Functional Materials

MS.3.P054
Characterization of laser-deposited Ge-Sb-Te thin films by Cs-corrected STEM

U. Roß[1], A. Lotnyk[1], E. Thelander[1], B. Rauschenbach[1]

[1]IOM Leipzig, Leipzig, Germany

ulrich.ross@iom-leipzig.de
Keywords: phase-change materials, STEM, Cs-correction

Phase-change materials refer to a class of intermetallic glass-formers with a reversible crystallization process between the amorphous phase and an intermediate metastable crystalline phase, accompanied by a large change in optical and electric properties. Common uses include optical data-storage discs as well as electrical phase-change memory devices. While the application in optical data storage is well-developed, the exact mechanism of crystallization that enables the rapid switching between amorphous and metastable crystalline state is not fully understood[1].

In the work presented here, we make use of the pulsed laser-deposition process (PLD) in order to produce thin films of the prototypical phase-change material system $(GeTe)_x$-$(Sb_2Te_3)_{1-x}$ (GST) on single-crystalline silicon substrate covered by a silicon oxide buffer layer[2]. These films are investigated by probe Cs-corrected analytical transmission electron microscopy (TEM) and spectroscopy (EDX/EELS) using a Titan^2 G2 80-300 kV STEM microscope equipped with Super-X fourfold EDX detector arrangement and a Gatan GIF post-column filter, in order to assess deposited quality, optimize the deposition process and gain insight into the crystallization process.

Due to the inherent beam sensitivity of the sample material, we had to optimize a suitable TEM sample preparation technique. Using a Zeiss dual-beam FIB and a liquid nitrogen stage-cooled Fischione Nanomill low-energy argon ion mill, we are able to prepare 10-20 nm thin FIB-lamella suitable for EDX/EELS spectroscopy as well as for high-resolution TEM and STEM imaging with minimal damage to the pre-existing sample crystal structure. In order to minimize the influence of the electron beam during investigation, we strive to apply minimal beam currents at 80 kV acceleration voltage. Since low beam currents are especially detrimental to the signal-to-noise ratio of the spectroscopic techniques, a compromise between beam settings and beam exposure times during the measurements had to be found.

In order to assess the crystallization behaviour, the as-deposited films are irradiated locally with excimer laser pulses of varying fluences, resulting in grids of recrystallized GST in amorphous matrix (Figure 1.a)). FIB-lamellas are cut from the edges of these irradiated spots in order to access the amorphous-crystalline transition zone as well as both pure phases. Layer thickness and crystallinity were investigated by bright-field TEM and selected area diffraction (Figure 1.b)), confirming the formation of the metastable phase. High-angle annular dark-field STEM in conjunction with quantitative EDX mapping allows us to determine the local mass-thickness as well as elemental distribution. Figure 2. shows a separation of the Ge content from the Sb_2Te_3 phase. At high magnifications, individual crystallites can be imaged down to their local atomic arrangement depending on crystallite orientation (Figure 3.) without major changes of the crystal structure during investigation. EELS spectra are recorded both in EFTEM imaging as well as STEM mode (Figure 4.), revealing both individual high-loss elemental edges as well as low-loss features related to the local band structure.

We have therefore successfully applied the FIB/low energy ion milling sample preparation and TEM/STEM investigation in order to reliably assess the as-produced and laser-crystallized GST thin films with minimal damage to the sample structure.

1. A. Kolobov, P. Fons, A. Frenkel, A. Ankudinov, J. Tominaga, T. Uruga, Nat.Mat. 3 (703-708) 2004
2. H. Lu, E. Thelander, J.W. Gerlach, D. Hirsch, U. Decker, B. Rauschenbach, Appl. Phys. Lett. 101 2012) 031905.
3. The financial support of the European Union and the Free State of Saxony (LenA project) is greatly acknowledged.

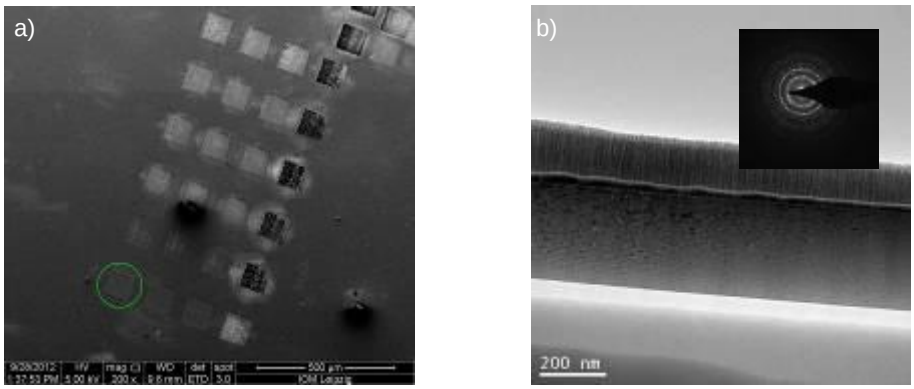

Figure 1. a) SEM surface image of GST film with laser-irradiated squares, green circle marks square selected for FIB preparation, b) bright-field image of amorphous-crystalline transition zone cut from area indicated in a), inset in b) shows local area electron diffraction pattern of the GST film.

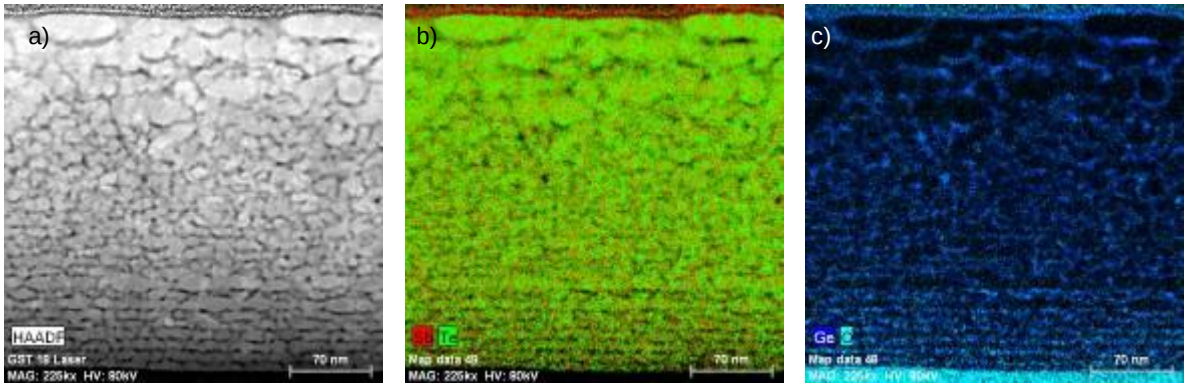

Figure 2. a) HAADF-STEM image of 50 mJ/cm² laser-recrystallized GST film as shown in Figure 1, b) EDX map of Sb/Te signal, c) EDX map of Ge/O signal

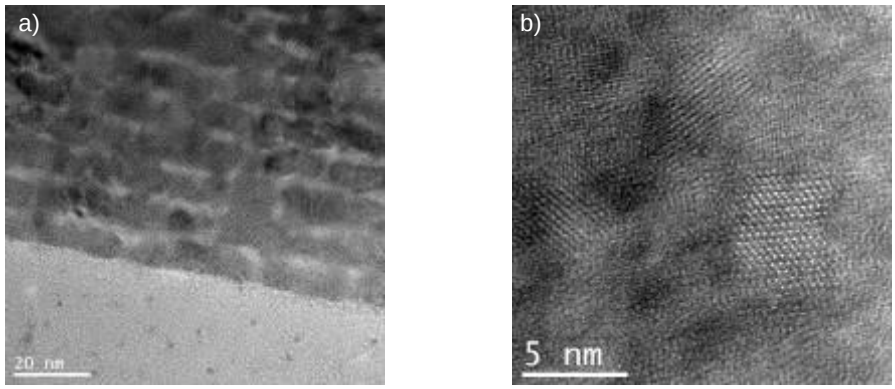

Figure 3. a) HRTEM image of interface SiO substrate – crystallized GST, b) HRSTEM image of GST crystallites

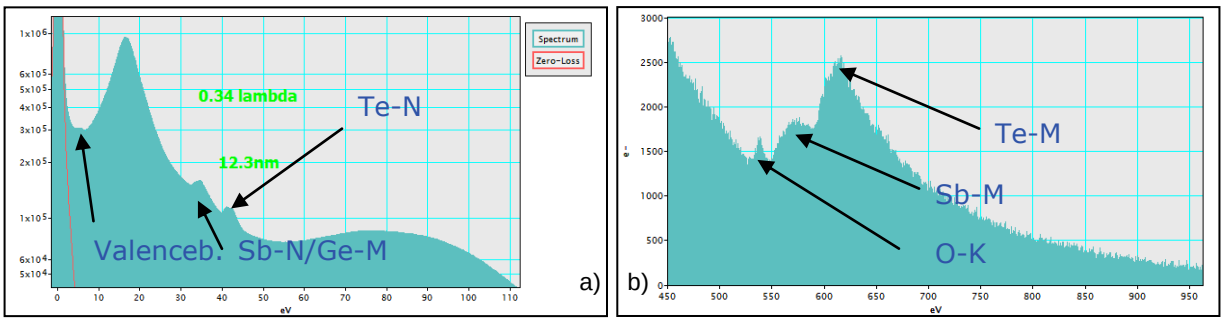

Figure 4. a) Low-loss region of GST EELS-spectrum from amorphous sample region, b) high-loss features (EELS edge fine-structure) from identical sample region.