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IT-7-P-1655 Characterization of Melting and Crystallization Behavior in the Au-Ge Eutectic System Using Au-catalyzed Ge Nanowires

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The catalyzed growth of nanowires (NWs) can provide us with a useful platform for studying nanoscale phase transformations that are readily observed using in situ transmission electron microscopy. For example, following vapor-liquid-solid (VLS) growth of Ge and Si NWs, the re-solidified catalyst, typically Au, remains at the end of the NW, with an abrupt, planar interface between the two materials. Fundamental behaviors of these nanoscale eutectic systems, such as melting and crystallization, as well as metastable phase formation, can be studied by heating and cooling the NWs in the TEM. The use of a MEMS based heating holder (Protochips Aduro™) allows for a large range of heating and cooling rates, including quench rates that are comparable to those used in more traditional rapid quench studies. Here we present details of the melting and crystallization behavior of the metastable hexagonal close-packed beta phase of the Au-Ge eutectic system.

We have previously shown that the metastable hcp phase, which occurs following NW growth [1], can also be formed by melting and rapid quenching of the Au nanocatalyst at the tip of a Ge nanowire [2]. Fig. 1 shows the melting behavior of the quenched-in metastable phase. Melting occurs over a timeframe of seconds; it begins at the edges of the Ge NW-catalyst interface (Fig. 1a and b). In Fig. 1b melting is also visible at the top of the catalyst indicating that initial melting continues along the surface. An abrupt formation of additional stacking faults (Fig. 1c), characterizes a transition to a large volume of melt regions that form parallel to the {0001} planes of the remaining crystal (Fig. 1d). As the melt volume grows, the crystal pulls away from the surface, adopting a spherical shape, and "floats" in the liquid, then moves to the interface (Fig. 1e), before abruptly dissolving into the liquid volume (Fig. 1f). This last process is accompanied by a notable darkening of the liquid as a result of mass contrast induced by the dissolved Au. We note that this same sequence of events has been observed a number of times in the hcp quenched structure, e.g. Fig 2. These results suggest that orientation of the crystal, and diffusion along the {0001} planes of the metastable hcp phase influence details of the melting process. We will also present results of cooling studies, which indicate a correlation between the formation and orientation of the metastable phase, and show that the amount of Ge that remains in the hcp structure can be controlled by the cooling rate and minimized by subsequent annealing.

[1] A.F. Marshall, et al, Nano Lett. 10 (2010), 3302. [2] A.F. Marshall, et al, Microscopy and Microanalysis 2013, Phoenix, AZ.

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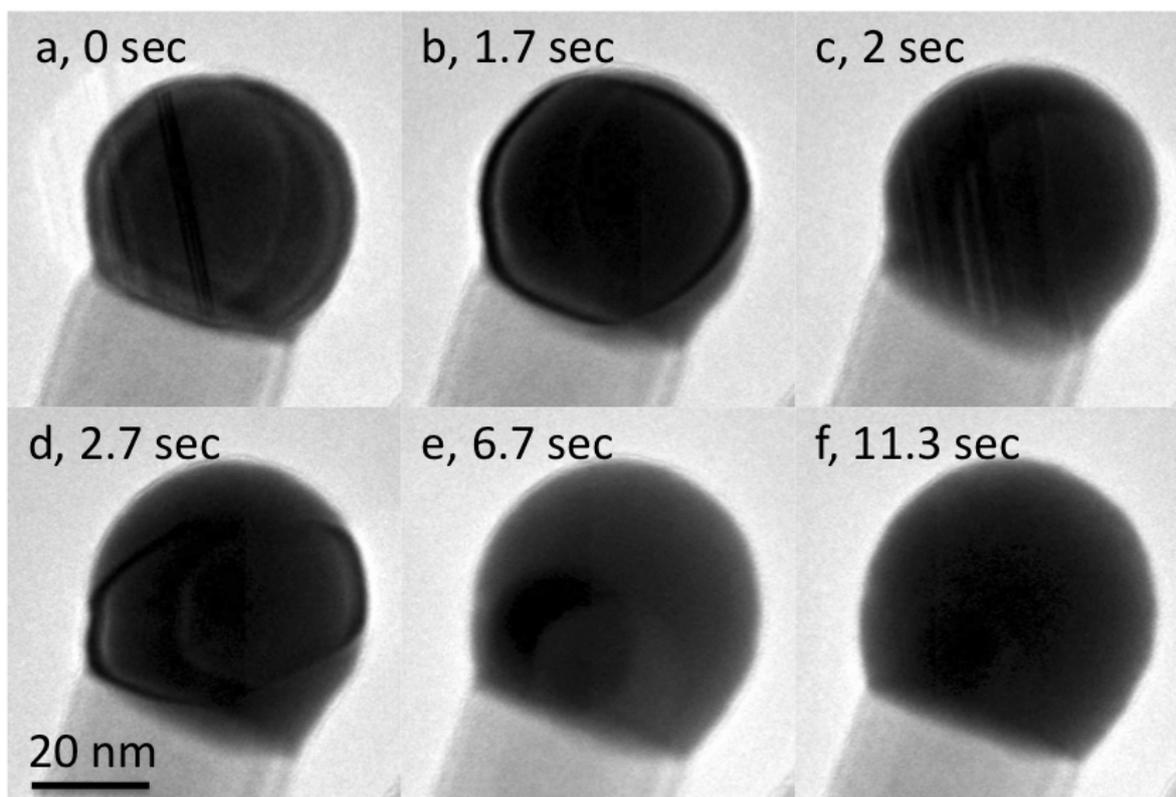


Fig. 1: Fig. 1: Selected timeframes from a melting video of a quenched nanocatalyst with the metastable phase. Melting begins at the edges of the interface in the first frame and proceeds through a series of morphological changes until final melting at about 11 seconds.

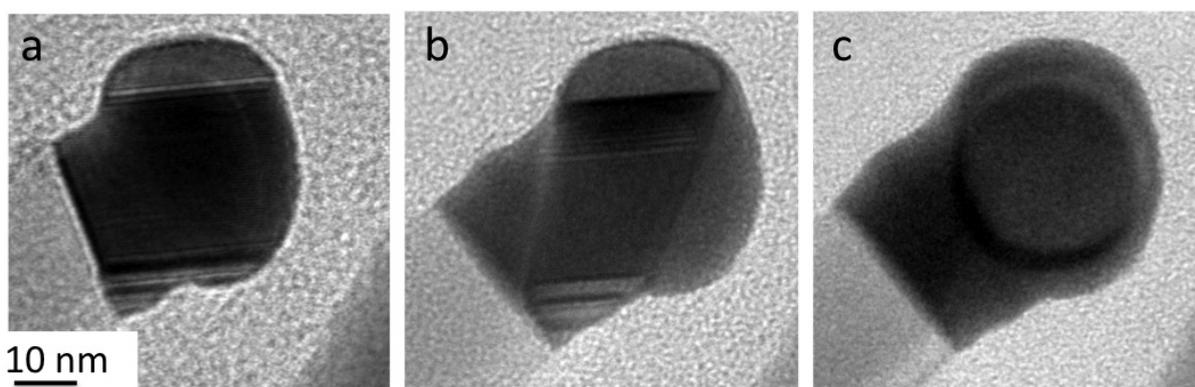


Fig. 2: Fig. 2: Still images from another quenched in metastable structure (a) shows the same preferred melting along the $\langle 0001 \rangle$ direction (b), and a detached crystal within the melt (c) prior to final melting.