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Efficient and stable photocatalyst for photoelectrochemical splitting of water

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The commercialization of photoelectrochemical (PEC) water splitting technology requires an efficient, stable, long service life, low price, and production scalable photocatalyst. Inspired by earlier publications of competing teams and knowledge of the material by the French partner, the project team is synthesizing and investigating the spinel-structured γ - Sn_3N_4 , expected to be suitable for the efficient PEC splitting of water. Two synthesis methods were tested. Firstly, the synthesis of microscopic amounts of the oxygen-free γ - Sn_3N_4 via the reaction of elemental tin with molecular nitrogen, $3 \cdot \text{Sn} + 2 \cdot \text{N}_2 \rightarrow \gamma\text{-Sn}_3\text{N}_4$, in a laser heated diamond anvil cell (LH-DAC) was successfully accomplished¹. The compound formed at pressures between 15.7 GPa and 20 GPa and temperatures $\sim 2000^\circ\text{C}$. However, the presence of unreacted tin was recognized even though nitrogen was introduced in excess.

Secondly, RF-magnetron sputtering was applied to deposit films of γ - Sn_3N_4 on various substrates such as monocrystalline Si-wafers, single crystals of spinel-structured MgAl_2O_4 , glass- and plastic plates. As followed from the preliminary XRD measurements based on a microfocus source delivering $\text{MoK}\alpha$ radiation (μS high Brilliance-Incoatec Microfocus Source Mo ELM33, Incoatec) and an imaging plate detector (MAR345, marXperts), crystalline products were obtained on the first two substrate types while amorphous products were found on the latter two substrate types. The spatially ($\sim 200 \mu\text{m}$) resolved XRD examination, performed in LSPM, showed that the phase composition of the crystalline films depends on their thickness. Those of $\sim 160 \text{ nm}$ in thickness contained γ - Sn_3N_4 and a presently unknown Sn_xN_y phase (patterns A and B in Figure 1). Also, the films deposited on monocrystalline MgAl_2O_4 exhibited a better crystallinity when compared with those on the monocrystalline Si-wafers. Thick films of $\sim 1.3 \mu\text{m}$ contained the unknown Sn_xN_y only (patterns C and D in Figure 1), independent of the substrate.

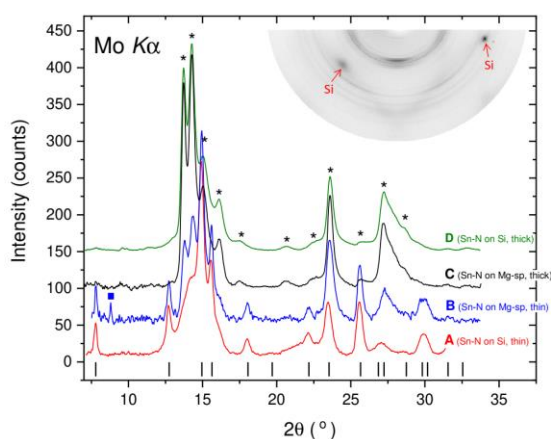


Figure 1: XRD patterns of thin (A, B) and thick (C, D) crystalline films. Vertical ticks indicate positions of the γ - Sn_3N_4 peaks. Stars indicate peaks of an unknown Sn_xN_y phase while a square the (111) peak of MgAl_2O_4 , the substrate material. The insert shows the 2D-XRD pattern of the thin film deposited on a monocrystalline Si-wafer.

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