Crystal structure and critical properties of HTSC 2G prototypes formed by pulsed laser deposition technique on the RABiTS tapes

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Abstract. HTSC 2G YBCO based short samples were formed by PLD (pulsed laser deposition) on the cube textured Ni-W tapes. The influence of buffer layer architecture on the critical properties of HTS films was investigated with scanning and transmission electron microscopy, X-ray diffraction and four point probe electrical measurement. The best critical properties were found in the HTS grown on tapes with triple buffer layers: Y$_2$O$_3$, YSZ and CeO$_2$. After CeO$_2$ layer deposition the blister-like defects appeared on the buffers surface. These defects being inherited by YBCO film could lead to degradation of critical properties.

1. Introduction
The formation of second generation of high temperature superconductor cables (HTSC 2G) on RABiTS (rolling-assisted-biaxially-textured substrates) require the intermediate buffer layers [1]. One of the widely used buffer layers architectures for YBCO growth on RABiTS is the 3-layered system consisting of the seed layer Y$_2$O$_3$, barrier layer yttrium stabilized zirconia (YSZ) and cap CeO$_2$ layer. The use of CeO$_2$ buffer is explained by good match of the crystallographic parameters of (001) CeO$_2$ and (001) YBCO compounds. In the present work we applied such 3 buffer layers for YBCO film formation on RABiTS. This system demonstrated 145 A/1 cm width current on short samples. The value of the current in our samples could be influenced by specific dome-like defects in YBCO films. Similar defects were found in [1-2], but their nature was not clearly understood. In this study we investigated the mechanisms of formation of such defects and their influence on critical parameters of YBCO films.

2. Experimental details
The EVICO RABiTS tapes with composition Ni + 5 %at W were used for the YBCO based coated conductors. The in-plane texture of substrates determined from the X-ray phi-scans was 6$. All films were grown using PLD (Kr-F, $\lambda$=248 nm, energy density 3 J/cm$^2$). The seed buffer layer Y$_2$O$_3$ was deposited in Ar+H$_2$ (P=1 mTorr) atmosphere to prevent Ni-W substrate from oxidation at initial growth steps. Other buffers were deposited in 1 mTorr O$_2$ atmosphere. O$_2$ pressure during YBCO deposition was P=350 mTorr. The deposition temperature of all layers have been controlled by IR-pyrometer and maintained at t=750°C. The sample sizes were 10x10 mm. The microstructure of the
systems were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), scanning/transmission electron microscopy (TEM/STEM) together with energy dispersive X-ray (EDX) microanalysis. SEM analysis were performed in Helios (FEI, US) dual beam instrument (electron - focus ion beam (FIB)), which was also used for cross-sectional sample preparation. A Pt layer of 2-3 µm thick was deposited on the surface of the sample prior to the FIB cross-sectioning. The protective layer was formed in two steps process: first it was deposited with of Plan-view samples were prepared by conventional method. All specimens were studied using a Titan 80-300 TEM/STEM (FEI, Oregon, US) equipped with a spherical aberration (Cs) corrector (electron probe corrector), a high angle annular dark field (HAADF) detector, an atmospheric thin-window energy dispersive X-ray (EDX) spectrometer (Phoenix System, EDAX, Mahwah, NJ, US), and post-column Gatan energy filter (GIF; Gatan, Pleasanton, CA, US). Measurements of the superconducting properties were made by standard 4 probe technique. Ag was DC sputtered for contacts formation with sequential etching of uncovered film in HNO₃ solution.

3. The buffer layers investigations.

The SEM study demonstrated different morphology of YSZ and CeO₂ surfaces and that is shown in Fig.1 (a) and (b). The blister-like defects appeared on the surface of CeO₂ (three buffer layers) Fig.1 (a). The diameter of blisters was 5-7 µm and that was stable within 10 - 100 nm of CeO₂ film thicknesses. The concentration of the defects was not changed either. However, we found different concentration of the blisters in different areas of the film, which were associated with differently oriented grains in the substrate. In the same time, the surface of YSZ was smooth, without any peculiarities Fig.1 (b). Close inspection of images of YSZ/CEO₂/protective Pt-C layer (Fig. 2 (a) and (b)) demonstrated, that during sample preparation by FIB, amorphization of CeO₂ surface occurred by Ga⁺ ions. That caused the formation of additional layer on the top of CeO₂. XRD and TEM data pointed that Ni oxidation occurs after CeO₂ film formation. The NiO layer is visible on cross-section HAADF STEM images on Fig. 2 (a) and (b). The blister area was filled with Ni: the HAADF contrast (Fig 2 (a)) together with EDX microanalysis unambiguously pointed to the absence of W in that area. The NiO layer was not flat (Fig.2 (b)). Surprisingly, that the thickness of NiO layer was higher in the areas, which were close to the perimeter of the blisters and lower in the epicenter. If the ambient atmosphere during cooling was changed to H₂, the blisters were not formed on the surface of CeO₂. All these data indicated that the formation of blister occurred due to oxidation of the Ni-W substrate through the channels, which appeared in the CeO₂/YSZ/Y₂O₃ buffers as a result of unevenness on Ni-W substrate surface from one hand and stress in the system caused by difference in thermal expansion coefficient from the other. The unevenness of the substrate resulted in the formation of grain boundaries in the buffer layers which served as O diffusion paths during the cooling of our system from the growth to room temperature.
The stress also triggered on the Kirkendall diffusion of Ni in the oxidized area and that initiated the formation of blisters.

4. YBCO film investigation

The YBCO films were deposited on RABiTS with three and two buffer layers under the same deposition conditions and SEM images of YBCO film surface are shown in Fig.3 (a) and (b) correspondingly. The blisters were found on the surface of YBCO/CeO$_2$/YSZ/Y$_2$O$_3$/Ni, however they looked smoother than those on CeO$_2$ surface. The diameter of blisters was similar to one on CeO$_2$ surface, namely 5-7 μm and we proposed that these defects were inherited by YBCO film. The SEM study did not reveal similar defects on YBCO/YSZ/Y$_2$O$_3$/Ni surface. The SEM shows better epitaxy and better surface morphology of YBCO films on CeO$_2$: slightly misoriented domains in the film and

Fig. 1 Secondary electron (SE) SEM images of buffer surfaces. (a) – CeO$_2$ buffer (3-layer buffer), (b) – YSZ (2-layer buffer).

Fig. 2 HAADF STEM cross-section image of the 3 layer buffer system in blister area. (a) – the blister was filled with Ni covered with NiO film. Dark area on the top of the blister corresponds to the hole in the TEM sample. (b) – enlarged image of NiW/Ni/NiO/Y$_2$O$_3$/YSZ/CeO$_2$ interfaces.
grain boundaries were clearly visible. Such contrast was not observed in YBCO films grown on YSZ buffer. YBCO films were not uniform in both cases, but overall porosity of YBCO films on YSZ buffer was higher.

The critical temperature of YBCO on 3 layers buffer was found to be $T_c = 88$ K with the transition width of 2 K and on 2 layers buffer $T_c = 85$ K. However, the transition width in the system with 2 buffer layers was narrower than 1 K. The reason of such critical temperature reduction was caused by changes of oxygen stoichiometry of the film. The X-ray analysis demonstrated that oxygen content in that sample was equal to $x = 6.7$, while in the samples with 3 buffer it was higher: $x = 6.9$. Critical current in the sample with 3 buffer layers was $I_c = 14.5$ A/(1 mm width), while in the system with 2 buffer layers $I_c = 7.5$ A, in both cases YBCO film thickness was 1 µm. That also could be explained by the difference in oxygen content. The difference in morphologies of YBCO films on different buffers might result variations in oxygen content.

Conclusion
Different aspects of the influence of buffer layers architecture on microstructure and electro physical properties of YBCO tapes on RABiTS substrate have been illustrated. The mechanism of blisters formation in 3 buffer layers system was proposed. The YBCO films on YSZ buffer layer demonstrated lower critical current density but narrower transition compared to the YBCO films on CeO$_2$ buffer layer. That was explained by the difference in film oxidation and film morphology.

References
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