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PROGRESS IN SCALE-UP OF SECOND-GENERATION HTS AT SUPERPOWER

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Abstract

SuperPower is focused on scaling up second-generation 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. Second-generation 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 6,000-7,000 amp-meters 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 has resulted in enhanced performance in magnetic fields. Industrial slitting and electroplating processes have been successfully adopted to fabricate tapes in 4mm width and with copper stabilizer for cable and coil applications. Our conductor configuration has yielded excellent mechanical properties and overcurrent carrying capability. Over 60 meters of such practical conductors with 100+ A/cm performance have been delivered to Sumitomo Electric Industries, Ltd. for prototype cable construction.

PACS codes:

Key words: 2nd-generation HTS wire, coated conductors, electropolishing, magnetic field, ac losses, narrow conductor, cable

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I. Introduction

2nd-generation (2-G) high temperature superconductor (HTS) wires have several important advantages over 1st-generation (1-G) HTS wires including potentially high engineering current density, better in-field performance at higher temperatures, potentially low processing costs, and lower ac loss. Research institutes and industries worldwide have shifted efforts towards the commercialization of 2-G HTS wires. At SuperPower, the development of long-length coated conductors is based on Ion Beam Assisted Deposition (IBAD) for textured buffer layers and MOCVD for HTS growth including $YBa_2Cu_3O_x$ (YBCO) while PLD is mainly employed 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 Amp-Meters by this mid-decade, our R&D 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 & processes suitable for long production runs;

(c) Continuous, reel-to-reel on-line & off-line quality-control and quality analysis tools to ensure high-quality production in a 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 a low-cost fabrication since capital equipment for coated conductor manufacturing is expensive [1]. With several equipment and process advancements, high linear tape speeds from

10-60 m/h have been achieved in three major steps: metal substrate polishing, IBAD-buffer and HTS deposition while maintaining HTS performance at 100 +A/cm level. These tape speeds were obtained in prototype, pilot-scale and preproduction manufacturing facilities that can handle tape lengths from 50 – 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 condition to achieve 6000-7000 Amp-Meters 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, practically 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.

II. Pre-HTS Processes: high throughput and long tape length handling capability in metal tape polishing and IBAD

Hastelloy®-C is used as the metal tape substrate. Smoothness of Hastelloy®-C surface is very important to achieve a good texture in buffer layers by IBAD process. The required average roughness (R_{avg}) of the substrate is usually at the level of a few nm for IBAD of yttrium-stabilized zirconia (YSZ) and $Gd_2Zr_2O_7$. But, a more stringently required at ~1 nm roughness is needed for IBAD-MgO process [2]. In collaboration with Los Alamos National Laboratory, SuperPower has established a preproduction-scale electropolishing facility which can handle more than 100 m tape lengths. Over 85 tapes in 100 m lengths have been polished in this facility at a linear speed of 20 to 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 66 cm long ion sources which provide a deposition zone 60 cm in length and 7 cm in width. 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_{avg}) less than 1.5 nm. (b) Average in-plane texture obtained on six 100 m long IBAD tapes fabricated in our Pilot IBAD facility ranges from 10.2 degrees to 10.8 degrees with a standard deviation from 2% to 3%, as measured rapidly in 0.25 m using a novel X-ray diffraction tool [3]. 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 [4], which reflects the robustness of our pre-HTS processes.

We recently reported that 10m/h tape speed was achieved in fabricating IBAD-MgO tapes up to 40 m long in a prototype facility equipped with two 22cm-long and 7cm-wide ion sources and a helix tape handling. 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, at least 3 fold tape speed increase in tape speed is expected.

II. High-rate MOCVD and performance improvement over long length

Some early work demonstrated that high quality YBCO films could be made by MOCVD [5, 6], and the development of 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 for MOCVD to be selected as the primary method for depositing HTS on IBAD substrates at SuperPower [7]. Throughput is determined by the size of the deposition area and the deposition rate. Deposition rates as high as 150 Angstroms/sec have been demonstrated by MOCVD with a performance level of 1 MA/cm^2 using photo-assist approach [8]. 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 which was installed in June 2004 for scale up to 100 m to 2 km lengths. Both systems use a liquid precursor delivery system that has been described in detail elsewhere [9]. The research MOCVD has a 20cm long deposition area and tape handling capability up to 50m for 100 μm thick substrates 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 photo assist. We recently reported that the deposition rate reaches 120 Å/s at a flow rate of 5 mL/min and critical current density (J_c) is over 1 MA/cm^2 [1]. 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 (30-100cm x 6cm) deposition zone and can handle tape length up to 2 km with interleaf. The projected tape speed is 50m/h. The functionality of this system has been quickly enabled as evidenced by I_c results of over 165A/cm-width and J_c over 1.1MA/cm^2 obtained on 1-5 meter long tapes.

In the research system, we started to test 50m continuous runs after an 18m long tape with end-to-end I_c of 111 A was fabricated [1]. 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 26th -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 1cm interval were tested by a novel continuous test rig constructed based on a technique developed at Los Alamos National Laboratory [10]. Fig. 3 (a) is the I_c in a field of 0.6 T perpendicular to the tape surface at every 1cm of a 75 A section. It is shown that I_c decreases gradually over ~30 cm length, and increases rapidly within a couple of centimeters to high values after reaching the minimum value. All 50-100 A sections show similar I_c variation as indicated by over 5000 data points. Microstructural analysis shows that at the lowest I_c value, the intensity of x-ray diffraction peak YBCO (006) is minimal as shown in the same figure (solid round symbols), surface morphology is rougher compared to the good area, and the in-plane texture of YBCO film is 7° while it is only 4° in the good region [11]. The worsened in-plane texture at the lowest I_c point can not be attributed directly to the buffer layer since its in-plane texture is comparable at both good and bad regions (11-12°). A composition analysis by Ion Coupled Plasma Spectroscopy 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 on-line quality control tools to monitor the stability of the system. Fig.4 (a) shows the two precursor parameters vary periodically in less than 40 minutes (a). We modified the hardware configuration and successfully extended the periodic time 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 length 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 total I_c. We reported an I_c value at 100 A/cm over 5 m tape length fabricated at 32 m/h for 3 passes when the precursor parameters were not stabilized [11]. After the precursor parameter stabilization improvement, we processed a 62m long tape using similar strategy recently. The I_c of every 1m is shown in Fig. 5. The end-to-end I_c is 100 A/cm as tested by winding the tape on a G-10 mandrel and this raises the performance level from the previous 2000 amp-meters to 6200 amp-meters for the best MOCVD-based long-length coated conductors. In that 3-pass run, precursor parameter instability was not observed. I_c of less than 100 A/cm was still observed at a few meter sections while it is as high as 172 A/cm for others. Reason for such variation is under investigation.

III. 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 Sm substitution in YBCO as described in our previous publications [1, 12], and the other to increase the zero–field I_c. Fig. 6 shows an example of our recent achievement on the enhancement of in-field performance. I_c was determined by transport measurement using a criterion of 1μV/cm. The sample was patterned into a 2 mm x 10 mm bridge. 10mm is the dimension along the tape length direction and transport current flow, and 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//c) to sample surface, Sm-substituted coated conductors exhibit only a factor of 4 to 5 drop in I_c from its zero-field value, compared to a 7 – 10 fold reduction for a typical YBCO coated conductors, and I_c is 86A/cm. As shown in the insert of this figure, I_c remains high at all field orientations, and the minimum is still above 65 A at middle angles ranging from 25-85 deg.

IV. 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 photolithographic method to reduce AC losses. These processes were described elsewhere [13]. 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 photolithograph has been yielded ac loss reduction, and results can be found elsewhere [13]. In this section, test data from evaluating the product from a specified route aiming at cable applications will be presented. This is the 4mm-wide tapes with surround stabilizer produced by slitting wider tapes followed by an electroplating process. Since this type of conductors is 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 μm Cu on all sides in a LN_2 environment that is pressurized to 10 atm for 10 hours, 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 [13, 14]. 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 I lists the test results on two 4 mm-wide conductors with 20 μm Cu on all sides in comparison with two samples with 40 μm only on the YBCO side of the conductor. The Table also shows results from two other tapes which had 20 μm Cu on all sides initially, but then the Cu on the substrate side etched out before the testing. All six samples have I_c around 60A. i.e., 150 A/cm-width, and both sides of the samples were exposed to LN_2 directly. Loading current was increased at a ramping rate of 50 Amp/min until the samples were burned out. It is shown from the table that the two samples with surround Cu can handle ~1.27 times more current and ~1.64 times more power density than the samples with Cu only on one side. This is due to the current sharing through the Cu on the back of the substrates and heat dissipation is more efficient. The overcurrent handling capability test in a pulse mode was conducted at Massachusetts Institute of Technology. The test was done on samples with ~37 μm Cu on all sides. The total Cu mass is comparable to having 75 μm Cu on one side. Two samples with Ic of 105A/cm-width and 135A/cm-width were subjected to 300 ms and 1 sec pulses, respectively. Fig.7 shows the voltage response when the 11th 300 ms pulse was applied to the sample, which is 9.1 times of the I_c of the sample. There was no substantial I_c degradation after the pulse. For 1 sec pulses, current up to 3.6 times I_c was applied and no obvious I_c degradation 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 [15]. Fig. 8 shows normalized I_c vs. axial tensile strain of three 4 mm-wide tapes tested at 76 K. Tapes with Cu stabilizer show higher irreversible strain (ε_{irr}) than the one with only a Ag overlayer, indicating that the existence of Cu might play a positive role by putting a pre-compressive stress or strain into HTS layer while samples are cooled down to LN_2 temperature for testing. The sample with 15 μm surround Cu has an ε_{irr} of 0.48% which is slightly less than that of the one with 30 μm Cu only on the YBCO side, meaning that the Cu thickness on the YBCO side might be the dominant factor. Despite the difference in ε_{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 been also tested. The yield strength as defined at 0.2% strain for tapes with surround Cu stabilizer is 454 MPa at room temperature and 640 MPa at 76K, only about 11-12 % lower than the substrate. This high yield strength can be attributed to the 100% I_c retention when such conductors were subject to tensile stresses up to 360 MPa at room temperature [13, 14], and indicate that the IBAD-based coated conductors are twice as strong as others - both 1G and 2-G wires. This type of conductor is also bend and tensile strain tolerant [13].

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

V. 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 100+ m long IBAD tapes have been fabricated with uniform in-plane texture of 11-12°. 10 m/h tape speed 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 and multi-pass approaches enabled us to fabricate a 62 meter-long tape with end-to-end I_c of 100A/m-width. SuperPower developed key processes for practical conductors. 4mm wide tapes 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 demo devices including 1m cable by SEI, race-track rotor coils for Rockwell Automation HTS motor and pancake coils. These data shows that our practical conductors are reaching readiness for use in HTS devices applications.

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REFERENCES

[1] V. Selvamanickam, Y. Xie, J. Reeves and Y. Chen, MRS Bulletin, August 2004, 29, p579.

[2] P.N. Arendt and S.R. Foltyn, MRS Bulletin, August 2004, 29, p543.

[3] V. Selvamanickam, J. Reeves D. E. Peterson, U.S. Dept. of Energy Annual Peer Review, Washington D.C., July 17-19, 2002.

[4] Y. Li, J. Reeves, X. Xiong, Y. Qiao, Y. Xie, P. Hou, A. Knoll, K. Lenseth, and V. Selvamanickam, Proc. ASC 2004, *IEEE Trans. Appl. Superconductivity.*

[5] S. Motsuno, F. Uchiawa and K. Yoshizki, Jpa, Appl. Phys., 29, L947 (1990).

[6] K. Watanabe, H. Yamane, T. Hirai, N. Kobayski, H. Iwasaki, K. Noto, and Y. Moto, Appl. Phys. Lett., 54, 575 (1989).

[7] V. Selvamanickam, G.B. Galinski, G. Carota, J. DeFrank, C. Trautwein, P. Haldar, U. Balachandran, M. Chudzik, J.Y. Coulter, P.N. Arendt, J.R. Groves, R.F. DePaula, B.E. Newnam, D.E. Peterson, Physica C 333, 155 (2000).

[8] P. Chou, Q. Zhong, Q. L. Li, K. Abazajian, A. Ignatiev, C. Y. Wang, E. E. Deal, and J. G. Chen, Physica C 254 (1995) p. 93.

[9] V. Selvamanickam, G. Carota, M. Funk, N. Vo, and P. Haldar, U. Balachandran, and M. Chudzik, P. Arendt, J. R. Groves, R. DePaula, B. Newnam, IEEE Trans. Appl. Supercond. 11, 3379 (2001).

[10] J. Y. Coulter, R. Depaula, P. C. Dowden, J. R. Groves, L. Hausamen, J. O. Willis, D. E. Peterson, S. R. Foltyn, P. N. Arendt, E. J. Peterson, L. Winston, M. P. Maley, "Position Dependent Characterization of YBCO Coated Conductors", 1999 Cryogenic Engineering Conference-International Cryogenic Materials Conference, Montreal, Quebec, Canada, July 13-16, 1999. LA-UR-99-3909.

[11] V. Selvamanickam, J. Reeves D. E. Peterson, U.S. Dept. of Energy Annual Peer Review, Washington D.C., July 27-29, 2004.

[12] Y. Xie, in Proc. Air Force Office of Sci. Res. MURI Coated Cond. Rev. [CD-ROM] (University of Wisconsin-Madison, 2004)

[13] V. Selvamanickam, A. Knoll, Y. Xie, Y. Li, Y. Chen, J. Reeves, X. Xiong, Y. Qiao, T. Salagaj, K. Lenseth, D. Hazelton, C. Reis, H. Yumura, and C. Weber, Proc. ASC 2004, *IEEE Trans. Appl. Superconductivity.*

[14] Y.-Y. Xie, A. Knoll, Y. Li, X. Xiong, Y. Qiao, Y. Chen, P. Hou, J. Reeves, T. Salagaj, K. Lenseth, C. Weber, and V. Selvamanickam, Proc. International Workshop on Coated Conductor for Applications, Nov. 18-20, 2004, Kanagawa, Japan, O1-13.

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

	Current & Power applied to burnout conductor			
Conductor Type	Conductor 1	Conductor 2	Conductor 3	Conductor 4
Single side Cu (40 microns)	233A – **18.6W/cm^2**	205A - **18.1W/cm^2**		
"Surround Stabilizer" Cu (20 microns all sides)	279A – **33.3W/cm^2**	277A – **27W/cm^2**	158A (Etched out Cu from substrate side) –**15.3W/cm2**	194A (Etched out Cu from substrate side) – **21.8W/cm2**

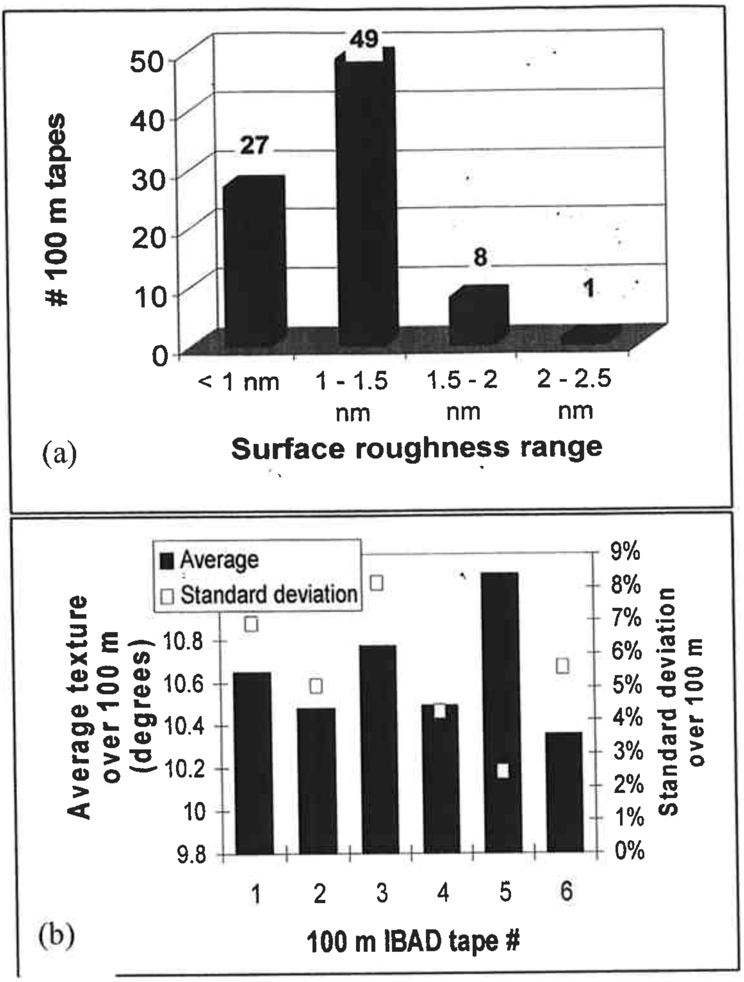


Fig.1. Statistic data from our pre-YBCO processes: (a) Electropolishing: surface roughness vs. number of polished 100 m long Hastelloy-C tapes. Surface roughness is measured on-line in the electropolishing facility in 1 mm intervals. (b) IBAD: in-plane texture measurements obtained every 0.25 m of six 100 m long tapes. The texture of the 100 m tapes is found to be uniform and reproducible.

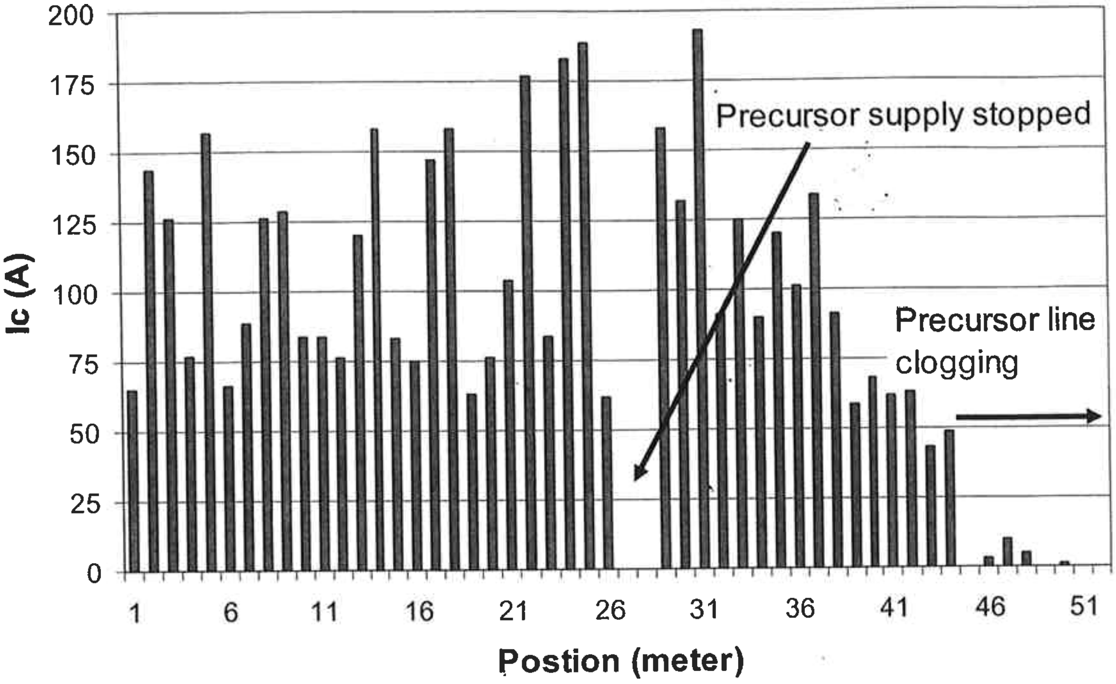
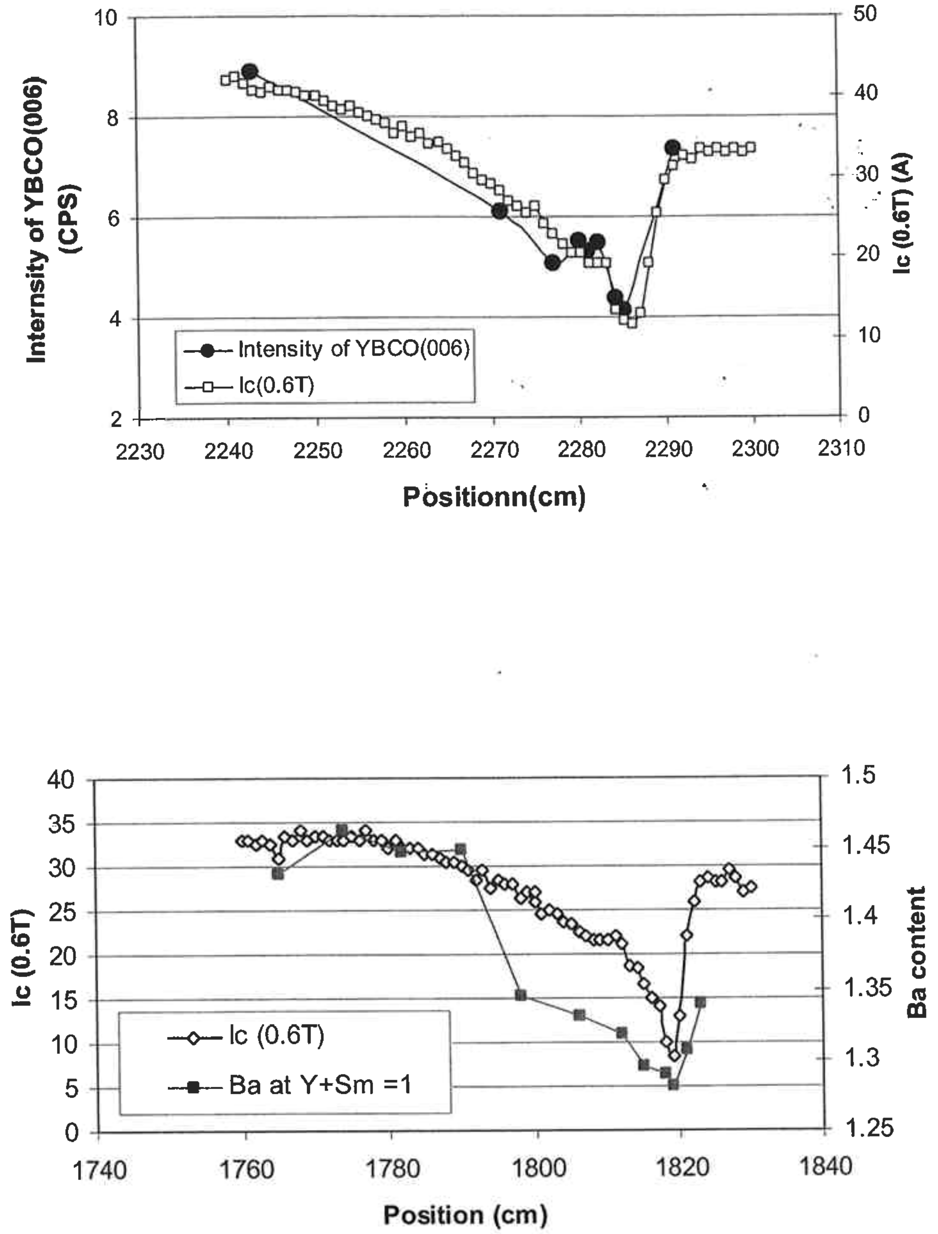


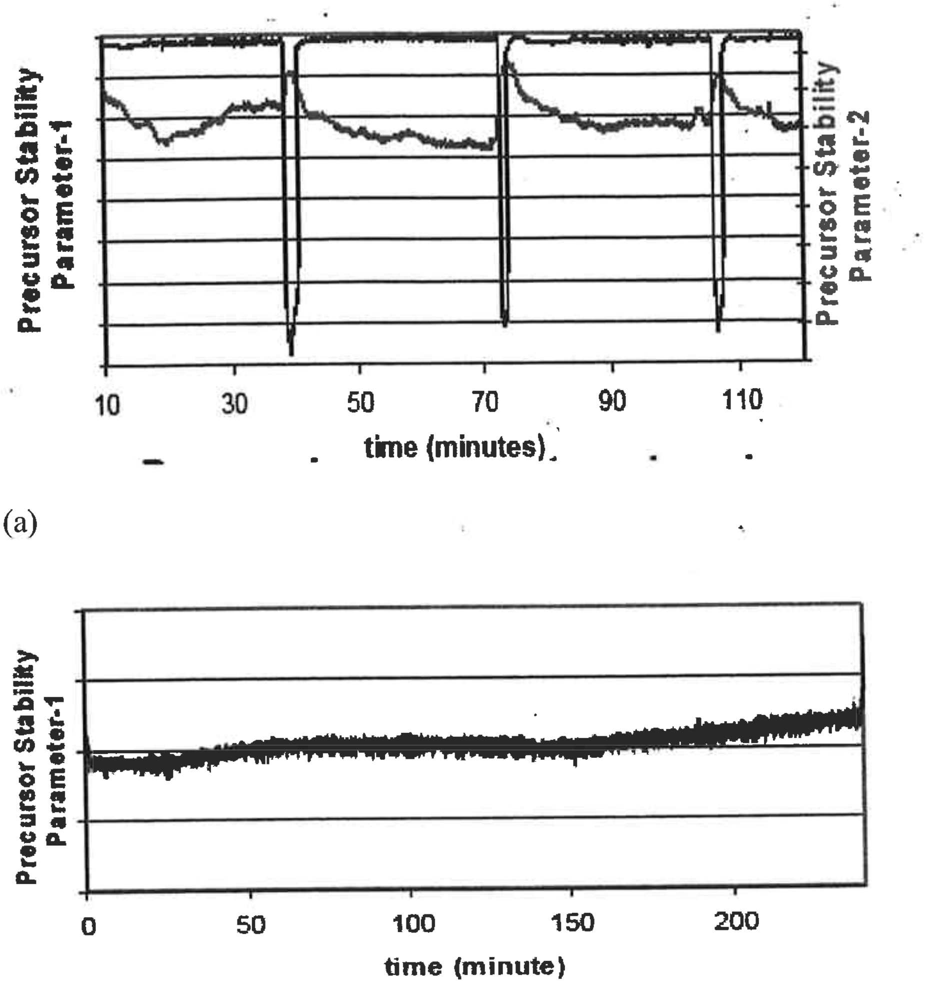
Fig. 2 Critical current (I_c) at every meter section of the first 50 m tape processed in the prototype MOCVD system. The voltage criterion is 1 μV/cm.



(a)

(b)

Fig. 3 Data on weak section from the 1st 50 m run in the prototype MOCVD system. (1)



(a)

(b)

Fig. 4 Precursor parameters monitoring results during MOCVD runs: (a) before system modification; (b) after modifications on the system to solve the precursor parameter instability problem.

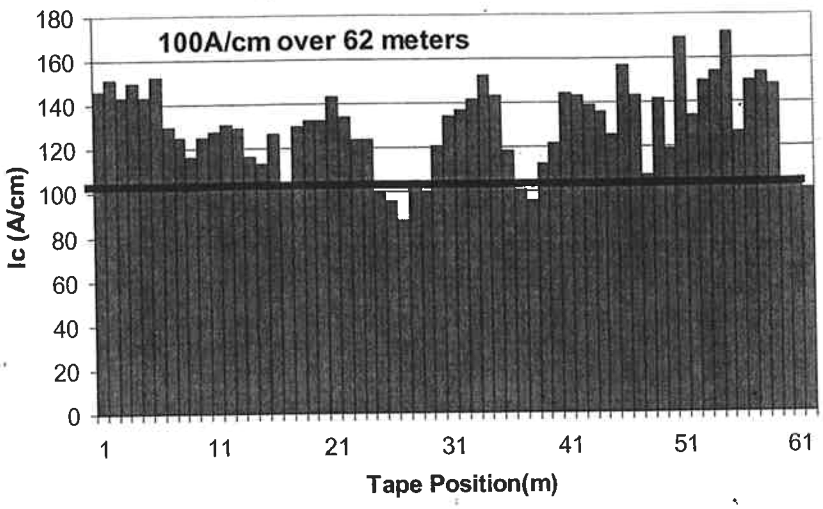


Fig. 5 I_c profile of a 62 meter long MOCVD tape. The end-to-end I_c is 100A/cm-width tested by winding the tape on a G-10 mandrel.

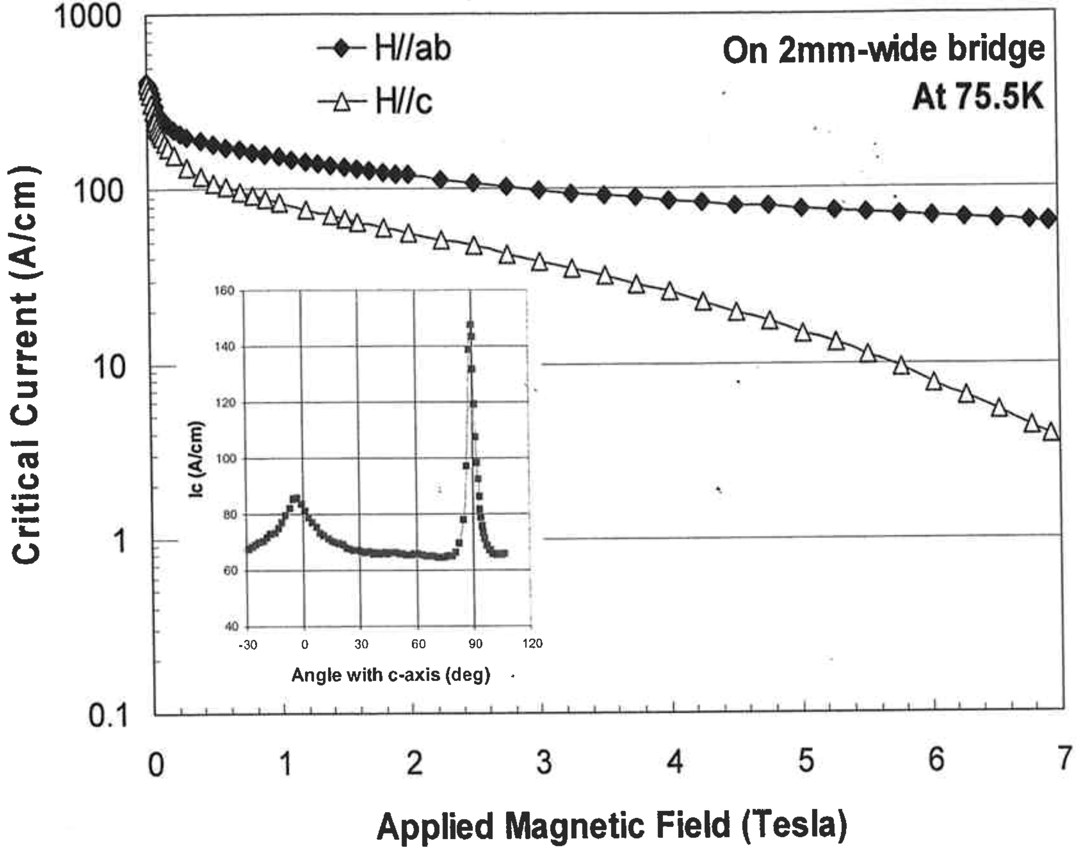


Fig. 6. Field dependence of critical of a Sm-substituted_YBCO coated conductor. The insert is the angular dependence of critical current at 1 T.

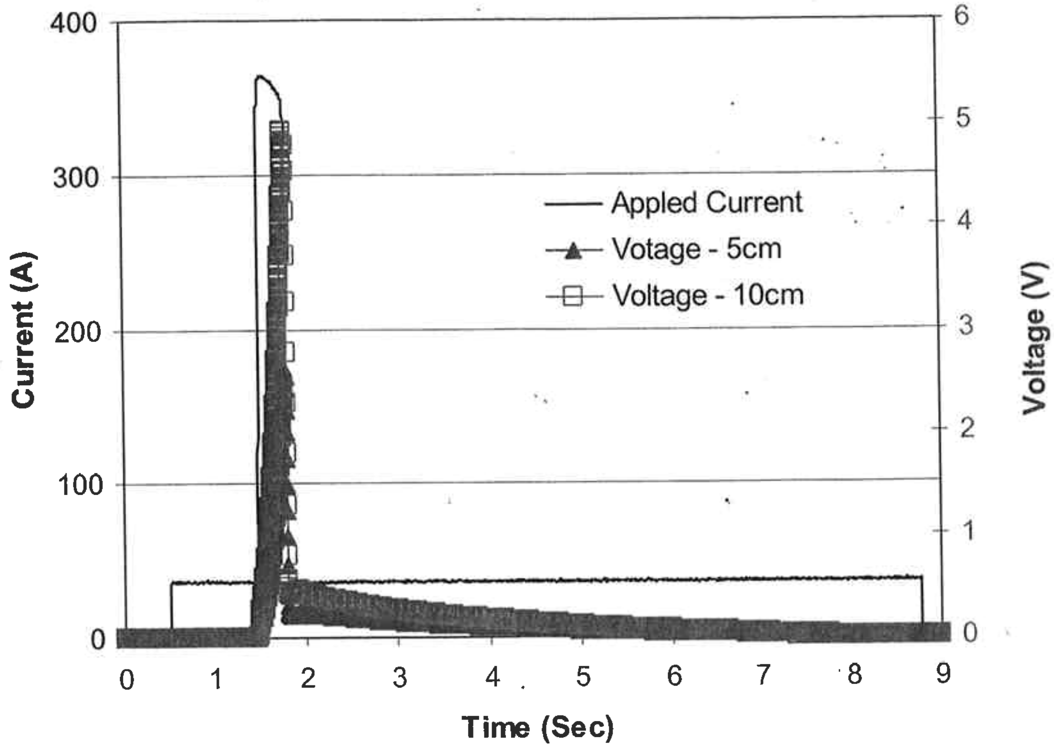


Fig. 7. The voltage response after the 11th 300 ms pulsed current was applied on a 4mm wide tape with 37 μm surround stabilizer. The pulsed current was 364 A which is 9.1 times of the I_c of the tape. No substantial I_c degradation was observed after this pulse.

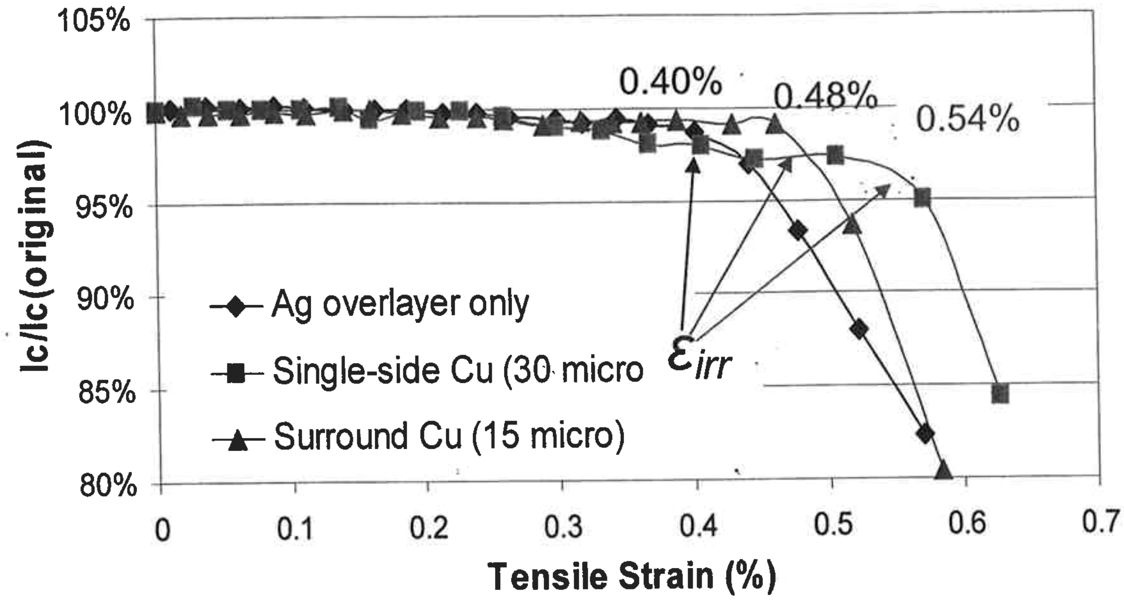


Fig. 8. Normalized I_c as function of axial tensile strain for three 4mm-wide coated conductors: (a) With only 3 μm Ag overlayer; (b) With 30 Cu on YBCO side; (c) With 15 μm Cu on all sides.