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In-Situ TEM Observation of Crystallization in Phase-Change Material

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Amorphous-to-crystalline phase transformations have been the subject of much research, particularly in the field of materials science. Yet the mechanism to track and measure the crystallization kinetics during the phase transformation can be challenging for materials that are poor glass formers, since processes with high nucleation rates and rapid crystal growth will need with higher spatial resolution and at faster time-resolution [1] to image. Chalcogenide-based phase-change materials such Ag-In-Sb-Te alloys, used for memory devices, are known to have fast switching between the amorphous to crystalline phases that varies with temperature [1, 2]. The tracking and measurements of such phase transformation is critical for the fundamental understanding of the phase transformation and could potentially impact applications in the faster memory devices. In-situ transmission electron microscopy (TEM) heating of the sample provides a powerful platform to track such phase transformation with higher resolution. The current capability to heat TEM specimens rapidly and with control and to acquire images at faster frame rates (~100 frames per second) can enable capturing of the transformation process with higher temporal resolution.

We used a specialized TEM heating holder with a capability to insert MEMS-based chip with a 9-pin biasing and heating configurations (Figure 1). The 30 nm thick amorphous films with a nominal composition of $Ag_3In_4Sb_{74}Te_{17}$ were directly deposited on the window of the chips on top of the heater elements. The temperature in the samples was ramped up until the material with amorphous phases started to crystallize. The TEM observations were carried with a JEOL JEM-2100 LaB6 operated at 200 kV. The images were recorded using Direct Electron DE-12 camera with acquisition rates of 20-30 fps.

A series of images showing the crystallization process in the Ag-In-Sb-Te alloy are shown in Figure 2. The crystal growth rate is on the order of 1 micrometer/second. This exceeds the growth rate measurement in the same alloy achievable with more conventional microscopic methods (such as optical microscopy) by more than an order of magnitude. These measurements begin to fill the gap between what may be achieved with conventional in situ optical and TEM experiments and measurements that may be made with high time resolution photo-emission TEM techniques, such a dynamic TEM (DTEM). Using a variety of in situ imaging techniques, the crystallization kinetics may be mapped more completely as a function of temperature. This will lead to a more comprehensive understanding of the crystallization process of technologically-important marginal glass formers.

References:
[1] S. Raoux *et al*, MRS Bulletin (2012) p.118.
[2] T Matsunaga *et al*, Nature Materials **10** (2011), p. 129.

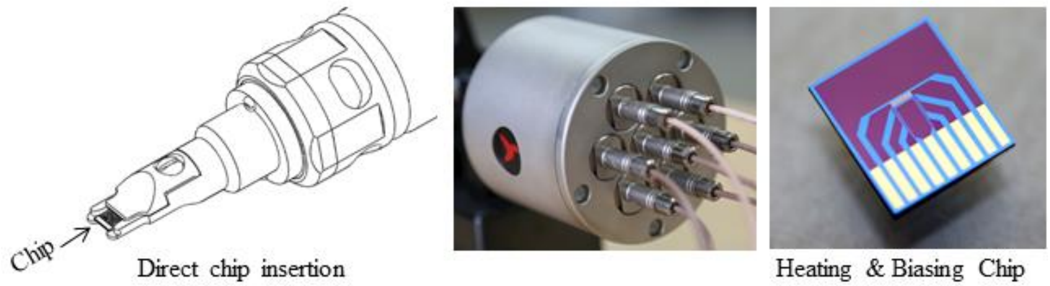

Figure 1. Electrical biasing and heating holder with a MEMS chip. The chip is directly inserted into the tip of the holder.

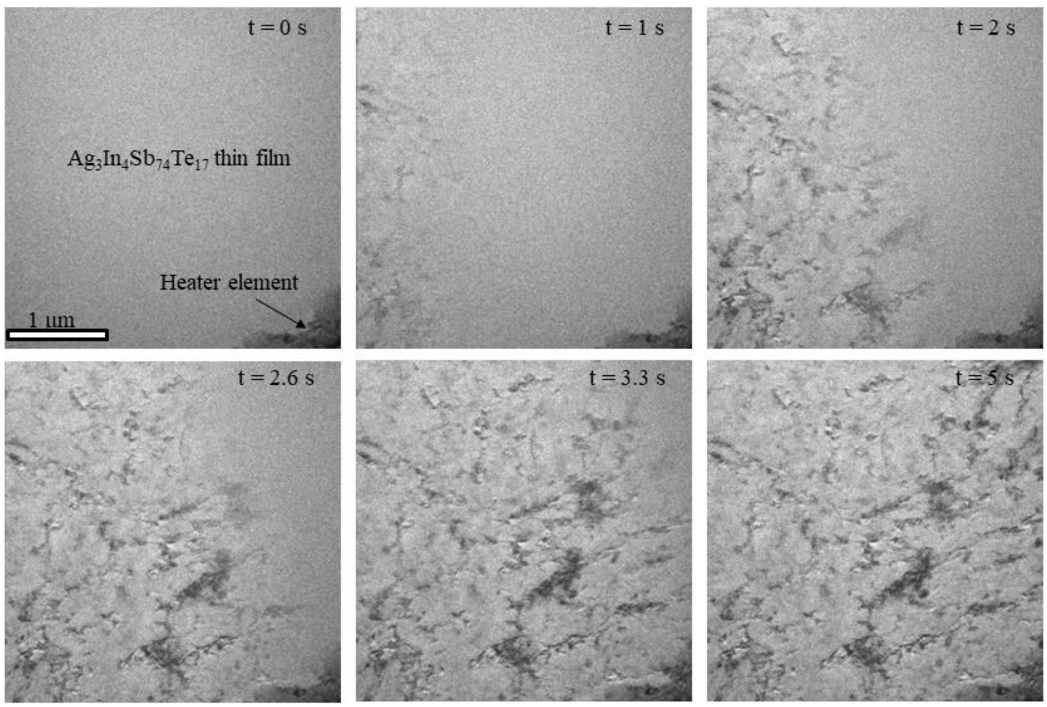

Figure 2. Tracking the evolution of amorphous → crystalline phase transformation in Ag-In-Sb-Te alloy thin film as a function of temperature. The phase transformation initiates when the temperature reaches around 90˚C.

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