## 1. Introduction

Commercial  $\text{REBa}_2\text{Cu}_3\text{O}_{7-x}$  (REBCO, RE; Y, Gd etc) coated conductors with high critical current ( $I_c$ ) values have been produced [1-3]. These coated conductors are promising candidates for industrial applications, such as superconductor cables, magnets and medical instruments. However, it is inevitable to develop a joint technique of the coated conductors for such practical applications [4-7]. Solder jointing [4,5], low resistance jointing studies using silver diffusion [6] and a superconducting joint technique [7] have been reported. However, these joint techniques were performed at higher temperatures (~500 °C) than the oxygen annealing temperature (~500 °C) of the REBCO crystals for carrier doping, so that REBCO crystals had an oxygen deficiency. Recently, a low electric resistance joint technique was developed using a paste containing nano-sized Ag particles at 150 °C [8]. The electric resistance of 6 nΩ at the joint was attained [8]. In this study, we characterized the nanostructures of the starting Ag particles and the jointed part of the coated conductors by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) in detail.

## 2. Experimental

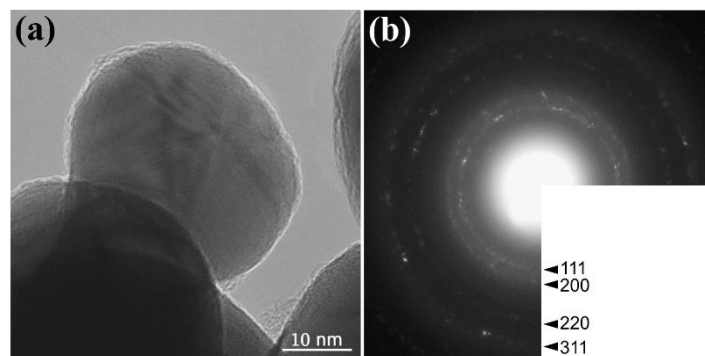
An Ag paste (L-Agl TeH, ULVAC Inc.) [8] was used as a starting paste. The paste was dispersed on a Cu grid with carbon thin film and dried. The Ag particles on the grid was characterized by TEM. A



Hastelloy™ tape with textured CeO<sub>2</sub>/LaMnO<sub>3</sub>/ion beam assisted deposition-MgO/Y<sub>2</sub>O<sub>3</sub>/Gd-Zr-O layers was used as a substrate for coated conductors. GdBCO superconductive layers were fabricated on the Hastelloy™ by pulsed laser deposition. Ag stabilizing layers were deposited by sputtering on the GdBCO layers. The surfaces of the Ag stabilizing layers were mechanically polished. These coated conductors were splice jointed by a face to face manner using a paste containing nano-sized Ag particles in air under a pressure of about 50 MPa at 150 °C for 1 hr [8]. The jointed coated conductors were sectioned by an Ar-ion beam at the accelerating voltage of 6 kV and then the cross-sections were further etched using the Ar-ion beam in a Hitachi IM4000 ion milling system. The cross-sections were observed by a Hitachi SU8000 scanning electron microscope (SEM) at the accelerating voltage of 1 kV. Furthermore, the jointed regions were thinned in a Hitachi NB5000 focused ion microscopy (FIB)-SEM system equipped with a micro-sampling system for preparation of a transmission electron microscopy (TEM) specimen, which was pasted on the edge of an Al foil support [9]. In addition, the cross-sectional specimen was further thinned using a Gatan PIPS ion milling system at an accelerating voltage of 1-0.5 kV to remove the FIB damaged layers formed on the foil specimen [9]. Both Ag particles in the starting paste and the specimen were examined in a TOPCON EM-002BF TEM with twin energy dispersive spectroscopy (EDS) detectors at the accelerating voltage of 200 kV.

### 3. Results and discussion

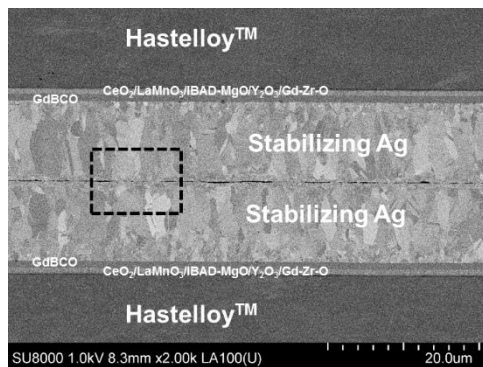
A TEM image of the Ag particles in the starting paste and a selected area diffraction pattern (SADP) of them are shown in Figs. 1 (a) and (b), respectively. The size of the Ag particles are a few tens of nanometers in diameter. However, these Ag particles are coated by some amorphous materials considered to be polymers included in the starting paste. These reflection rings in Fig. 1 (b) correspond to Ag metal.



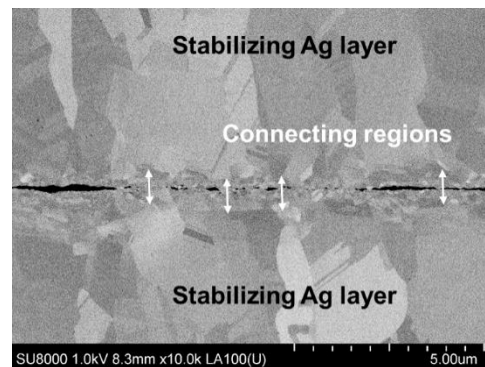
**Figure 1.** (a) TEM image of Ag particles in a starting Ag paste. (b) SADP corresponding to the Ag particles with plane indexes of Ag.

A cross-sectional SEM image of the jointed coated conductors using back scattering electrons is shown in Fig. 2. Figure. 3 shows a higher magnification SEM image of the jointed region corresponding to the broken square in Fig. 2. The stabilizing Ag layers are partially connected by the Ag particles indicated by arrows in Fig. 3. The size of Ag particles changing from the starting paste is much smaller than that of Ag grains in the stabilizing layers. A cross-sectional TEM image and a SADP of the connected region are shown in Figs. 4 (a) and (b), respectively. The size of Ag particles in the connecting region are about 50 nanometers, so that Ag particles have grown larger during the jointing process. Reflection rings correspond to Ag metal as shown in Fig. 4 (b). A cross-sectional high angle annular dark field (HAADF) image of an unconnected region of the jointed coated conductors is shown in Fig. 5. Figures. 6 (a) - (c) show the results of EDS analysis of the regions A - B in Fig. 5, and Ag particles of the starting pastes. Oxygen cannot be detected in the region A and the starting Ag particles, but can be detected in the region B. (Since the TEM specimen was pasted on the Al foil support [9], the Al peak of around 1.5 keV in Figs. 6 (a), (b) was caused from the Al supports. Therefore, neither the Ag particles nor the stabilizing

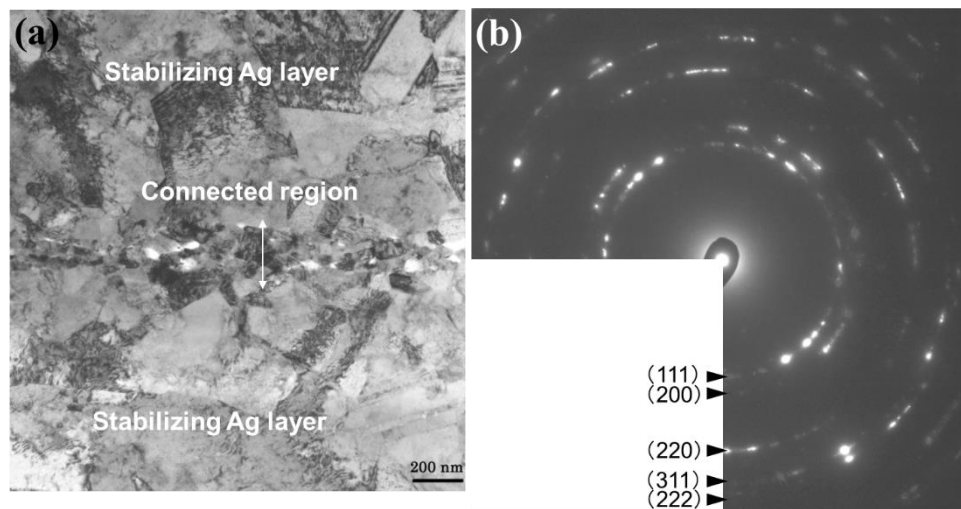
Ag layers dose not include Al. The Ag particles was put on a Cu grid with carbon thin film. The Cu peak of around 0.9 keV in Fig. 6 (c) was from the Cu grid.) Figure. 7 shows a SADP acquired from such the region containing oxygen. Reflection spots in Fig. 7 mainly correspond to Ag metal, but some spots, indicated by arrows, correspond to the (110) of  $\text{Ag}_2\text{O}$ . These oxides are consider to be formed on the unconnected regions during the jointing process. With increasing the splice jointed regions, nucleation of such the oxides on the unconnected regions could be suppressed. In addition, the electrical joint resistance between the coated conductors should be lower.



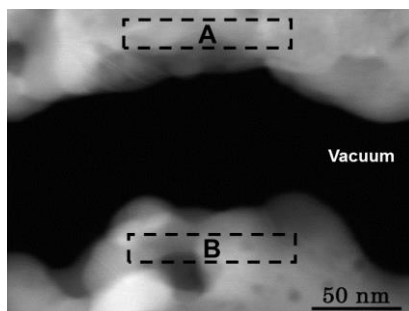
**Figure 2.** Cross-sectional SEM image of jointed GdBCO coated conductors using back scattering electrons.



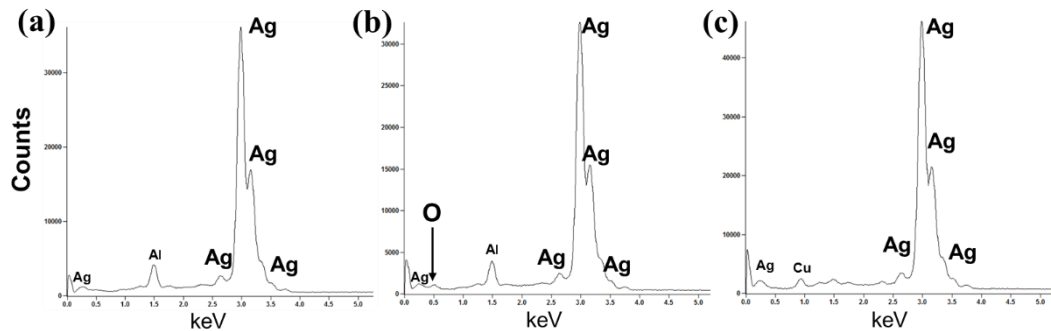
**Figure 3.** Cross-sectional SEM image of bonding regions corresponding to the broken square in Fig. 2.



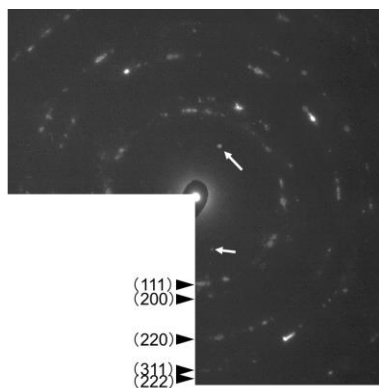
**Figure 4.** (a) Cross-sectional TEM image of a connected region and (b) SADP corresponding to the region of (a) with the plane indexes of Ag.



**Figure 5.** HAADF image of an unconnected region of the jointed coated conductors.



**Figure 6.** Results of EDS analysis of Ag particles. (a) and (b) corresponds to the regions A and B in Fig. 5, respectively. (c) Ag particles of the starting pastes.



**Figure 7.** SADP corresponding to Ag particles containing oxygen with the plane indexes of Ag. Arrows indicate reflection spots of the (110) of  $\text{Ag}_2\text{O}$ .

#### 4. Conclusions

We observed the nanostructures of the Ag particles in the starting pastes and jointed regions of GdBCO coated conductors using the Ag pastes in which the low electrical resistance of  $6 \text{ n}\Omega$  was attained. The size of Ag particles in the starting paste was a few tens of nanometers in diameter. According to the selected area diffraction patterns, no Ag oxides were found in the starting paste. Both the surfaces of the stabilizing Ag layers were partially bonded by the Ag particles. The larger the splice jointed area, the lower the electrical joint resistance.

#### Acknowledgements

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