Center for Electron Microscopy and Nanofabrication
Promoting Nanotech Education and Research

Portland State University

# Effect of Cryo-Quench on the Microstructure of Copper

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Center for Electron Microscopy and Nanofabrication

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# Microstructures of materials can determine macroscopic applications

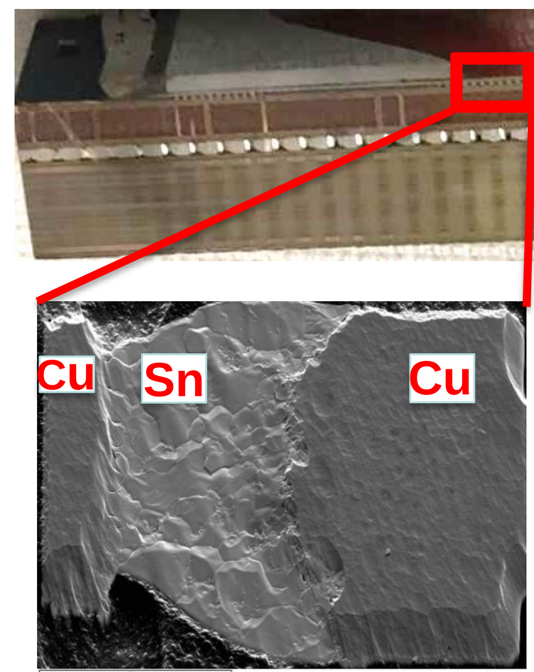
- Copper is an essential interconnect in the semiconductor industry
- Nanocrystalline metals exhibit a promising combinations of conductivity, strength, and ductility[1]
- Refining the microstructure of materials via thermomechanical processing
  - Hot and cold rolling
  - Forging and Extrusion
- Disadvantages include:
  - Extreme heat and pressure systems with large power consumption
  - Cannot be applied to device manufacturing

# Why cryogenic treatment?

- Electronic packaging and semiconductor manipulation cannot be exposed to heavy deformation
- Quantum computers with quantum bits function at temperatures near zero Kelvin[2]
- How do cryogenic environments affect the microstructure and properties of materials, specifically for application in future computing systems?

# Materials

1. Bulk Copper
   - ❖ Diamond cut from copper ring
2. Microchip
   - ❖ Copper and tin interconnect



# Preparation and experiment

- Tube Furnace
  - ❖ Annealed bulk copper at 750 $^{o}$C for two hours
  - ❖ Re-annealed after quench at 100 $^{o}$C for three minutes

- Hitachi IM4000 Plus
  - ❖ Milled in two consecutive 45-minute sessions

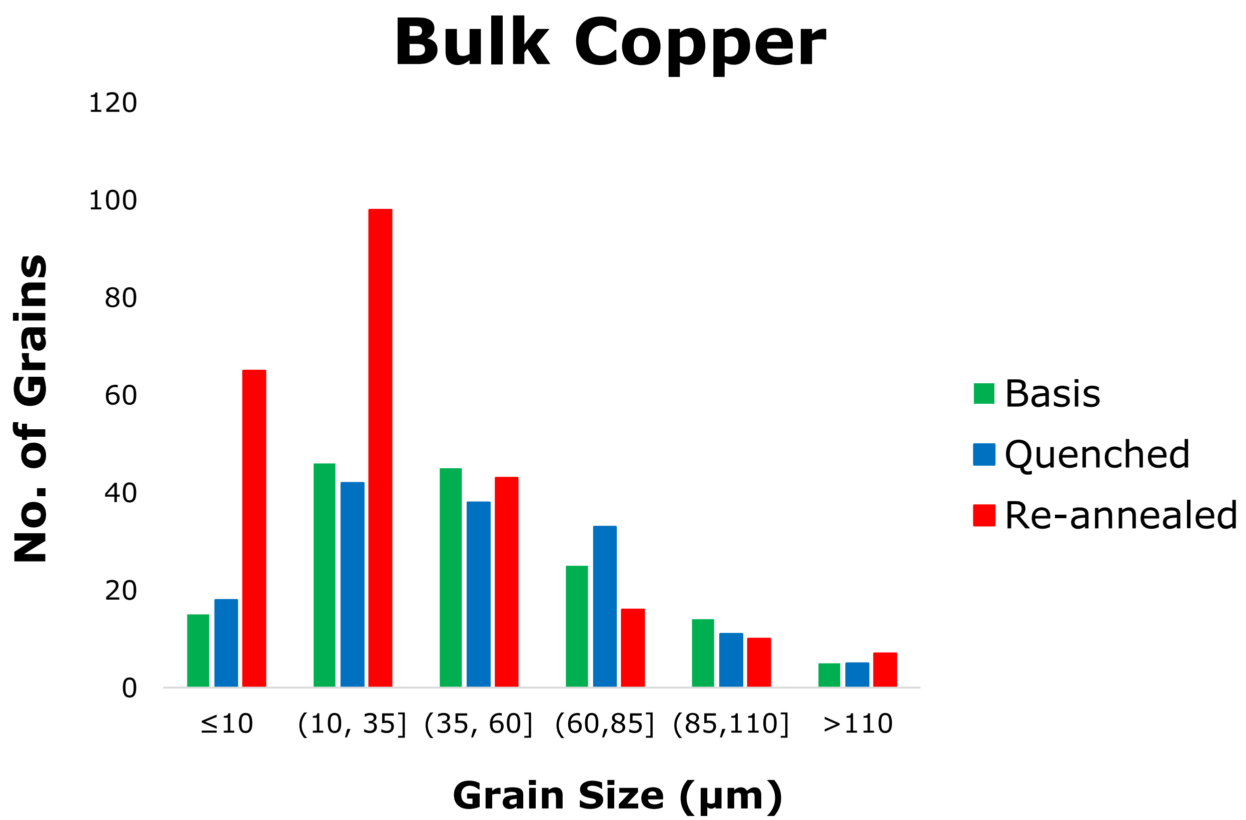

- Cryo-Quench
  - ❖ Liquid Nitrogen (-193oC)
  - ❖ Reached steady-state temperature in cryo bath and then dunked in distilled water bath


Liquid Nitrogen Bath
Distilled Water Bath
FEI
250 mL
KIMAX®
KIMBLE
200
150
100
50

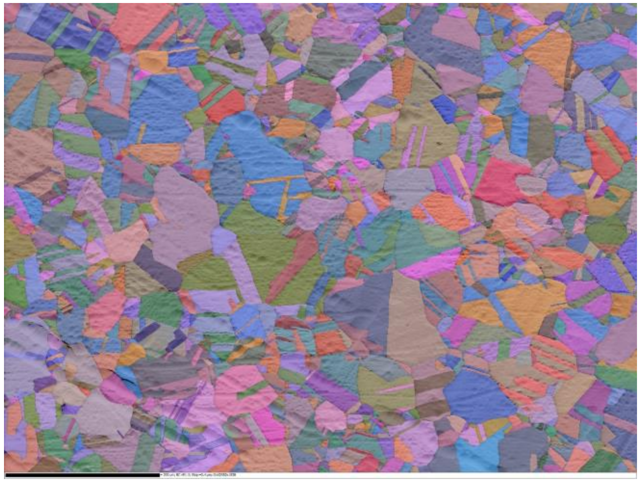
# Results

| | No. of Grains | Average Grain Size (µm) |
|---|---|---|
| Bulk Copper Basis | 150 | 46.377 |
| Bulk Copper Quenched | 147 | 46.455 |
| Bulk Copper Re-annealed | 239 | 31.384 |
| Cu Microchip Basis | 5104 | 0.740 |
| Cu Microchip Quenched | 3840 | 0.854 |
| Sn Microchip Basis | 148 | 2.893 |
| Sn Microchip Quenched | 245 | 2.093 |

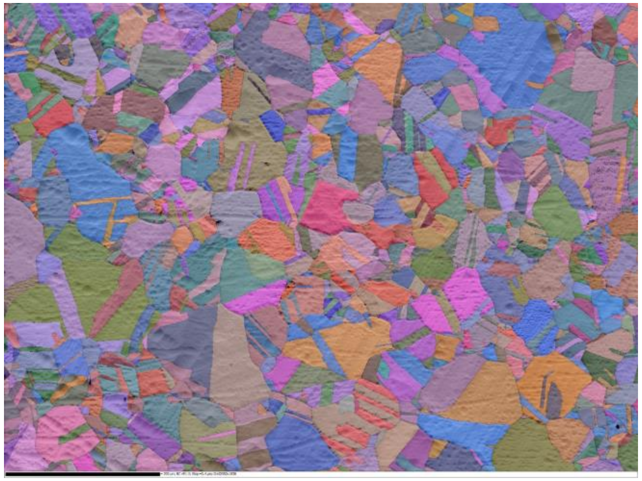
# Average grain size in bulk copper decreased by 32% after re-annealing

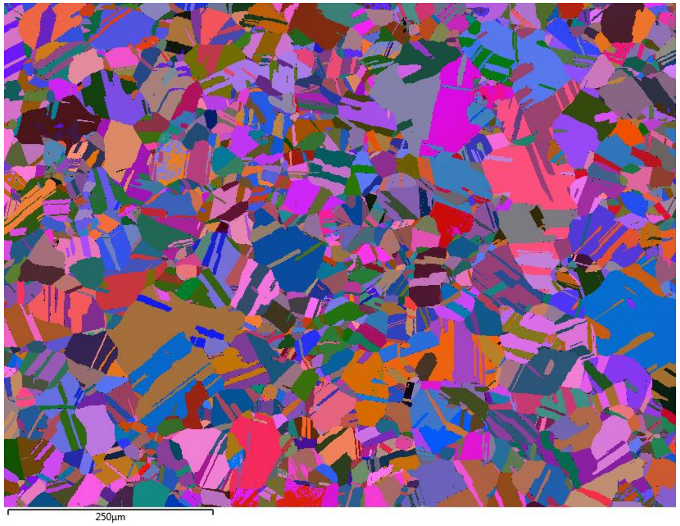


750°C/2h



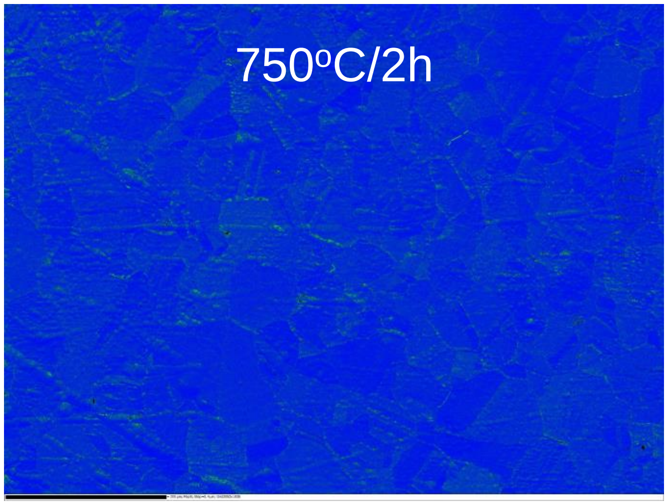
750°C/2h + -193°C/sec

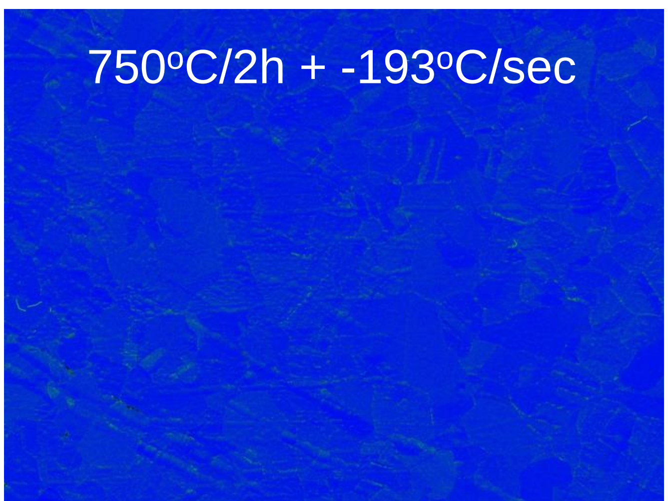


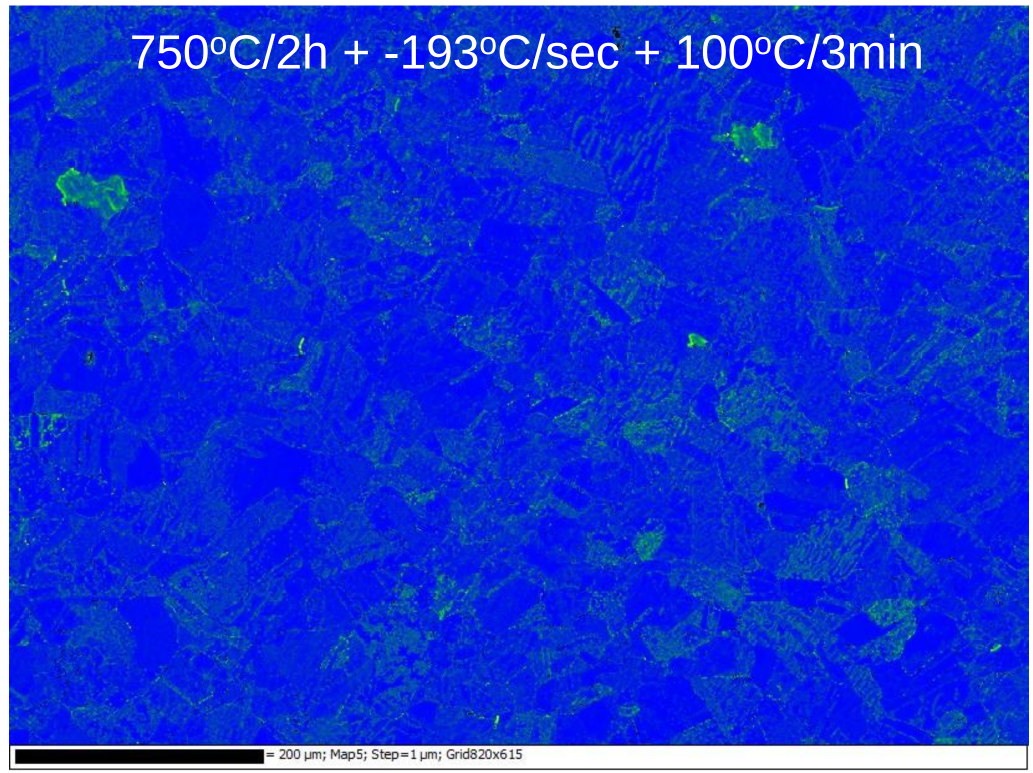



750°C/2h + -193°C/sec + 100°C/3min

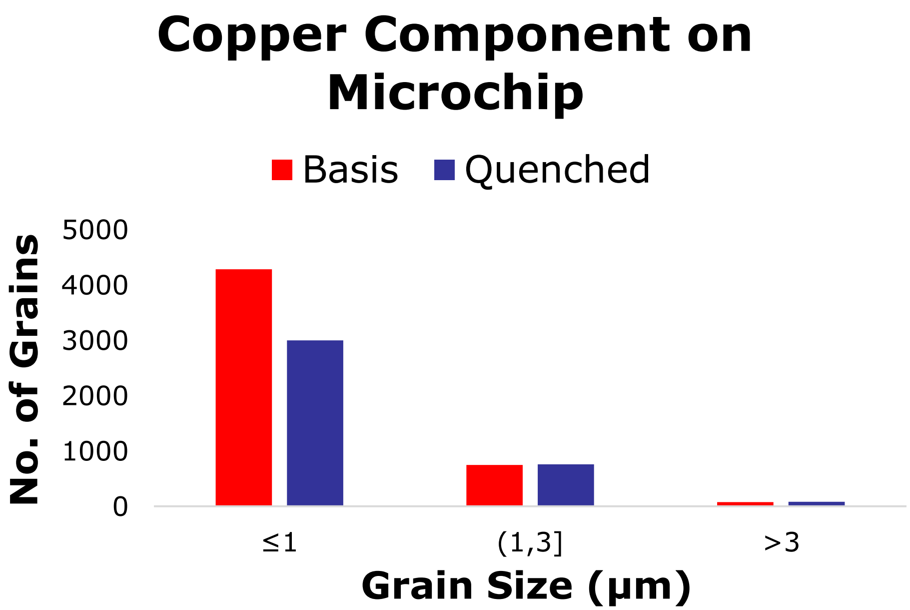
# Local misorientation maps indicate sites of deformation

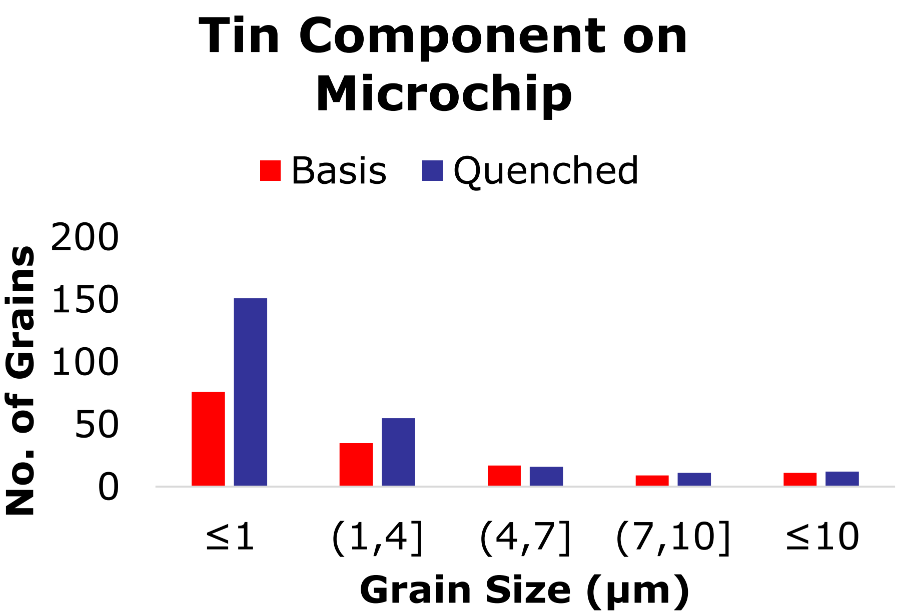


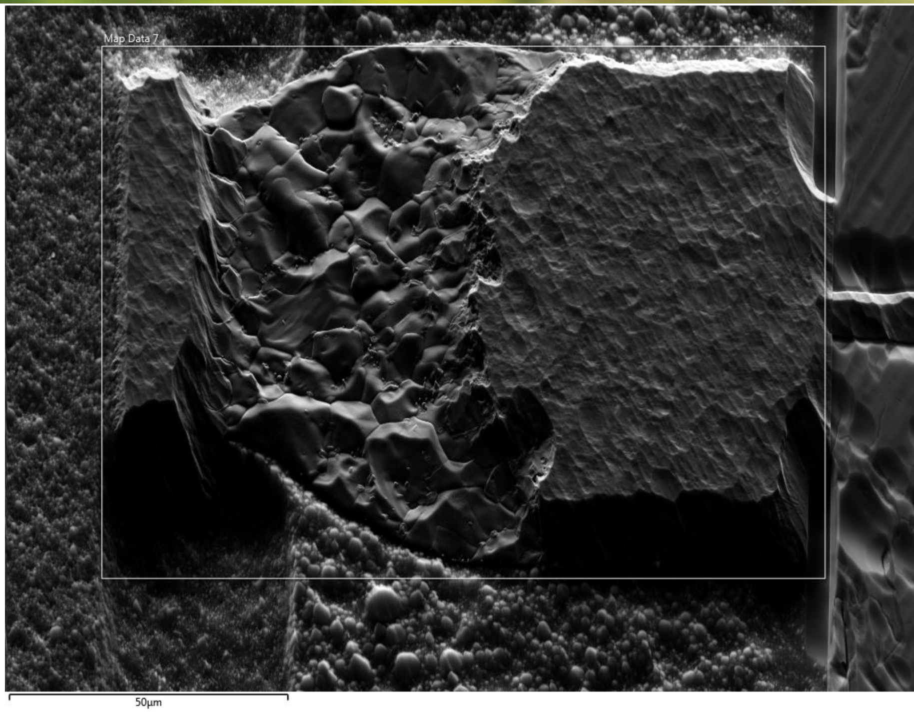



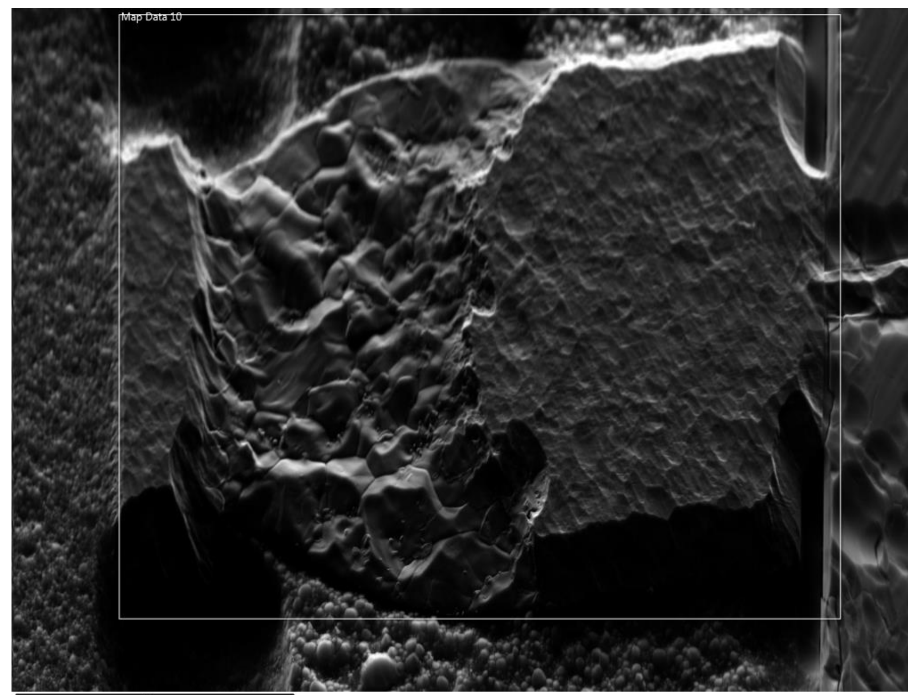
# Microchip before and after cryo-quench





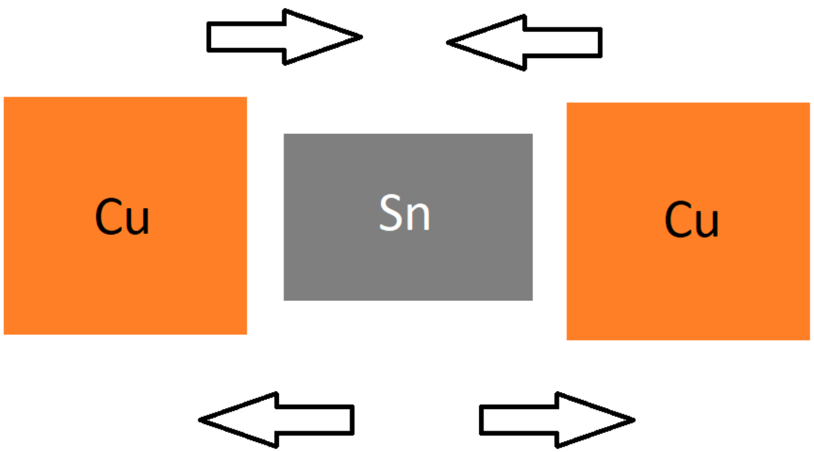

Forescatter diode (FSD) image of computer chip basis



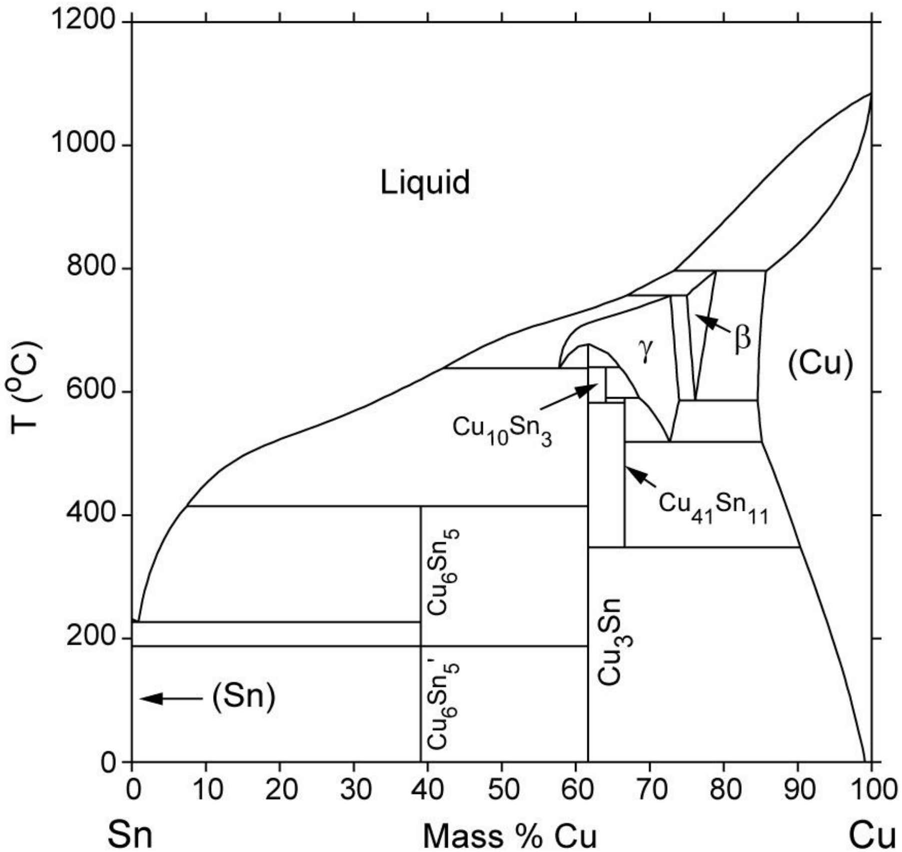

FSD image of computer chip site after quenching

# Tin has a greater thermal expansion coefficient than copper

Cryo-Quench induced tensile stress



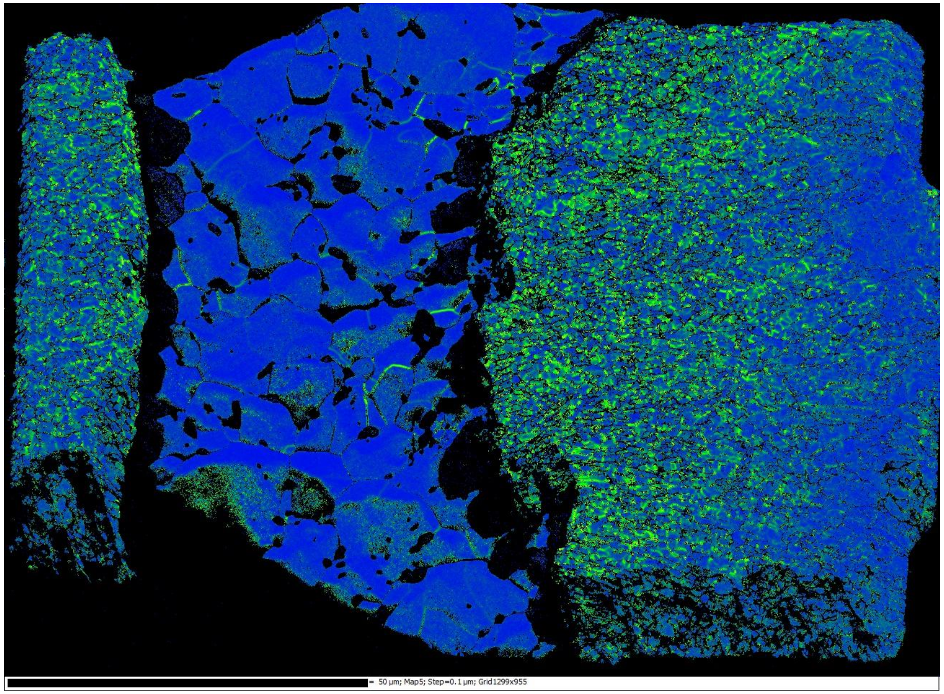

Water Bath induced compressive stress



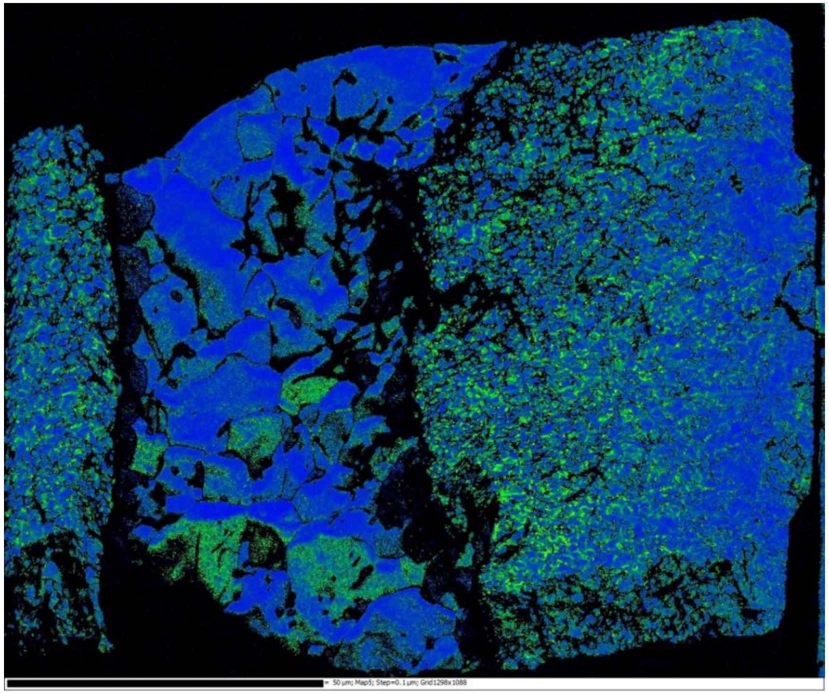

J.-H. Shim, C.-S. Oh, B.-J. Lee and D.N. Lee, Z. “Phase Diagram and Computational Thermodhnamics” *Metallkde*

Phase diagram of Sn and Copper

# Deformation observed in tin may have led to average grain size reduction



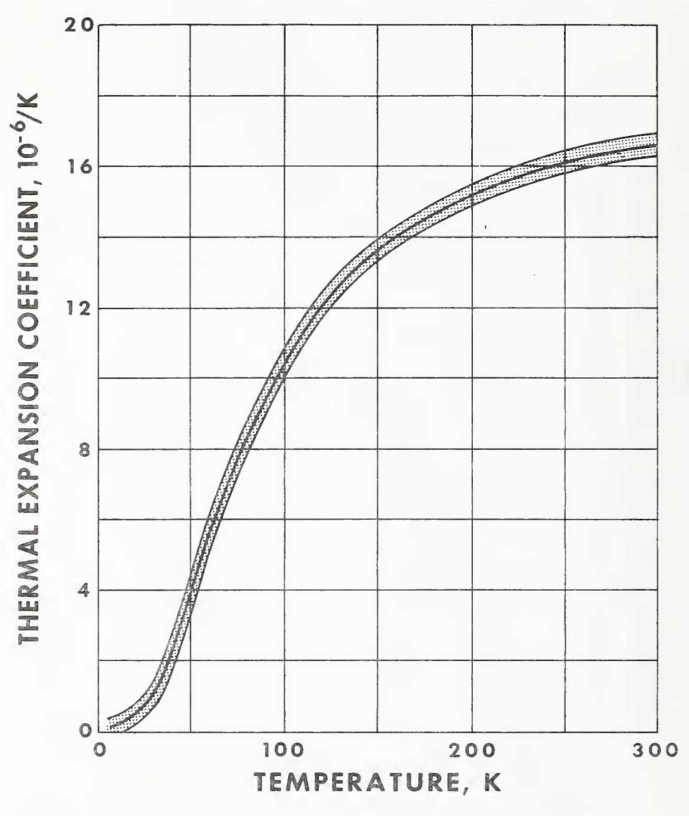

Local misorientation map of computer chip before quenching




Local misorientation map of computer chip after quenching

# To be determined…

- Insufficient driving force
- Re-annealing may release stress and cause recrystallization
- Residue stress analysis
  - X-ray diffraction
- Thermal analysis




N. J. Simon, E. S. Drexler, and R. P. Reed "Properties of Copper and Copper Alloys at Cryogenic Temperatures." *International Copper Association, Ltd,*

# Summary

- Bulk Copper
  - ❖ Avg. grain size increased by <1%
  - ❖ After re-annealing avg. grain size decreased by 32%

- Microchip
  - ❖ Cu avg. grain size increased by 15%
  - ❖ Sn avg. grain size decreased by 27%

# Acknowledgements

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# References

1. Lei Lu et al., “Ultrahigh Strength and High Electrical Conductivity in Copper”
2. E. Charbon *et al*., "Cryo-CMOS for quantum computing," *2016 IEEE International Electron Devices Meeting (IEDM)*, Pages 13.5.1-13.5.4. (2016)
3. N. J. Simon, E. S. Drexler, and R. P. Reed “Properties of Copper and Copper Alloys at Cryogenic Temperatures.” *International Copper Association, Ltd,* Page 264 (1992)
4. J.-H. Shim, C.-S. Oh, B.-J. Lee and D.N. Lee, Z. “Phase Diagram and Computational Thermodhnamics” *Metallkde*. 87 (1996) 205-212