Fermilab

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Update on low RRR niobium vs flux expulsion

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U.S. DEPARTMENT of ENERGY
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Talk outline

1. motivation for flux expulsion
2. baking???
3. the low RRR problem
4. materials study
5. next steps
6. questions/discussion

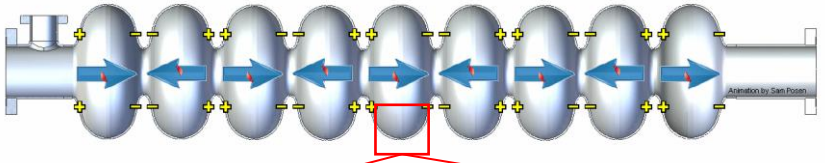
1

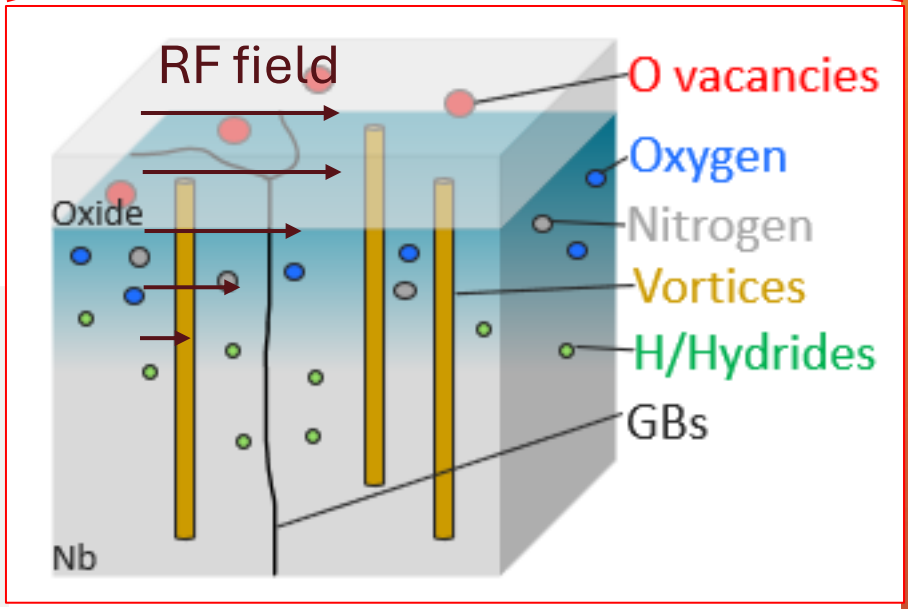
motivation for flux expulsion study

trapped flux

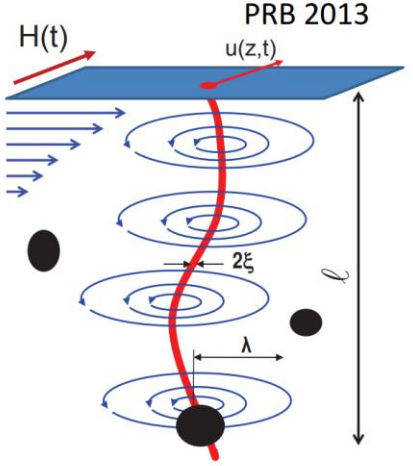
component of interest within larger study of impurities/defects in Nb

- renewed interest in community on material correlations with flux trapping/expulsion
- discussions surrounding microstructure as pinning centers
  - impurities
  - ‘texture’ (orientation)
  - strain
  - grain boundaries/grain size

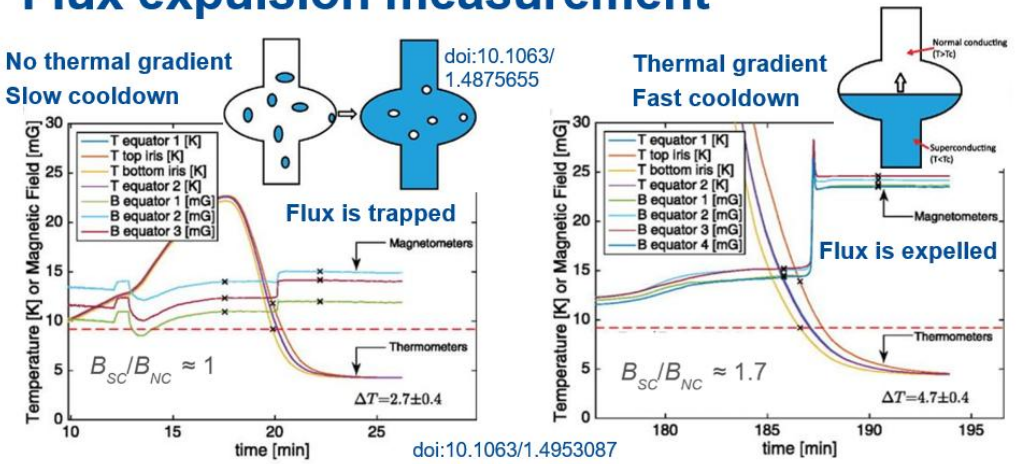




Gurevich & Ciovati
PRB 2013



Flux expulsion measurement



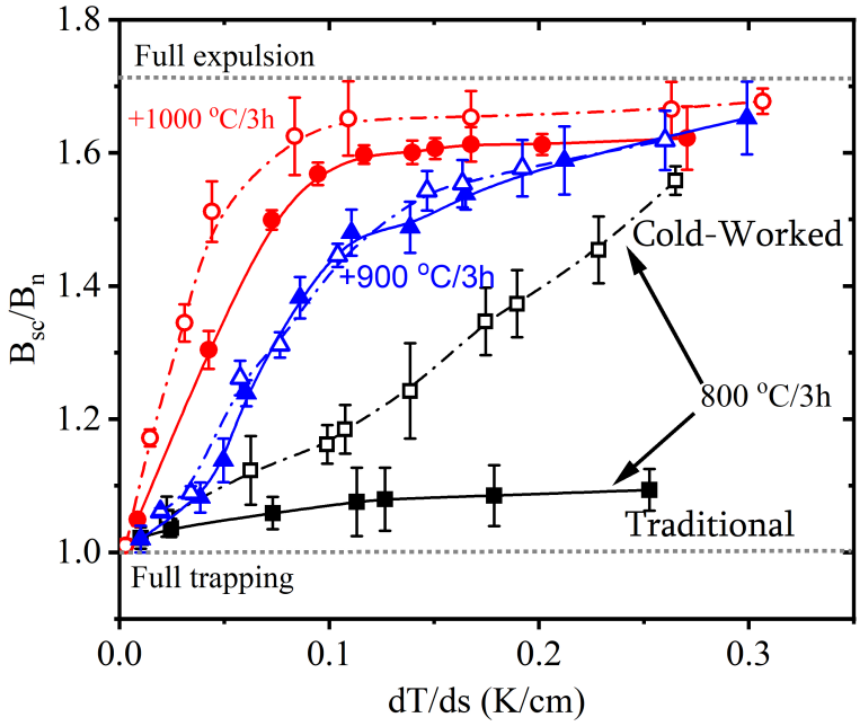
2

what about baking?

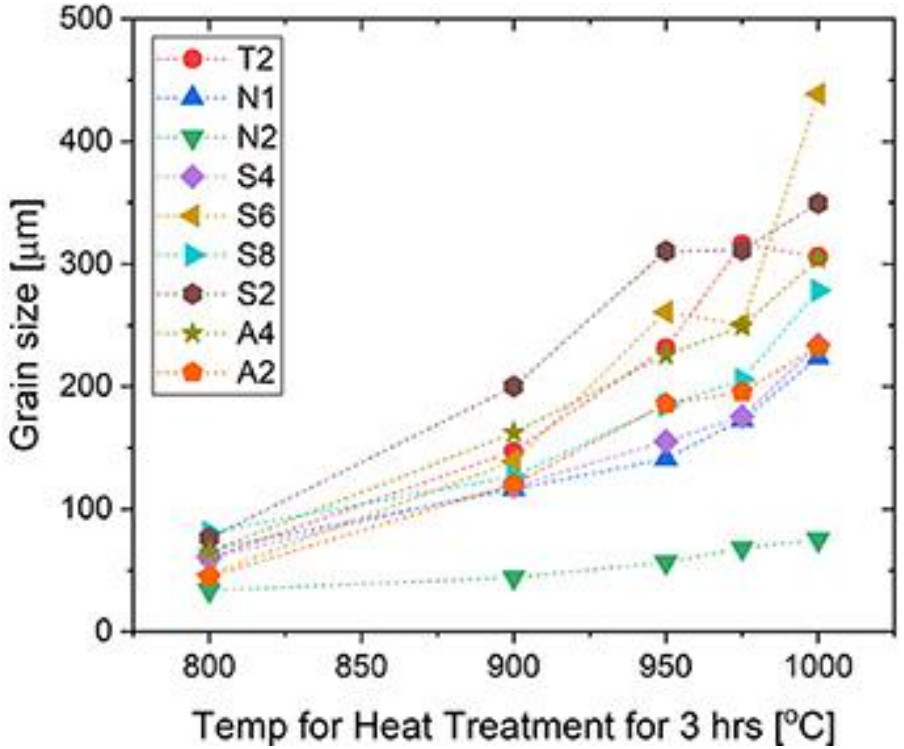
the usual solution

a 900 C bake is typically enough to solve our flux problems

1000 C almost always works



B D Khanal *et al* 2025 *Supercond. Sci. Technol.* **38** 015015



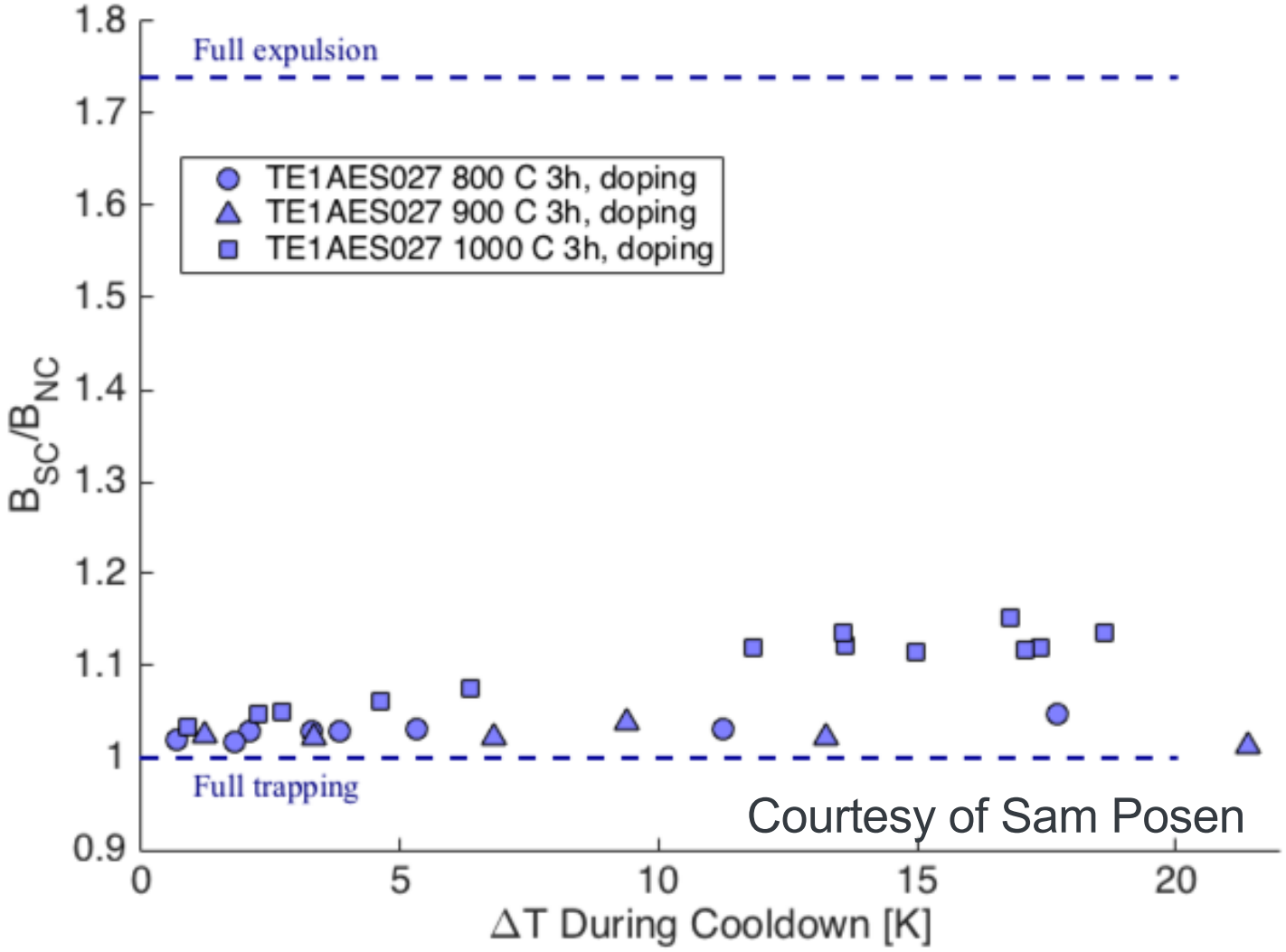
Zu Hawn Sung *et al* 2023 *Supercond. Sci. Technol.* **36** 095015

2

the problem(s) with low RRR material

Low RRR cavity has poor flux expulsion

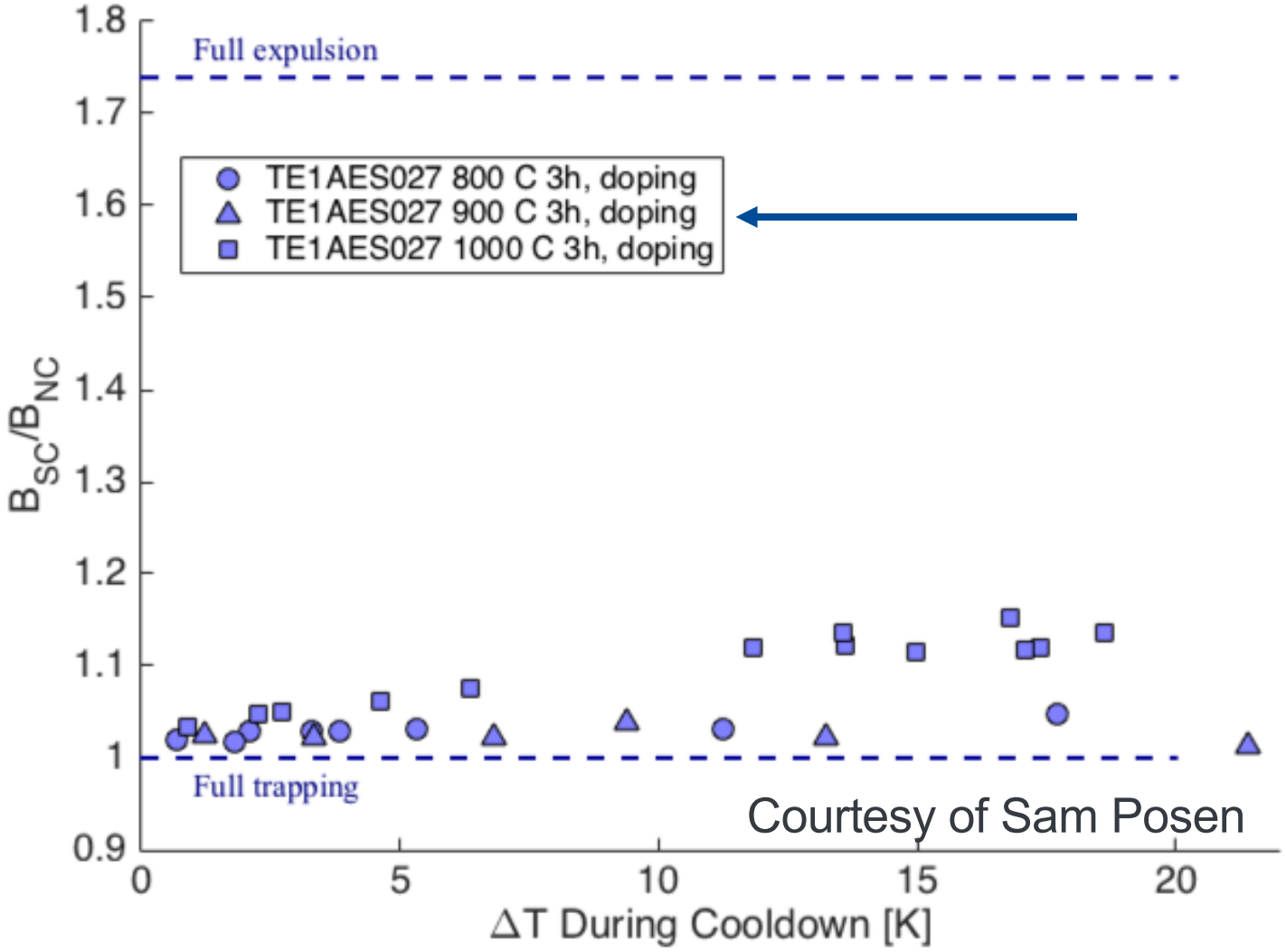
even after high-temperature annealing



Courtesy of Sam Posen

Low RRR cavity has poor flux expulsion

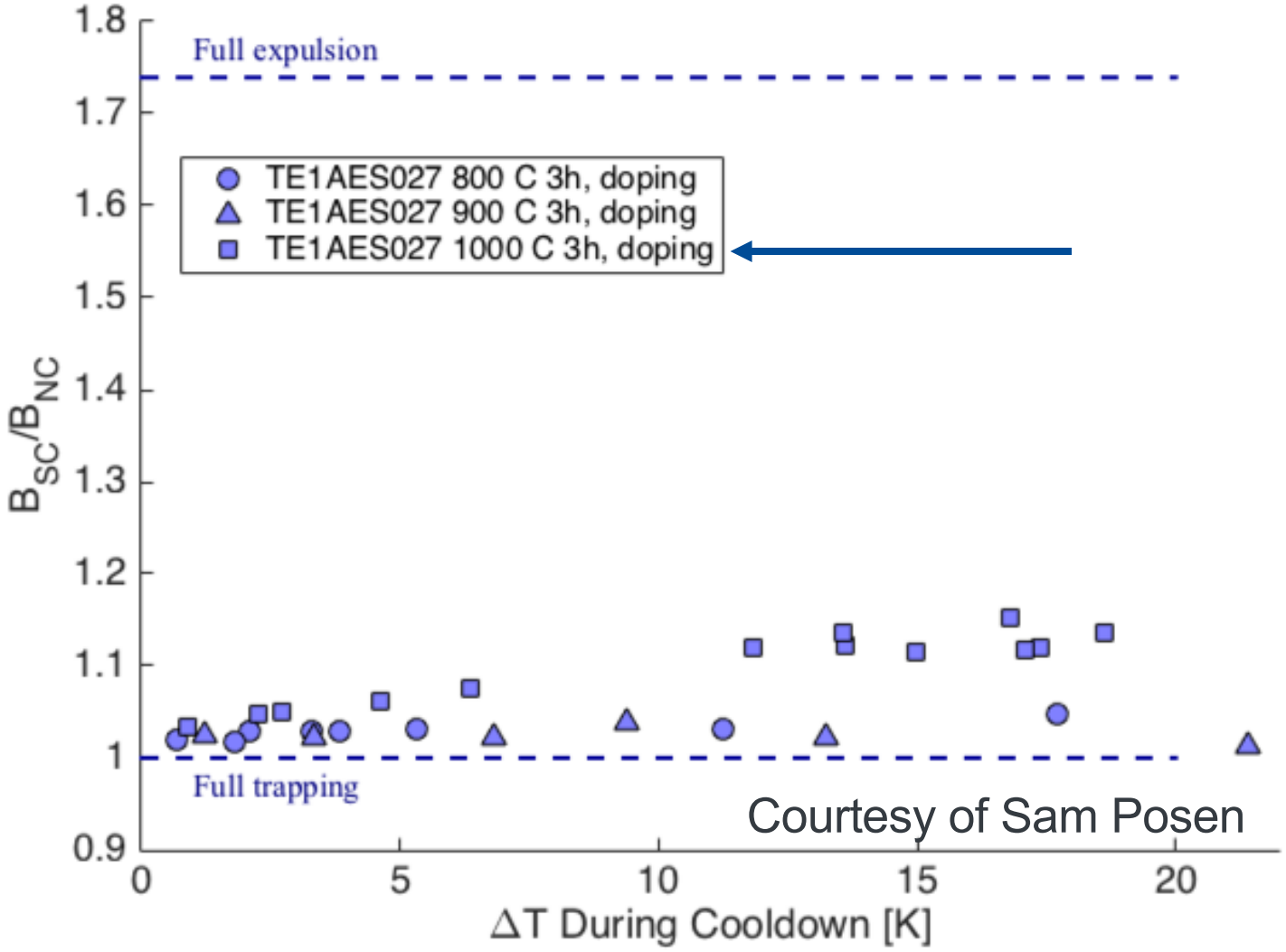
even after high-temperature annealing …. 900 C not enough



Courtesy of Sam Posen

Low RRR cavity has poor flux expulsion

even after high-temperature annealing … 1000 C not enough???

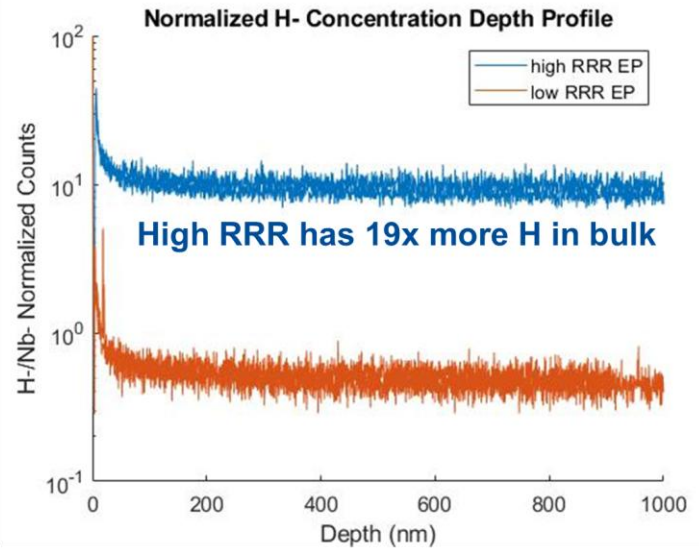


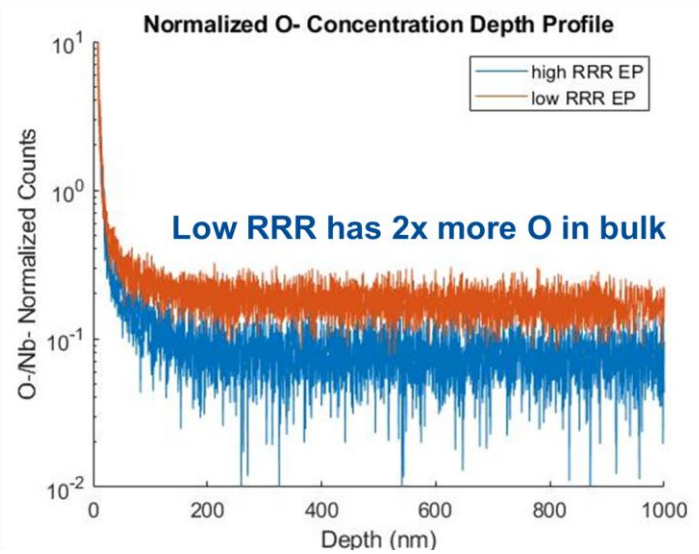
Courtesy of Sam Posen

What is different about this material?

RRR = 63 after 800 C x 3 hour bake

- Originally focusing on collaborative effect between intrinsic and extrinsic impurities through lens of optimizing BCS resistance
- What are the intrinsic impurities in low RRR material?
  - Not trivial! wild goose chase regarding data sheet and impurity content
  - RRR is a bulk property so only care about deep in bulk
  - SIMS: no “key” impurity found
  - Impurity concentration is **not** primary reason for low RRR
- Look at lattice itself to search for mechanism of RRR
- **New** focus on crystal structure through flux expulsion lens
  - Going to correlate grain growth/misorientation/RRR change with flux expulsion
- **What is going on in the material on a microscopic scale that is preventing flux expulsion?**
- **What is driving the poor flux expulsion and how can we fix it?**





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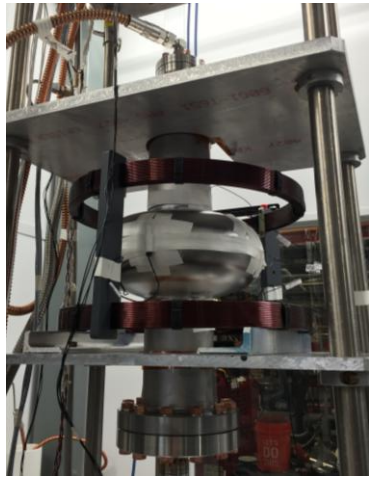
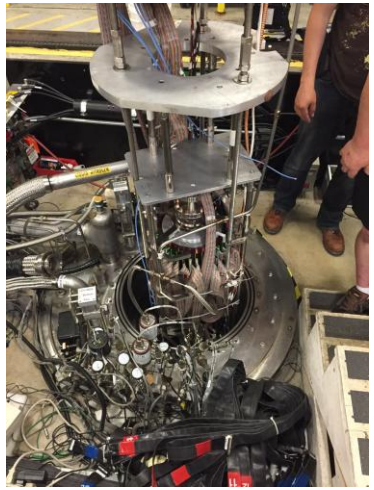
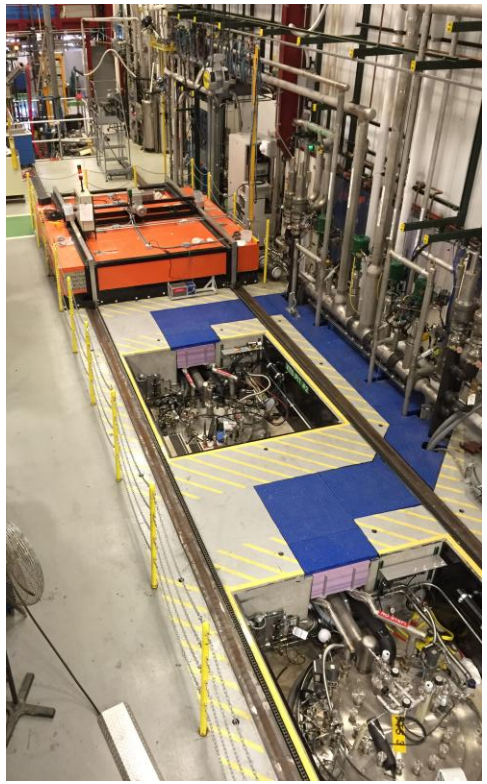
Sample study on low RRR material

Materials Science to Identify Origins of Limitations

RF performance of cavities







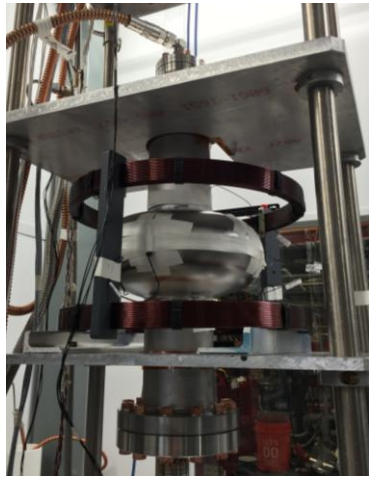
Material science measurements

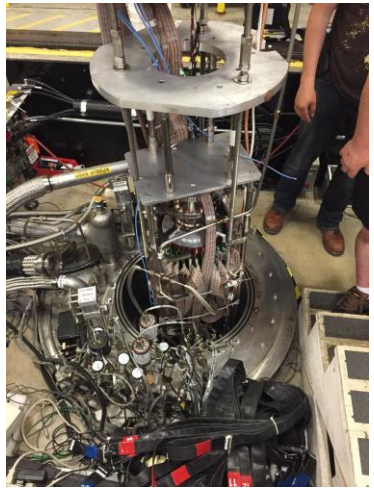
XRD

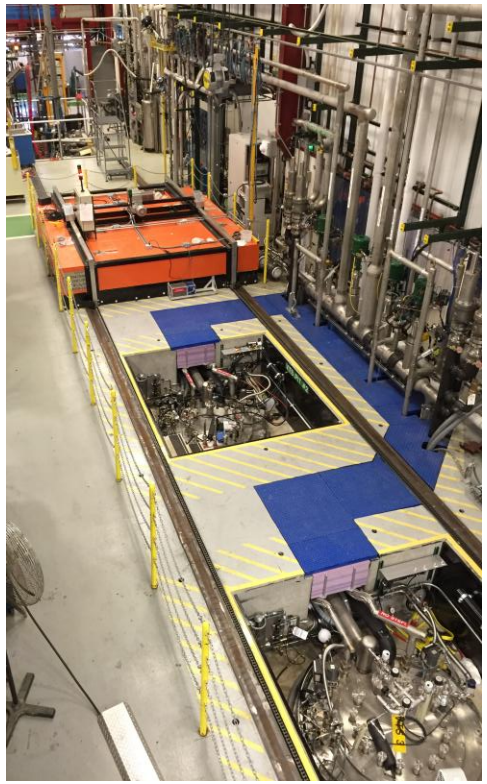
PPMS

Materials Science to Identify Origins of Limitations

RF performance of cavities







Material science measurements

XRD

PPMS

sample study plan

samples cut from same Nb sheet as the cavity

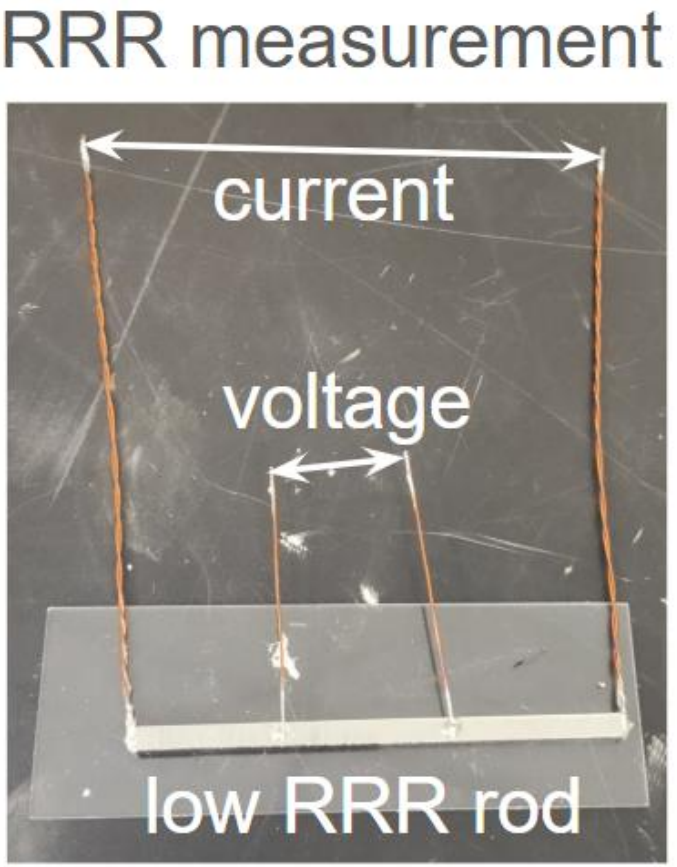
- samples in 6 conditions:
  - as received (bulk EP only)
  - 800 C x 3 hours
  - 900 C x 3 hours
  - 1000 C x 3 hours
  - 1100 C x 3 hours
  - 1200 C x 3 hours
- measurements:
  - RRR: 4 point resistance measurement $= \frac{\rho(293\ K)}{\rho(10\ K)}$
  - PPMS: magnetization hysteresis loops to get Hc1, Hc2, Tc
  - XRD: measure 2theta, FWHM, intensity of peaks
  - EBSD: grain size, grain orientation
  - SIMS: impurity concentration depth profile

RRR measurement

4 point resistivity measurement of bulk

- RRR $= \frac{\rho(293\ K)}{\rho(10\ K)}$
- see increasing RRR with bake temperature
- requires LHe for test

Sample treatment	RRR
As received (EP only)	35
800 C x 3 hour bake	63
1000 C x 3 hour bake	128
1100 C x 3 hour bake	TBD
1200 C x 3 hour bake	TBD

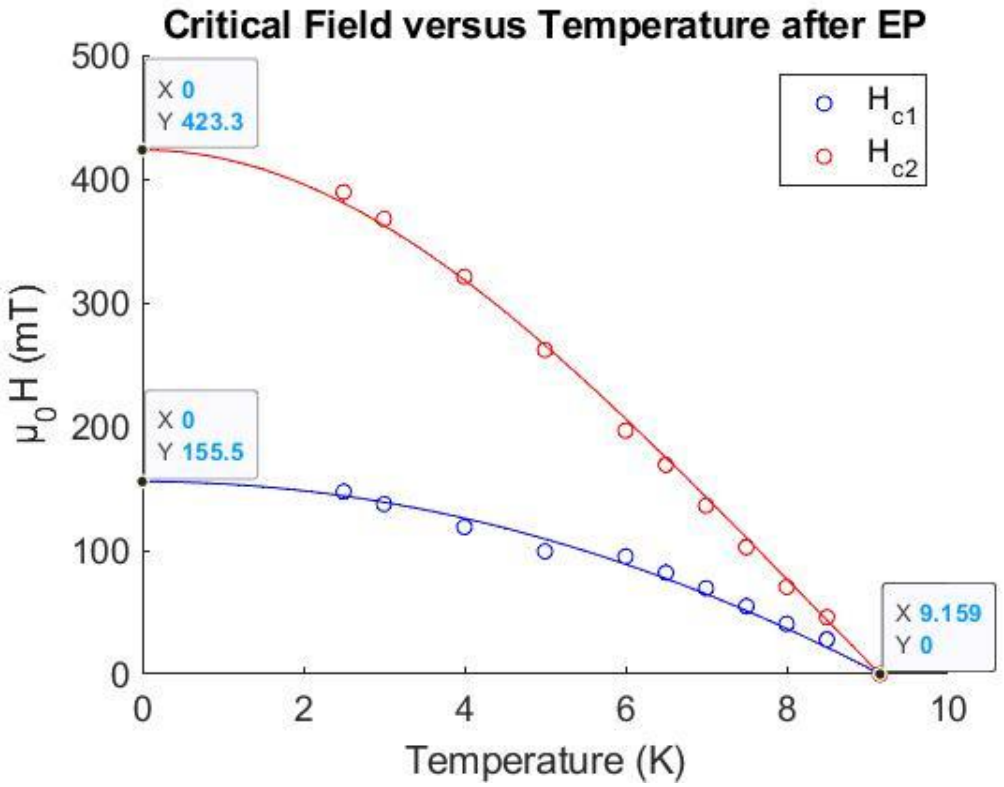


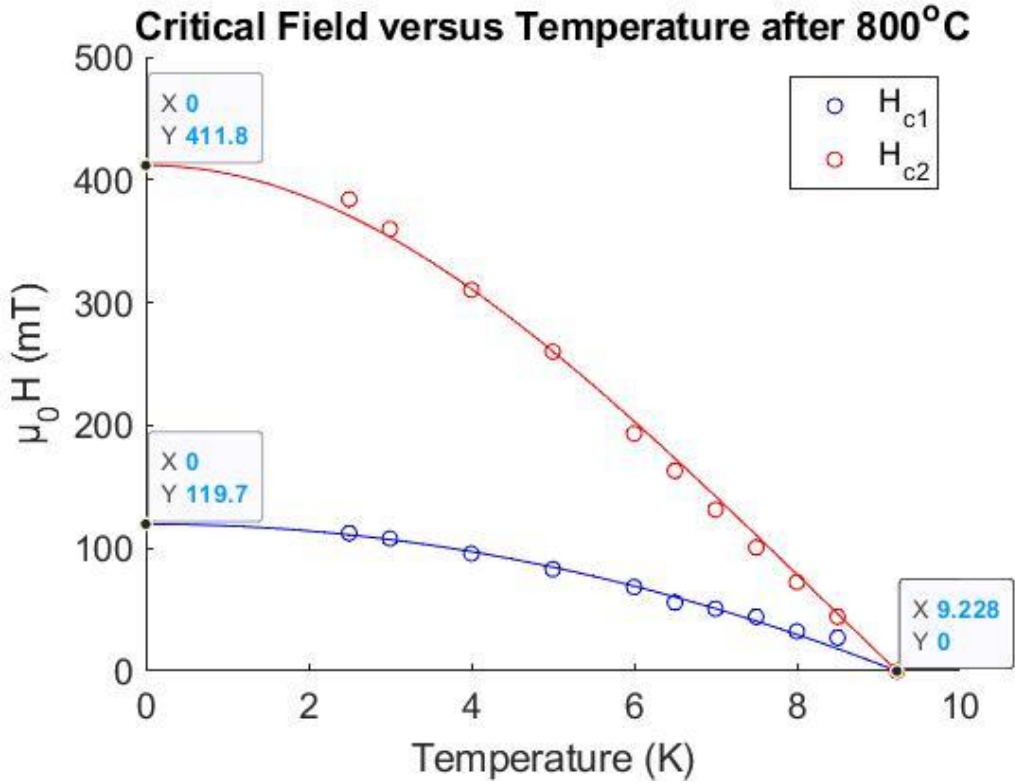
Magnetization measurements

measurements of magnetization vs applied magnetic field

extract Hc1,Hc2, and Tc

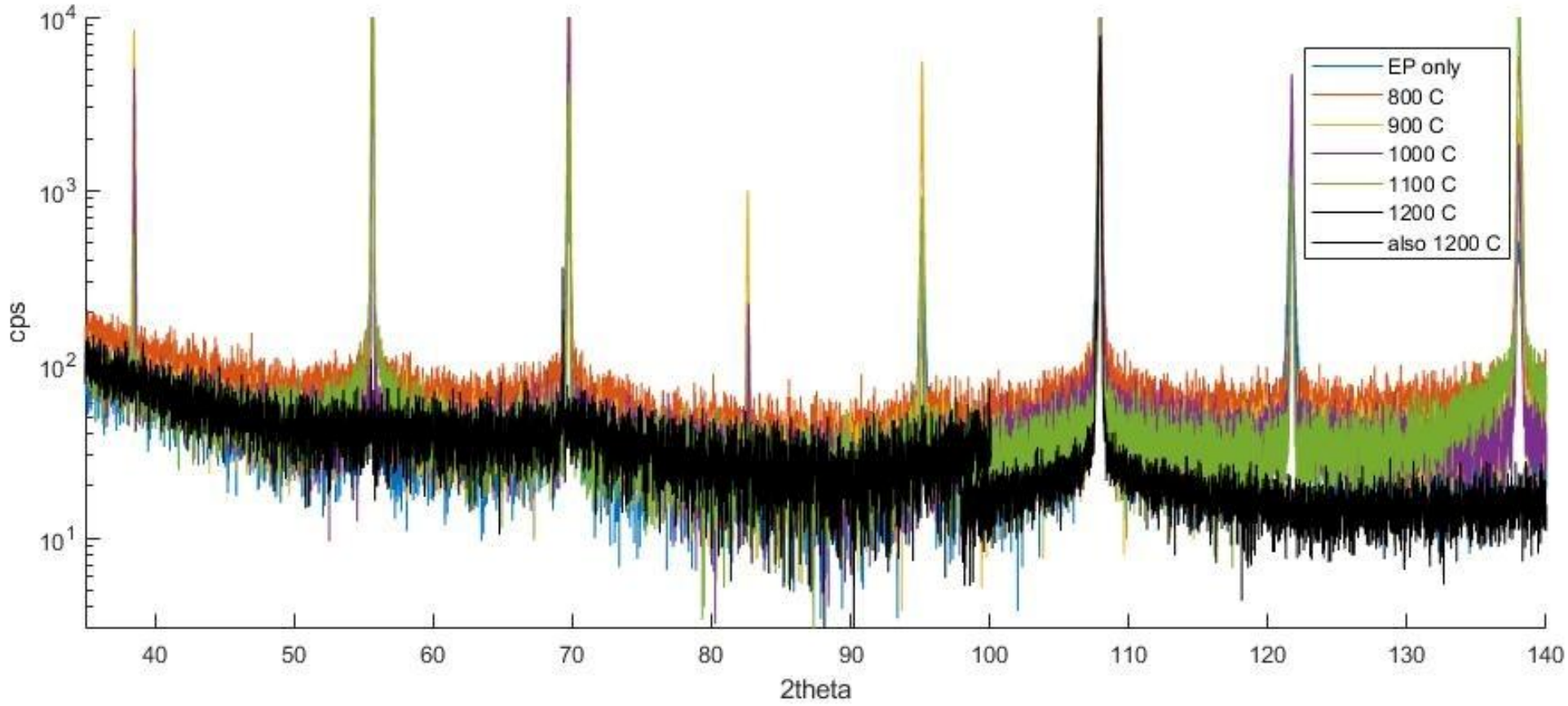
preliminary results:





X-ray diffraction

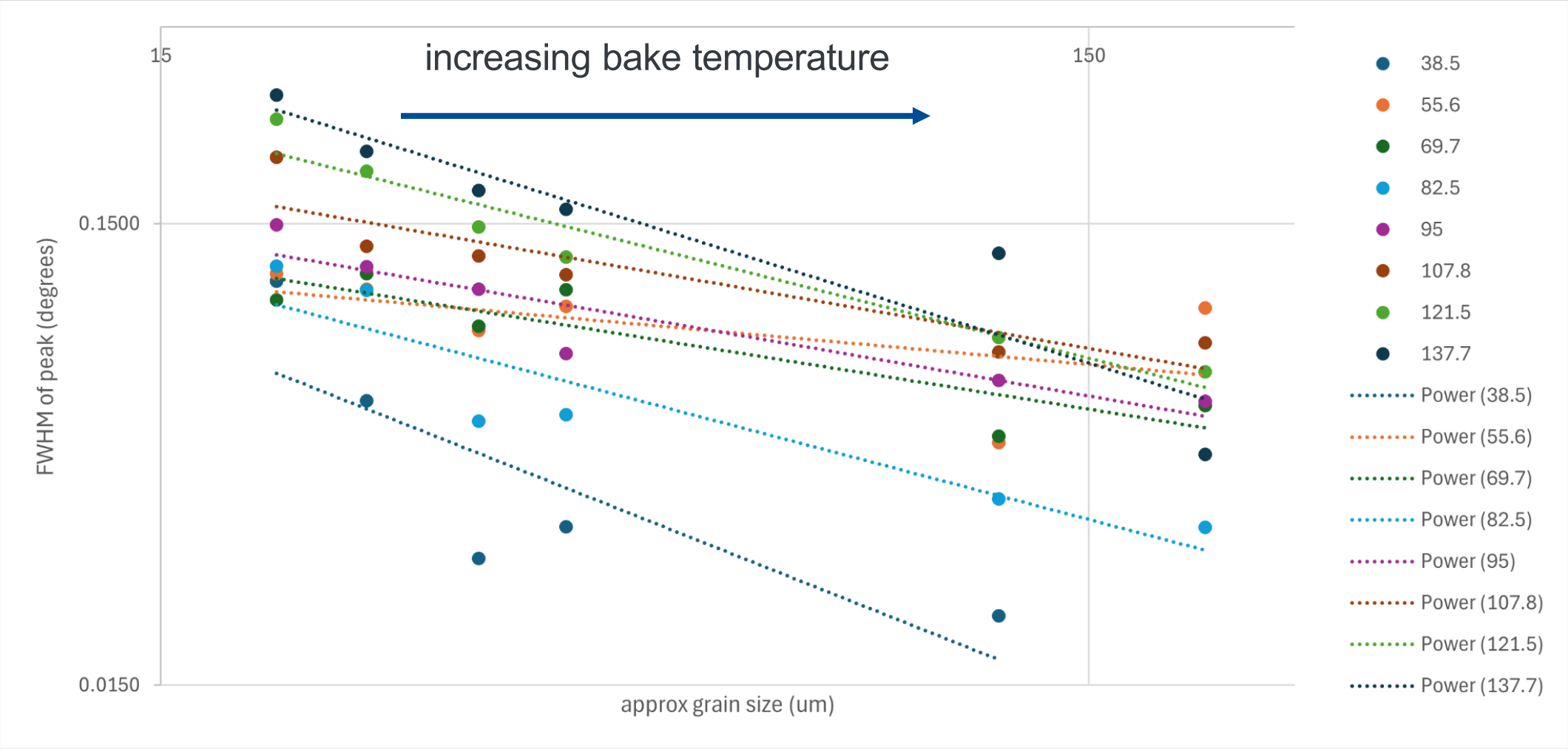
becomes more difficult to measure all peaks after high temperature anneal because grain size is so large → have to do rocking measurements at each peak individually



X-ray diffraction

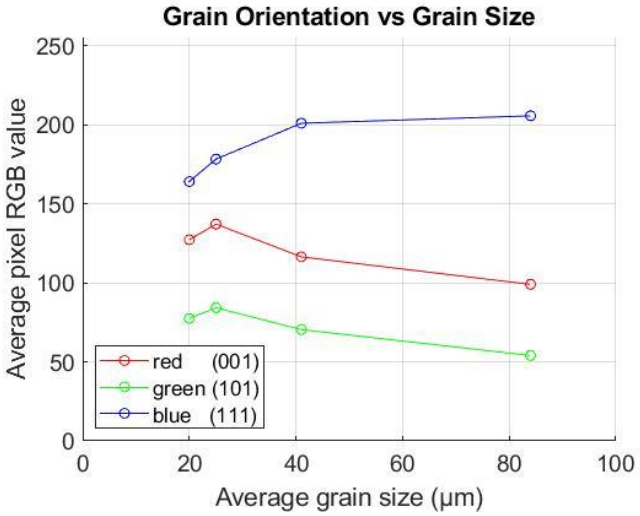
full width at half maximum of each diffraction peak

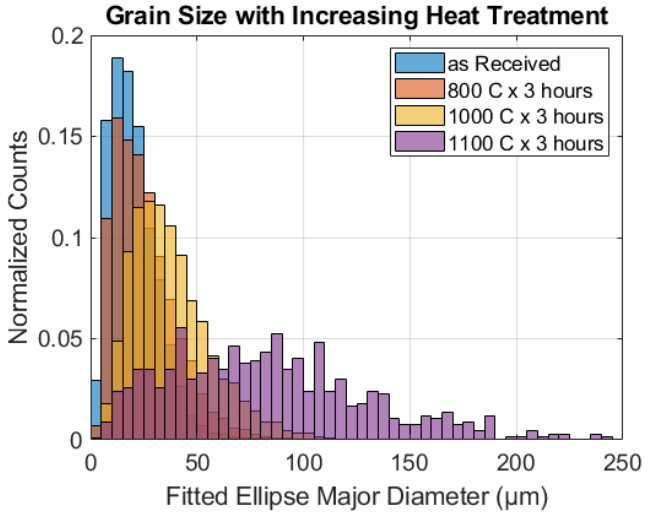
observe general decrease in peak width at higher bake temperature (proxy for strain)

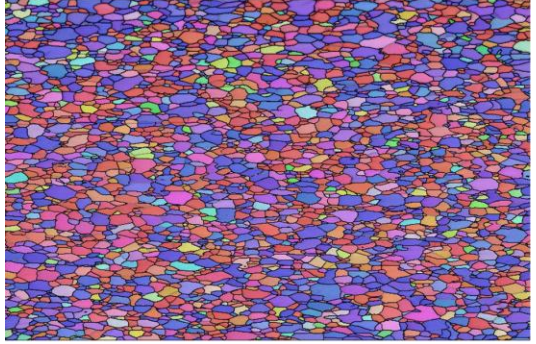


electron backscatter diffraction

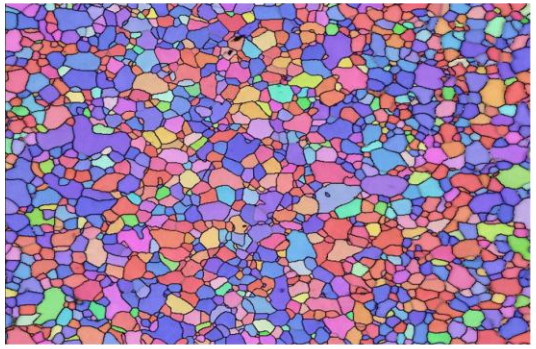
- observe increase in grain size with bake temp
- observe shift in orientation toward 111



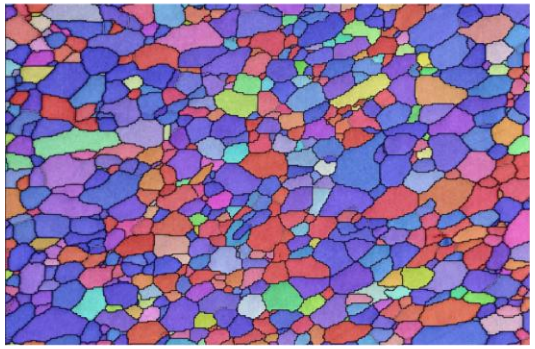




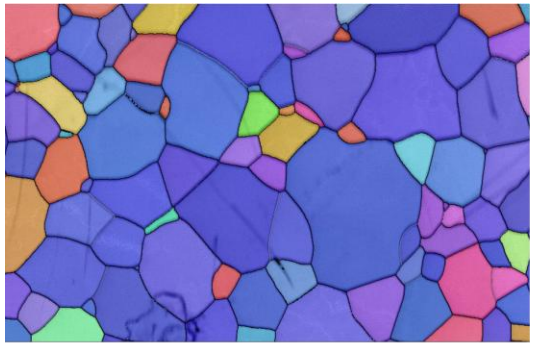
As received (EP only)
avg grain size = 20 um
avg pixel = [127,77,164]
RRR = 35



800 ˚C x 3-hour anneal
avg grain size = 25 um
avg pixel = [137,84,178]
RRR = 63



1000 ˚C x 3 hour anneal
avg grain size = 41 um
avg pixel = [116,70,201]
RRR = 128



1100 ˚C x 3-hour anneal
avg grain size = 84 um
avg pixel = [99,54,206]
RRR = TBD

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Summary

- Relationship between bake temperature and grain **growth**
  - Slow growth up to 1000 C → rapid growth after 1100 C
- Relationship between bake temperature and grain **orientation**
  - Shift toward 111 orientation with increased temperature
- Relationship between bake temperature and lattice **strain**
  - decrease in diffraction peak width corresponds to decrease in strain

- We do see similar improvement in microstructure of low RRR material after baking
  - just need higher temperature
  - is 1200 C going to be enough to improve flux expulsion?

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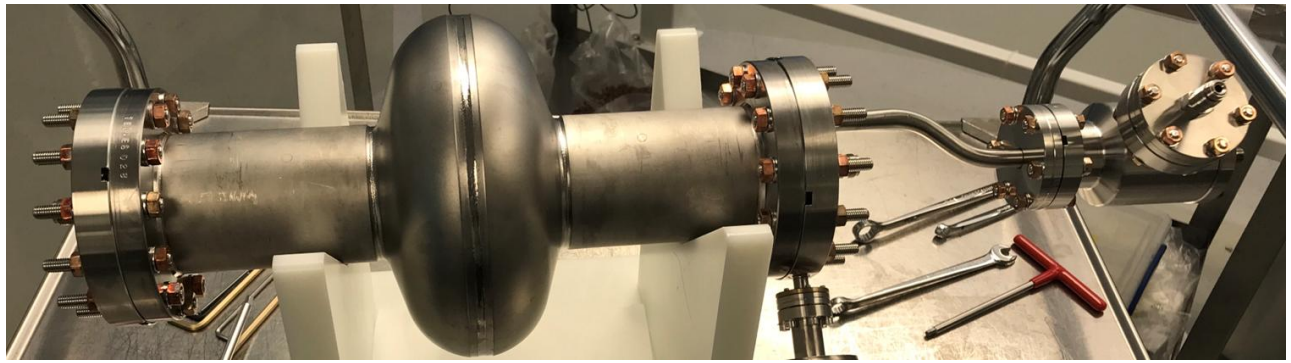
next steps

Low RRR next steps

- Sample study
  - EBSD on remaining samples
  - SIMS measurements
  - lots of data analysis
- Cavity study
  - Bake low RRR cavity to 1200 C
  - Flux expulsion measurement on cavity



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discussion/question time

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