Direct observation of crystallization in phase-change materials using in-situ TEM

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Chalcogenide-based phase change materials (PCMs) such as Ag-In-Sb-Te are known to show rapid transformation between the amorphous to crystalline phases with increase in the temperature. This faster rate of transition between the phases can perform as a switch, and can be used in various applications including solid-state memory devices [1, 2]. However, the tracking and understanding of this nanoscale phase transformation, which evolves rapidly with temperature, is important and yet challenging to acquire *via* traditional characterization methods [2]. 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 n/aframes per second) can enable capturing of the transformation process with higher temporal resolution. Here, we present the direct observation of nanoscale phase evolution from amorphous to crystalline phase in the thin film of Ag-In-Sb-Te using in-situ TEM microfabricated heating devices and a controller.

We used a specialized TEM heating holder with a capability to insert microfabricated-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.

The evolution of the crystalline phase from the amorphous thin film of Ag-In-Sb-Te alloy as a function of temperature is presented in Figure 2. The sequence of images shows that the rate of crystal growth 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 variety of in-situ imaging techniques, the crystallization kinetics may be fully mapped, and may potentially be applied in 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.

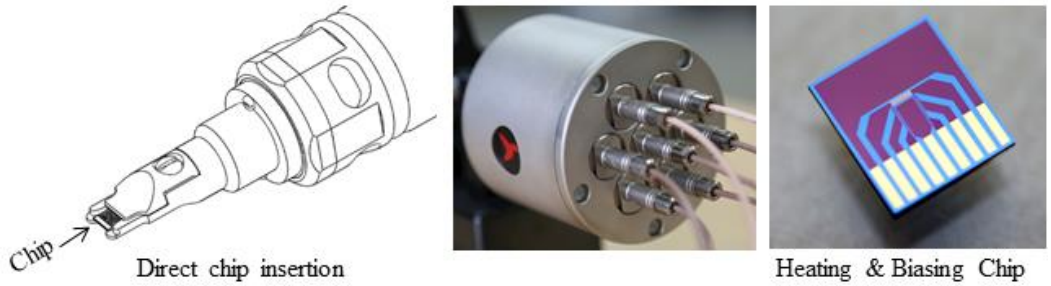

Figure 1. Electrical biasing and heating holder with a microfabricated chip. The chip is directly inserted into the tip of the holder.

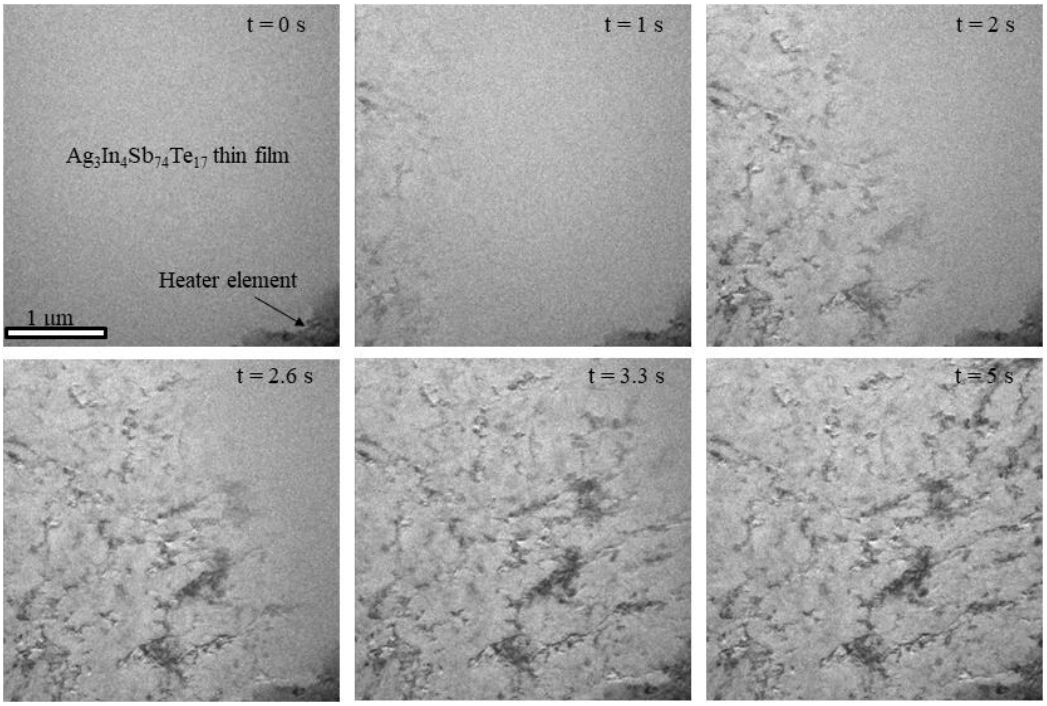

Figure 2. Tracking the evolution of amorphous → crystalline phase transformation in Ag-In-Sb-Te alloy thin film as a function of temperature. The phase transformation initiates when the temperature reaches around 90°C.