TEM investigations of mixed ionic/electronic conducting $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ materials

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Due to the continuing increase of electrical power consumption, the reduction of CO_2-emissions in coal or gas power plants is a major issue. This goal can be reached by enhanced combustion processes applying sophisticated new materials as oxygen separation membranes. One promising candidate is the mixed ionic/electronic conduction (MIEC) $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF) perovskite. However, there is little knowledge about the long term structural stability of the material under application-relevant conditions.

Two series of BSCF samples were examined in the present study in order to determine phase compositions and crystal structures: (i) Thin layers of BSCF were fabricated by pulsed laser deposition (PLD) on Gd-doped ceria substrates (CGO), and (ii) dense BSCF bulk samples were produced by powder processing and sintering. Both series were annealed at different temperatures (700°C, 800°C, 900°C and 1000°C for 10 h). TEM sample preparation was performed by focused ion beam (FIB) milling (PLD samples) and conventional preparation applying mechanical grinding and polishing (bulk samples). TEM investigations were carried out using a Philips CM200FEG/ST microscope.

TEM investigations of the PLD thin films show, as expected, growing grain sizes with increasing annealing temperature (Fig. 1) ranging from 30 nm to 500 nm, respectively. To determine the crystal structure, selected area electron diffraction (SAED) was performed. The diffraction patterns of an adequate number of different grains within one sample, oriented in several different zone axes were compared to simulations performed with JEMS [1]. Crystal structure data of the cubic BSCF perovskite phase given in reference [2] and proposals for a hexagonal phase given in reference [3] were used for the simulations. Fig. 3 shows two examples of these investigations. First results show evidence of the coexistence of a hexagonal and cubic phase in the samples annealed below 900°C. X-ray diffraction (XRD) measurements of the same samples suggest independently the existence of a hexagonal phase below 900°C (Fig.2).

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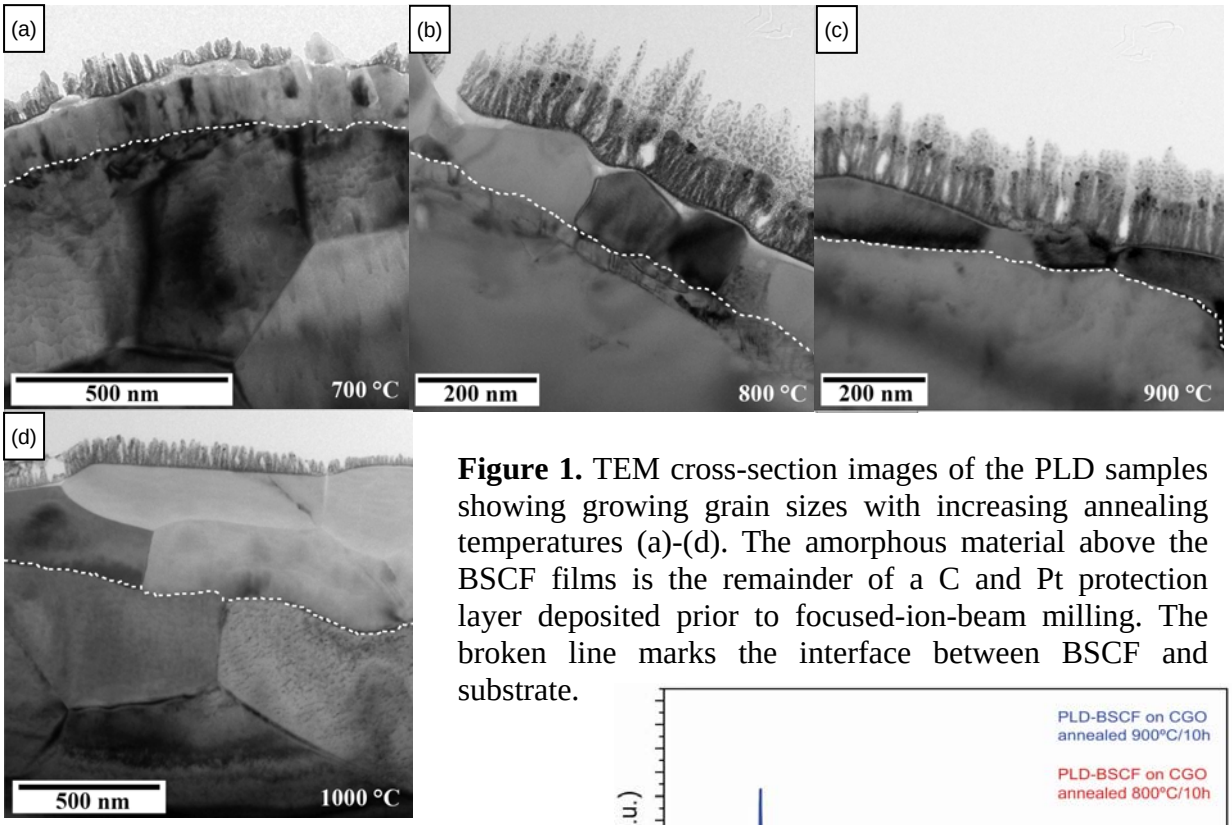

Figure 1. TEM cross-section images of the PLD samples showing growing grain sizes with increasing annealing temperatures (a)-(d). The amorphous material above the BSCF films is the remainder of a C and Pt protection layer deposited prior to focused-ion-beam milling. The broken line marks the interface between BSCF and substrate.

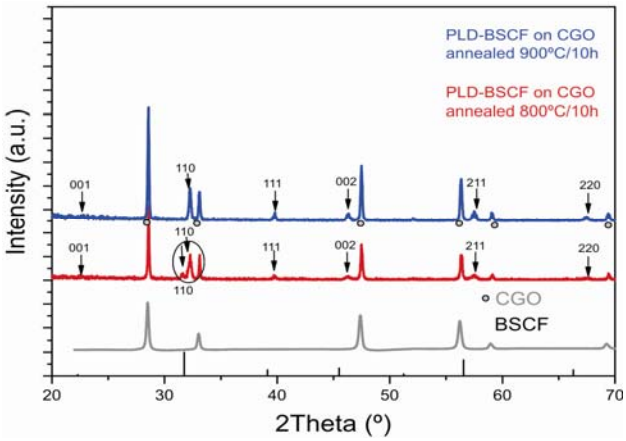

Figure 2. X-ray diffraction spectra of the PLD samples annealed at 800°C and 900°C indicating the formation of a phase with hexagonal structure below 900°C. The corresponding peaks are marked with the circle.

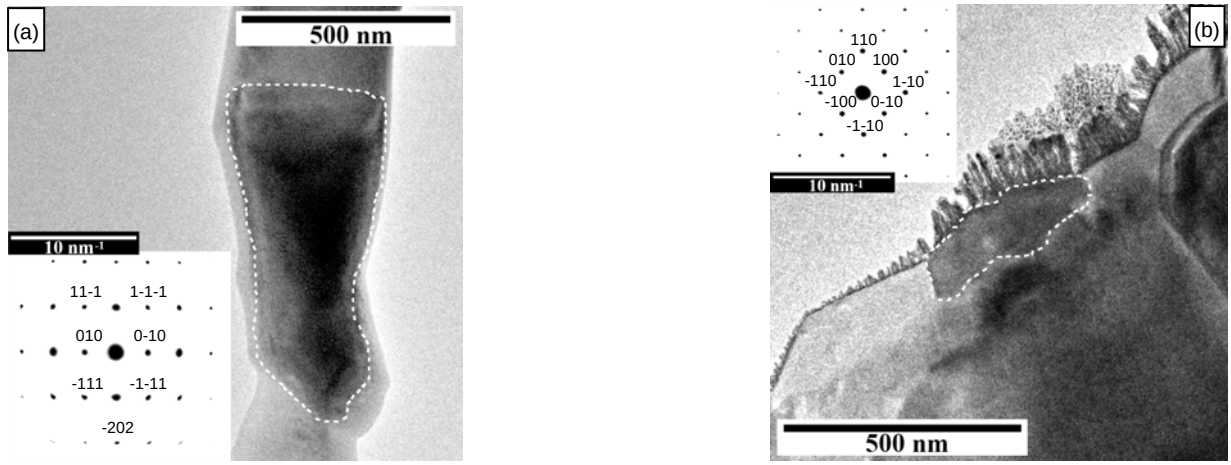

Figure 3. SAED investigations of the marked crystallite (a) of the bulk sample annealed at 1000°C in [101]-zone axis and (b) the PLD film annealed at 900°C in [001]-zone axis. Both show cubic crystal structure with a lattice parameter of 3.93 nm ± 0.03 nm which is in good accordance with Ref. [3].

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