

Highly reproducible large-area and double-sided pulsed laser deposition of HTSC YBCO:Ag thin films for microwave applications

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Abstract. Large-area pulsed laser deposition (PLD) producing high-quality $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) thin films on both sides of R-plane sapphire substrates with CeO_2 buffer layer is used routinely to optimize planar microwave stripline filters for satellite and mobile communication systems. A relatively simple PLD arrangement with fixed laser plume and rotating substrate, with an offset between the laser plume and the center of the substrate is employed to deposit laterally homogeneous 3-inch-diameter Ag-doped YBCO thin films.

The YBCO:Ag films are about 250 nm thick and have laterally homogeneous critical current densities of more than 3.5 MA/cm^2 at 77 K and homogeneous maps of microwave surface resistance R_s of about $40 \text{ m}\Omega$ at 145 GHz and 77 K. The R_s at 8.5 GHz and 77 K, determined in the center position of the YBCO:Ag films, remains constant at about $370 \mu\Omega$ up to a microwave surface magnetic field of about 10 mT. After experience with more than 700 double-sided 3-inch-diameter films, a high degree of homogeneity and reproducibility of j_c and R_s is reached. The PLD-YBCO:Ag films are suitable for microwave applications envisaged for future communication systems.

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High- T_c superconducting (HTSC) $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) thin films on low-dielectric-loss substrates are suitable candidates for applications as passive microwave devices in future communication systems. Many countries are highly active in the development of microwave devices using HTSC thin films, because in this field real market applications of HTSC subsystems seem to be possible in the near future [1, 2].

Devices like microwave stripline filters for satellite communication systems require HTSC thin films on both sides of large-area single-crystal wafers. Therefore, scaling-up of established HTSC deposition techniques, such as pulsed laser

deposition (PLD), thermal coevaporation, and sputtering to substrate diameters of at least 3 inch is of high relevance. The first attempts to deposit $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) on 3-inch-diameter substrates by using PLD was reported as early as three years after the discovery of the YBCO high- T_c superconductor [3]. Large-area PLD of two-sided YBCO thin films on 2-inch-diameter LaAlO_3 wafers has been performed by using a rotating substrate with an offset between the fixed laser plume and the substrate [4]. PLD of YBCO on 3-inch-diameter LaAlO_3 by using a large rotating target of 90 mm in diameter and rastered by the laser beam results in excellent homogeneity of the superconducting properties [5]. For an information about different arrangements of large-area PLD see the reviews [6, 7].

A highly reproducible PLD process for large-area 3-inch-diameter and double-sided YBCO films on sapphire substrates for microwave applications has been developed and is being continuously improved at the University of Leipzig [8–12]. The electrical and microwave performance of these PLD-YBCO films [10] is comparable to that of high-quality films deposited by other techniques such as thermal coevaporation.

Much research has been concentrated on YBCO films on thermally stable substrate materials like LaAlO_3 or MgO [1, 2]. Sapphire is a less expensive substrate material of lower dielectric permittivity and loss tangent, higher mechanical strength, and good epitaxial surface quality, even over large areas. Therefore it has some advantages as a substrate material of HTSC thin films to be used at microwave frequencies. However, the high-temperature YBCO deposition on sapphire substrates is more complicated because of the necessity of a thin epitaxial CeO_2 buffer layer in between the YBCO films and the sapphire substrate. Moreover, the different thermal expansion of YBCO and sapphire reduces the maximum microcrack-free YBCO thickness on sapphire substrates to about 250 nm to 330 nm, depending on the YBCO deposition technique [13].

This paper describes the state of the art of the development of a highly reproducible large-area PLD technique for HTSC YBCO thin films on sapphire wafers by comparing maps of the critical current density and of the microwave sur-

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face resistance. The results are illustrated by some structural data and selected TEM cross sections. Despite the simple deposition principle of PLD, there are a huge number of difficult-to-handle influences which may affect the quality of the deposited films. For example, accurate control of the laser pulse energy and substrate temperature, homogeneous target ablation, and the cleaning of the deposited laser entrance windows are extremely important for a highly reproducible PLD process.

1 Experiment

YBCO:Ag thin films and CeO_2 buffer layers were deposited by PLD by using a KrF excimer laser operating at a wavelength of 248 nm onto both sides of 3-inch-diameter R-plane sapphire wafers of 430 μm thickness [8–11].

According to the nomenclature proposed by Greer [6, 7], a simple “off axis” PLD technique is used for deposition of CeO_2 , YBCO, and gold. In the “off axis” approach the center of the rotating substrate is offset a fixed amount from the center of the ablation plume. An offset of about 30 mm is used with a target–substrate distance of 110 mm. Figure 1 schematically shows the applied PLD arrangement. Polycrystalline laser targets with diameters of only 18 to 25 mm are used for the PLD on the much larger 76-mm substrates. The Ag content of the PLD YBCO target was optimized with respect to the critical current density (j_c), microwave surface resistance (R_s), and mechanical film properties. High-quality films were deposited by using hot-isostatic pressed or sintered YBCO targets with 7 and 4–5 wt. % Ag, respectively, whereas sintered targets with 7 wt. % Ag resulted in mechanically softer and therefore more sensitive YBCO films. Therefore, the optimized Ag content of the YBCO target seems to be below 5 wt. %, which corresponds to the results obtained by the Pinto group [14]. Compared to Ag-free PLD YBCO films, Ag doped YBCO has reduced surface roughness, slightly lower R_s , and improved homogeneity of R_s [11, 12].

The double-sided films are deposited subsequently at substrate temperatures around 760 °C. The rotating substrate is radiatively heated by a fixed heater element of KANTHAL wire. An additional heater element is applied at the front side at the edge of the wafer in order to improve the ho-

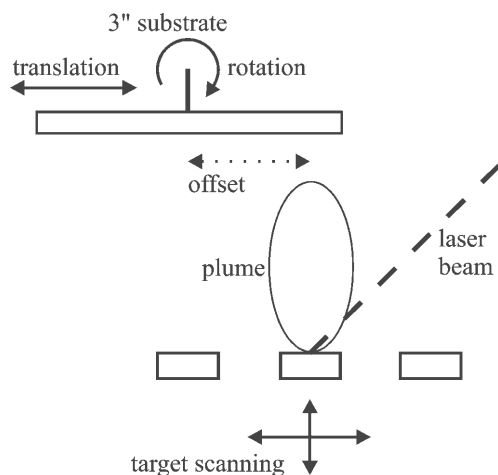


Fig. 1. Diagram of large-area PLD on 3-inch-diameter substrates

mogeneity of the substrate temperature. The deposition rate averaged over the whole 3-inch substrate was optimized to about 0.3 nm/min for CeO_2 and about 10 nm/min for YBCO and gold when a laser fluence of 2 J/cm² was used for CeO_2 and YBCO and a fluence of 5 J/cm² was used for gold. For double-sided deposition on optically transparent 3-inch-diameter sapphire wafers, the heater power must be decreased during the deposition of the first YBCO film in order to oppose the added absorption from the heater radiation by the growing film. The real substrate temperature is measured by a pyrometer operating at an optical wavelength of about 8 μm , which corresponds to the optical absorption edge of sapphire. All the parameters of the large-area PLD process are controlled by sophisticated PC software and a log file is produced for each deposition run. The deposition of several hundreds of 3-inch-diameter sapphire wafers leads to a high degree of film quality and reproducibility.

Figures 2 and 3 show photos of our new homemade PLD system with a 4-inch-diameter substrate heater. This second large-area PLD chamber allows very flexible movement of the substrate with the substrate heater, which is fixed

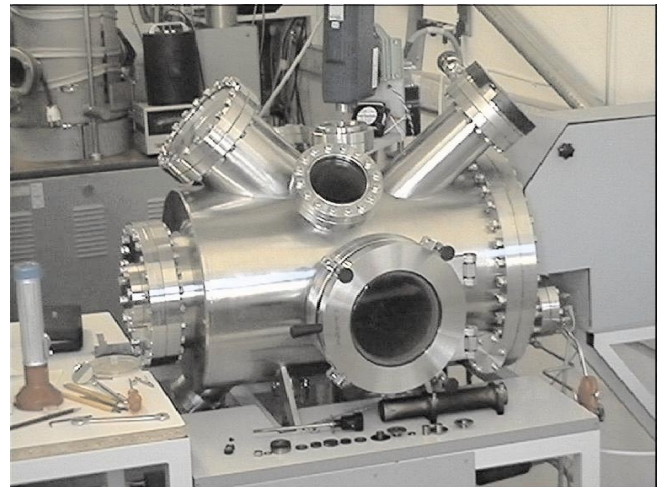


Fig. 2. Front view of homemade PLD system with flexible 4-inch-diameter heater

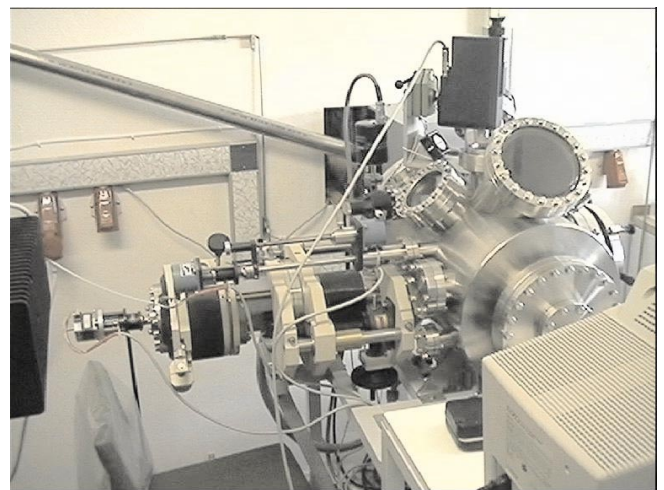


Fig. 3. Side view of 4-inch PLD chamber with rotation and X-Y-Z translation feed-throughs for substrate heater and target holder

to a UHV X-Y-Z manipulator system as shown in Fig. 3. A sputtering chamber for gold films and a substrate manipulator will be added to the PLD chamber in order to allow full in situ deposition of space-qualified double-sided $\text{CeO}_2/\text{YBCO:Ag}/\text{Au}$ film systems on sapphire. In order to further improve the efficiency of the PLD process as a series deposition method, we have developed an 8-inch-diameter substrate holder, which is shown in Fig. 4. This substrate holder will allow simultaneous PLD of three 3-inch-diameter YBCO films.

Most of the PLD YBCO:Ag films are used to make passive microwave filters for the advanced satellite and communication technique at Robert BOSCH GmbH, Stuttgart. Therefore, in the optimization of PLD process we use the critical current density and the microwave surface resistance as important film properties. Mapping of the critical current density j_c at 77 K, employing the inductive and side-selective method [15], is used as a simple and fast routine control of the YBCO film quality. R_s mapping using an open resonator technique at 145 GHz [16] provides information on the lateral R_s homogeneity over the 3-inch-diameter film surface. This information is very important in the control of the lateral homogeneity of the deposition process [12]. The dependence on the microwave surface magnetic field at 8.5 GHz and 77 K was measured with a 16-mm-diameter sapphire resonator in the center of the 3-inch-diameter films [17], giving direct information on the power-handling capability of the films.

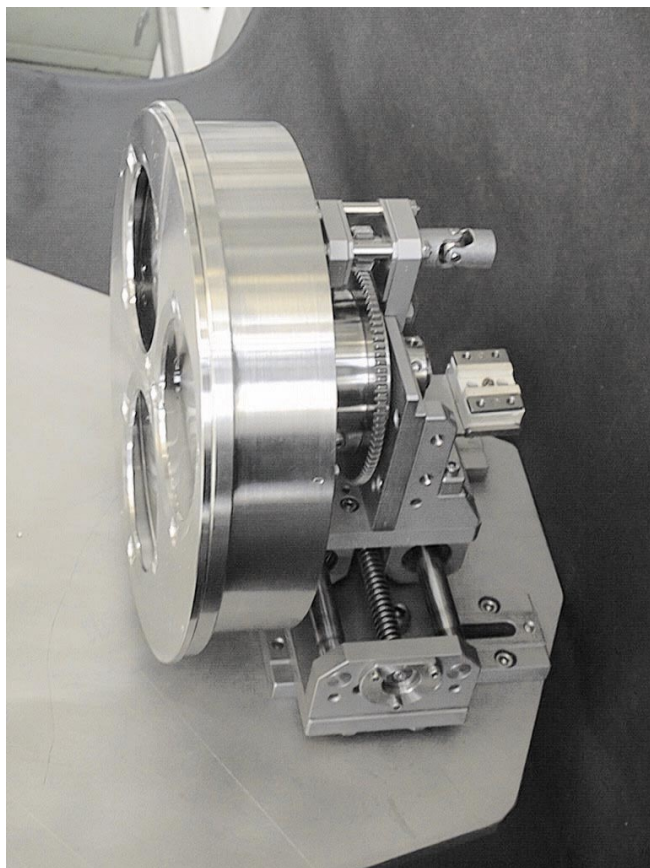


Fig. 4. Homemade 8-inch-diameter substrate heater for simultaneous PLD of three 3-inch-diameter films

Samples for cross-sectional TEM were ground and ion-milled according to the technique described in [18]. TEM was carried out with a PHILIPS CM 30 T microscope equipped with an EDX analyzer at the National Center for HREM (NCHREM), The Netherlands.

2 Reproducibility of large-area PLD

Figure 5 demonstrates the obtained lateral homogeneity and reproducibility of the critical current density of the double-sided 3-inch-diameter YBCO films on sapphire. In more detail, Fig. 5 shows the j_c scans of 10 consecutively deposited double-sided YBCO films on samples S 100 to S 110. High critical current densities of 4 to 5.5 MA/cm² at 77 K with a YBCO thickness of about 250 nm have been routinely obtained by the large-area PLD technique described here. The variation of j_c from sample to sample is nearly the same size as for one single 3-inch-diameter YBCO film. Figure 5 shows the very good reproducibility of the large-area PLD, which has been obtained after more than 5 years of optimization work on the process.

More important than the electrical film properties at low frequencies (some kHz) is the film performance at microwave frequencies (some GHz). Figure 6 shows typical maps of the microwave surface resistance of two double-sided 3-inch-diameter YBCO films, S 070 and S 100, at 145 GHz and 77 K as measured at the Research Center, Karlsruhe. Because of the application of the PLD YBCO films as microwave devices, the microwave properties are definitive for optimization of the PLD process. Figure 6 demonstrates good lateral homogeneity of R_s over single 3-inch-diameter films, which is within the statistical variation of R_s values due to the measuring system. However, from side one (Fig. 6 left) to side two (Fig. 6 right) of the same sample and from sample to sample (Fig. 6 top and bottom) there are higher differences in the levels of R_s , which are due to very small variations in the PLD conditions.

Our PLD process, with sequential deposition of YBCO:Ag films on sides 1 and 2 of the samples, typically produces slightly higher R_s values on side 1 than on side 2 (see Fig. 6). This feature possibly arises from thermal degradation of the first YBCO film on side 1 during the deposition of the second YBCO film on side 2. However, the planar stripline structures of the microwave filters are patterned always on side 2, whereas side 1, which is the ground plane, does not effect the total filter performance so much. Indeed, C-band filters with very good bandpass performance suitable for satellite communication subsystems have been structured from PLD YBCO:Ag thin films at Robert BOSCH GmbH, Stuttgart [10–12].

The microwave power-handling capabilities of selected PLD YBCO:Ag films shown in Fig. 7 have similar characteristics and good reproducibility; the R_s remain constant up to a microwave surface magnetic field of about 10 mT [11, 12]. Considering the rather small film thickness, the best R_s values are comparable to those of films prepared by other techniques and on other substrates. Furthermore, the power-handling capability, as shown in Fig. 2, is as good as for films on structurally better matched substrates like LaAlO_3 or MgO [17].

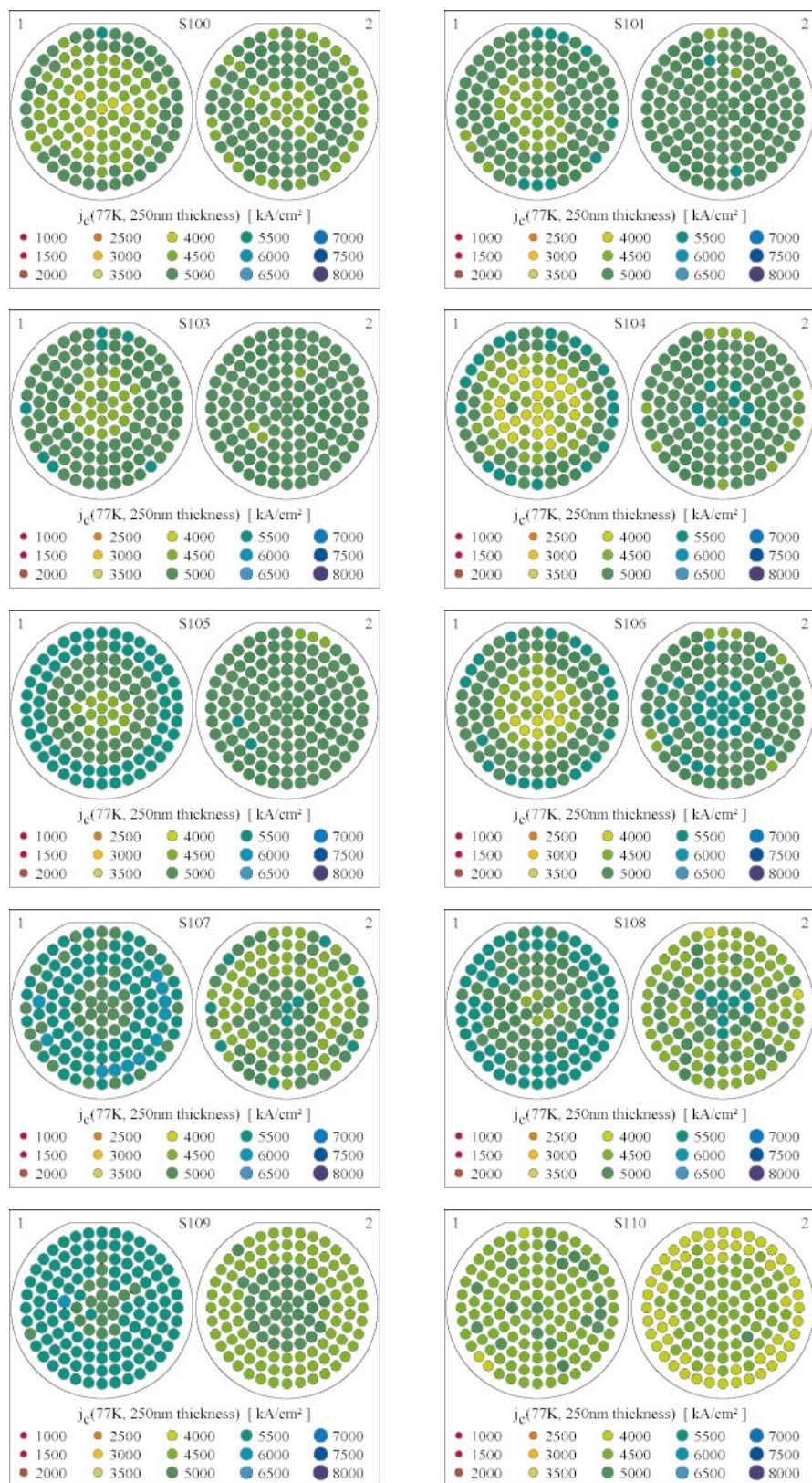


Fig. 5. Reproducibility of the large-area PLD process: j_c maps of 10 consecutively deposited double-sided 3-inch-diameter YBCO:Ag thin films on R-plane sapphire with CeO_2 buffer layer

3 Structure and microwave properties

In order to further optimize the microwave properties of the PLD YBCO films it is important to find correlations to the

chemical composition, the microstructure, and the surface morphology of the films. Table 1 lists the oxygen partial pressure $p(\text{O}_2)$ during PLD, the critical current density j_c at 77 K and $B = 0$, the orthorhombic splitting $\Delta a - b$, and the c -axis

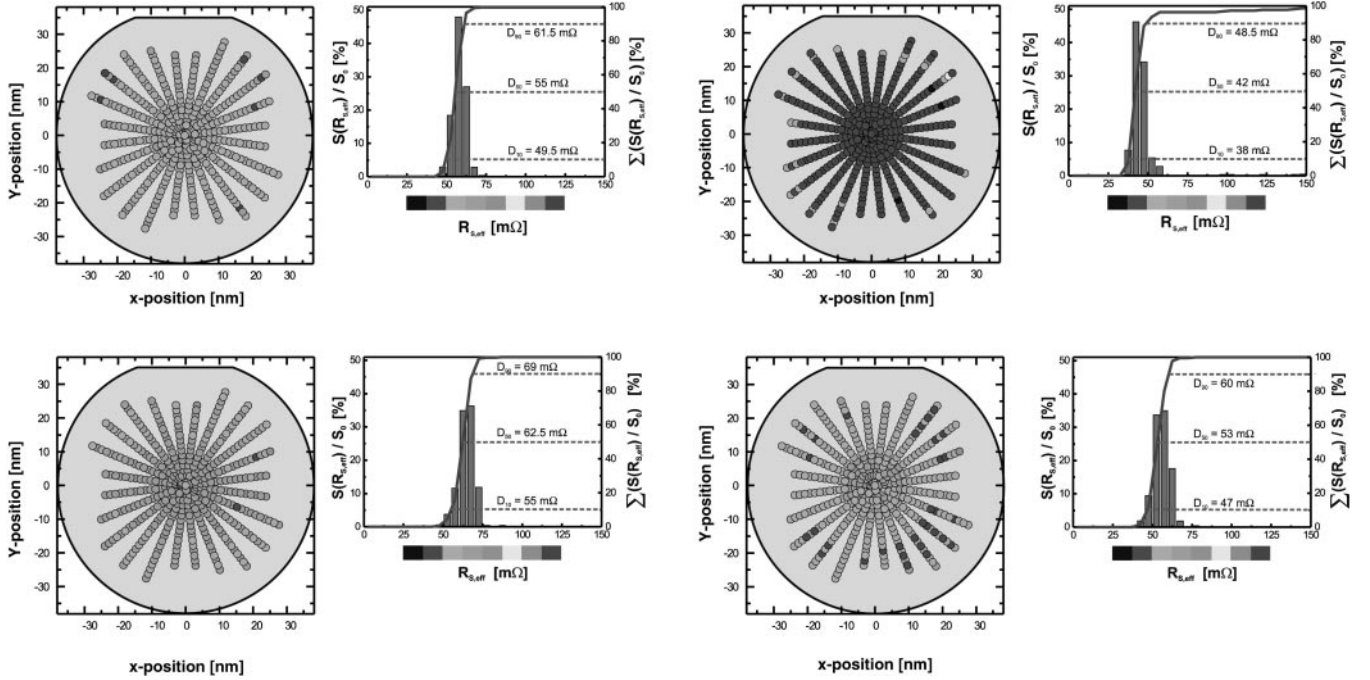


Fig. 6. Lateral homogeneity of microwave surface resistance: R_s mapping with histograms, taken at 145 GHz and 77 K, of two typical double-sided PLD YBCO:Ag films, S 070 (top) and S 100 (bottom), each for side 1 (left) and side 2 (right) of the sapphire wafer (measured by R. Schwab, Research Center Karlsruhe)

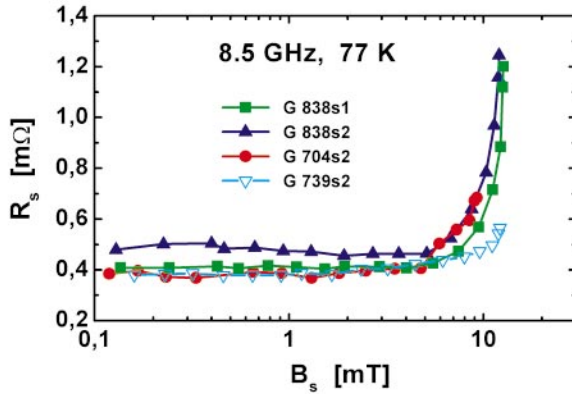


Fig. 7. Power-handling capability of selected PLD YBCO:Ag films on sapphire substrate. R_s is measured at 8.5 GHz and 77 K at different microwave surface magnetic fields B_s (by T. Kaiser at the University of Wuppertal)

lattice constant of YBCO as determined from XRD, the degree of a-orientation (from XRD), the mean size of particulates on the film surface as depicted by SEM, and the uncorrected Y/O and Y/Cu intensity ratios from SNMS depth profiling, together with R_s at 8.4 and 145 GHz (for experimental details see [8–11]) for two 3-inch samples G 702 and G 704. The lower oxygen partial pressure used for sample G 704 corresponds to slightly increased c -axis length, which is the usual measure of oxygen content of YBCO. Incidentally, the reduced orthorhombic splitting of side 1 of G 704 correlates to a reduced Y/Cu SNMS intensity ratio. The J_c of all the films investigated were above 3–4 MA/cm² at 77 K. The degree of in-plane epitaxy as determined by polarized Raman spectroscopy with laser spot sizes of about 0.02 mm² [9] was

Table 1. Structural and microwave properties of two typical large-area double-sided PLD YBCO:Ag thin films on sapphire wafers. The lattice constants and a-orientation were determined by C. Schäfer, MPI Microstructure Physics, Halle/S

Sample no.	G 702		G 704	
	side 1	side 2	side 1	side 2
p(O ₂) /mbar	0.25	0.25	0.16	0.16
j_c^a /MA cm ⁻²	2 to 4	3 to 4	3 to 4	3 to 4
$\Delta a - b$ axis /Å	0.054	0.054	0.049	0.055
c -axis /Å	11.660	11.662	11.665	11.664
a-orientation /%	1.0	0.1	0.1	0.0
size of droplets /nm	500	350	430	280
SNMS Y/O	14.4	15.6	14.6	16.2
SNMS Y/Cu	1.71	1.76	1.67	1.78
R_s^a (8.5 GHz) /μΩ	640	450	470	380
R_s^a (145 GHz) /mΩ	70	65	65	50

^a at 77 K

about 85% to 90% for all the investigated samples, which corresponds to good epitaxial film quality.

The microwave properties are also given in Table 1, which shows a clear correlation between a decreasing R_s and the decreasing size of the particulates on the YBCO film surface, the degree of a-orientation, and the increasing Cu/O and Y/O concentration ratios. Therefore, a reduced oxygen content seems to assist in the optimization of the R_s values of the PLD YBCO films.

4 TEM cross-sections of PLD-YBCO films

Two selected results of the cross-sectional transmission electron microscopy (TEM) investigations of several specimens taken from the YBCO films G 702 and G 704 are shown in

Figs. 8 and 9. The interface between YBCO and the CeO_2 buffer layer was found to be atomically flat despite some imperfections in the sapphire substrate. At the substrate– CeO_2 interface, an amorphous or polycrystalline reaction layer was occasionally found.

Sample G 702 shows on both sides and over the whole 3-inch-diameter film area a wavy TEM contrast with uniform stress distribution in the (001) planes (Fig. 8). The amorphous interface reaction layer in G 702 does not cover the whole substrate and is found to be crystalline rather than amorphous. The broken CeO_2 buffer layer facilitates the formation of interface precipitates which, however, do not introduce any considerable film surface roughness. On the film surface unit cell high singular micro-steps are present. The texture of the *c*-planes parallel to the substrate surface seems to be perfect in Fig. 8, which corresponds to the good properties of the PLD YBCO films. The surface and interface particles in sample G 704 were examined by EDX. It was found that the surface particles are copper oxide, whereas the interface particles are the product of a chemical reaction between Al_2O_3 and

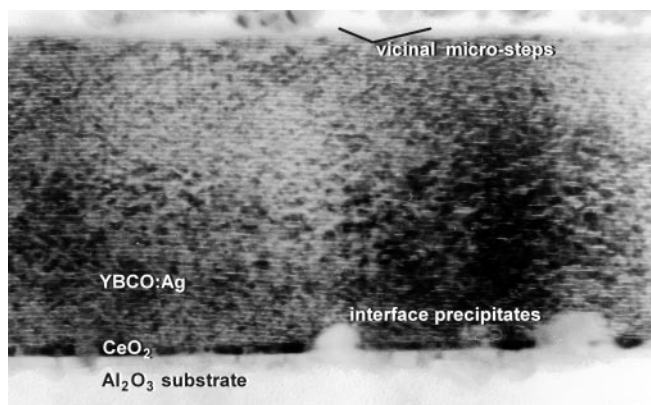


Fig. 8. Cross-section TEM image of PLD YBCO:Ag film G 702 (on side 2, 28 mm from the wafer rim). The surface of the film has unit-cell high singular microsteps and an interface precipitate does not worsen the quality of the film surface. TEM contrast variation corresponds to the stress modulation in the film structure (measured by V. Svetchnikov at NCHREM Delft, The Netherlands)

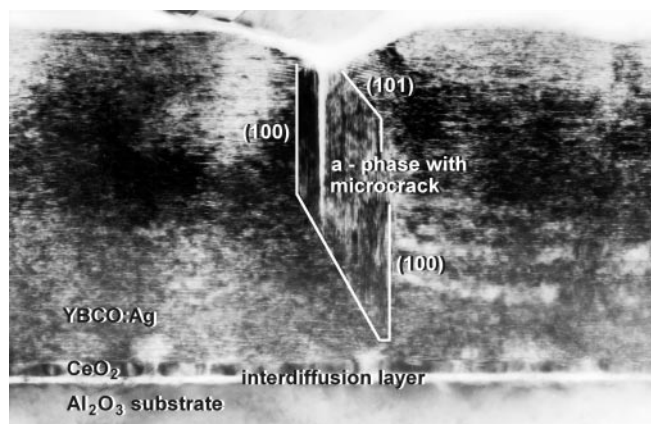


Fig. 9. Cross-section TEM micrograph of sample G 704 (side 2, 20 mm from the rim) showing a microcrack along an *a*-axis-oriented grain. Note that this grain nucleates near the CeO_2 buffer layer and does not exceed the surface of the film (measured by V. Svetchnikov at NCHREM Delft, The Netherlands)

$\text{Ba}_2\text{Cu}_2\text{O}_5$. It was found that the YBCO film in the vicinity of interface precipitates is Cu- and Y-depleted. From SNMS depth profiles we have estimated the Ag content in the PLD YBCO:Ag films to be only about 0.02%, despite the much higher Ag content of 4% in the PLD targets used [11, 12].

Accommodation of the mismatch stresses in YBCO films grown on CeO_2 buffer layers proceeds by dislocation formation in the 103 YBCO film planes [19], and in the case of prevailing mismatch stresses from the buffer layer one can expect that the stresses are mostly in these planes. With long-range stresses from the substrate, it is quite common that the stresses are in (001) YBCO planes. Our TEM results revealed that the stresses in G 702 are mostly concentrated in basal planes with a low-amplitude TEM contrast modulation (in other words, at low stress amplitude), and in G 704 it is expected that the high-amplitude uniformly distributed stresses are in both (pyramidal and basal) planes.

As shown in Table 1, some *a*-oriented grains are found sometimes within the *c*-axis-oriented films. *A*-orientation starts either in film regions with many stacking faults, or near the buffer layer, as shown in Fig. 9. It is interesting to note that in the investigated films, *a*-axis-oriented grains never grew over the level of the film surface, but usually formed a concavity on the film surface (see Fig. 9). The growth rate ratio in YBCO $v_{c\text{-orientation}}:v_{a\text{-orientation}}$ varies from 1:8 to 1:3 depending on $p(\text{O}_2)$ [20]. In our case high $p(\text{O}_2)$ in PLD provides a low growth rate ratio and makes it possible to confine the *a*-oriented grains to the limit of the film thickness.

The microcracking observed by TEM (see Fig. 9) demonstrates only the ability of the thin-film structure to crack, but it is not the same crack found in the bulk film due to the different strain field in the structure [21]. In addition, microcracking was found to strongly decrease the microwave performance and especially the microwave power-handling capability of the PLD YBCO films if they are thicker than the critical thickness of about 250 to 330 nm [22].

5 Conclusions

A PLD technique is presented (Figs. 1–4) that allows fully reproducible double-sided coating of 3-inch-diameter sapphire wafers by thin Au/YBCO/ CeO_2 multilayers with laterally homogeneous critical current densities of 3.5 to 5 MA/cm^2 at 77 K and with about 250 nm of film thickness. The lateral variation of j_c within each 3-inch-diameter YBCO film is of the same quantity as the j_c variation from wafer to wafer for consecutively deposited samples (Fig. 5). Laterally homogeneous maps of the microwave surface resistance R_s of about 40 m Ω at 145 GHz and 77 K have been measured for selected PLD YBCO films. Homogeneous PLD YBCO films show lateral variations of R_s in the range of the statistical fluctuation of the measurement (Fig. 6). However, the variation in the microwave surface resistance from wafer to wafer is considerably higher (Fig. 6). The R_s at 8.5 GHz and 77 K, determined in the center position of the YBCO:Ag films, remains constant at about 370 $\mu\Omega$ up to a microwave surface magnetic field of about 10 mT (Fig. 7). The PLD YBCO:Ag films are suitable for microwave applications envisaged for the future communica-

tion systems of Robert BOSCH GmbH, Stuttgart, Germany. The flexible PLD technique seems to have advantages over other deposition techniques, particularly if more complicated multilayer systems, such as, for example, YBCO/SrTiO₃ films on sapphire substrates with a CeO₂/YBCO buffer layer for use as electrically tunable microwave filters, are deposited.

Detailed inspection of TEM cross sections (Figs. 8, 9) and comparison with the structure and microwave properties (Table 1) of large-area and double-sided YBCO:Ag thin films on R- plane sapphire with a CeO₂ buffer layer revealed correlations that support the optimization of the PLD process.

Further work seems to be necessary to correlate the microstructure and the microwave properties of YBCO thin films on technologically important substrate materials like sapphire, in particular to the effect of special defects in the YBCO structure, and to develop in this way HTSC thin films which meet the requirements of advanced HTSC microwave devices in future satellite and mobile communication systems. At present, even highly reproducible deposition processes result in YBCO thin films with a large number of defects. The microwave surface resistance of very pure YBCO single crystals is the about 4 times lower than that of thin films [23], so there seems to be great potential for improvement in the microwave performance of YBCO thin films. However, the results presented here show the principal suitability of PLD as a series deposition technique for complicated multielement multilayers for advanced applications in the microwave communication technology of the future.

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