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In-situ TEM irradiation induced amorphization of $Ge_2Sb_2Te_5$

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$Ge_2Sb_2Te_5$ (GST) is a material that changes phase from amorphous to crystalline at 120-140 °C [1]. The phase transition occurs rapidly and results in a large change in optical properties, which makes this material well suited for optical disk memory applications [2]. *In situ* transmission electron microscopy (TEM) is useful for gaining insight at the nanoscale into the mechanism responsible for the change in microstructure and resulting properties of materials. The *in situ* ion irradiation transmission electron microscope at Sandia National Laboratories is uniquely suited to observe irradiation induced phase change in real time [3, 4].

In this work several thin films of GST with varying carbon content are prepared via magnetron sputtering. Amorphous films 90 nm-thick are deposited on top of 40 nm-thick SiN windows for TEM analysis. They are annealed *in situ* at a rate of 10 °C/min until crystallization then held at 200 °C (450 °C for 12% C samples) for 5 minutes. Specimens are then irradiated *in situ* with a 2.8 MeV Au^{4+} beam with beam flux 1×10^{11}-1×10^{12} ions/$cm^2\cdot s$ and current density of $7\times10^{-8} – 6\times10^{-7}$ A/cm^2. Ions impact the film at a 30° angle and changes in either the microstructure or diffraction pattern are observed simultaneously during the irradiation. The electron beam is at a 200 keV accelerating voltage and diffraction patterns are collected using parallel beam selected area electron diffraction. Displacements per atom (dpa) damage thresholds are calculated with stopping range of ions in matter (SRIM) models where film density is estimated using the rule of mixtures between GST (6.35 g/cm^3) and graphite (2.65 g/cm^3) [5]. As seen in previous studies, we show a condensed electron beam results in crystallization producing small grains [6], which is most apparent in low carbon specimens. All films crystallized into an FCC lattice with no notable change in lattice parameter, and grain size decreased with increasing carbon content in the films. The films are analyzed before and after vacuum anneal using energy dispersive x-ray spectroscopy (EDS) and no change in relative concentration of the elements are observed. Increasing carbon content resulted in increased crystallization temperatures, but reduced irradiation amorphization thresholds. This phenomenon is contradictory to the trends seen in bulk scale experiments.

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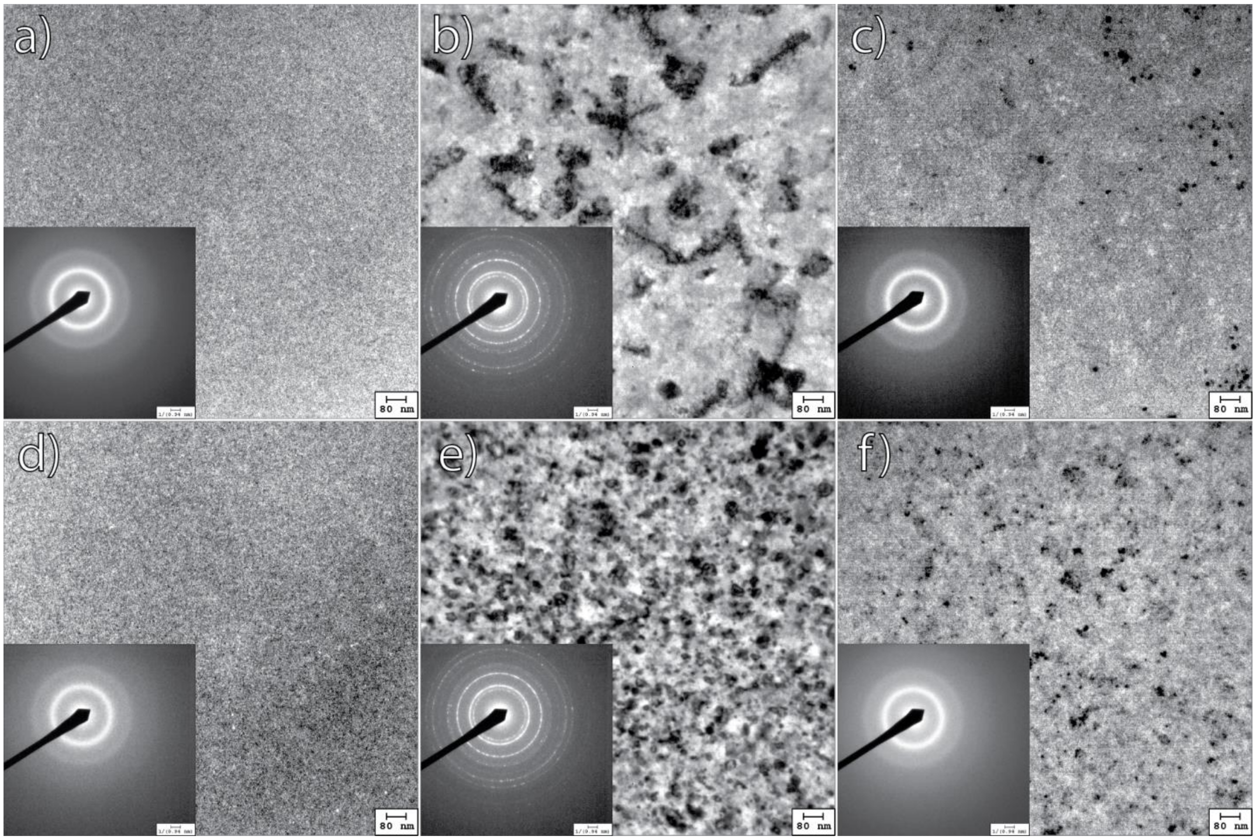

Figure 1. Representative microstructures and diffraction patterns (inset) of GST-C films 0% C (a-c) 2% C (d-f) as deposited (a, d), after anneal (b, e), and after irradiation (c, f).

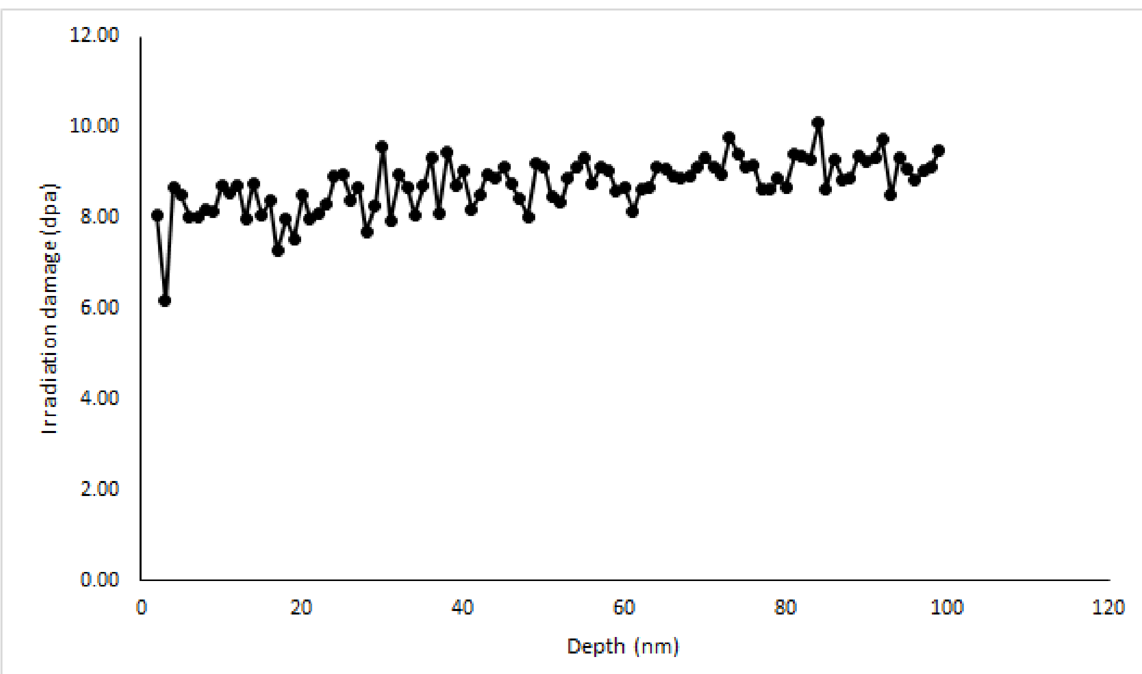

Figure 2. SRIM calculation of the damage profile from 2.8 MeV Au ions at a fluence of 9x1014 ions/cm² through a Ge2Sb2Te5 thin film showing uniform damage profile across the film at 30°

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References

[1] S. A. Kozyukhin *et al.*, "Direct observation of amorphous to crystalline phase transitions in Ge–Sb–Te thin films by grazing incidence X-ray diffraction method," *Journal of Materials Science: Materials in Electronics*, vol. 31, no. 13, pp. 10196-10206, 2020/07/01 2020, doi: 10.1007/s10854-020-03565-7.

[2] N. Yamada, E. Ohno, K. Nishiuchi, N. Akahira, and M. Takao, "Rapid-phase transitions of GeTe-Sb2Te3 pseudobinary amorphous thin films for an optical disk memory," *Journal of Applied Physics*, vol. 69, no. 5, pp. 2849-2856, 1991.

[3] J. A. Aguiar *et al.*, "In-situ Ion Irradiation and Recrystallization in Highly Structured Materials," *Microscopy and Microanalysis*, vol. 25, no. S2, pp. 1572-1573, 2019.

[4] K. Hattar, D. C. Bufford, and D. L. Buller, "Concurrent in situ ion irradiation transmission electron microscope," *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms,* vol. 338, pp. 56-65, 2014.

[5] R. E. Stoller, M. B. Toloczko, G. S. Was, A. G. Certain, S. Dwaraknath, and F. A. Garner, "On the use of SRIM for computing radiation damage exposure," *Nuclear Instruments and Methods in Physics Research B,* vol. 310, pp. 75-80, 2013, doi: http://dx.doi.org/10.1016/j.nimb.2013.05.008.

[6] T.-T. Jiang *et al.*, "Progressive amorphization of GeSbTe phase-change material under electron beam irradiation," *APL materials,* vol. 7, no. 8, p. 081121, 2019.

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