ESRF	**Experiment title: "In situ study of crystallization mechanisms of metastable amorphous GeTe and GeSb6 thins films used in phase change random access memory (PCRAM) "**	**Experiment number**: 02-02-775
Beamline: BM02	**Date of experiment**: from: 10 November 2010 to: 14 November 2010	**Date of report**: 14/02/2012
Shifts: 6	**Local contact(s)**: Jean-Paul SIMON	*Received at ESRF:*
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Proposal summary:

Phase Change Materials (PCM) such as GeTe or Ge2Sb2Te5 (GST) are currently excellent candidates for use in non volatile memories in Phase Change Random Access Memory (PC-RAM). In PC-RAM, the material switches from a high resistance amorphous state to a low resistance crystalline state, which allows to store information. For applications a high retention time, a high thermal stability of the amorphous phase and a high cyclability are mandatory. GeTe and GeSb6 have shown promising results with respect to thermal stability. The aim of this proposal was to anneal in situ during the diffraction experiments some films of GeSb6 and GeTe, 30 and 150nm thick. Although GeSb6 was a promising phase change material (REF 3 of proposal: Cabral et al., Appl. Phys. Lett. 93 (2008) 071906.), difficulties met later in its integration in PCRAMs have lead to discard its use. Therefore we focused only on the study of the crystallization of GeTe. Morover, confining the phase change material has a strong impact on the crystallization mechanism. For GST thin films it has been demonstrated that the crystallization temperature increases when reducing the thickness of the film under 30nm, with a clear influence of the interface material (R. E. Simpson et al., *Nano Lett.*, 2010, 10 (2), pp 414–419). Considering the continuous scaling of devices and recent studies performed at CEA-LETI, we extended the thickness range down to 5 nm and studied the impact of the material interface.

Experiment & results:

Samples of GeTe thin films with different material interfaces (Ta, TiN and SiO_2) have been prepared. For each interface material, GeTe film of various thicknesses has been deposited: 5, 10, 30 and 100 nm. The incident X-ray energy has been chosen equal to 17.485 keV and the 2D detector was the CCD Princeton@ camera (16 bits dynamics & 1340x1300 pixels of 50x50 μm^2).

In order to find the best conditions for the in situ experiments and check the feasability of measurements on the thinnest samples, we first performed X-ray diffraction on samples annealed ex situ (one sample of each kind) for 15mins@450℃. The 2θ range has been chosen between 10° and 25° and each sample has been measured both in the θ-2θ configuration (angle of incidence 8.5°) and in the same 2θ range with an incident angle of 2°. From these ex situ experiments we found that crystalline films interfaced with SiO_2 are strongly textured, those interfaced with TiN are only slightly textured while those interfaced with Ta are isotropic. We validated the possibility of measuring the thinnest samples (5 nm).
To perform X-Ray diffraction experiments during in situ annealing we used an Anton Paar DHS900 furnace equipped with a PEEK plastic dome. We had difficulties in installing the experimental setup and in choosing the best dome. Finally we have been able to measure films of 5, 10 and 30 nm interfaced with SiO_2 and films of 10 and 30 nm interfaced with TiN. For each sample the temperature has been raised by steps of 5℃ each 10 minutes and three measurements were performed for each temperature step. Some runs were spoiled by bad command transmission to the furnace current supply. Quantitative analysis of the images is in progress.