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Microstructure correlations and flux-trapping in SRF Nb- Role of initial microstructures

Shreyas Balachandran

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This work is a part of a collaboration between: FSU, MSU, and JLAB

Outline of this talk

Background: Great progress has been made in SRF Nb.

- Flux-trapping and subsequent incomplete expulsion during cool down of cavity through T_c (9.2 K) increases residual resistance of SRF cavity by 5-10 nΩ, is expected to be 1-2 nΩ [1].
- Came to the fore with the **possibility of high Q at gradients of 15-20 MV/m with N doping**.
- **Why is this an important issue?**
  - **High Q, High G push. Example: $Q_0 \geq 2.7x\ 10^{10}$… G, as high as possible.**

What have we done?

- Performed systematic studies on evolution of microstructure after deformation and subsequent heat treatment in RRR 170 Nb. Magneto-Optical- Imaging to relate microstructure to superconducting properties.
- Cross-sectional Microstructure studies of cavity half cell after deformation and heat treatment.
- Recrystallization curves of SRF Nb material from two different vendors with different deformation history.

1. D. Gonnella, S. Aderhold, A. Burrill,et.al. "INDUSTRIAL CAVITY PRODUCTION: LESSONS LEARNED TO PUSH THE BOUNDARIES OF NITROGEN-DOPING", Proceedings of SRF2019, Dresden, Germany

Key points of this talk

Metallurgical results reported here:

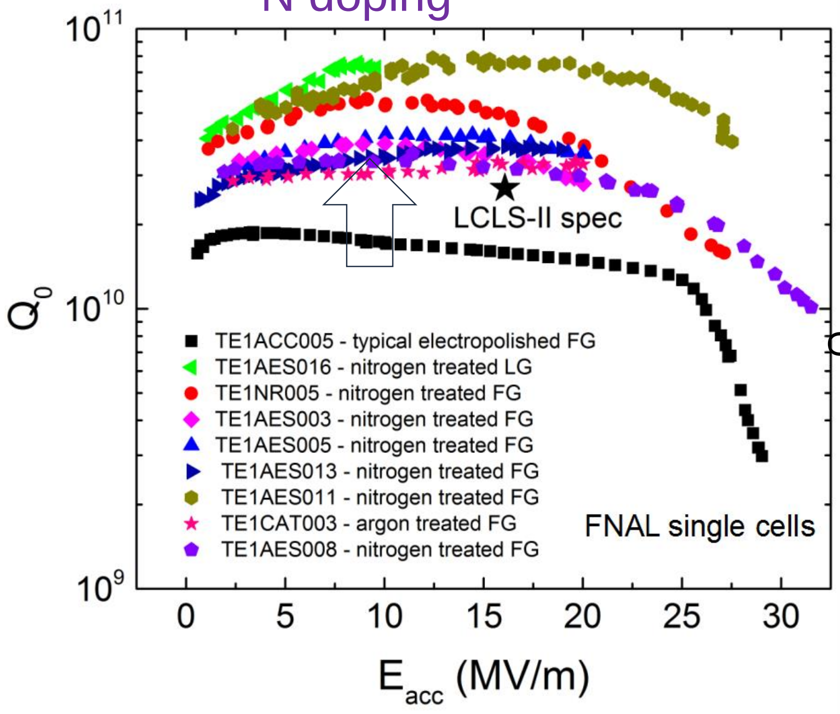
- Spatial variations in trapped flux depends on microstructure. Flux is trapped in regions where the grain size is fine (un-recrystallized) whereas flux was expelled selectively in regions where the grain size is ≥100 μm (recrystallized).
- Spatial variations in grain size occur in SRF Nb half cells after deformation and 800 °C/3 h heat treatment.
- Strain distribution in cavity structures are important to understand, as it drives recrystallization.

So what?

- Full recrystallization could be used to improve flux expulsion.
- Attention in upstream processing history is needed and important.

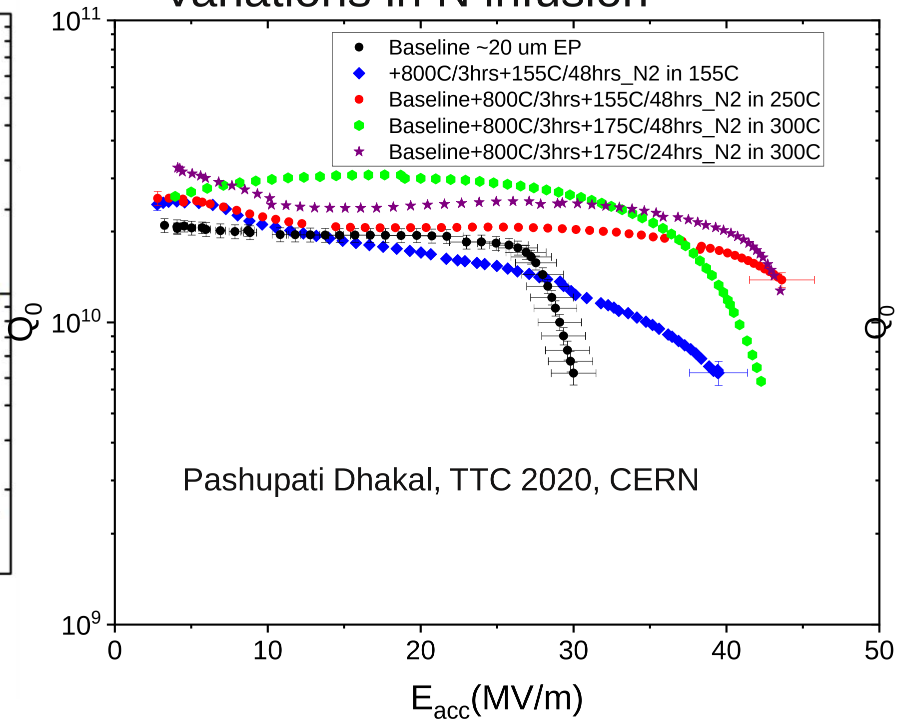
Background- Performance of SRF Nb cavities have been improving (Q, and E_{acc}).

N doping

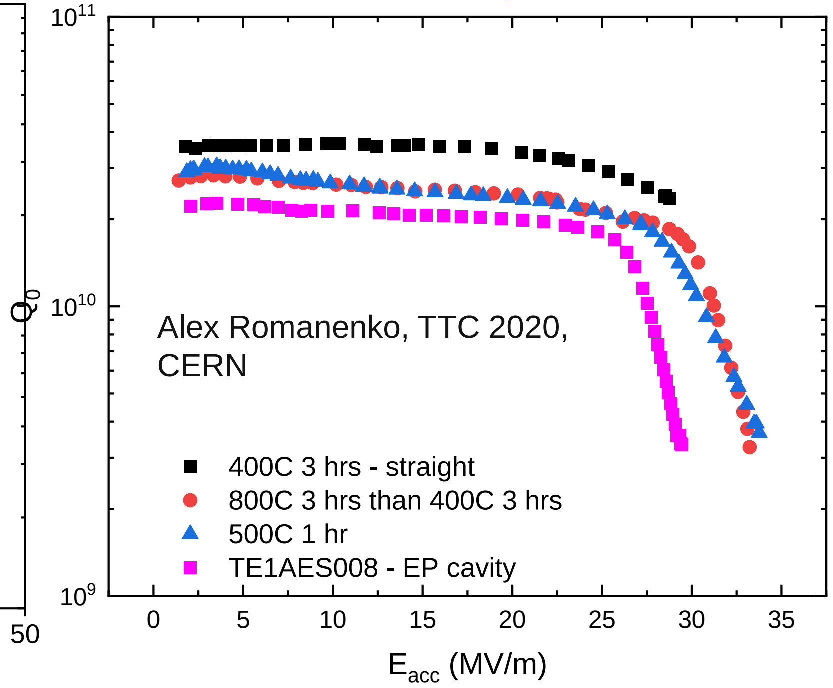


A Grassellino , A Romanenko, *et al.*, Supercond. Sci. Technol. 26 (2013)

Variations in N infusion



Modified baking treatments



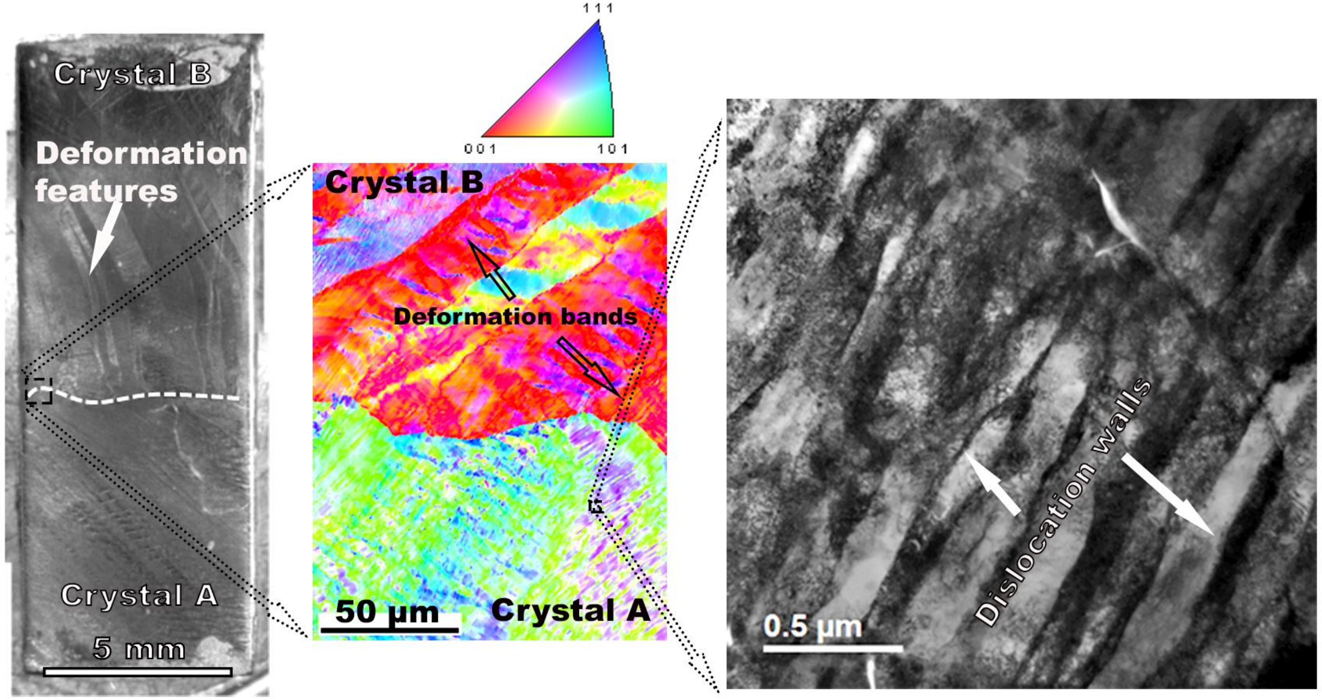
- Clean room, electro-polishing improvements.
- Material Science on cavity cut-outs and coupons have started to provide understanding of surface and bulk Nb.

SRF Nb cavities can reach unprecedented quench fields.

Part I: Direct evidence of microstructure and flux trapping

Deformation of RRR 170 Nb bi-crystal by simple shear (corresponds to 67% Rolling deformation)

- Deformation complexity in microstructure at different scales.

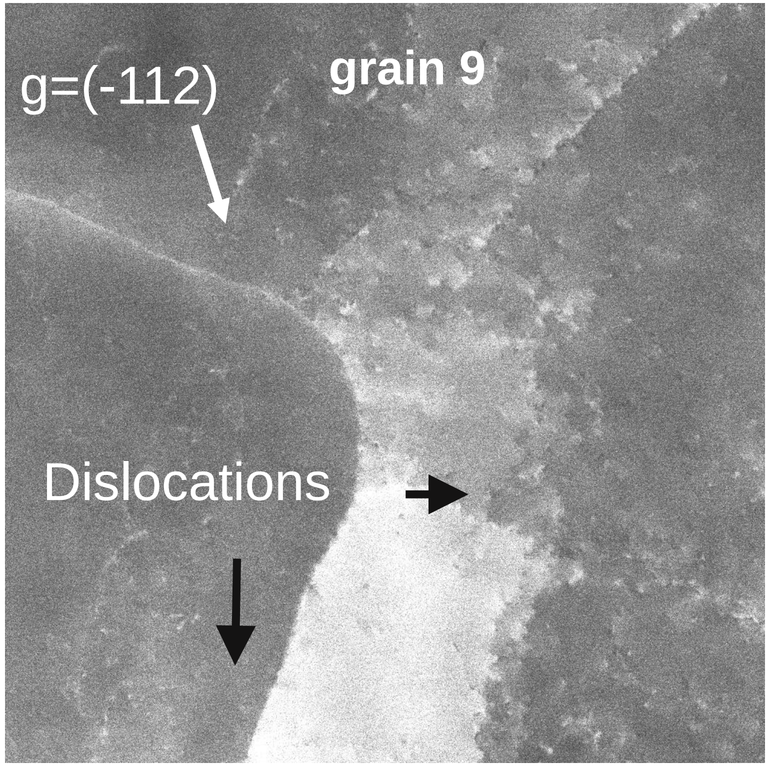


a — Macroscale (mm's)

b — Microscale (10's of µm)

c — Sub-micron scale (10-100's of nm)

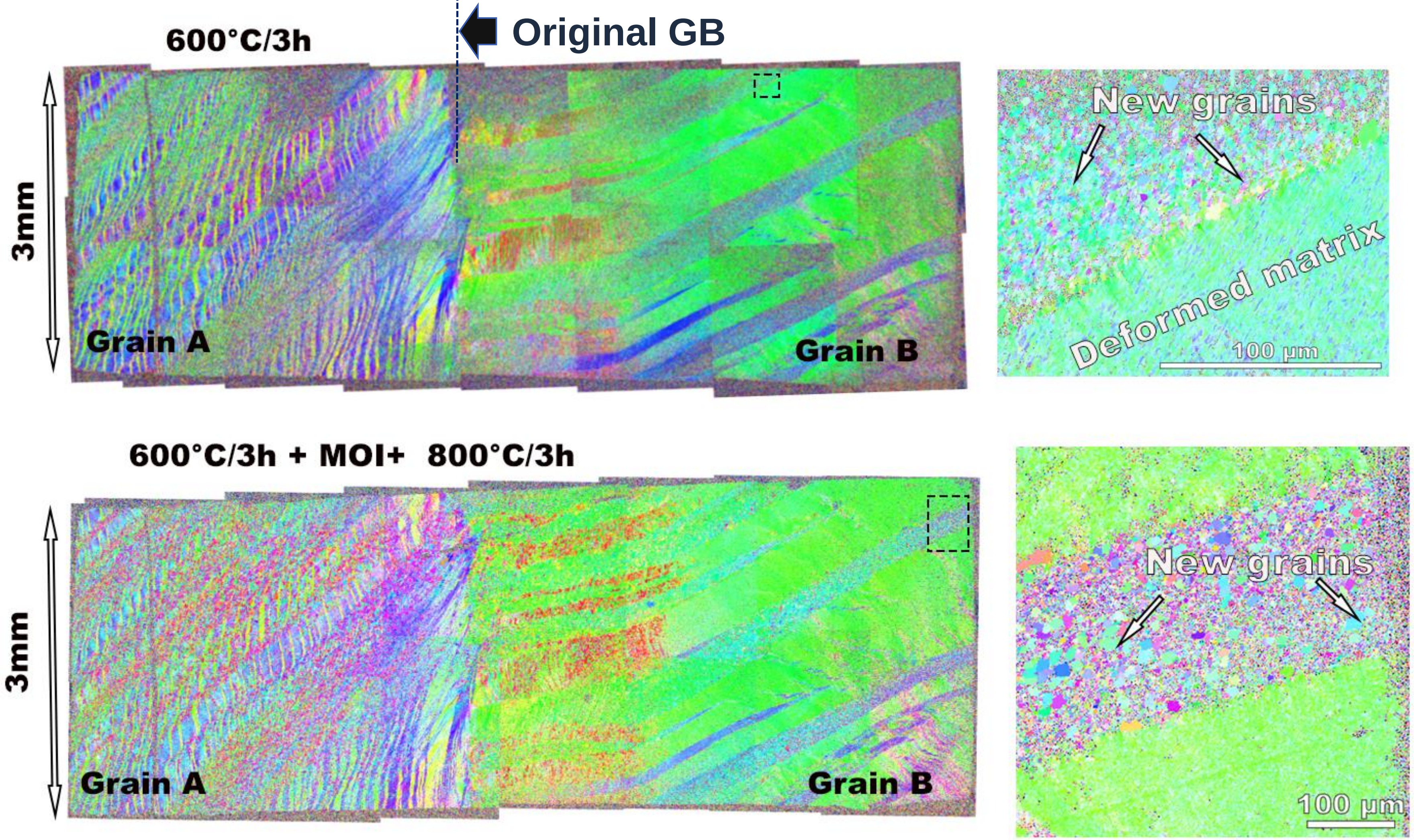
Electron Channeling Contrast Imaging (ECCI)



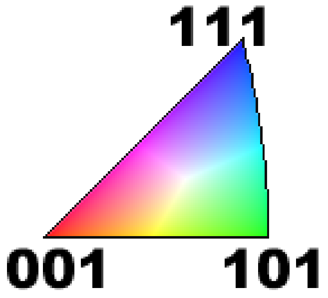
Mingmin Wang (PhD advisor: Tom Bieler) recent MSU graduate (soon joining SLAC)

MICHIGAN STATE UNIVERSITY

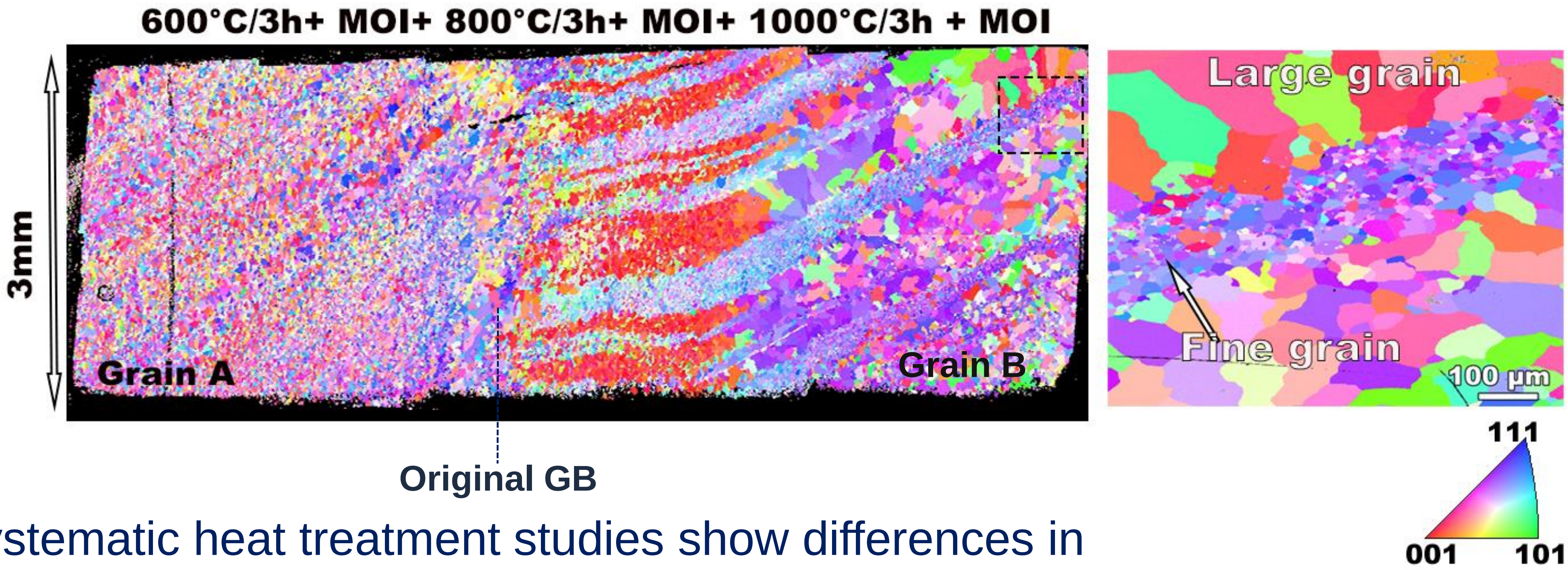
Systematic heat treatment studies indicate complex recovery and recrystallization process - Depends on grain orientation



- Thickness of sample is ~300 µm



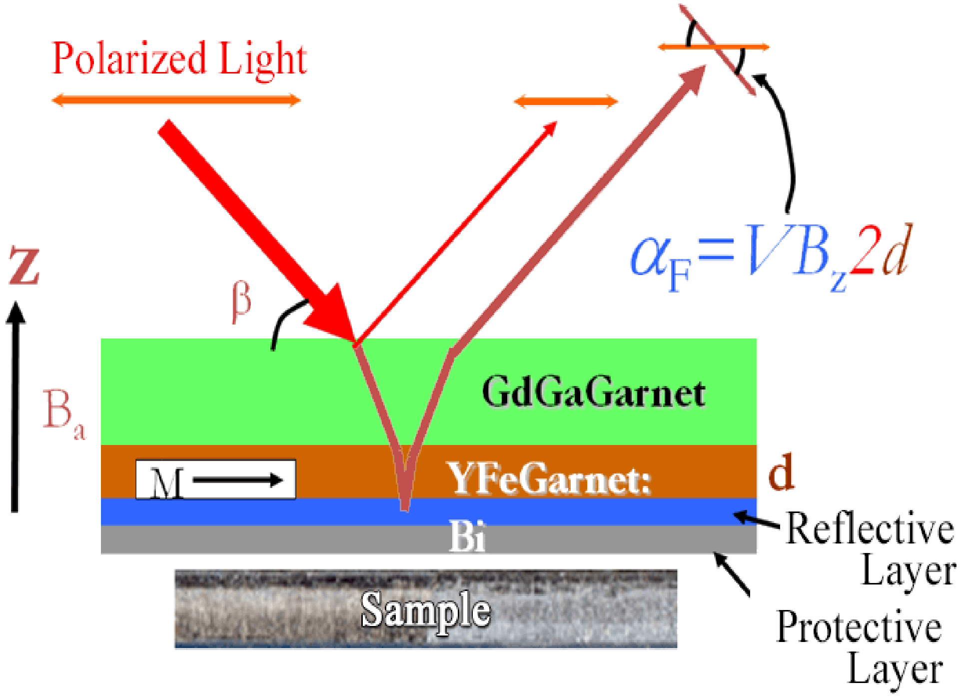
After 1000°C/3h heat treatment recovery and recrystallization processes occurred throughout the deformed material.



- Systematic heat treatment studies show differences in microstructure evolution in deformed Nb.
- After a 1000°C/3h heat treatment, a bi-modal microstructure appeared in Nb.

MO imaging- Qualitative understanding of flux behavior in SRF Nb

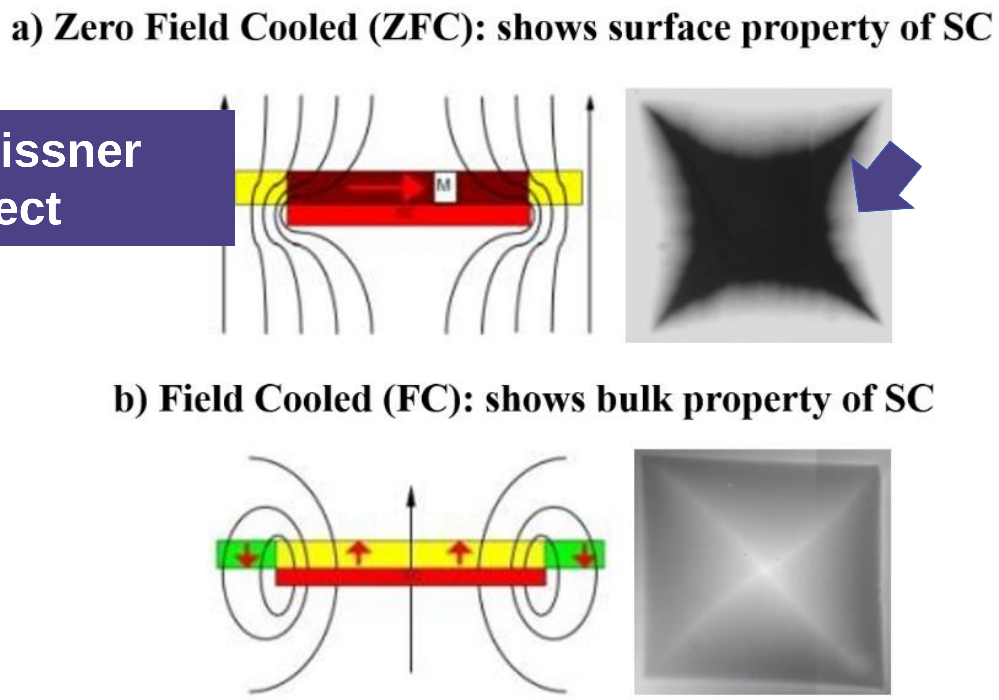
All the MOI work was done by Anatolli Polyanskii



MOI Schematic and principle

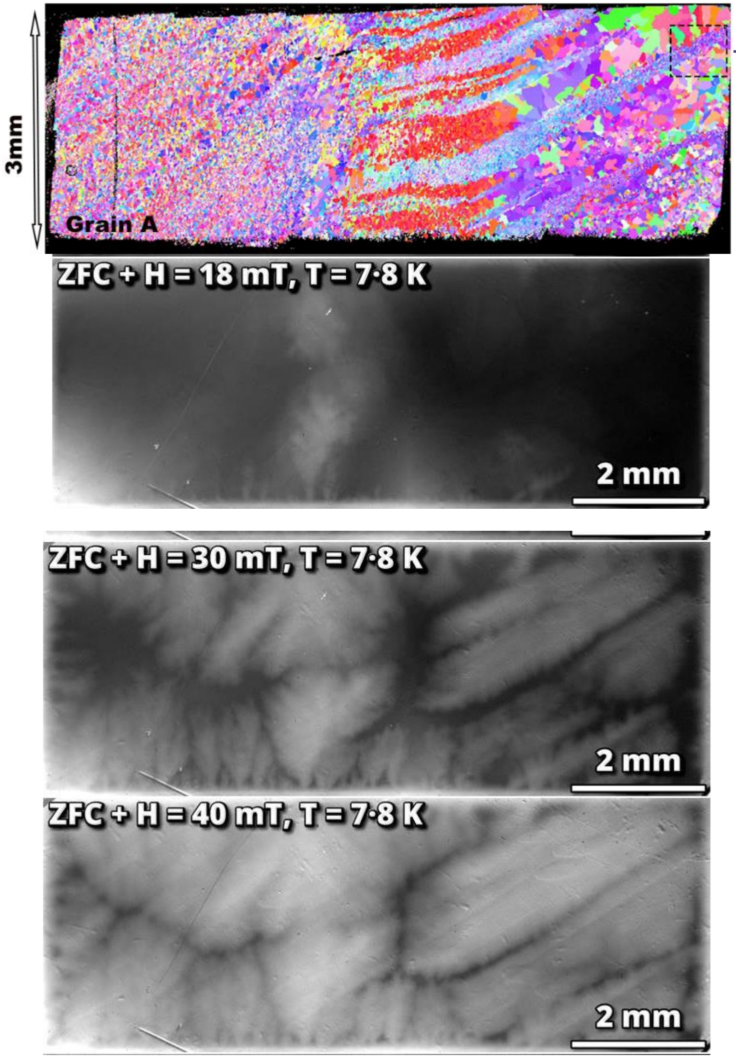
- Allows direct Imaging of Bz in plane above superconductor
- Double Faraday effect occurs in reflective mode using Bi-doped YFe indicator film with in-plane magnetization

Meissner effect



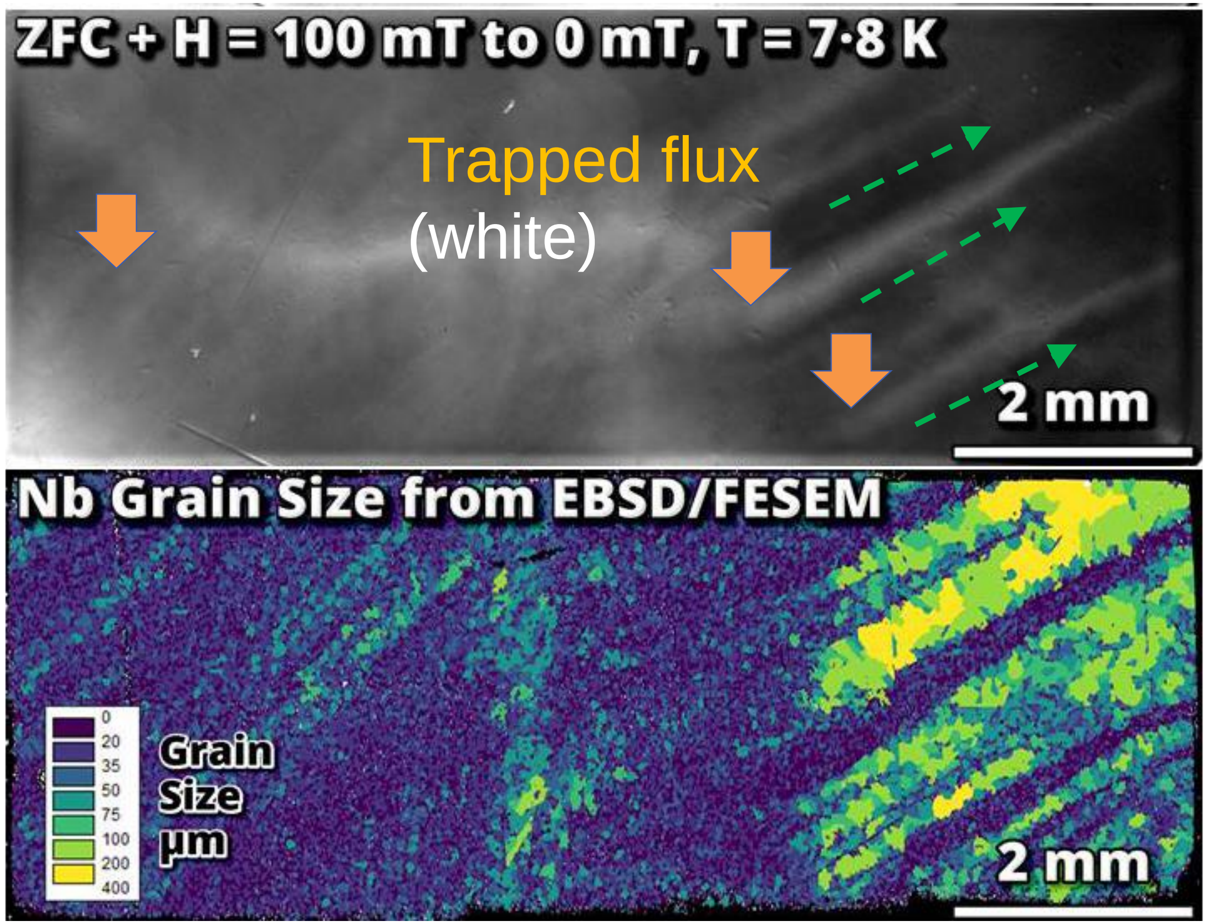
Cooled in the absence of external fields to below T_c, at $T < T_c$ visualized in an external magnetic field.
FC- Sample cooled below Tc in the presence of a magnetic field, and visualized after removing the external field

MOI indicates flux penetration and trapping in Nb varies spatially
Direct evidence flux of trapped flux in fine grain size regions



H_{c1}(7.8 K)= 13 mT

Increasing field



Summary

- Grain orientations are important because deformation features differ depending on orientation.

- Recovery and recrystallization during heat treatment depends on the details of the worked Nb state.
  - Influences grain size after heat treatment, and also flux trapping.

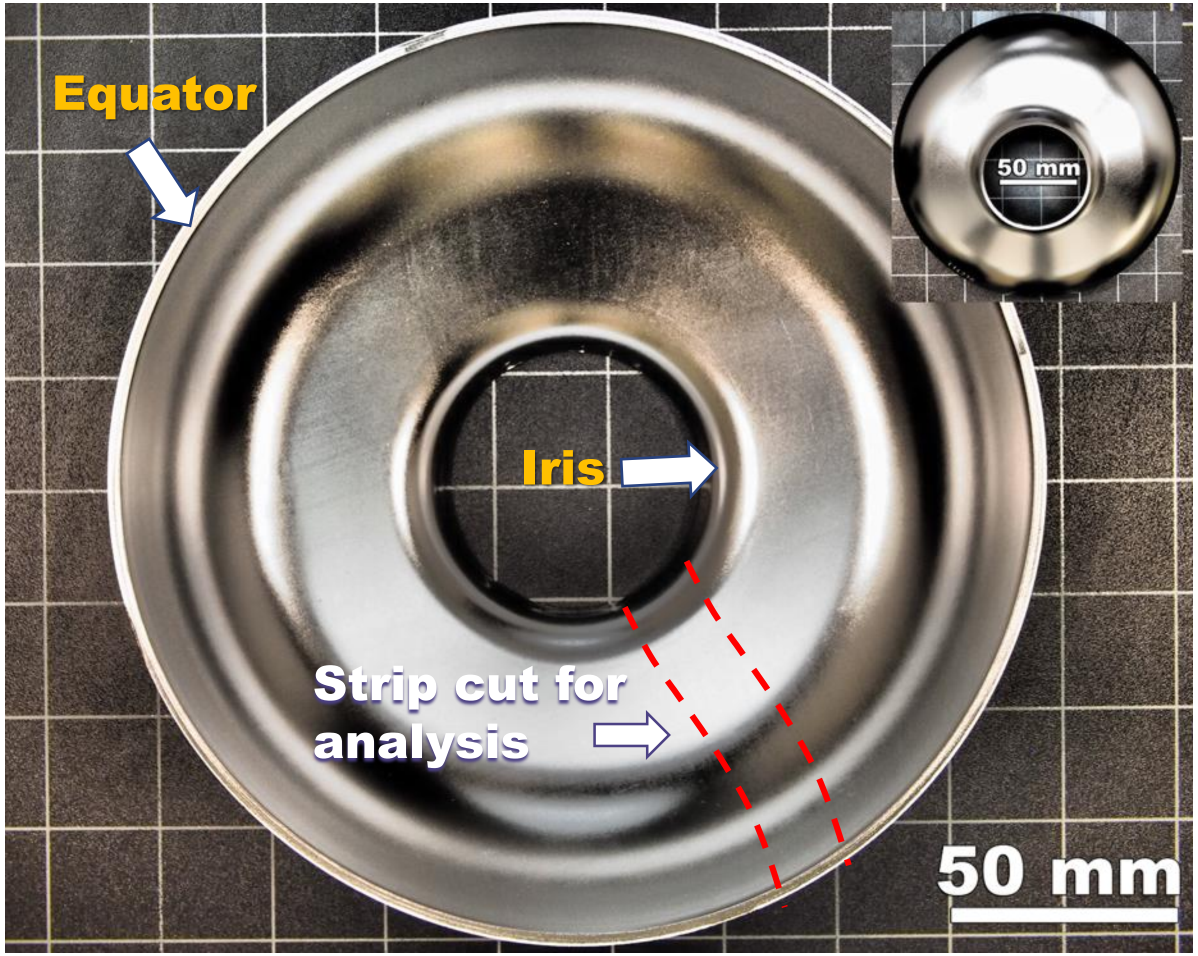
- **Flux trapping can be reduced by selecting heat treatment conditions so that recrystallized grain size exceeds 100 μm.**
  - **But these conditions will be sensitive to details of deformed microstructure**

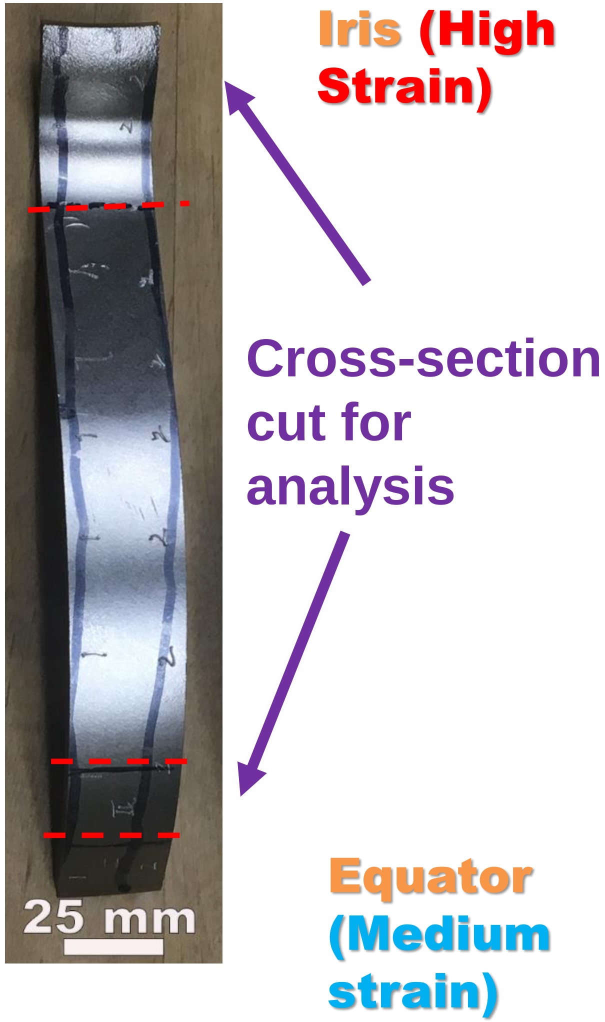
Part II: Strain, Recovery, and Recrystallization in SRF Nb half cells

Collaboration with Pashupati Dhakal (JLAB)

Jefferson Lab

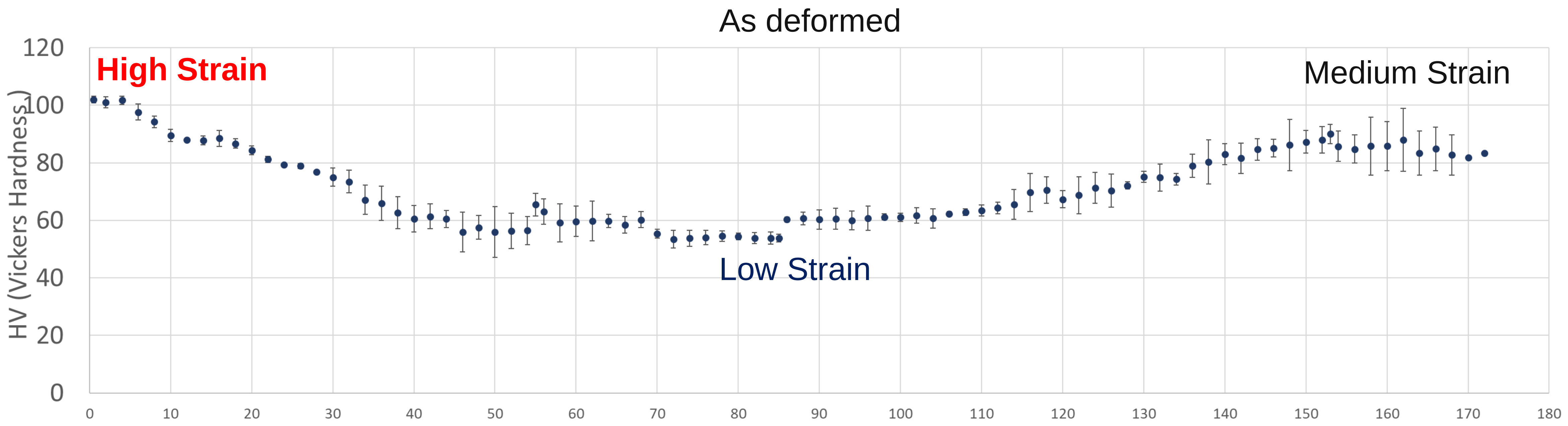
Description of procedure - Cavity Half cell fabrication and sample details (LCLS-II sheet material)



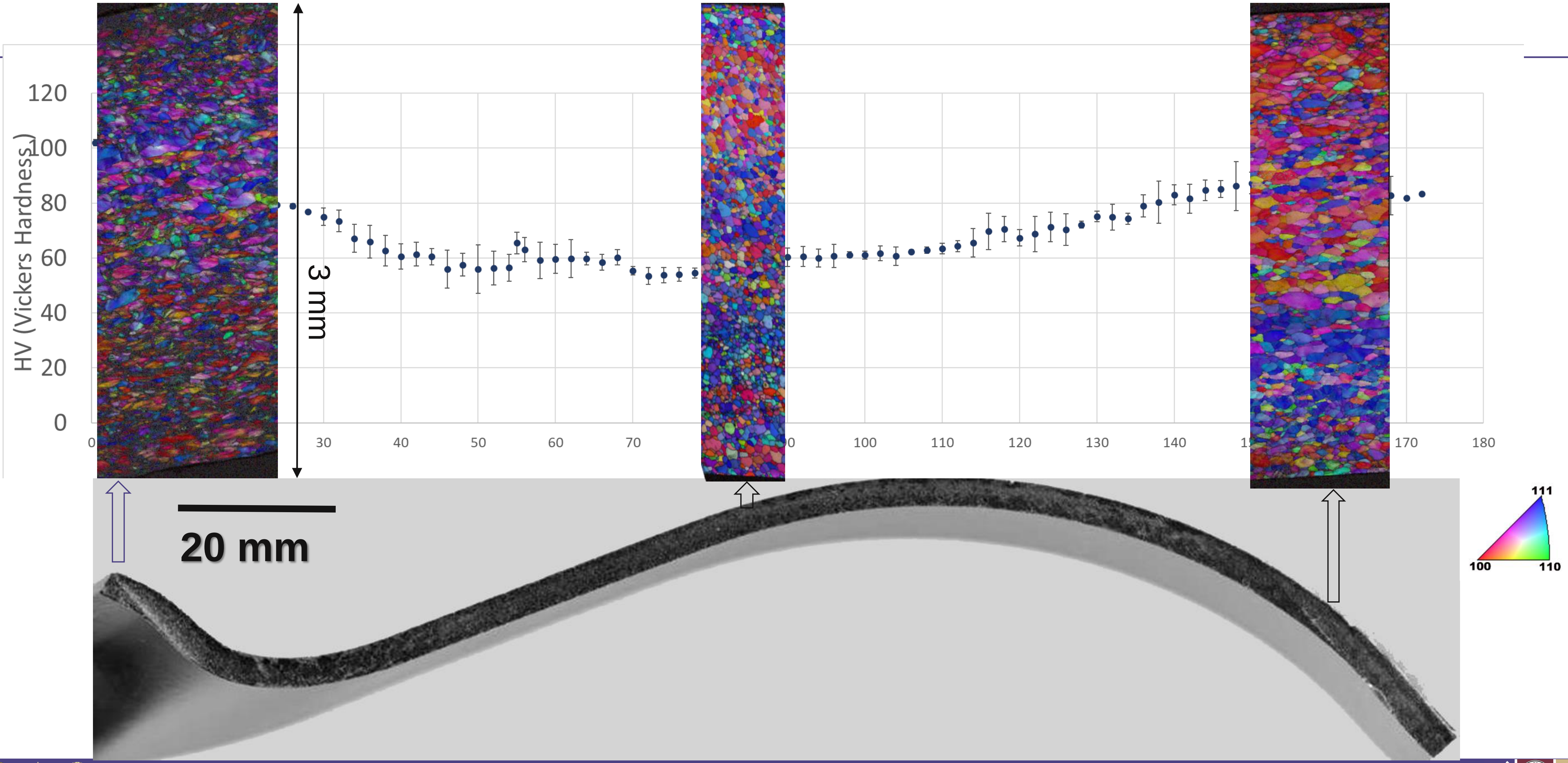


- The material has differing strain paths from iris to equator.
- According to Finite Element simulations, bi-axial strain varies from a maximum 15% at iris to less than 5% at the equator.
- Microstructure was assessed at high strain and low strain regions in as-deformed and **after 800°C/3h** condition

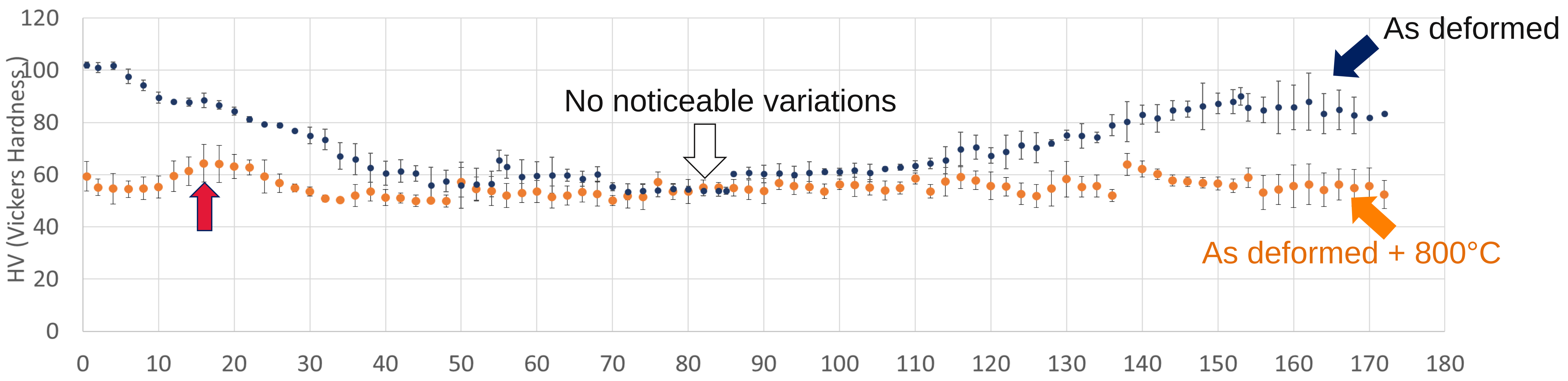
Result- Cavity strain is dependent on location, we do not still understand the strain path (microstructure based simulations needed)



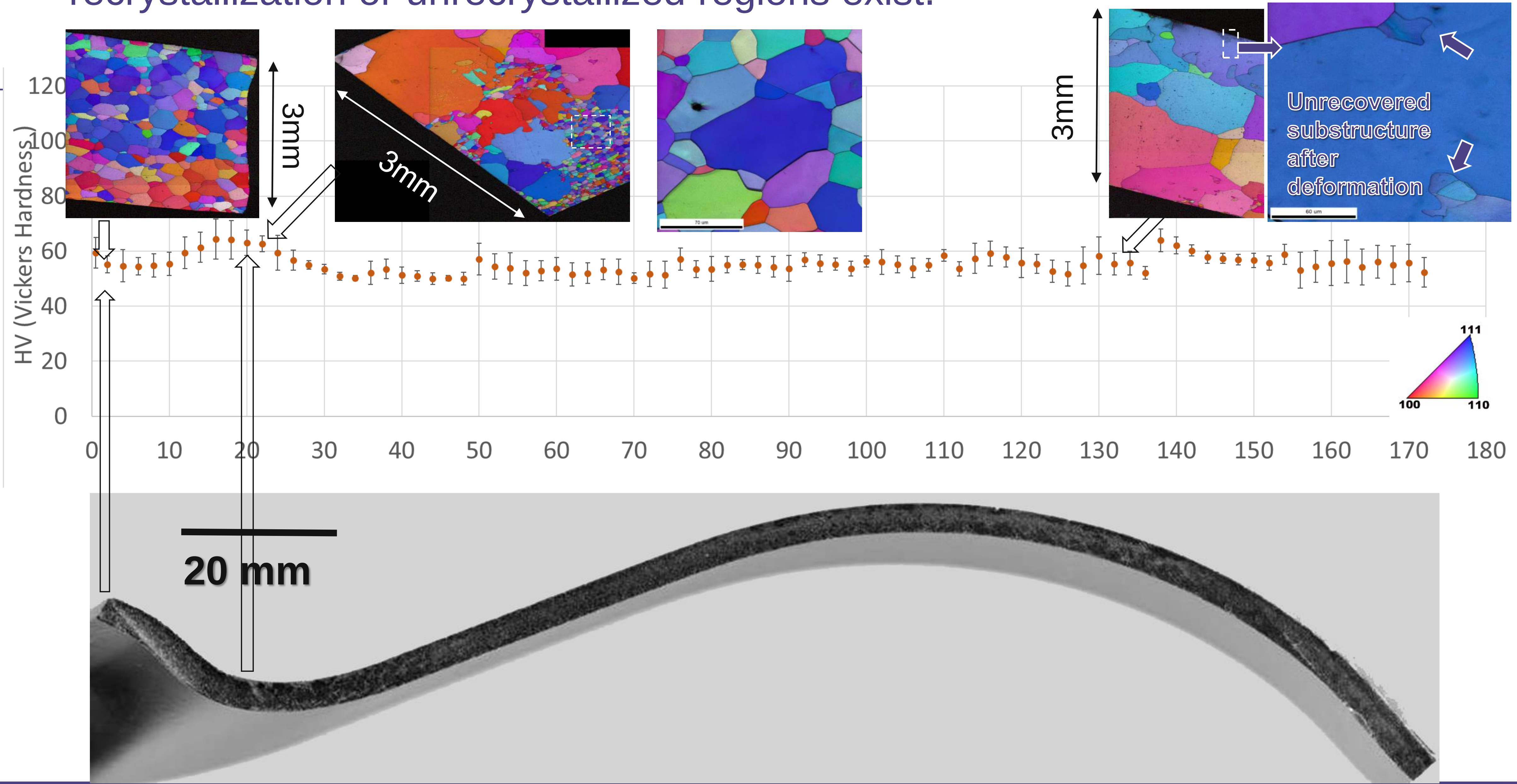
Result- Cavity microstructure is dependent on location



Result- Cavity recovers and anneals differently depending on location after 800°C/3h.



Cavity anneals and recovers differently at different locations after 800°C/3h- Partial recrystallization or unrecrystallized regions exist.

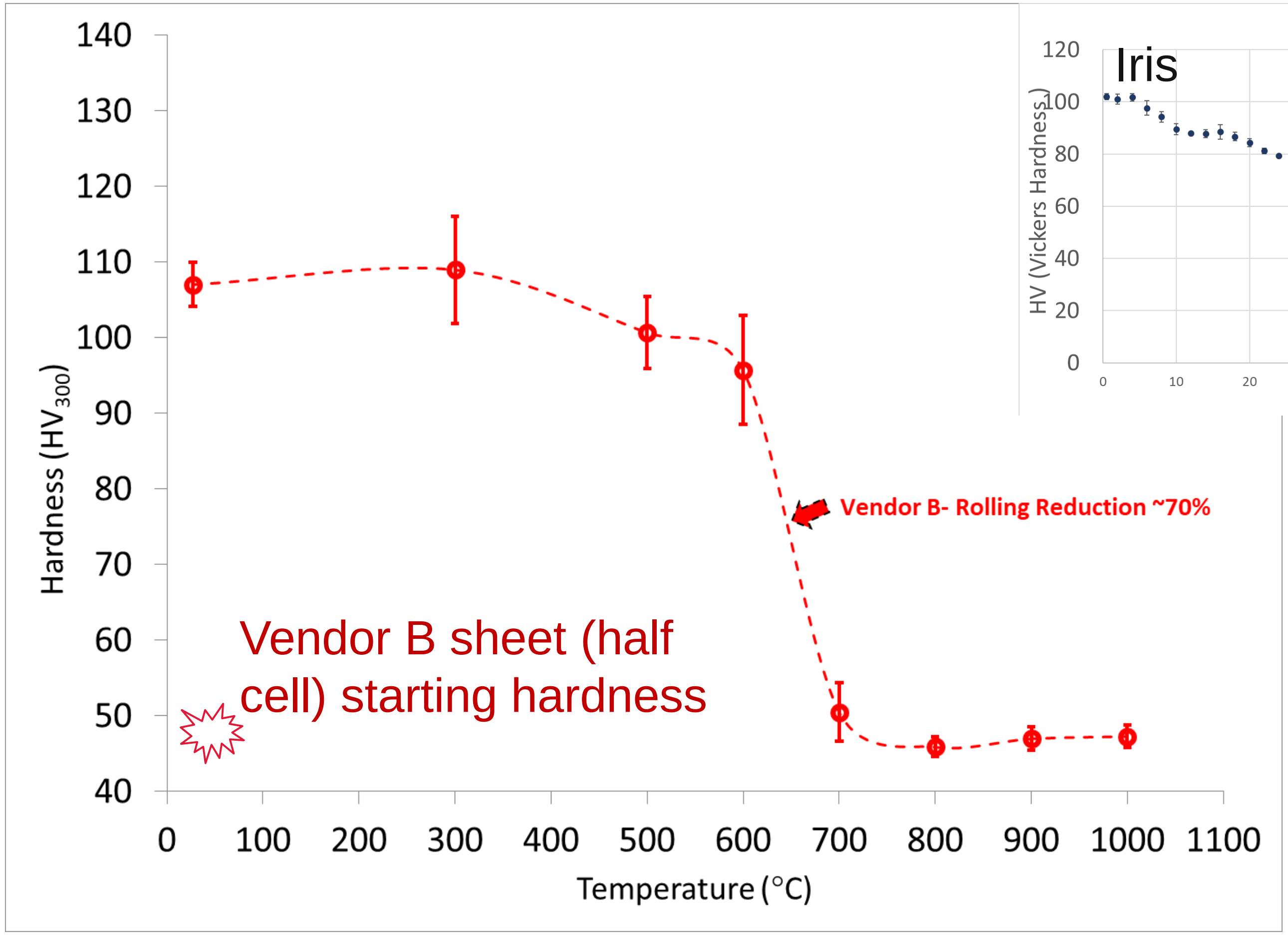


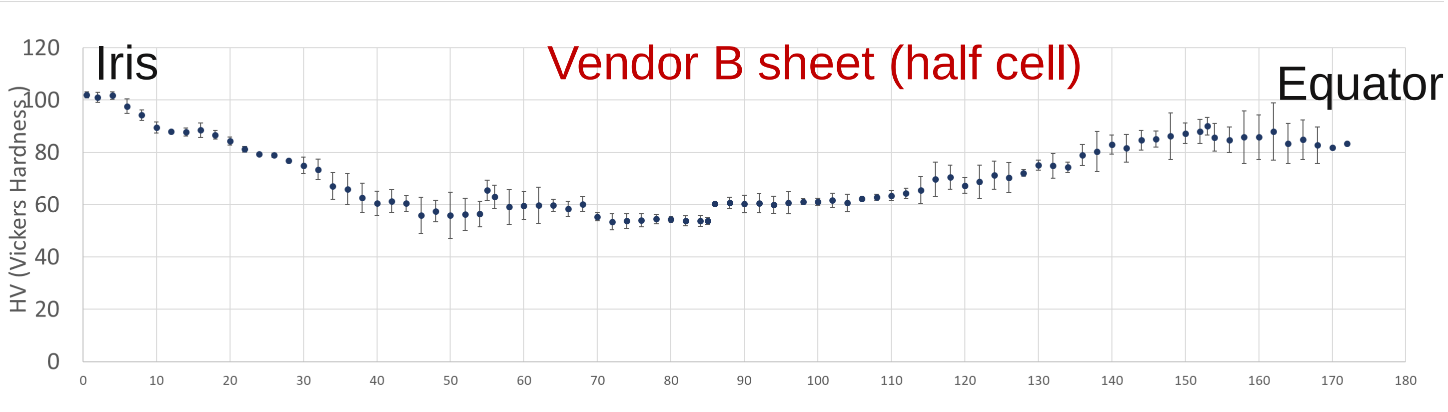
Summary

- Deformation strain varies with location in cavity half cell, and so does recovery and recrystallization.
  - Highly strained regions around the iris have equiaxed recrystallized grains
  - Regions about 10mm from the equator have large grains with retained recovered deformation substructure.
- 800°C/3h heat treatment does not fully anneal equator regions.
  - Higher temperature heat treatments could improve the situation.
  - GB is the main strengthening mechanism in Nb. A larger the grain size lowers is the material strength
  - However, grain size strengthening increases exponentially with decreasing grain size, so there is not much difference between moderate grain growth and much grain growth.
  - How much grain growth is tolerable?

Part III: Recrystallization of SRF Nb and its dependence on the initial strain state (In Progress)

Recrystallization of 70% cold rolled sheet from Vendor B shows that at 800°C provides complete recrystallization.

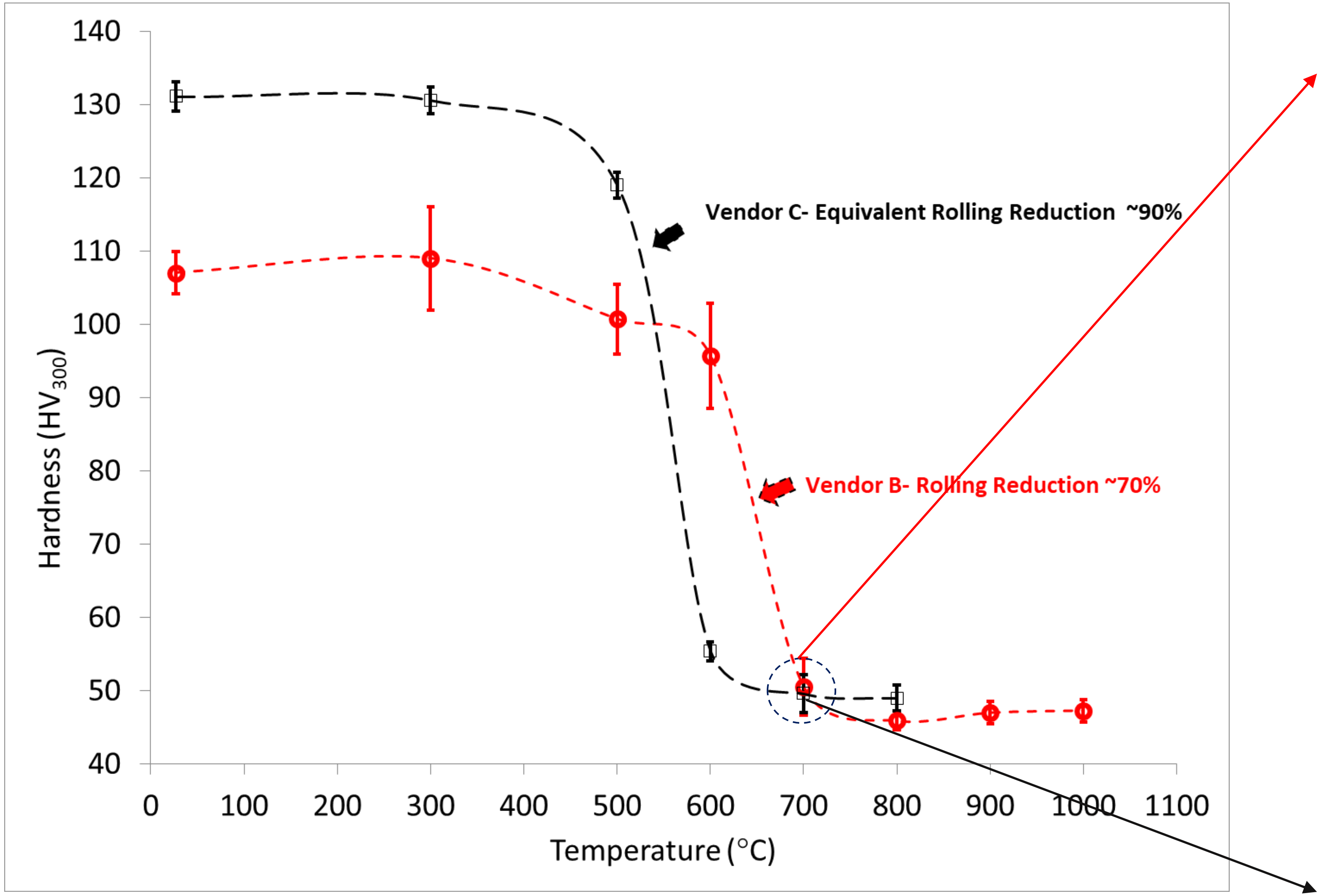


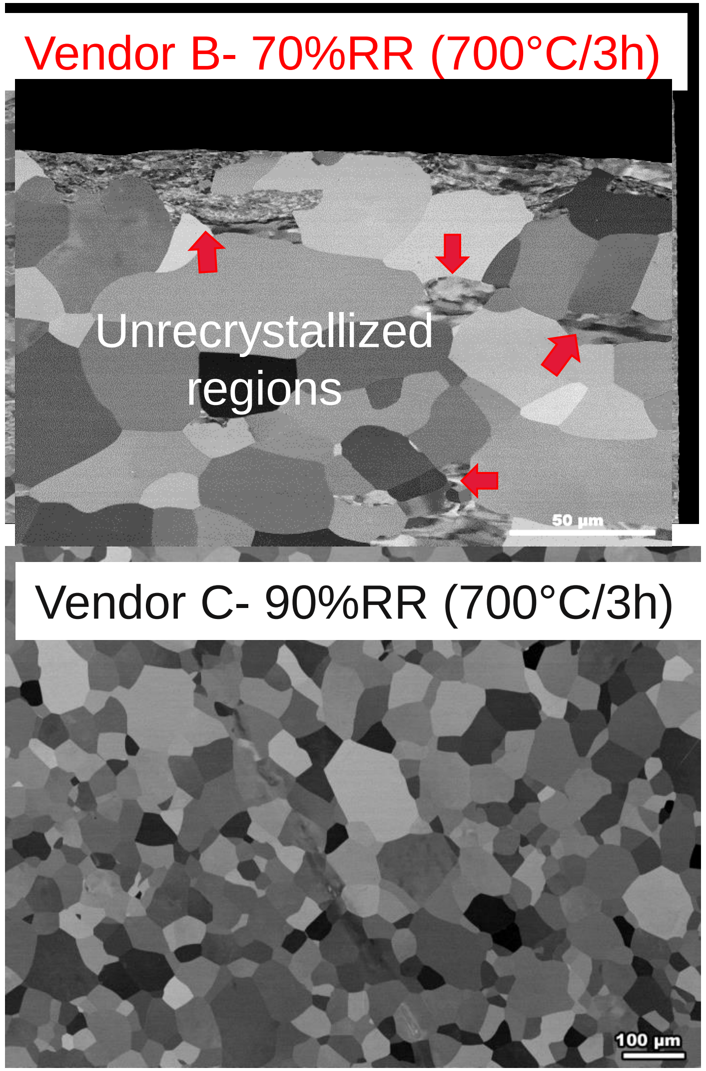


As rolled sheet from Vendor B after 3h heat treatments.

- The iris region likely receives a similar strain as 70% rolling reduction.
- This result is consistent with full recrystallization at 800°C in the iris region.
- **Different percent cold rolling on Nb sheets from same vendor could be used to evaluate recrystallization behavior and small scale superconducting sample measurements.**

Recrystallization of SRF Nb depends on the initial strain state- The same 700º heat treatment gives incomplete recrystallization with 70% reduction, but complete recrystallization with 90% reduction





Summary and Conclusions

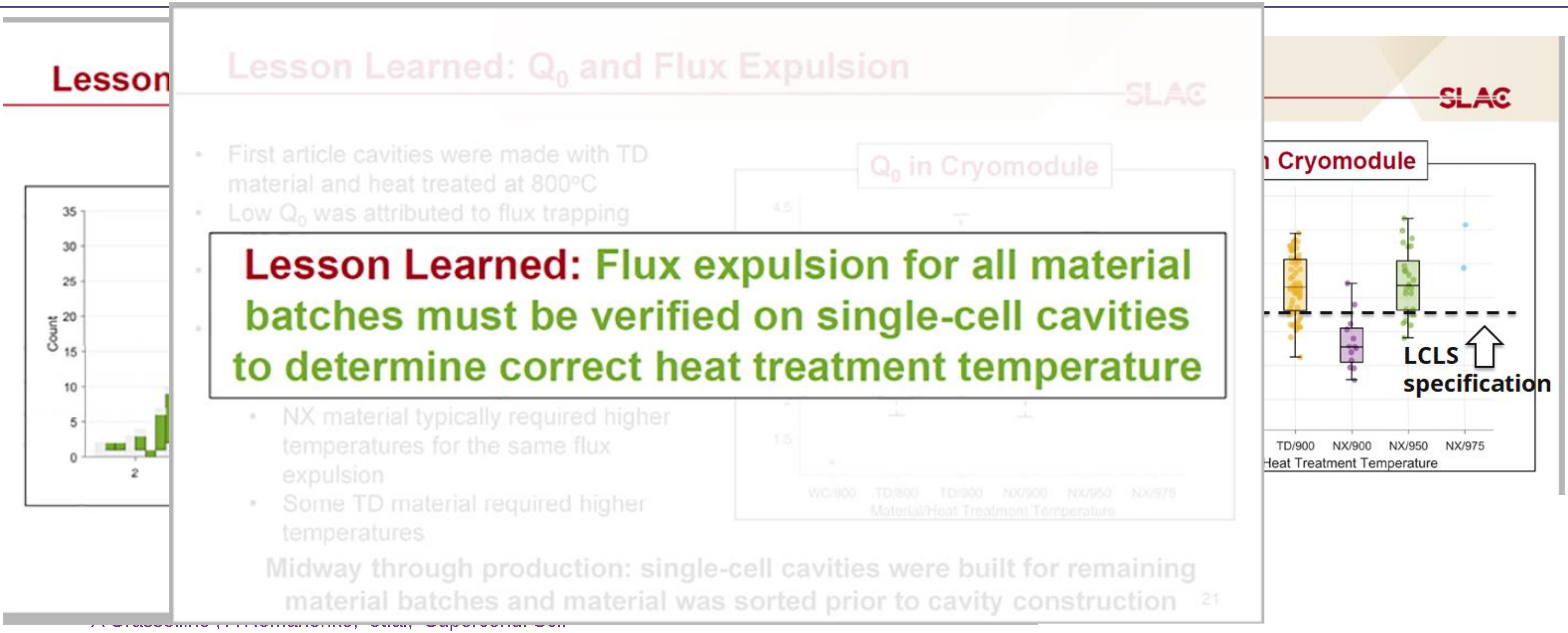
- **Clear evidence between flux trapping and microstructure found.**
  - Recovered substructure (lack of recrystallization) observed even after 1000°C/3h.
  - Bi-modal grain size distribution in a deformed sample after heat treatment with flux trapping mainly in fine grain regions (incomplete recrystallization) and flux expulsion in large grain regions (>100 µm) .
- **Half cell experiments also indicate the presence of un-recrystallized microstructure even after 800°C heat treatment.**
  - The deformed state is closely related to dislocation recovery and recrystallization temperature.
  - Some deformation structures could persist even after 900°C, 950°C... **Strength of the structure and mechanical stability needs to be considered while increasing heat treatment temperature.**
- **Upstream processing activities need attention for future high-*Q* high-*G* cavities.**
  - We are working on establishing vendor relationships to create variations in Nb sheet material.

Thank you, for your attention

Extra slides

LCLS-II specification is demanding- $Q_0 \geq 2.7 \times 10^{10}$ at 16 MV/m, cavities suffered from increased flux trapping and poor expulsion- Performance was limited.

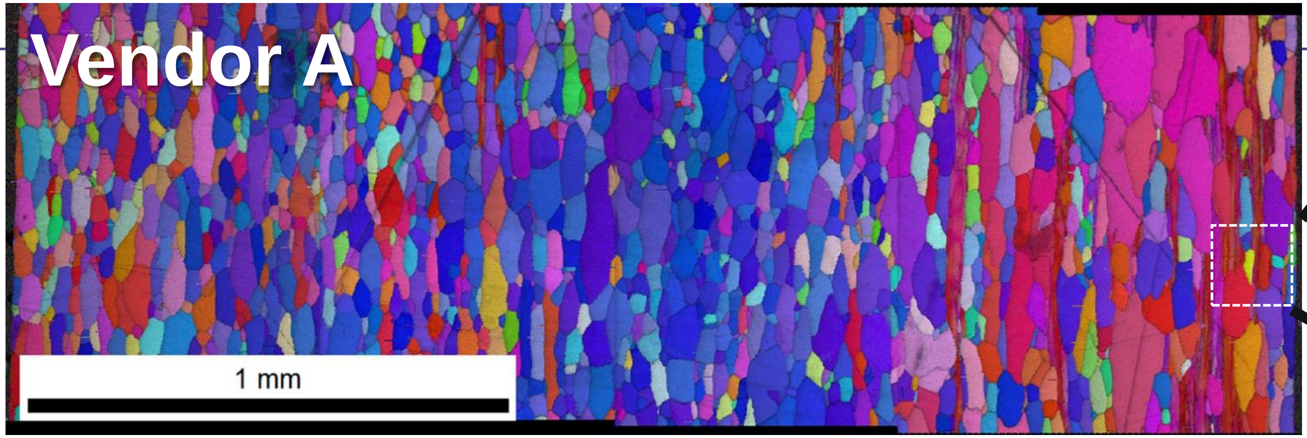
Fixes found!- Different batches need different heat treatment

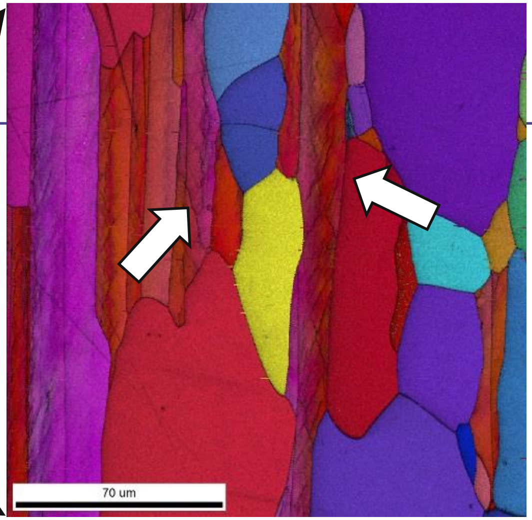


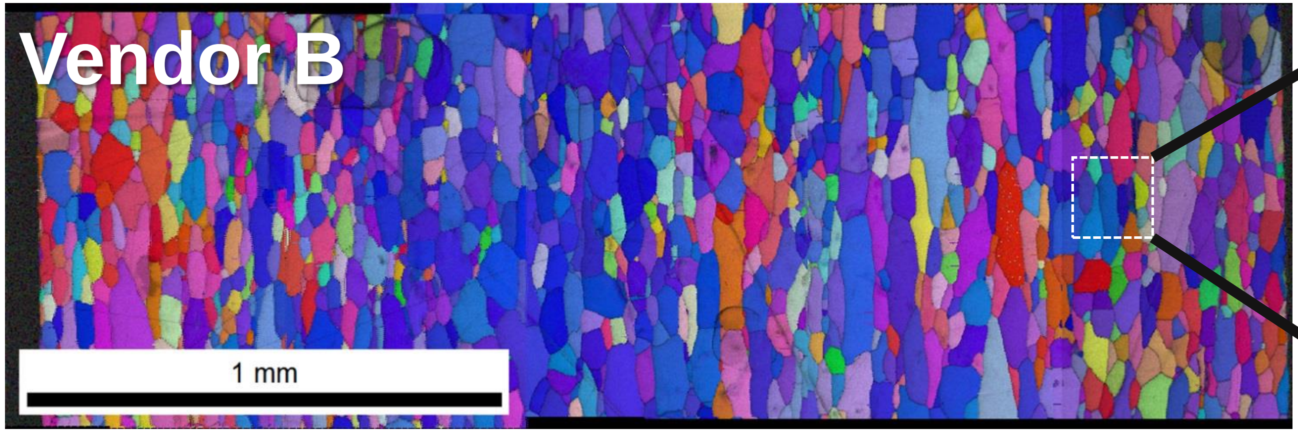
Technol. 26

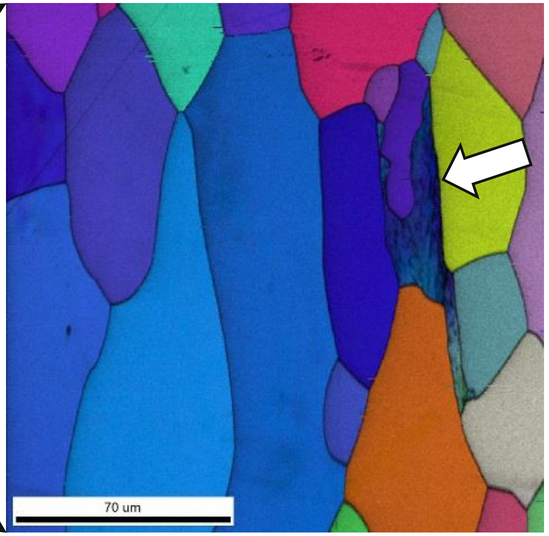
Dan Gonnella (on behalf of LCLS-II team) , Industrial Cavity Production: Lessons Learned to Push the Boundaries of Nitrogen-Doping, SRF 2019, Dresden, Germany

Deformation structures present in as received Nb sheets before any cavity level deformation



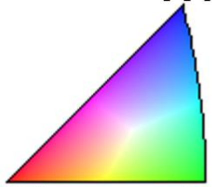






Recrystallized grain have uniform color.

Variations in color and black line networks identify dislocation substructures in recovered grains (lots of low angle GBs)



High resolution EBSD. Inverse pole figure (IPF) maps show crystallographic orientations in Nb sheet material