



## Summary for TTC WG 4

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21 April 2010

\*Opinions, interpretations presented here are my own

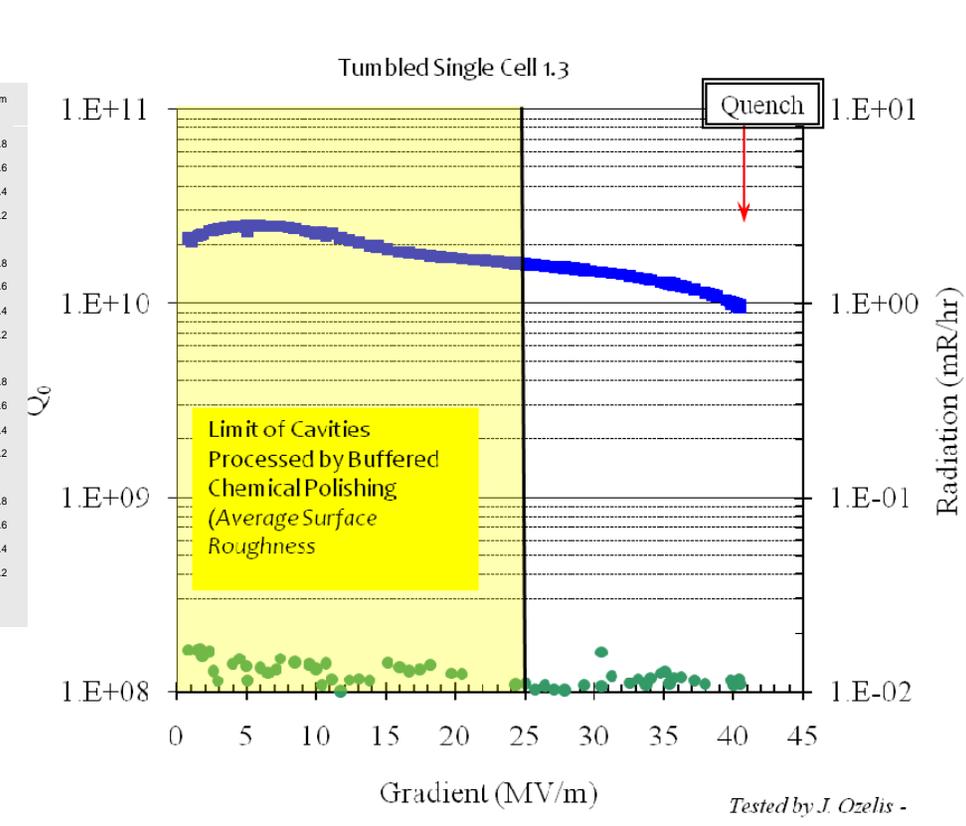
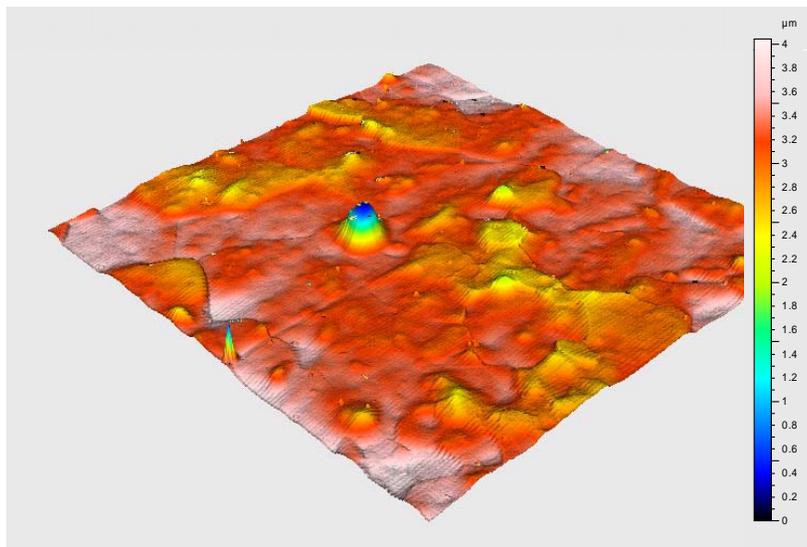
## Workshop at a glance

- **6<sup>th</sup> Workshop in a series started by P. Bauer, H. Edwards, G. Wu, C. Antoine**
- **56 Attendees**
- **8 Sessions**
  1. **Recent cavity results and drivers**
  2. **Recent coupon results and drivers**
  3. **Ideal limits to SRF**
  4. **Surface processing – bulk removal**
  5. **Surface processing – final processing, coating, and repair**
  6. **Q(E) and Rs measurements**
  7. **Forming and welding**
  8. **Alternate processes (films)**
- **<http://indico.fnal.gov/conferenceDisplay.py?confId=3118>**

# DRIVERS FROM CAVITY TEST RESULTS

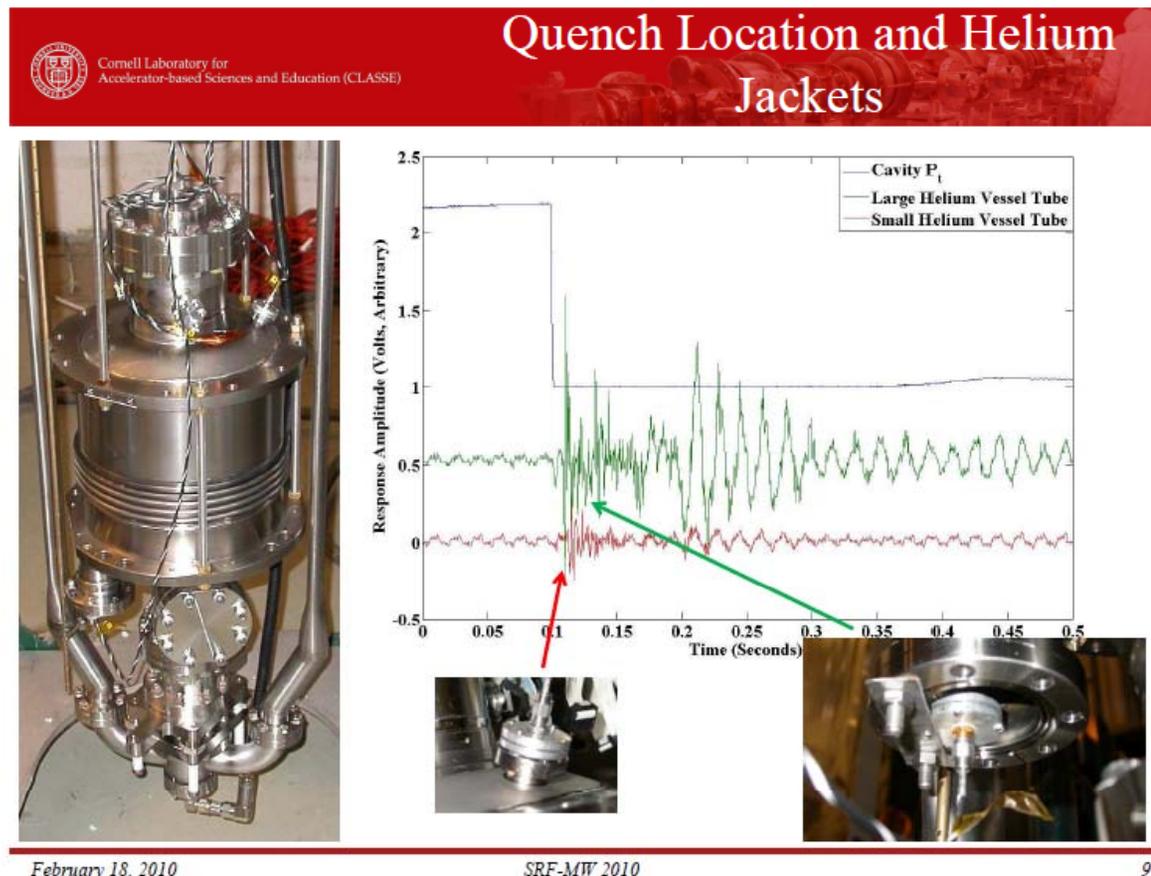
# 40 MV/m in tumbled cavity

- ...Yet the surface looks very rough. Does this imply that sub-surface chemistry is more important than topography?
  - C Cooper, session 4



# Locating quenches inside He jackets

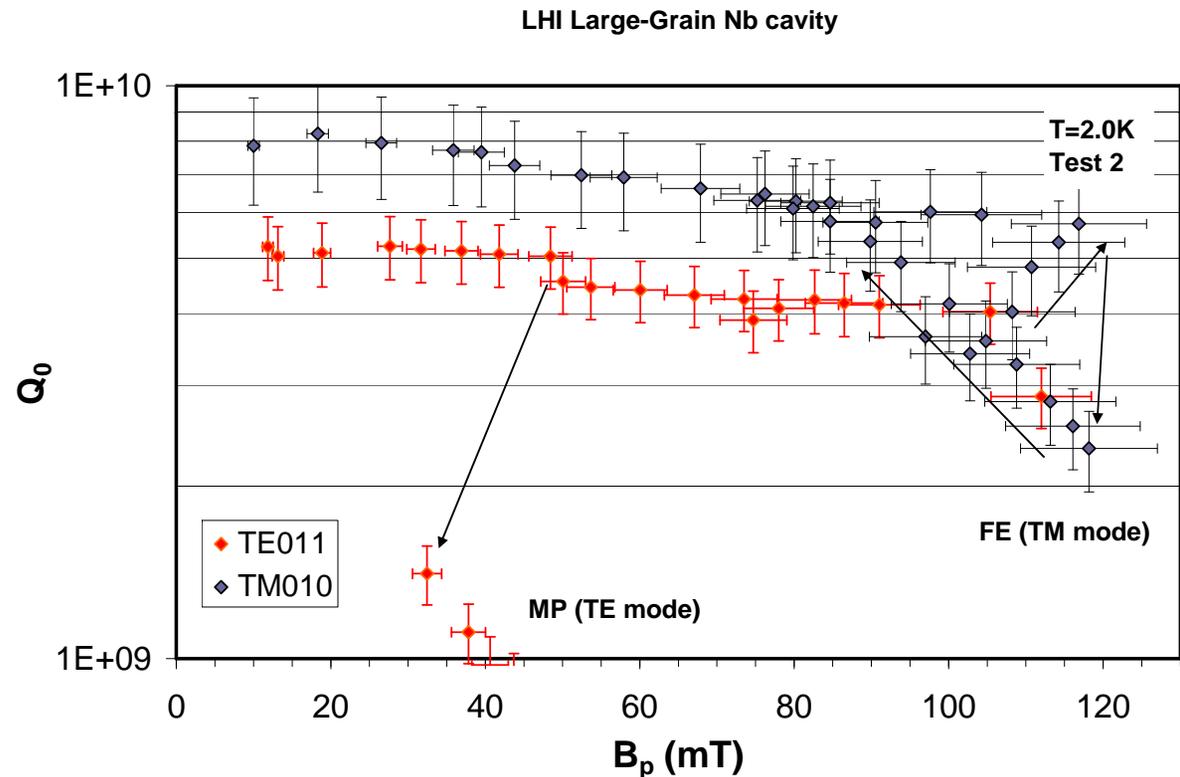
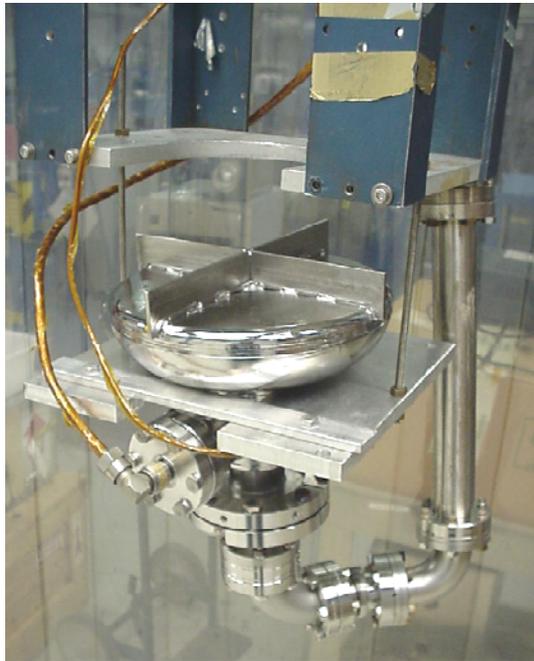
- Second sound works even in He jacket (Conway, §1)
- Can second sound replace T-mapping for other needs too?



# Laser-induced flux sweeping

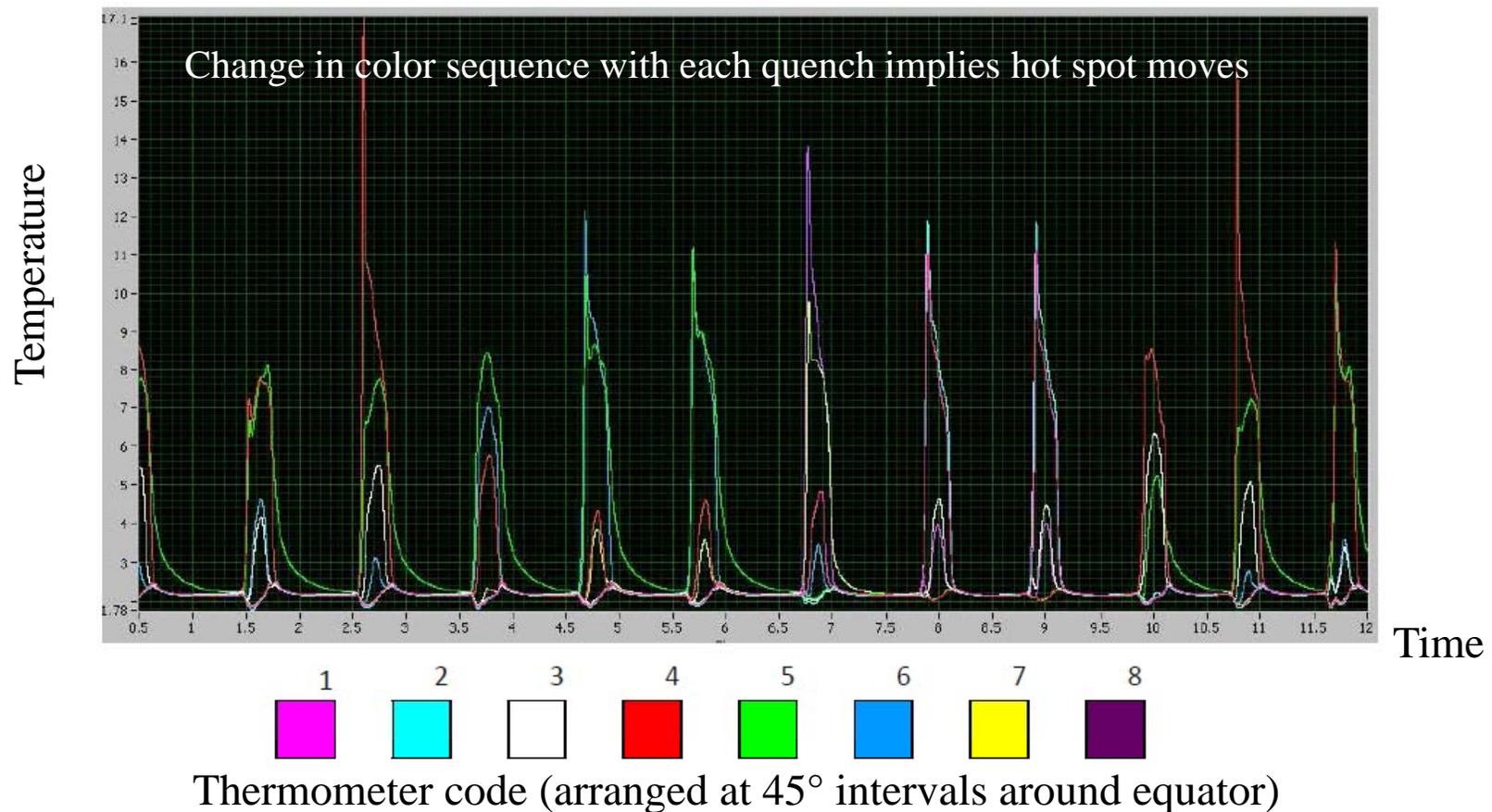
Ciovati, session 1 – FE and MP prevent evaluation of laser-induced flux sweeping

- To try: He processing, Operate in a different mode (for example  $TM_{020}$  at 2.66 GHz), Analyze MP trajectories with a 3D code



## Quench locations that move

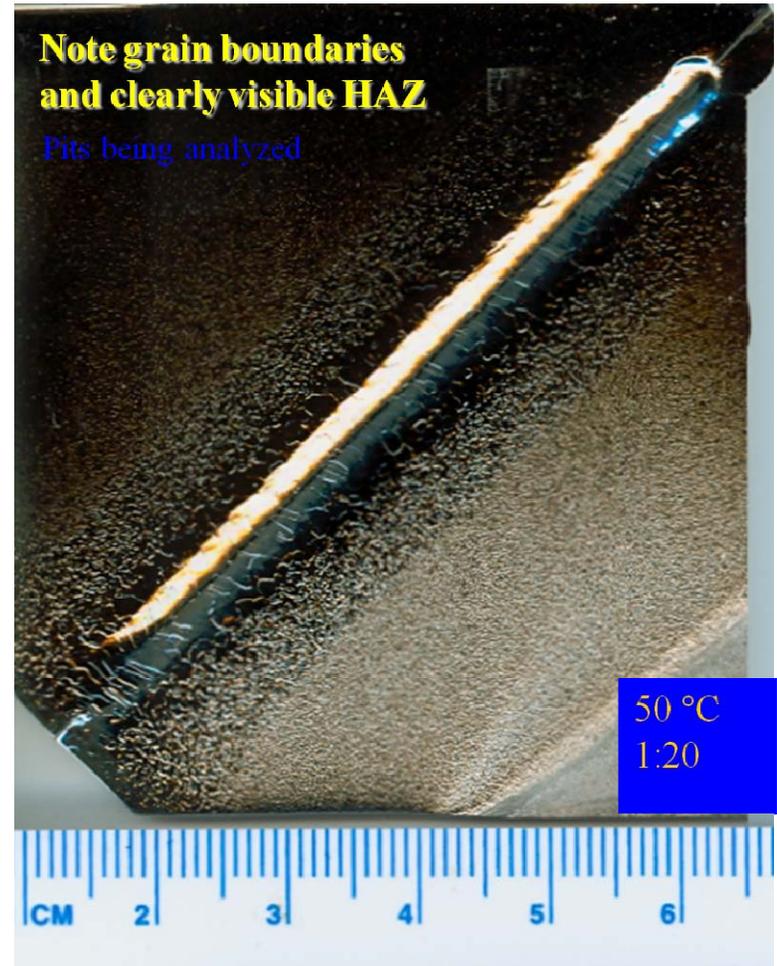
- Above 2.2 K, quench location moves. Do thermal gradients sweep flux around equator? (Sergatskov, session 1)



# DRIVERS FROM COUPON RESULTS

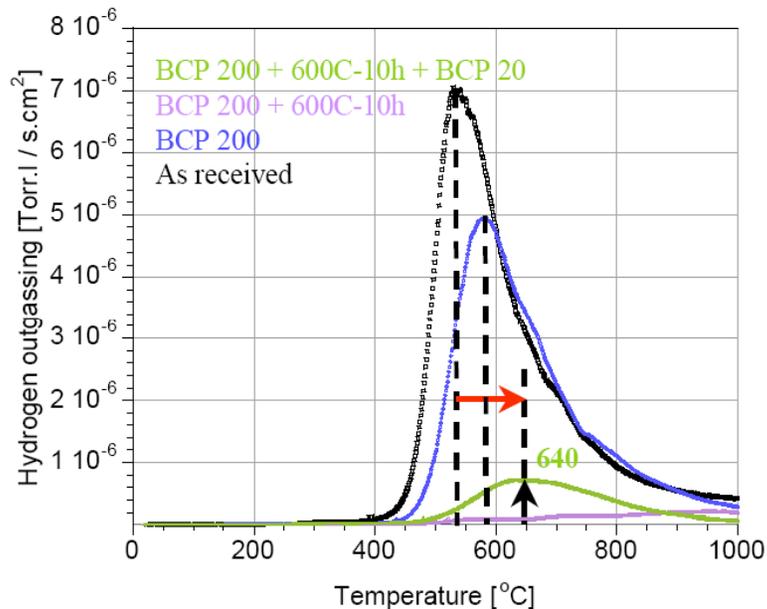
# Electrochemistry - Drivers from coupon results

- **First coupon EP that looks like cavity EP (Cooley, §2)**
  - Need 1:20 area ratio of cathode to anode
  - Need warmth (50 °C)
  - Need flow (1 L/min)
  - Unwanted reactions may then occur at cathode
  - Viscous layer might be removed
  - See Reece §3 talks – is electro-etching component present? If so, what is doing the etching?

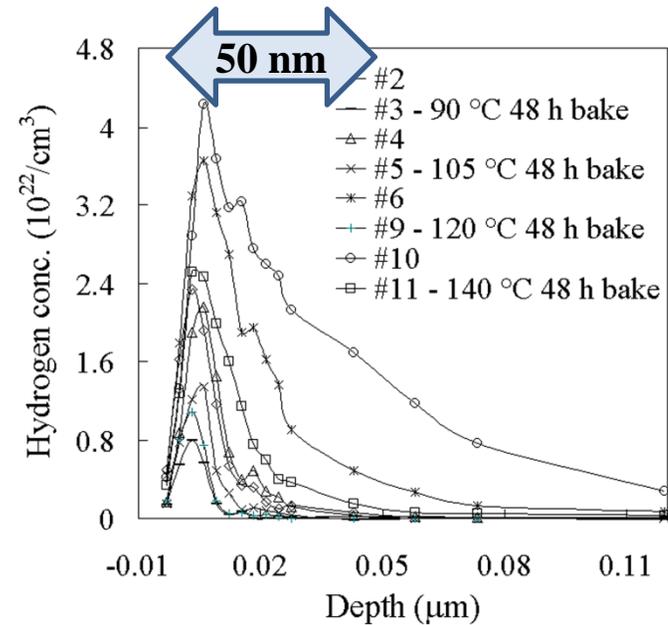


## Few words about Hydrogen

- **Bulk hydrogen vs. surface hydrogen (Ciovati, session 1):**



*P. Chiggiato, G. Chuste, I. Wervers, A.-M. Valente, JLab Technical Note, TN-09-056 (2009).*

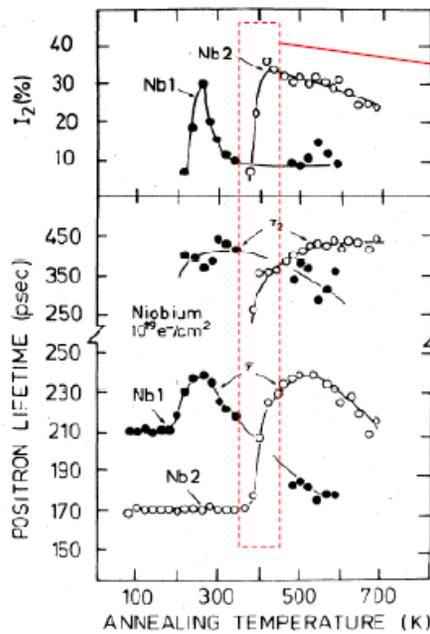


*G. Ciovati, J. Appl. Phys. 96, 1591 (2004)*

- 600 °C Bake not sufficient to remove H from surface (SIMS data); need 800 °C
  - **H presence correlates with performance reduction.**
  - **Is this tied to dislocation motion?**
- **Propose 800°C, 3 hr plus 120 °C, 12 hr in UHV with no final chemistry**

## Debate over 120°C baking (and implications for high-field Q-drop or quench)

- **It's not oxygen** (Romanenko, §2)
  - Baking works 100% of time for EP, less so for BCP
  - HF + HPR + air exposure do not remove benefits
  - No oxygen-enriched layers, oxides go away and come back like before bake
- **It's hydrogen + dislocations** (next slide)
  - Surface hydrogen?
  - *Need new probes for H*
- **It's oxygen** (Zasadzinski, §2)
  - Point-contact tunneling shows clear difference for material from a “hot spot” vs a “cold spot” from T-map
  - Grain boundary full of something magnetic (oxygen spin?)
  - Nb<sup>4+</sup> is evident from EPR
    - {121} defect
  - Does HF attack sub-oxides?
- **(2<sup>nd</sup> slide following)**

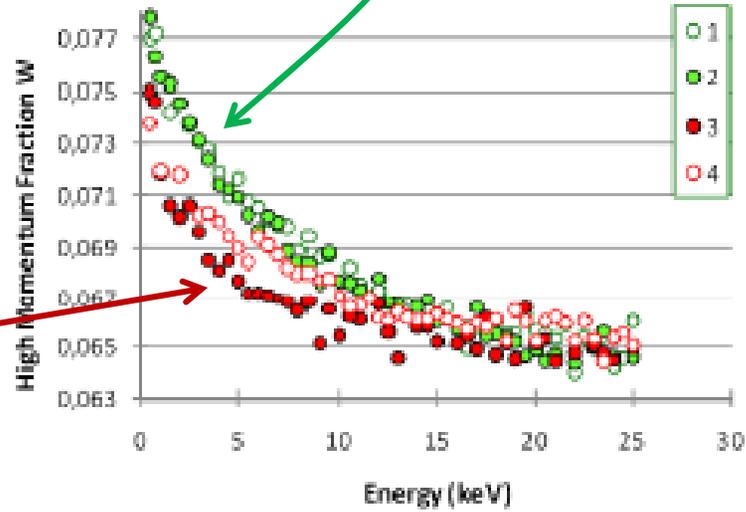


- Vac-H complexes in niobium containing some H (Nb2 in plots) dissociate at ~380K (107C) – compare to baking temperatures
- Mobile vacancies => dislocation climb becomes possible

*Positron annihilation study:  
After bake there is a high fraction of mobile vacancies because H is unbound from them. Dislocations can then move.*

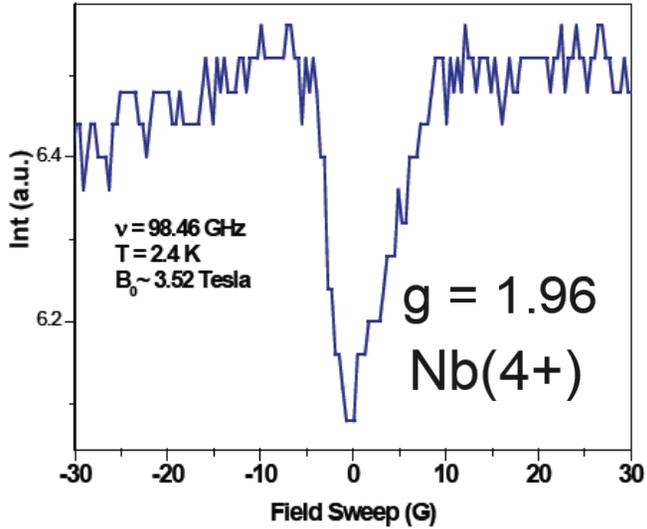
[P. Hautojarvi et al., Phys. Rev. B., Vol.35, Num.7, 1985]

*Before bake vacancies are bound with H, so they cannot move and will not permit dislocations to move, either!*

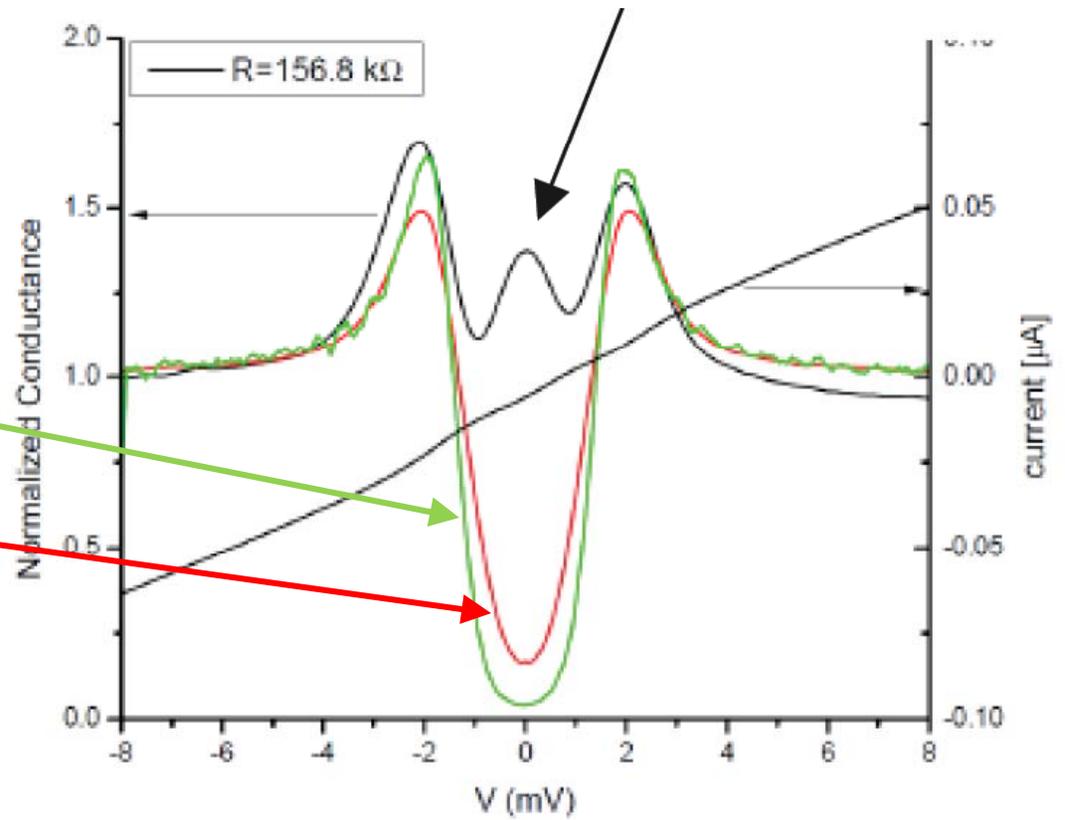


B. Visentin et al., Proceedings of SRF'09

Niobium Powder Electron Paramagnetic Resonance (not baked)



**Hot spot, near grain boundary**  
(Conduction at 0 mV is due to broken Cooper pairs)



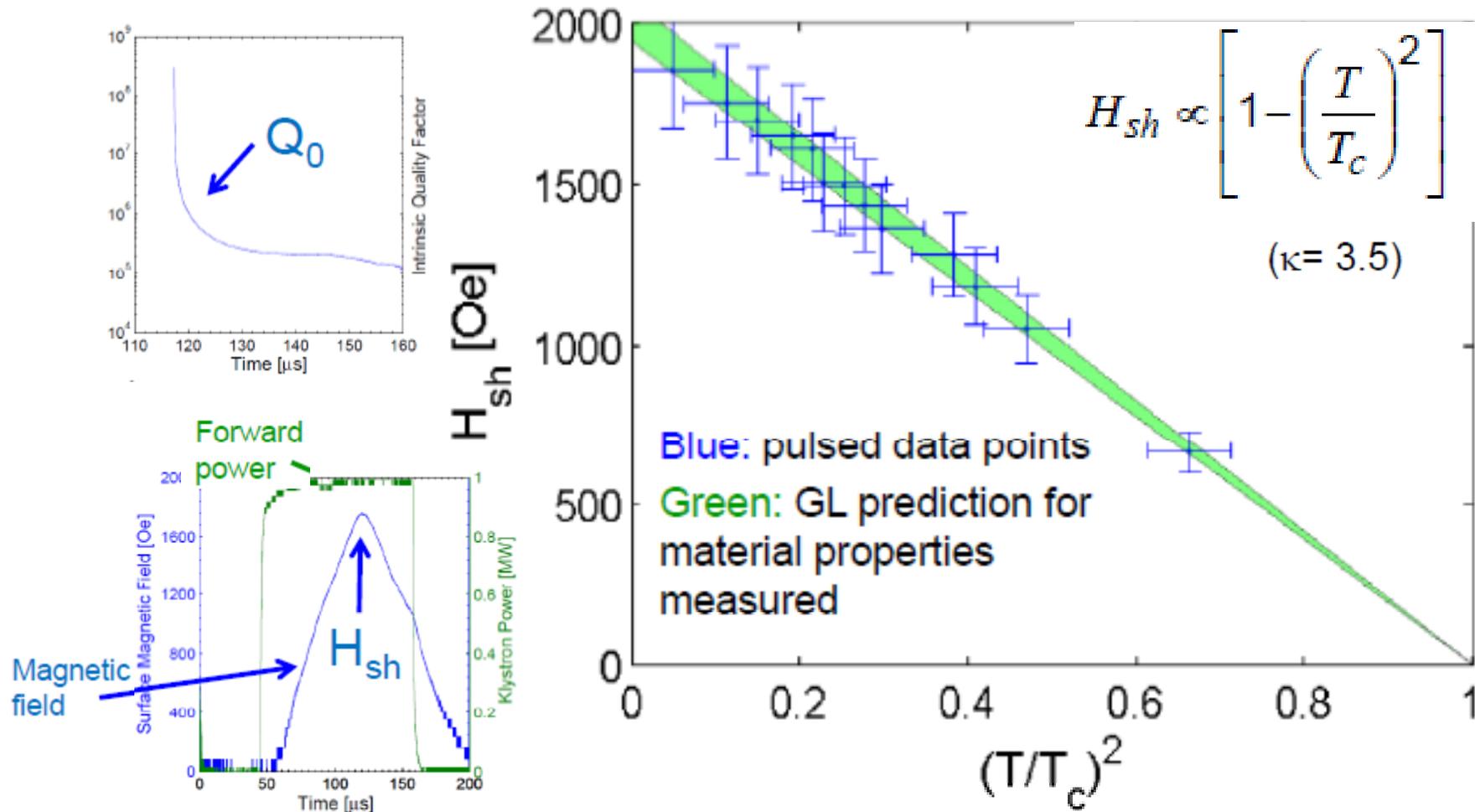
Hot spot, far from grain boundary

Cold spot, near grain boundary

# IDEAL LIMITS

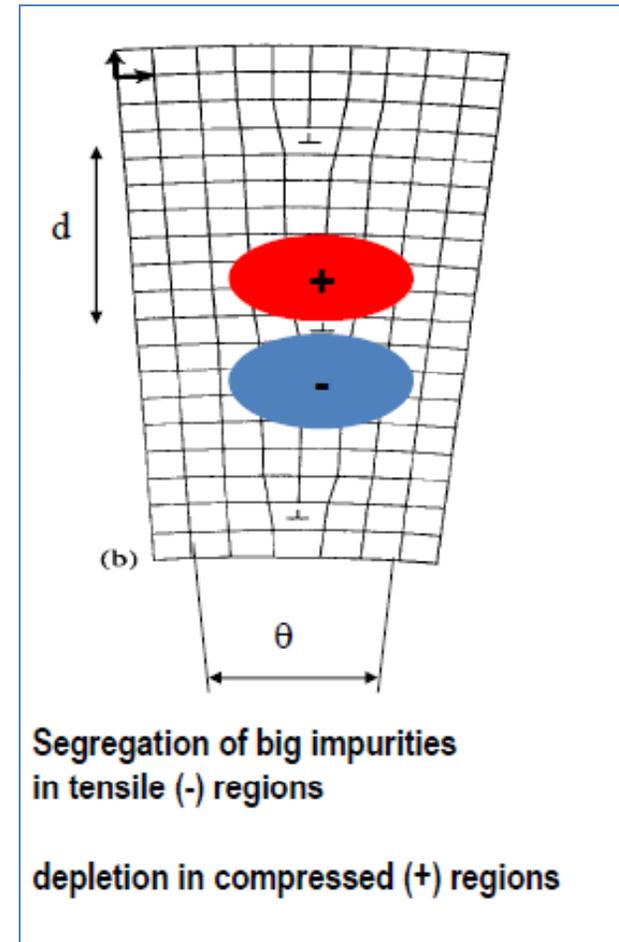
# Pulsed tests – $H_{sh}$ limit was verified

Matthias Liepe, 6th SRF Materials workshop, Tallahassee

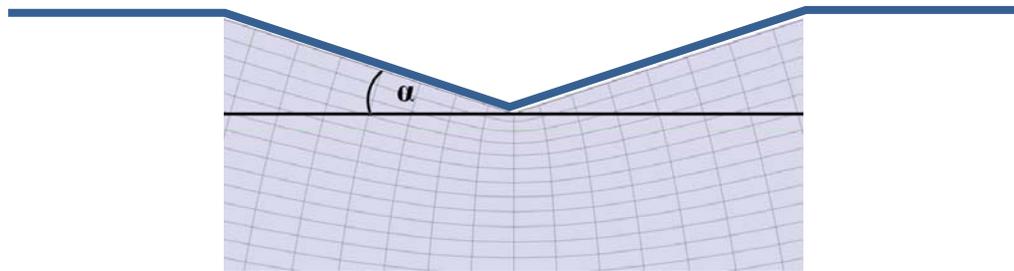


# Cottrell Atmospheres

- Impurity “clouds” around defects
- If hydrogen, then we must re-tool our surface science approach
  - NMR
  - $\mu$  - SR
  - Positron annihilation
- GB triple points can be collectors
  - Dislocations in Nb tend to pile up
  - Clouds follow
- Thus, some GBs can be benign, some can limit RF current!

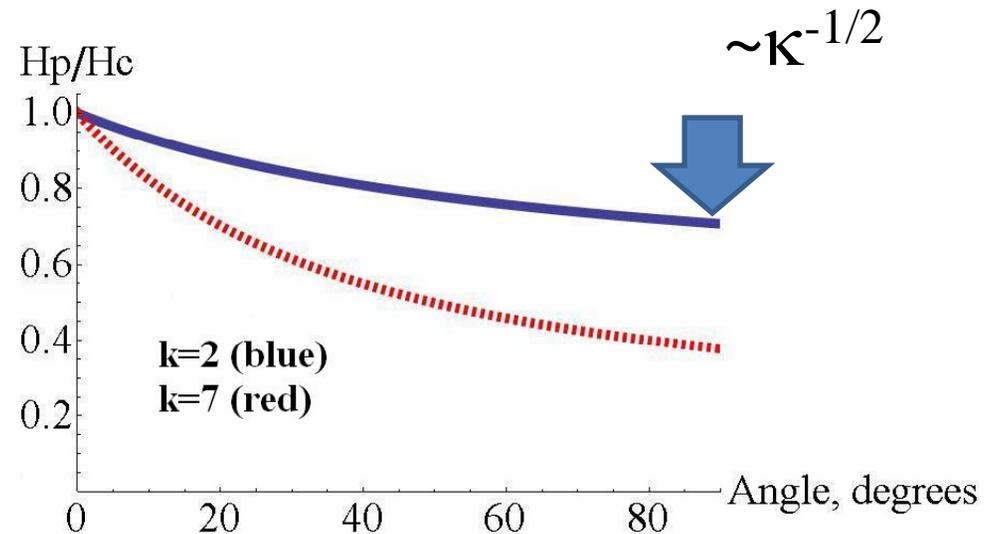
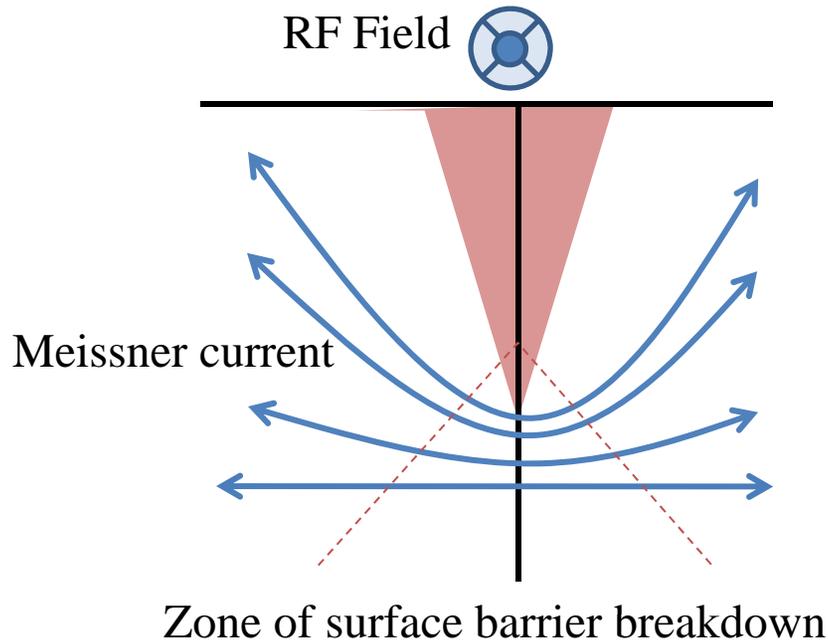


# Onset of Q-drop due to flux penetration at grooves



A. Dzyuba, FNAL

$$H_{pen} = \left( \frac{\xi}{\lambda} \right)^{1 - \frac{\pi}{\pi + 2\alpha}} H_{crit}$$



## Other fundamental topics

- **Pinning interactions cannot overcome Meissner current until 300-400 nm depth**
  - Surface zones have oscillating flux
  - Sweeping effects require deeper thermal gradients
- **Non-linear Meissner effect: current can suppress gap**
  - Now has been measured at 20 GHz PRB 81, 020504R
- **Optimization for low  $R_s$  is different than that for high E**

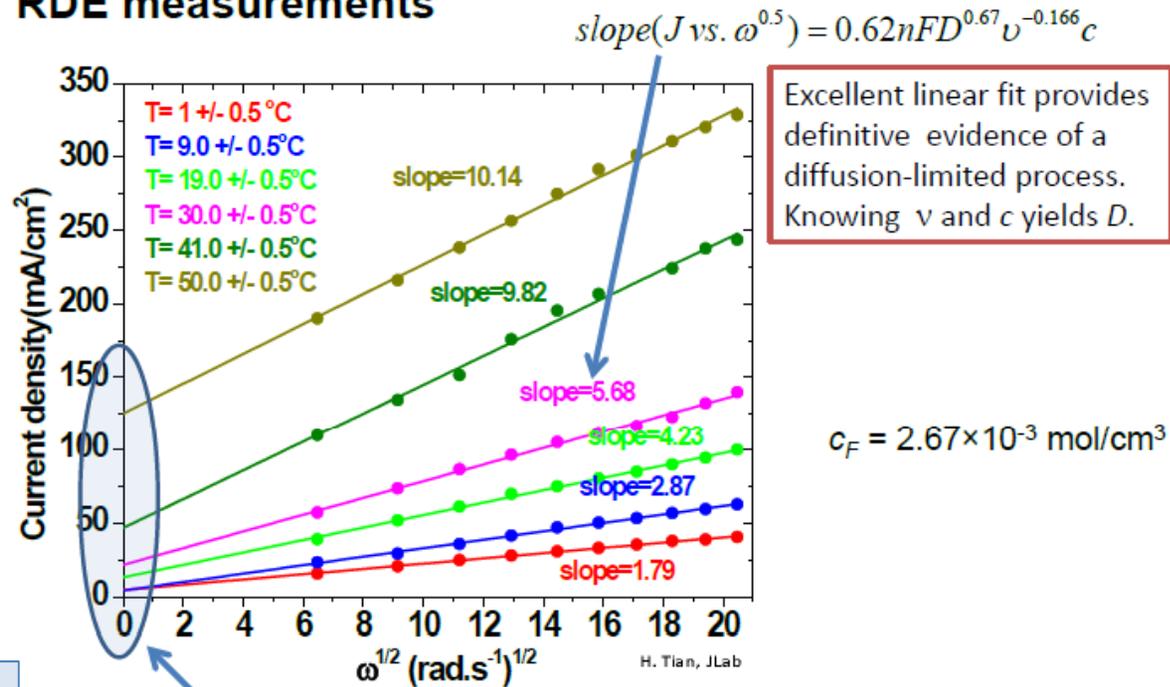
# PROCESSING R&D AND REPAIR

# Two electrochemical processes

- Tian, Reece

Diffusive

RDE measurements



Convective?!

Strong evidence for temperature-dependent electrochemical etching in parallel with the diffusion-limited process. For analysis, we must separate these current contributions.

# Control of EP temperature

## Horizontal EP

- **Present: control  $T$  by controlling flow, but 1 Hz temperature swings remain**
  - “Stable, but hot” – Reece
  - Electrolyte viscosity falls by 2x for 20°C rise!
- **Difficult to control  $T$  by controlling voltage**
- **Water spray works well!**

**Action: Apply external cooling to horizontal EP**

## Vertical EP

- **Water spray works well!**
- **Present: top-bottom asymmetry**
- **Paddles, screws, other strategies to circulate electrolyte**
- **Also cavity flip**

# Repair by laser re-melting

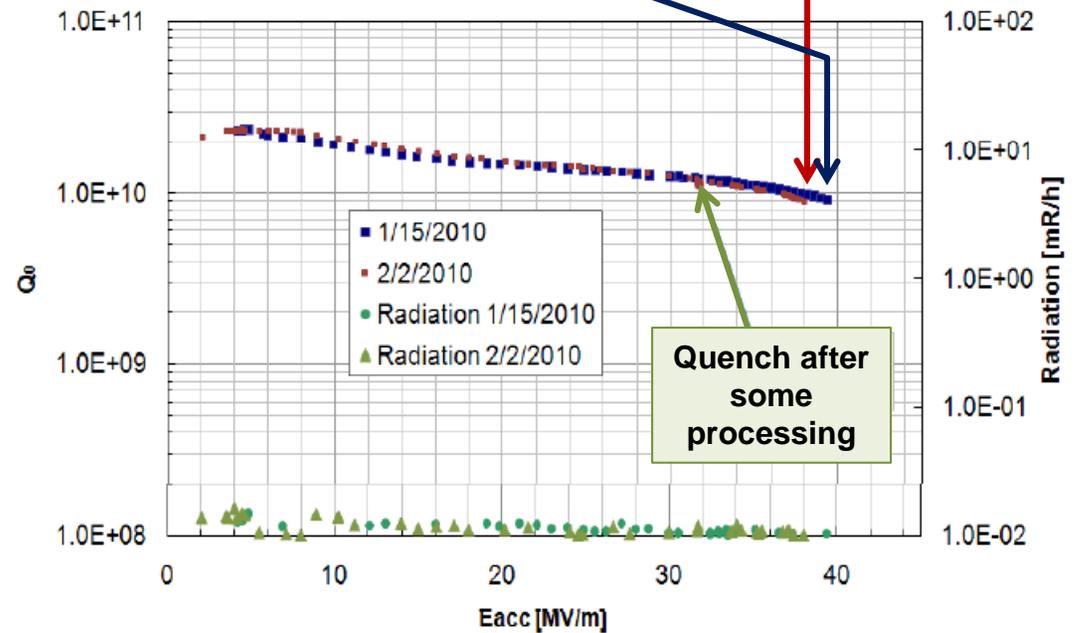
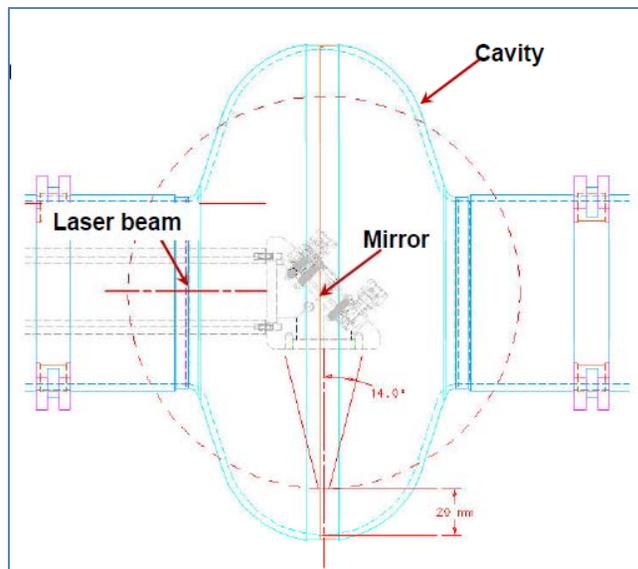
- **First attempts restored max gradient (Ge, §5)**
  - Quench location remains at repaired spot
  - Argon purity can improve



The Pit before re-melting



After re-melting



## Alternate processing

- **Tumbling + light EP works well, 9-cell experiments soon**
- **Non-HF processing ideas ripe for support – tools designed, processes laid out**
  - Faradayic electropolishing – large pulsed currents
  - Lactic acid, sulfonic acid, other electrolytes
  - Chemical-mechanical processing
  - Jet-slurry polishing
- **New film deposition routes**
  - Self-sustained metal plasmas
  - Other ALD

# NEW FACILITIES PROCESSING AND TESTING

# New Facilities

## Processing

- **JLab – integrated cavity processing facility**
  - Vertical processing, cavity never moves from fixture, services are brought to cavity
- **FNAL – integrated cavity processing apparatus**
  - 1-cell R&D

## Testing

- **Coupon Q(E)**
  - SLAC is operating “mushroom” style cavity
  - Texas A&M and Jlab have sapphire resonators
  - Maryland has point-RF probes and laser-scanning RF experiment
- **Proposed**
  - Dual laser interferometry plus re-melting
  - Surface resistance measurement

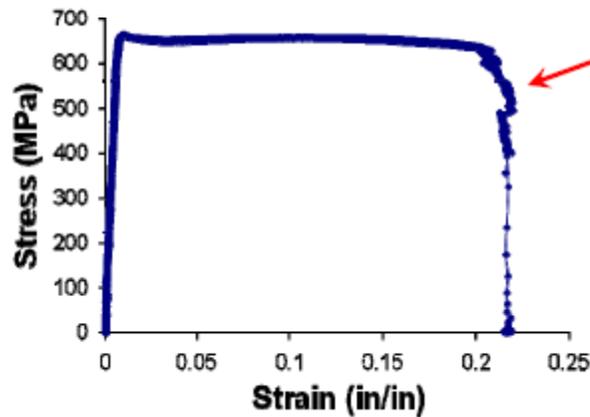
# RAW MATERIALS

# Niobium

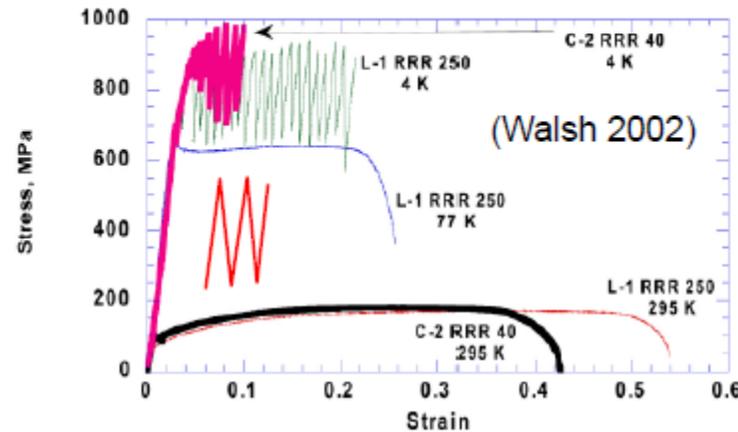
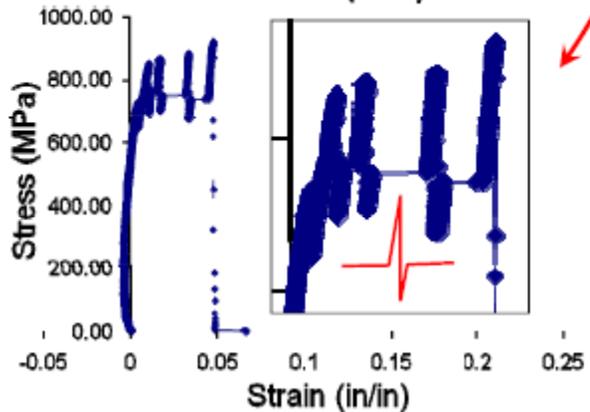
- **Should we modify the specification?**
  - 2% flatness, RRR = 300, ASTM 6 interfere with each other
  - No batch is the same as any previous batch
- **Stockpiles of pieces, end groups, formed items might be useful**
- **Tubes for hydroforming are now a product offered by vendors!**
  - 140 mm OD, 1.2 m long, 3-6 mm wall
  - ECAE of 6" tubes may be possible soon
- **Special textures can be prepared, might be interesting**
- **Excessive cold work can be applied during forming**
  - Does this lead to trouble later?

# Nb testing at 4.2 K

Polycrystalline samples deformed at 77 and 4K agree with data from literature



- High RRR Nb has same yield and flow behavior at 77 K
- At 4K, Sample showed 5 instances of jerky flow before fracture, at lower strain than samples from literature



## Conclusions - Recommendations

- Pay as much attention to sub-surface contamination as topography, especially impurity clouds at dislocations and grain boundaries
- Understand hydrogen as much as we now understand oxygen (and understand oxygen better, too)
  - Re-tool surface science for hydrogen
- Water cool the outside of cavities during EP
  - Two electrochemical processes, maybe one is convective!
  - Decouple temperature control from acid flow
- Several repair routes are feasible
- Continue progress on alternate processing and alternate forming
- Relax the Nb spec?