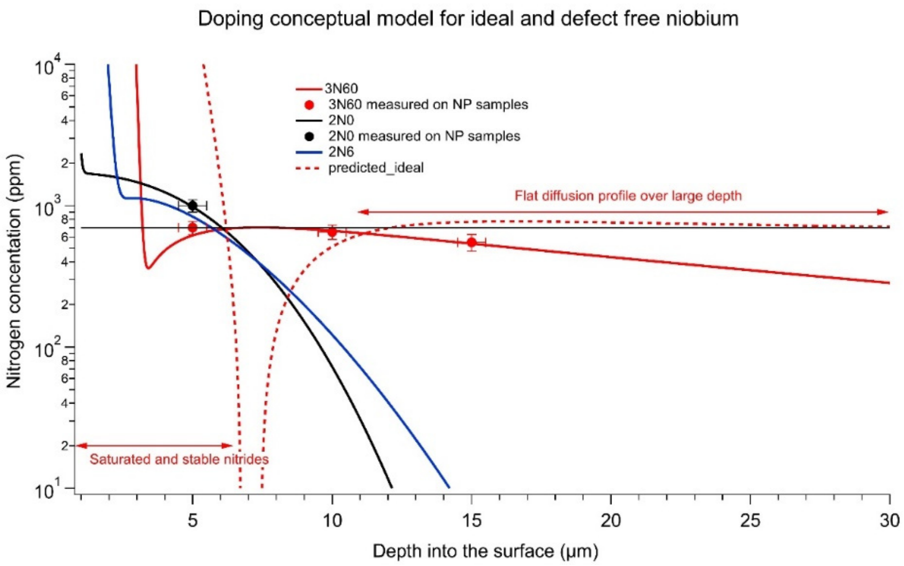
Development of a Qualitative Model for N-Doping Effects on Nb SRF Cavities

Can we make a predictive model of quench fields and high Q0 in high temperature doped SRF cavities

- Nitride growth morphology
- Diffusion profiling
- Nitrogen's hydrogen blocking effects
- Nitride, carbide, and nitrided niobium chemistry effects



Ari Palzewski – SRF Scientist

Monday, July 1, 2019

Contributing information

- Sample EP Hui Tian - JLab
- SEM images by: Joshua Spradlin - JLab
- Analysis by : Joshua Spradlin, Charlie Reece, and Ari Palczewski - JLab
- SIMS measurements by Natalie Sievers and Jonathan Angle @ Nanoscale Characterization and Fabrication Lab (NCFL) at Virginia Tech with Cameca IMS 7f GEO dynamic SIMS
- Coordination between facilities and input from Michael Kelley – College of William & Mary and Virginia Tech

Outline

- History and developing the initial model
  - Low doping long anneal times
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  - 3N60 initial results
  - Quench field expectations Comparison to theory
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- New data
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  - Post doping EP defects
  - FNAL single cell EP
  - Failure of HE 9 cell cavities
- Qualitative model rethink

Comment on Nomenclature

- Historically for doping at we have used N#A##, N# is the time for nitrogen at 25mTorr and A## was the post doping vacuum annealing time.
- Then we dropped the “A” and then went to #N## where #N is the time in nitrogen and ## is the anneal time
- Now we sometimes use #/## for doping time/vacuum annealing time

N3A60=3N60=3/60

Pressure always at ~20-25mTorr unless noted at all Labs/manufactures

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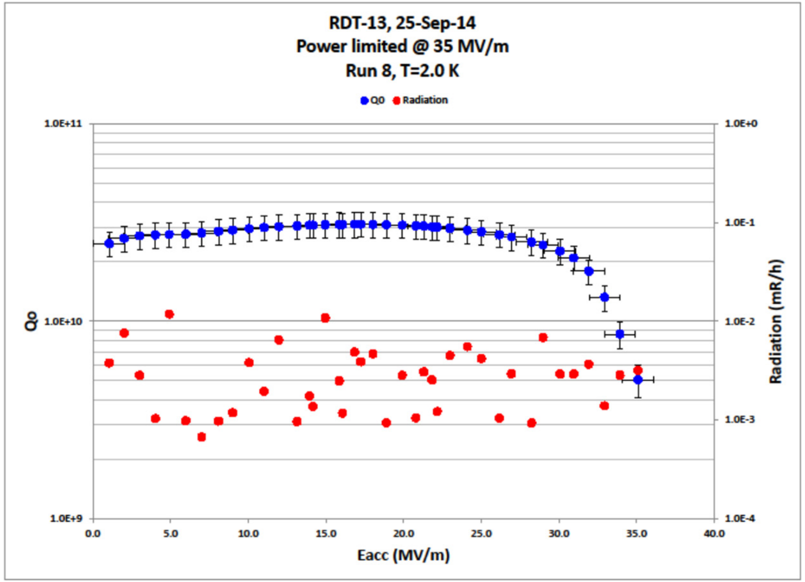
Low doping studies 2014

2N60 – EP5

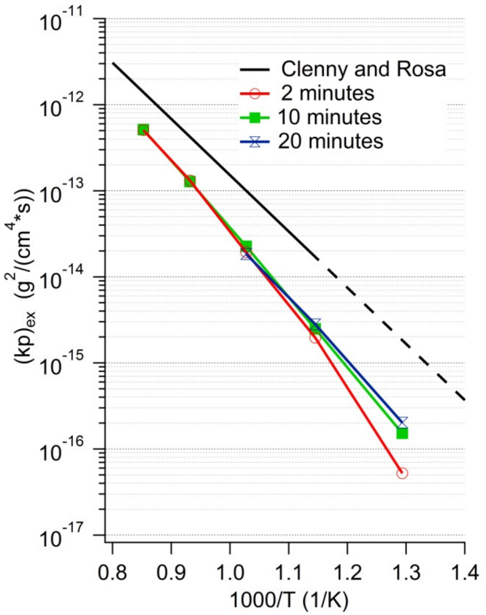


- Low doping, long anneal times was first evaluated in 2014
- Low doping was supposed to solve two problems
  - One – wide and flat diffusion profile to allow wider production variances in EP.
  - Two – block hydrogen deeper in the bulk, predicted by modeling *
- Why were these cavities Q-slope and not quench limited?

*Ford, Denise C., Lance D. Cooley, and David N. Seidman. "Suppression of hydride precipitates in niobium superconducting radio-frequency cavities." *Superconductor Science and Technology* 26, no. 10 (2013): 105003.

Nitride growth model 2016

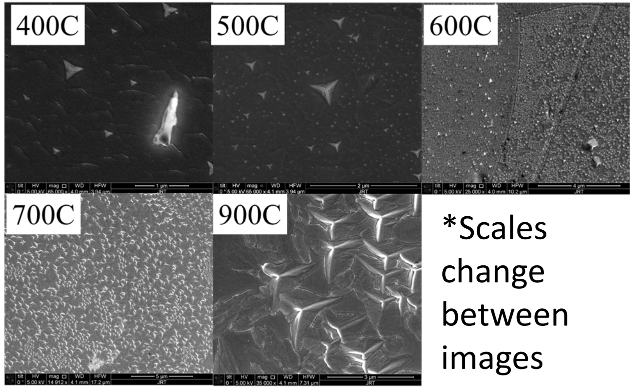
Arrhenius plot – total absorption calculated using the furnace pressure drop on largest niobium tests plates - black line atmospheric pressure study



A. D. Palczewski, et al., "Investigation of Nitrogen Absorption Rate and Nitride Growth on SRF Cavity Grade RRR Niobium as a Function of Furnace Temperature", in *Proc. 28th Linear Accelerator Conf. (LINAC'16)*, East Lansing, MI, USA, Sep. 2016, pp. 744-747. doi:10.18429/JACoW-LINAC2016-THOP02

- Within errors– i.e. very small amount of total nitrogen actually diffuses into the bulk at all temperatures – most form nitrides.
- Nitrides form at all temperatures down to 400°C – SEM images

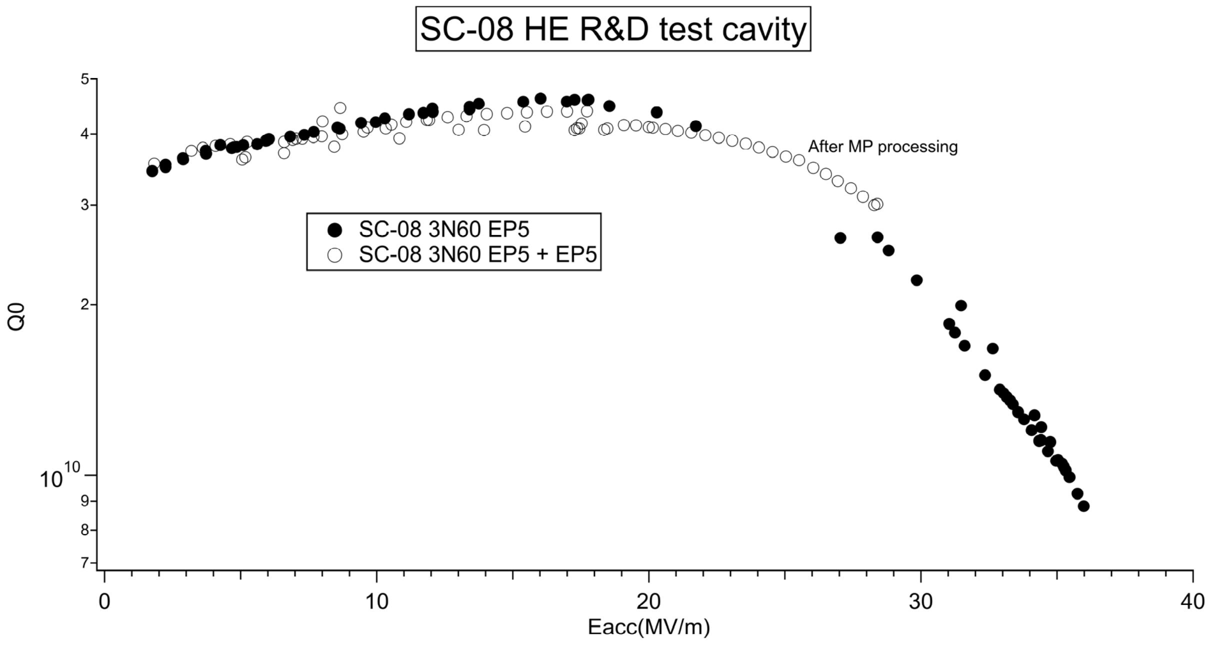
<u>Sample SEM images* looking for Nitrides – 20 minutes doping</u>



*Scales change between images

Jefferson Lab

3N60 doping 2018 – LCLS-II HE R&D

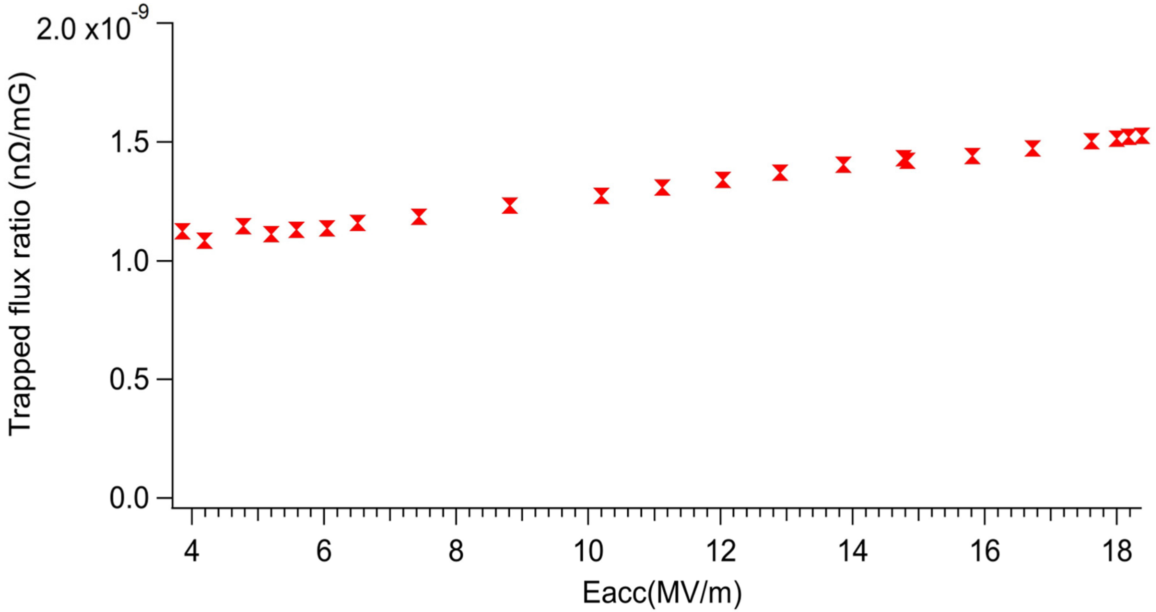


- One other cavity went to 37MV/m after 10 micron EP

Palczewski, "High Q0 and Gradient R&D at JLab", Vancouver, Canada on the campus of the University of British Columbia and hosted by TRIUMF on February 5 - 8, 2019

- Low doping, long anneal times' RF properties duplicated with "new" 3N60 recipe.
- Data again suggested the hydrogen blocking effects of nitrogen were probably in play.

3N60 EP8 – Flux trapping losses

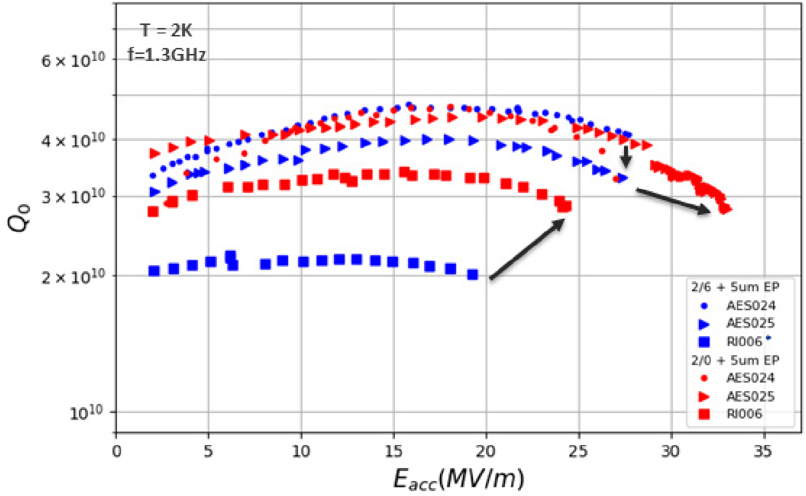


*M. Checchin *et al.*, "Frequency dependence of trapped flux sensitivity in SRF cavities," *Applied Physics Letters*, vol. 112, no. 7, p. 072601, 2018.

- Flux trapping losses from the 3N60 suggest the mean free path is about the same as 2N6 EP5 doping level ~100nm*
- Conclusion - Higher Q0 and Quench field in 3N60 suggests hydrogen blocking no RF surface doping level

2N0 doping 2015/2018 – LCLS-II HE R&D @ FNAL

Sequential Cavity Recipe Study: Test 2/3



2/6 + 5um EP (Baseline):

+40um EP reset

2/0 + 5um EP:

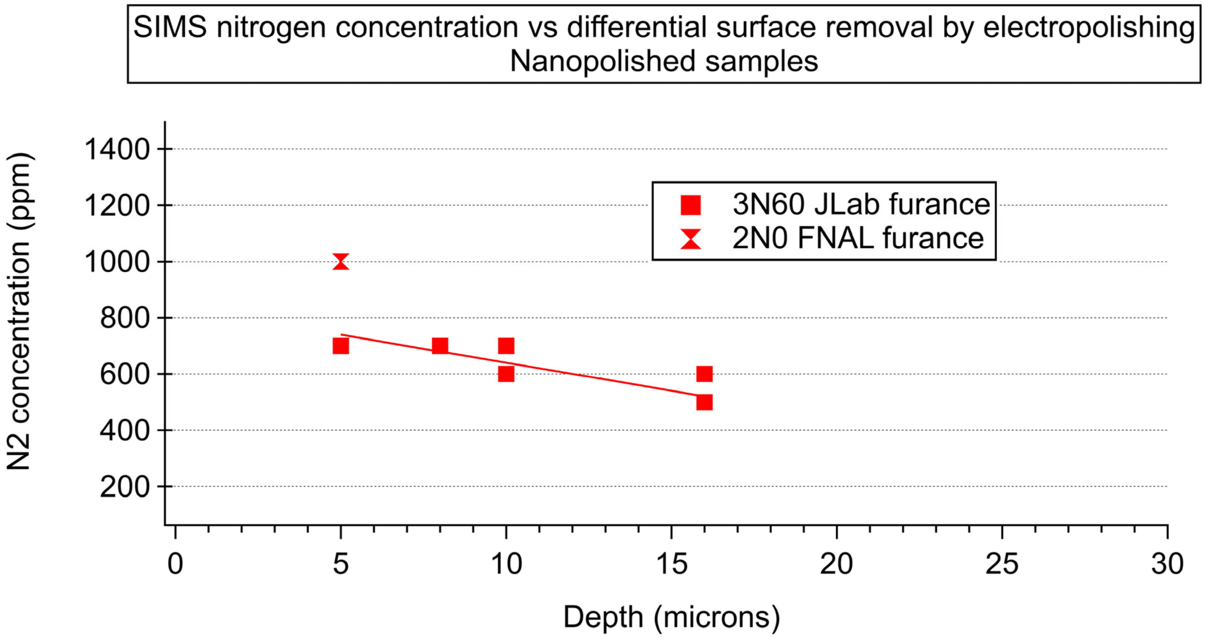
- AES025 & RI006: higher Q and Quench increases by +6MV/m
- AES024 yields similar results

* RF test had a bad FG

- 2/0 recipes show higher quench fields than 2/6.
- FNAL analysis said the higher quench field came from low doping level i.e. higher mean free path.

A. Grassellino, "N Doping: Progress in Development and Understanding", in *Proc. 17th Int. Conf. RF Superconductivity (SRF'15)*, Whistler, Canada, Sep. 2015, paper MOBA06, pp. 48-54.

SIMS 2018 supporting the initial conceptual model

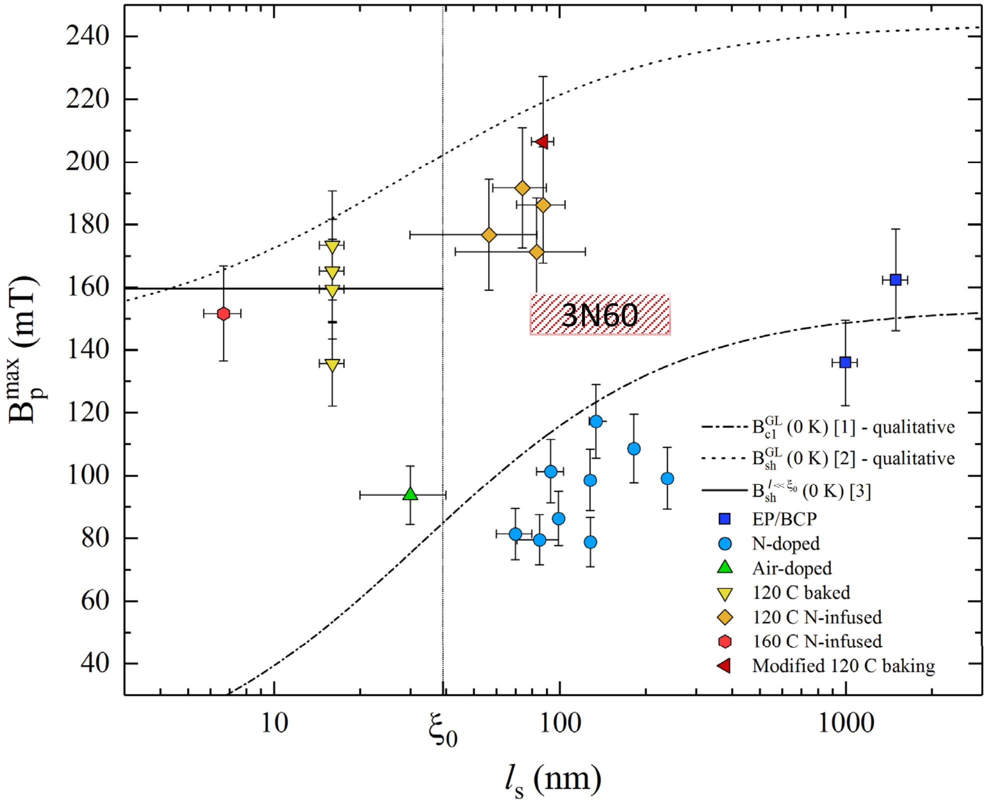


- 3N60 samples have a lower doping and therefore higher mean free path and higher H_{C1}
- The hydrogen blocking effect of nitrogen allows for higher than expected Q0

Theoretical insights at the time – 3N60

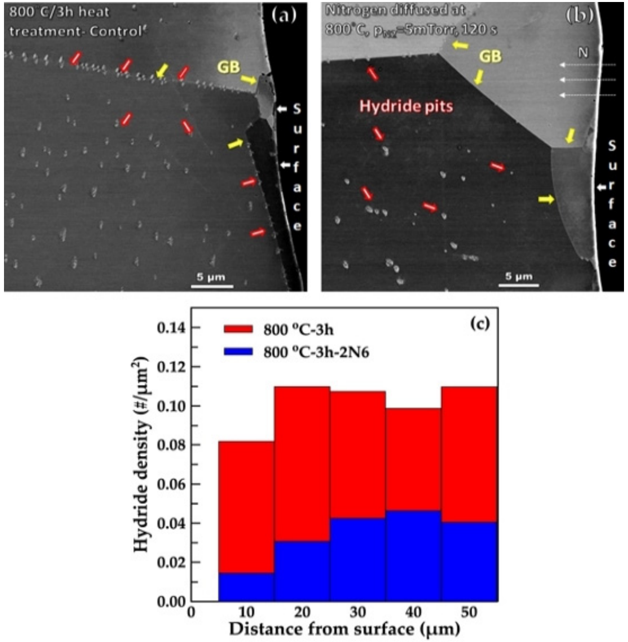
Plot from M. Checchin *et al.*, to be published (2018) – taken from TTC2018

Quench field vs superficial mean-free-path



- First time doped cavities with High Q0 at mid-field exceed the HC1 quench limit
- These new data points again point to hydrogen blocking as a key parameter similar to explanation of N-infused cavities, and 120°C baked cavities

Hydrogen blocking effect of nitrogen



Doped surface suppress hydrogen nobility toward the surface during cooling to 2K.

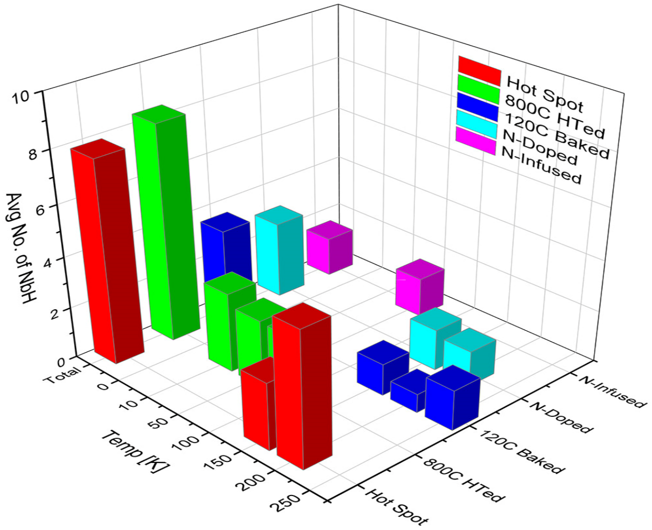
Taken from - P Garg et al 2018 Supercond. Sci. Technol. 31 115007 doi:10.1088/1361-6668/aae147

Theory - Ford, Denise C, Lance D Cooley and David N Seidman, *Suppression of hydride precipitates in niobium superconducting radio-frequency cavities.* Superconductor Science and Technology, 2013. **26**(10): p. 105003.

Niobium hydride studies using AFM/MFM

Statistical comparison of NbH precipitation morphology I

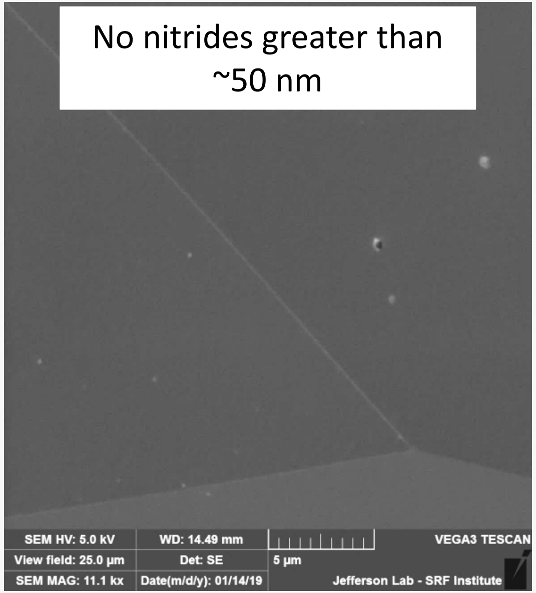
Avg. No. of NbH appearance within 10 x 10 µm² unit area during cooling



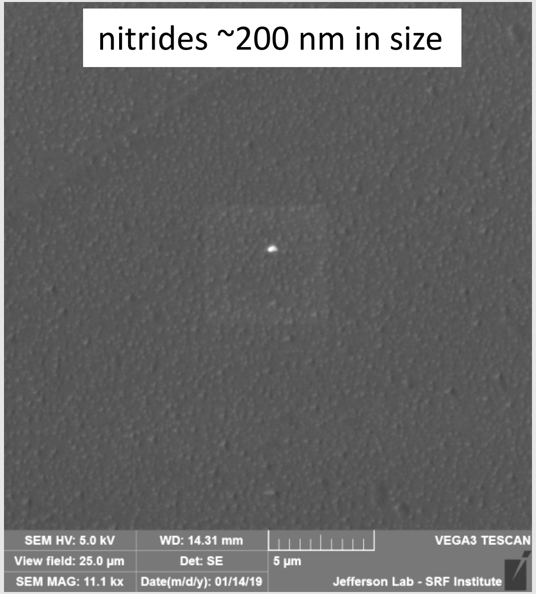
Taken from ZHSUNG | LCWS 2018 Arlington

SEM images late 2018 – JLab doped samples

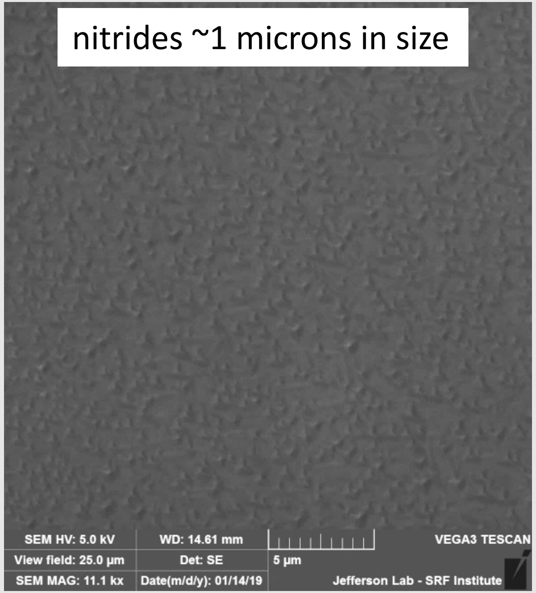
L92 – 2N0



L94 – 2N6

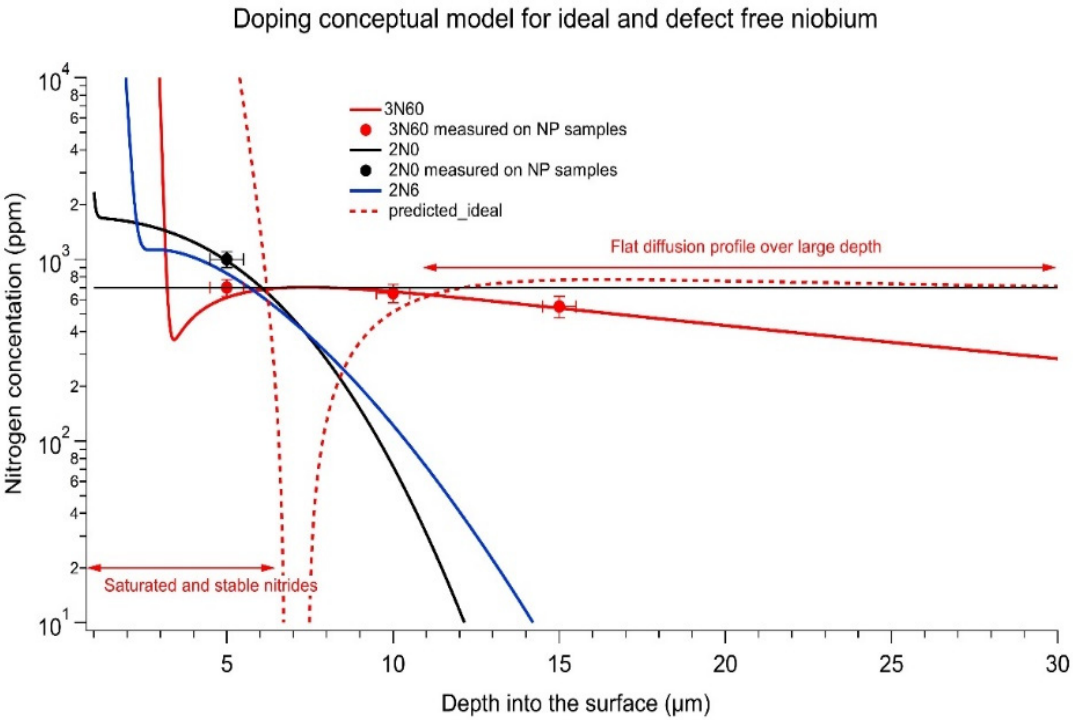


L91 – 3N60



- It appears nitrides have a seed time where diffusion occurs before crystal form
- Continued nitride growth in the annealing phase strongly suggest diffusion from the bulk to the surface – lower the doping level and elongating the tale into the bulk

Initial conceptual model – fall 2018/spring 2019



- Conceptual model of the envisioned - predicted ideal doping level for gradients up to 40 MV/m while maintain high Q0 at midfield
- Curves scaled to fit nano-polished SIMS measurements of nitrogen concentration.
- Can we make a scalable model to allow us to mathematically calculate our "ideal" doping?

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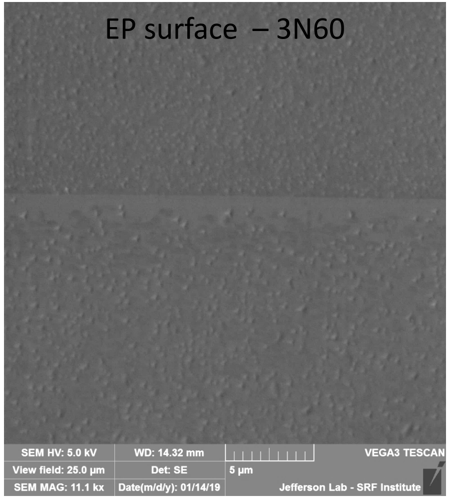
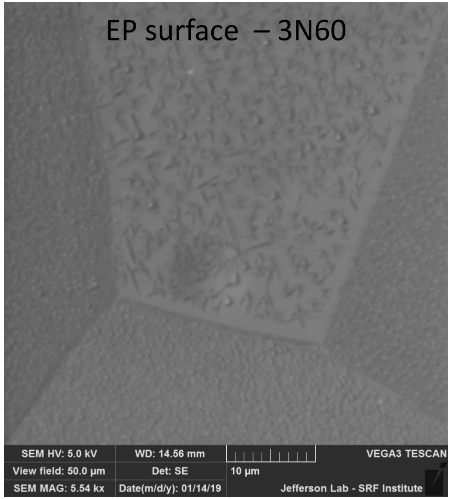
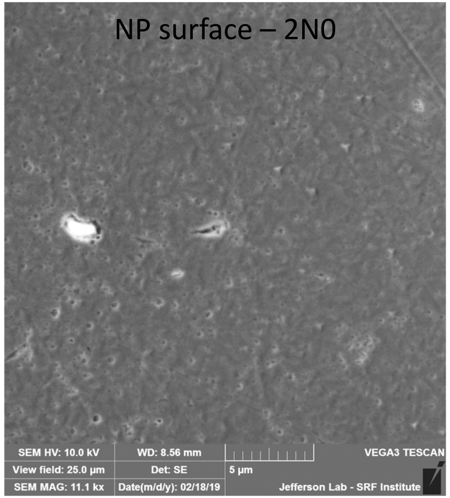
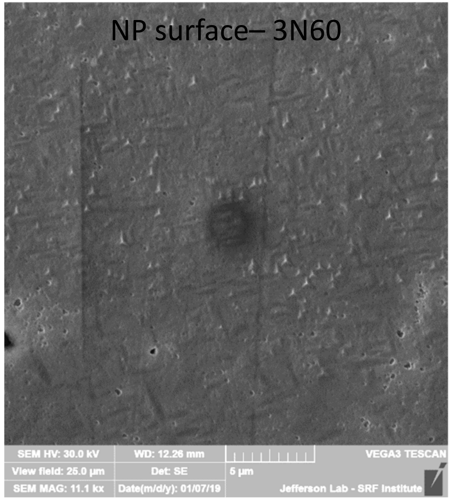
Nitride growth vs crystal structure and sample prep

- Nitrides grow during doping are highly grain dependent.
- Each specific grain orientation appears to change the size and density of the nitrides
- Nano-polished samples without additional EP severely restrict nitride
- SIMS data points on model – slide 10 - likely not, representative of the “real” cavity surface.

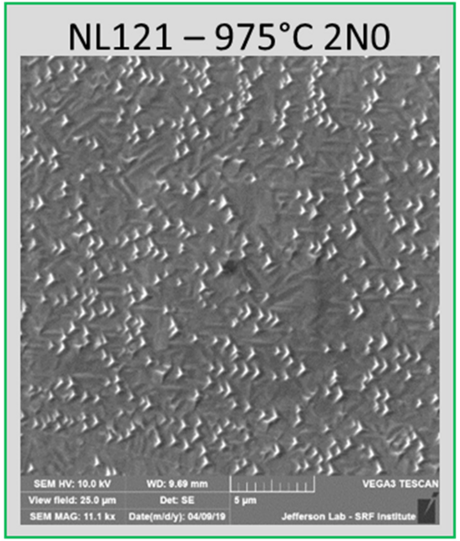


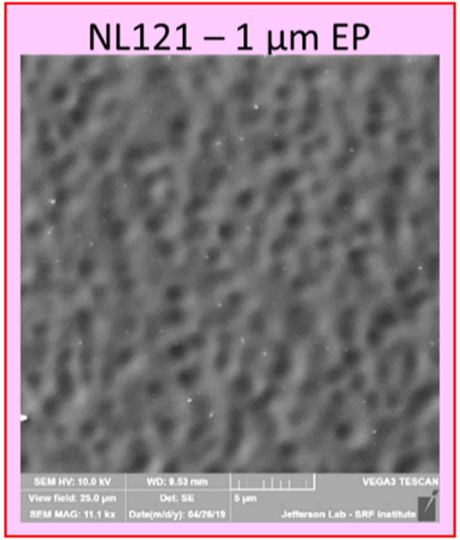


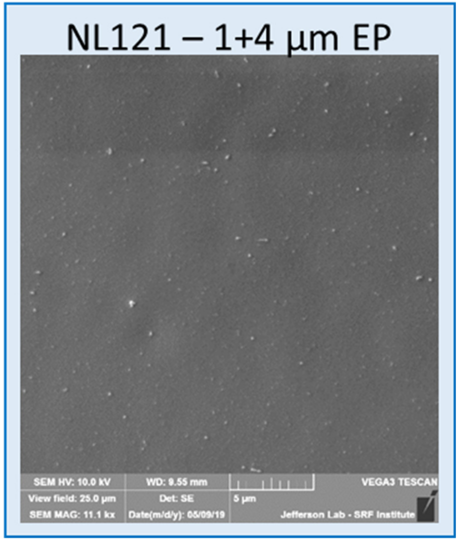




EP evolution - See J. K. Spradlin MOP030



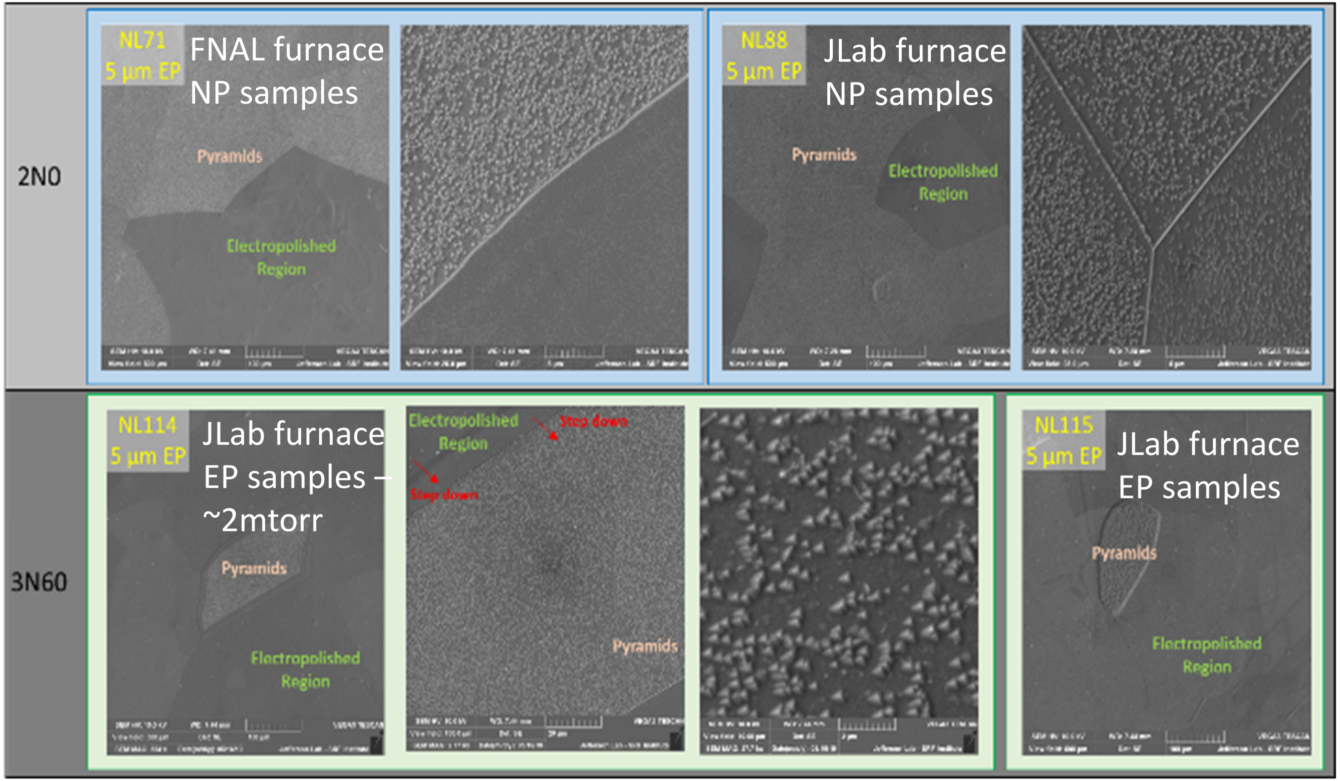




- Cathode to sample area 20:1 (cavity EP is ~1:10)
- Electro-polish performed at ~ room temperature

- Pit density is the same at nitride density (crystals may be carbides as well)
- EP appears to carve out surface crystal– “excavate”
- Defect free inter-grain EP appears to smooth out the surface on these samples, may not be very where.

Nitrides? after EP - See J. K. Spradlin MOP030



Post 5 µm EP, select grains show "Pyramids" ~ 5× smaller than standard nitrides.

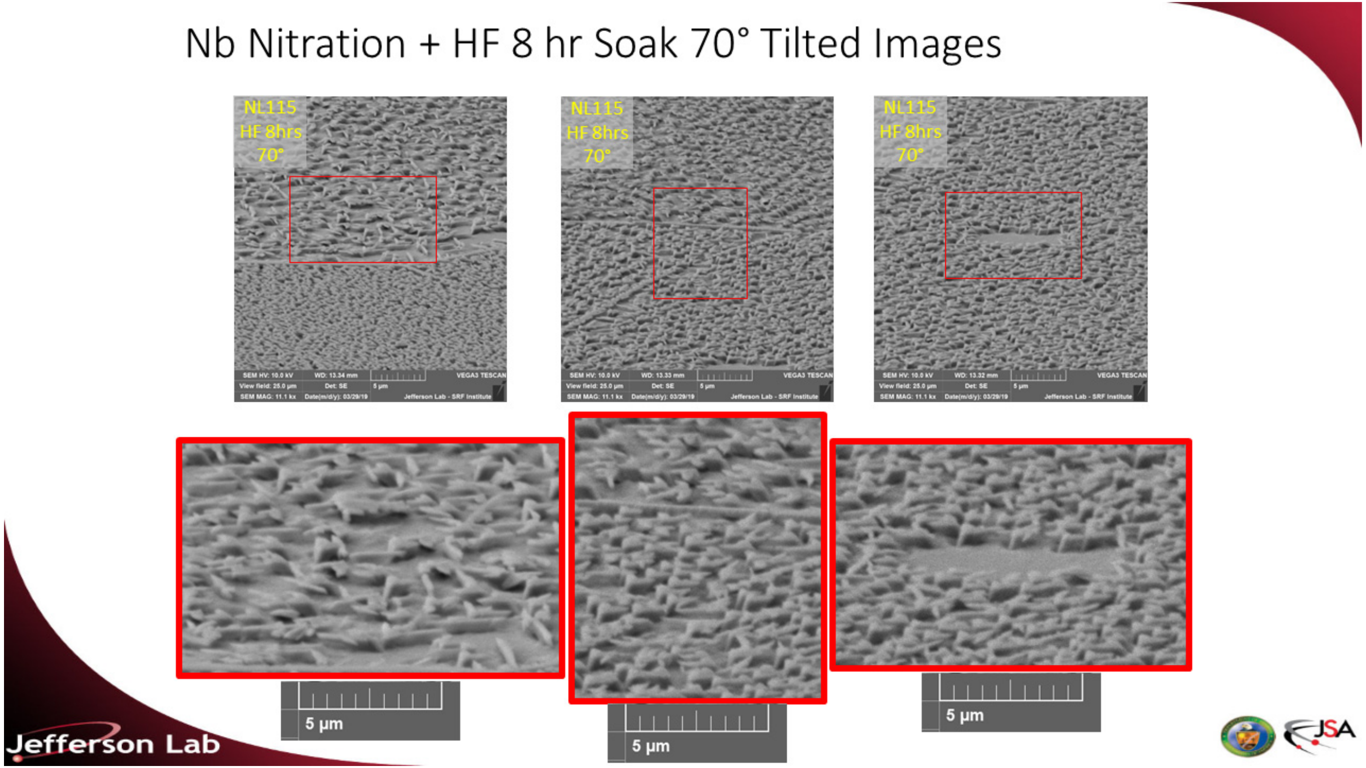
Do they grow below a thin top crystal and are only exposed after EP.

Do these Pyramids cause premature quench?

Do these pyramids cause Q-slope?

J. K. Spradlin, A. D. Palczewski, C. E. Reece, and H. Tian, "Analysis of Surface Nitrides Created During Doping" Heat Treatments of Niobium"", presented at the 19th Int. Conf. RF Superconductivity (SRF'19), Dresden, Germany, Jun.-Jul. 2019, paper MOP030.

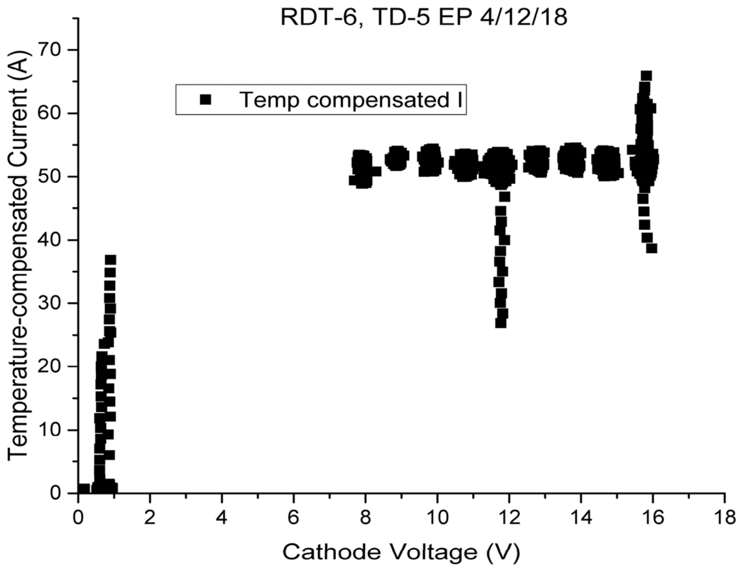
Nitride HF soaking – See J. K. Spradlin MOP030



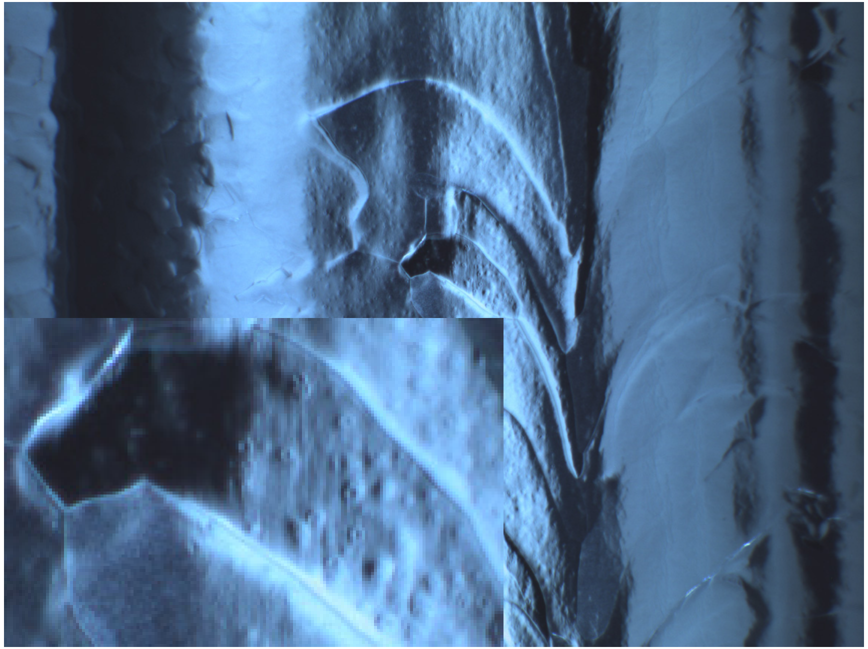
- HF soaking reveals nitrides above the surface plane.
- Nitride are barely affected by HF
- Etching of niobium between the nitrides strongly suggest the niobium's chemical potential
- EP close to the surface would have a different etching vs polishing plateau because of the nitrogen density. – see next slide

EP plateau for non-doped cavities - JLab TN-18-027 - and doped cavities

C. Reece, "Exploration of EP Plateau with two Single cell cavity Processing," *JLab Tech note,* no. TN-18-027, 2018.



Un-doped cavities EP'ed in the same way the image on the right. This EP has a polishing plateau down to at least 8V, ~6V theoretical – there should be zero pitting and very smooth surface



3N60 EP8 pitting while on non-doped cavity EP plateau – heavy nitrogenized niobium is not in the EP Plateau region (etching rather than polishing).

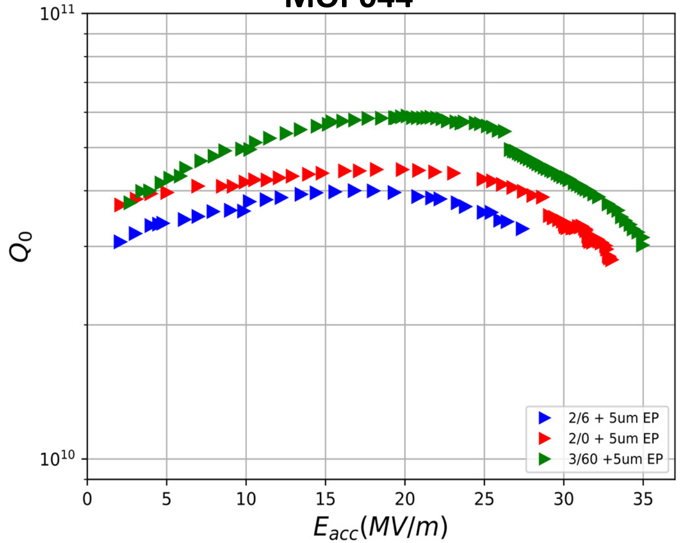
Interesting note about FNAL single cell EP studies – serendipitously key to higher quench fields?

For more details see *F. Furuta presentation TUPO022*

For more details see *D. Bafia's presentation* TUFUA4 and MOP044

<u>FNAL EP</u>

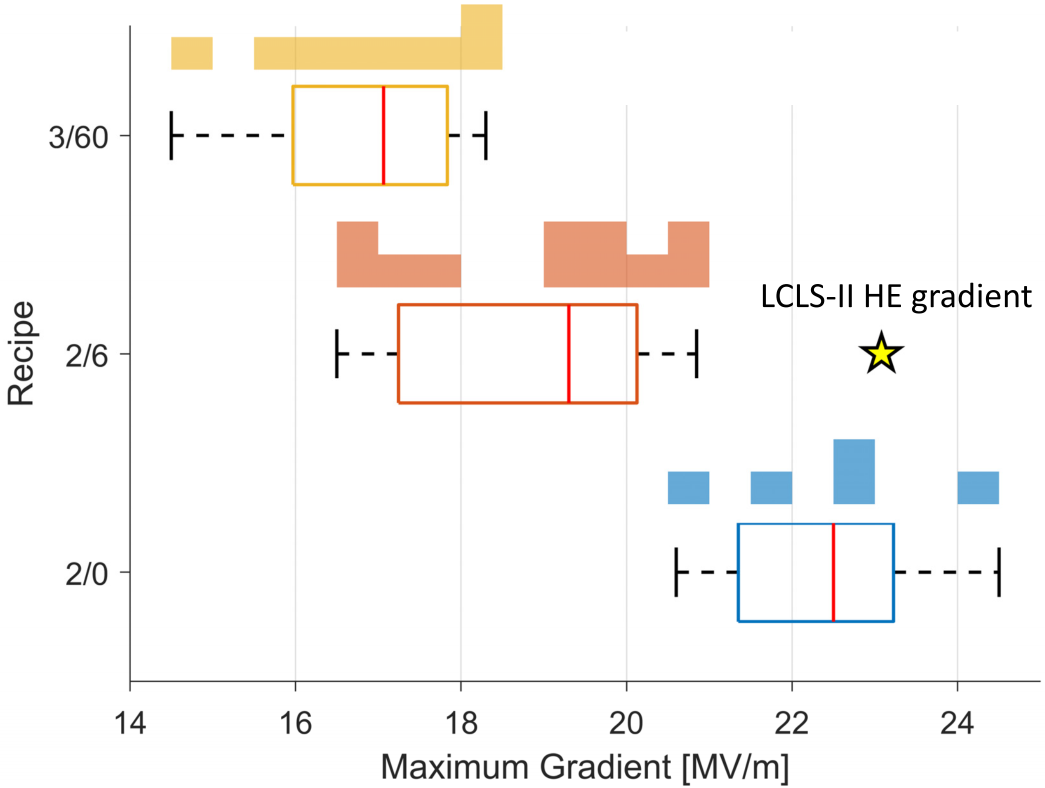
- Higher voltage than JLab/ANL and LCLS-II EP
- Colder than all others
- Lower current
- Longer EP



Doping comparison – using FNAL EP

FNAL's EP appears to improve high temperature doped cavity performance

LCLS-II HE 9 cell cavities – more details MOP045 Gonnella for group



- Zanon cavities heat treated at Research Instruments
- Many cavities heat treated at 975°C which we now know is incompatible with high gradient in RI's furnace*
- Nitrided niobium chemistry - 3/60 heavier surface doping (lowest quench), and 2/0 lowest surface doping (highest quench)

*This in not just RI each furnace will have its own characteristic maximum temperature

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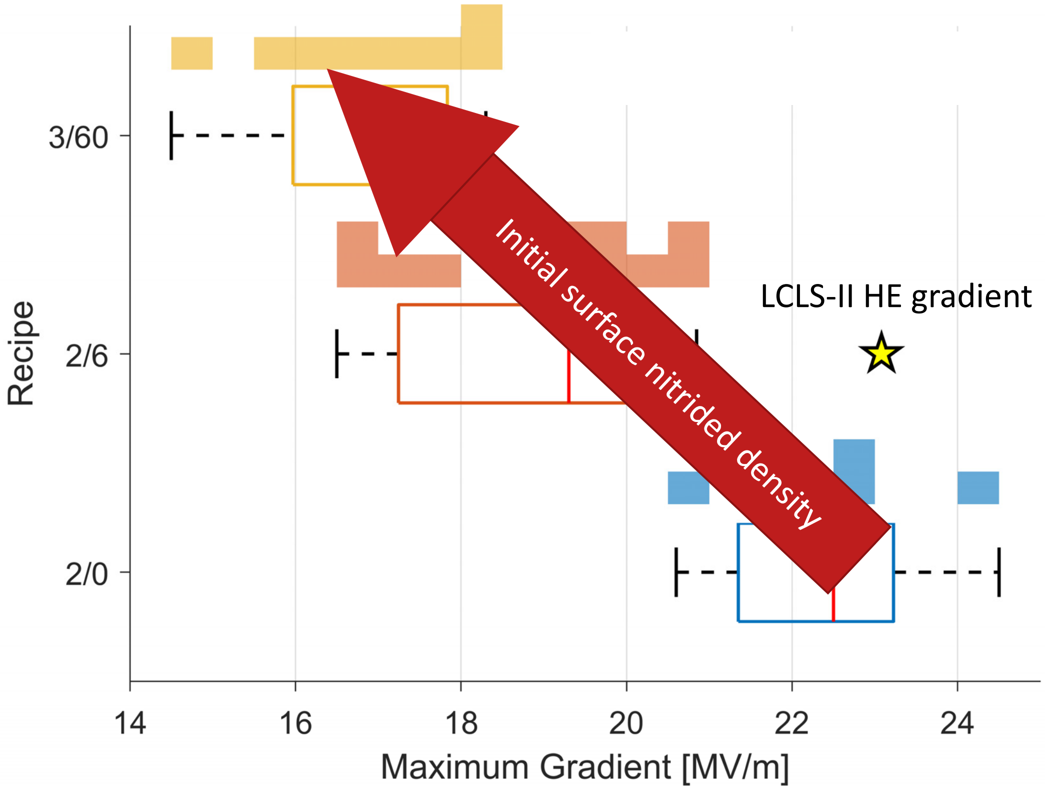

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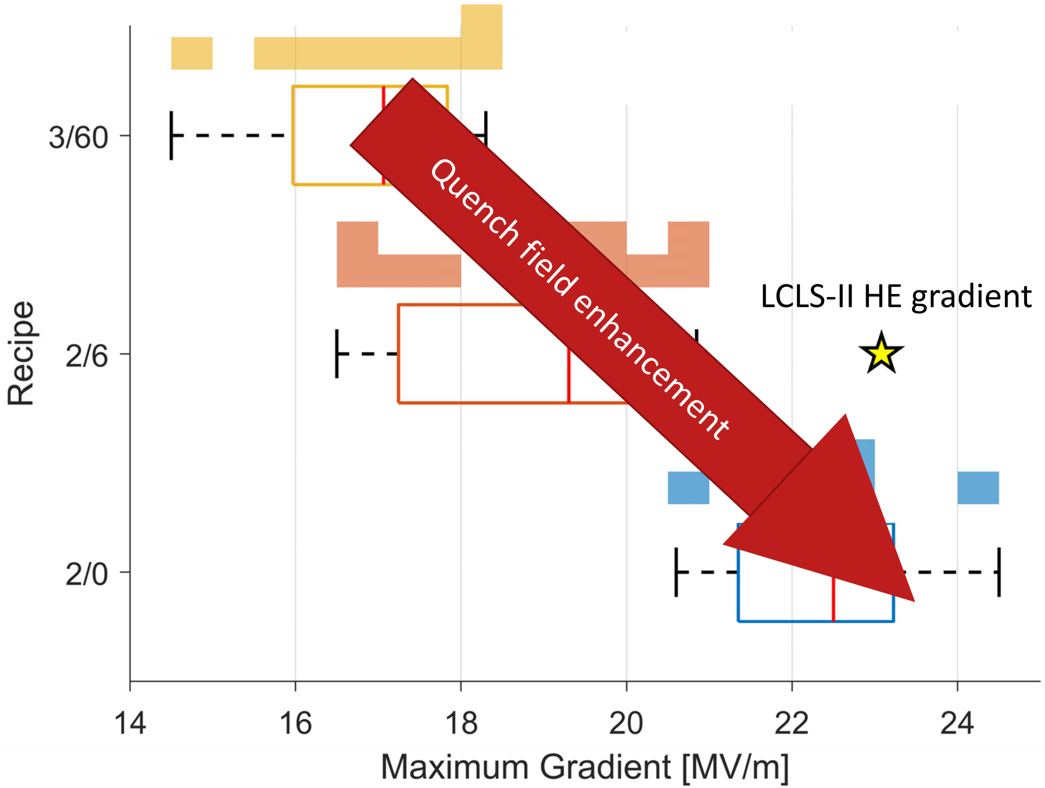

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Jefferson Lab

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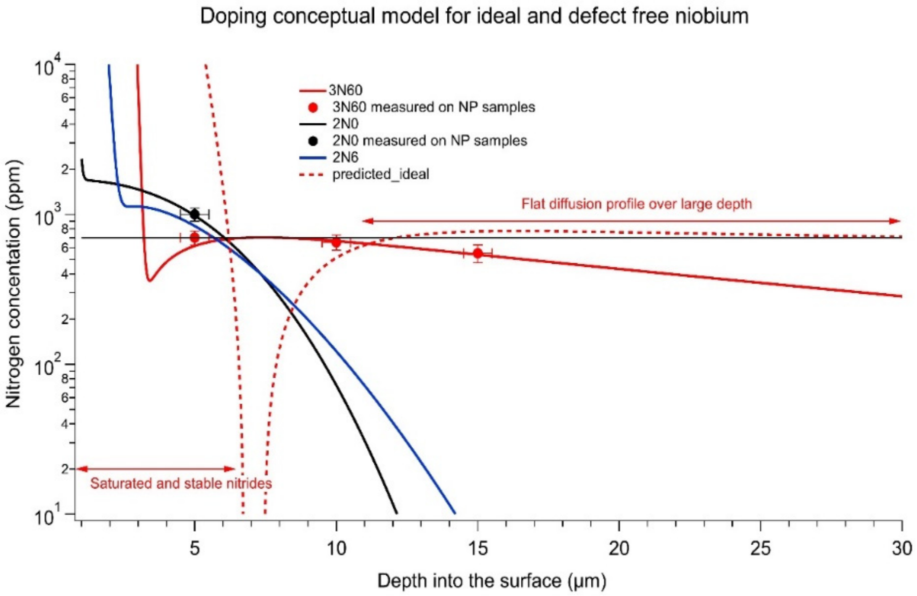
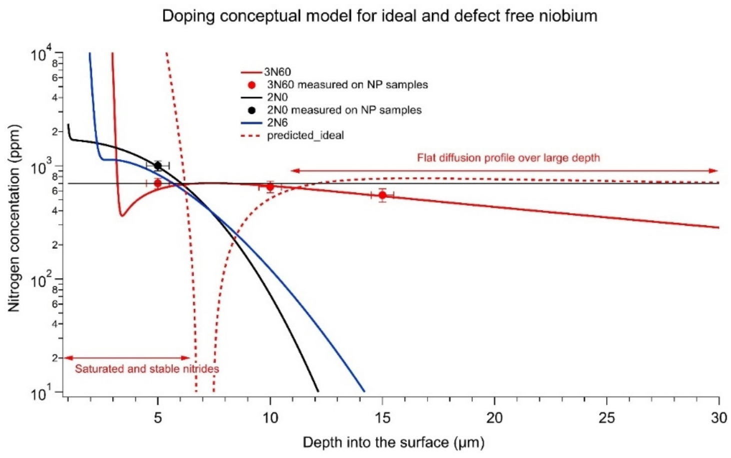
Model as of right now – still good?



- We think this type of model is still good
- Ideal doping yet to be found
- **But first**
  - Nitride chemistry must be fully understood – removal vs excavating.
  - Heavily nitrogen loaded niobium chemistry – EP plateau not fully understood.
  - Benign vs. troublesome furnace contamination levels must be understood – furnace dependent.

Path to maximizing quench field and Q0 in doped cavities

- Smoother pre-doping surface
  - Also requires better manufacturing.
- Higher voltage for post-doping EP.
- Lower temperature for post-doping EP.



Questions?

Ari Palczewski – SRF scientist

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- Funding partially provided by LCLS-II and LCLS-II HE
- Contributing information
  - Sample EP Hui Tian – Jlab
  - SEM images by: Joshua Spradlin – Jlab
  - Analysis by : Joshua Spradlin, Charlie Reece, and Ari Palczewski – Jlab
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  - Coordination between facilities and input from Michael Kelley – College of William & Mary and Virginia Tech