Direct nanoscale 3D characterisation of Ge-Sb-Te and Ge-Te phase change films.

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ABSTRACT

Over the years of research and development Phase-Change Memory has demonstrated its potential as a non-volatile storage media capable of overtaking Flash memory. And as this new technology pushes its way into the high volume manufacturing phase the experimental validation of the scalability becomes of great importance. Direct investigation of failure mechanisms for instance, such as heater/PCM void formation or delamination and pre-existing nucleation site configurations leading to rapid current path formation, becomes a crucial aspect of the technology's constant development. In this paper we propose a new methodology for PCM investigation and characterisation on the nanoscale. Results on $Ge_2Sb_2Te_5$ thin film samples investigation via Beam Exit X-section Polishing (BEXP) and material sensitive Ultrasonic Force Microscopy (UFM) are presented. The results on GeTe sample investigation will be presented elsewhere.

1 INTRODUCTION

In the last decade Phase-Change Memory (PCM) technology went from a perspective concept to a ready consumer device piloted by Samsung [1]. And being a solid-state type memory, a major concern for it is scalability, in particular that of the cell structure and of the electrical parameters (mainly the reset current) without any degradation of the reliability aspects With this thought in mind we quickly come to dozens of different designs and threir experimental validation [2],[3],[4],[5]. A great deal of work has been carried out with FIB and TEM on structure investigation and characterisation [6],[7],[8],[9] and seeing the limitations of these techniques (cost, accessibility, time consumption, properties investigation) it seems an alternative approach is needed.

In this study we propose such a methodology, based on material sensitive imaging via SPM, in particular Ultrasonic Force Microscopy [10] and Ion Beam Exit X-section Polishing [11]. Due to the probe-sample surface contact constriction BEXP was implemented to produce atomically flat sections of the different phase films in order to enable sub-surface, or 3D, profiling and property mapping.

Keywords: Ultrasonic Force Microscopy, Phase-Change Materials, Beam Exit X-section Polishing, $Ge_2Sb_2Te_5$

2 EXPERIMENTAL DETAILS

2.1 PCM array sample preparation

Thin films (100 nm) of $Ge_2Sb_2Te_5$ (GST) were deposited at room temperature on Si and Glass substrates by RF magnetron sputtering (Moorfield MiniLab 25) using single alloy targets, 2" diameter, with the 2:2:5 composition. The background pressure inside the chamber was typically 3×10^{-5} mbar and Ar pressure of 4×10^{-3} mbar. Typical power applied was 25 W. The resulting deposition rate was 3.5 Å/s. XRD data confirmed a predominantly amorphous phase in the as-deposited films. Film samples to be amorphized were anealed prior in ambient conditions at 160°C. Diffraction measurements were carried out during the process in order to affirm the as-deposited amorphous-to-fcc phase transition. 4Ω measurements were performed on GST/glass samples with a 4 order sheet resistivity drop upon phase change. An Ar Blue (457-514 nm) continuous laser source together with an optical chopper and focusing optics system was used to produce sub-micron crystalline arrays in as-deposited matrix with pulse times of 200 μs and power outputs ranging from 5-30 mW for individual dots up to 60 mW for continuous lines. A EKSPLA pump pulse Nd:YAG (532 nm) 30 ps laser system was used to produce sub-micron amorphous dot arrays in crystalline matrix with pulse energies ranging from 100 to 200 nJ. In both laser setups a lens of 0.65 NA was used to focus the laser light down to a 1 μm diameter spot on the film surface.

2.2 Beam Exit X-section Polishing

The equipment used for BEXP in this study is the Leica EM TIC020 Ion Beam Cutter with a modified sample positioning apparatus and a different ion milling operation. An argon ion beam is produced and directed towards the sample and shield plate to mill the unshielded part of the sample. This results in a sample section that is of the order of 1-2 mm in width and which is produced perpendicular to the plane of the shield plate as shown in Fig. 1(a). A stereo microscope together with a CCD camera are used for sample aligning before the initiation of the procedure and controlling the progress of the milling. The process reaches a final stage when the geometrically sharp lower part of the ion beam defined by the mask mills its way out of the

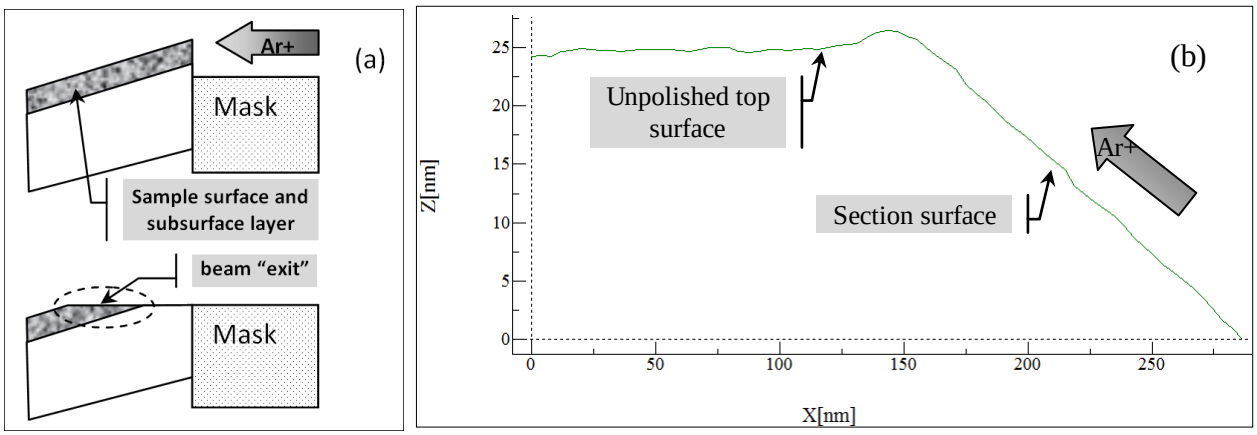

Figure 1: a) Schematic illustration of the principle of BEXP; b) an AFM topography image of a GST film surface sectioned by BEXP shows a close to perfect cut with flat prismatic surfaces and dimensions well in the sub-micrometre range.

sample, or "exits", from underneath the top face creating an atomically sharp edge (Fig.1 b). This edge is defined by the newly polished facet adjoining the top face of the sample at a $\sim\pi + \alpha$ angle, where α, or tilt, is the angle between the top face plane and the ion beam axis. This tilt is usually chosen between 2-10° for producing an edge angle closest to an open one. The purpose of this is to prevent extreme cantilever movement in the Z axis during subsequent probe scanning (which is limited by the dynamic range of the piezoelectric scanner) and the creation of unbalanced forces on the scanning microscope that is known to deteriorate image quality, especially for ones generated in the ambient environment where a water meniscus between the probe and the sample might be present. The method is developed and patented by the authors together with Lancaster University. The method is tailored for SPM studies specifically, as the close to open angle ridge profile of the ion beam polished and pristine surfaces does not adversely affect probe scanning.

A PMMA masking procedure was adopted for the purpose of pushing the sputtering edge effects from the GST film onto the sacrificial layer. After the photoresist deposition and a quick 30 second annealing step at 100°C the GST sample's edge of choice for sectioning was lapped with diamond paper in order to adjust the samples geometry to the holder and mask stage inside the BEXP chamber. After the Ion Beam process the PMMA film was removed via ultrasonication of the sample in a photoresist remover solution for 15 minutes, followed by a standard semiconductor cleaning procedure: 10 min. ultrasonication in Acetone → 10 min. in IPA → Clean Nitrogen blasting.

2.3 Ultrasonic Force Microscopy

The Equipment used for UFM is the DI Multimode with the addition of standard lab test equipment (lock-in, oscilloscope, wave generator).In UFM mode a sample is placed on top of a piezoelectric plate that oscillates at a frequency, that far exceeds the normal Atomic Force Microscopy cantiliver's non-contact and contact resonance frequencies. This produces the effect of a dynamically stiff cantilever during contact with the sample, which in turn allows the tip to indent or elastically deform sample materials that are of same or higher stifness in a normal contact mode. And although there is no detectable deflection of the cantilever on the high frequency scale (2-10 MHz) due to the high Force vs. Distance nonlinearity of a tip-surface contact a net force, or "ultrasonic" force, is detectable by the SPM at a superimposed low frequency modulation (usually of a saw tooth amplitude modulation, 1-3 KHz). This signal is intertwined with the normal "linier" deflection signal and can be separated and analysed with help from additional lab test equipment (Fig. 2). The nonlinear deflection signal is directly related to local stiffness inhomogeneities of the sample surface and subsurface allowing for mechanical characterization of a wide range of solid state materials and structures.

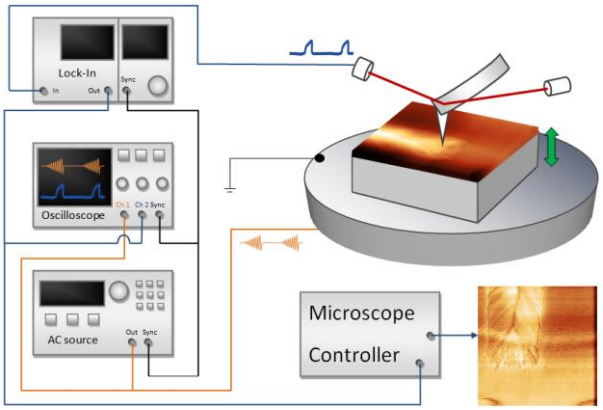

Figure 2: Schematic illustration of a UFM setup.

3 RESULTS AND DISCUSSION

3.1 GST surface imaging

Due to the density shifting upon phase switching the UFM imaging can be complimented by contact mode Atomic Force Microscopy topographical contrast. As expected the crystalline regions have compressed by a few nm (Fig.3a) and the amorphous region expanded by a few nm (Fig.3c) in the Z axis. The UFM and AFM are taken simultaneously, the Microscope operation mode is Contact mode and the cantilever used is a conventional full contact one with a spring constant of 0.2 N/m. Figures 3b and 3d clearly show a big difference in nanomechanical elasticity maps of different phase regions demonstrating the UFM's phase sensitivity in the application to PCM surface characterisation.

3.2 GST section imaging

No phase contrast was found upon investigating the GST section surface in regards to the shielded top surface of the sample, thus we can state that the normal IB operation does not induce a phase switch (Fig. 4). The slight surface/section contrast discrepancies can be attributed to the partially different surface roughness of IB polished and sputter-deposited surfaces and the section area tilt which might change the AFM probe/surface contact area thus producing a slightly different ultrasonic response. Comparing the two with the silicon substrate contrast the difference is much more profound.

Figure 5 shows a crystalline line sectioned by BEXP. The topographical contrast of the crystalline phase is lost upon the IB step procedure but the nanomechanical contrast is clearly visible in the UFM image. The crystalline phase extends 40-50 nm into the thickness of the 100 nm film. The Si contrast is lost due to the different sample orientation and the fast scanning axis of the AFM, UFM Silicon contrast not seen due to the offset flattening needed to extract the different GST phase contrast. The sample structure is identical to the one in Fig.4.

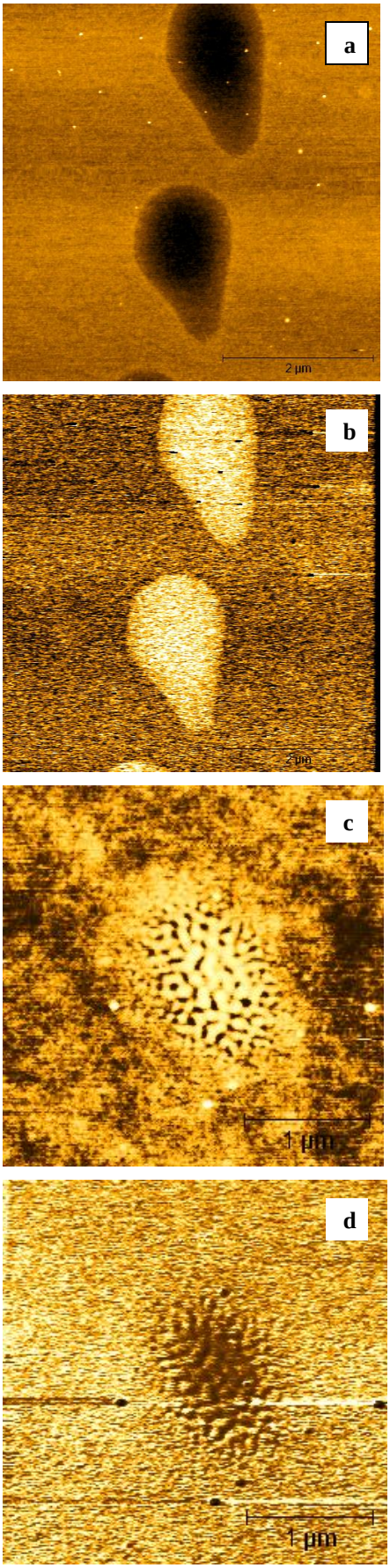

Figure 3: a) AFM topography image of crystalline bits in an as-deposited GST matrix, z range is 5 nm; b) UFM image taken simultaneously with (a) revilling good material sensitivity with the matrix being more compliant, the mark – more stiff ; c) AFM topography image of an amorphous bit in a crystalline GST matrix, z range is 8 nm; d) UFM image taken together with (c) again showing good contrast and resolution capabilities inside the mark with an inverse local stiffness distribution compared to the (b) image. In both cases the marks seem elongated, which is due to the slight optomechanical misalignment during GST switching.

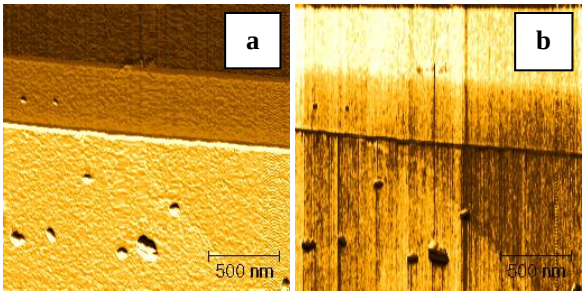

Figure 4: a) AFM topography image of Si substrate/GST section/GST top surface from top to bottom respectively. Si and GST sectioned at a slight relative angle due to different sputtering yield angular dependence. The image is flattened, so the contrast is exaggerated, the actual tilt being ~1°; b) UFM image of the same structure. The sectioned GST and top surface GST showing identical conrast.

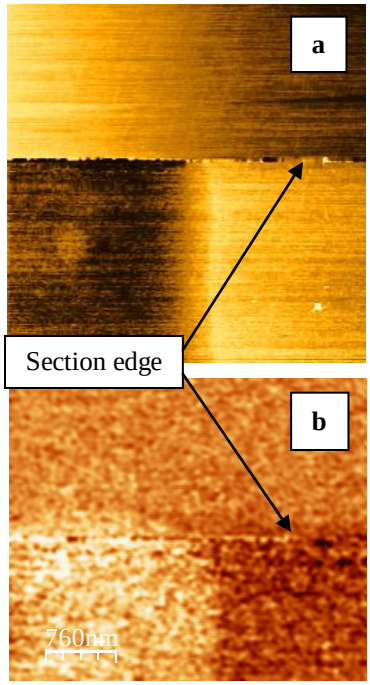

Figure 5: a) AFM image of GST crystalline region section, flattened; b) UFM image of the same region, only scale is different, the crystalline phase contrast clearly extends beyond the top surface and into the section area of the scan, offset flattened.

4 CONCLUSSIONS

The results clearly demonstrate the exceptional material sensitivity of Ultrasonic Force Microscopy in application to Phase-Change materials, in particular to GST. This proves to be rather useful especially when caring out 3D SPM characterisation, as the topographical information related to different phase formation is lost upon Ion Beam sectioning. Future work will aim at investigating sections of electrically switched bits, both amorphous and crystalline, as well as investigating phase-change/heater (electrode) interfaces in working memory cells.

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