BNL-98269-2012-CP

Post-growth annealing to improve the performance of CdZnTe detectors

Ge Yang

Presented at the SNM Movement Detection/Radiation Sensors and Advanced Materials Portfolio Review RadSensing 2012 Conference

Albuquerque, New Mexico
June 5 – 7, 2012

Nonproliferation and National Security Department

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SNM Movement Detection / Radiation Sensors and Advanced Materials Portfolio Review

RadSensing2012

Post-growth annealing to improve the performance of CdZnTe detectors

Ge Yang

Brookhaven National Laboratory

June 5, 2012

Project Overview

- **Project title:** *Post-growth annealing to improve the performance of CdZnTe detectors*

- **Participating Laboratory:** *Brookhaven National Laboratory*

- **Co-Principal Investigators:** *Ge Yang and Ralph James*

- **Supporting BNL Investigators:** *Aleksey Bolotnikov, KiHyun Kim, Giuseppe Camarda, Anwar Hossain and Yonggang Cui*

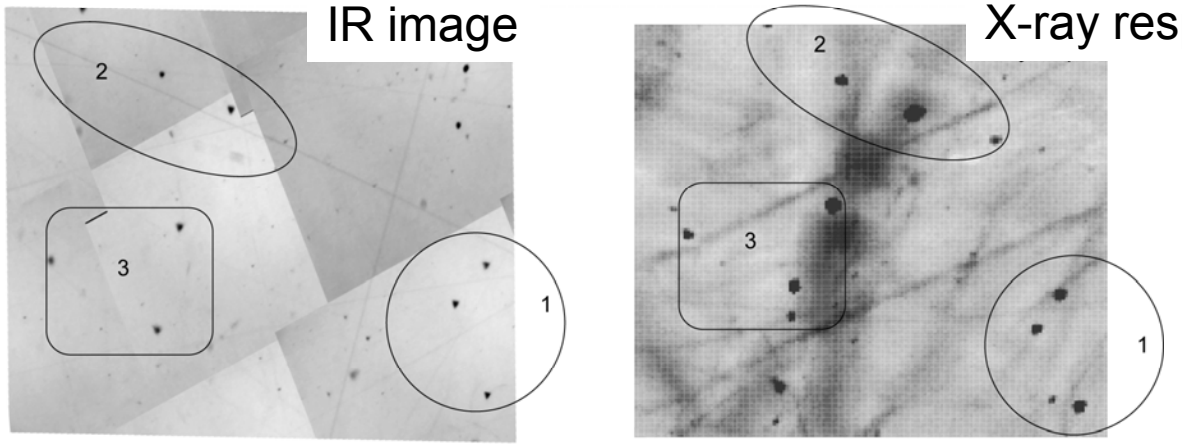
- **External Participants:** *Endicott Interconnects, Redlen Technologies (Canada), FLIR Radiation, Fisk University, Alabama A&M University, Charles University (Czech Republic), Chernivtsi University (Ukraine), IMEM (Italy) and Acrorad (Japan)*

- **Budget:** *FY10: $450K, FY11: $470K, FY12: $470K*

Project Background

Material quality is inextricably linked to supply, performance and cost of large-volume CdZnTe (CZT) gamma-ray detectors

- Tellurium inclusions and other extended defects (e.g., dislocations) are found in typical CZT crystals provided by commercial vendors and research institutions
- These defects are less important in thin detectors, but their role is critical for thick devices



The area is ~ 1.2 x 1.0 mm^2

Goal and Capability Improvement

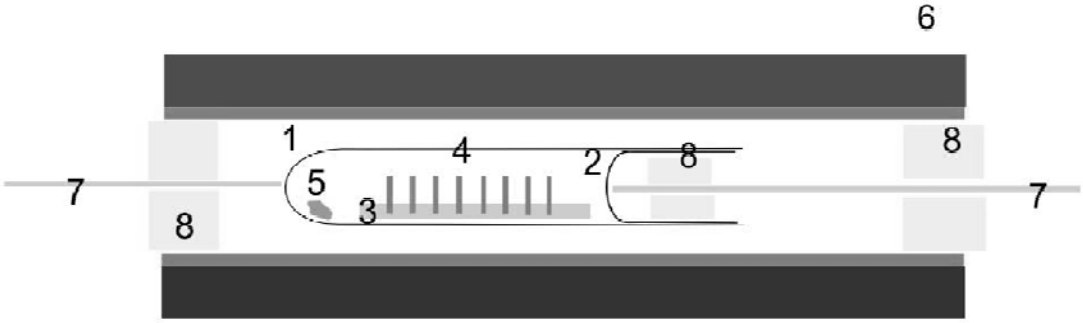
Goal: **Determine suitable thermal annealing conditions to increase the uniformity of charge-transport for large-volume detector-grade CZT crystals and devices**

Capability Improvement: **The realization of the project is critical for the mass production of inexpensive, high efficiency, high energy-resolution radiation detectors**

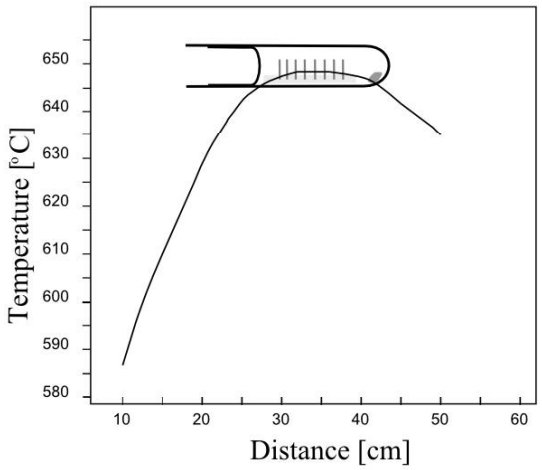
When uniform inclusion-free CZT material with good electrical properties is produced routinely, then a new class of efficient, high-resolution gamma detectors useful for spectroscopy and imaging will become available to a wide variety of nonproliferation users

Technical Approach

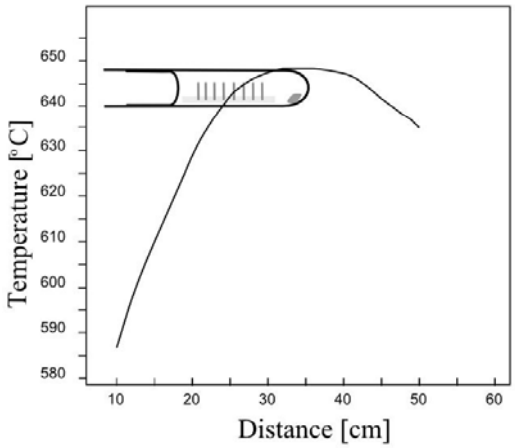
Basic Design of Annealing Experiments



1 – quartz ampoule
2 – quartz plug
3 – quartz sample holder
4 – samples
5 – annealing source
6 – furnace
7 – thermocouple
8 – thermal insulation



Uniform temperature field



Temperature gradient field

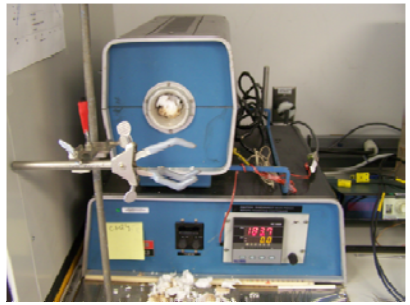
Annealing Facilities at BNL

We created a new laboratory devoted to annealing studies

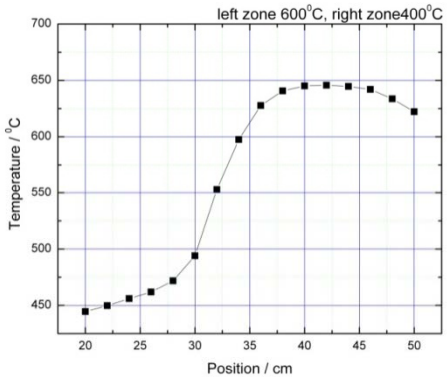
(Two-zone furnace for annealing of temperature-gradient & annealing with separate control of component pressure, one-zone furnace for uniform temperature annealing)

Two-zone annealing furnace

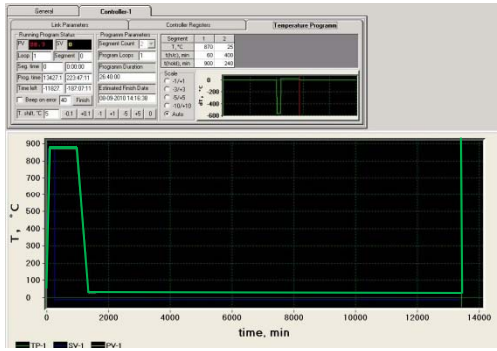
Single-zone annealing furnace # 1



Single-zone annealing furnace # 2



Temperature profile of the annealing furnace (two-zone)

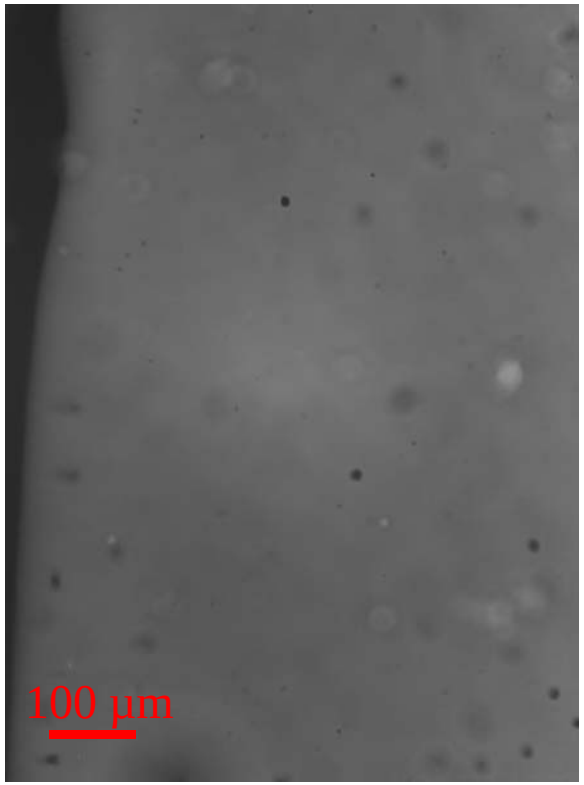


Customized computer-controlling interface

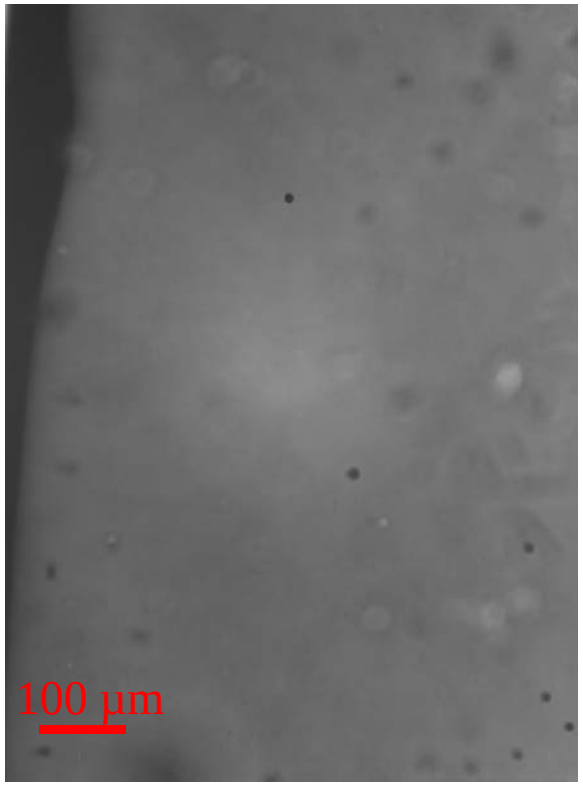
Sealed quartz tubes for annealing experiments

Results and Findings

Cd Over-Pressure Annealing at 500 °C (Uniform Temperature Field)



Before annealing

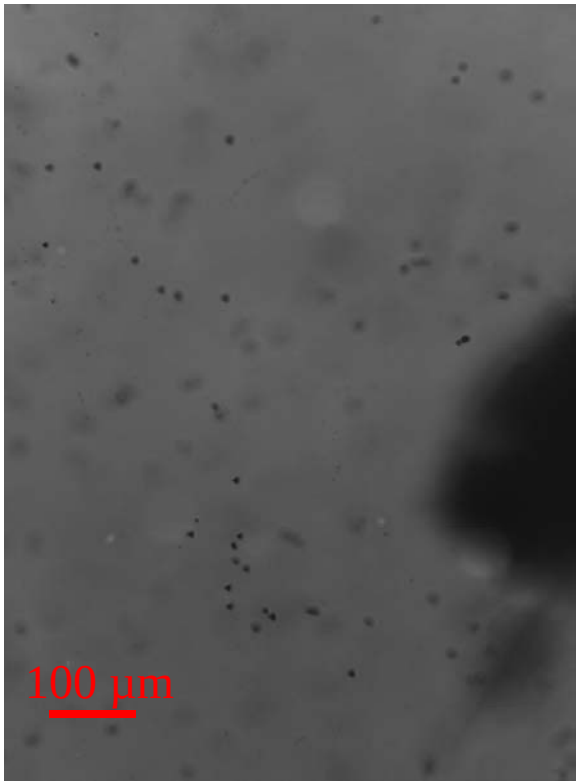


After annealing

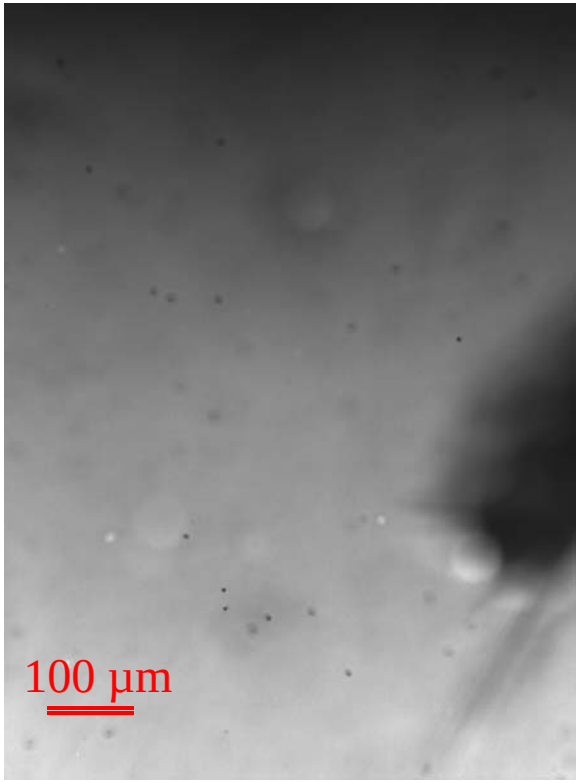
Small-size Te inclusions were removed, and large-size Te inclusions remained.

Cd Vapor Annealing at 700 °C

Cd Over-Pressure, CZT Temperature at 700 °C (Uniform Temperature Field)



Before annealing

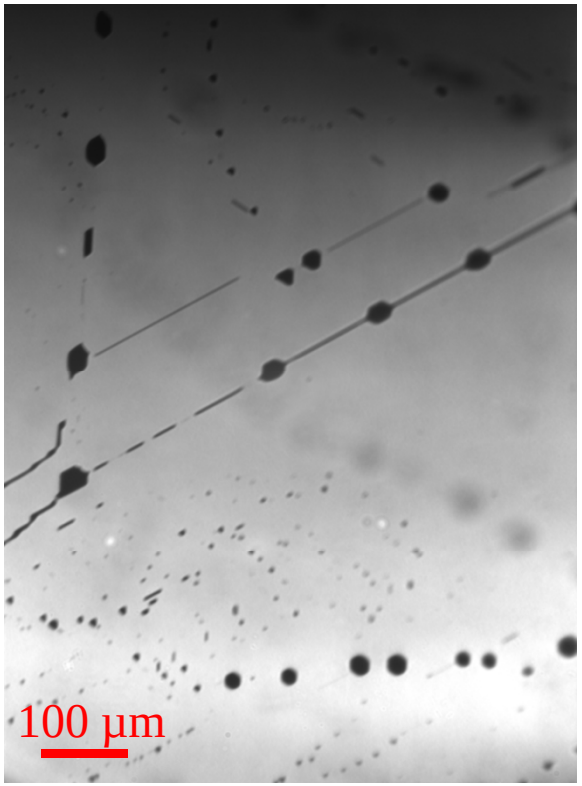


After annealing

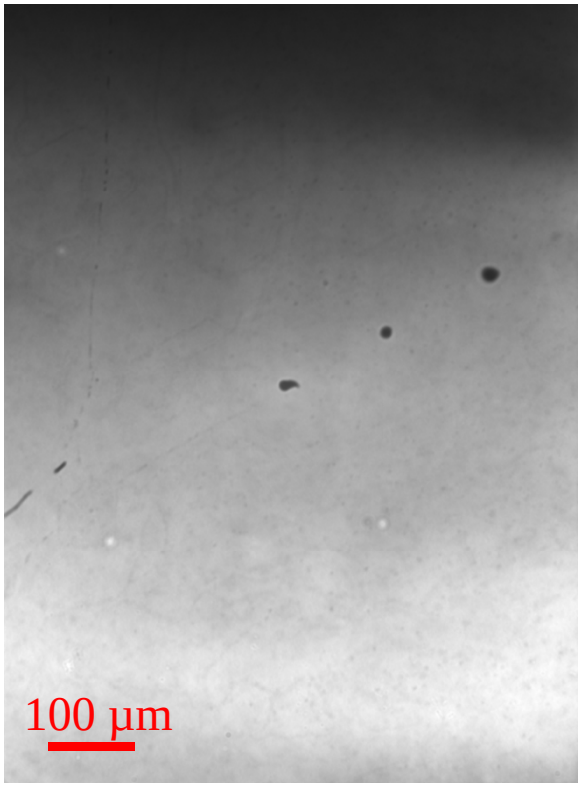
Some of large-size Te inclusions were removed at 700 °C

Cd Vapor Annealing at 823 °C

Cd Over-Pressure Annealing at 823 °C (Uniform Temperature Field)



Before annealing

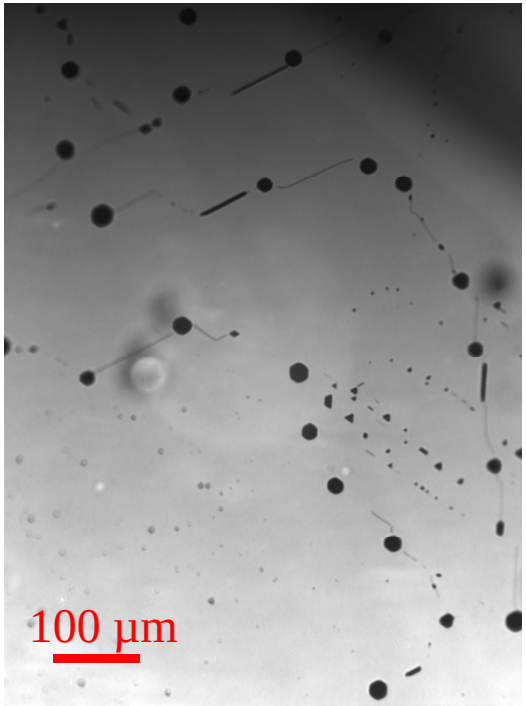


After annealing

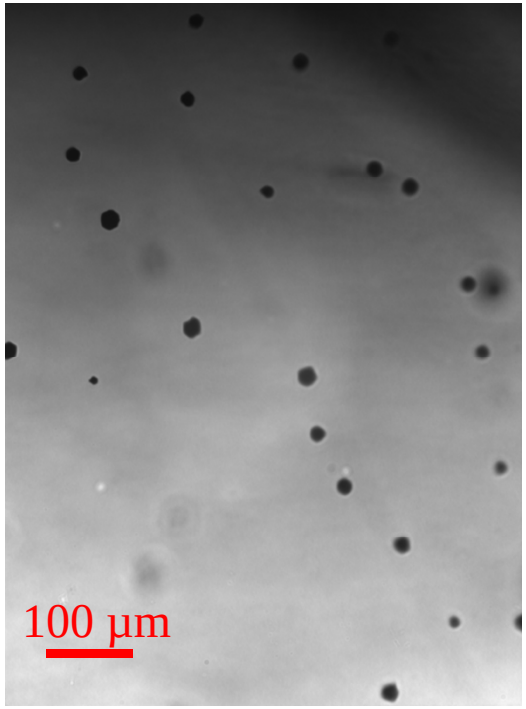
Te Inclusions were significantly removed --- Higher temperature annealing has stronger effect

Removing Te inclusions with Increasing Annealing Time

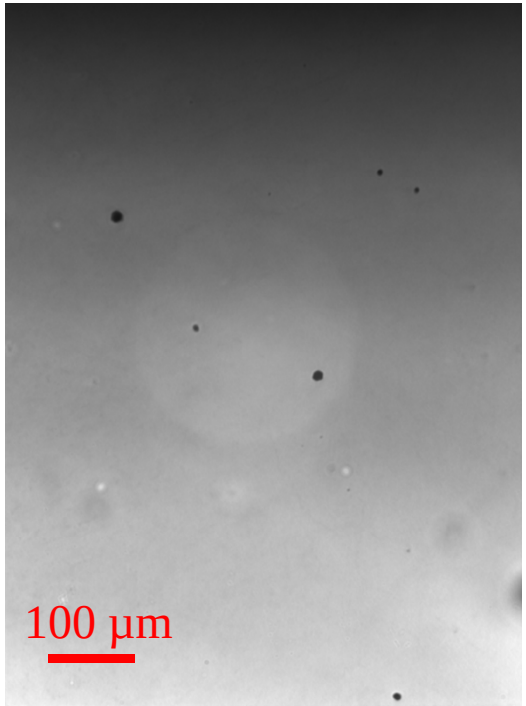
Te inclusions were continuously decreased with increasing annealing time



Before annealing



After 1st annealing



After 2nd annealing

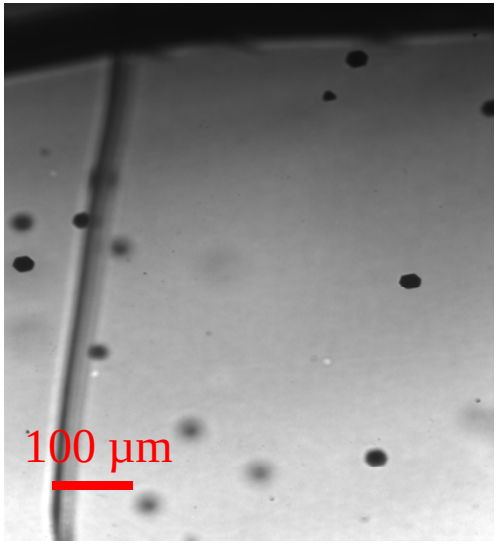
1st annealing conditions: T=827 ℃, t=15 min

2nd annealing conditions: T=827 ℃, t=15 min

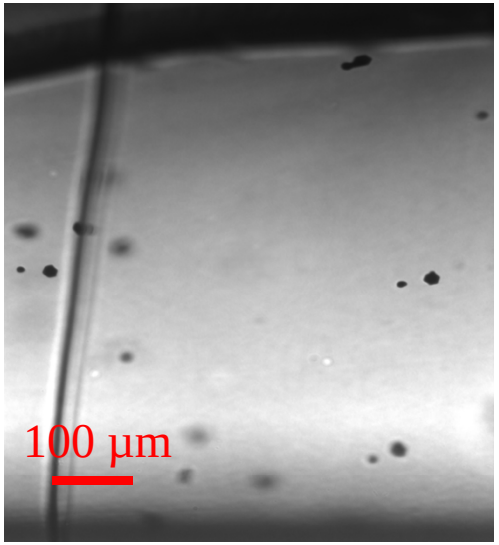
Migration of Te Inclusions in a Temperature Gradient Annealing

700 °C, Temperature gradient of 10 °C/cm, Cd over-pressure

Low High



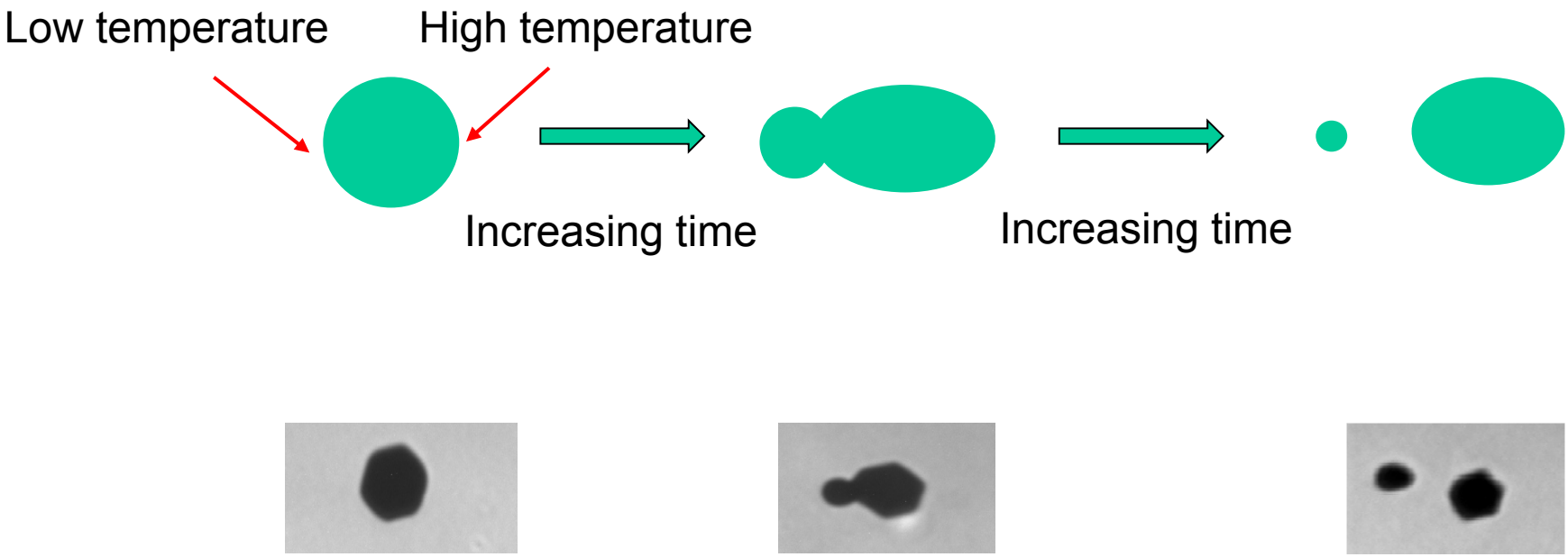
Low High



Thermo-migration velocity of Te inclusions is 4~5 µm/h

While small-size Te inclusions (less than 5 µm) are relatively difficult to move, large-size ones (20 to 40 µm) can easily migrate, possibly due to lower surface tension and larger temperature difference between hot and cold ends of the inclusions

Thermo-migration mechanism of Te inclusions

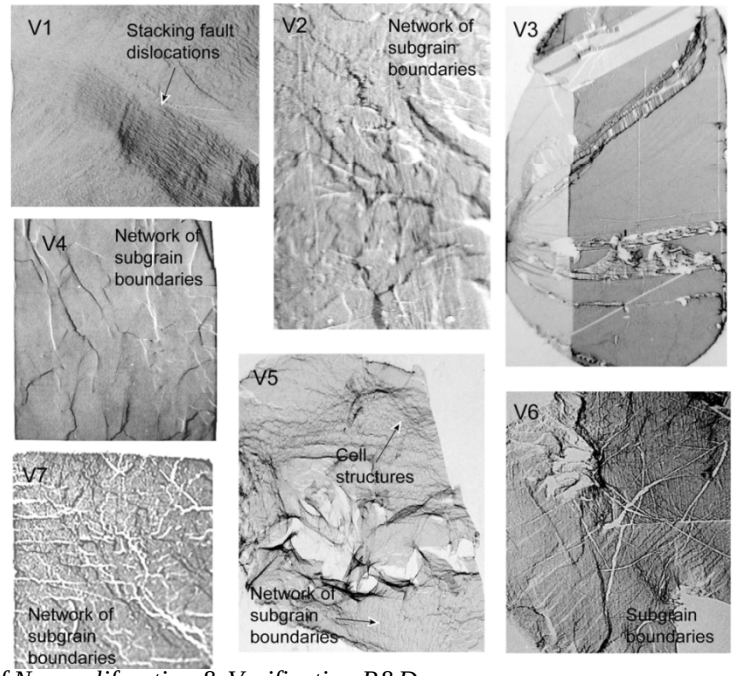


Note: Some dark spots were left behind after the migration of large-size Te inclusions. Possibly, the dark spots are empty space in these large-size Te inclusions.

Results and Findings

- Up to date, we successfully eliminated Te-rich inclusions in CZT by post-growth annealing.
- Next step: We are investigating if the post-growth annealing can remove other extended defects.

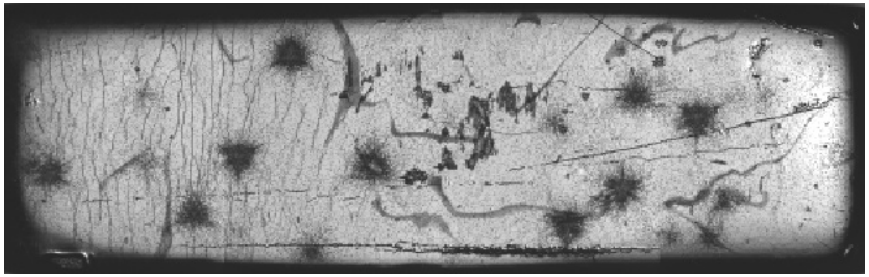
Note: It was observed that the post-growth annealing (usually high temperature, e.g. 900℃) indeed reduces or eliminates extended defects (e.g. dislocations) in other II-VI compounds like ZnO, which also has the same zincblende crystal structure as CZT. (Guillaume et al., *J. Appl. Phys.* 109, 023513 (2011))



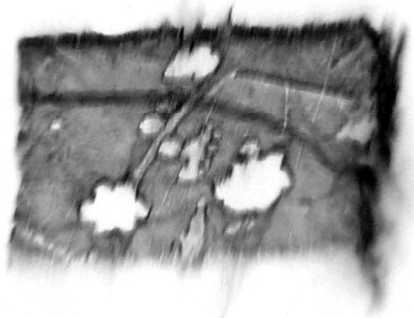
Extended defects (e.g., sub-grain boundaries) can be another important performance-limiting factor besides Te-rich inclusions.

A. E. Bolotnikov et al., IEEE Trans. in Nucl. Sci. 58, 1972 (2011)

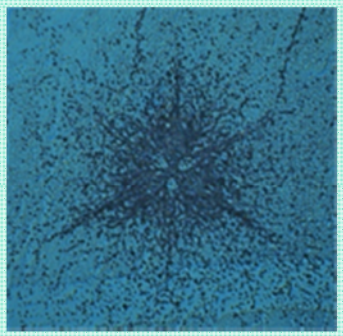
‘Star-like’ Defects in Cd Annealed CZT



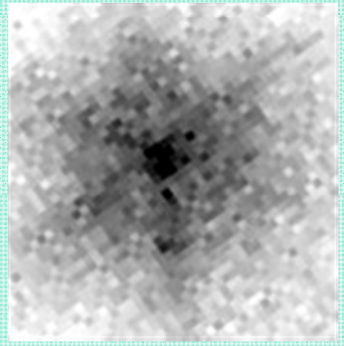
Etch pit pattern



White beam X-ray diffraction topography (WBXDT) image



Optical image



X-ray response map

‘Star-like’ defects are typical features in Cd annealed CdTe and CZT crystals. Such ‘star-like’ defects deteriorate the uniformity of charge transport.

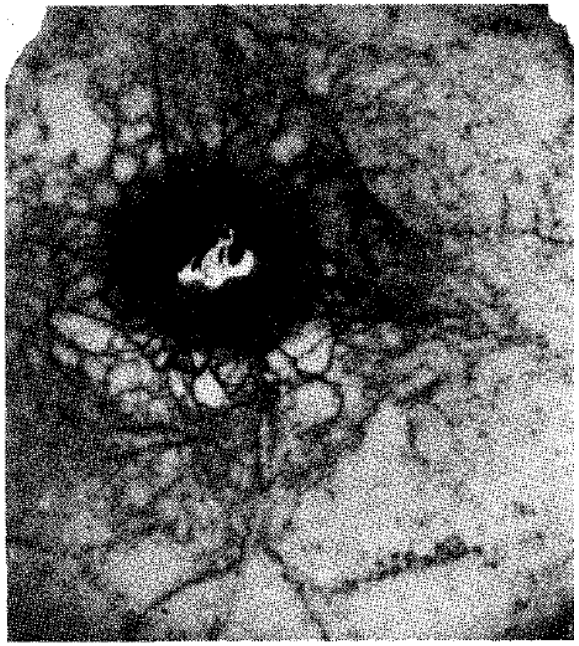
Origin of 'Star-like' Defect

No core in the middle of the 'stars' can be revealed by IR imaging

These 'star-like' defects may be the remnant of Te-rich inclusions, after they have been eliminated by Cd annealing

However, sizes of 'star-like' defects are much larger (100 times) than those of Te-rich inclusions and the concentration of 'star-like' defects is much lower than that of Te-rich inclusions before annealing. Why?

Possibly, the 'star-like' defects are results of the movement, consolidation and reactions of the dislocations surrounding the inclusions, which are driven by the heat release and the pressure release when eliminating large-size Te inclusions during annealing



Catho-luminescence image

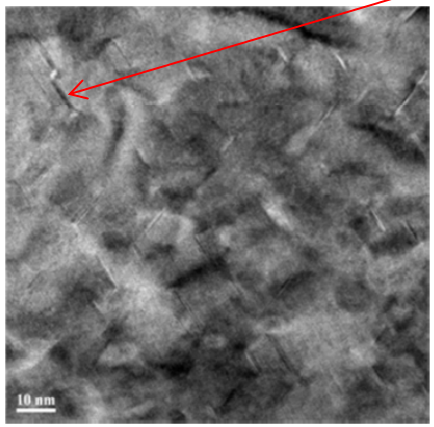
100 μm

(J. L. Pautrat et al., J. Appl. Phys., 53, 8668, 1982)

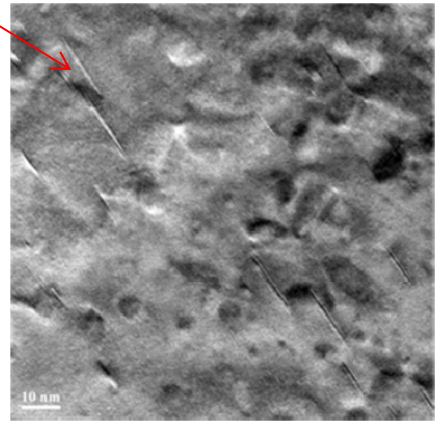
TEM Observation of Extended Defects

Transmission electron microscopy (TEM) observation of extended defects of CZT before and after annealing

Micro-twins



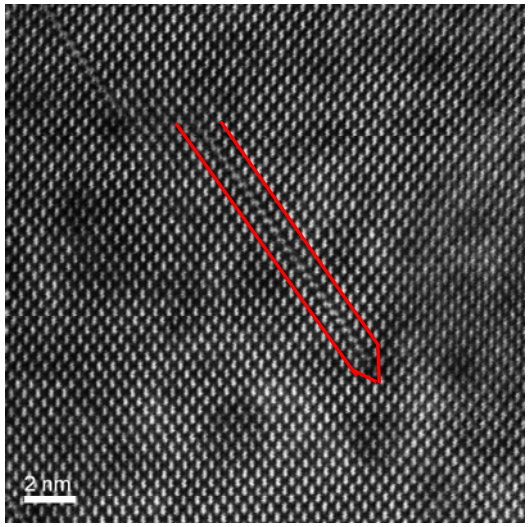
Before annealing



After annealing

TEM images of CZT before and after annealing in Cd vapor (600 ℃)

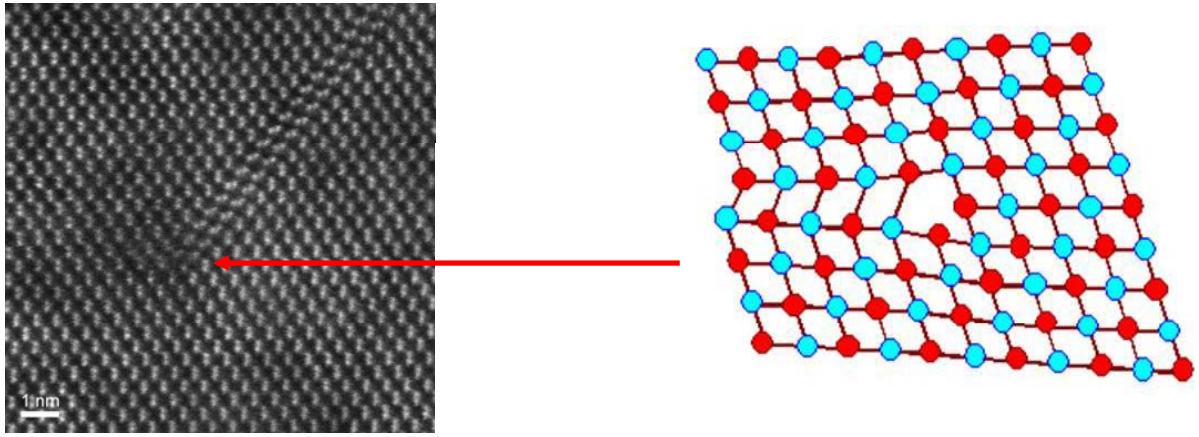
Micro-twin density was reduced after the annealing, while the physical size of individual 'micro-twins' was enlarged



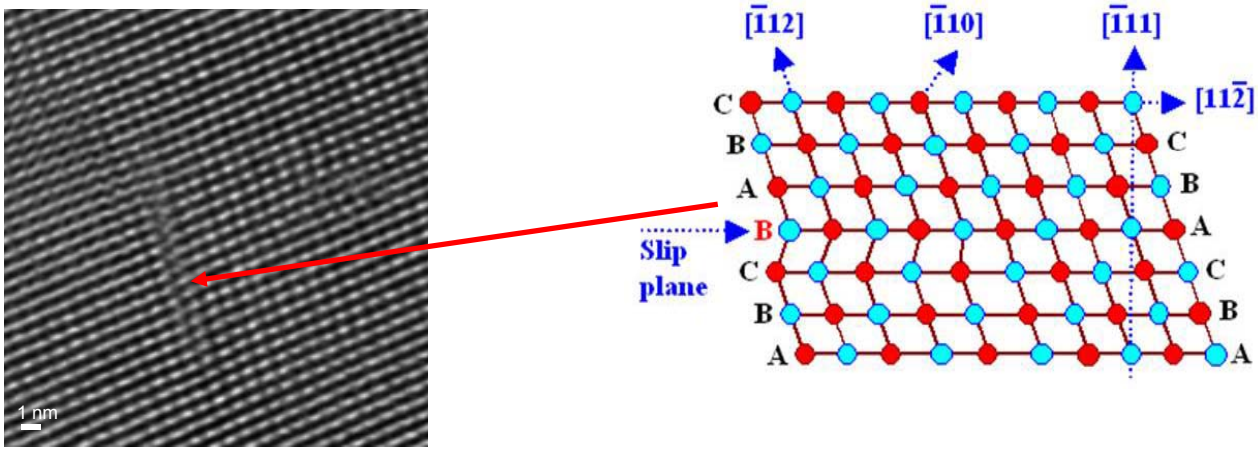
Details of micro-twins revealed by STEM, showing dislocations at the ends of micro-twins

Dislocation Dissociation in CZT

Dislocation dissociation phenomenon in CZT, revealed by high-resolution transmission electron microscopy (HRTEM)



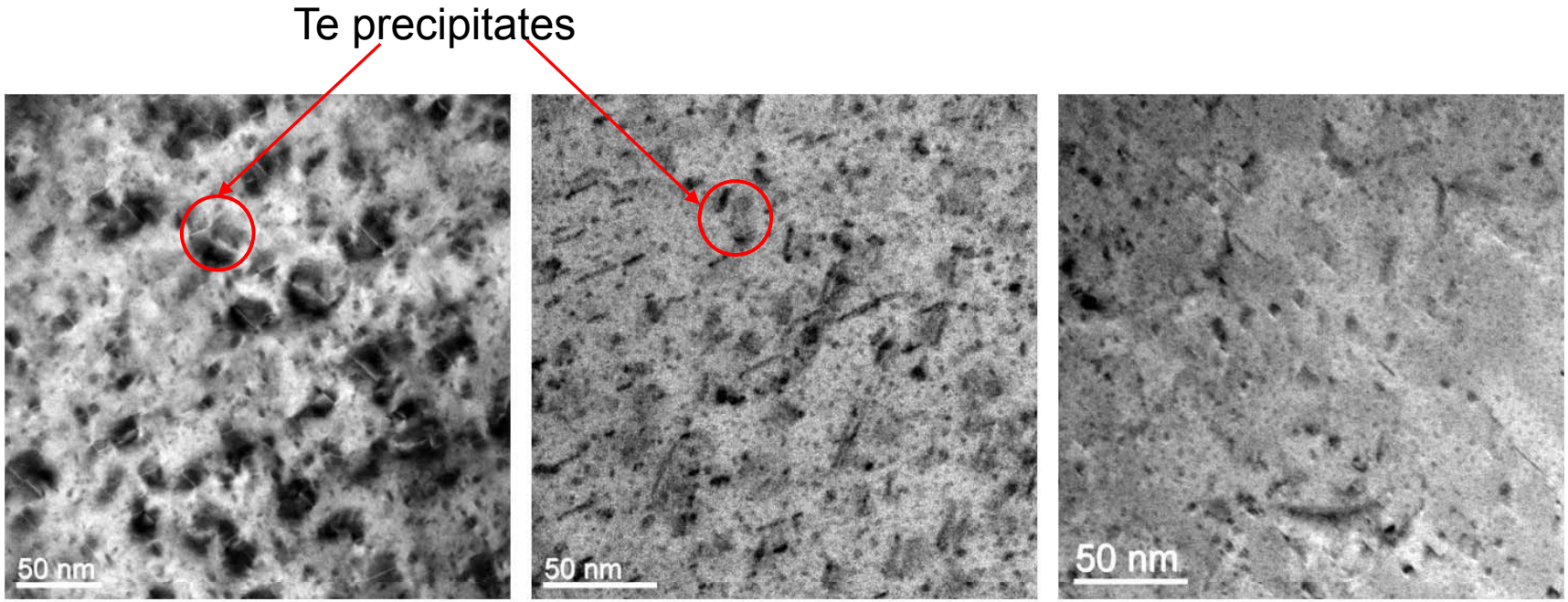
Frank partial dislocation b = a/3 <111>



Shockley partial dislocation b = a/6 <112>

Change of Te Precipitates in Cd Vapor Annealing

Te precipitates of as-grown and annealed CZT crystals with different Cd over-pressure



As-grown

Annealed
CZT/Cd – 700 ^{o}C / 490 ^{o}C

Annealed
CZT/Cd – 700 ^{o}C / 600 ^{o}C

Te precipitates can be eliminated by Cd annealing (Higher Cd over-pressure helps to promote this effect)

Resistivity Change after Annealing

After only-Cd annealing, the loss of resistivity can be 5 orders of magnitude

Possible Reason: The introduction of excess Cd atoms changes the compensation condition between deep donor Te antisites, Cd interstitials, and the acceptor Cd vacancies, which pins the Fermi level and results in high resistivity of CZT

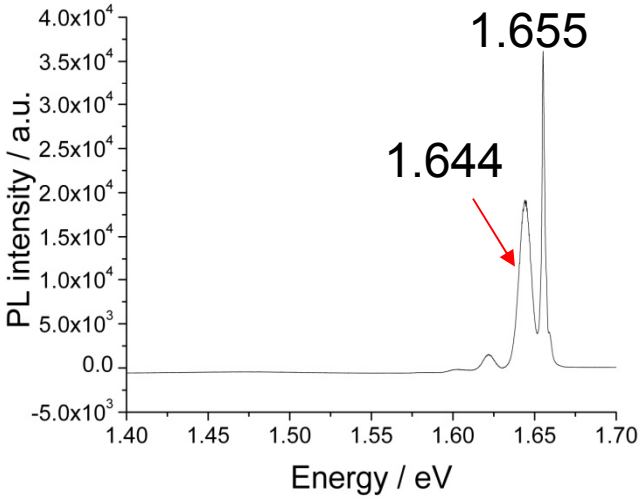
Proposed solutions:

I: Two step annealing: (1) under Cd overpressure to remove Te inclusions; (2) under Te overpressure to restore high resistivity

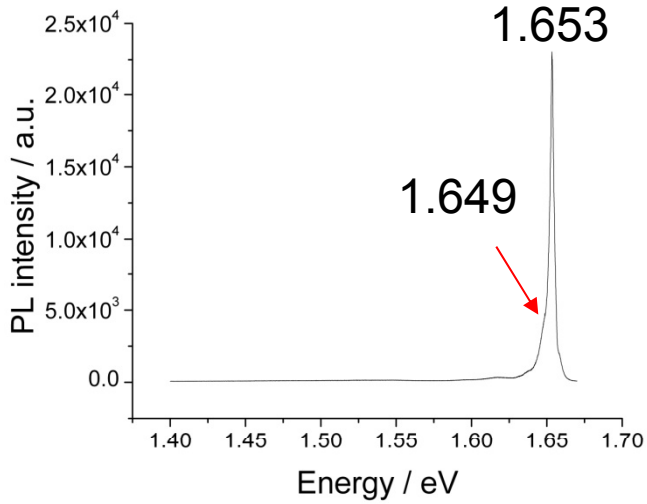
II: One step annealing with suitable Zn pressure control (using Cd + Zn sources)

We have demonstrated both ways work (the resistivity was only lowered 1-2 orders). We are concentrating on optimizing the annealing conditions to further improve the resistivity

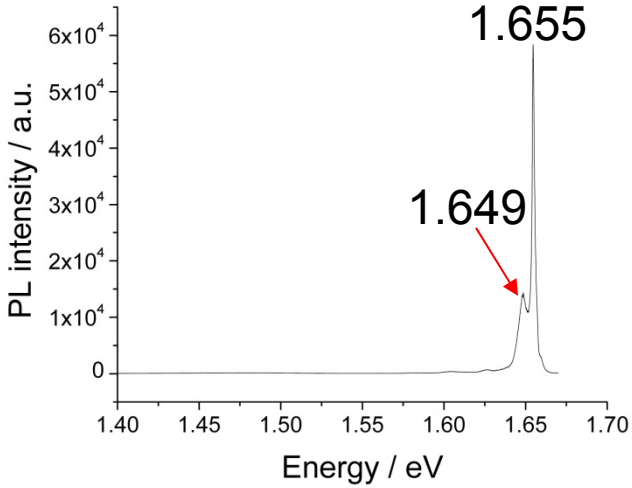
Photoluminescence Spectroscopy Measurement (4.2 K)



As-grown, resistivity: 10^{10} Ω·cm



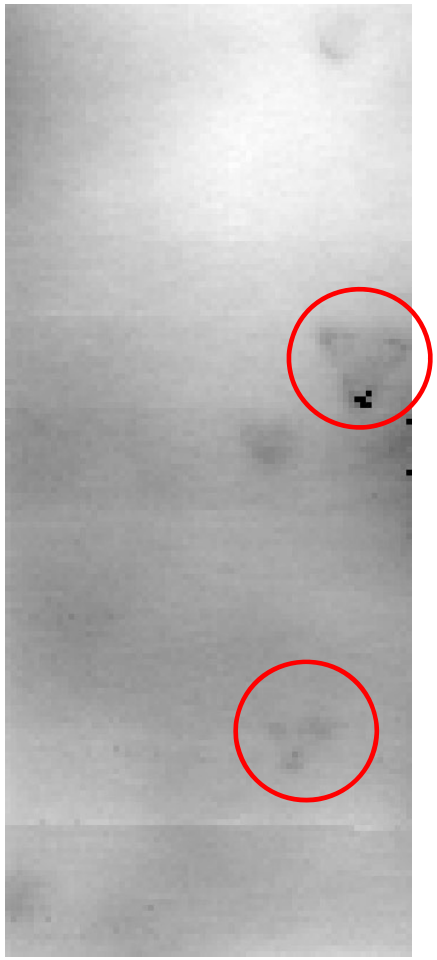
After Cd annealing, resistivity: 10^{4} Ω·cm



After Cd + Zn annealing, resistivity: 10^{8} Ω·cm

The intensity of the acceptor-bound exciton (A^0,X) peak at ~1.649 eV is significantly strengthened when the resistivity is high! Possibly this phenomenon is related to the change of Cd vacancies during the annealing

X-ray Response Map of An Annealed Sample



There are several triangular features (~ 100 μm), which have much less charge loss than regular large-size Te inclusions. These features are possibly the remnant of large-size Te inclusions after they were eliminated by the annealing

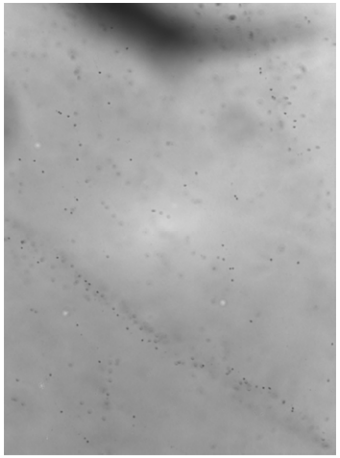
Two step annealing conditions:

1st annealing: Sample/ Cd, 800 ℃ / 750 ℃, 24 hours

2nd annealing: Sample/ Te, 700 ℃ / 550 - 600 ℃, 24 hours

The area of X-ray response map is 0.70 x 1.68 mm^2

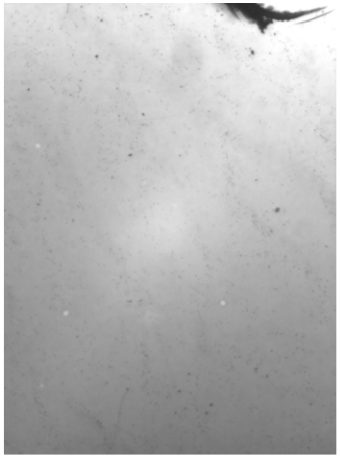
Interesting Phenomenon During Cd + Zn Annealing



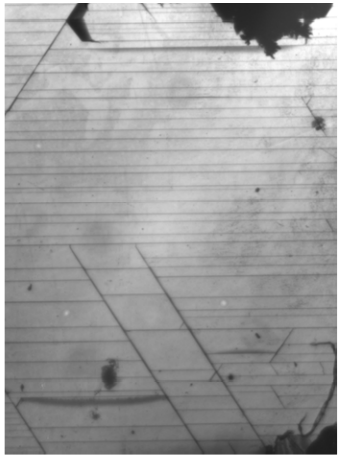
As-grown

After annealing

IR images of as-grown and annealed CZT crystal (750 ℃, under Cd + Zn vapors), showing the reduction of Te inclusions. The area is 0.66 x 0.90 mm^2.



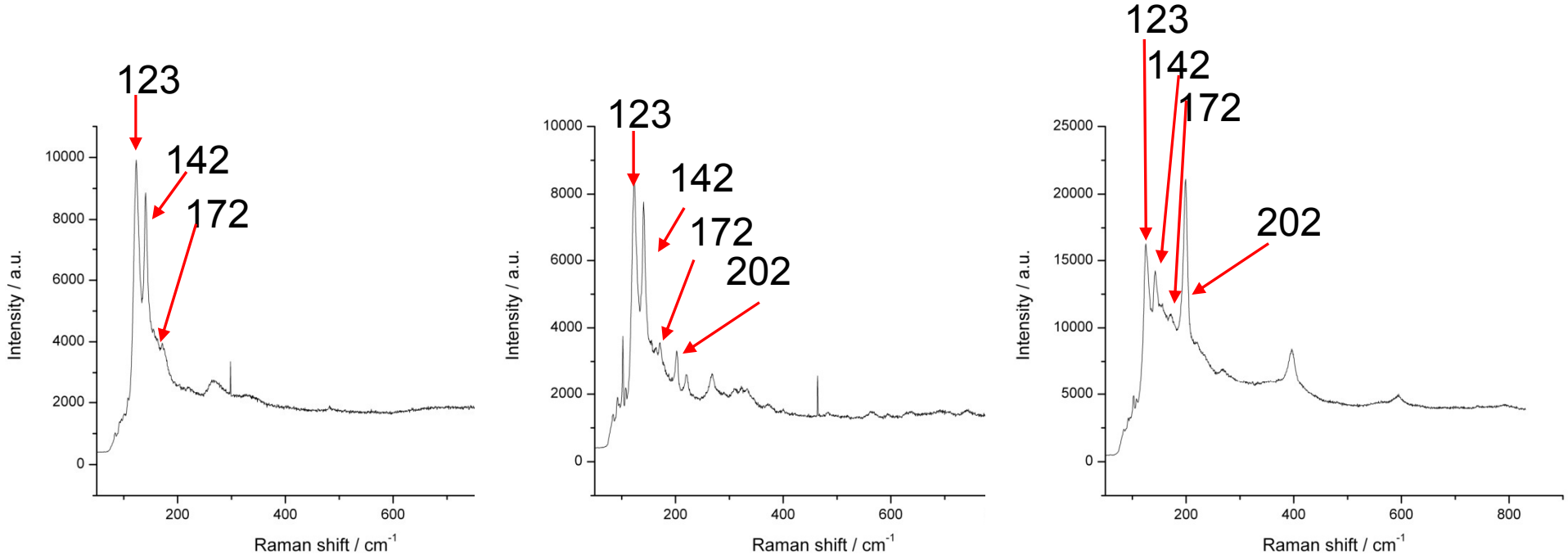
As-grown



After annealing

Surface morphology of as-grown CZT and annealed CZT (750 ℃ under Cd + Zn vapors). New twin patterns were formed on the surfaces of CZT during the annealing. The area is 0.66 x 0.90 mm^2.

Raman Spectroscopy Before and After Annealing



As-grown | After Cd annealing | After Cd + Zn annealing

The peaks at 123 cm^{-1} and 142 cm^{-1} correspond to Te

The peak at 172 cm^{-1} has been assigned to the longitudinal optic (LO) mode of CdTe

The origin of the peak at 202 cm^{-1} is not clear, while this peak dominates the spectrum after Cd + Zn annealing

Summary

- We developed single-zone and two-zone annealing furnaces with customized software to automatically control the temperature field
- We successfully eliminated Te-rich inclusions/precipitates in CZT by post-growth annealing
- We observed thermo-migration of Te inclusions in a temperature-gradient field for Cd/Te vapor annealing
- We observed 'star-like' extended defects in CZT after Cd vapor annealing
- We observed the evolution of nano-scale defects like Te precipitates, micro twins and dislocations during annealing
- We achieved relatively high resistivity after two-step annealing and one-step annealing with suitable Zn pressure control
- We rapidly disseminated data on annealing experiments to the radiation detector community (15 publications and 20 presentations)

Future Work

- Clarify the evolution processes and mechanisms of material defects (especially dislocations and sub-grain boundaries) during annealing
- Improve the electrical properties, e.g. high resistivity and the carrier mobility-lifetime products for thermally-annealed, inclusion-free CZT
- In-situ TEM observation of CZT during the annealing process
- Correlate material properties (inclusion content, dislocations, resistivity, and mobility-lifetime products) and device performance (resolution and stability) with the annealing conditions
- Apply understanding of annealing effects to large-volume CZT crystals, considering the influence of sample dimensions on processing conditions
- Continue to disseminate data on annealing experiments and material and detector characterization measurements

Acknowledgement

This work was supported by U.S. Department of Energy, Office of Nonproliferation and Verification Research & Development, NA-22.

Thank you for your attention!

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