DEPOSITION OF PROTECTIVE THIN LAYERS ON STRIATED REBCO SUPERCONDUCTING TAPES

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Abstract

High-temperature superconducting tapes made from rare-earth element barium copper oxide have significant potential for use in high-field magnet applications, owing to their outstanding superconducting properties.However, they experience high magnetization losses when subjected to transient or alternating magnetic fields. To tackle this issue, it is crucial to reduce the width of tapes, which can be achieved through the striation of superconducting layer. During this process, the superconductor becomes exposed at the edges of the filaments to ambient, potentially leading to its degradation. In this study, we provided additional protection to these exposed areas in striated superconducting tapes by applying multilayer coatings using magnetron sputtering. Adhesion tests and measurements of electrical resistance were conducted on samples featuring 12 different coating systems, based on AlN. Their quality was evaluated using scanning electron microscopy. The selected system Ti / AlN was sputtered onto striated superconducting tapes, which underwent electrical measurements to comprehensively evaluate their performance.

Keywords: High-temperature superconducting tapes, AC losses, magnetron sputtering, AlN, thin films

1. INTRODUCTION

$REBa_2Cu_3O_{7-\delta}$ (REBCO) tapes, where RE stands for rare earth element such as yttrium or gadolinium, belong to a group of high-temperature superconducting (HTS) materials [1]. These materialshold significant promise for use in high-field magnet applications such as pulse magnets and particle accelerators, thanks to their high capacity for carrying electrical current and their robust resistance to strong magnetic fields [2]. However, due to unsuitably large aspect ratio in the thickness and width of the superconductor within the tape (1:10,000), REBCO tapes are susceptible to alternating current (AC) losses, resulting from changing magnetic fields. The thickness, typically about 1 to 3 µm, cannot be significantly increased, as larger thicknesses lead in an increase in lattice defect density without any increase in critical current (I_c) density. According to the theories of Brandt and Zeldov [3, 4], magnetization loss values are approximately proportional to the width of the superconducting layer. Therefore, a clear approach to reduce part of AC losses, hysteresis losses, is to manufacture narrower tape widths, or to segment superconducting layer into filaments.

Currently, commercially produced HTS tapes are available in narrow widths of 4, 3, or even 2 mm, primary intended for cable production. In such narrow tapes, further narrowing of the superconducting layer can be achieved by creating filaments, which are separated from each other through a process known as striationing [5]. Such a structure can be created by several methods: laser ablation [5-9], chemical etching [10, 11], mechanical scratching [12, 13], photolithography [14]or filaments created by deposition [15,16].

During the striation process, the edges of the grooves in the superconductor became exposed, rendering them susceptible to degradation. Therefore, safeguarding these areas is crucial. To improve the functionality of REBCO tapes, we applied protective multilayers to the striated HTS tapes via magnetron sputtering (MS). We selected AlN ceramics as the base for the coatings because this material possesses a high electrical resistance and high thermal conductivity. Such coatings can serve multiple purposes: they protect the superconductor from environmental damage, enhance its mechanical strength, and offer tailored thermal stabilization without increasing coupling magnetization losses.

2. EXPERIMENTAL PROCEDURE

2.1 Coating of samples and electrical resistance measurements

For this study, a commercially available Ag-stabilized REBCO tape from SuperPower was used, which has a width of 12 mm and a thickness of 100 µm, with an average critical current (I_c) of 367 A ± 14 A. From this tape, 14 samples with dimensions 20 mm x 12 mm were cleaned and coated with a different system material using magnetron sputtering at parameters for particular layers listed in **Table 1**. The targets for sputtering have 7.6 cm and base pressure in both DC and RF chamber was 7×10^{-5} Pa. The prepared systems are shown later, in chapter 3.

The samples had two distinct sections; one half remained in its initial state (Ag layer on the top), while the other section was coated with a protective system, as depicted in **Figure 1(a)**. The coating was applied either on the Ag stabilization or on the REBCO layer, while the Ag layer was removed by etching in saturated aqueous solution of KI_3. The quality and adhesion of the sputtered layers were evaluated through a simple peel test (**Figure 1(b)**), which involved applying a strong adhesive tape to the coated surfaces. Some of the samples were examined in a scanning electron microscope (SEM) JEOL JSM7600F. For this purpose, cross-sections were prepared using a JEOL SM-09010 polisher.

Table 1 Sputtering parameters of layer used for REBCO tapes modification

Layer	**Thickness** (nm)	**Mode**	**Target**	**Gas**	**Gas flow** (sccm)	**Pressure** (Pa)	**Power** (W)	**Time**
AlN	1000	RF	AlN	Ar	50	5×10^{-1}	290	200 min
Cu	450	DC	Cu	Ar	50	5×10^{-1}	300	7 min 18 s
Cu	900	DC	Cu	Ar	50	5×10^{-1}	300	18 min 15 s
Ti-Cu	150	DC	Ti, Cu	Ar	50	5×10^{-1}	300	2 min
Ti	30	DC	Ti	Ar	50	5×10^{-1}	300	2 min
Si_3N_4	30	RF	Si_3N_4	Ar	50	5×10^{-1}	300	3 min 15 s
Al_2O_3	30	DC	Al	Ar/O_2	40/10	5×10^{-1}	200	2 min

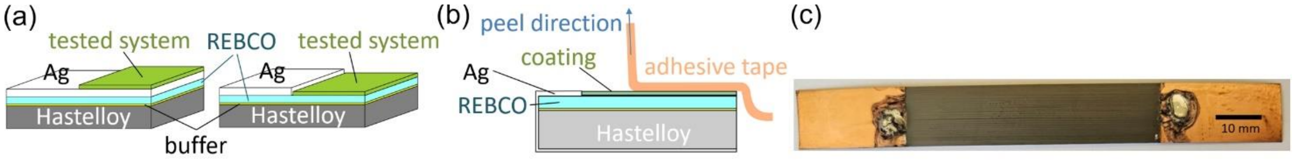

Figure 1(a)Scheme of the samples with deposited coatings for electrical resistance measurements, (b) scheme of peel test, (c) sample of the striated tape with the deposited protection coating Ti/AlN

To assess the electrical resistance of deposited layers, measurements were conducted using a HIOKI 3545-01 resistance meter. This method involved the use of a two-probe technique. To ensure the integrity of thin layers during testing, graphite electrodes were employed. Graphite was chosen for its soft, conductive properties, which help to establish a reliable contact without penetrating or otherwise damaging the delicate layer.

2.2 Preparation of striated and coated HTS tape

The samples were prepared from 10 cm long HTS tapes. For I_c measuring, electroplated copper was applied over 2.5 cm at each end of the tape to ensure better electrical contact. This left a central section of 5 cm in length with a silver stabilization layer available for striation, as illustrated in **Figure 1(c).**

After cleaning the HTS tape using ethanol, their surface was deposited with a 30 nm thin Ti layer using magnetron sputtering. Then the striation process was carried out as follows. Edding T25 ink, diluted to a 1:2 ratio with absolute ethanol, was evenly distributed on sample surface for filament masking using a spin coater operating at 2800 rotations per minute until the mask was completely dry. The grooves among 30 filaments, reaching the Ag layer, were fabricated manually using a diamond pen with a 57° tip angle, maintaining a consistent speed of about 2 cm/s. For etching of grooves, a 10 wt.% solution of $(NH_4)_2Ce(NO_3)_6$ was utilized. The samples were stirred in the solution for 60 seconds. Subsequently, the samples were washed in water, and a stream of absolute ethanol was used to clean residual mask. To protect the areas of samples intended not to be deposited or etched, covering with a Kapton tape was applied. Finally, a 1 µm thick layer of AlN was deposited to the striated surfaces.

2.3 Electrical measurements

Measurements of critical current and *n*-value (the steepness of the transition to the normal state) were performed by measuring the voltage-current characteristics of non-striated HTS tapes with coatings deposited onto superconducting layer, as well as on striated HTS tapes with and without protective coatings, immersed in a liquid nitrogen bath.

3 EXPERIMENTAL RESULTS

3.1 Electrical resistance and adhesion of deposited systems

The samples with deposited systems listed in **Table 2** underwent measurement of electrical resistance to verify the expected high electrical resistance. Simultaneously, a peel test of the samples was performed to assess the quality of adhesion. The results are shown in **Table 2.**

The adhesion of the AlN layer to the Ag and Cu surfaces in samples 1 and 2 was evaluated as very poor. An addition of a thin Ti layer at the Ag/AlN interface in samples 3 and 4 significantly improved this property, however; it was not sufficient for the AlN/Cu interface. Therefore, a 150 nm thin transition Ti-Cu layer sputtered simultaneously from both targets, was added to this interface, and then the adhesion (sample 5) was satisfactory. The adhesion of the AlN layer to the REBCO layer was good in all samples (for example samples 6 and 7).

The measurements of electrical resistances were a little bit confusing. The expected high electrical resistance of systems containing AlN ceramics, which was about 12 700 Ω, was only observed in case of sample 6, where the AlN layer comes into direct contact with the REBCO superconductor. If there is a good conductive layer such as Ag between them, the resistance decreased by about two orders of magnitude (samples 1 and 3). This reduction is even more pronounced when conductive layers were deposited above AlN layer, as seen in samples 2, 4, and 5. The measured electrical resistance of these layers is surprisingly comparable with sample 8, where only a Cu layer was deposited on the tape with the Ag stabilization. Similarly, in sample 7 with AlN/Ti/Ti-Cu/Cu system sputtered on REBCO surface, the electrical resistance was very low (4.3 Ω). In our experiments, the Ag layer was removed using a KI_3 selective etchant. Due to the results of resistance measurements, it was suspected that this selective etchant leaves some residue in the form of thin conductive layer. This was indeed confirmed with measurements of sample 9, in which one half of Ag layer was etched to the REBCO superconductor, and sample 10, in which additionally the superconducting layer edges were completely etched away with 33 vol.% aqueous solution of HNO_3, thus isolating it from the Ag layer on the other section of the sample. It was found that adding of further 30 nm thin interlayer from Al_2O_3 or Si_3O_4 can significantly increase the resistance of multilayer system deposited on sample 9 (see samples 11-14).

Table 2 Properties of investigated systems

Sample	Layer system	R(Ω)	Adhesion
1	Sample with Ag/1 µm AlN	150	×
2	Sample with Ag /1 µm AlN/ 450 nm Cu	0.3	×
3	Sample with Ag / 30 nm Ti/1 µm AlN	145	✓
4	Sample withAg/30 nm Ti/1 µm AlN/30 nm Ti/450 nmCu	0.5	×
5	Sample withAg/30 nm Ti/1 µm AlN/30 nm Ti/150 nm Ti-Cu / 450 nmCu	0.2	✓
6	Sample with REBCO / 1 µm AlN	12 700	✓
7	Sample with REBCO / 1 µm AlN / 30 nm Ti/150 nm Ti-Cu / 450 nmCu	4.3	✓
8	Sample with Ag / 450 nm Cu	0.2	✓
9	Sample with REBCO	60	-
10	Sample with REBCO with perfectly isolated edges	1 250 000	-
11	Sample with REBCO / 30 µm Al_2O_3 / 1 nm AlN	150 000	✓
12	Sample with REBCO / 30 µm Al_2O_3 / 1 nm AlN/30 nm Ti /30 nm Ti/150 nm Ti-Cu / 450 nmCu	1200	✓
13	Sample with REBCO / 30 µm Si_3O_4 / 1 nm AlN	135 000	✓
14	Sample with REBCO / 30 µm Si_3O_4 / 1 nm AlN/30 nm Ti /30 nm Ti/150 nm Ti-Cu / 450 nmCu	390	✓

3.2 SEM examination of deposited layers

Some of the samples were examined in SEM. The chemical composition of AlN layer, determined using energy dispersive X-ray (EDX) analysis, was 50.8 at.% of Al and 49.2 at.% of N, which is nearly a 1:1 ratio as indicated by chemical formula. **Figure 2** shows examples of cross-sections prepared from sample 2 and 5. In the sample coated with AlN / Cu, delamination of the Cu layer is apparent. Conversely, in the sample featuring the Ti / AlN / Ti / Ti-Cu / Cu system, the individual layers appear compact.

Figure 3 presents striated HTS tape protected with Ti / AlN coating. As indicated by the EDX maps of the surface, the Ti / AlN coating was homogenously distributed over filaments, while in grooves AlN was concentrated.

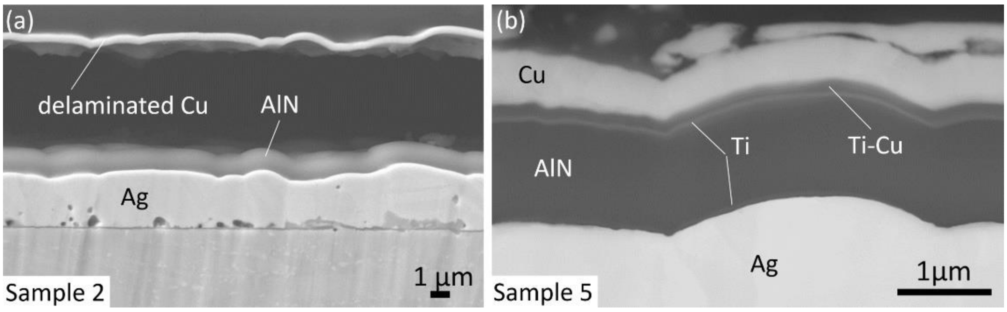

Figure 2 SEM images of cross-sections prepared from (a) sample 2 and (b) sample 5

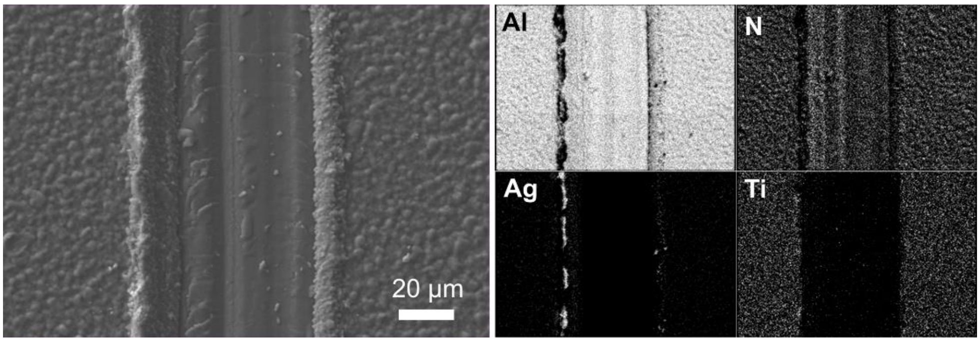

Figure 3 SEM image with EDX maps of striated HTS tape protected with Ti / AlN coating

3.3 DC measurements

Selected samples underwent electrical measurements to assess the impact of the coatings on their critical current and *n*-values. These results are presented in **Table 3**. The best result among three different coating systems applied onto superconducting layer of HTS tape was achieved in the sample 6, which was deposited with AlN, demonstrating of 78% for I_c and 92% for *n*-value, while the other systems degraded the samples significantly. Consequently, AlN coating was chosen for protect the HTS layer at filaments edges in striated samples, while Ti / AlN system was deposited on the filaments with Ag surface. Left side of **Table 3** indicates that this coating improved the electrical properties of striated HTS tapes by more than 20% compared to striated HTS tape without coating.

Table 3 DC Measurements of coated and HTS striated tapes

Non-striated HTS tapes with coating deposited on REBCO surface				**Striated HTS tapes (30 filaments)**			
Sample	**Coating**	**I_c retention** (%)	***n* retention** (%)	**Coating**	**I_c retention** (%)	**I_c^*retention** (%)	***n* retention** (%)
6	AlN	78.1	92.5	-	31.9	45.1	53.0
11	Al_2O_3 / AlN	7.6	50.6	Ti/AlN	51.4	64.6	62.8
13	Si_3O_4 / AlN	6.8	50.8	Ti/AlN	65.0	78.2	59.4

I_c* means retention of critical current under consideration of HTS material loss from grooves

4 CONCLUSION

In this study, 12 various coating systems were evaluated as potential protective layers for striated REBCO HTS tapes. The selection was based on their electrical resistance and adhesion properties. The chosen materials (Ti and AlN) were then sputtered onto striated tapes. For comparison, striated tape without any sputtered material was prepared. All samples underwent DC measurements to assess how the deposition affected the critical current of the tapes. These measurements are essential prior to evaluating AC losses, as tapes with degraded I_c are unsuitable for practical applications. The results demonstrated that the protective layer improved performance, with I_c retention approximately 20% higher than that of the non-sputtered tapes, validating the effectiveness of the protective coating.

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