

## MS13. New instrumentation, methods and approaches in inorganic crystallography

Chairs: Anton Meden, Damien Jacob

### MS13-P1 Nano-diffraction in STEM and fluctuation electron microscopy of phase-change material

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We investigated the phase-change materials  $\text{Sb}_2\text{Te}_3$  by nano beam diffraction in a scanning transmission electron microscope (nano-diffraction in STEM) and  $\text{Ag}_4\text{In}_3\text{Sb}_{67}\text{Te}_{26}$  by fluctuation electron microscopy (FEM). Both methods rely on illuminating the areas of interest of the sample by a small (probe size is around 2 nm) and almost parallel (convergence angle of 0.54 mrad) electron probe. This creates nano diffraction patterns with the spatial resolution of the probe size. We also extract information about the nanometer scale medium range atomic order (MRO) of amorphous materials by calculating the variance out of 500-1000 nano-diffraction patterns of amorphous material. This technique is called STEM-FEM [1]. The nano diffraction in STEM is used to identify crystalline regions of  $\text{Sb}_2\text{Te}_3$  samples. The FEM is used to gather information about the MRO of  $\text{Ag}_4\text{In}_3\text{Sb}_{67}\text{Te}_{26}$  samples. To help to understand the reaction mechanism of the wet chemical synthesis of hexagonal  $\text{Sb}_2\text{Te}_3$  platelets, we investigated  $\text{Sb}_2\text{Te}_3$  intermediates of the reaction with nano diffraction in STEM. By scanning the intermediates with the electron probe and collecting nano diffraction patterns at the same time, it is possible to identify the crystalline areas of the intermediate. The investigation by nano diffraction helped to reveal part of the reaction mechanism of  $\text{Sb}_2\text{Te}_3$  platelets in the wet chemical synthesis [2]. These platelets would have promising application in memory applications or as model systems. A deeper understanding of the crystallization kinetics of  $\text{Ag}_4\text{In}_3\text{Sb}_{67}\text{Te}_{26}$  is needed, because the crystallization speed is the limiting factor of the writing speed of possible phase change memory devices using  $\text{Ag}_4\text{In}_3\text{Sb}_{67}\text{Te}_{26}$  [3]. The MRO of

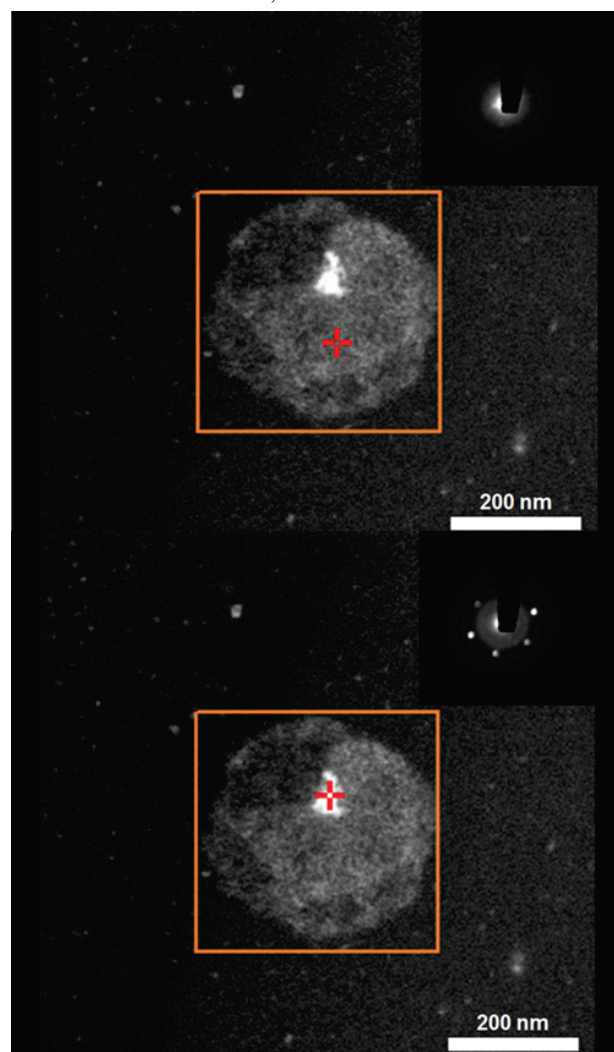
the amorphous phase of  $\text{Ag}_4\text{In}_3\text{Sb}_{67}\text{Te}_{26}$  could play an important role in the difference of crystallization speeds of as-deposited and melt-quenched  $\text{Ag}_4\text{In}_3\text{Sb}_{67}\text{Te}_{26}$ . The normalized variance is calculated by doing FEM in STEM in a STEM dedicated Titan.

References:

[1] Voyles, P.M. and D.A. Muller, Fluctuation microscopy in the STEM, in *Ultramicroscopy*. 2002. p. 147-159.

[2] Saltzmann, T., et al., Shape without Structure: An Intriguing Formation Mechanism in the Solvothermal Synthesis of the Phase-Change Material  $\text{Sb}_2\text{Te}_3$ . *Angewandte Chemie International Edition*, 2015.

[3] Salinga, M., et al., Measurement of crystal growth velocity in a melt-quenched phase-change material. *Nature Communications*, 2013. 4.



**Figure 1.** The boxes are marking the region scanned by nano-diffraction. The crosses are marking the actual positions of the electron probe. The nano-diffraction patterns shows, that the main body of the intermediate is amorphous. Only a small part of the bright core part of the intermediate is crystalline.

**Keywords:** Nano-diffraction, TEM, STEM, phase-change, FEM