Long length coated conductor fabrication by inclined substrate deposition and evaporation

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Abstract. The commercial development of coated conductors is rapidly progressing. As a result we present an economic route to produce second generation HTS tape from the initial substrate preparation to the final metal coating. The most important and technically challenging steps are the deposition of an oriented buffer layer and the superconductor film in a reel-to-reel configuration. New evaporation techniques have been developed to enable reliable, high rate tape coating. Highly oriented MgO - buffer layers are realized by inclined substrate deposition (ISD) and DyBCO is deposited by simple e-gun evaporation yielding critical currents beyond 200 A/cm. Coated conductors have been fabricated up to 40 m length and are currently tested in a variety of applications.

1. Introduction
In order to foster commercial applications of coated conductors (CC) the main focus of industrial research is directed towards increasing tape length, demonstrating perspectives of low production cost, and improving performance. Inclined substrate deposition (ISD) is one of the most promising techniques, which offers both fast processing and excellent alignment [1]. Neglected for quite a while to RABiTS and IBAD [2,3] latest progress render it a serious and economic fabrication route for coated conductors. Since cost go hand in hand with throughput our recent activities were focused on improving the reliability of tape processing and increasing the speed of all involved fabrication steps. Prototype production equipment is now operational to manufacture CC tape for the first real applications.

2. Tape fabrication process
2.1. Substrate preparation
The flexible metal substrate used here is commercially available, non-magnetic Hastelloy® C276 cut in 10 mm wide tape of 100 μm thickness. The as - received tape is degreased and passed through a mechanical grinding stage for surface conditioning. Subsequently, the tape is electro-polished, cleaned with de-ionized water, and dried in airflow at a processing speed of 30 m/h. The resulting substrate tape has a thickness of 90 μm. Maximum surface slopes, which could affect the buffer alignment are less than 0.8° so that the inclination angle of the ISD process is well preserved even on a microscopic scale. In all following processing steps a 23 μm thin polyimide interleaf tape is used to protect the sensitive surface from damage.
2.2. ISD buffer deposition technique

A well-aligned MgO buffer layer is deposited by ISD where MgO is electron beam evaporated from a turntable feeding stage. To remove water from the tape surface and to facilitate adhesion of the buffer the tape is passed through a short heating stage at 400°C before entering the deposition area. The vacuum deposition process is performed at room temperature without additional heating. Details of the ISD texturing technique have been published previously [4]. Essentially, the substrate is inclined at an angle of 25°-30° towards the MgO vapor. Due to the high deposition rate > 4 nm/s and growth selection by shadowing only MgO grains with good in plane alignment and a surface tilt of about 20° survive. The optimum MgO film thickness is about 3 μm which yields an in plane alignment < 12° FWHM. Our current equipment can handle 300 m of tape and allows a tape processing speed of about 15 m/h. However, even higher deposition rates of up to 28 nm/s have been successfully realized already, which gives additional margin to speeding up.

On top of the thick ISD-MgO layer a 200 nm thin MgO cap layer is grown homo-epitaxially at 720°C and at perpendicular incidence. This layer serves to close gaps between the large MgO columns and to establish appropriate surface quality for the subsequent HTS coating. For efficiency reasons the tape passes through 15 loops of a helical tape winder to utilize a large deposition area (e.g. 400 cm² in case of the MgO cap and the HTS layer). This arrangement also guarantees better uniformity in film thickness and performance than a single, wide web. Integrated into the tape winder is a heater and an oscillating oxygen shuttle for the epitaxial coatings [5]. Since the MgO cap layer is thin, the transfer speed is already 10 m/h. The buffered tape is manufactured with a standard length of 35 - 40 m.

2.3. HTS – evaporation

DyBCO is deposited by an electron gun. A water-cooled copper turntable is extracting the granular DyBCO material from a funnel and conveying the evaporation material in form of a track into the hot zone of the electron beam. Details have been described previously [6]. Previous attempts to use such a mechanism were not successful since the YBCO powder was pre-melted to larger droplets that tend to fractionate [7]. However, fine-grained material (grain size < 0.5 mm) can be continuously evaporated this way, preserving the composition of the source material [8]. This new deposition scheme offers a variety of advantages. A large volume of powder can be stored in the reservoir and continuously evaporated over long periods of time. The deposition rate is just given by the geometry of the track and the rotation speed of the feeding turntable. Since the stoichiometry is essentially preserved, no sophisticated composition control is required and the film composition is uniform within a very larger area. Due to the reduced complexity of the DyBCO-deposition, the new e-beam deposition technique has greatly improved the process reliability.

Currently, the deposition rate is 1 nm/s translating into a volume deposition rate of 8 m μm cm/h. However, the e-beam source can be easily operated faster and 5 nm/s have already been demonstrated in short sample depositions.

2.4. Final steps

To establish superconducting properties the HTS tape is oxygen loaded by transferring it in 800 mbar of oxygen atmosphere through a tube furnace with a temperature ramp from 600°C to 300°C at a speed of 10 m/h.

To enable good electrical contact to the HTS layer and between the superconductor and the Hastelloy® substrate several μm thick metal layers are evaporated in the same chamber without exposure to ambient atmosphere. Again, the tape is wound in loops to use a large deposition area. The metal coating consists of copper or silver, which is continuously wire fed to a hot boat source for flash evaporation. This technique can be operated at high rate (currently > 5 nm/s, i.e. 25 m/h) and minimizes the thermal load of the superconductor tape. Even if just 5% of the source metal is deposited onto the tape, the material yield is more than 80% since the pure metal can be easily recovered from the shields of the evaporation chimney for recycling. In a final step the tape is roll-slit to a custom designed width and passed through a Tapestar™ scanner for spatially resolved critical current measurement [9]. Table I summarizes the key figures for processing 10 mm wide tape.

Table I: Summary of key figures for processing 10 mm wide tape.

- **Superconducting Property**: Critical Current (Ic)
- **Yield**: 80%
- **Speed**: 25 m/h
Table I

| Process       | Max. Length | Deposition rate | Speed  |
|---------------|-------------|-----------------|--------|
| E-polish      | 300 m       | -               | 30 m/h |
| ISD-MgO       | 300 m       | > 10 nm/s       | 15 m/h |
| MgO cap       | 40 m        | 0.2 nm/s        | 10 m/h |
| DyBCO         | 40 m        | 1.0 nm/s        | 8 μm/h |
| O₂-anneal     | > 100 m     | -               | 10 m/h |
| Metal coating | > 100 m     | > 5.0 nm/s      | 25 m/h |
| Slitting      | > 300 m     | -               | > 500 m/h |
| Tapestar™     | > 1000 m    | -               | 200 m/h |

3. Results

The crystalline quality and alignment of the buffer and DyBCO layers have been evaluated by X-ray diffraction (XRD). The in-plane FWHM of the MgO (200) peak is typically in the range between 10° – 12°. However, XRD constitutes an integrated measurement of the entire film thickness. Since the alignment improves with increasing MgO thickness the surface alignment is about 2° - 3° better than the above average figures, which is reflected in the better values for the DyBCO layers that exhibit in-plane FWHM values around 7° - 8°. In any case, the ultimate measure is the critical current performance of the CC. Critical currents have been evaluated by end-to-end transport measurements and reel-to-reel with 0.5 mm spatial resolution by a Tapestar™. The operating principle and defect monitoring has been described in detail elsewhere [9].

CC tape has been fabricated up to 40 m length, which is currently established as the standard length for our prototype production. The alignment technique yields critical current densities up to 2.5 MA/cm² at 77 K even in 2-3 μm thick DyBCO films. Performance data on different length scales are summarized in Table II. While short tape sections exhibit up to nearly 500 A/cm, effective end-to-end values are normally in the range between 200 – 250 A/cm for longer tape. A new e-polishing line has just been taken into operation and will considerably reduce the number of point defects, which gave rise to local jc – deterioration in the past. Consequently, we expect an improvement of the standard specs beyond 300 A/cm in the near future.

Table II

| Length (m) | Thickness (μm) | Average Ic (A) | Scatter σ (A) | Max Ic (A) | Min. Ic (A) |
|------------|----------------|----------------|---------------|------------|-------------|
| 1          | 2.0            | 422            | 47            | 486        | 227         |
| 5          | 1.8            | 337            | 32            | 370        | 240         |
| 10         | 1.8            | 332            | 24            | 381        | 200         |
| 40         | 2.0            | 158            | 75            | 371        | 0           |

4. Summary and outlook

Within the last two years we have introduced and demonstrated a consistent physical vapor deposition route for the fabrication of coated conductors. Highly oriented MgO buffer layers are deposited at high rate by ISD yielding critical current densities up to 2.5 MA/cm². A revolutionary simple electron beam deposition configuration allows fast and reliable deposition of the HTS layers. All involved processing steps, from initial substrate polishing to final metal coating, are performed in reel-to-reel mode. Most of our equipment can handle tape at a transfer speed in excess of 10 m/h, has a reel capacity well beyond 100 m, or can be easily scaled up. The standard length currently processed is 40 m and is scheduled to increase beyond 100 m in 2006.

The main volume of such tape is currently produced for motor applications, which will consume 5 – 6 km within the next two years. Due to the unique physical properties of the underlying Hastelloy
substrate, however, the CC material is also ideally suited for a variety of other applications. Cryogenic current leads will profit from the low thermal conductivity (7 W/m-K) and the high electrical resistivity (130 μΩcm) of the substrate make it the perfect choice for fault current limiters (FCL). First FCL tests in collaboration with Forschungszentrum Karlsruhe were extremely successful yielding electrical fields of 2.6 V/cm in the switched state. Due to substrate heating the quenching occurs along the tape and hot spots are prevented [10]. The high mechanical strength (500 MPa tensile) and excellent adhesion of the layers [11] was essential for the successful fabrication and tests of first Roebel assembled cable strands at FZ Karlsruhe [12].

The rapid progress in CC development and the large number of practical tests underway will certainly lead to a variety of promising applications and prototypes within the coming years. We believe that eventually a lot of traditional engineering and technology will profit from coated conductors justifying the huge development effort invested.

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