Hot Topic 1

Is large grain/single grain an alternative to fine grain?

First Part : Short Talks

- 5 presentations
  - 5 minutes each, 5 slides maximum each,
  - Quick questions for information only
- Presenters:
  - Please take a clear position about how your remarks show an advantage (or disadvantage) for large grain/single crystal over fine grain
- Please no status reports
  - address a specific issue

Discussion after the Short Talks

- Short Comments on the talks
- General Discussion
  - Separate discussions for
    - Large grain and Small grain
  - Address a series of questions and Try to answer:
    - Not an issue
    - more R&D needed
    - yes, no,
    - make a list

General Discussion: 30 minutes
What are the benefits of large grain/single grain?

- Is the material cost lower?
- Is fabrication or treatment simpler (cost effective)?
- Is the material better?
  - Less defects? Larger elongation?...
- Is performance better ?
  - High gradients, smaller spread?
- Do we absolutely need single grain?
- Caution: We do not need to discuss <u>how</u> to make large grain/single grain here…

Large Grain Questions

Is the material cost lower?

- Is there a solid evaluation of the cost benefit?
- Any studies in progress?
- Do we know the best way to cut sheets?
- Which companies can provide large grain now

Is fabrication or treatment simpler?

- Are the fabrication issues solved? List the remaining issues
  - Earing, thinning at iris, holes during ebw
  - What issues remain to be solved? List these
  - What r&d is need to solve these
- Is EP necessary for large grain to reach 35 MV/m? or is BCP sufficient ?
- Do the large grain sheets need to have a central single crystal? Why?
  - How do we get the ingot with central grain

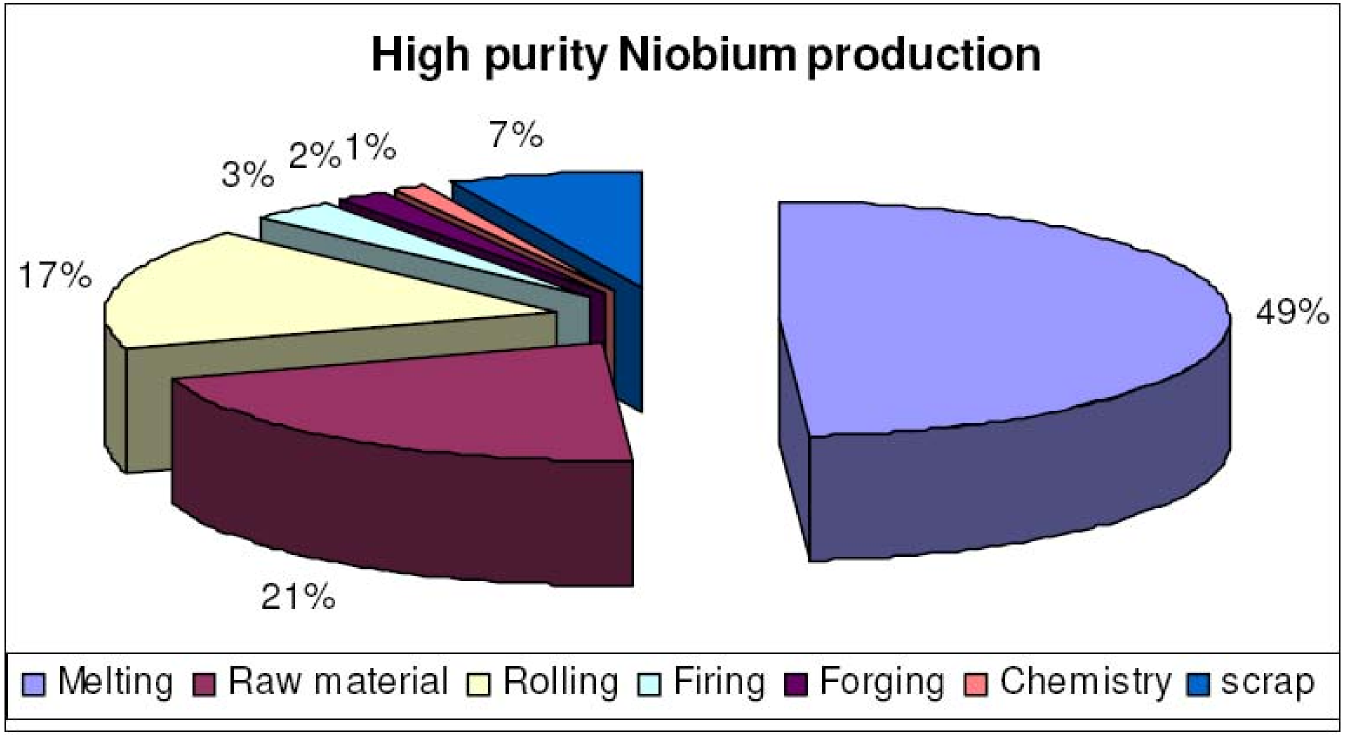
Is performance better ?

- Is there less field emission with large grain?
- Are there fewer defects?
- Are material properties better?
  - Elongation? Bulge test
- Are the intrinsic properties (e.g. Hc) better in one orientation?
- Are there other reasons for a preferred grain orientation?
- Does the lower density of grain boundaries help?
- Does the phonon peak due to large grains help?
- Does the existing data show that the spread of large grain cavity performance better than fine grain?
-

Single Grain Questions

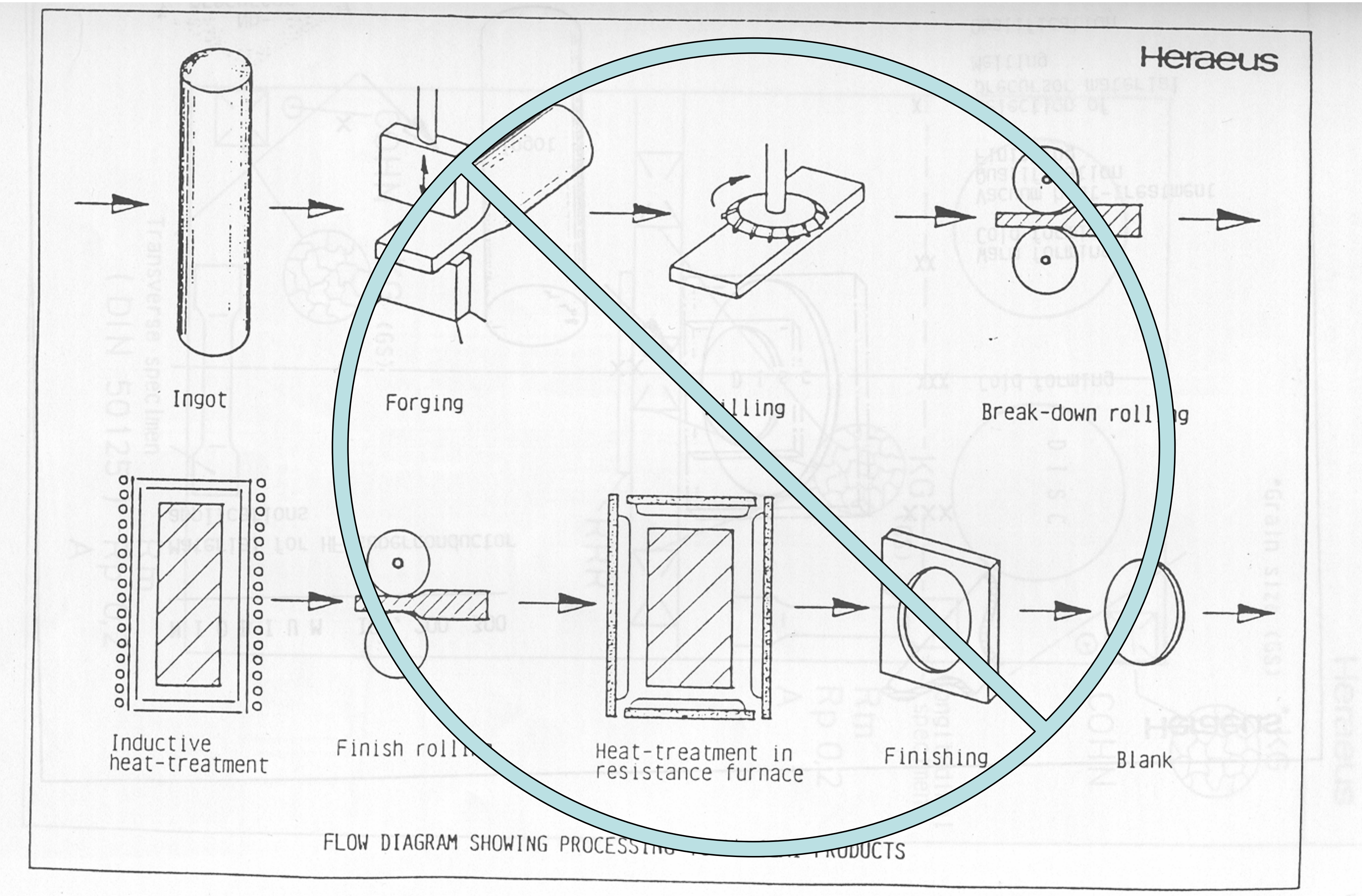
- Advantages over large grain? List
  - Don’t need EP, smoothness with BCP gives good finish
- What are the added difficulties? List
- Which is the better path?
  - Rolling an ingot slice
  - Starting with tube
    - Gives seamless
- Who is working on single grain?

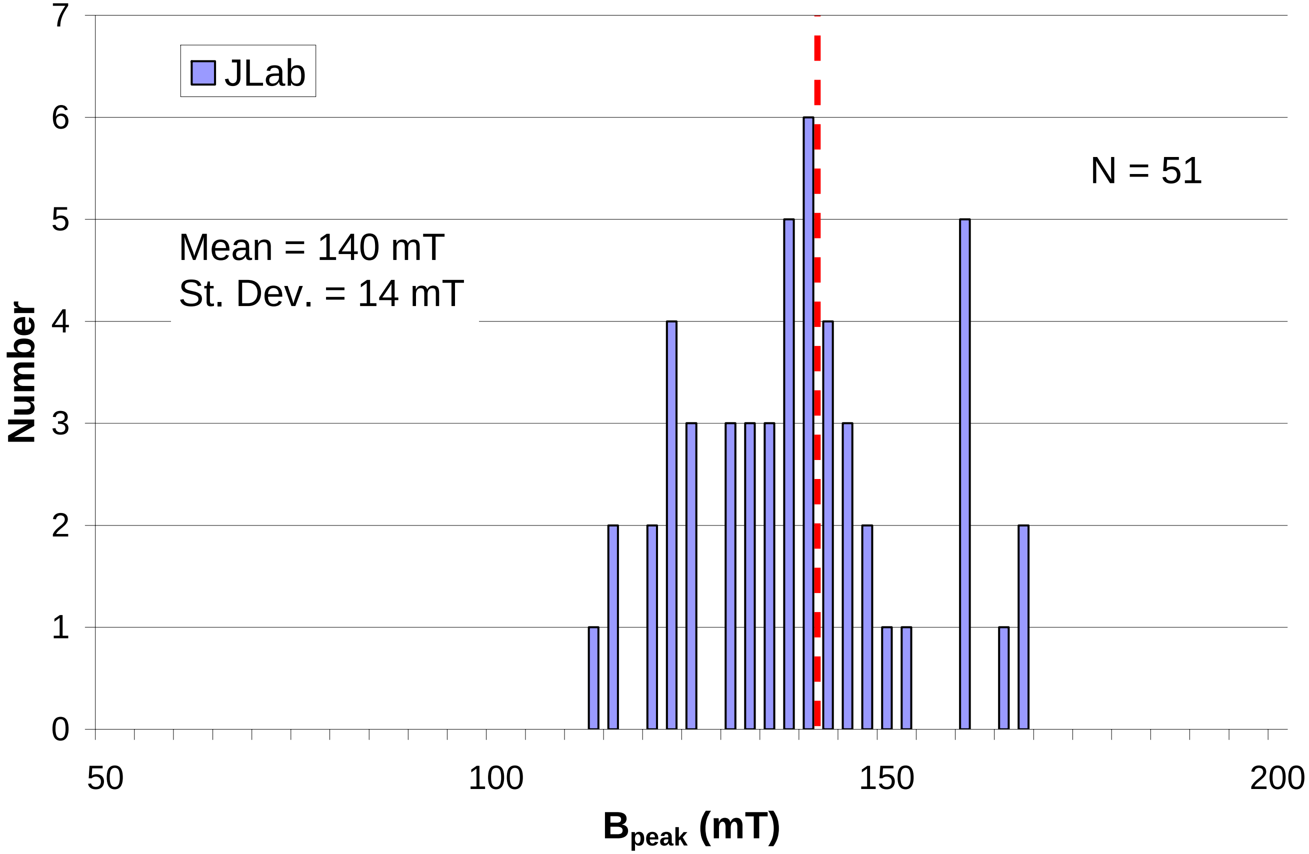
Cost Analysis of Production



SFR 2005, Industrialization
Symposium, D.Proch, DESY

Can We go Directly from Ingot to Sheet?





JLab
Mean = 140 mT
St. Dev. = 14 mT
N = 51
Number
B_{peak} (mT)
0
1
2
3
4
5
6
7
50
100
150
200

13th International Workshop on RF Superconductivity

Peking University, Beijing, China 2007

Large Grain Niobium

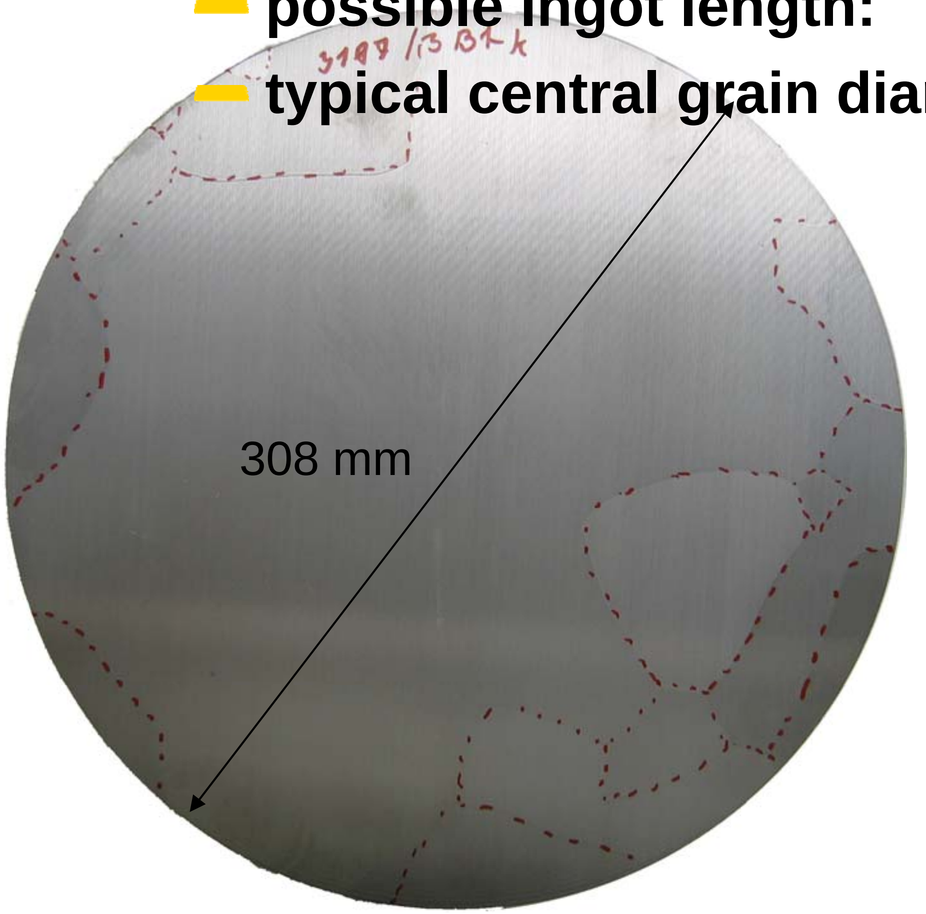
- **State of the Art at Heraeus**

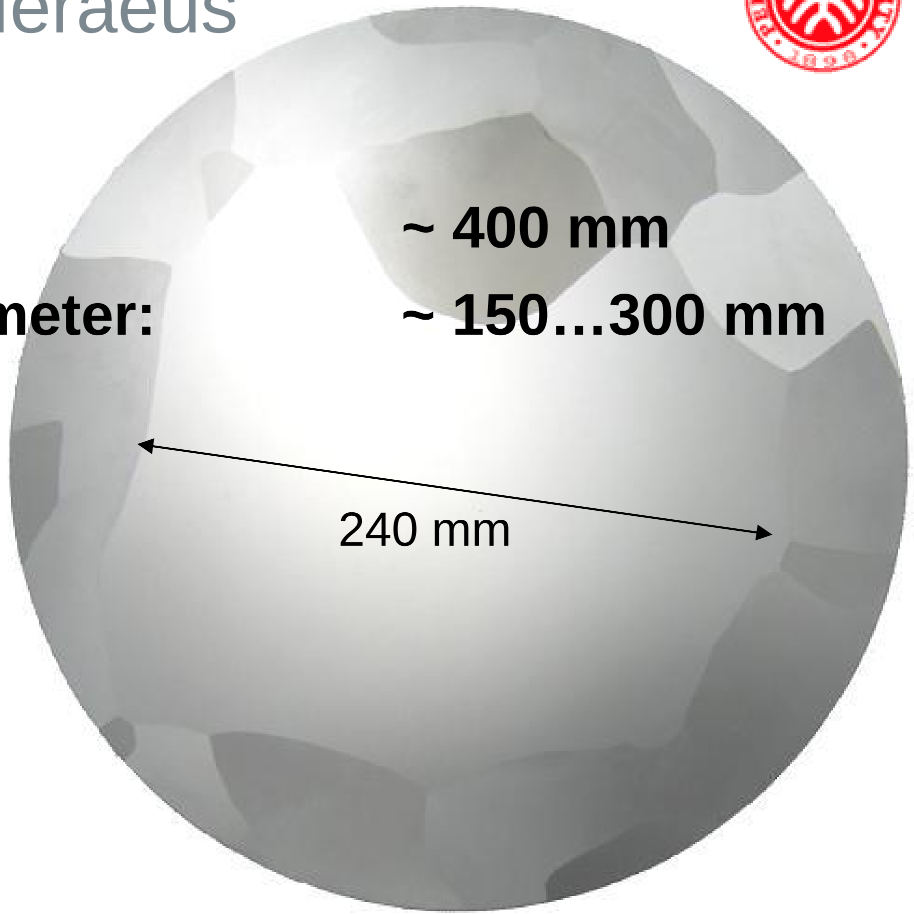
- **Comparison**
  Discs directly cut from Ingot vs rolled Sheets

Heraeus

State of the Art at Heraeus

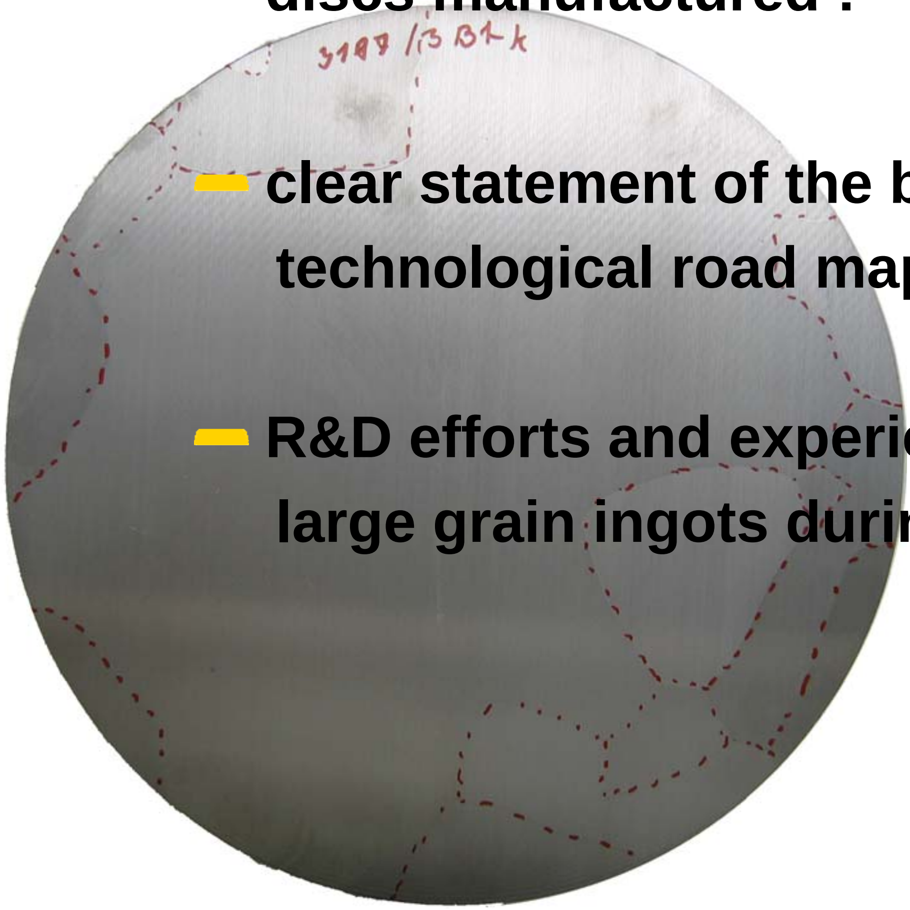
- **possible Ingot length: ~ 400 mm**
- **typical central grain diameter: ~ 150…300 mm**





Heraeus

State of the Art at Heraeus



- **discs manufactured : ~ 300 pcs**
- **clear statement of the board Nb RRR is part of the technological road map of W.C.Heraeus**
- **R&D efforts and experience for several tons of large grain ingots during the last 2 years**

Comparison
Discs directly cut from Ingot vs rolled Sheets

Property	Disc	Sheet
RRR value	>300	>300
thickness tolerance	0,06 mm	0,2 mm
surface roughness	better	
mechanical properties		
Rm	80 MPa	80 MPa
Rp0,2	50 MPa	50 MPa
Al 30	85%	65%
risk of contamination	low	higher
workability	ACCEL	
cavity performance	DESY / CORNELL	
reliability	few cavities "only" a few results	many cavities experience over years
price	estimated 80…85%	100%
yield Ingot to sheet / disc	higher	lower
time in process	lower	higher

- Disadvantages Disc directly cut from Ingot
- **Reliability:**
- **till now, experience of only a few cavities is available**

Advantages Disc directly cut from Ingot

- **Technical :**
- **thickness tolerance**
- **elongation/ductility**
- **lower risk of contamination**

- **Commercial advantage for the Projects**
- **estimated 15…20% below common technology**

Heraeus

Large Grain Niobium

- **thank you**
- **and don't hesitate to send your order**

Hot Topic I – Single Crystals

Input from Fermilab

Lance Cooley, Hairong Jiang, Genfa Wu, Robert Schuessler, Mike Foley*

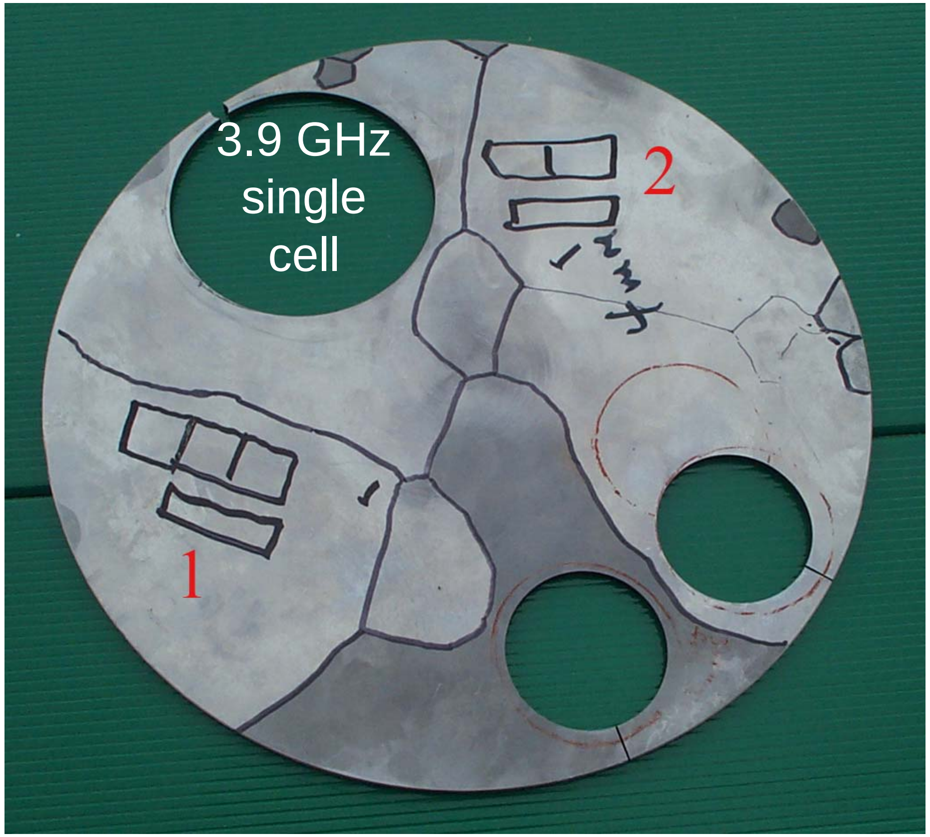
Technical Division, Fermilab

**Accelerator Division, Fermilab*

Challenging questions for workshop discussion

- Large central grains probably need to be seeded; zone refining methods need huge amounts of power
  - Zone-refined single crystal tubes will work, however
  - Are seamed single-crystal tubes possible?

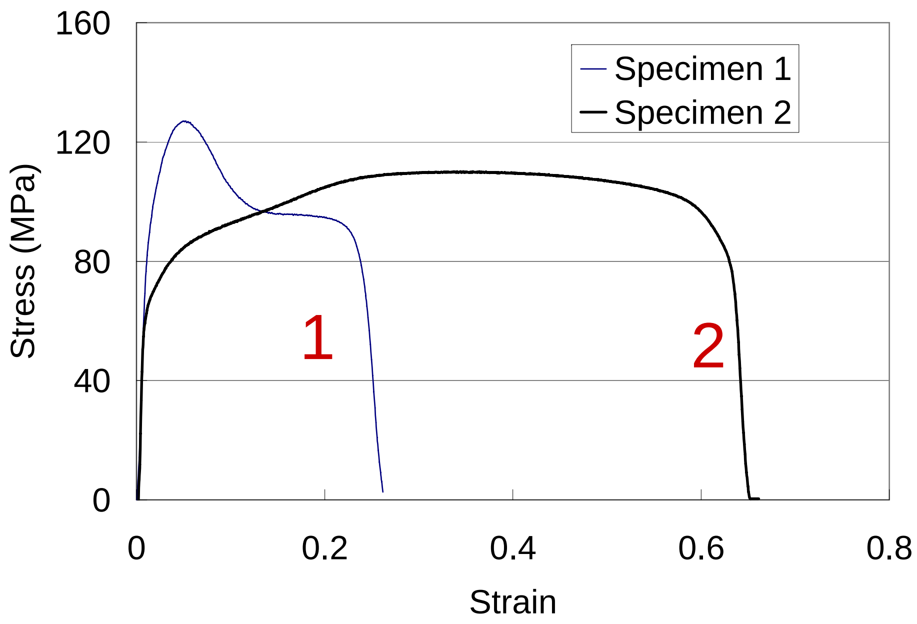
- *How tightly can we control the orientation of seeds and the subsequent crystals?*
- *Do we care? Just align and cut…*
- *Do properties vary with direction?*



Hairong Jiang
Fermilab

3.9 GHz cavity in forming process at the moment

RRR
Grain 1: 254
Grain 2: 242
Crystal orientation has no big effect on RRR value

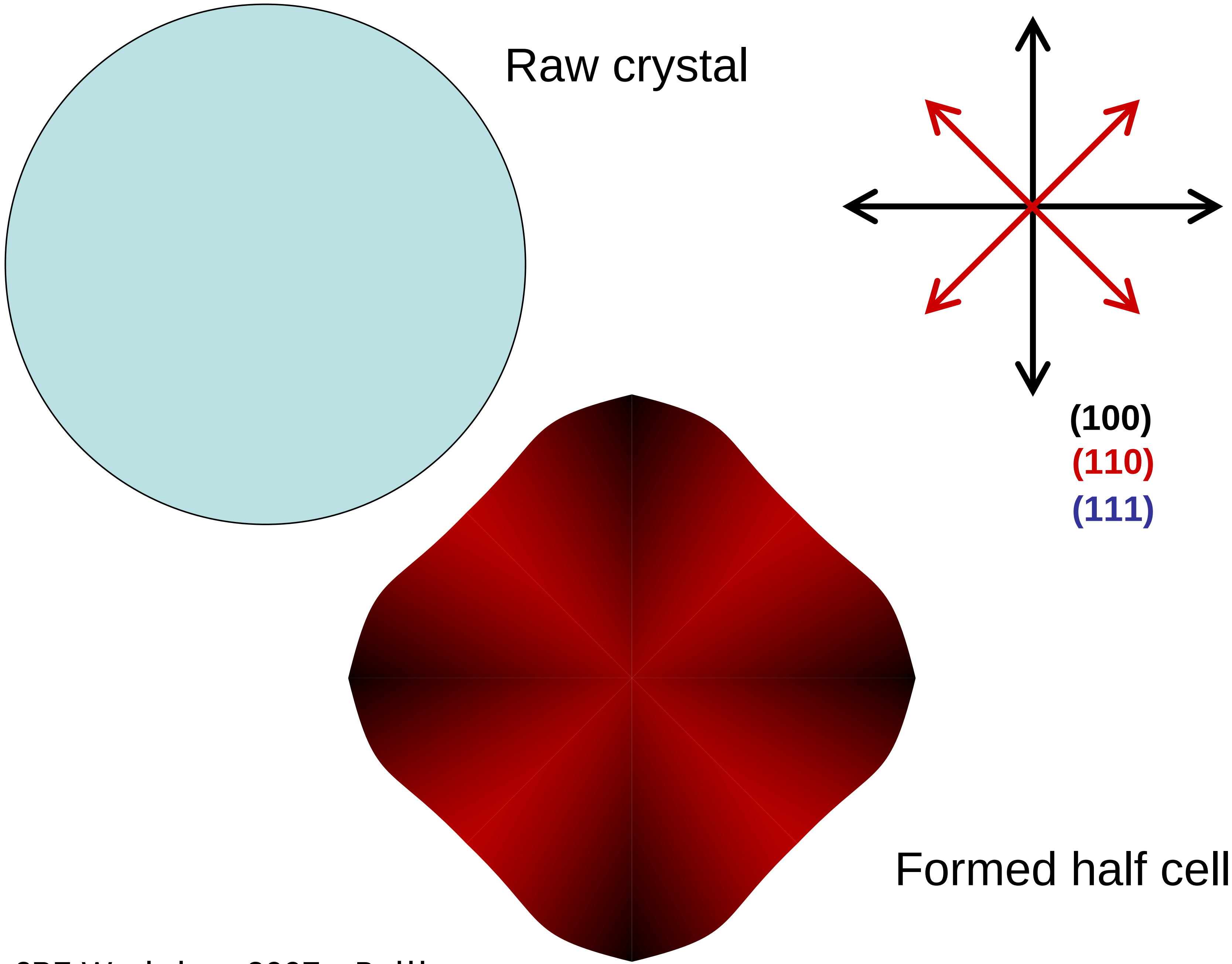


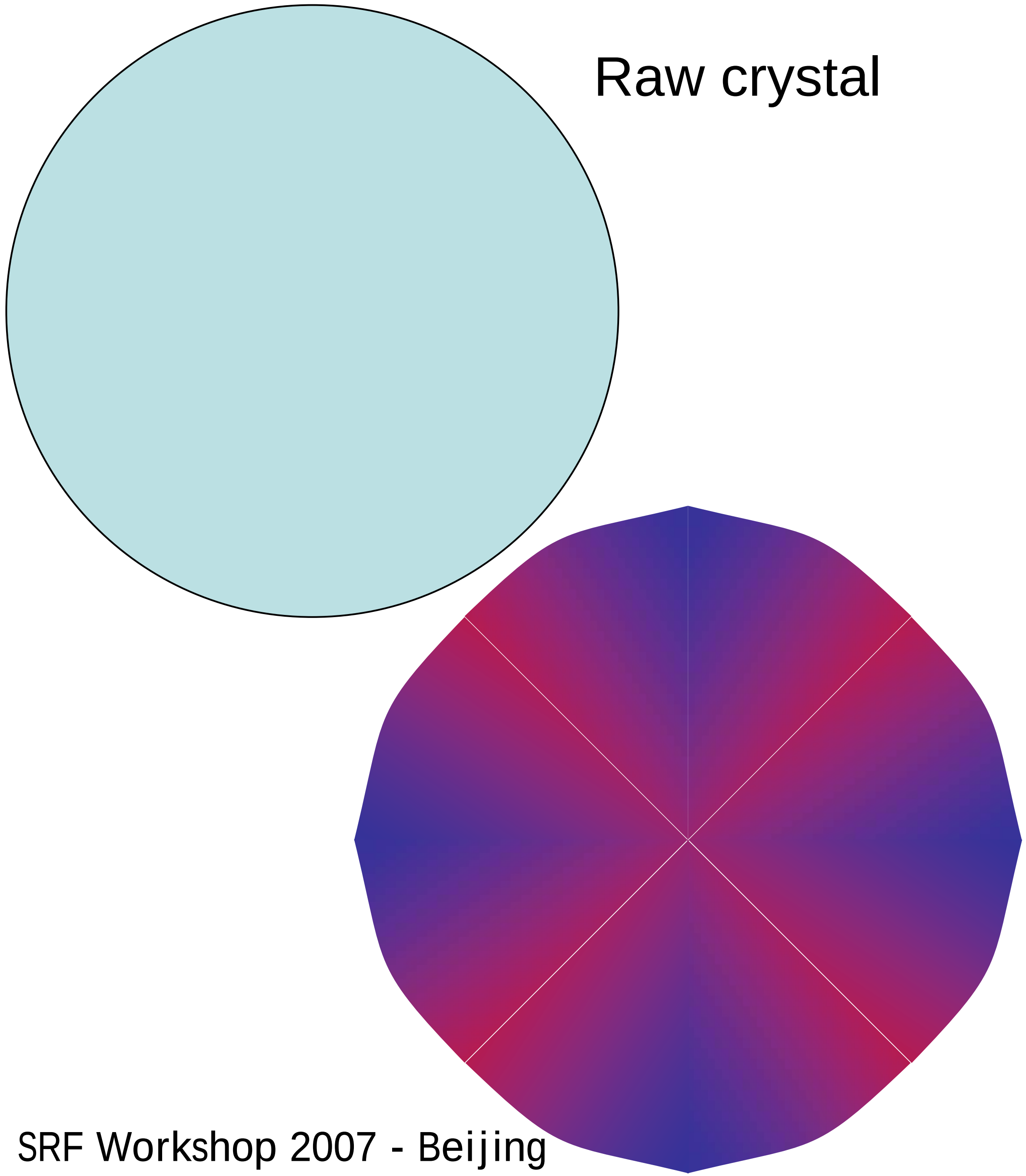
	Yield Strength (MPa)	UTS (MPa)	Elongation
Grain 1	89	126	26%
Grain 2	55	109	65%

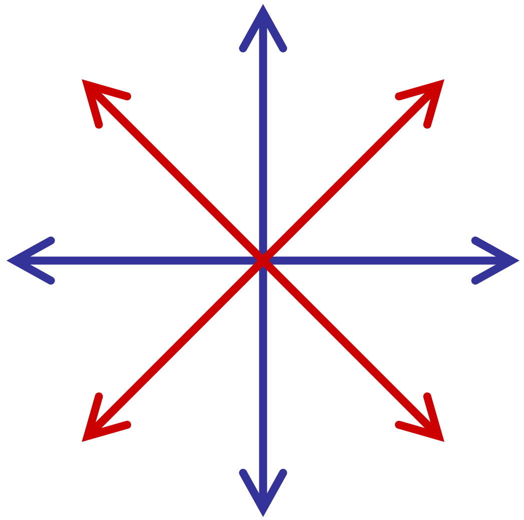
Grains not indexed yet
Grain 1 mixed
Grain 2 mostly (100)

Mechanical properties of Nb are greatly influenced by crystallographic orientation

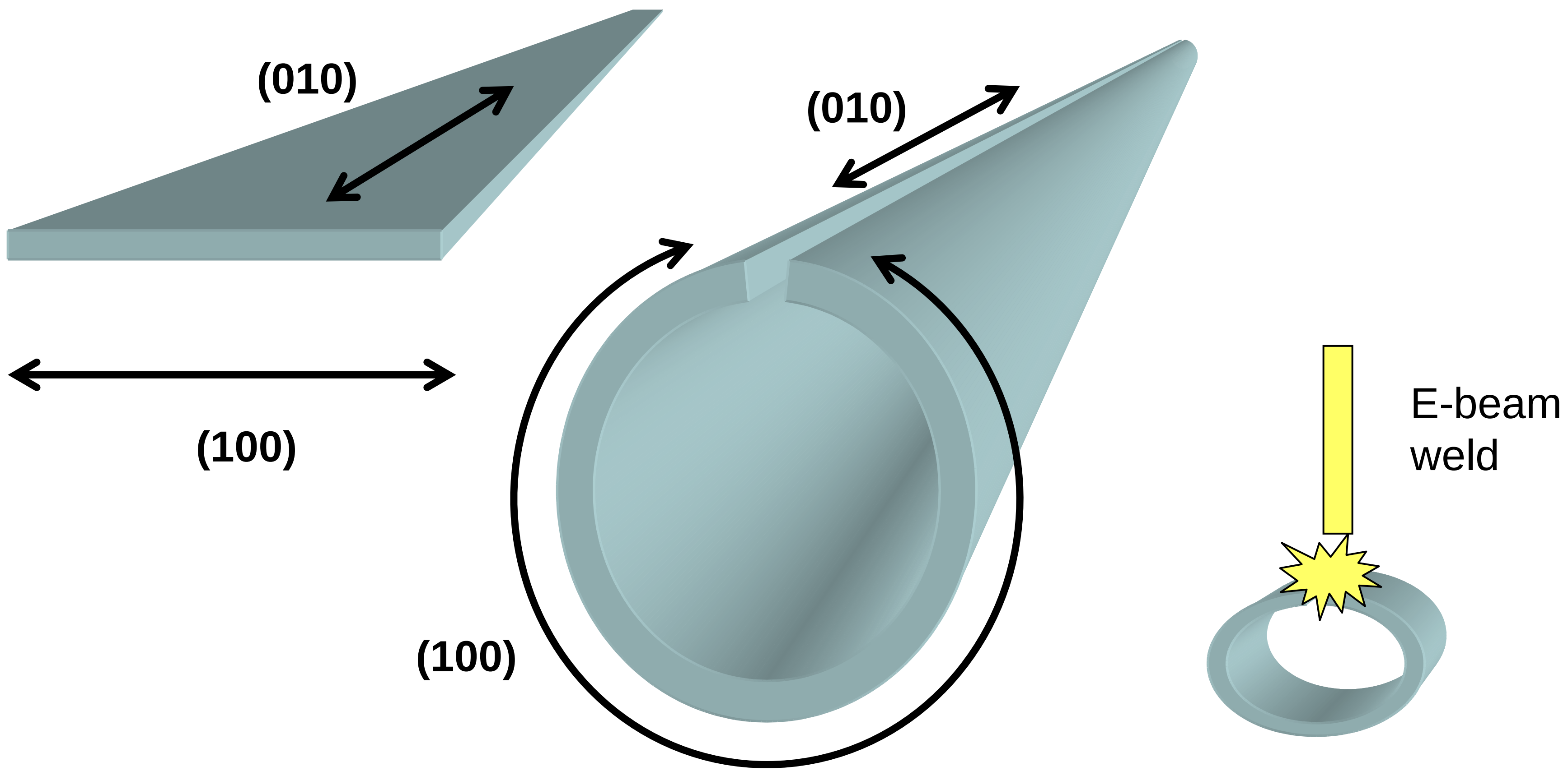
Hairong Jiang & Genfa Wu
FermilabC


Raw crystal
(100)
(110)
(111)
Formed half cell


Raw crystal
Formed half cell


(100)
(110)
(111)

Single crystal seamed tubes for hydroforming



Hot Topic I
Single and Large Grain Niobium

Cavity fabrication experience and recrystallization studies for large grain and single crystal niobium

A. Aizaz, D. Baars, T. Bieler, J. Bierwagen, S. Bricker, C. Compton, T. Grimm, W. Hartung, M. Johnson, P. Kneisel, J. Popielarski, L. Saxton, N. Wright

13th International Workshop on RF Superconductivity
Beijing, China
10/18/2007

NSCL

MICHIGAN STATE UNIVERSITY

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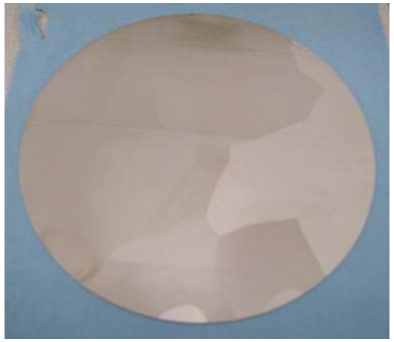
Fabrication of Large Grain Cavities

- Deep drawn
  - Fine Grain – 200 tons, 100 tons coining
  - Large Grain – 50 tons, 40 tons coining
- Spring back
  - Fine Grain – 0.25mm (0.010”)
  - Large Grain – 0.13mm (0.005”)
- Earing – Allow extra material
- Grain boundaries – Polishing interior surface due to grain boundary roughness
- Large grain, large strain → ease movement
  - Spring-back from machining fixtures
  - Miss-match in e-b welding alignment (additional fixturing), same weld parameters
    - Shrinkage of equator
      - Fine Grain – 0.51mm (0.02”)
      - Large Grain – 0.76mm (0.03”)
- Multi-cell fabrication
  - Parallelism
    - Fine Grain Δ= 0.76mm (0.03”)
    - Large Grain Δ= 1.77mm (0.07”)



Niobium from JLAB

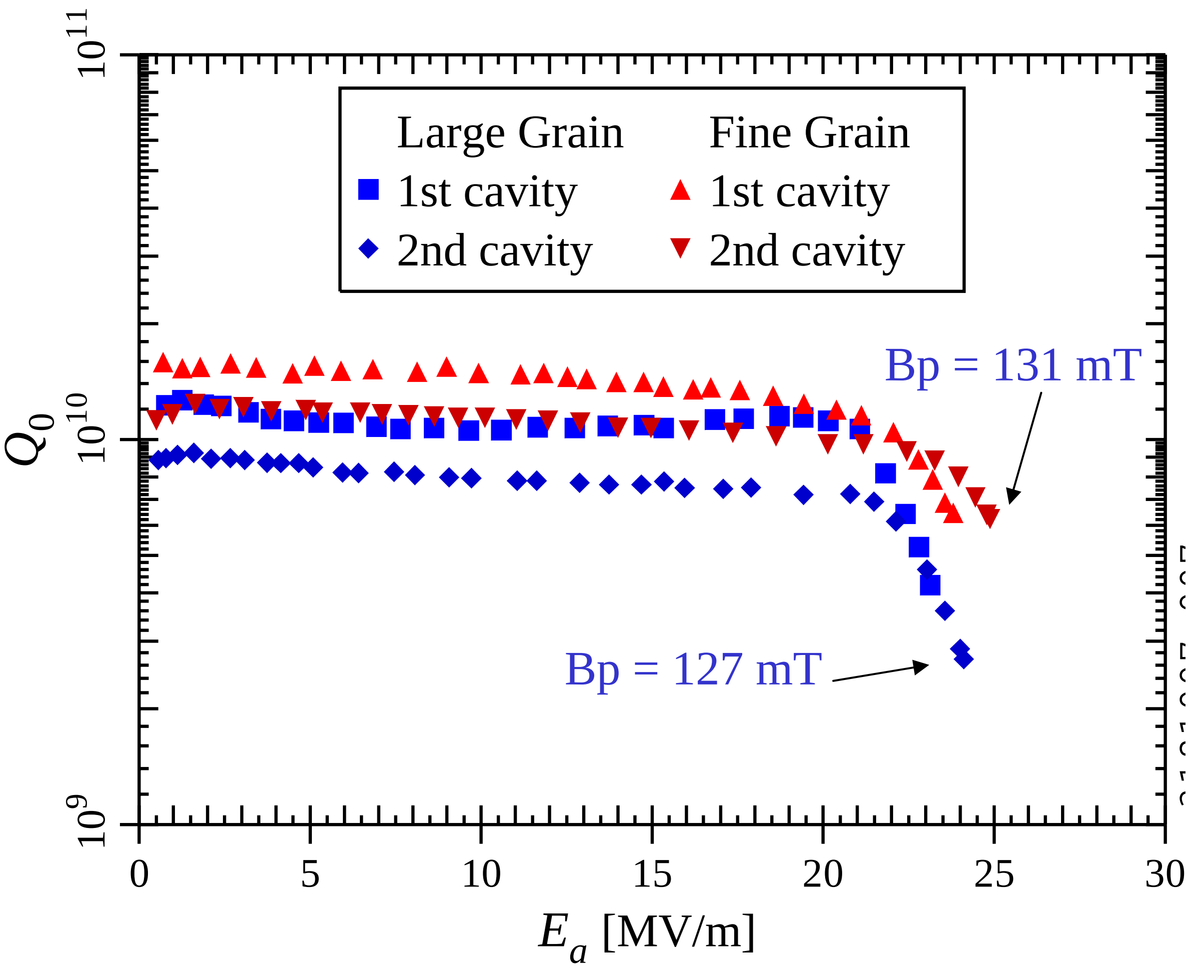
RF Performance

Fine Grain Cavity

- BCP (180 μm)
- HPR (60 min.)
- 1250° C firing for 3 hours in Titanium box
- BCP 50 μm, HPR

Large Grain

- Fired at 600° C for 10 hours for H degassing
- BCP (50 μm before firing, 50 μm after firing)
- HPR (60 min.)
- 1250° C firing for 3 hours in Titanium box
- BCP 50 μm , HPR



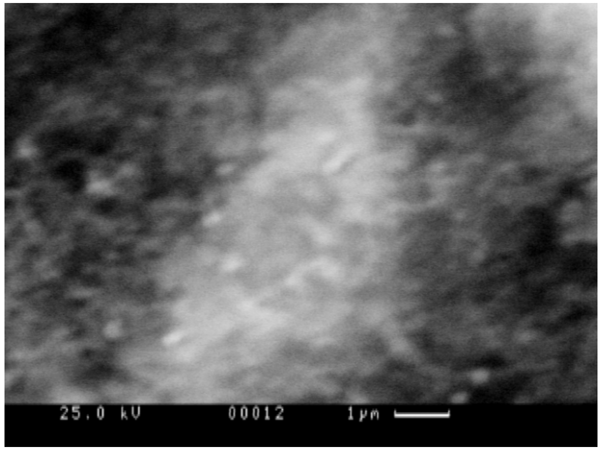
See TUP55 for more information

As-received Ingot Niobium

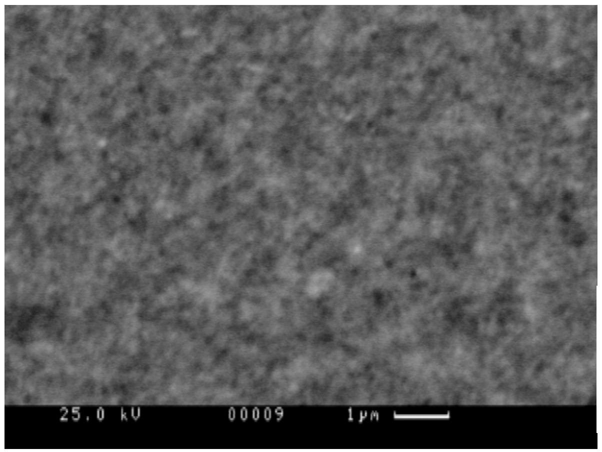
As-received ingot has lots of dislocations

→ affects thermal conductivity (phonon peak)

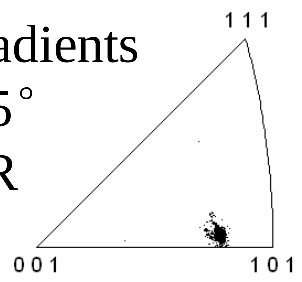
As-received → many entangled dislocations in cell walls



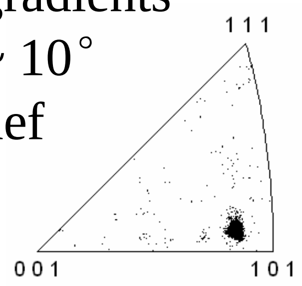
Rx grain → fewer dislocations, evenly distributed



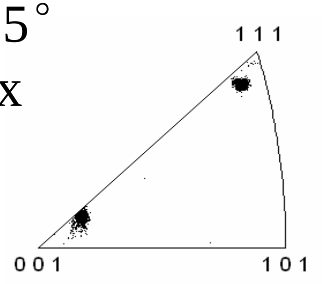
gradients ~ 5° AR

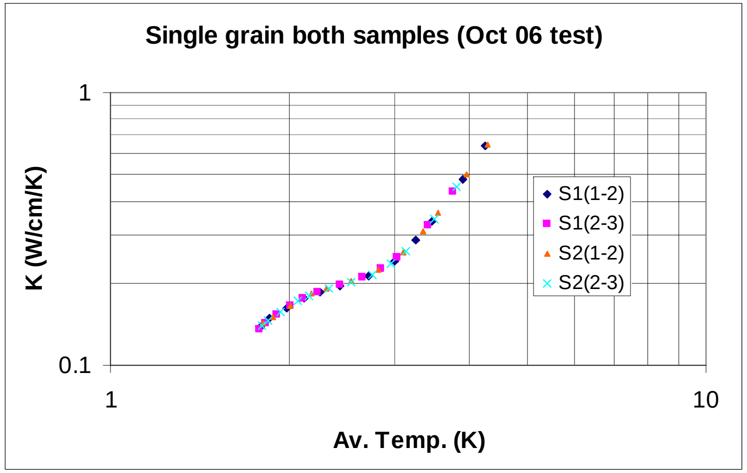


gradients ~ 10° def



gradients ~ 5° Rx

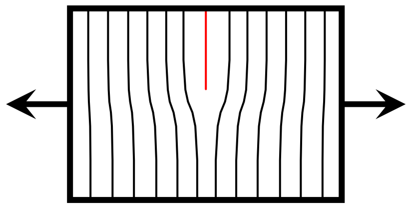




Phonon peak is restored with annealing in a polycrystal

Peak ***spread*** is about the same in as-received and Rx material

→ Nb likes to form dislocations



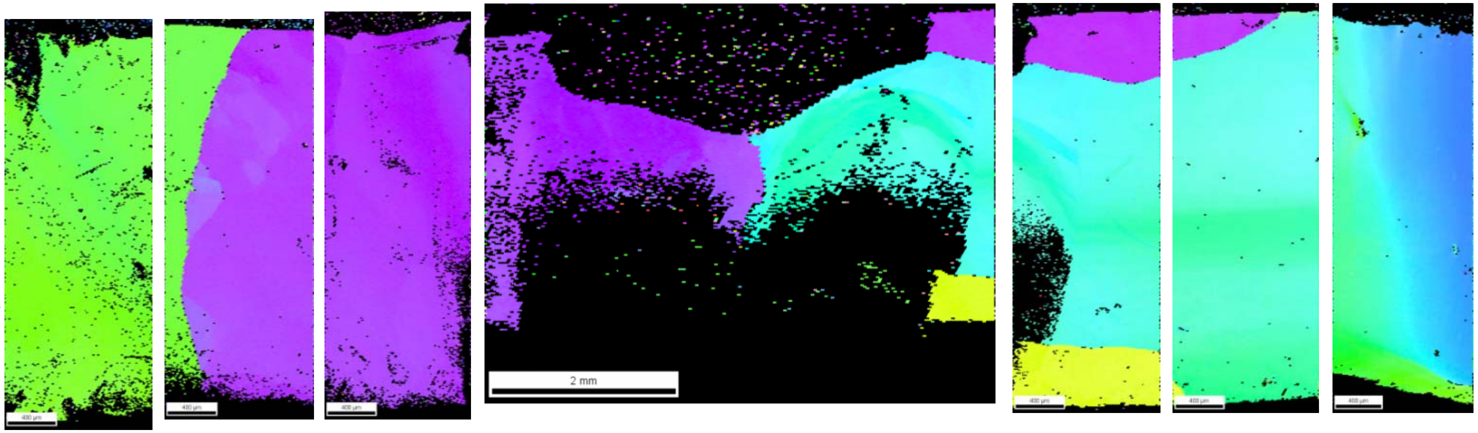
Most compliant direction

See TUP05 for more information

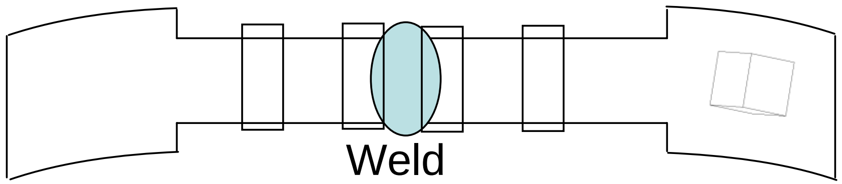
Recrystallization of Single Crystals

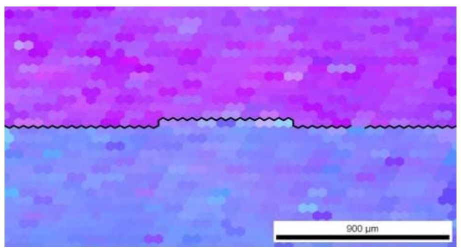
Two deformed (ε~0.4) single crystals with different orientations were cut, then welded together

Original orientation



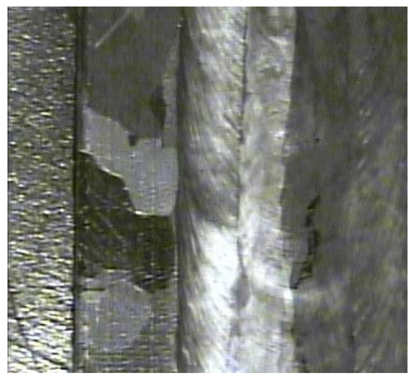
Original orientation





Single Crystal weld of as-received ingot niobium, No Rx

- Original crystal orientations were present from the ends to halfway across specimen
- Middle section recrystallized
- Rx nucleus was between weld and terminal Rx front, determined orientation in weld
- Deformed region has shear bands



Bi-crystal equator weld showing Rx in a Large grain cavity

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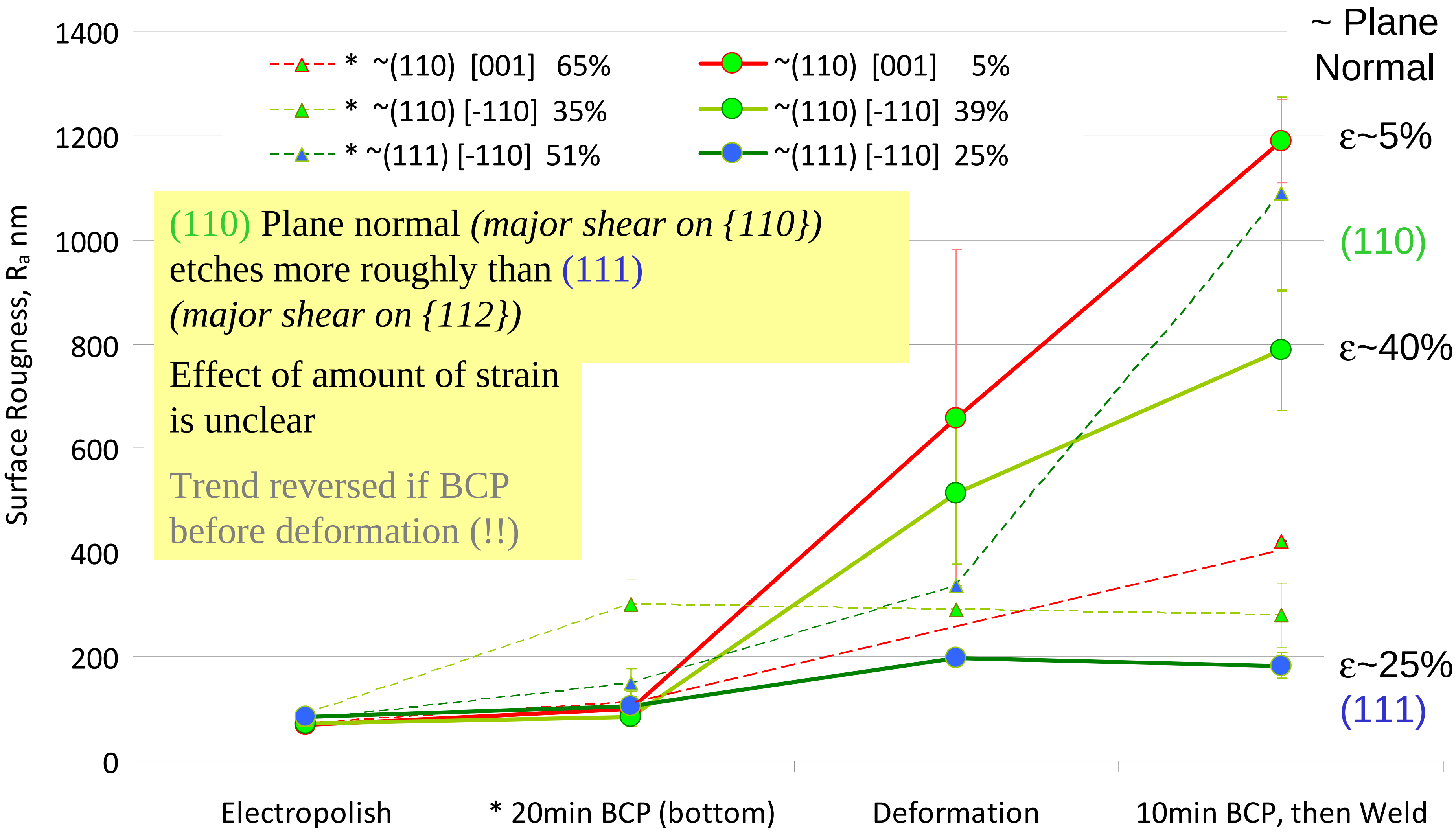
See TUP05 for more information

Processing effects on Single Crystals



See TUP05 for more information

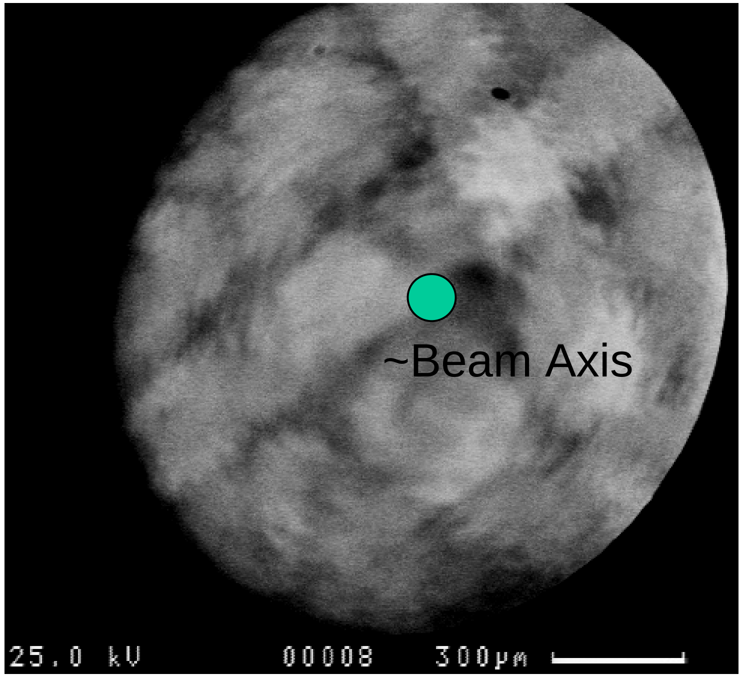
Materials Science

- Crystal orientation and dislocation/defect structure in single crystal Nb may affect
  - Cavity fabrication
    - What are beneficial crystal orientations for deep drawing/hydroforming?
    - More/less dislocations generated within differing crystal orientations upon deformation?
    - Recrystallization upon localized heating has been observed; could appropriate annealing step prevent by reducing dislocation density?
  - Surface Quality
    - Significant difference in etch rate/surface roughness of crystal orientations?
    - Do dislocations/deformation bands at surface lead to significant roughness after chemical treatments?
  - Cavity performance
    - Relation to reduction/loss of phonon peak, effect on heat transfer?
- Research is underway at MSU to investigate these questions
  - Samples of various crystal orientations are being studied before and after steps similar to the cavity forming process

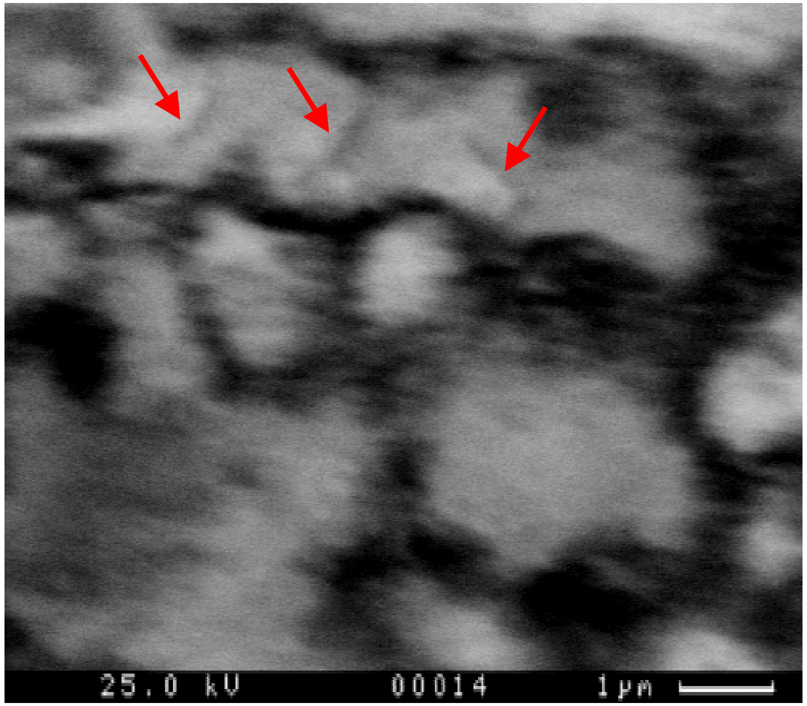
Materials Science

ECCI technique used to infer dislocation orientation and density

By first aligning the SEM beam to a particular crystal direction (left), electron channeling contrast imaging (ECCI, right) is used to image dislocations (noted by red arrows)



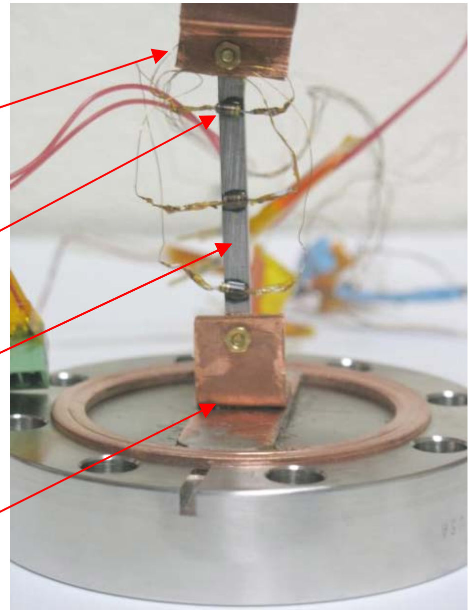
Grain I SACP Image ~-5° tilt

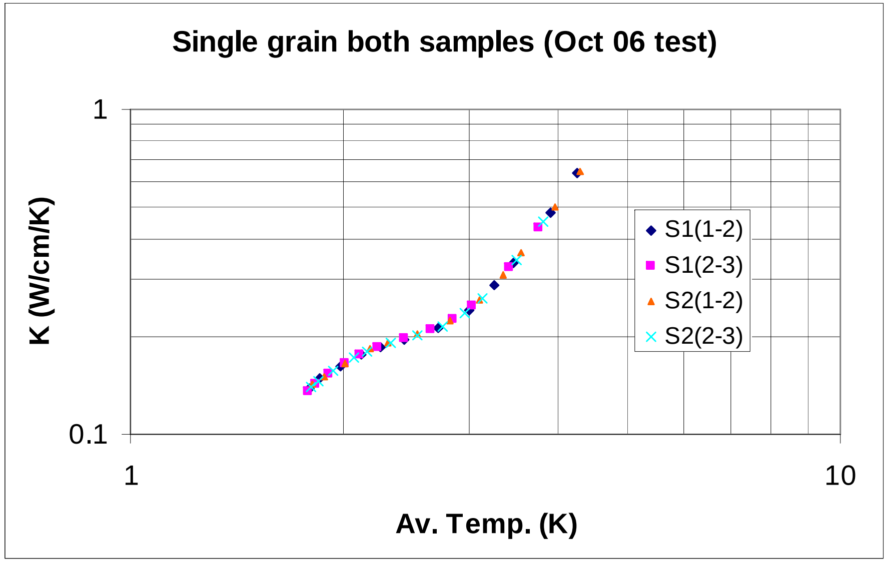


Fundamental research of single crystal Nb will determine the relationship between dislocation density/orientation and recrystallization while extending knowledge of how these impact cavity fabrication and performance.

Heat Transfer Studies [1]

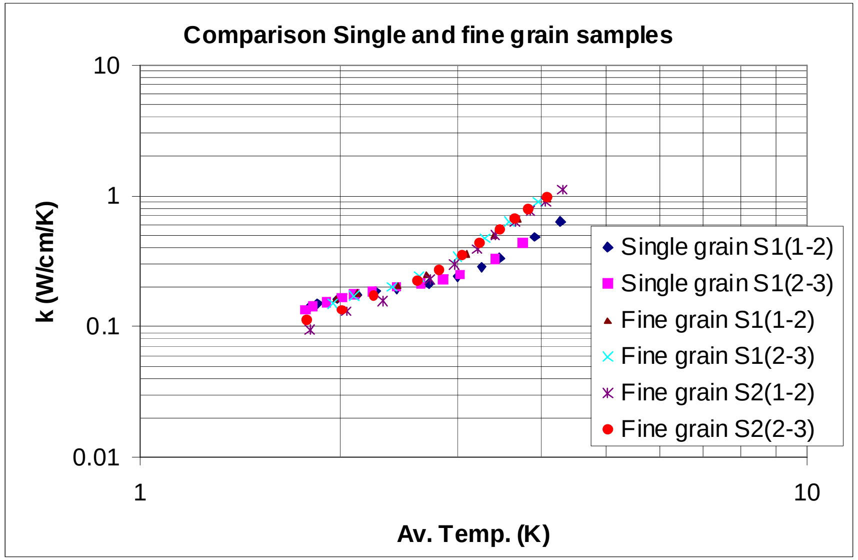
- Below 3 K, heat transfer is dominated by phonon movement
- Elements that may slow phonon movement
  - Grain Boundaries
  - Strain (dislocations)
- Heat Transfer Results
  - Single Crystal
  - Bi-Crystal

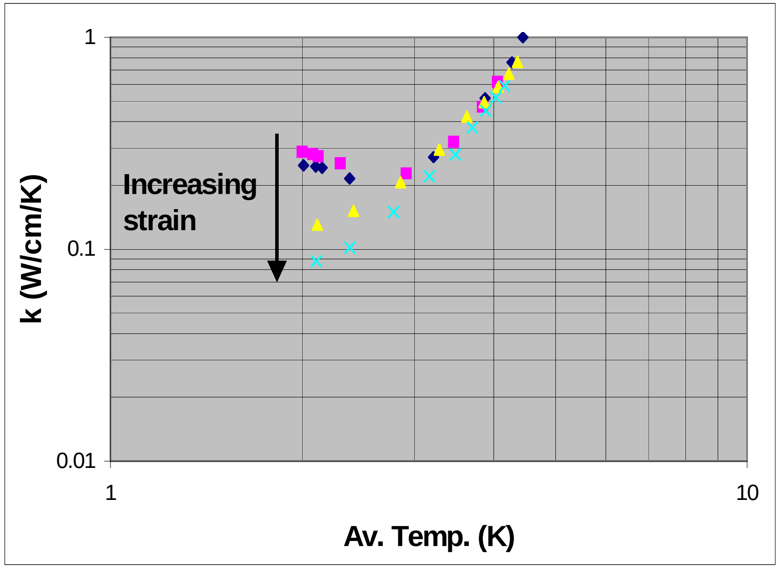




Heat Transfer Studies [2]

- Comparing Single Crystal to Fine Grain
  - Fine Grain (50 µm) [as received]
  - Single Crystal
  - Bi-Crystal
- Samples not annealed, lost phonon peak due to strain?





*Paper presented at ASC 06

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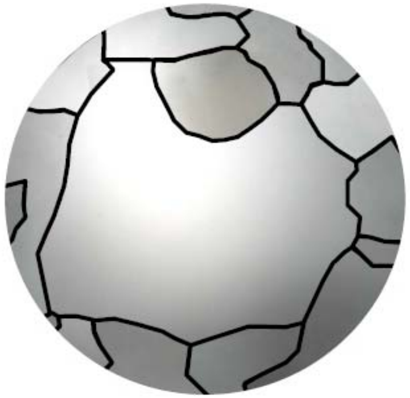
to Hot Topics Session

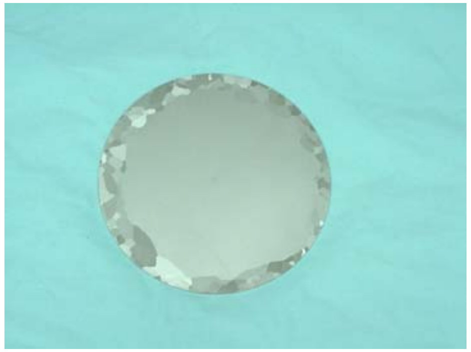
Is large grain/ single crystal Nb an alternative material to polycristallline niobium ?

Waldemar Singer

DESY

- Is grain orientation important for fabrications or performance?



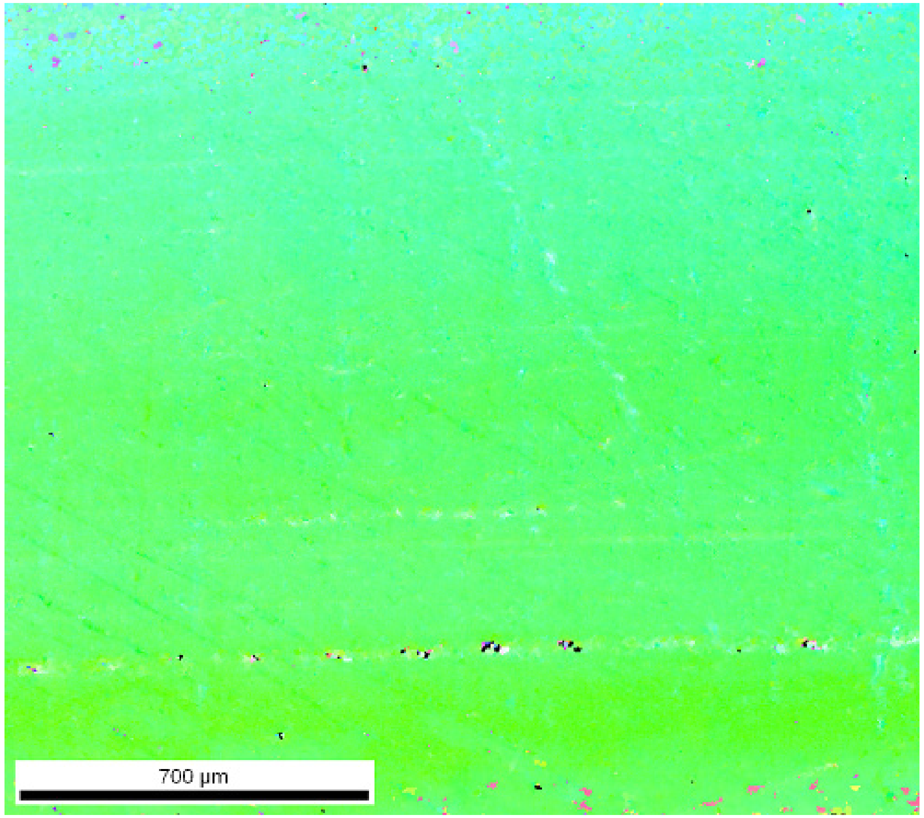


Performance: Experiences are not sufficient:

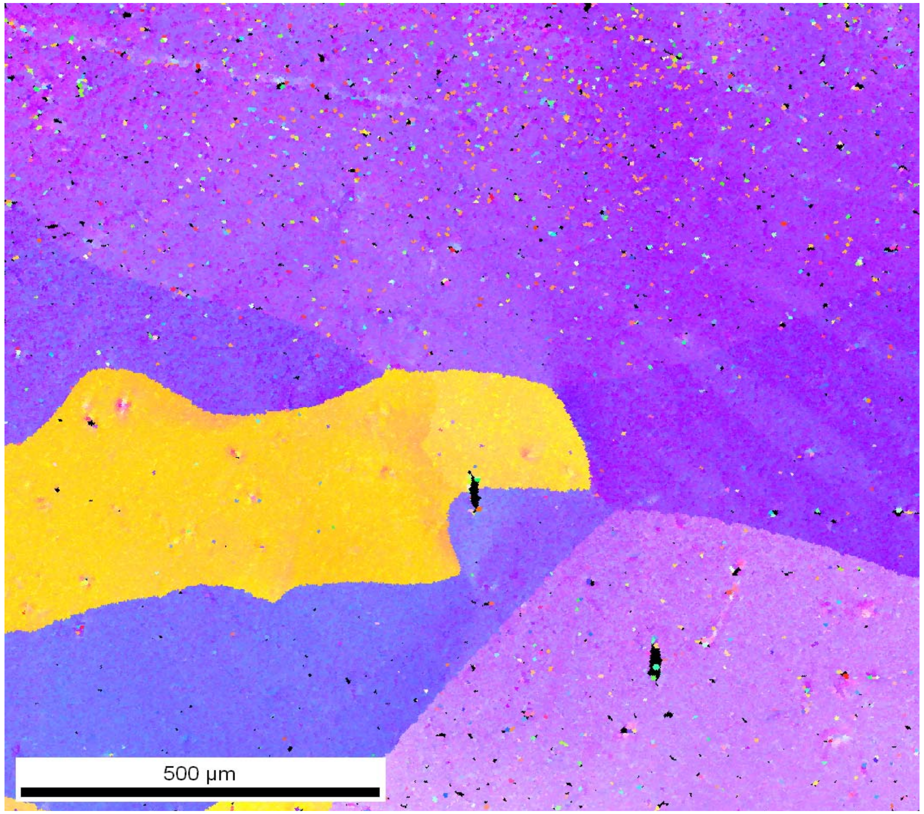
HERAEUS SC: (100) like orientation
Hquench 166 mT after BCP

CBMM SC: (111) like orientation
Hquench 174 mT after EP

Few preliminary results of different Labs get on the at DESY prepared set of Nb single crystal samples



Orientation stability of Nb SC (100) after 50% cross rolling <110> and annealing at 1200 ^{o}C for 3 hours

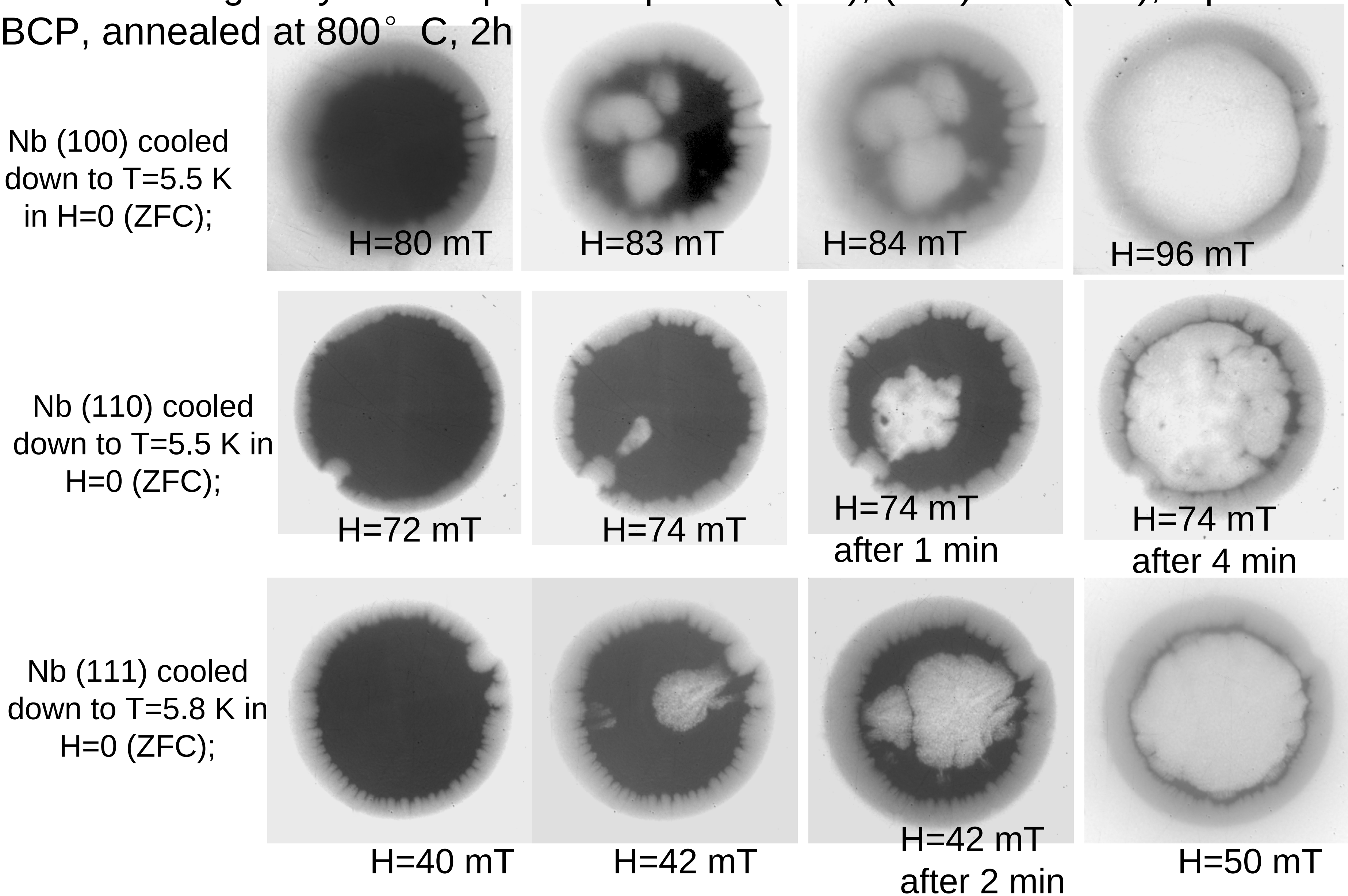


Recrystallization in Nb single crystal (111) after 50% rolling <110> and annealing at 1200º C for 3 hours

Fabrication from sheets with the orientation plane (100) parallel to sheet surface is more preferable

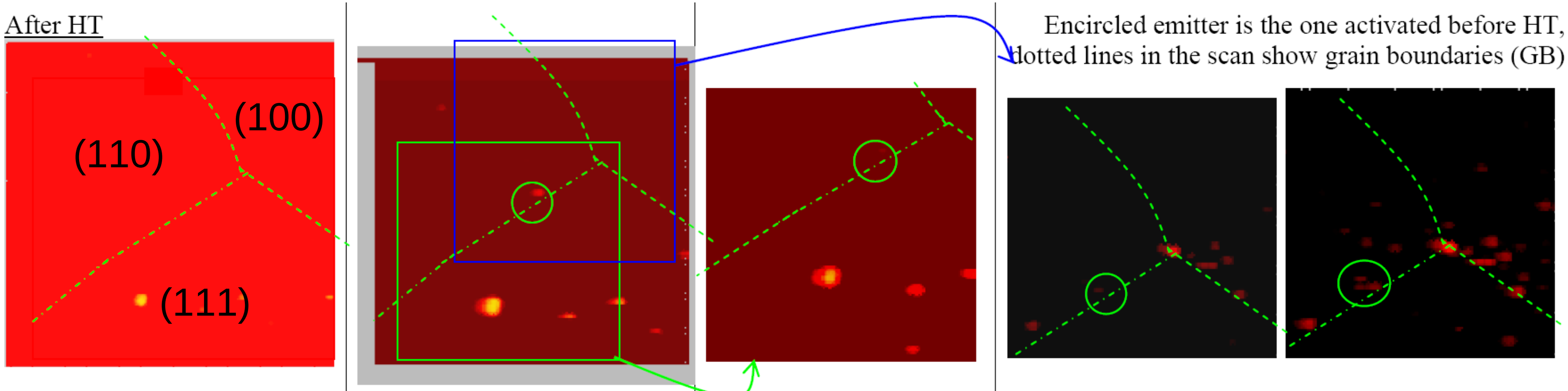
A.Ermakov, V.Levit etc. EUCAS2007

Three Nb single crystal samples with planes (100), (110) and (111); 5 µm BCP, annealed at 800° C, 2h



Orientation plane (100) parallel to surface is more preferable

(A. Polyanskii etc. Poster TUP12)



@ 90MV/m, (10 mm)^2 Emitters activated during V-scans before HT.

@ 150 MV/m, (7.5 mm)^2

@ 200 MV/m, (5 mm)^2 4 old +1 new emitters observed.

@ 250 MV/m, (5 mm)^2 @ 300 MV/m, (5 mm)^2 Increased number density after HT, all emitting sites appear near GB. Strongest emission observed at the intersection of three grain boundaries.

FE scans on three LG after 100 µm BCP and baking 150° C, 14hs

Sample	Number of emitters			
	@ 120 MV/m over (10mm)^2	**@ 150 MV/m over (7.5mm)^2**	**@ 200 MV/m over (5mm)^2**	**@ 250 MV/m over (5mm)^2**
SCNb3, 100µm (110)	0	0	3	9
SCNb4, 100µm (111)	0	0	2	7
SCNb5, 100µm (100)	0	0	2	3

Poster

A.Dangwal etc. WEP24 SRF2007

Orientation (100) is more preferable.