SAND2022-10387C

Sandia National Laboratories

In-situ TEM laser heating for manipulation of cooling rates and observation of precipitate dissolution kinetics

M&M 2022 MICROSCOPY & MICROANALYSIS July 31-August 4 • Portland, OR

Kathryn Small, Ryan DeMott, Erin Barrick, Jeff Rodelas, Khalid Hattar

August 3, 2022

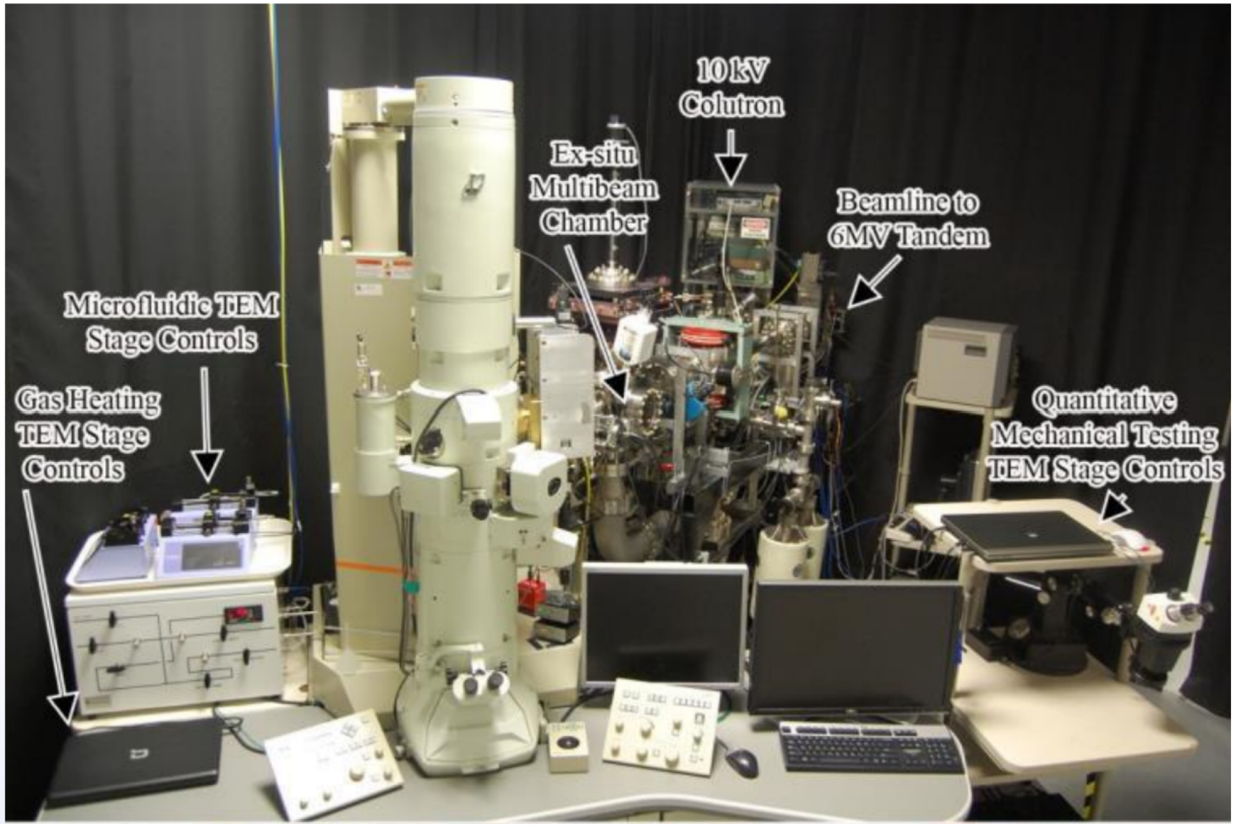
This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, 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 Unit

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

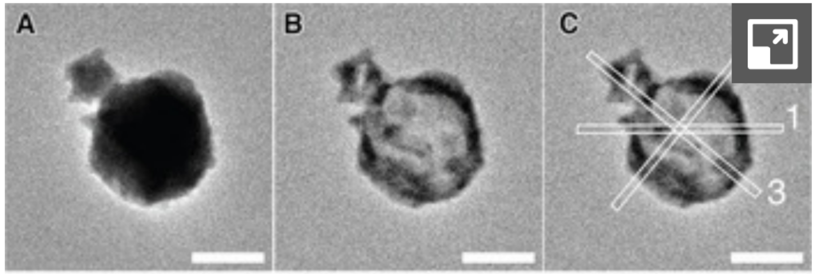
In-situ TEM: Capabilities

- The In-situ Ion Irradiation TEM (I3TEM) at the Ion Beam Laboratory at Sandia National Labs
- Variety of holders
  - Thermal: heating, cryo
  - Mechanical: straining, fatigue
  - Electro/chemical: liquid/gas cell with biasing
- Recent WAVIKS installation
- IR laser 1064nm wavelength, 20W

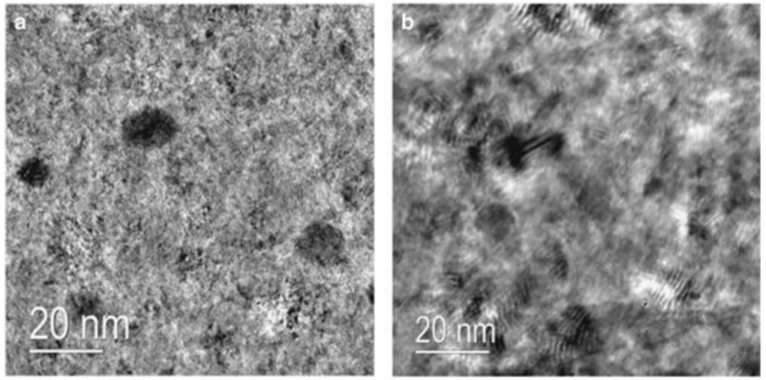


Current heating work with lasers

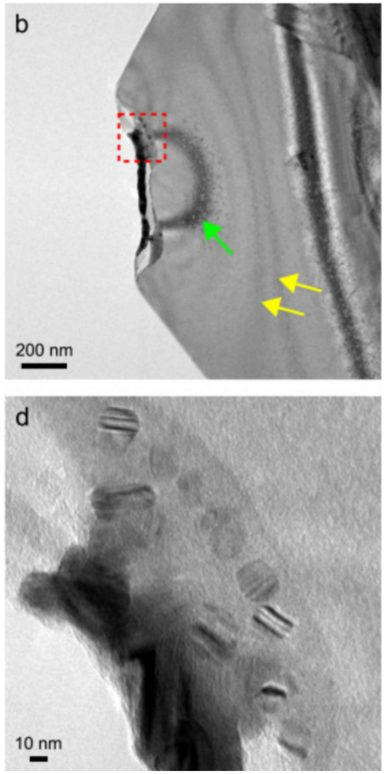
- Observe thermally activated microstructural processes
- Crystallization of amorphous materials
- Synthesis of nanowires
- Nanoparticle synthesis through laser ablation
- Hollow zinc oxide nanoparticle generation using infrared heating



Mehraeen, Shareghe, et al. *Microscopy and Microanalysis* 19.2 (2013)



Wu, Yueying, et al. *Microscopy and Microanalysis* 24.6 (2018): 647-656.



Allen, F. I., et al. *Ultramicroscopy* 178 (2017)

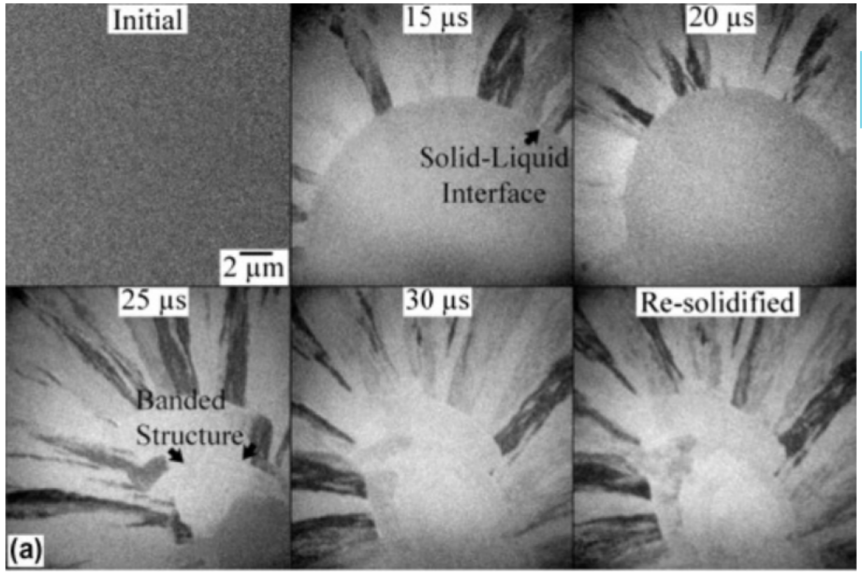
Taheri, Mitra L., et al. "Laser-based in situ techniques: Novel methods for generating extreme conditions in TEM samples." *Microscopy research and technique* 72.3 (2009): 122-130.

Observations of cooling

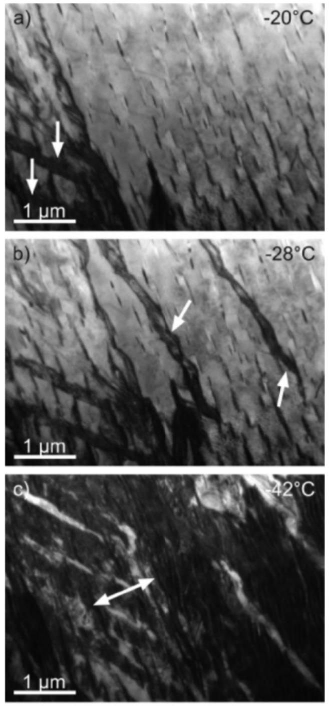
- Rapid solidification after laser processing
- Martensitic transformations
  - Shape memory alloys
- Phase transformation at various cooling rates

In-situ TEM lasers can be used to observe microstructural changes in real time during ultrafast cooling

Dlouhy, Antonin, Jafar Khalil-Allafi, and Gunther Eggeler. *Philosophical Magazine* 83.3 (2003): 339-363.



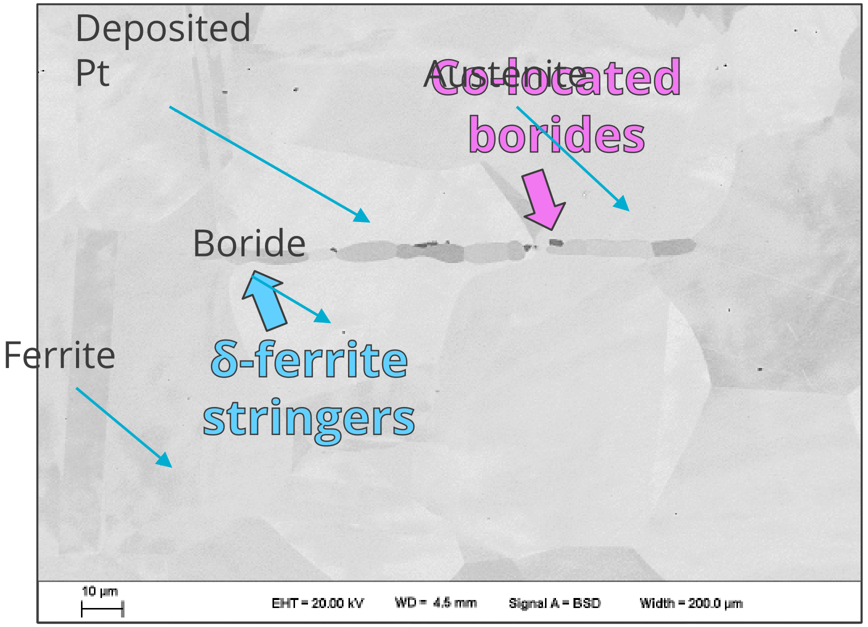
McKeown, Joseph T., et al. *Acta Materialia* 65 (2014): 56-68.



Kröger, A., et al. *Materials Science and Engineering: A* 481 (2008): 452-456.

Methods and Materials

- FIB lift-out of 304L VAR stainless steel
- Known to contain boride (CR2B) precipitates at Austenite/Ferrite phase boundaries
- After heat treatment, GB borides cause liquation cracking in welded SS



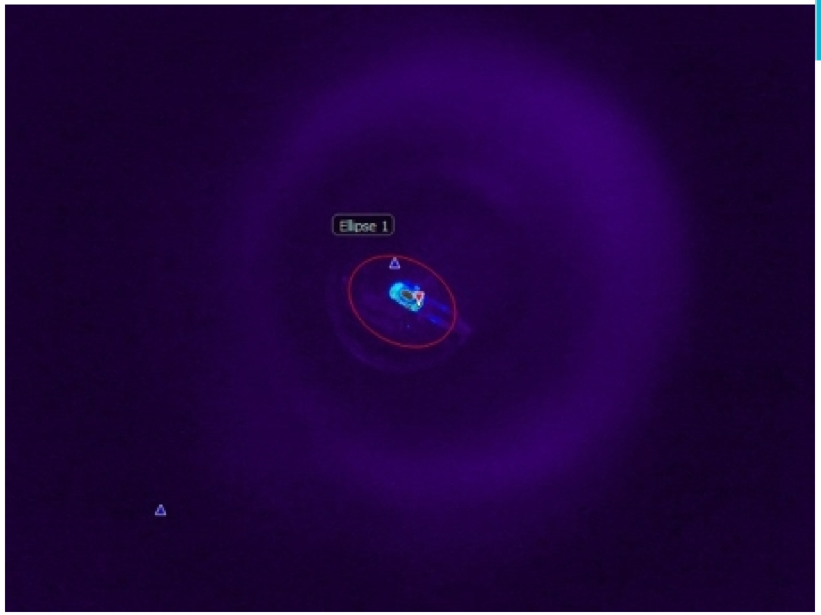
- Increased laser power incrementally to minimize thermal drift and allow equilibration
- Waited for features indicating melting of Pt
- Peak laser power differed between holder temperatures

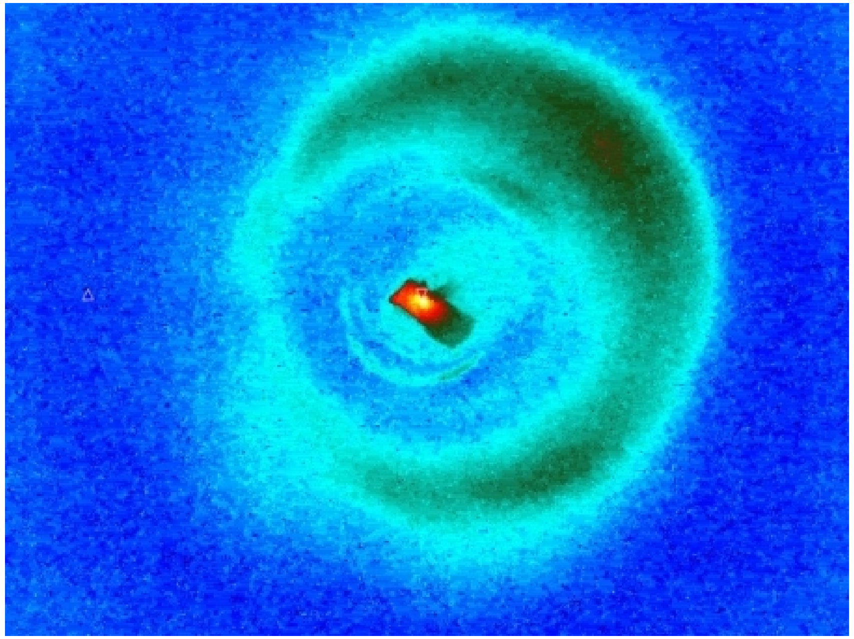
Experiment Temperature	Holder Used	Peak Laser Power (W)	Laser Power Increment (W)
Room	Double Tilt	4	1
Cryogenic	Cryo DT	10	1

Combining in-situ laser heating with heating and cooling holders results in varying cooling rates

Temperature Estimation

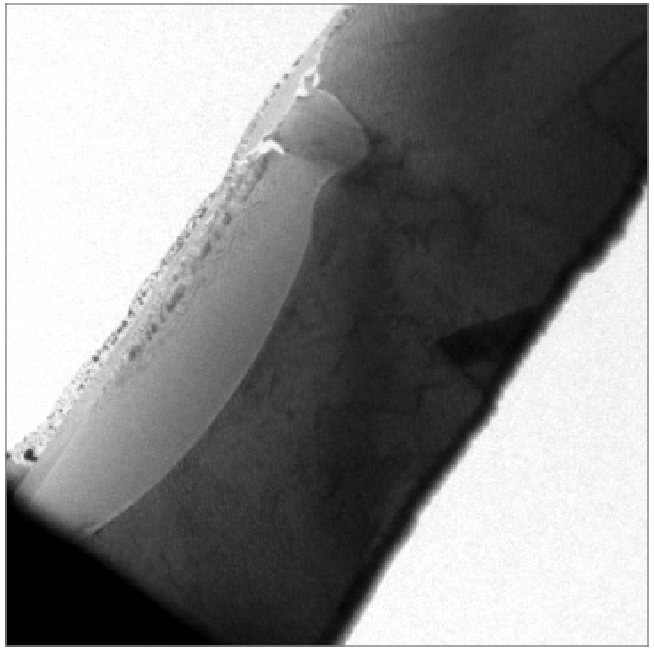
- FLIR Camera
  - Not high enough resolution to distinguish sample from laser beam
  - Calibration required for each phase due to different emissivity and absorptivity
- Diffraction pattern
  - Measurement of d-spacings at higher laser power
  - MATLAB code determines lattice strain through identification of DP spot centroids
    - Manually choose DP spots for each image
  - Strain and temperature results vary widely due to manual spot selection





Change during laser exposure

- Further/faster change with increase from 4.8-5.4W
  - Ferrite degrades more than boride



10 fps

- Overall change from pre- to post-exposure
  - Some recovery of Austenite phase visible
  - Degradation of ferrite and, to a lesser extent, boride
  - Most degradation of Pt deposition
    - may be helpful to remove Pt layer prior to laser heating to make results more clear

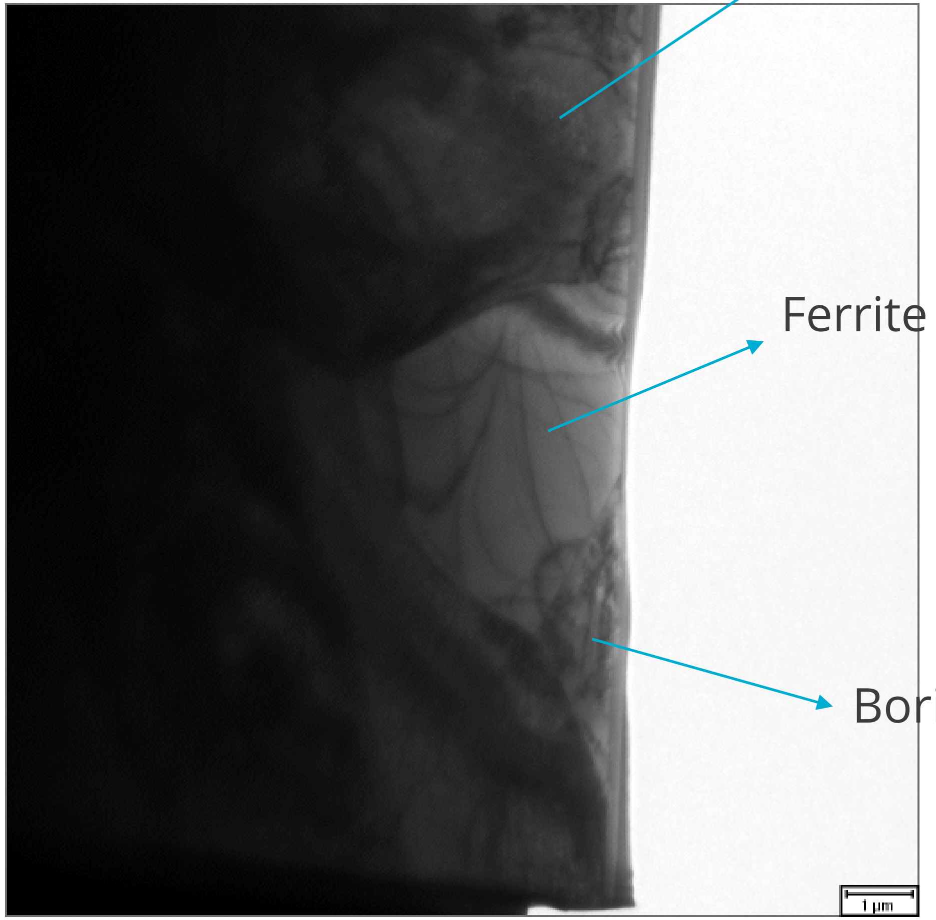
Post-laser heating (up to 5.4W laser power for 10 minutes) at [$\bar{1}$12] zone axis

Pre-laser heating at [$\bar{1}$12] zone axis

Room temperature

At 3W

Sample at RT before laser exposure



Austenite

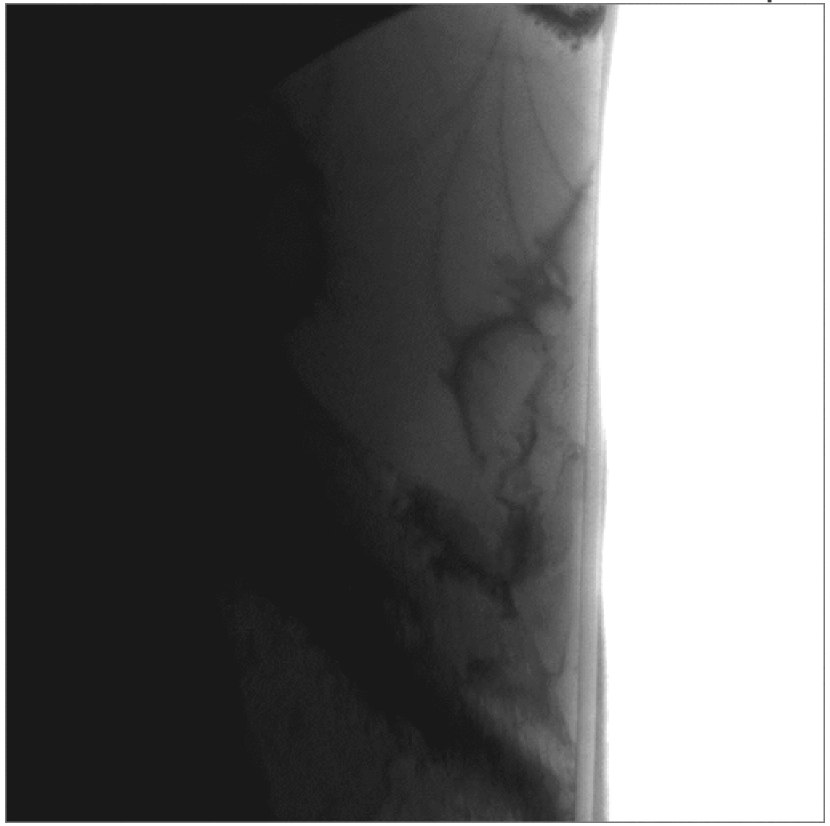
Ferrite

Boride

At 0W

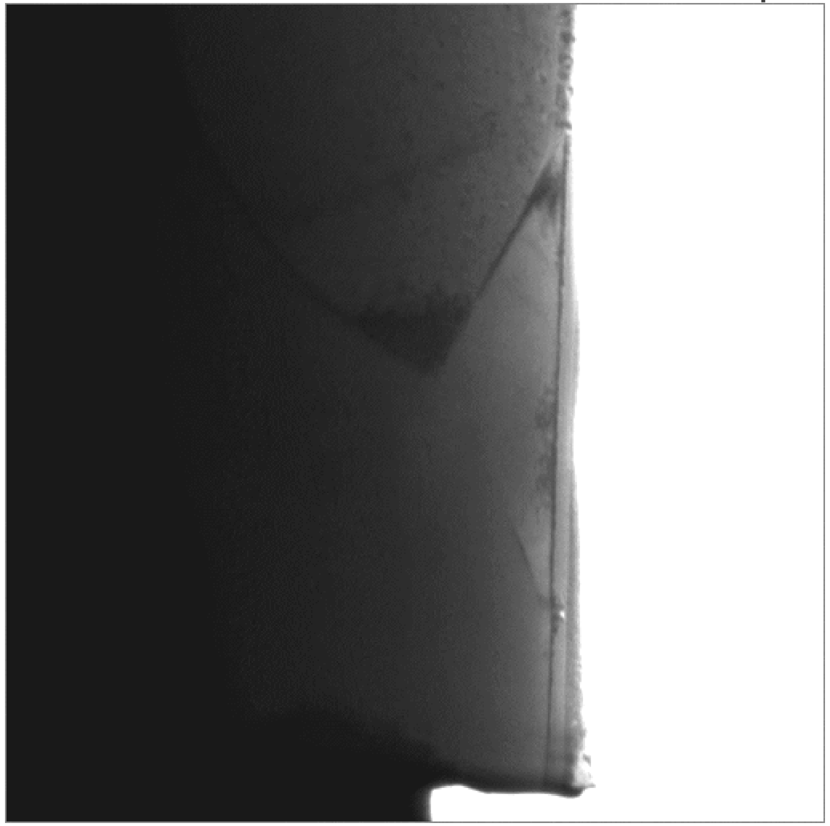
Cooling to Room Temperature

20 fps



3W → 4W → 0W

5 fps



4W → 0W

Cryogenically cooled

At 9W

Sample at -170°C before laser exposure

Austenite

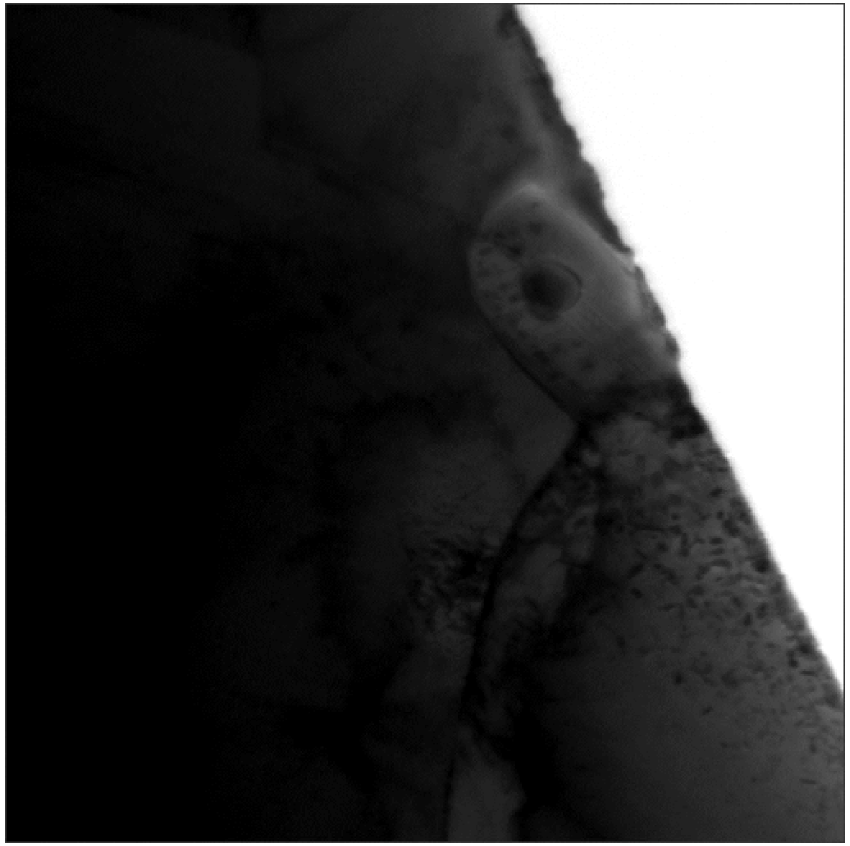
Boride

At 0W

Ferrite

Cooling to Cryogenic Temperature (-170C)

28 fps



9W → 10W → 0W

5 fps

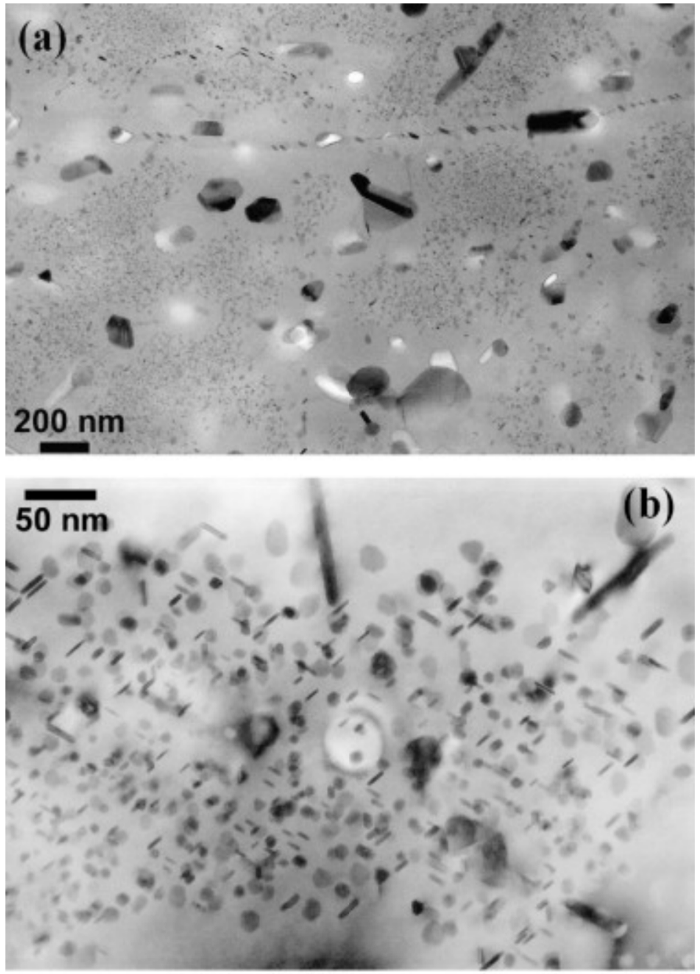
10W → 0W

Microstructural Effects

Cooled to RT after 10W laser power

Cooled to -170C after 10W laser power

Precipitates only form in ferrite; size seems to increase with increased cooling rate

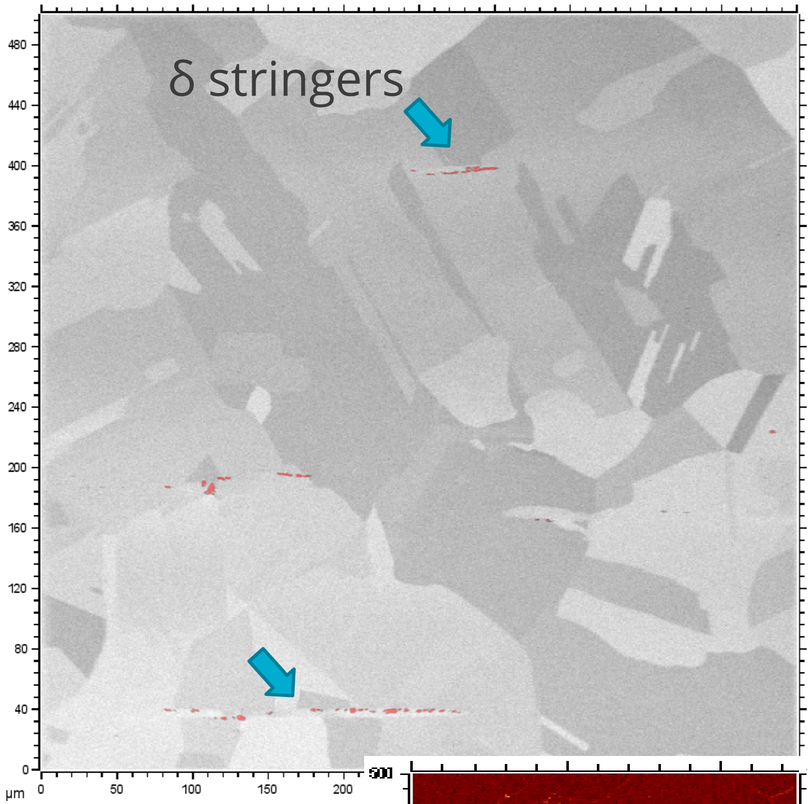


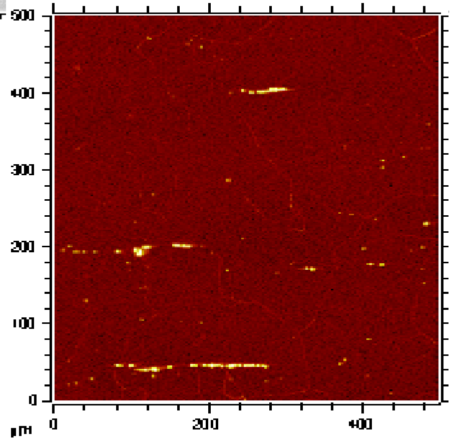
Al-Zn-Mg alloy quenched at 10C/min

Deschamps, A., et al. *Materials Science and Engineering: A* 501.1-2 (2009): 133-139.

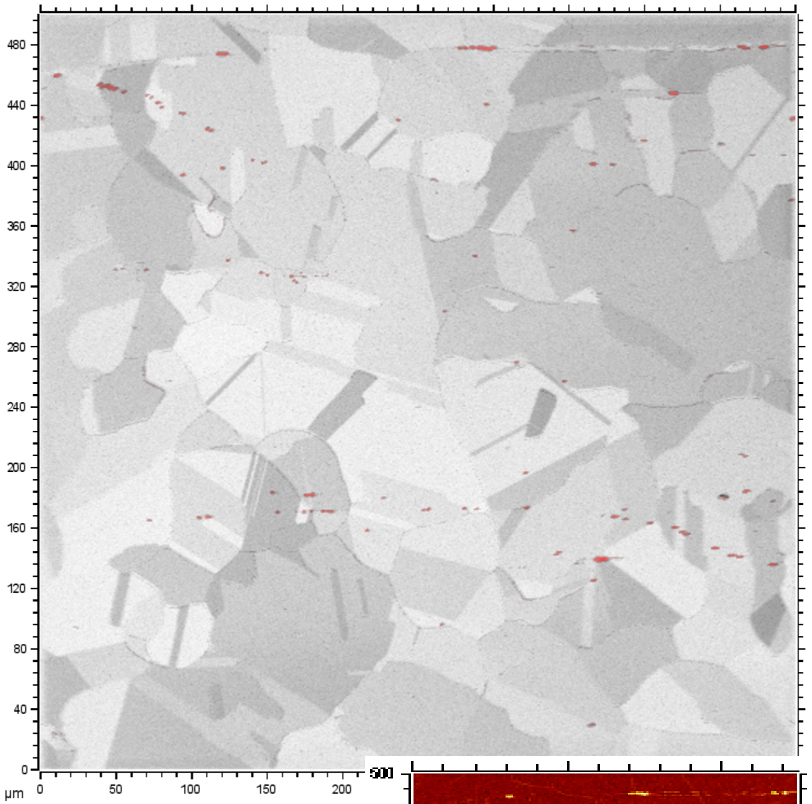
Boride dissolution occurs between 1000°C and 1100°C

As-Received

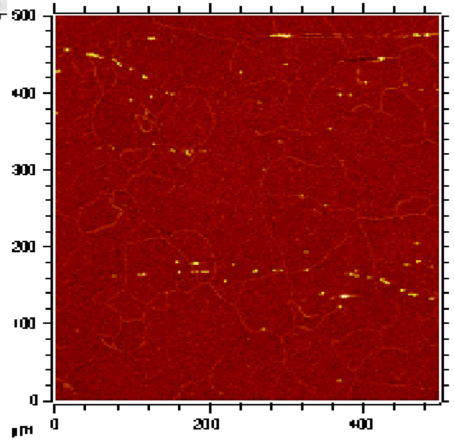




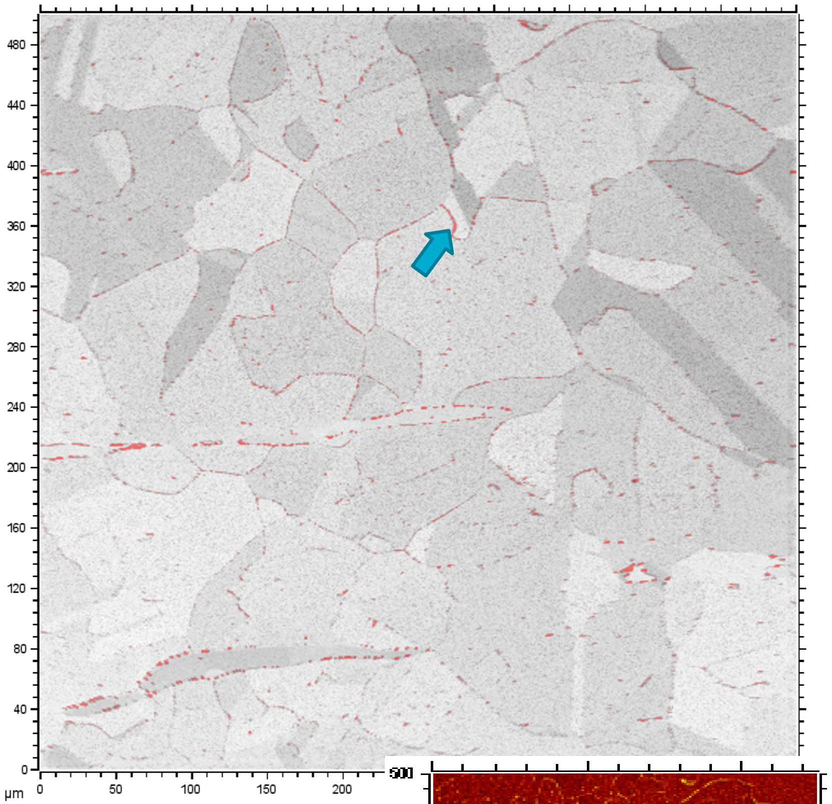
1000°C 32 min



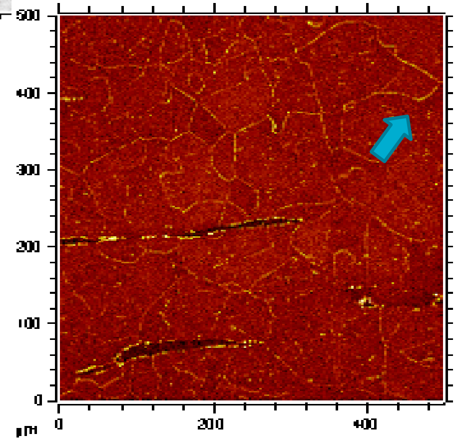
Little (if any) observable changes between as-received and 1000°C 32 min



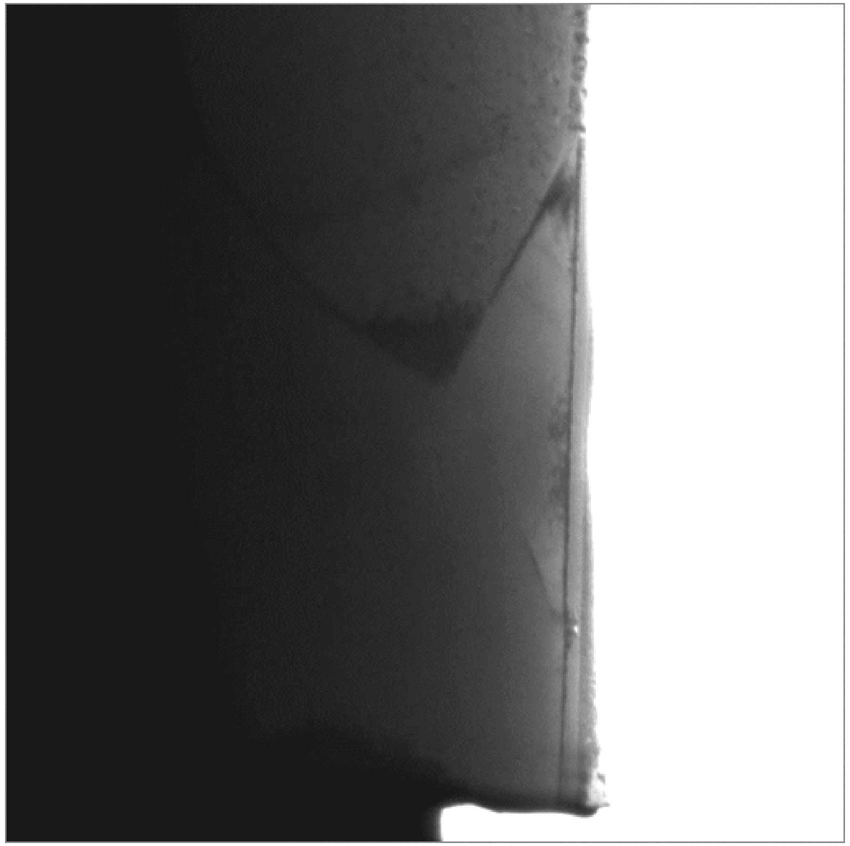
1100°C 1 min



Some boron migration to γ grain boundaries; some remains on δ



Approximation of Cooling Rate Observed



Using RT holder, cooling lasted between 3-5 seconds
← ~366°C/s
(457K/s)

Using cryo holder, cooling lasted no longer than .6 seconds →
~1833°C/s
(2288K/s)

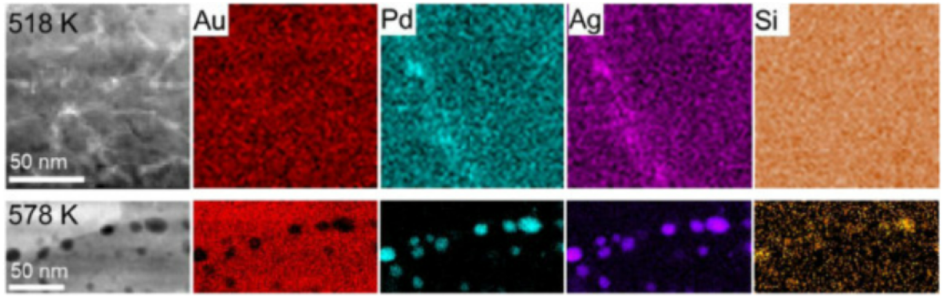
4W → 0W

Decreasing temperature of holder is an easy way to increase cooling rate of sample

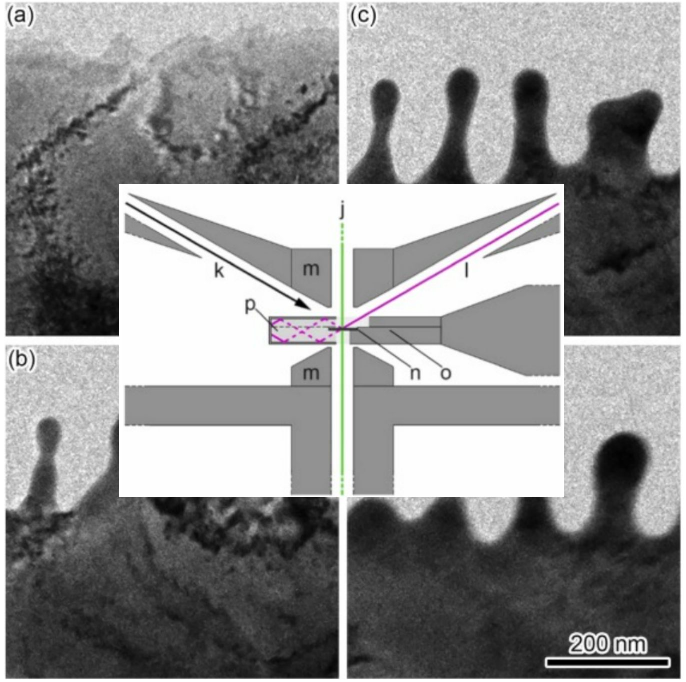
10W → 0W

Future cooling work with lasers

- Temperature measurement
- Precipitate identification in ferrite
- Application to laser fabrication techniques
  - Discrepancy between thermal conductivities (e.g. vacuum vs. metal powder)
- Crystallization of metallic glasses
  - Requires cooling rates ~1000s of K/s
- Development of heat resistant alloys
  - Protection of TEM components with specialized holders



Ivanov, Yu, et al. *Acta Materialia* 196 (2020) 52-60



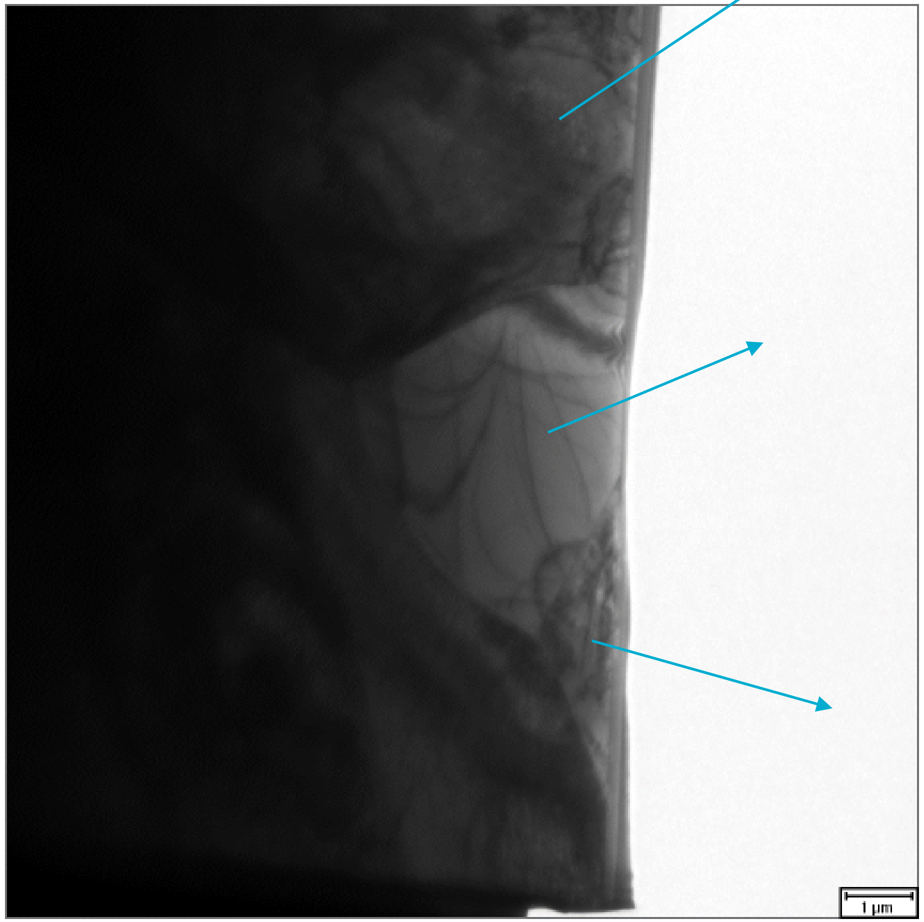
Uemura, Naoki, et al. *Micron* 157 (2022): 103244.

Extra Slides

Room temperature

At 3W

Sample at RT before laser exposure



Austenite

Ferrite

Boride

At 0W

Cryogenically cooled

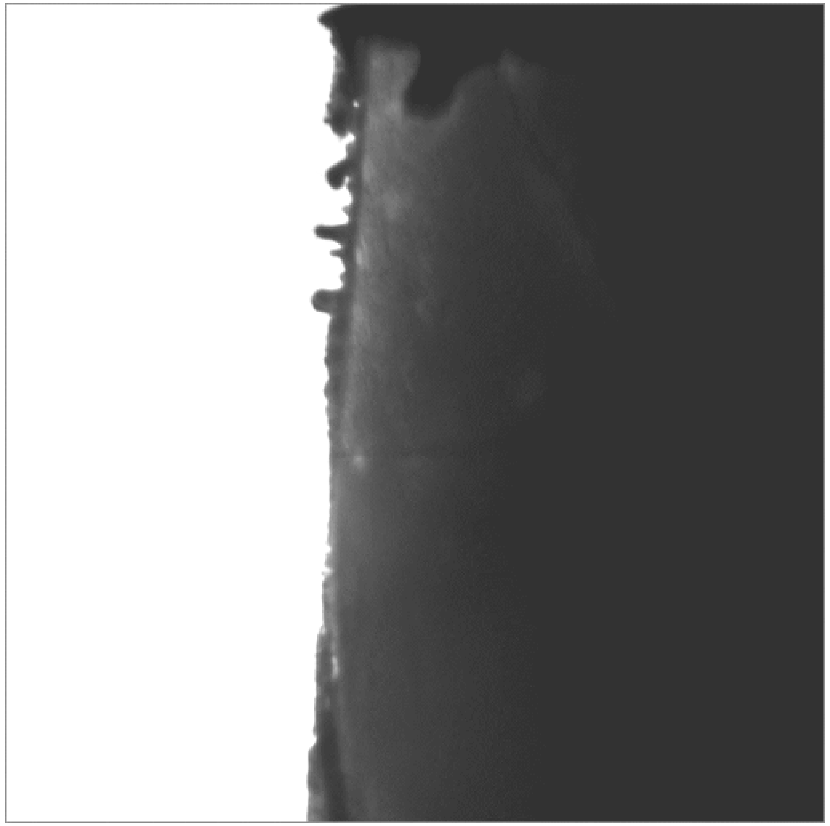
At 9W

At 0W

$[\bar{1}22]$
Austenite

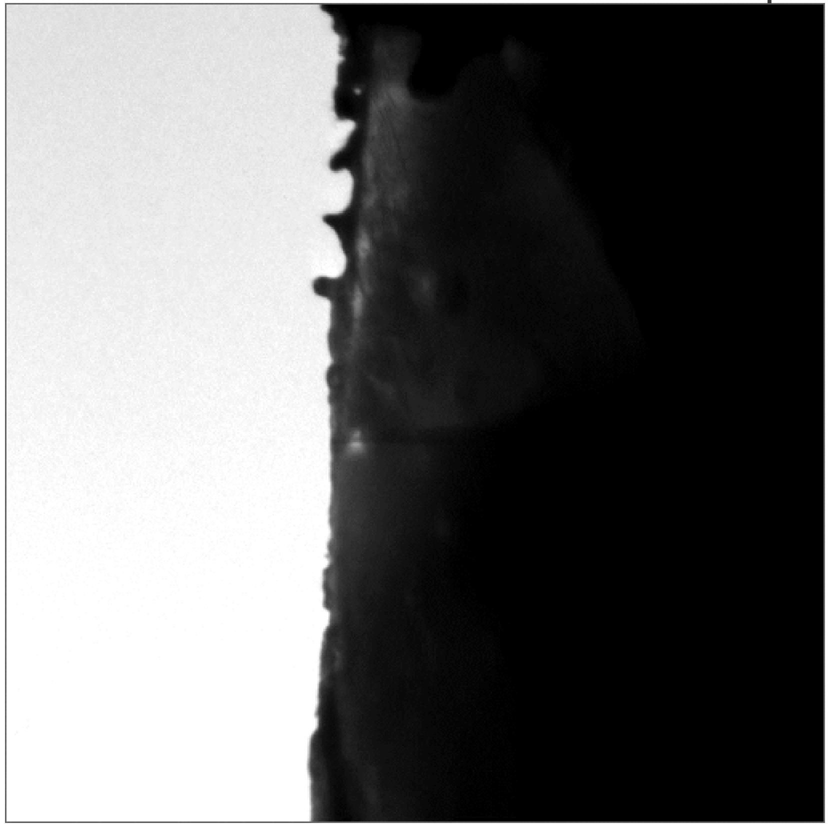
[001]
Ferrite

Heating Holder Cooling Experiment



Heating Holder Cooling Experiment

30 fps



10 fps