Progress in scale-up of second-generation high-temperature superconductors at SuperPower Inc

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Abstract

SuperPower is focused on scaling up second-generation (2-G) high-temperature superconductor (HTS) technology to pilot-scale manufacturing. The emphasis of this program is to develop R&D solutions for scale-up issues in pilot-scale operations to lay the foundation for a framework for large-scale manufacturing. Throughput continues to be increased in all process steps including substrate polishing, buffer and HTS deposition. 2-G HTS conductors have been produced in lengths up to 100 m. Process optimization with valuable information provided by several unique process control and quality-control tools has yielded performances of 6000–7000 A m (77 K, 0 T) in 50–100 m lengths using two HTS fabrication processes: metal organic chemical vapor deposition (MOCVD) and pulsed laser deposition (PLD). Major progress has been made towards the development of practical conductor configurations. Modifications to the HTS fabrication process have resulted in enhanced performance in magnetic fields. Industrial slitting and electroplating processes have been successfully adopted to fabricate tapes in width of 4 mm and with copper stabilizer for cable and coil applications. SuperPower’s conductor configuration has yielded excellent mechanical properties and overcurrent carrying capability. Over 60 m of such practical conductors with critical current over 100 A/cm-width have been delivered to Sumitomo Electric Industries, Ltd. for prototype cable construction.

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1. Introduction

Second-generation (2-G) high-temperature superconductor (HTS) wires are referred as the YBa$_2$Cu$_3$O$_x$ (YBCO)-type superconducting films coated conductors while first-generation (1-G) HTS wires as Bi(Pb)SrCaCuO superconductor made in a powder-in-tube process. 2-G HTS wires have several important advantages over 1-G HTS wires including potentially high engineering current density, better in-field performance at higher temperatures, potentially low processing costs, lower ac loss, and significantly improved mechanical properties. Research institutes and industries worldwide have shifted efforts towards the commercialization of 2-G HTS wires. To obtain high critical current density in YBCO superconductors, an outstanding problem is that its grains are difficult to align. Therefore, biaxially textured buffer films or layers are necessary for successful deposition of textured YBCO films on metallic substrates. So far, a few techniques have been developed for textured buffer layers, among which ion beam assisted deposition (IBAD) [1,2], rolling assisted biaxially textured substrates (RABiTS) [3,4], and inclined-substrate deposition (ISD) [5,6] have shown the potential in commercialization. At SuperPower, the development of long-length coated conductors is based on IBAD for textured buffer layers and metal organic chemical vapor deposition (MOCVD) for HTS growth including YBa$_2$Cu$_3$O$_x$ (YBCO), whereas PLD is employed mainly to confirm the quality of layers underlying HTS. In order to meet the milestone of scaling up coated conductor processes to produce tape in piece-lengths greater than 1 km with performance greater than 100,000 A/m by mid-decade, our research and development activities have been directed towards the following key objectives:

(a) High throughput at every step of the process to demonstrate a route for low cost production.
(b) Equipment and processes suitable for long production runs.
(c) Continuous, reel-to-reel on-line and off-line quality-control and analytical tools to ensure high-quality production in fast and long runs.
(d) Robust manufacturing process for a practical conductor for prototype device demonstration.

A high throughput is essential to a large-volume production at a given equipment base, and thus to low cost fabrication, since capital equipment for manufacturing coated conductors is expensive [7]. With several equipment and process advancements, high linear tape speeds from 10 to 60 m/h have been achieved in three major steps: metal substrate polishing, IBAD-buffer and HTS deposition, while maintaining HTS performance at a level above 100 A/cm. These tape speeds were obtained in prototype, pilot-scale and preproduction manufacturing facilities that can handle tape lengths from 50 to 100 m in continuous runs. Feedback from several improved and newly added on-line and off-line quality-control and quality-analysis tools has led us to a better understanding of the relationship between microstructure and performance. This feedback has enabled optimization and stabilization of process conditions to achieve 6000–7000 A/m performances over those tape lengths. Major progress has been also made towards the development of a practical conductor configuration. Improved in-field performance has been achieved by chemical modification of the HTS layer; practical useful tape width has been enabled by slitting; and excellent mechanical properties and electrical stability have been achieved by electroplating surround Cu. These processes result in a product of an application-ready conductor.

2. Pre-HTS processes: high throughput and long tape handling capability in metal tape polishing and IBAD

Hastelloy®-C is used as the metal tape substrate. The smoothness of Hastelloy®-C surface is
very important to achieve a good texture in buffer layers by IBAD process. The required average roughness ($R_{\text{avg}}$) of the substrate is usually at the level of a few nanometers (nm) for IBAD of yttrium-stabilized zirconia (YSZ) and Gd$_2$Zr$_2$O$_7$ (GZO). However, a more stringently required roughness at $\sim$1 nm is needed for the IBAD-MgO process. This is because films only $\sim$10 nm thick are needed to optimize its texture for IBAD-MgO thus a smooth deposition surface is required to achieve an acceptable and continuous texture where as films 1–2 $\mu$m thick for IBAD-YSZ or GZO thus they are more tolerant to surface roughness [8]. In collaboration with Los Alamos National Laboratory, SuperPower has established a preproduction-scale electropolishing facility that can handle tape lengths of more than 100 m. Over 85 tapes in 100 m lengths have been polished in this facility at linear speeds of 20–60 m/h, which is more than a 10-fold increase compared to the speed of a chemical mechanical polishing process (CMP). Deposition of buffer layers on the electropolished substrates is then conducted in a pilot IBAD facility. This facility has been fitted with dual ion sources 66 cm long that provide a deposition zone 60 cm long and 7 cm wide. In order to maximize the linear speed of the IBAD process, we use a helix tape-handling system. In addition to increased linear tape speed, the helix tape-handling system enables uniform deposition over a 7 cm width. Fig. 1 depicts the recent results of the two processes: (a) 90% of the 85 polished substrates exhibit an average surface roughness ($R_{\text{avg}}$) less than 1.5 nm. (b) Average in-plane texture obtained on six IBAD tapes 100 m long fabricated in our pilot IBAD facility ranges from $10.2^\circ$ to $10.8^\circ$ with a standard deviation from 2% to 3%, as measured rapidly every 0.25 m using a novel X-ray diffraction tool [9]. We recently achieved good performance of 70 A/cm-width end-to-end over 100 m as well as 105 A/cm-width end-to-end over 57 m on coated conductors [10], which reflects the robustness of our pre-HTS processes.

We recently reported that 10 m/h tape speed was achieved in fabricating IBAD-MgO tapes up to 40 m long in a prototype facility equipped with two ion sources 22 cm long and 7 cm wide and a helix tape handling system. The in-plane texture of the MgO layer over 40 m is less than 7°. When the IBAD-MgO process is transferred to the pilot system, an increase of at least 3-fold in tape speed is expected.

3. High-rate MOCVD and improvement of performance over long length

Some early work demonstrated that high-quality YBCO films could be made by MOCVD [11,12], and the development of a liquid-precursor delivery system made this method attractive for continuous deposition. MOCVD also has the potential for the highest throughput among the techniques that are being currently pursued for deposition of the HTS layer, which was one of the key reasons MOCVD was selected as the primary method for depositing HTS on IBAD substrates at SuperPower [13]. Throughput is
determined by the size of the deposition area and the deposition rate. Deposition rates as high as 150 Å/s have been demonstrated by MOCVD, with a performance level of 1 MA/cm² using a photo-assist approach [14]. Deposition area with MOCVD can be as long and as wide as the showerhead, which is essentially unlimited.

We have established two MOCVD systems; one is a research system dedicated to process optimization for continuous runs, and the other a pilot system that was installed in June 2004 for scale-up to lengths of 100 m–2 km. Both systems use a liquid precursor delivery system that has been described in detail elsewhere [15]. The research MOCVD has a deposition area 20 cm long and a tape handling capability up to 50 m for substrates 100 μm thick with interleaf. In this system we evaluated the possibility of increasing the growth rates of HTS films for coated conductors just by increasing the precursor flow rate in our liquid-delivery system, without employing any assist mechanisms such as photoassist. We recently reported that the deposition rate reaches 120 Å/s at a flow rate of 5 mL/min, and the critical current density \( J_c \) is over 1 MA/cm² [7]. This makes it possible to fabricate tapes with \( I_c > 100 \) A/cm-width in a speed of 10 m/h. The pilot system has a larger (30–100 cm × 6 cm) deposition zone and can handle tape length up to 2 km with interleaf. The projected tape speed is 50 m/h. The functionality of this system has been quickly enabled as evidenced by \( I_c \) results of over 165 A/cm-width and \( J_c \) over 1.1 MA/cm² obtained on tapes 1–5 m long.

In the research system, we started to test 50 m continuous runs after an 18 m long tape with an end-to-end \( I_c \) of 111 A [7]. Fig. 2 shows the \( I_c \) profile over the entire length of the first 50 m run. The profile was tested by a reel-to-reel \( I_c \) test rig designed and built in-house at SuperPower. This 50 m tape showed periodic sections of low (65–100 A) and high (120–193 A) \( I_c \) within meter-long sections. Zero \( I_c \) at 26–28th m and after 46th m can be attributed to hardware issues related to precursor supply. To investigate the reasons for the periodic appearance of 50–100 A sections, detailed \( I_c \) profiles at every 1 cm interval were tested by a novel continuous test rig constructed based on a technique developed at Los Alamos National Lab-

Fig. 2. Critical current \( (I_c) \) at every meter section of the first 50 m tape processed in the prototype MOCVD system. The voltage criterion is 1 μV/cm.

Fig. 3. Data on weak section from the first 50 m run in the prototype MOCVD system.
Microstructural analysis shows that at the lowest $I_c$ value, (a) the intensity of X-ray diffraction peak YBCO(006) is minimal as shown in the same figure (solid round symbols), (b) surface morphology is rougher compared to the good area, and (c) the in-plane texture of YBCO film is $7^\circ$, whereas it is only $4^\circ$ in the good region [17]. The degraded in-plane texture at the lowest $I_c$ point cannot be attributed directly to the buffer layer since its in-plane texture is comparable in both good and bad regions (11–12°). A composition analysis by inductively coupled plasma–atomic emission spectrometry shows that the $I_c$ can also be correlated to the cation ratio of Ba in the film as shown in Fig. 3(b). These data have led us to install online quality-control tools to monitor the stability of the system. Fig. 4(a) shows that the two precursor parameters vary periodically in less than 40 min. We modified the hardware configuration and successfully extended the time period beyond 240 min as indicated in Fig. 4(b).

Besides directly solving the instability problems, the other strategy we employed to improve performance over long lengths was to process tape at high linear speed to reduce the number of instabilities encountered in a long run and then to run multiple passes to add up the total $I_c$. We reported an $I_c$ value at 100 A/cm over 5 m tape length fabricated at 32 m/h for three passes when the precursor parameters were not stabilized [17]. After the improvement in precursor parameter stabilization, we recently processed a tape 62 m long using a similar strategy. The $I_c$ measured every 1 m is shown in Fig. 5. The end-to-end $I_c$ is 100 A/cm-width tested by winding the tape on a G-10 mandrel.

**Fig. 5.** $I_c$ profile of a 62 m long MOCVD tape. The end-to-end $I_c$ is 100 A/cm-width tested by winding the tape on a G-10 mandrel.

4. Improved in-field performance

To increase the in-field $I_c$ of our coated conductors and satisfy the application requirement in magnetic fields of 1 T and higher, we developed two solutions: one to improve the retention of critical current in high magnetic fields at all field orientations by substitution of Sm in YBCO, as described in our previous publications [7,18], and the other to increase the zero-field $I_c$. Fig. 6 shows...
an example of our recent achievement in the enhancement of in-field performance. The $I_c$ was determined by transport measurements using a criterion of 1 $\mu$V/cm. The sample was patterned into a 2 mm $\times$ 10 mm bridge. Ten millimeter is the dimension along the tape length direction and transport current flow, and the field direction is always perpendicular to transport current. This Sm-substituted YBCO has a critical current value of 407 A/cm-width at zero field and 75.5 K. For a 1 T field perpendicular ($H \parallel c$) to the sample surface, Sm-substituted coated conductors exhibit a drop by only a factor of 4–5 in $I_c$ from its zero-field value, compared to a 7–10-fold reduction for typical YBCO coated conductors. The $I_c$ is 86 A/cm. As shown in the inset of this figure, $I_c$ remains high at all field orientations, and the minimum is still above 65 A at middle angles ranging from 25° to 85°.

5. Development of a practical conductor

In order to enable application readiness of coated conductors, SuperPower has developed three key processes: slitting for a suitable conductor width, Cu-plating for a surround stabilizer, and striation by a photolithographic method to reduce ac losses. These processes were described elsewhere [19]. Conductors that are suitable for different applications can be produced by varying the combination of these three post-YBCO processes and parameters for each one. Striation by photolithography has yielded reduced ac loss, and results can be found in Ref. [20]. In this section, test data evaluating the product from a specified route aiming at cable applications will be presented. These are the tapes 4 mm-wide with surround stabilizer produced by slitting wider tapes, followed by an electroplating process. Since these conductors are encapsulated by Cu, HTS as well as the entire layered structure is protected. Hermetic test is one way to examine the level of protection. We have tested more than 80 m of such conductors with about 20 $\mu$m Cu on all sides in a liquid-nitrogen environment that is pressurized to 1 MPa (10 atm) for 10 h, which are typical test conditions for 1-G HTS wires. We have observed no change in tape width, thickness and $I_c$ after the test [19,20]. Such conductors also show excellent electrical stability and mechanical properties as described in the following.

Overcurrent handling capability in a continuous mode was tested at SuperPower. Table 1 lists the
test results on two 4 mm-wide conductors with 20 \mu m Cu on all sides in comparison with those on two samples with 40 \mu m on only the YBCO side of the conductor. The table also shows results from two other tapes that had 20 \mu m Cu on all sides initially, but then the Cu on the substrate side was etched before the testing. All six samples have $I_c$ around 60 A, i.e., 150 A/cm-width, and both sides of the samples were exposed to liquid nitrogen directly. Transport current was increased at a ramp rate of 50 A/min until the samples were burned out. The table shows that the two samples with surround Cu can handle \sim 1.3 times more current and \sim 1.6 times more power density than the samples with Cu on only one side. This is due to the current sharing through the Cu on the back of the substrates and more efficient heat dissipation. The overcurrent handling capability test in a pulse mode was conducted at the Massachusetts Institute of Technology. The test was done on samples with \sim 37 \mu m Cu on all sides. The total Cu mass is comparable to having 75 \mu m Cu on one side. Two samples with $I_c$ of 105 A/cm-width and 135 A/cm-width were subjected to 300 ms and 1 s pulses, respectively. Fig. 7 shows the voltage response when the 11th 300 ms pulse was applied to the sample, which current is 9.1 times the $I_c$ of the sample. There was no substantial degradation of $I_c$ after the pulse. For 1 s pulses, current up to 3.6 times $I_c$ was applied and no obvious degradation in $I_c$ was observed.

The $I_c$ retention properties of the conductors with surround Cu stabilizer under axial tensile strain were evaluated at the National Institute of Standards and Technology (NIST). The detailed description of the test method can be found elsewhere [21]. Fig. 8 shows normalized $I_c$ vs. axial tensile strain of three tapes 4 mm-wide tested at 76 K. Tapes with Cu stabilizer show an irreversible strain ($\varepsilon_{irr}$) higher than the one with only a Ag

Table 1
Overcurrent handling capability of conductors with surround Cu stabilizer tested in continuous dc loading mode

<table>
<thead>
<tr>
<th>Conductor type</th>
<th>Conductor 1</th>
<th>Conductor 2</th>
<th>Conductor 3</th>
<th>Conductor 4</th>
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<tbody>
<tr>
<td>Single side Cu (40 \mu m)</td>
<td>233 A–18.6 W/cm²</td>
<td>205 A–18.1 W/cm²</td>
<td>158 A–15.3 W/cm² (etched out Cu from substrate side)</td>
<td>194 A–21.8 W/cm² (etched out Cu from substrate side)</td>
</tr>
<tr>
<td>“Surround stabilizer” Cu (20 \mu m all sides)</td>
<td>279 A–33.3 W/cm²</td>
<td>277 A–27 W/cm²</td>
<td>(etched out Cu from substrate side)</td>
<td></td>
</tr>
</tbody>
</table>
overlayer, indicating that the existence of Cu might play a positive role by putting a pre-compressive strain into the HTS layer while samples are cooled down to liquid nitrogen temperature for testing [21,22]. The sample with 15 \( \mu \text{m} \) surround Cu has an \( \varepsilon_{\text{IRR}} \) of 0.48\% which is slightly less than that of the one with 30 \( \mu \text{m} \) Cu only on the YBCO side. Despite the difference in \( \varepsilon_{\text{IRR}} \), the axial-strain performance of all three configurations may comfortably meet the most severe benchmarks for applications. The “tensile stress vs. strain” properties have also been tested [23]. The yield strength, as defined by a 0.2\% strain offset criterion for tapes with surround Cu stabilizer, is 454 MPa at room temperature and 640 MPa at 76 K, lower by only about 11–12\% than that for the substrate [23]. This high yield strength indicates that the IBAD-based coated conductors are twice as strong as others—both 1-G and 2-G wires. \( I_c \) retention was 100\% when such conductors were subject to tensile stresses up to 360 MPa at room temperature [19,20]. This type of conductor is also tolerant to bending and tensile strain [19].

Our practical conductors have already been used for different demonstration devices. First, 61 m of tapes 4 mm wide with surround Cu stabilizer were supplied to Sumitomo Electric Industries (SEI) to fabricate a coated conductor cable 1 m long as a part of the Albany Cable Project. The 61 m of tapes were tested and fully qualified. The 1 m cable was constructed using 48 segments 1.2 m-long. Tests at SEI showed a total dc \( I_c \) of 2150 A, which is consistent with the average \( I_c \) of all segments and self-field effects. Transport current ac losses of 0.1 W/m and 0.4 W/m were measured with fiber reinforced polymer (FRP) former and metal former, respectively, at 1000 A, 60 Hz loading current. Second, four race-track rotor coils were constructed from 26 m of tapes 1.2 mm wide plated with Cu stabilizer and used to fabricate a HTS motor by Rockwell Automation. The motor was operated as a generator at 1800 rpm and 1.2 HP. Third, we fabricated a pancake coil using 7.4 m of coated conductor with a small internal diameter of 14 mm. The coil consisted of 83 turns. \( I_c \) for the coil at 77 K was 55 A, at which current a magnetic field of 0.28 T was generated.

6. Summary

The overall progress of the scale-up of 2-G HTS wires at SuperPower is provided here with emphasis on high throughput and robust pre-YBCO processes, improved performance over long length by high-rate MOCVD, and application-ready practical conductors. Multiple IBAD tapes over 100 m long have been fabricated with uniform in-plane texture of 11–12°. A tape speed of 10 m/h has been realized in the IBAD-MgO process to process up to 40 m length with in-plane texture better than 7°. High-rate MOCVD is capable of producing 100 A/cm-width conductors at a tape speed of 20 m/h. Solving the precursor parameter instability problem together with multi-pass approaches enabled us to fabricate a tape 62 m long with an end-to-end \( I_c \) of 100 A/m-width. SuperPower has developed key processes for practical conductors. Tapes 4 mm wide with surround Cu stabilizer fabricated by a slitting process followed by electroplating show excellent environmental stability, electrical stability and electro-mechanical properties. Our practical conductors have also been used in demonstration devices, including a 1 m cable by SEI, race-track rotor coils for Rockwell Automation HTS motor, and pancake coils. These data show that our practical conductors are reaching readiness for use in HTS devices applications.

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References