Characterisation of multilayer ramp-type REBa$_2$Cu$_3$O$_{7-\delta}$ structures by scanning probe microscopy and high-resolution electron microscopy

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Abstract

We studied the morphology of ramps in REBa$_2$Cu$_3$O$_7$ (REBCO) epitaxial films on SrTiO$_3$ substrates, fabricated by RF magnetron sputter deposition and pulsed laser deposition (PLD), by scanning probe microscopy (SPM) and high resolution electron microscopy (HREM). The ramps were fabricated by Ar ion beam etching using masks of standard photoresist and TiN. AFM-studies on ramps in sputter deposited films show a strong dependence, i.e. formation of facets and ridges, on the angle of incidence of the ion beam with respect to the substrate surface as well as the rotation angle with respect to the crystal axes of the substrate. Ramps in pulsed laser deposited films did not show this dependence. Furthermore, we studied the effect of an anneal step prior to the deposition of barrier layers (i.e. PrBa$_2$Cu$_3$O$_7$, SrTiO$_3$, CeO$_2$) on the ramp. First results show a recrystallization of the ramp surface, resulting in terraces and a non-homogeneous growth of the barrier material on top of it. The thickness variations, for thin layers of barrier material, can even become much larger than expected from the amount of deposited material and are dependent on the deposition and anneal conditions. HREM studies show a well defined interface between barrier layer and electrodes. The angle of the ramp depends on the etch rate of the mask and REBCO, and on the angle of incidence of the ion beam. TiN has a much lower etch rate compared to photoresist, resulting in an angle of the ramp comparable to the angle of incidence, resulting in a low etching rate on the ramp. These results will lead to improved electrical characteristics of ramp-type junctions.

Keywords: Ramps; SPM; HREM

1. Introduction

Ramp-type Josephson junctions are suitable for various applications, by virtue of their artificial barrier. In this type of junction the barrier material and thickness can be used to tune the junction performance [1]. Josephson junctions with a ramp-type geometry are prepared by structuring a ramped edge in a REBCO base electrode. A barrier layer and a top electrode are epitaxially grown over this structured ramp. The coherence length $\xi$ is extremely short in high-$T_c$ materials and, therefore, barrier layers with a thickness of only a small number of unit cells have to be used to obtain a significant d.c.-Josephson current. Consequently, surface morphology and growth of barrier material on the ramp will influence the junction properties seriously.

The product of the critical current $I_c$ with the normal state resistance $R_n$ (product), which is an important figure of merit for Josephson junctions, has a systematically higher value for lower PrBa$_2$Cu$_3$O$_7$ thicknesses [2]. The junction properties, however, become irreproducible and the current–voltage characteristics show large excess current when the barrier thickness is decreased below 10 nm. In this contribution we study the surface morphology of the etched ramp using a soft (photoresist) mask or hard (TiN) mask.

2. Experimental

Epitaxial DyBa$_2$Cu$_3$O$_7$ films were deposited on 510 mm$^2$ (001) SrTiO$_3$ substrates using off-axis RF-magnetron sputter deposition and pulsed laser deposition from stoichiometric targets. Before structuring, a photoresist mask was made by spinning Shipley S-1813 for 30 s at 4000 rpm and was then baked on a hot plate at 90 °C for 10 min. Thickness of the mask was about 1.2 μm. Next, the sample was exposed for 15 s and developed. For the TiN mask a lift-off technique was used. In this study a 40 nm thick TiN
layer was deposited by bias sputter deposition of Ti in an Ar/N₂ atmosphere. Partial pressures were 2.0×10⁻² mbar for Ar and 0.5×10⁻² mbar for N₂ (RF-power=300 W, self bias=650 V and substrate bias=100 V). The etching of the ramps was done with a Kaufmann Ar ion gun (3 cm in diameter, V=500 V, I=10 mA) at an Ar pressure of 2×10⁻³ mbar in a pulsed mode (8 s on, 10 s off) to prevent overheating of the sample. The geometry during the etching process is sketched in Fig. 1(b). The plane of incidence is perpendicular to the edge of the mask.

For morphology studies on ramps an "open air" Nanoscope III was used in AFM contact mode with commercial Si₃N₄ tips.

3. Results and discussion

Fig. 1(a) shows the etch rate of DyBa₂Cu₃O₇ (DBCO) versus the angle of incidence (Ψ) of the ion beam. The etch rate at Ψ=45° is 12 nm/pulse for DBCO and 24 nm/pulse for photoresist. Due to this difference in etch rates, the resulting ramp angle in the DBCO layer will be smaller than Ψ, e.g., an angle of incidence of Ψ=45° will yield a ramp angle of approximately 20°. As a result, the effective angle between the angle of incidence and the ramp angle will be 25°. This will lead to a high ion bombardment and, consequently, an etch damage of the ramp surface during structuring of the ramp. To avoid this damaging effect, we used a hard mask made of TiN. TiN has an etch rate which is 10 times less than photoresist.

Fig. 2 shows the calculated effective angle versus the desired ramp angle for masks made of photoresist and TiN (note: the ratio of the etch rate of DBCO, photoresist and TiN is 5:10:1). The effective etch rate on the ramp surface, for ramp angles between 10 and 30°, is significantly smaller for masks made of TiN, resulting in a small etch rate of the ramp surface and, therefore, less damage on the ramp.

Fig. 3(a) and Fig. 3(b) show AFM micrographs of a ramp made in a sputter deposited DBCO film. The mask, in this case made of photoresist, is aligned (ϕ=0°) with respect to one of the crystal axes of the SrTiO₃ substrate. Fig. 3(b) shows an enlargement of the ramp surface. Subtracting the average plane, the concave appearance of the ramp can clearly be seen. This curvature of the ramp, due to the shift of the photoresist edge during etching, is also seen in HREM studies [3]. Besides this curvature, facets are observed, which are formed parallel to the ramp. These facets are caused by continuous surface reconstruction of the ramp during etching. The facets are about 20 nm wide and up to 500 nm long [4]. Misaligned ramps (ϕ≠0°) show a less regular surface. Ramps, fabricated with photoresist masks, in films made by PLD, with grain sizes much smaller than sputter deposited films, do not show these facets. Observation of the ramp surface in more detail shows another disadvantage due to the effective etching of the ramp surface during ramp milling. The surface shows irregularities all over the ramp area. These irregularities become even more pronounced after annealing the sample at 780 °C. In our opinion these anomalies are formed due to preferential sputtering on the REBCO surface. Cleaning the surface with a soft etch of 50 V did not show any improvement.

These facets and irregularities do not influence the growth of the barrier layer from a microscopic point of view. HREM studies show a well defined interface between the electrodes and the barrier. No extra CuO-layer or different phases are observed. However, problems occur if barriers less than 10 nm are required [2].

Fig. 3. (a) AFM micrographs of a ramp made in a sputter deposited DBCO film. (b) Enlargement of the ramp surface with the average plane subtracted.
To avoid preferential sputtering, the hard (TiN) mask is a good alternative. The ramp surface shows no facets and the irregularities almost disappear. Before annealing, only a smooth surface can be seen. After annealing, the unit cells become visible. Fig. 4 shows a cross-section of an AFM image after annealing at 760 °C.

4. Conclusions

In this contribution we studied the effect of the masks used in multilayer ramp-structures. Using photoresist, a concave shape of the ramp area is observed. The area consists of facets and irregularities, even after etching at ambient temperature. Annealing of these structures will increase these anomalies. This damaged ramp surface is caused by the preferential sputter etching, due to a large angle between the angle of incidence and the ramp angle. The use of hard masks prevents this difference in angles. Furthermore, the effective etch rate for small angles minimizes this damaging effect. Steps of unit cell height are observed after annealing ramps fabricated using hard masks.

References