Poster

Particle- and Phase-selectivity with Micro and Macro X-ray beam at DESY P23 beamline: The Case of $(SmS)_{1.19}TaS_2$ Nanotubes

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Nanotubes (NT) are considered promising materials for future electronics, sensors and nanomechanical devices. However, it is known that the properties of the NT depend on their structure. During the NT synthesis unwanted phases often occur (platelets, amorphic content, etc.) [1, 2] and it is hard or even impossible to disentangle the structural information about the NTs from the data achieved by conventional X-ray diffraction methods, because of the similarity of the lattice constants. The purpose of this study is to show the advantages of anomalous X-ray powder diffraction and microbeam diffraction techniques for the analysis of $(SmS)_{1.19}TaS_2$ NT powders containing a sufficient number of different phases. The $(SmS)_{1.19}TaS_2$ NT belongs to the new class of the NT formed by misfit layered compounds (MLCs) and, in particular, chalcogenide-based MLCs of the general formula $[(MX)_{1+y}]_m[TX_2]_n$ (abbreviated here as MX-TX_2; M=Pb, Sb, Bi, Sn, rare-earth atom; T = Ta, Nb, Ti, Cr, V; X=S, Se). The diffraction anomalous fine structure (DAFS) technique, based on the measurement of the diffraction peak intensity in the vicinity of the X-ray absorption edge, brings spectroscopic information about the structure of the tubes in the powder mixture. Contrary to conventional XAFS spectroscopy, with the DAFS technique it is possible to measure the XAFS-like signal from a certain phase or crystallographic site separately by choosing the proper diffraction peak, thus providing phase- and site-selectivity [3]. Microbeam spectroscopy and diffraction allow one to focus the X-ray beam on a certain microparticle and perform a single-particle structural and XANES analysis.

Microbeam X-ray diffraction analysis of single NTs has shown (Fig. 1), that they have several diffraction features, that distinguish them from the conventional 3D crystals and single 2D layers, that are based on the same structural units/layers (Carbon nanotubes vs Graphite, etc). Due to the lack of out-of-plane symmetry, the diffraction patterns of the NTs don't show the distinct reflections of the *h*0*l* and 0*kl* type, which distinguish them from the bulk particles; contrary to the single 2D layers the multilayered NTs show the reflections of 00*l* type due to diffraction from the basal planes. These 00*l* and 0*kl* reflections can be used to get information about the NTs using DAFS from NT powder.

To understand the growth and stability of $(SmS)_{1.19}TaS_2$ NT a set of powders were synthesized by the chemical vapor transport method (CVT) [2, 4] at different temperatures and annealing times. The powder X-ray diffraction, single NT microbeam X-ray diffraction and DAFS methods were used to evaluate the structural changes and suitable reaction conditions.

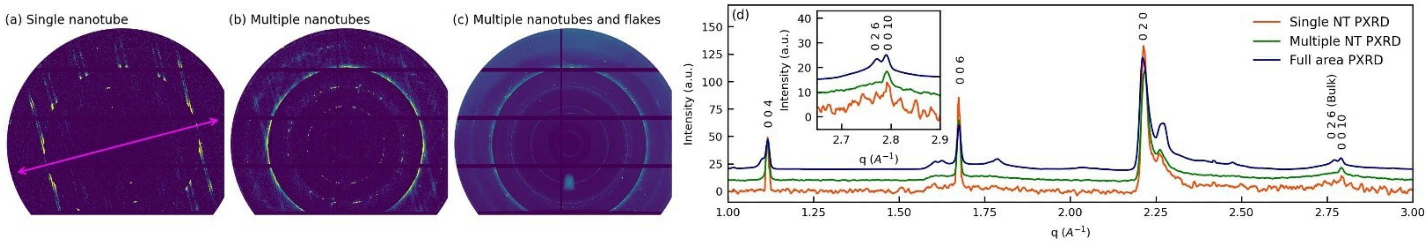

Figure 1. XRD patterns obtained with sub-μ X-ray synchrotron beam from $(SmS)_{1.19}TaS_2$ prepared at 825 °C. (a) XRD pattern from a single nanotube, the nanotube axis is marked with a pink double arrow; (b) Integrated XRD pattern collected from many different nanotubes; (c) Integrated XRD pattern from the entire $(SmS)_{1.19}TaS_2$ sample containing both nanotubes and flakes. (d) Azimuthally integrated (a), (b) and (c) patterns imitating filtered 1D X-ray diffractograms from single nanotube, pure nanotube powder and full sample containing nanotubes and MLC flakes.

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