SRF Materials

Perspectives on what we have learned
Pathways forward for linear colliders

Lance Cooley – Head, SRF Materials Group, Fermilab

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Acknowledgments:

M. Champion, C. Cooper, A. Dzyuba, D. Ford, C. Ginsburg, D. Hicks, R. Kephart, J. Kerby, T. Prolier, C. Reece, A. Romanenko, A. Rowe, D. Sergatskov, C. Thompson, E. Toropov, J. Zasadzinski

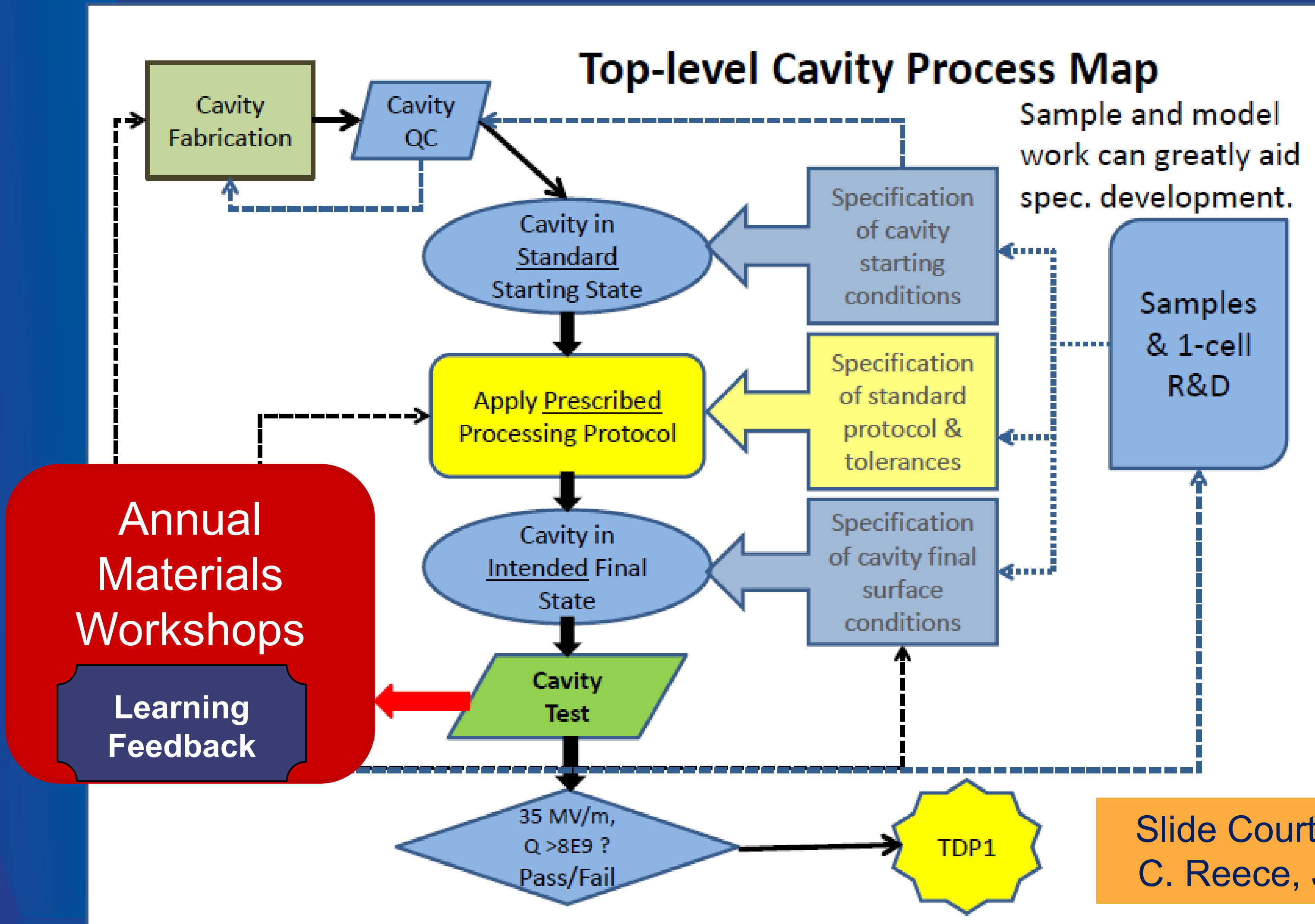
U.S. DEPARTMENT OF ENERGY

Fermilab

Goals of SRF Materials R&D

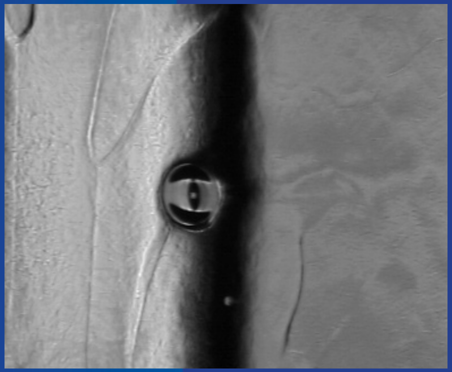
- Provide understanding beneath the baseline industrial process used to make SRF cavities
  - Validate or recommend modification of present steps
  - Specify requirements for success at each step
  - Integrate steps to serve the final RF surface
- Explore alternate routes
  - Higher gradient and higher Q
  - Simplify, reduce cost, increase yield
  - Reduce hazards, lessen environmental impact
- Conduct basic materials science
  - What is the ideal topography, structure, composition?
  - What is actually made?
  - How do real surfaces change properties?

Improving the present industry baseline



Slide Courtesy of C. Reece, J-Lab

FNAL activities to improve the baseline

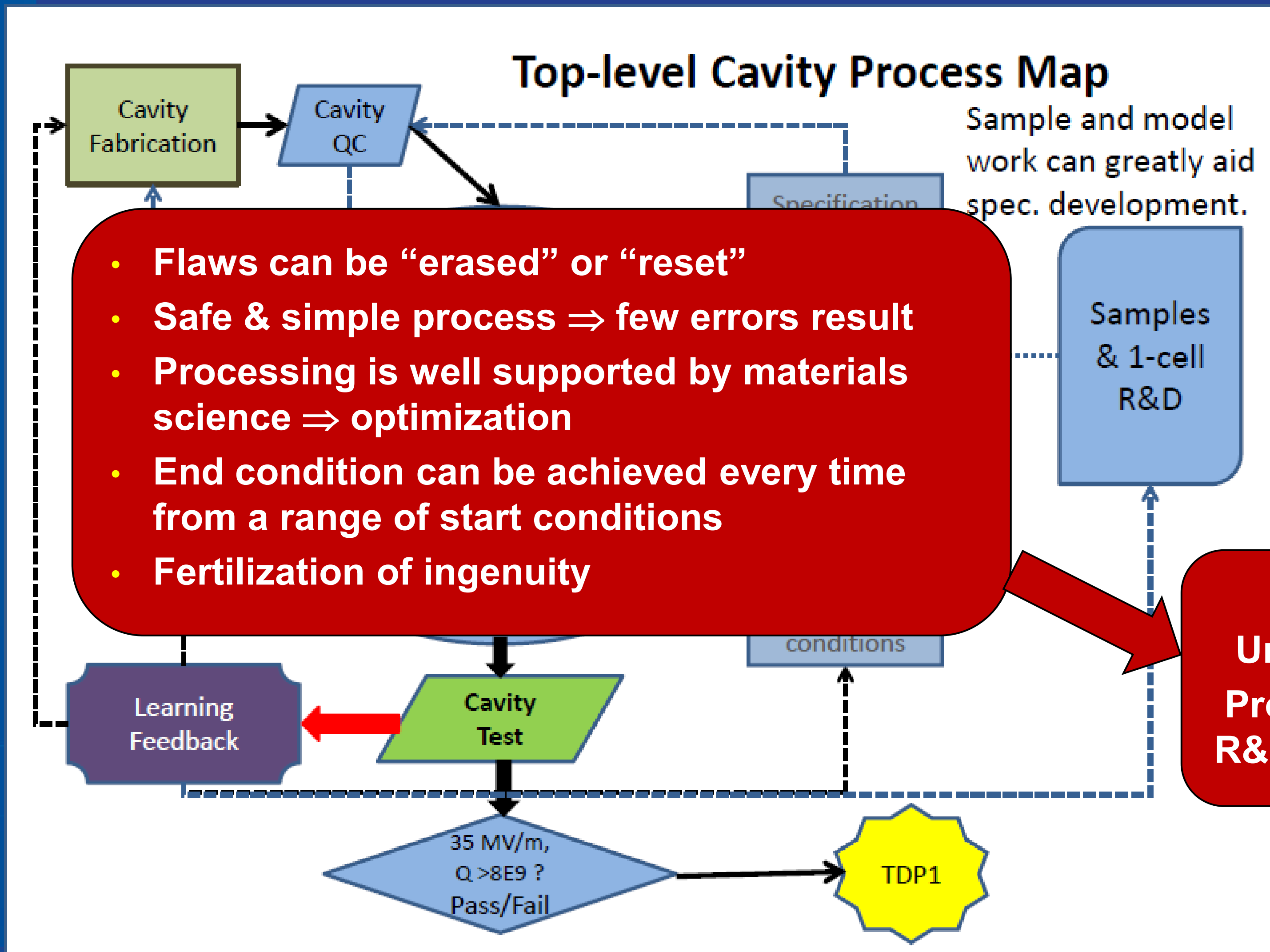


Did the cavity weld form a bubble?

Or, is it an etch pit?

- Annealing to prevent defects in the weld heat-affected zone
  - Stress relief / recovery anneal before EP
    - 10 single-cell cavities in fabrication now
  - Hydrogen de-gas after weld-prep BCP
    - Materials science motivates this, too
- Fine-tune electropolishing (EP) steps
  - Custom built single-cell R&D apparatus
  - Use external cooling instead of acid flow as the primary cooling method
- Local repair by melting with a laser

Alternate processes that may be more forgiving, adaptable, simpler, cheaper



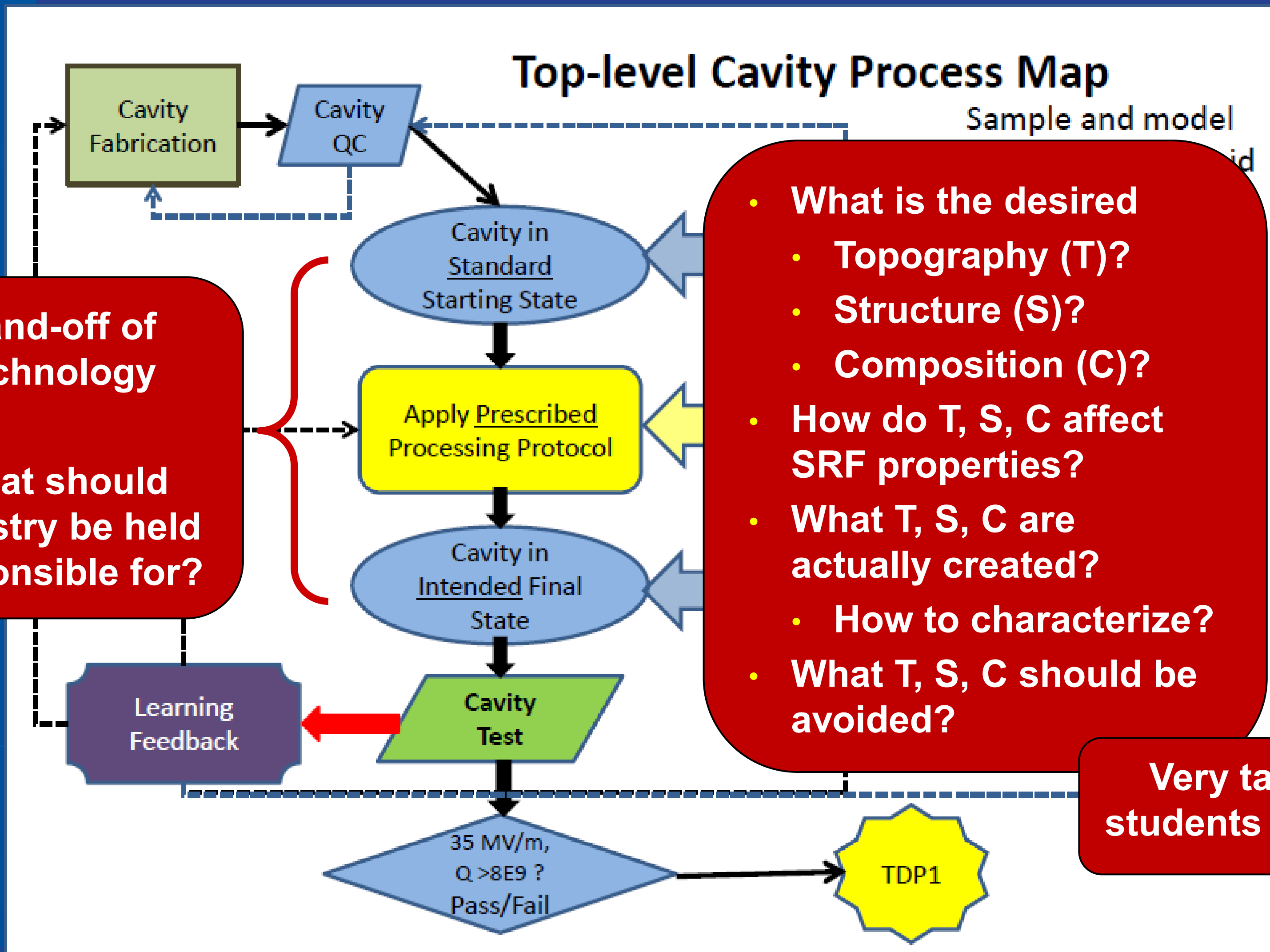
- **Flaws can be “erased” or “reset”**
- **Safe & simple process ⇒ few errors result**
- **Processing is well supported by materials science ⇒ optimization**
- **End condition can be achieved every time from a range of start conditions**
- **Fertilization of ingenuity**

SBIRs
Univ. R&D
Processing R&D at Labs

Promising Alternate Processes

- Cavity Hydroforming and Nb Tube R&D
  - Need a coordinated program
- Extended CBP / tumbling / mechanical polishing
  - Transformational — Very smooth surfaces, forgives / repairs flaws, low-tech, eco-friendly
  - Can the process be completely free from toxic acids?
  - Adapt polishing to other cavity shapes?
- High-power deposition of films
  - Recent breakthroughs bring RRR up above 300

Topics of basic materials science



Hand-off of technology

What should industry be held responsible for?

- **What is the desired**
  - **Topography (T)?**
  - **Structure (S)?**
  - **Composition (C)?**
- **How do T, S, C affect SRF properties?**
- **What T, S, C are actually created?**
  - **How to characterize?**
- **What T, S, C should be avoided?**

Very talented students & Ph.D.s

Ideas motivated by materials science

- Dry cleaning techniques
  - Plasma cleaning just after 800 °C bake
- Protective coatings
  - Develop an atomic layer deposition process that integrates with regular cavity processing
- Hydrogen control strategies
  - Large-grain (ingot-slice) niobium: few dislocations
    - Recent workshop at J-Lab
  - Fluorocarbons instead of water during processing

ACTIVITIES TO IMPROVE THE BASELINE PROCESS AT FNAL

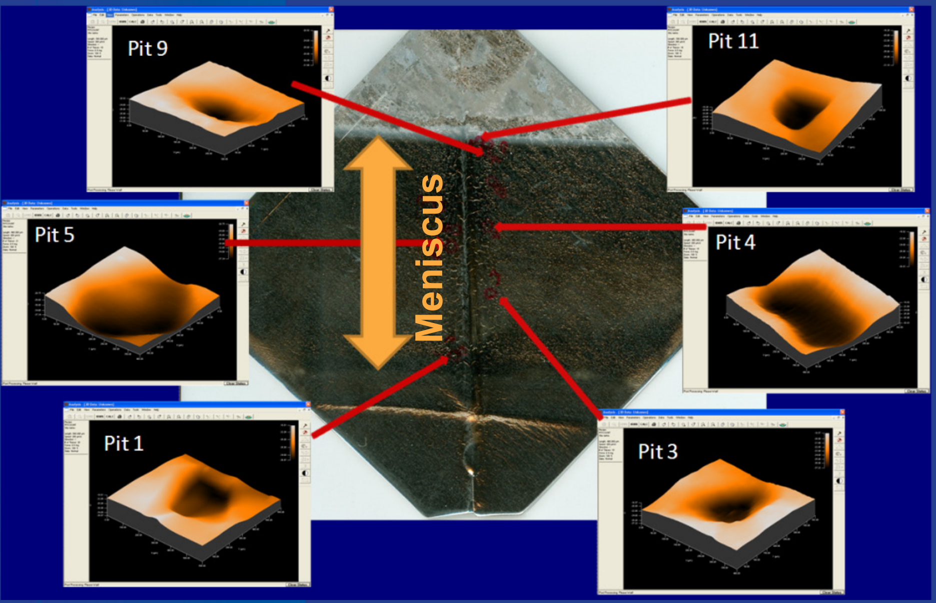
Single-cell Processing R&D

- Integrated Cavity Processing Apparatus
- Electropolishing and high-pressure rinsing
  - First acid expected May to June
  - Does water cooling improve EP?
  - Is fluorine ion monitoring feasible?
    - Raman spect: $H_2SO_4 + HF \leftrightarrow H_2O + HFSO_3$

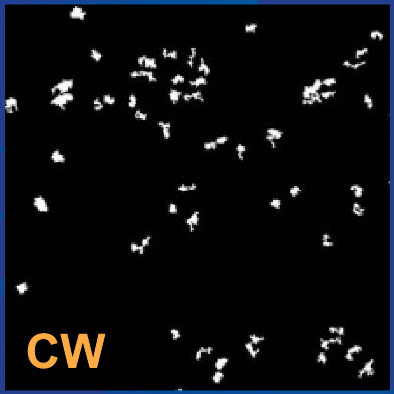
L Cooley
C Cooper

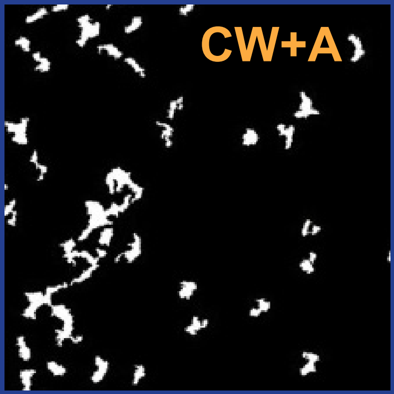
TD Instrum. & Ctrl. Grp.
TD Support Dept
TD Cavity Processing Grp.

Implications of coupon EP experiments



LD Cooley et al., IEEE Trans ASC **21**, June 2011 to appear

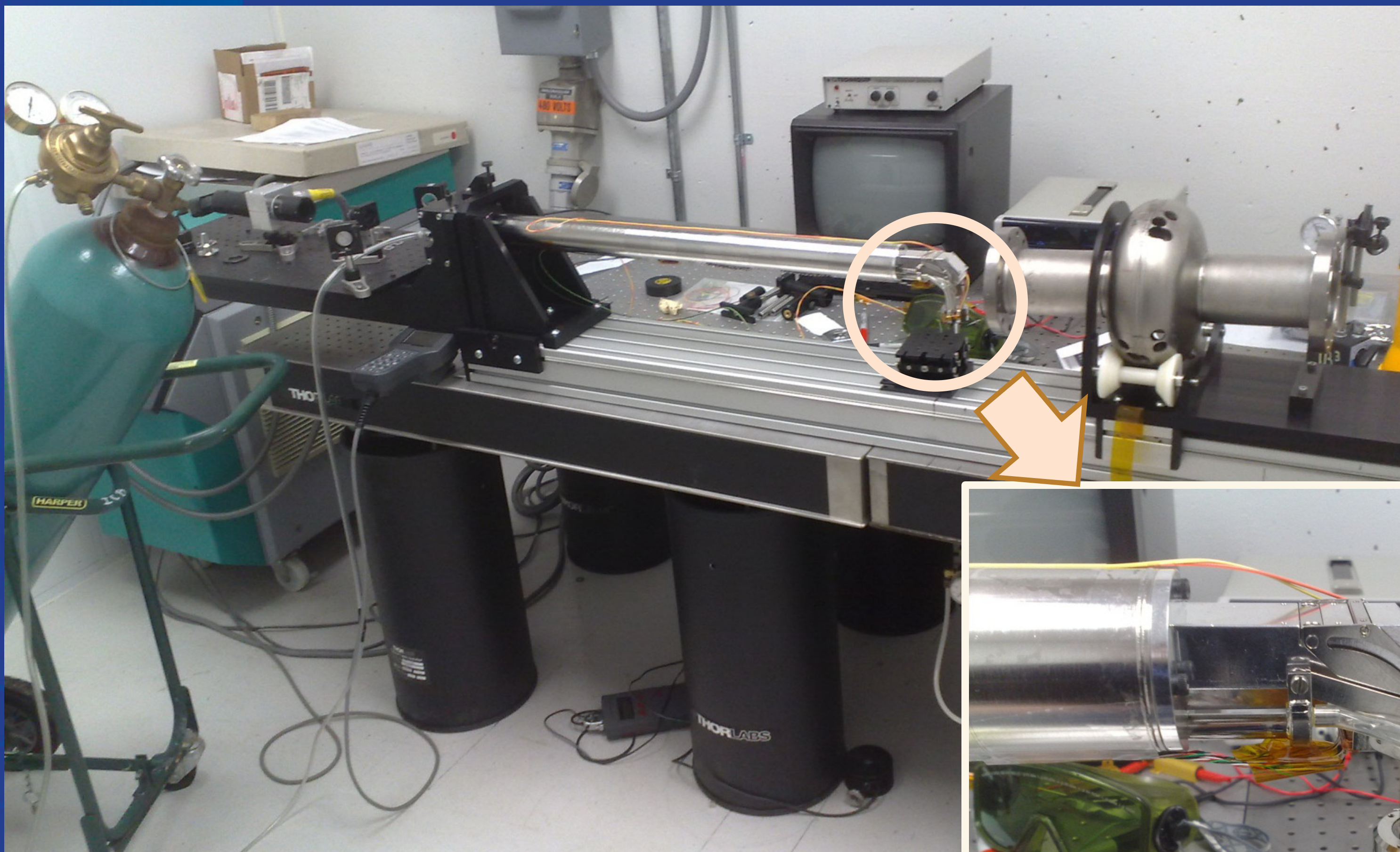




- Agitation promotes etching over leveling
  *H Tian & C Reece, PR-STAB **13**, 083502 (2010)*
  - Pitting may occur in meniscus
  - Keep electrolyte cold and thick
- More pits occur in cold worked than annealed Nb
  - Dislocation-assisted pitting?
  - Anneal before EP?
    - So far, CW + anneal is only partly successful

Sample areas from 2 cm x 2 cm binary scans using laser confocal scanning microscopy with particle counting algorithm – Donna Hicks & Chad Thompson (FNAL)

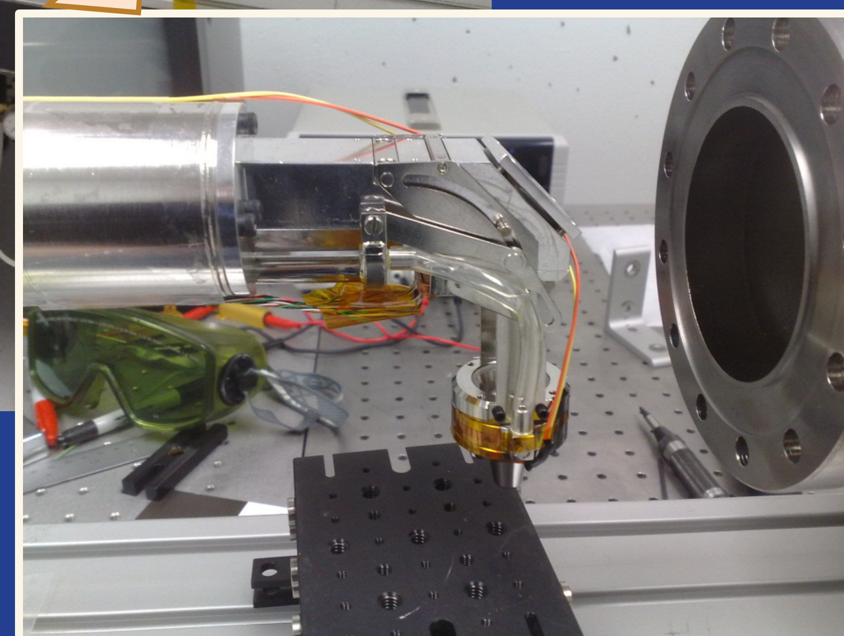
Laser re-melting system by Ge and Wu



Defect in 9-cell cavity has been re-melted and is being processed now.

Mingqi Ge (now at Cornell), Genfa Wu (now at ANL), et al., Proc. SRF 2009

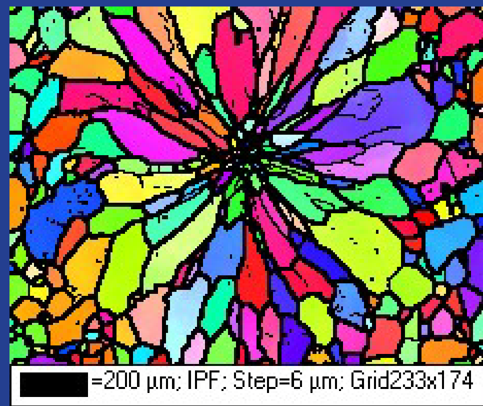
A. Dzyuba (FNAL) photographs

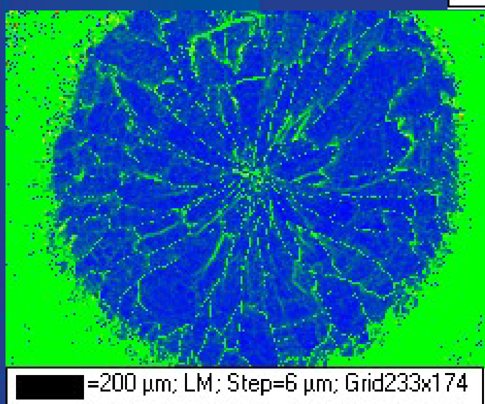


Complex articulation of nozzle for cover gas arranged around mirror and lens for laser focus

Optimization of laser technique

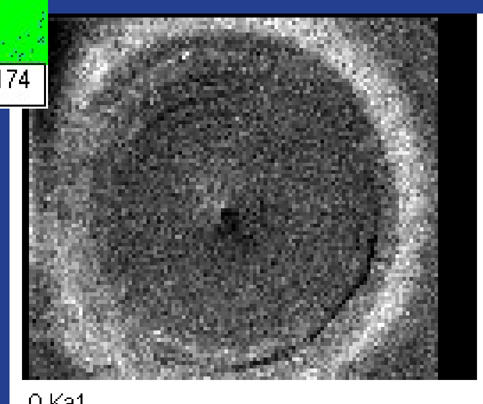
Solidification along thermal gradient





Evidence for stress along boundaries

Oxidation ring around single high-power shot



- Why melt?
  - Whole-cavity grinding might uncover new defects
  - Therefore melt locally
- Molten zone details
  - HAZ like EB weld – Does this affect post process?
  - Oxidation <10 µm thick
    - OK for final EP
  - One high power shot: “impact crater” topography
  - Multiple low power shots: desirable flat pool

A. Dzyuba and E. Toropov, (FNAL)

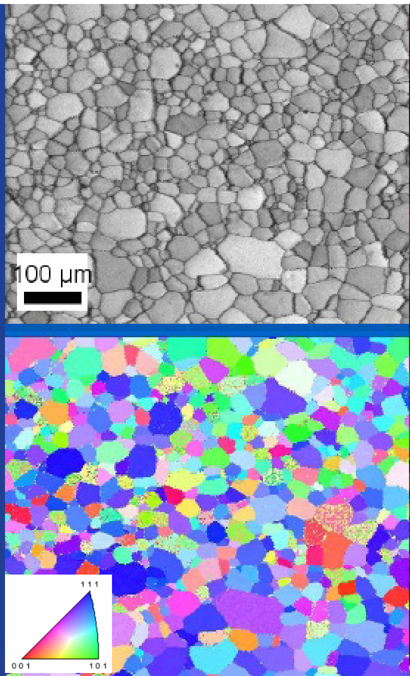
PROMISING ALTERNATE PROCESSING ROUTES

Hydroforming

- DESY has proven the principle with 3x3 cavities
  - Several review presentations by Waldemar Singer
- Extensive exploration by J-Lab & KEK, too
- Tube technology has emerged, is now ready
  - ATI Wah Chang now sells 150 mm x 1.5 m x 4 mm wall fine-grained seamless tubes (see next slide)
    - Billet process integrated with extrusion & forming
- FNAL has an active RFP with ILC-ART funding
  - Motivation: Reduce welds and weld defects
  - Goal: complete 9-cell hydroformed cavities
    - Workshop on 1 Sept. 2010, several vendors interested
  - 9-cell cavities hoped for by September, 2012

Successful DOE-SBIR with follow-on:
Black Labs / Dynamic Flow Forming / ATI Wah Chang / J-Lab / FNAL

13 µm grains
Random orientation



R. Crooks et al.

Billet forging + back extrude + flow form

DOE SBIR DE-FG02-04ER83909

6" I.D. RRR Tubes at ATI Wah Chang

As flow-formed

For DESY Hydroforming Machine Size

1.65 m length
~ 3 mm wall

9

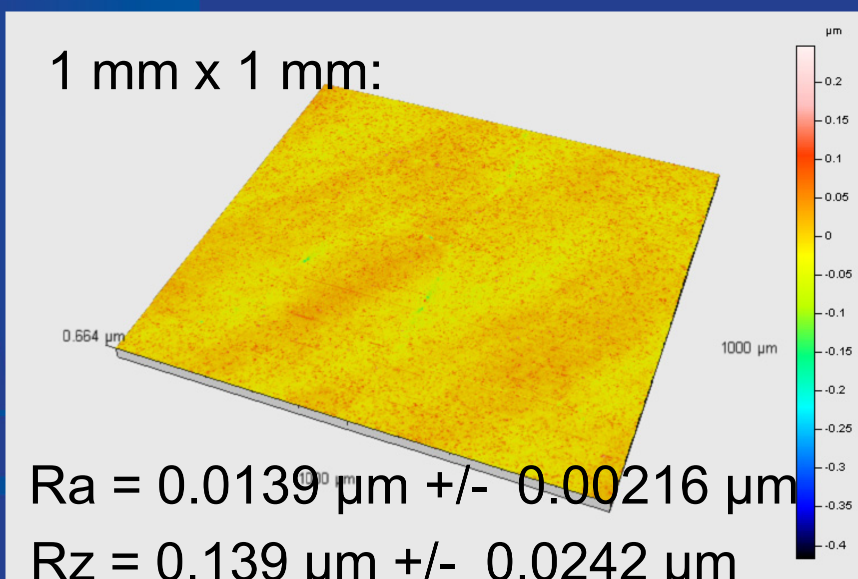
Niobium tube R&D

- Labs have ability to image grain orientations now
  - Very powerful at debugging raw material (e.g. tubes)
  - R&D: do grain orientations affect downstream processing?
- Reprocessing ideas
  - Texas A&M / Shear Form: Grain refinement, texture control, and weld healing by equal channel angular processing (SBIR)
    - Does not reduce diameter of tube while refining grains
    - Can cheaper tubes with inferior initial microstructures be tailored for hydroforming?
  - Nev.-Reno / Mich. State / Wah Chang: Zone induction annealing
    - Can reactor grade tubes be refined to RRR grade?
    - Highly ductile single crystal tubes have been demonstrated
- Long term: are re-entrant hydroformed cavities possible?

Tumbling / Extended CBP

- Order-of-magnitude improvement in surface finish over EP
  - By a tech in a lab coat & safety glasses working in typical lab space.
- Thanks to pioneering work by Higuchi, Saito, Singer, others – Many groups already have a machine !
  - Assuredly, many exciting innovations are still to come

1 mm x 1 mm:



Ra = 0.0139 µm +/- 0.00216 µm

Rz = 0.139 µm +/- 0.0242 µm

Courtesy C. Cooper, TTC Milan slides

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Extending CBP* to fine polishing

Grinding

Step 1
Cutting

+ Soap & Water

Step 2
Intermediate Polishing

+ Soap & Water

CBP

Micro-machining

Step 3
Intermediate Polishing

400 grit Alumina + Water (~20 μm)

Step 4
Intermediate Polishing

800 grit Alumina + Water (~10 μm)

De-laminating

Step 5
Final Polishing

Colloidal Silica (0.05 μm)

Cooper's key observation: must soak the hardwood blocks in the slurry†

*Higuchi T et al., 1995 Proc. 7th Wkshp RF Supercond. (Saclay, Gif sur Yvette, France) p. 723.

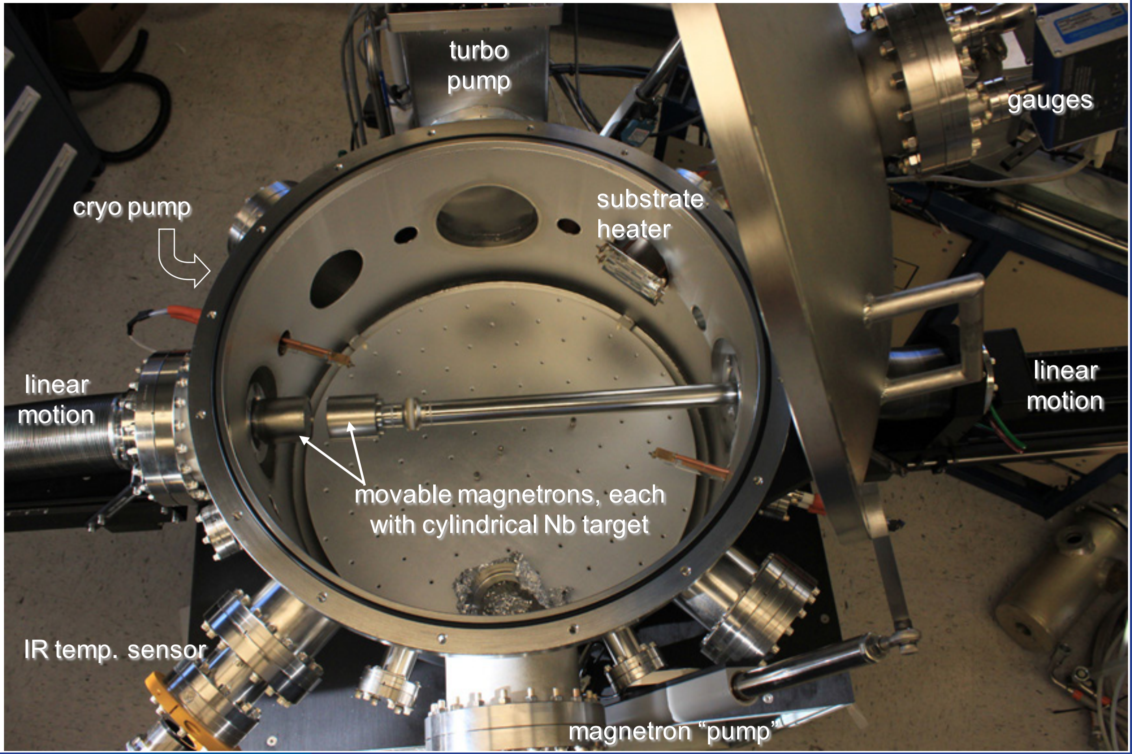
†Cooper C and Cooley L, Superconductor Science and Technology, submitted

Comments about tumbling

- Parallel and scalable, few control “knobs”
- Mirror smooth after step 5, best Q(E) after step 4 plus final EP so far
  - Replace “bulk” EP right now with 4-step process
  - De-laminating smears material over grit – will solving this (metallography) remove need for toxic acid?
- More hydrogen – longer bake at 800 °C needed
  - Bake to p(H_2) and p(H_2O) standards?
  - Use fluorocarbons instead of water (Saito)?
- Does tumbling encourage re-entrant cavity R&D?
  - Higher electric fields may require mirror-smooth finish
- (Long term) Can we construct the RF surface from films and coatings on mirror-smooth cheap Cu?

High power impulse magnetron sputtering

A state-of-the-art HIPIMS system for 1.3 GHz,
Offering optional dual-HIPIMS and two-material HIPIMS



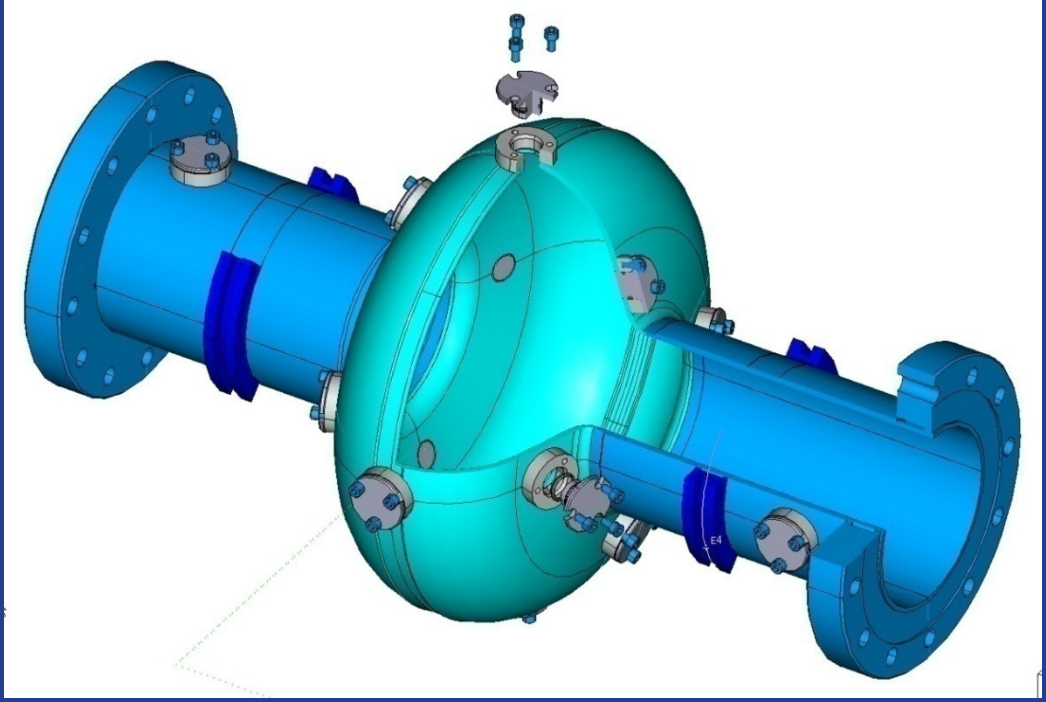
A. Anders, J. Vac. Sci. Technol. A 28 (2010) 783

- Zhou (J-Lab), with Alameda Applied Sciences: Nb films with RRR well above 300 attained for first time, Oct 2010
  - Flat films on hot sapphire
- Anders (LBL): HiPIMS system ready for cavities
- Do the new breakthroughs make Nb/Cu or Nb/Al work for ILC? Project X?

All Labs should use coupon cavities

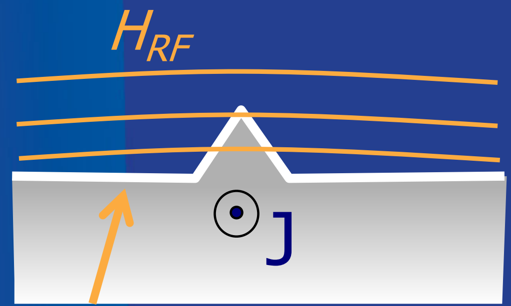
- Give materials scientists opportunities to provide process feedback without dissecting cavities
  - No other way to obtain spectroscopy from inside cavity
- Is a thick-wall version possible ?

C Cooper's design, similar to several others already being used



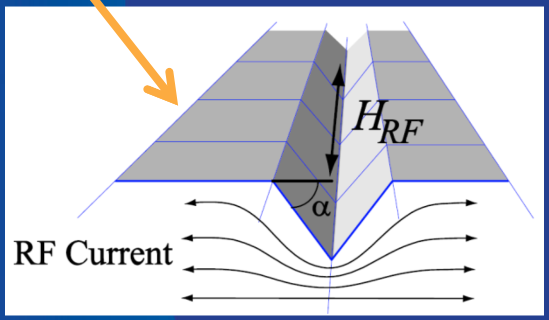
IDEAS MOTIVATED BY MATERIALS SCIENCE

General aspects of the SRF surface



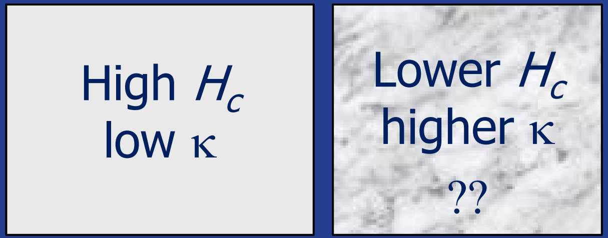
Knobloch et al

Surface Barrier

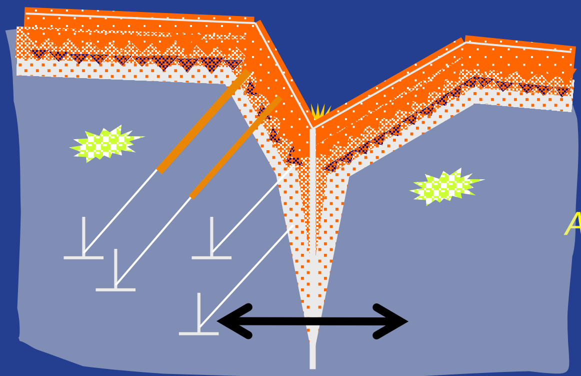


Dzyuba et al

RF flux should not penetrate due to topography: **Smoother surfaces are better** (but how smooth is smooth enough?)



Base superconducting properties should be ideal: **Less contamination**



Halbritter, Antoine, et al, Tian et al, Many others

RF current should not be induced across non- or weakly superconducting boundaries, precipitates, defects and damage: **Less contamination**

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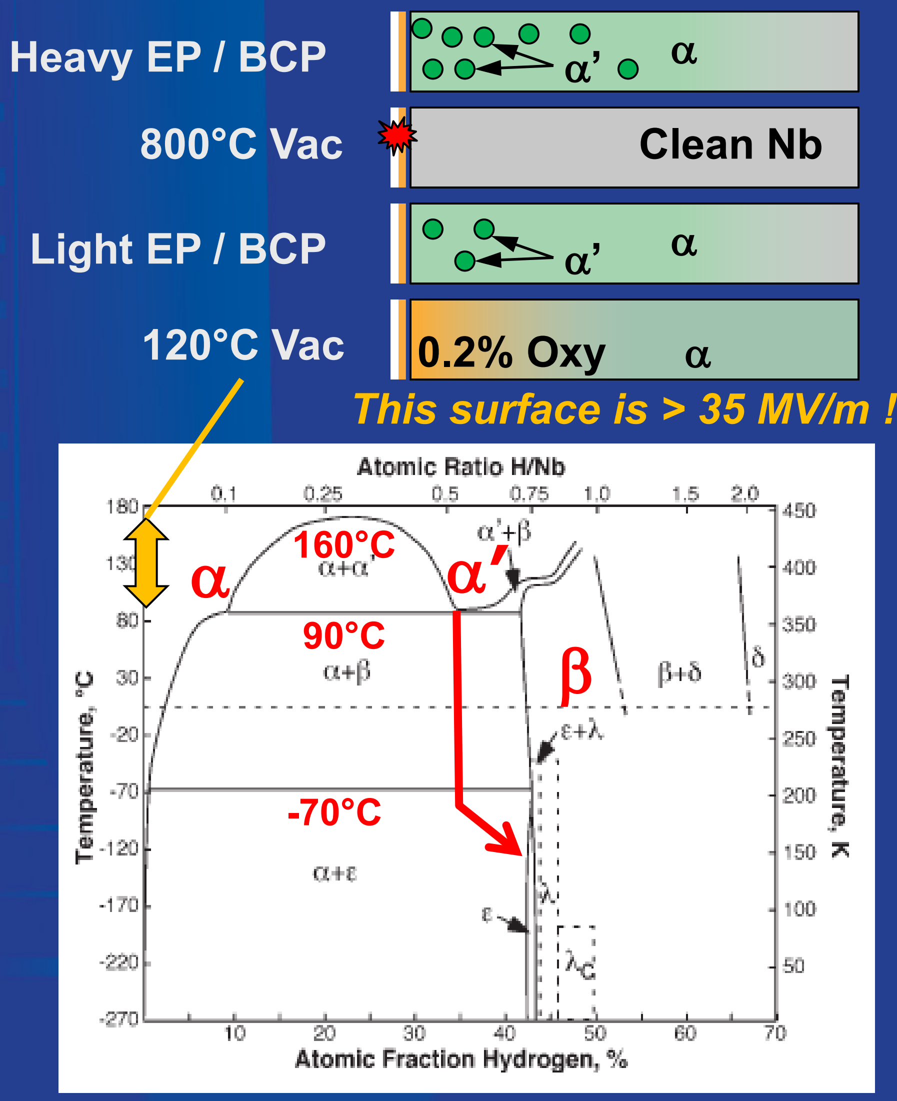
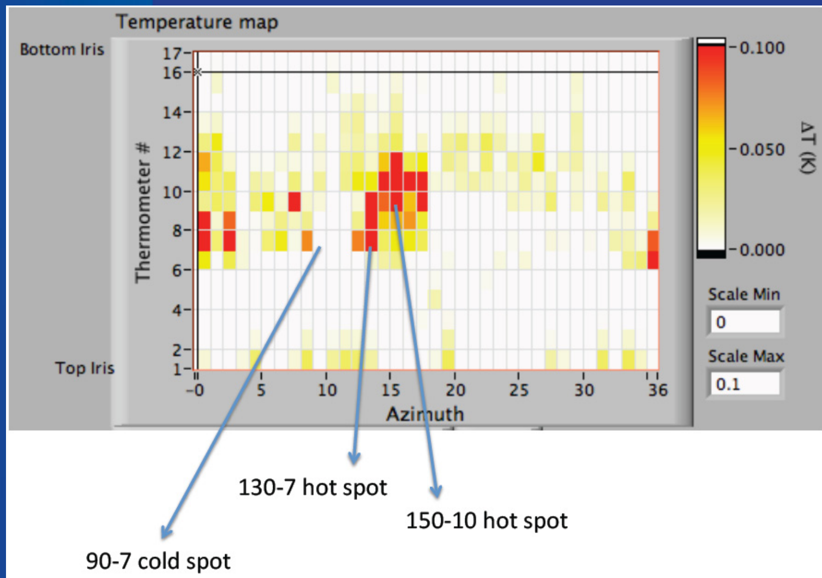
Latest models of the Nb surface region



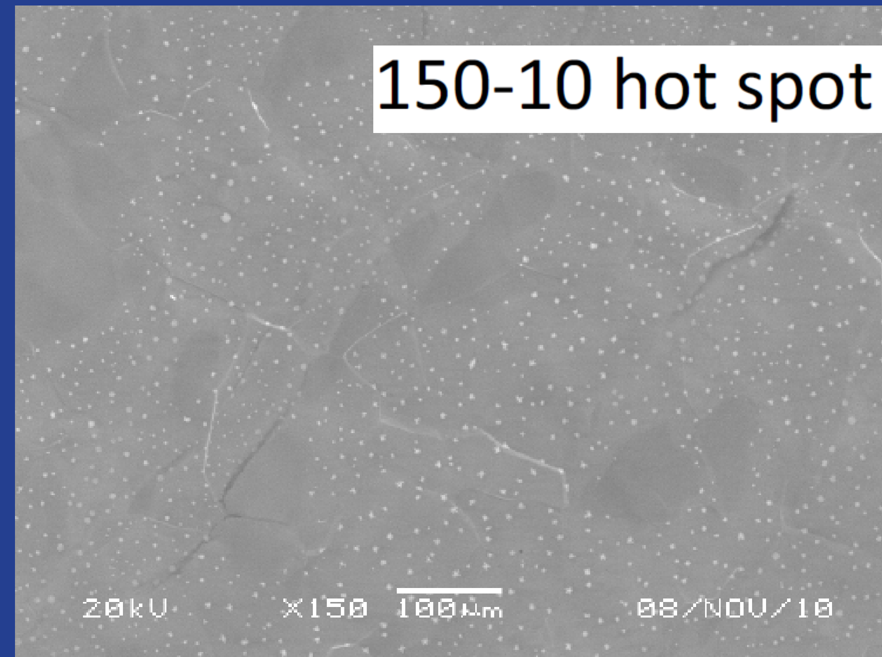
- 10-40% H at / under surface
  - BCP, EP, warm water …
  - $\alpha' \rightarrow \beta$
    - Nucleation on defects
    - What change after 800°C bake is more important: reduction of H or removed nucleation sites?
  - Lots of β: Q sickness
  - Some β: Q slope?
    - Are mid- and high-field Q slopes different aspects of same nanostructure?

Ricker Myneni J.Res.NIST 2010
Ciovati – Ingot niobium workshop at J-Lab Sept 2010
Romanenko - He atom recoil spectroscopy with U.Mich. & W.Ontario

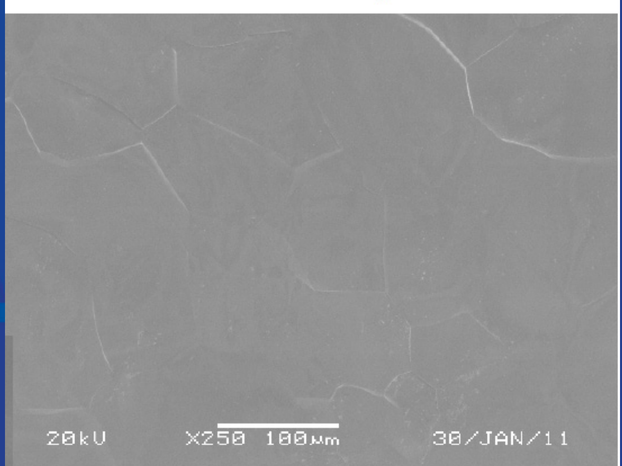
Connection between Q-slope (onset above 100 mT) and small near-surface hydride precipitates!



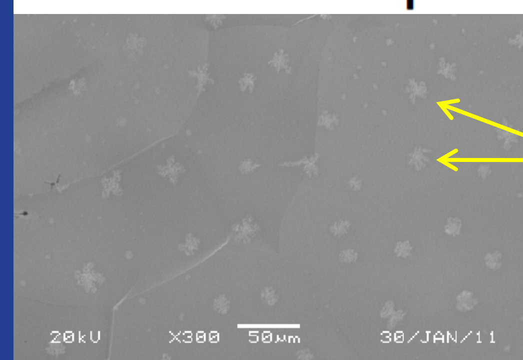
150-10 hot spot



90-7 cold spot



130-7 hot spot

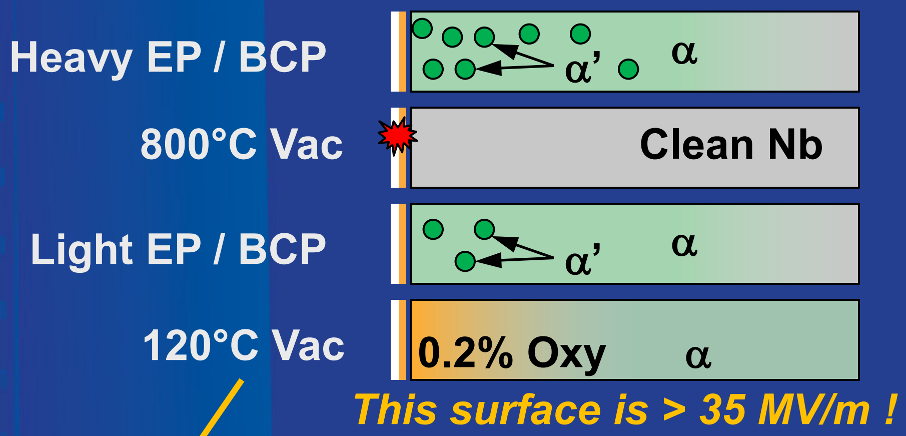


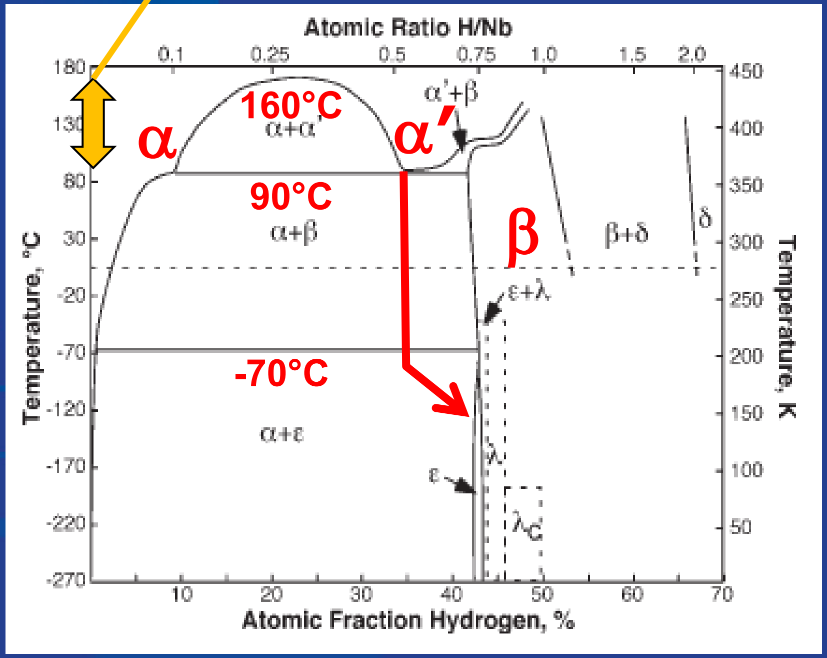
A. Romanenko, Fermilab, Unpublished work

These are hydrides: Vinnikov and Golubok, Phys. Stat. Sol. 69:631 (1982) and others

Fermilab

Latest models of the Nb surface region





- 90°C to 160°C mobilizes H
  - H clusters (α′) disperse from vacancies, defects [1]
  - Oxygen enters from decomposition of surface oxide [2]
    - Does O bind to nucleation sites and disperse H, thereby suppressing β? [3]

[1] Visentin et al, e+ / e- annihilation, SRF2009; Romanenko Ph.D. Thesis – Cornell,

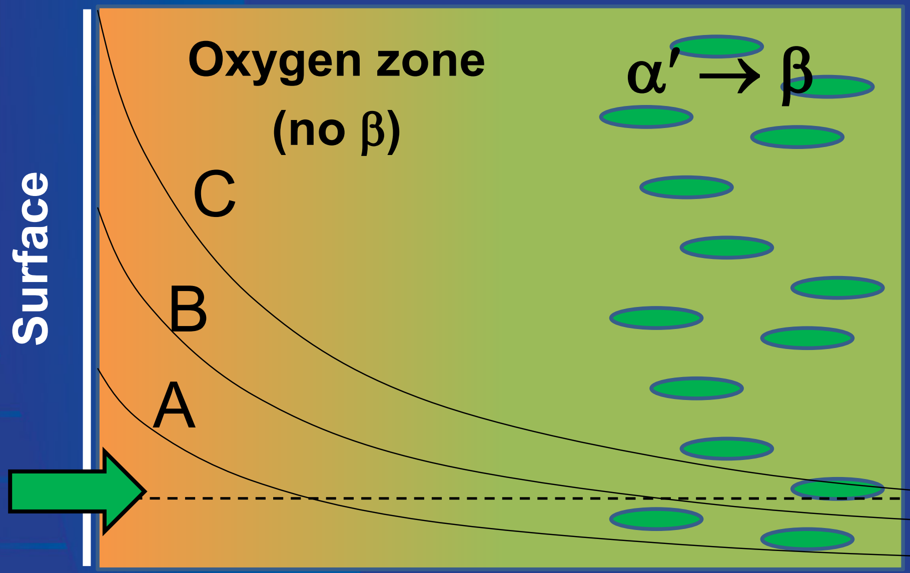
[2] Casalbuoni et al, Nuc.Inst.Meth.PR-A 538, 45 (2005)

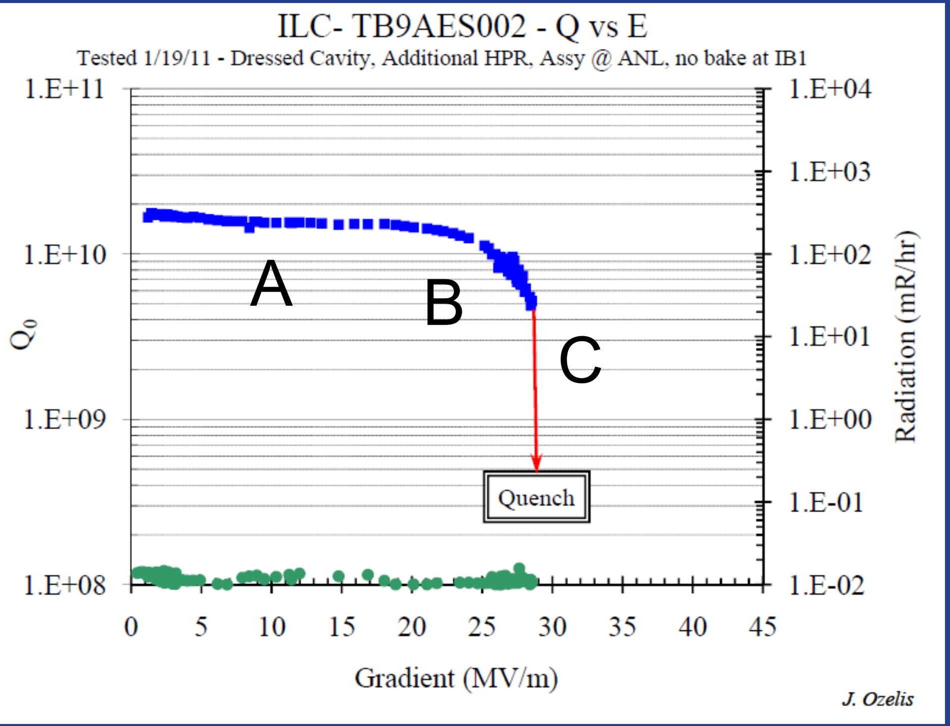
[3] D. Ford (NWU Ph.D. student) DFT + VASP at Fermilab

Do sub-surface hydrides explain Q(E)?

- There is always some oxygen just below surface
  - Large precipitates = strong loss at low onset
  - Small precipitates = weak loss at high onset

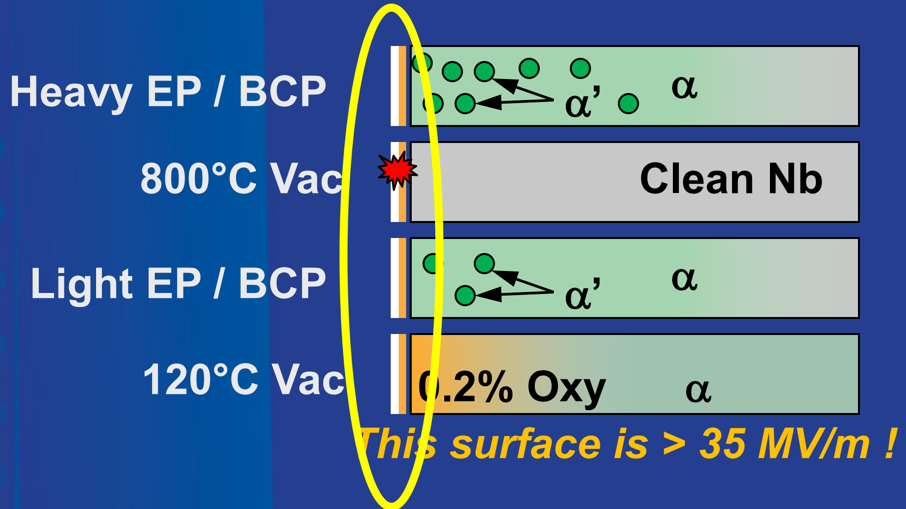
London profile of RF field H(x)

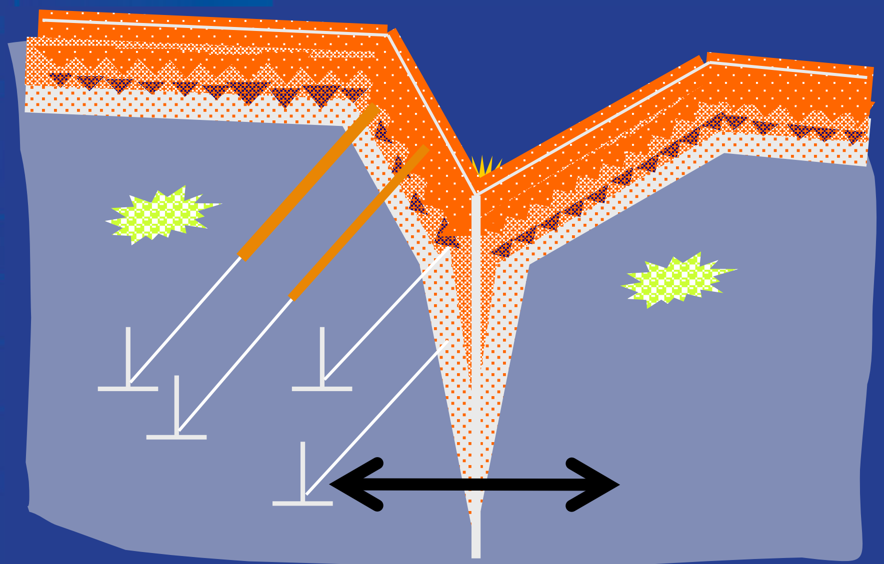




Green arrow: Local field at which currents induced across precipitates create losses

Latest models of the Nb surface region





- Nb-oxides contain magnetic defects [1,2]
  - Break Cooper pairs, reduce Q
  - Oxides are often amorphous[3], full of defects
  - Do dislocations transport magnetic ions inward?
- Are "wet" oxides worse than "dry" oxides?
  - Wet = water vapor, aqueous
  - H stabilizes O defects [2]

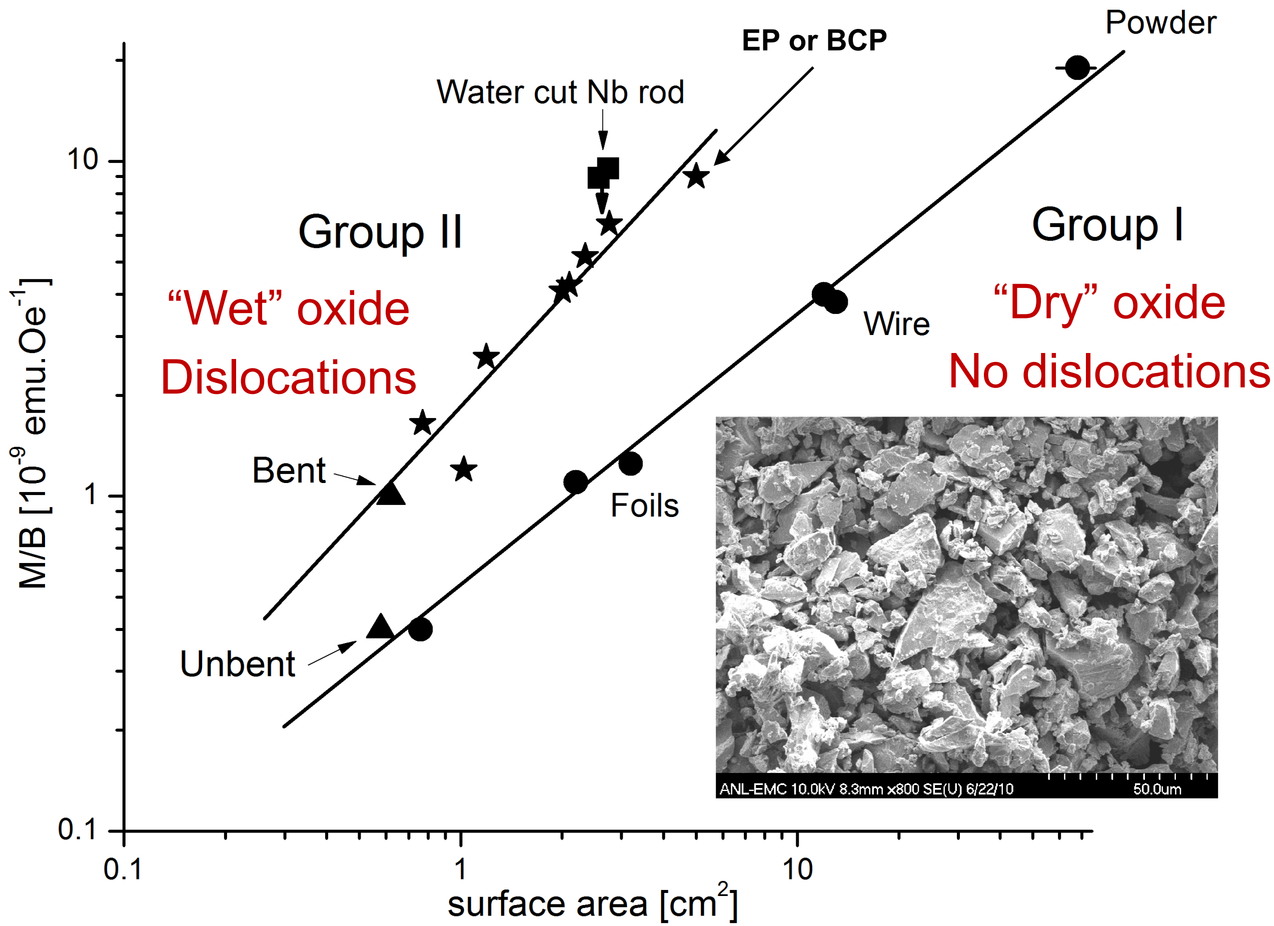
[1] Point-contact tunneling data: Zasadzinski group, IIT, and Prolier group, ANL, Appl. Phys. Lett. 2008, 2009.

[2] Density-functional calculations: W. Walkosz, ANL

[3] TEM data: R Klie, UIC

Dislocations affect magnetic signature

Slide Courtesy of T. Prolier, ANL and J. Zasadzinski, IIT



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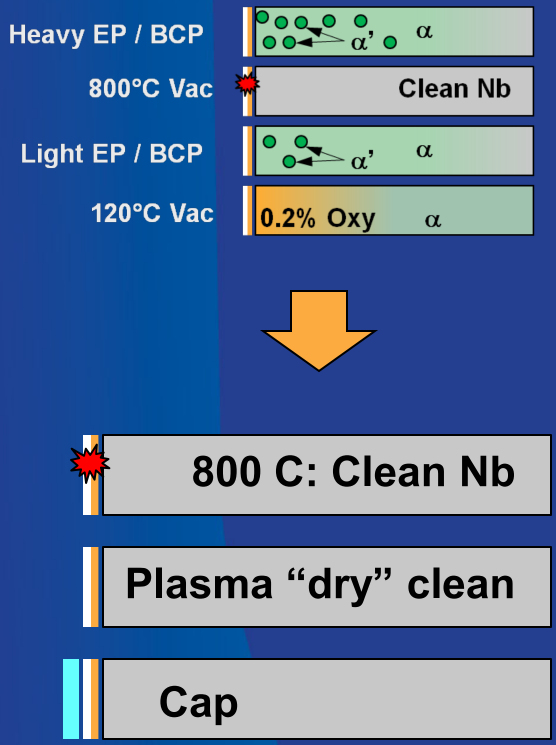
Implications for final processing R&D



No HPR?

- The 800°C high-vacuum anneal is important – preserve the metal in this state (control its nanostructure)
  - It erases side-effects of bulk removal
  - It forgives aspects of forming, welding
  - Low concentrations of O and H
  - Low concentrations of defects, nucleation centers, and dislocations
- Remove any debris by a dry technique, then cap immediately
  - Dry plasma cleaning
  - Dry deposition, e.g. modified ALD
  - Peel off dust with non-aqueous coating

Summary of SRF Materials R&D Areas

- EP with water cooling
- Local repair (laser melting)
- Coupon cavities
- 9-cell Hydroforming
- Tubes for hydroforming
  - ATI Wah Chang product
  - Equal channel angular process
  - Zone annealing
- Tumbling
  - 4-step + final EP
  - Mirror-smooth 5th step
    - As preparation for thin films
    - For re-entrant cavities
- Films & coatings
  - High power impulse sputtering
- Hydride nanostructure control
  - Dry etch, e.g. plasma cleaning
  - Dry protective coating, e.g. by Atomic Layer Deposition

The present ILC processing baseline

S0 Team, 2010 SCRF Cavity Technol. & Industrialization Wkshp. / Kyoto

	Standard Cavity Recipe
Fabrication	**Nb-sheet (Fine Grain)**
	Component preparation
	Cavity assembly w/ EBW
Process	**BCP + 1st (Bulk) Electro-polishing (>120um)**
	Ultrasonic degreasing with detergent, or ethanol rinse
	High-pressure pure-water rinsing
	Hydrogen degassing at > 600 C
	Field flatness tuning
	2nd Electro-polishing (~20um)
	Ultrasonic degreasing or ethanol rinse
	High-pressure pure-water rinsing
	Antenna Assembly
	Baking at 120 C
Vertical Test	**Performance Test with temperature and mode measurement →inspection, reprocessing, other remediation**

Industry

Inspection & remediation

Conclusion:
Possible processing baseline in 5 years

	1 TeV Cavity Recipe
Fabrication	**Nb tubes (Fine Grain)**
	Single-piece end-group preparation
	Hydroform tubes and assemble end groups w/ EBW
Process	**Tumbling**
	Ultrasonic degreasing with detergent, or ethanol rinse
	High-pressure pure-water rinsing
	Field flatness tuning
	Material recovery and hydrogen degassing at > 800 °C
	Antenna Assembly
	Plasma Cleaning
	Capping by Atomic Layer Deposition
Vertical Test	**Performance Test with temperature and mode measurement →inspection, reprocessing, other remediation**

Industry

Inspection & remediation