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Assessing the growth window of stannous oxide by ion beam sputter deposition (IBSD)

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SnO_x films were fabricated on soda-lime glass and **sapphire substrates** by reactive ion beam sputter deposition (IBSD). A detailed growth series on *c*-plane sapphire was prepared using heater temperatures from room temperature to 600°C with different oxygen-to-argon ratios to investigate the impact of those parameters on thin film characteristics. By applying X-ray diffraction analysis and Raman spectroscopy an operating window was defined, in which SnO was grown without inclusions of parasitic Sn-related phases. The chemical bonding of the films was examined by X-ray photoelectron spectroscopy (XPS). Only a very narrow regime of oxygen flux was found with a composition close to stoichiometry. Furthermore, variation of the composition in SnO_{1±δ} as well as the growth temperature were found to influence the morphology of the grown layer. The surface morphology was investigated by scanning electron microscopy (SEM) and atomic force microscopy (AFM). An average grain size of ≈ 10-50 nm was revealed for films grown at 200°C, which increases for annealed samples and samples grown at elevated temperatures. In addition to the expected influence on grain growth and surface roughness, disintegration of the SnO crystallites was found for inappropriate oxygen flux and heater temperature, giving way to incorporation of Sn or SnO₂ species.

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I. INTRODUCTION

Tin oxides are sustainable, non-toxic and earth-abundant semiconductors, which makes them an attractive material system for applications such as electronic devices. SnO₂ crystallizes in the tetragonal rutile structure and is an *n*-type semiconductor with a direct band gap of 3.6 eV. It is already widely used as a TCO, gas sensor material, and catalyst¹. The corresponding monoxide, SnO, however is not comparably well described. Mainly this is due its reactivity and instability against temperature. SnO is a *p*-type semiconductor with an indirect band gap of 0.7 eV and a direct optical gap in the range between 2.5 eV and 3.4 eV^{2,3}, the latter being significantly lower than observed in similar compounds like ZnO (3.2 – 3.4 eV). The thermodynamically most stable form α -SnO crystallizes in space group *P4/nmm* being isostructural to PbO. This yields a tetragonal unit cell, cf. Fig. 1, with lattice constants $a = b = 3.8029 \text{ \AA}$ and $c = 4.83824 \text{ \AA}$ ⁴. The layered structure can formally be derived by removing layers of oxygen ions from SnO₂ as described by Seko and co-workers⁵. In the same work, Seko et al. suggest that structures of intermediate stoichiometries can be constructed in a similar fashion, but with fewer missing oxide layers. In a joint experimental and ab initio work, recently published concerning tin oxides, we were able to identify the intermediate phase of tin oxide proposed in several works as Sn₃O₄⁶. To focus on SnO, in this layered network the positive charge of the Sn²⁺ ions is shielded by an electron cloud between

the layers. Coulomb repulsion is therefore reduced⁷⁻⁹. Due to its layered structure SnO shows a strong elastic anisotropy¹⁰. In addition to the tetragonal configuration a orthorhombic modification ($a = 5.00 \text{ \AA}$, $b = 5.72 \text{ \AA}$ and $c = 11.12 \text{ \AA}$) is stated with phase transitions occurring upon high pressure¹¹⁻¹³. Different color impressions observed in literature are most likely triggered by the different shapes and surfaces of the SnO crystals at hand¹⁴.

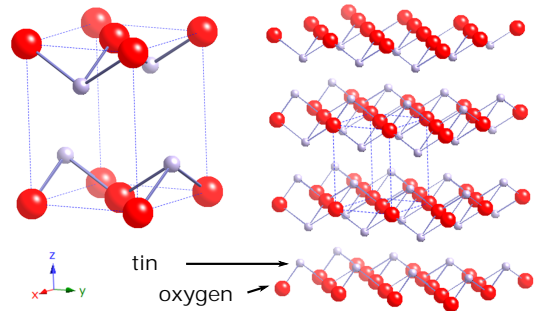


FIG. 1. Litharge structure of α -SnO. Each tin atom is four-fold coordinated (bond length 2.23 Å). Along [001] the crystal appears to consist of layers with Sn_{1/2}-O-Sn_{1/2} sequence and a Van-der-Waals spacing of 2.52 Å between adjacent tin planes¹.

Several fabrication methods have been employed to grow SnO thin films, which include pulsed laser deposition (PLD)¹⁵ and different sputtering techniques. The commonly used substrates are soda-lime glass¹⁶, quartz¹⁷ and (001) yttria-stabilized zirconia (YSZ) substrates¹⁵. When SnO is thermally heated in the absence of ex-

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