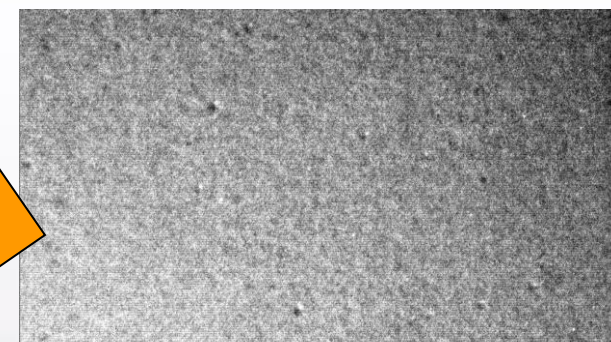
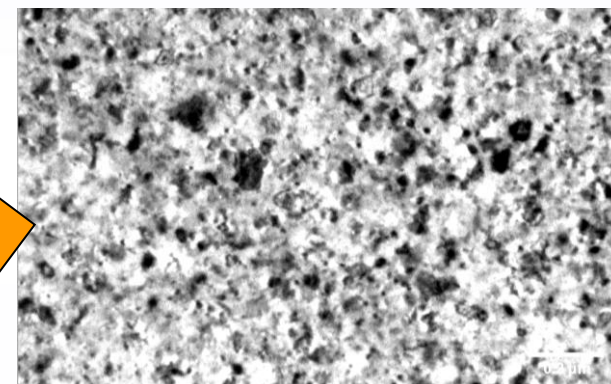
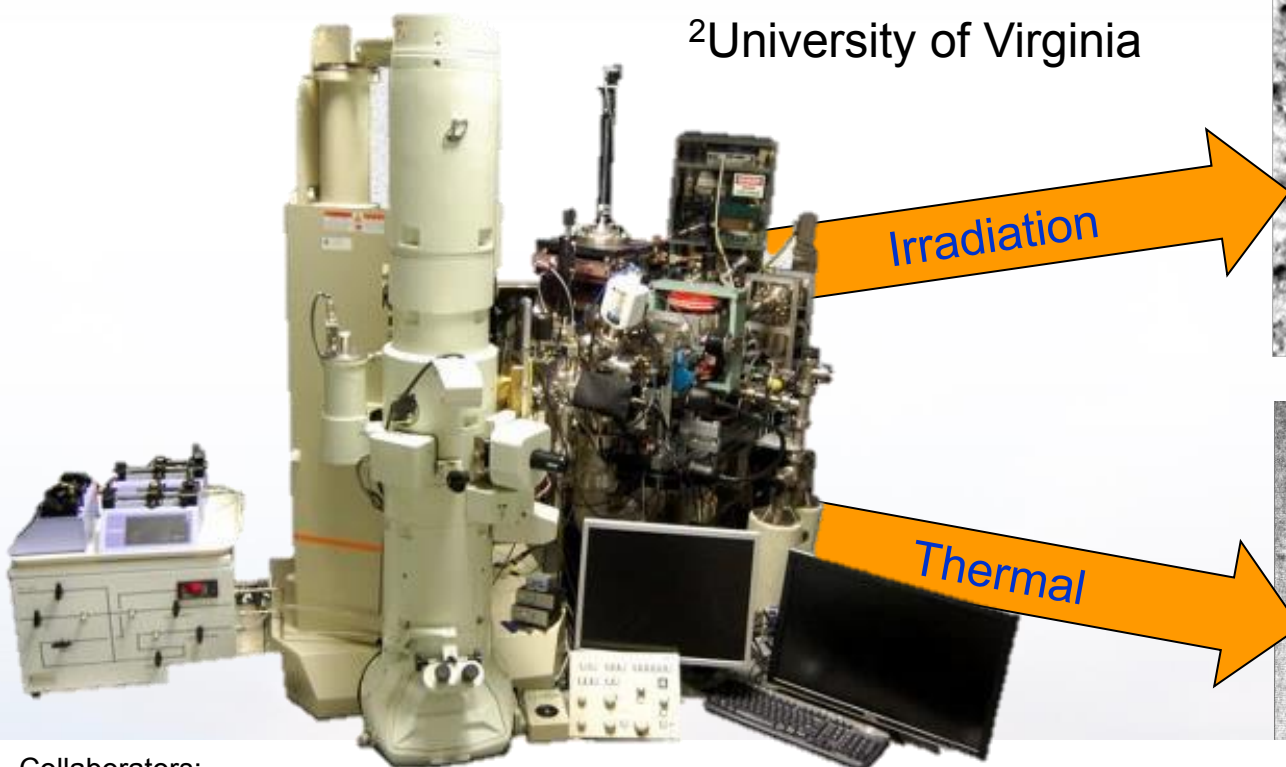




In-situ TEM Irradiation Induced Amorphization of $\text{Ge}_2\text{Sb}_2\text{Te}_5$

Trevor Clark¹, Ethan Scott^{1,2}, David Adams¹, Khalid Hattar¹¹Sandia National Laboratories

²University of Virginia



Collaborators:

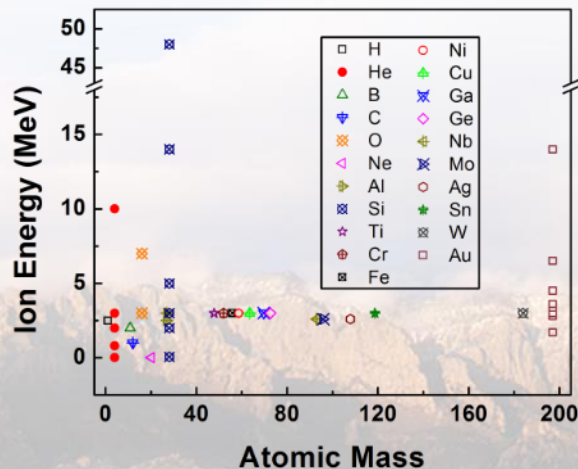
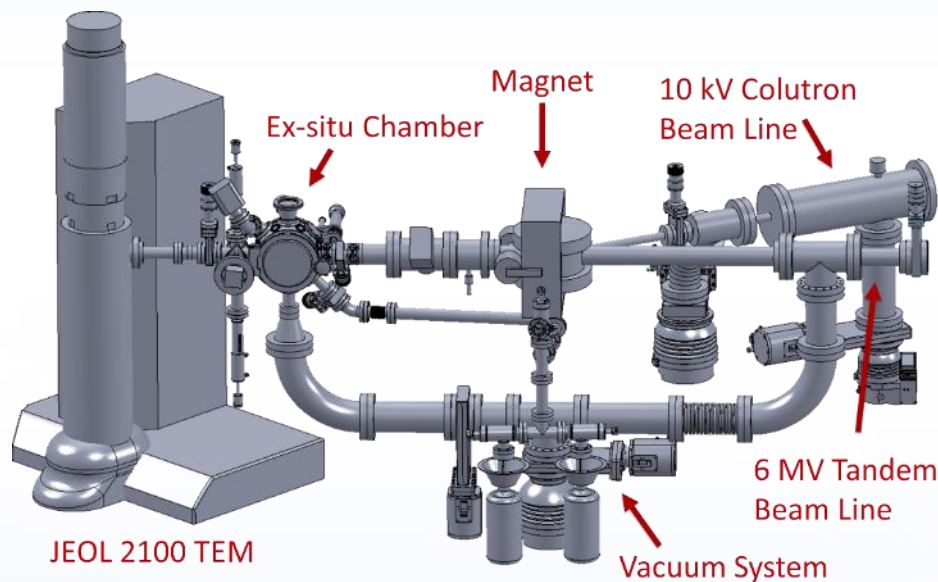
- D.L. Buller, D.C. Bufford, S.H. Pratt, T.J. Boyle, B.A. Hernandez-Sanchez, S.J. Blair, B. Muntifering, C. Chisholm, P. Hosemann, A. Minor, J. A. Hinks, F. Hibberd, A. Ilinov, D. C. Bufford, F. Djurabekova, G. Greaves, A. Kuronen, S. E. Donnelly, K. Nordlund, F. Abdeljawad, S.M. Foiles, J. Qu, C. Taylor, J. Sugar, P. Price, C.M. Barr, D. Adams, M. Abere, L. Treadwell, A. Cook, A. Monterrosa, IDES Inc, J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, W. Mook, Hysitron Inc., G.S. Jawaharram, S. Dillon, R.S. Averbach, N. Heckman, J. Carroll, S. Briggs, E. Carnes, J. Brinker, D. Sassaki, T. Nenoff, B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan, K. Jungjohann, & Protochips, Inc.

This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. This work was performed in part at the Center for Integrated Nanotechnologies, an Office of Science User Facility, operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

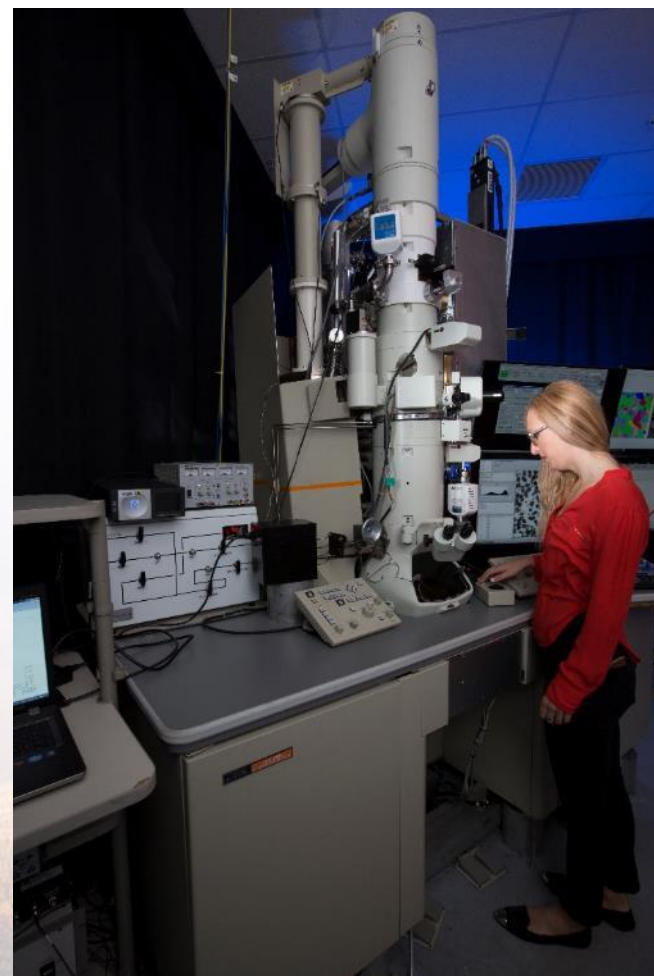
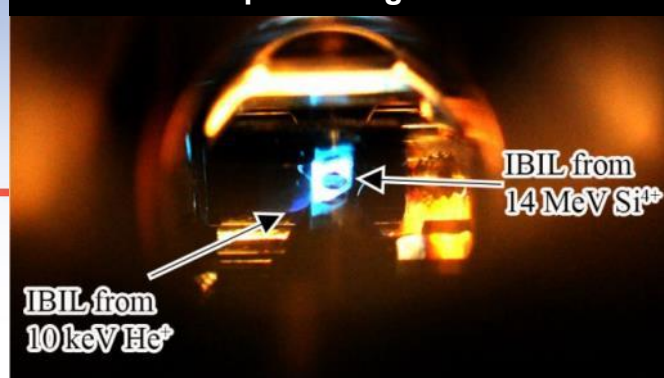
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



IBIL from a quartz stage inside the TEM



Sample Preparation and Experimental Conditions

■ 90 nm-thick GST with 0 % C - 20 % C

■ Plan View:

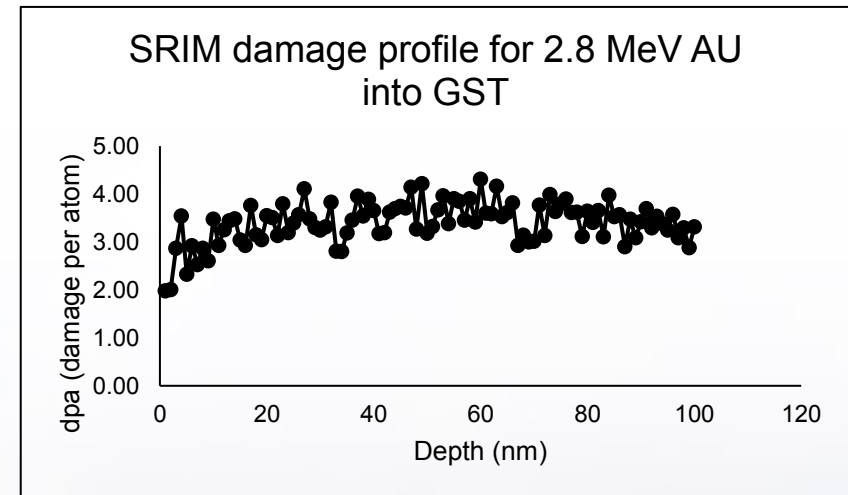
- ♦ Anneal (5 °C/min)
- ♦ RT irradiation (4dpa)
 - Anneal (5 °C/min)
- ♦ 100 °C hold & irradiation (4dpa)

■ Cross section FIB lift-outs:

- RT Irradiation
 - ♦ 200 °C & 300 °C Hold

■ Irradiation Conditions

- 2.8 MeV Au⁴⁺
- Up to 4 dpa

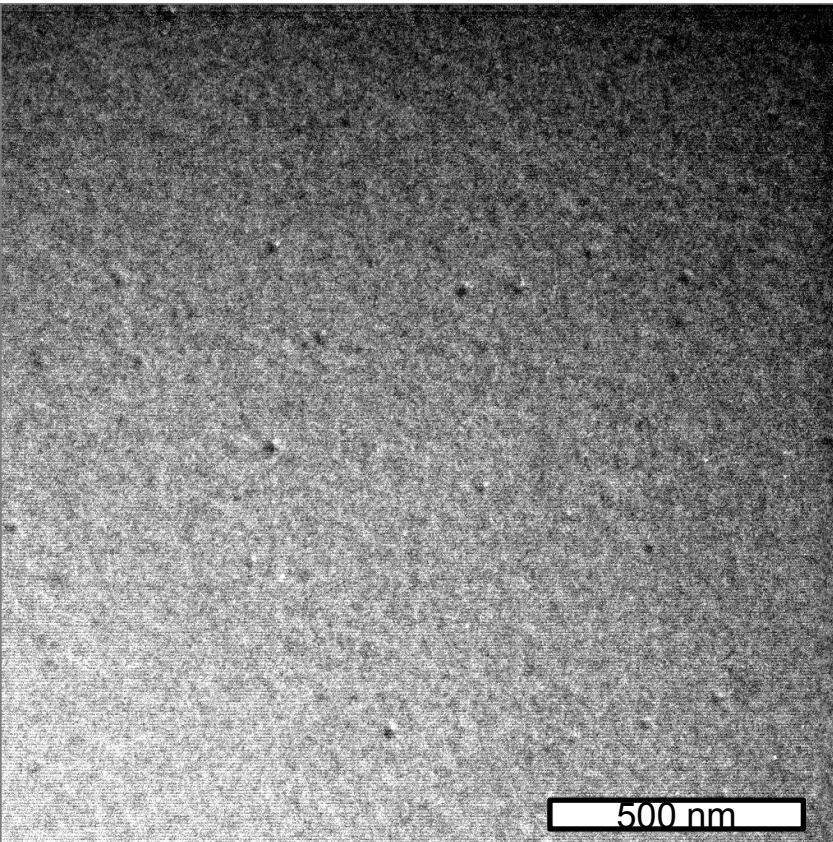


■ Data Analysis

- SRIM simulation data is used to calculate damage level (DPA) from this fluence
- Manual rain size analysis done

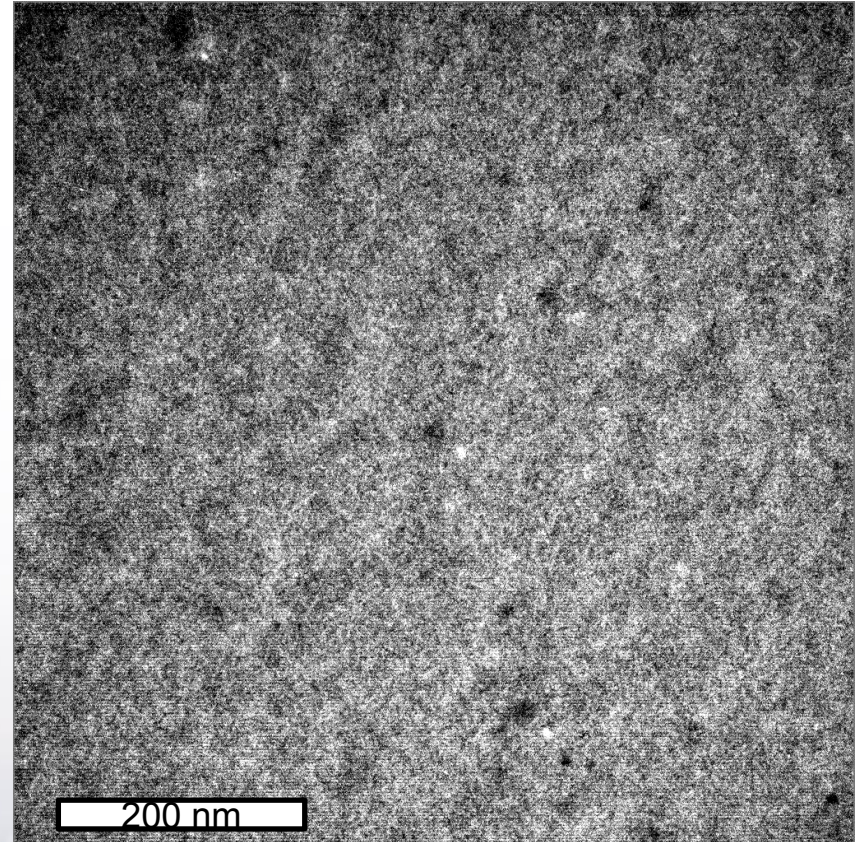
In-situ Annealing of Pristine and Irradiated GST

No irradiation



GST 0 %C
Anneal 5°C/min
100-150 °C
15x speed

4 dpa

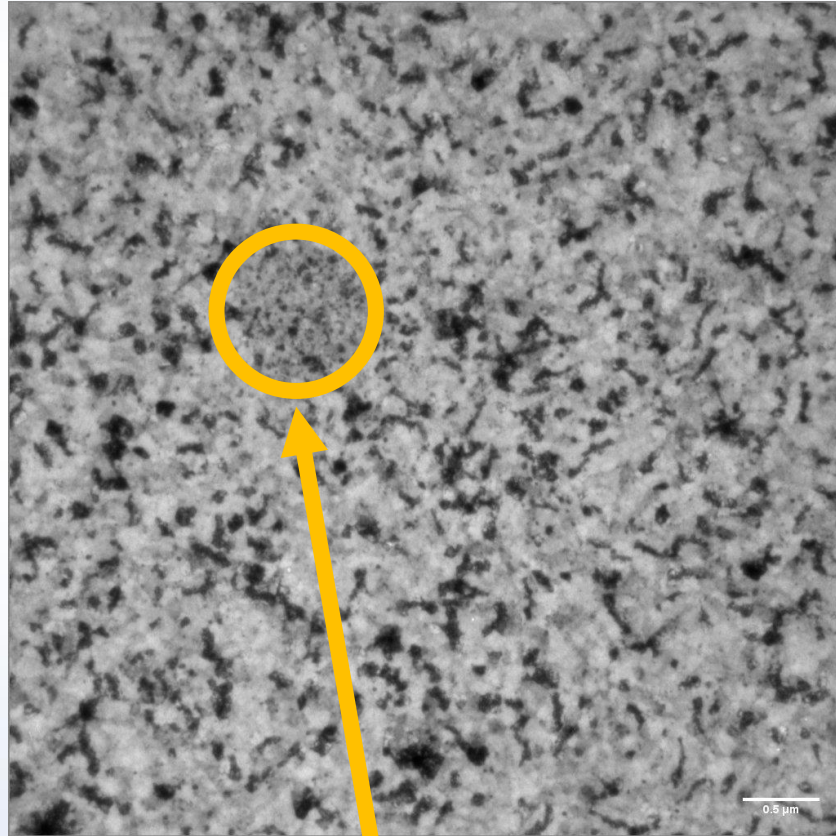


**Crystallization temperature doesn't seem
to change with ion irradiation**



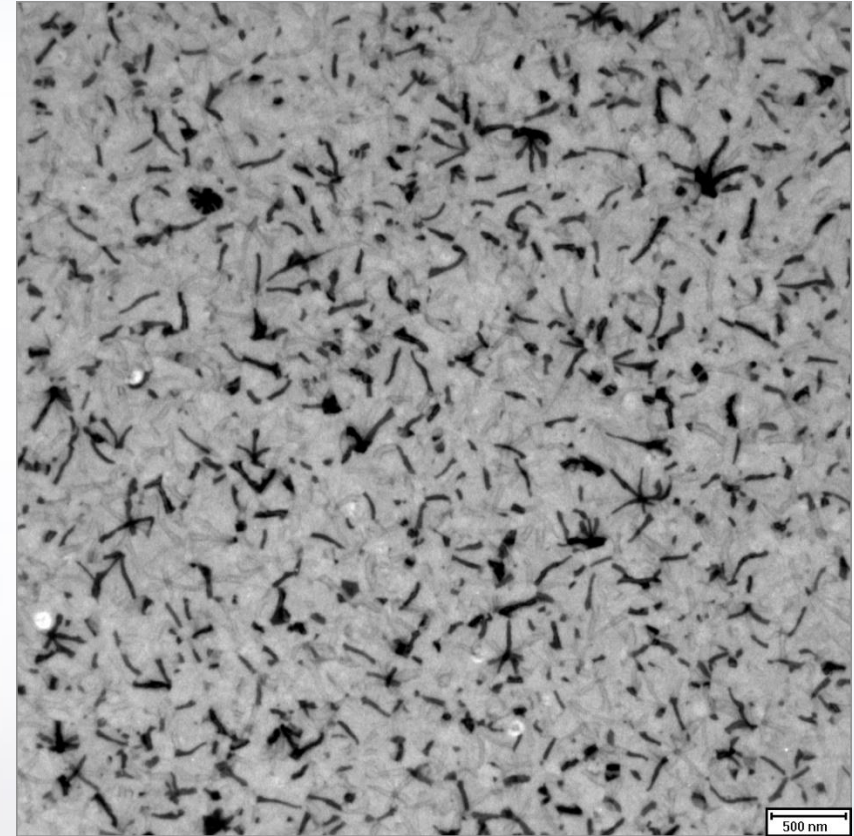
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Electron Beam Induced Crystallization



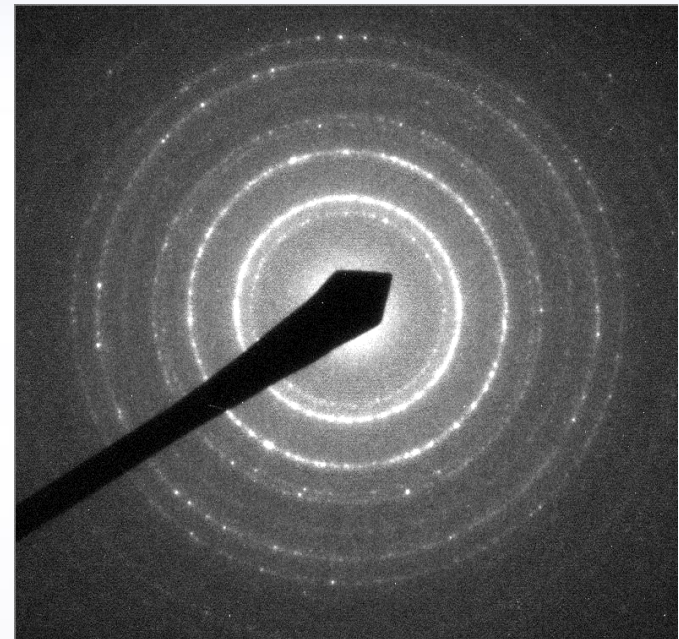
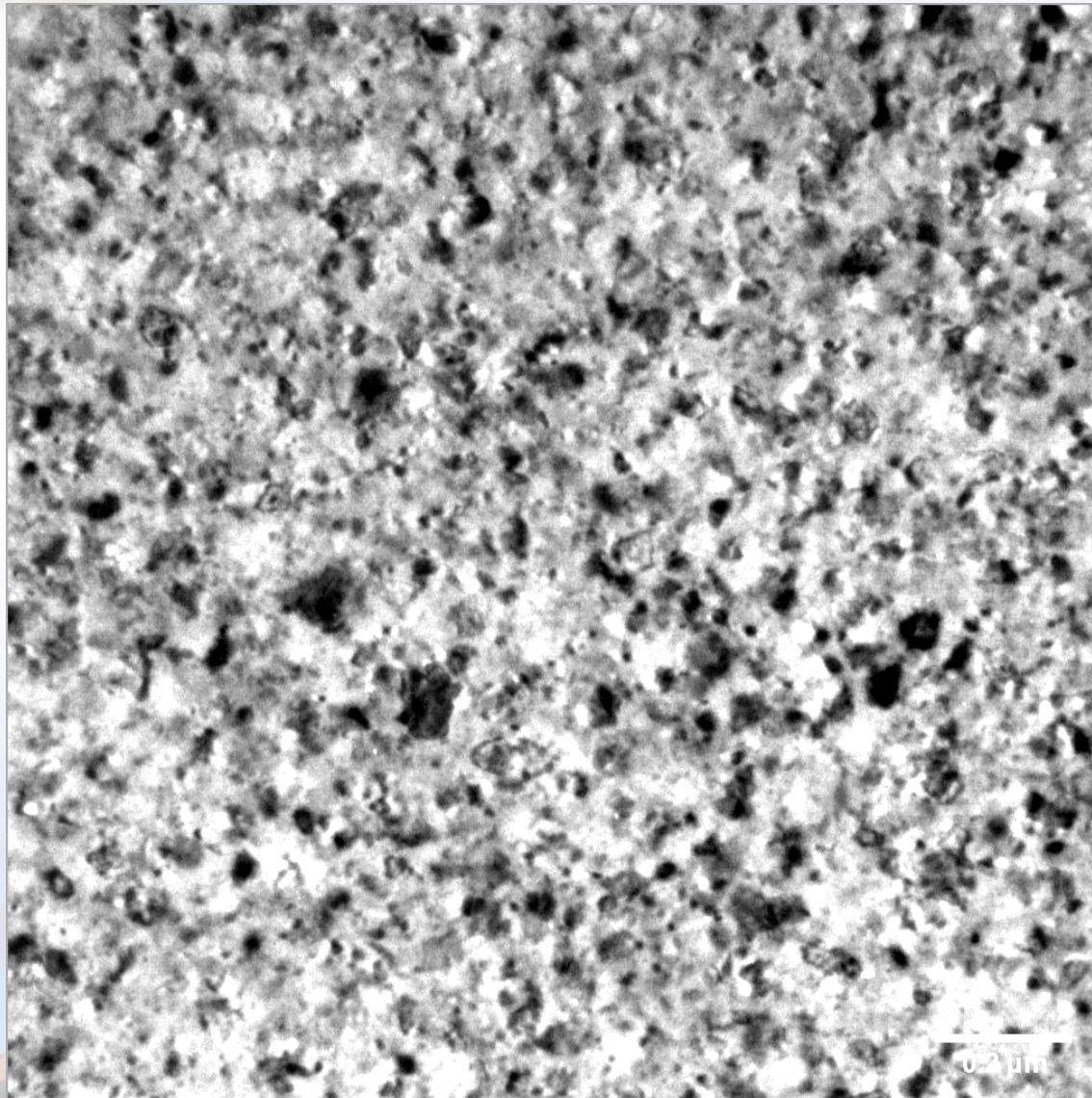
Electron beam damage

**Atypical crystallization nucleated by
condensed electron beam at RT
Nanoscale grains ~20 nm.**



Typical crystallization. Highly strained film with large grains ~500 nm. Contrast bands are bend contours.

GST 4 % C irradiated to dose of $1.5E14$ ions/cm²



Displacement damage not visible by post-mortem investigation can be clearly seen during in-situ TEM ion irradiation

Sped up 24x



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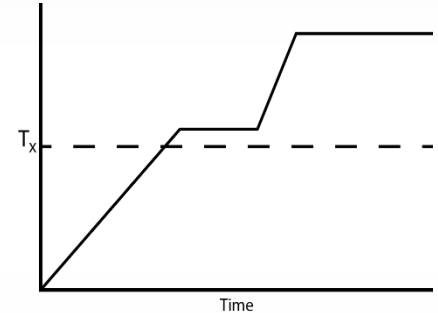
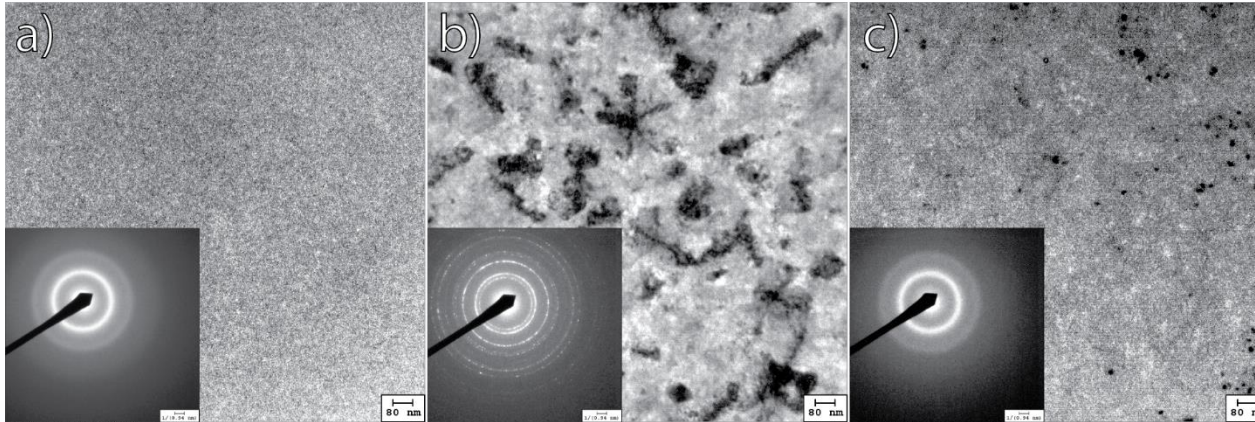
Representative Results

As-deposited

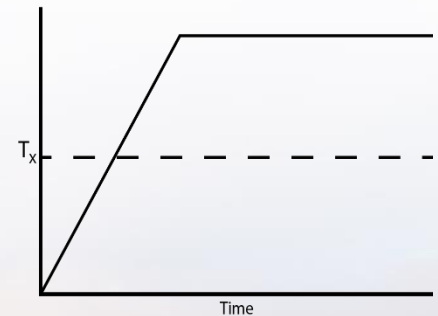
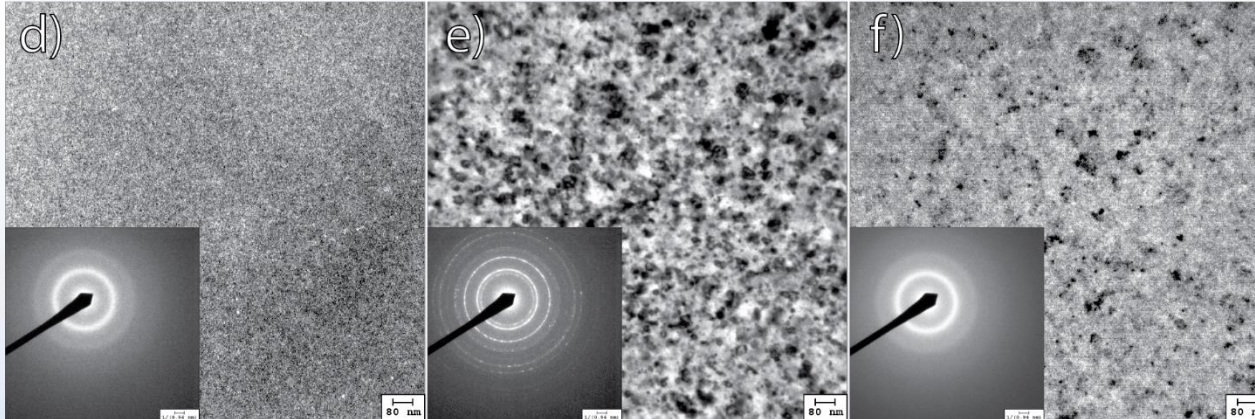
After Anneal

After Irradiation

0% C



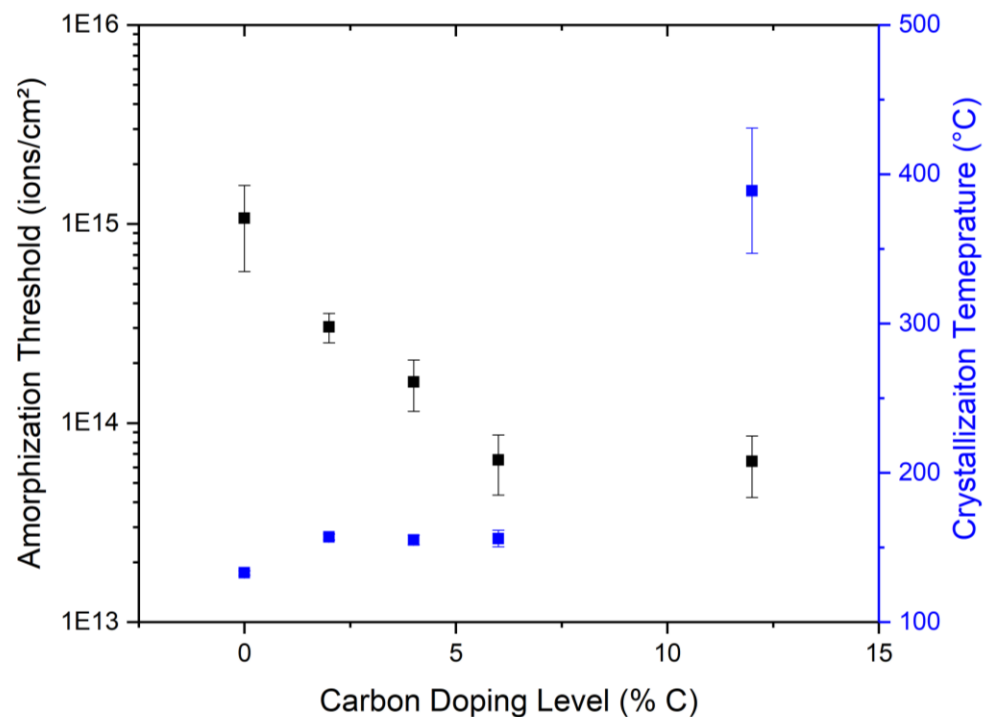
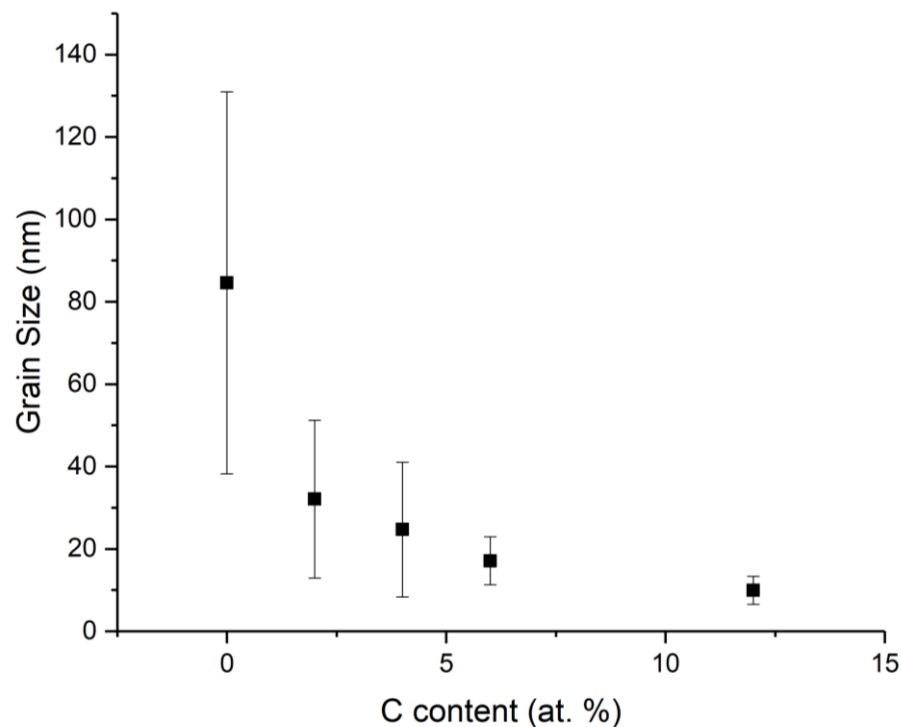
2% C



When annealing, holds close to crystallization temperature before high temperature anneal results in larger grains (b vs e)

Defects remain in the film after irradiation but produce no change to SAD pattern

Grain Size and Phase Evolution as a Function of Carbon Concentration



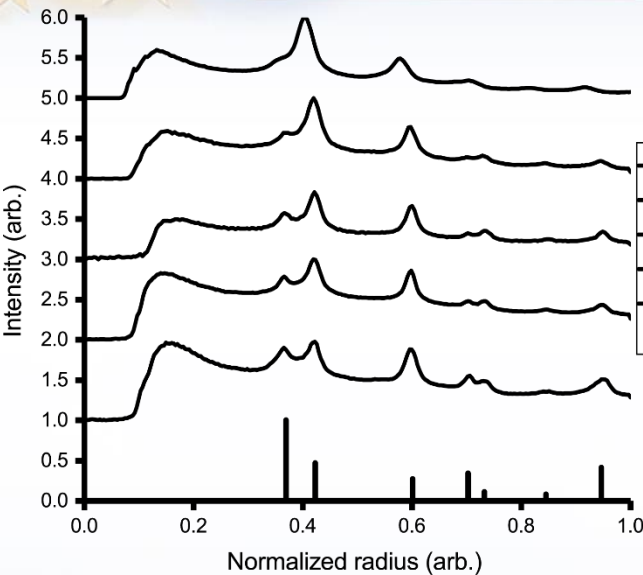
Grain size in the annealed state decreases with increasing C content

Amorphization and crystallization temperature are carbon dependent



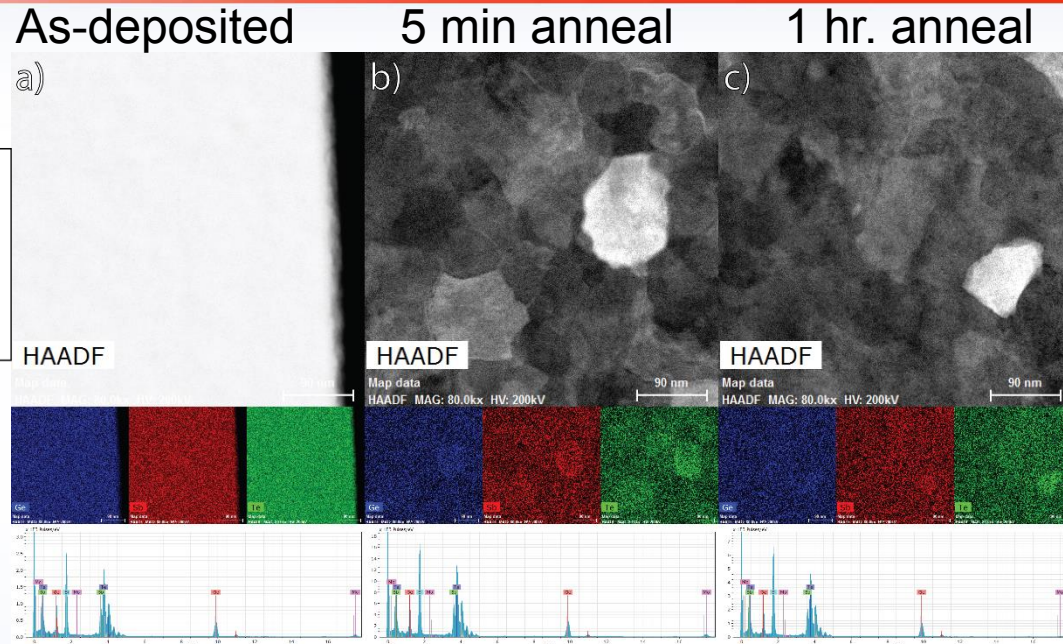
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Phase and Compositional Analysis



- Calculated by integrated radial intensity of SAED patterns.
- Peak indices: 111, 200, 220, 311, 222, 400, 420 (legend is nominal C content)

- **Single phase FCC crystals**
- **Slight decrease in lattice constant with increasing C content**



200 °C vacuum anneal at heating rate ~ 20°C/min

- Top: HAADF image
- Middle: EDS elemental map Ge (blue), Sb (red), Te (green)
- Bottom EDS spectra

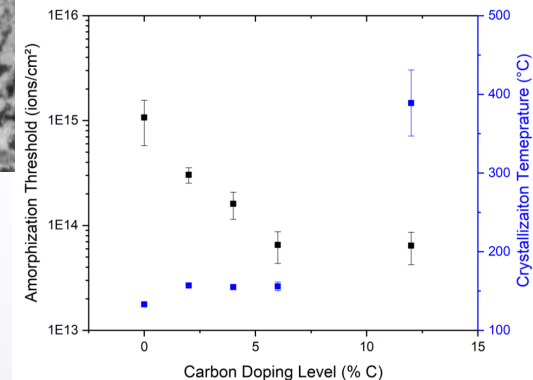
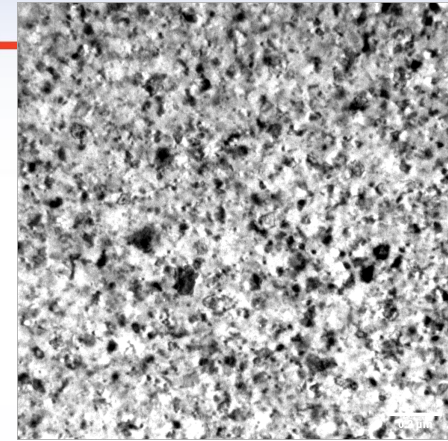
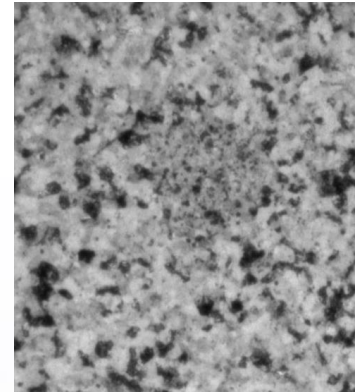
- **X-ray count ratios of Te/Ge and Sb/Ge did not change after the annealing.**
- **Indicating negligible loss of Te.**



Summary

- Amorphization damage threshold decreases with increasing C doping level
- Crystallization temperature increases with increasing C doping level
- Grain size in the annealed condition increases with increasing C doping level
- Potential beam heating of sample ($7e-8 - 6e-7$ A/cm² current density). COMSOL calculations can be used to estimate temperatures
- Increasing C may stabilize the amorphous phase

The stability of GST can be extensively explored via *in situ* TEM irradiation and heating



Collaborators:

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Sandia's User and Position Opportunities



D. Hanson, W. Martin, M. Wasiolek

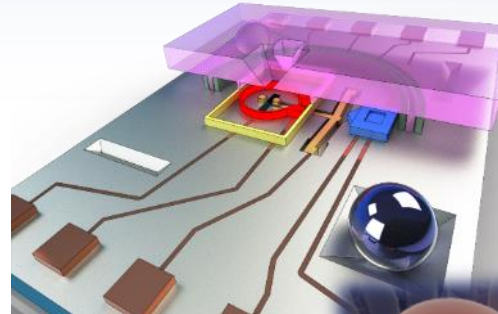
www.cint.lanl.gov

- Spring and Fall proposals for 18 months
- Rapid Access proposal anytime for 3 months

Core Facility - SNL

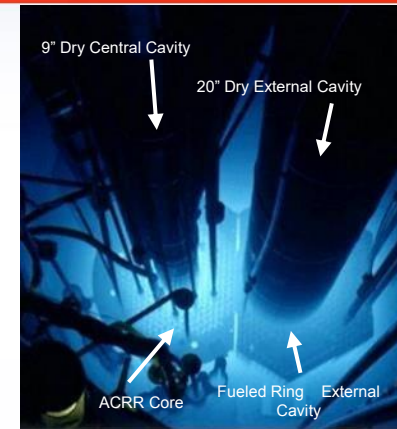


Gateway Facility - LANL



www.nsunf.inl.gov

- Three proposal a year for 9 months



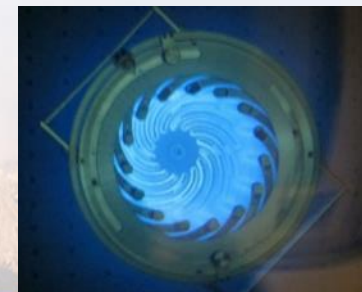
Position Oppurtunities at:

<https://www.sandia.gov/careers/>

Post-doc = 671121

Grad Student = 670865

Undergrad Student = 670864



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