

DE LA RECHERCHE À L'INDUSTRIE



# PHYSICAL PROPERTIES OF NIOBIUM:

*Origin of the specifications for  
fabrication of SRF cavities.*

TUTORIAL

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CEA, Irfu, SACM, Centre d'Etudes de Saclay, 91191 Gif-sur-Yvette Cedex,  
France

## Purity issues (*RRR, thermal properties...*)

## Mechanical properties of high purity Nb

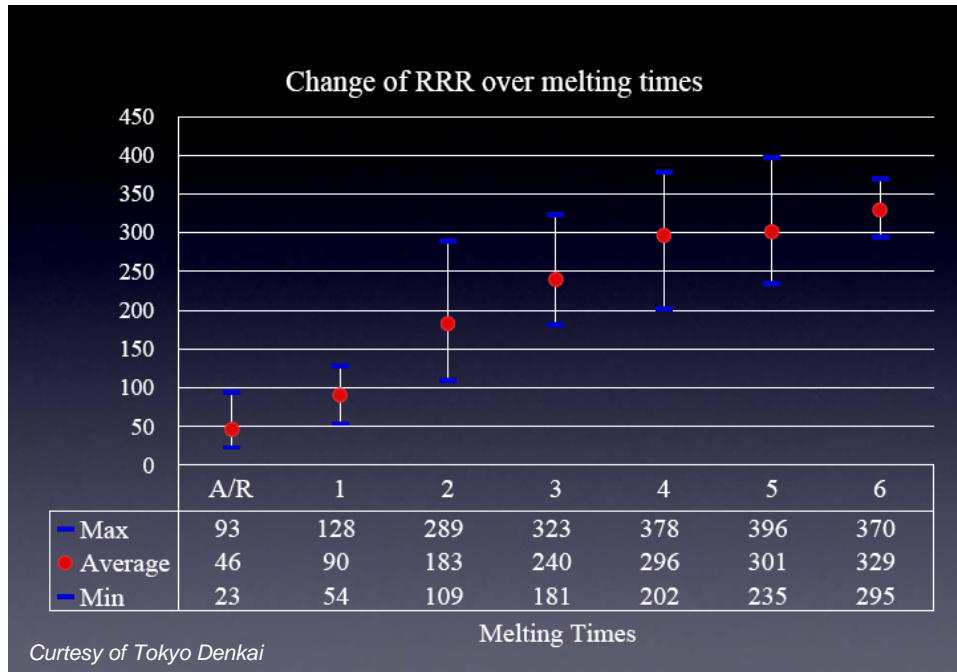
*Elastic vs plastic properties,  
Recrystallization and recovering, influence of grain size,  
Cavity forming (strain hardening, tensile curves...), influence of welding  
Specifications, reception controls,  
Low temperature behavior,  
Examples of problem in industrial production  
Large grain issues*

## Surface state

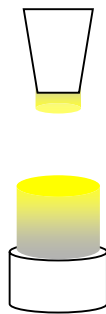
*Damage layer  
Chemistry aspects  
Surface morphology and Quenches*

# PURITY ISSUES

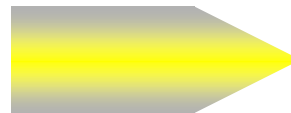
# HIGH RRR MATERIAL



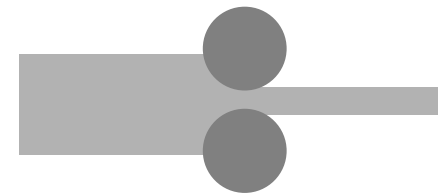
Electron  
beam melting



Hot Forging (air)



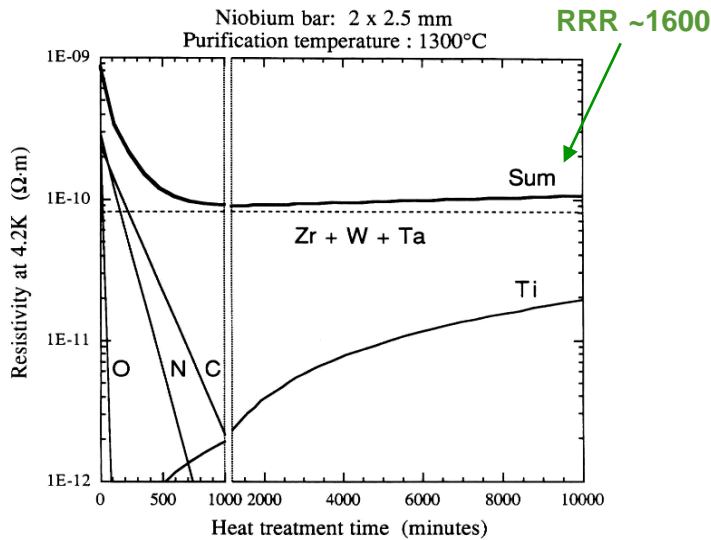
Rolling + recovering



# NIOBIUM PURIFICATION: $\lambda_T \propto RRR \propto \text{PURITY}$

## Purification annealing with a getter (Ti)

- $\Delta G^\circ (\text{TiO}_2) < \Delta G^\circ (\text{Nb}_2\text{O}_5)$
- Moderate vacuum, temperature
- Diffusion limited  $\Rightarrow$  **Issues for macroscopic objects :**



Impurity	$\Delta\rho/\Delta C$ (nΩ.m/At ppm)
N	0.52
O	0.45
C	0.43
H	0.08
Ti	0.096
Ta	0.025

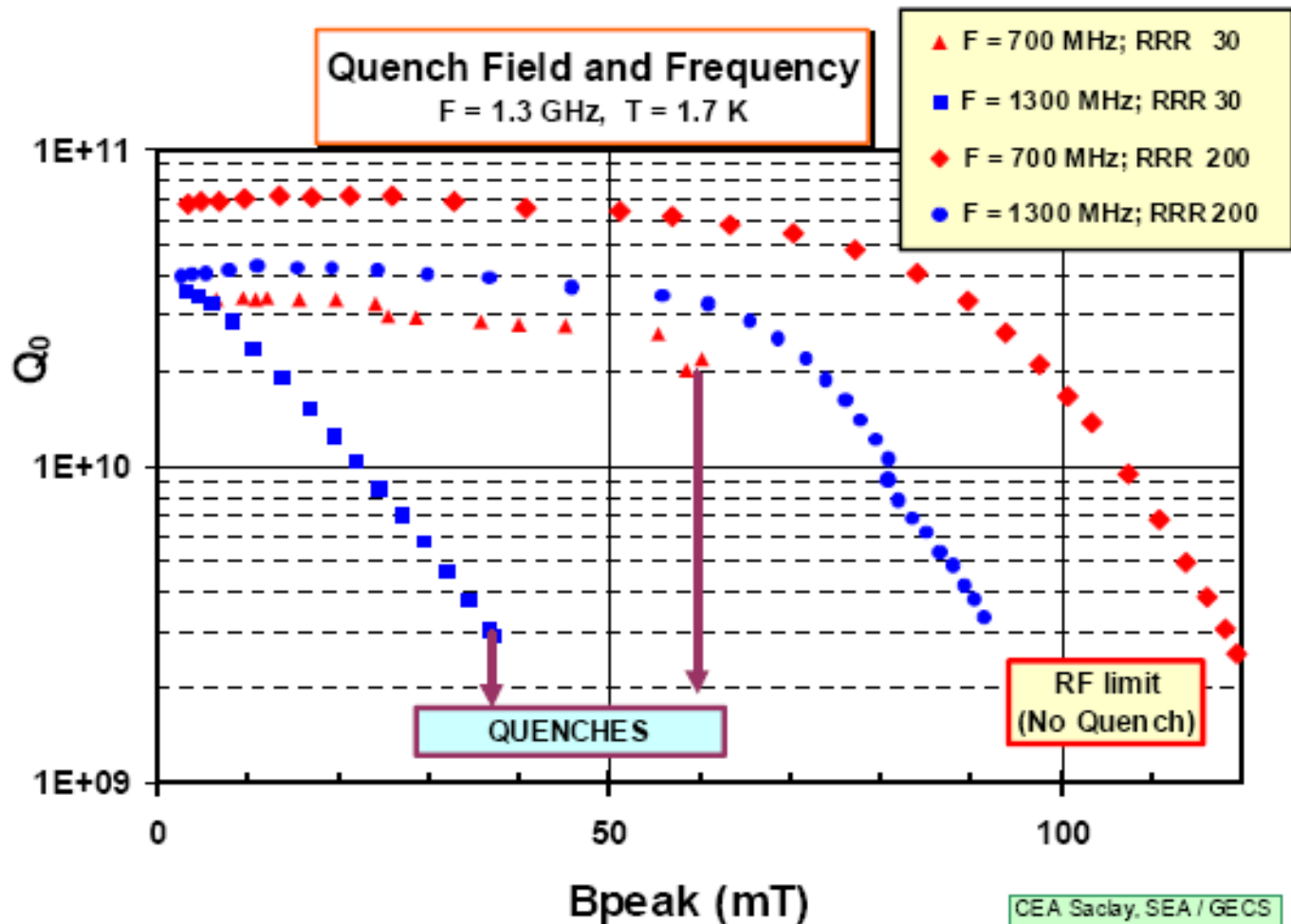
- Only light elements contribute to thermal behavior (e<sup>-</sup> scattering) up to RRR ~800
- metallic impurities : homogeneous after EB melting
- Inclusions appear during manipulation (e.g. dust embedded in soft Nb)
- Ta content : plays on RRR, not on thermal behavior

Origin	Commercial	RF application	Post-purified (cavities)	Post-purified (samples)	Other preparation	Theoretical
RRR	<b>30-50</b>	<b>200-300</b>	<b>600-800</b>	<b>Up to 1800</b>	<b>5-6000</b>	<b>33000</b>

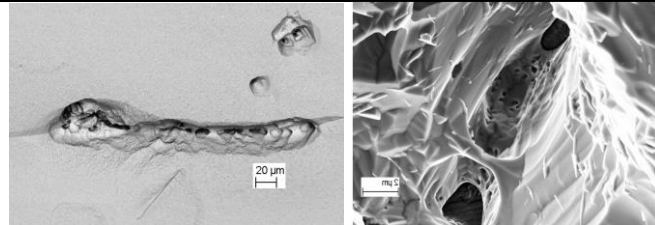
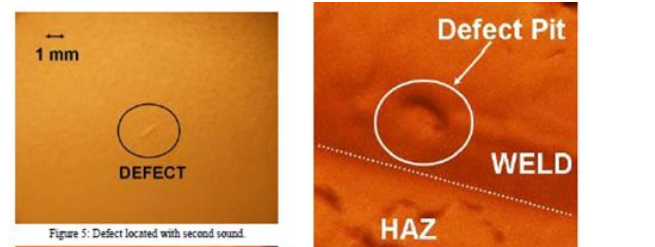
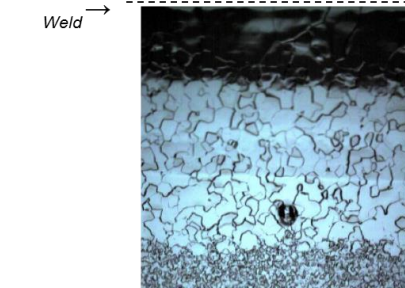
*light impurities* ←

→ *metallic impurities, lattice defects*

- Quench field depends a lot on RRR



# SURFACE DEFECTS AND QUENCH

Defect type	origin	Quench field
Bubble, seen by XR $\varnothing$ 0.5 mm	Saclay, Bad EB welding	$\sim 12$ MV/m
Bad vacuum during EB welding	Experience at Saclay and DESY	20-25 MV/m
	Desy Bad vacuum EB welding ?	$\sim 16$ MV/m
	DESY Bump, defect in the deep drawing die	$\sim 20$ MV/m
Ta inclusion (un-cleaned rolling machine)	DESY	8 to 14 MV/m
	FNAL Pit, in the HAZ	$\sim 15$ MV/m

(tesla shape)

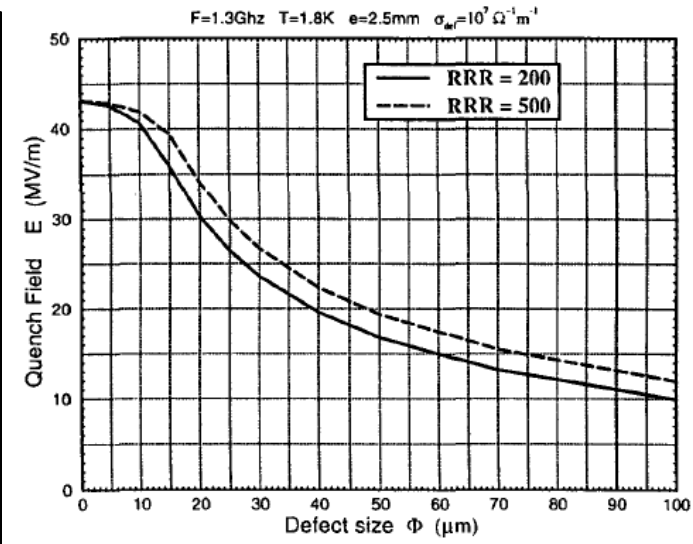


Figure 8- Quench field as a function of defect size.

[H. Safa, 1995]

<http://accelconf.web.cern.ch/accelconf/SR/F95/papers/srf95c10.pdf>

Quench field < 15-20 MV/m:

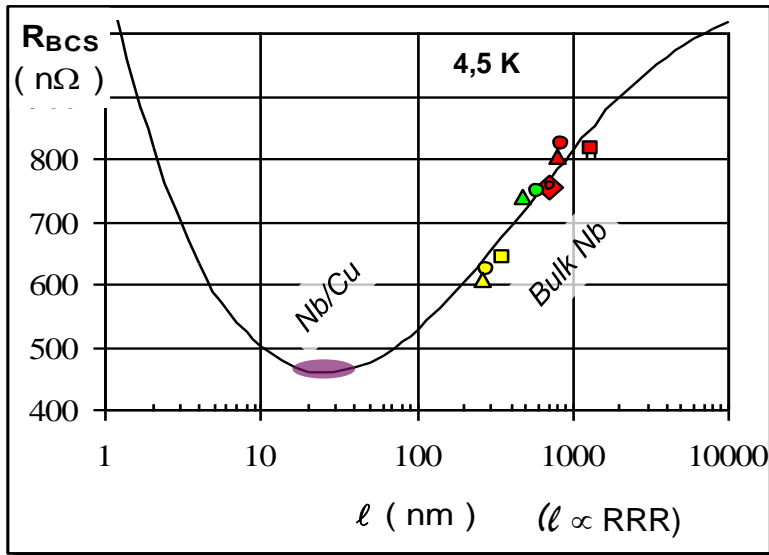
- Defect  $\sim 50$ -100  $\mu$ m
- You can see it with the eye !

# WHY HIGH RRR ?

Surface resistance:

$$R_S = R_{BCS} + R_{Res}$$

$$R_{BCS} = A(\lambda_L^4, \xi_F, \ell, \sqrt{\rho_n}) \frac{\omega^2}{T} e^{-\Delta/KT}$$



Thermal conductivity

$$K_s(T) = R(y) \left[ \frac{\rho_{295 K}}{L RRR T} + aT^2 \right]^{-1} + \left[ \frac{1}{D \exp(y)T^2} + \frac{1}{BIT^3} \right]^{-1}$$

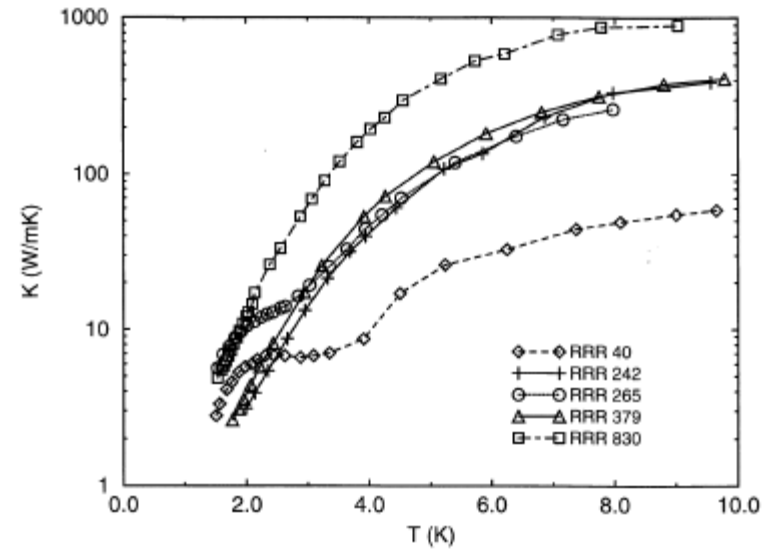
$$L = 2.45 \times 10^{-8} \text{ W K}^{-2}$$

$$a = 2.30 \times 10^{-5} \text{ m W}^{-1} \text{ K}^{-1}$$

$$\alpha = 1.76$$

$$B = 7.0 \times 10^3 \text{ W m}^{-2} \text{ K}^{-4}$$

$$1/D = 300 \text{ m K}^{-3} \text{ W}^{-1}$$

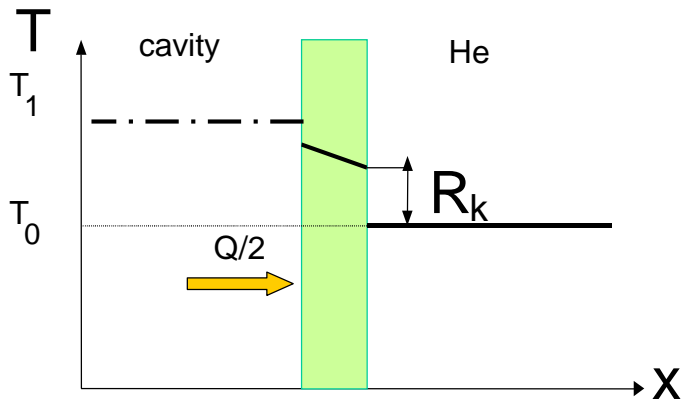


[Koechlin, Bonin 1996]

High RRR not required for superconductivity  
But for thermal stabilization of defects



# THERMAL TRANSFER: $\lambda_T \propto RRR \propto \text{PURITY}$

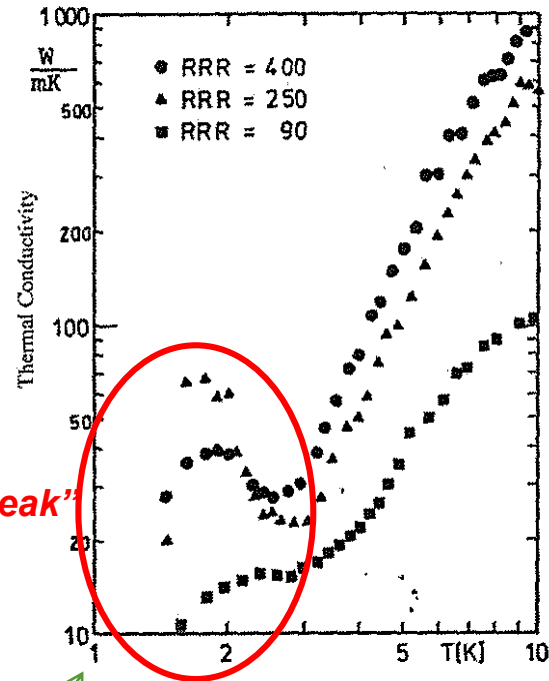


$$\lambda_T = 1/K$$

[See J. Amrit, Orsay, and publications from MSU]

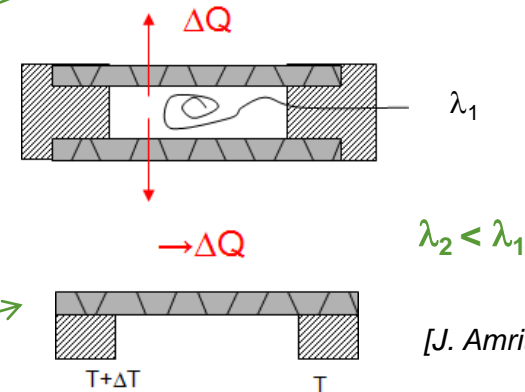
$$\Delta T = R_{tot} \frac{\dot{Q}}{2S}$$

$$R_{tot} = 2R_K + \left(\frac{e}{K}\right)$$



“phonon peak”

- Thermal resistance depends on the thermal conductivity and on interface Kapitza resistance
- Kapitza resistance varies in function of surface state
  - Its influence is negligible in polycrystalline material (thermal conduction is dominant)
  - becomes noticeable in large grain material (high thermal conduction)
- Phonon peak depends on the crystalline quality of the material (disappears with  $\sim <10\%$  deformation)
- Thermal conductivity is influenced by GB (large grain material)



[J. Amrit]

# **MECHANICAL PROPERTIES**

Mechanical properties depend strongly of :

- The material purity
- Its deformation/crystallization state
- Very narrow freedom to monitor it (grain size, cold work, annealing)
- => **compromise!**

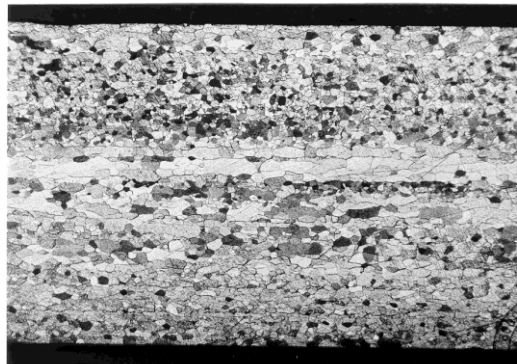
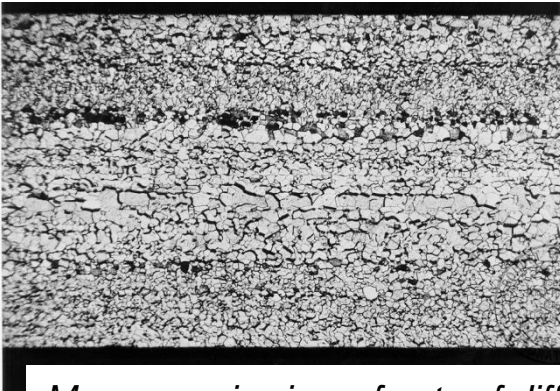
Mechanical properties in cause :

- For forming of material (deep drawing, machining) => plastic parameters
- For mechanical behavior of the completed cavity/object => elastic parameters (=  $f(T)$  !)
- => **not the same parameters in concern ! Opposite requirements**
- => **compromise again !**

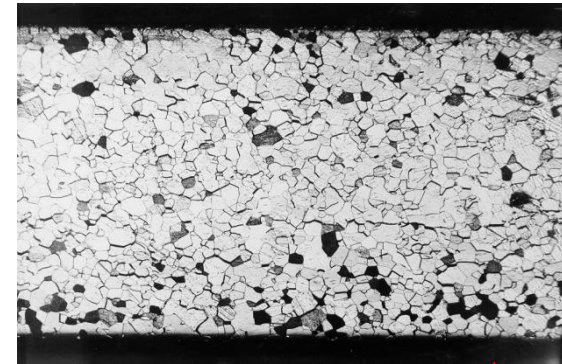
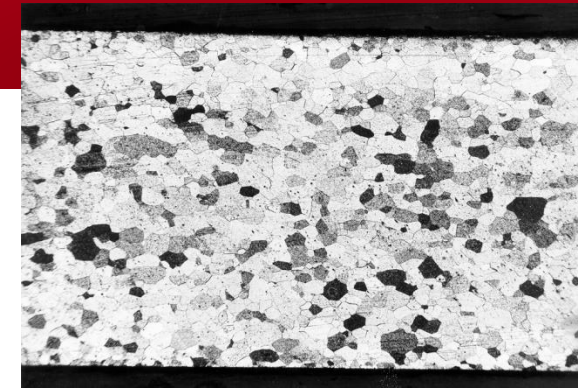
**FORMING:  
WHAT KIND OF MATERIAL  
IS NEEDED**

# GRAIN SIZE SPEC. => FORMABILITY

- **> 90% recrystallized :**
  - For pure Nb : recrystallization  $\equiv$  recovering  
=> full plasticity
- **ASTM 5 (0.65mm) or ~~finer~~**
  - Deformation is more uniform with small grains
  - Orange peel  $\downarrow$
  - Small grain.  $\uparrow$  Y. S. (Hall-Petch Law  $\sigma = \sigma_0 + K(d)^{-1/2}$ )
- **uniform grain size !!!!**
  - $\downarrow$  Risk of tearing



Macroscopic view of cuts of different niobium sheets (2 mm thick)

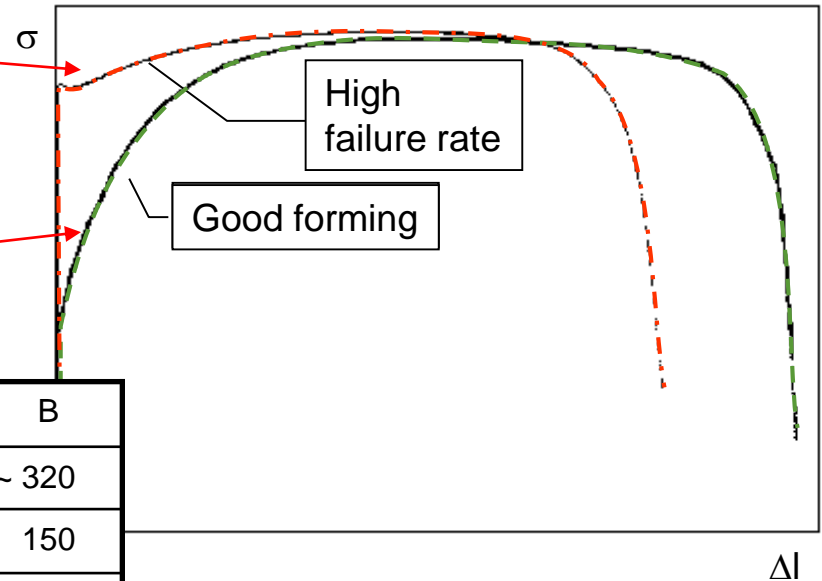
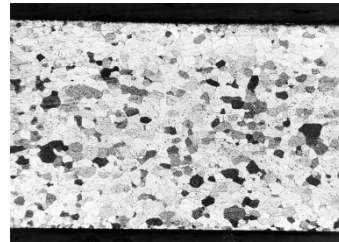
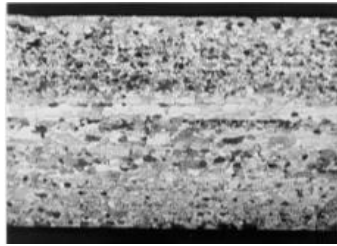


*normal forming.*

*high level of tearing.*

**But... too small grains => no improvement LTB => compromise !**

# TRACTION CURVES & FORMABILITY

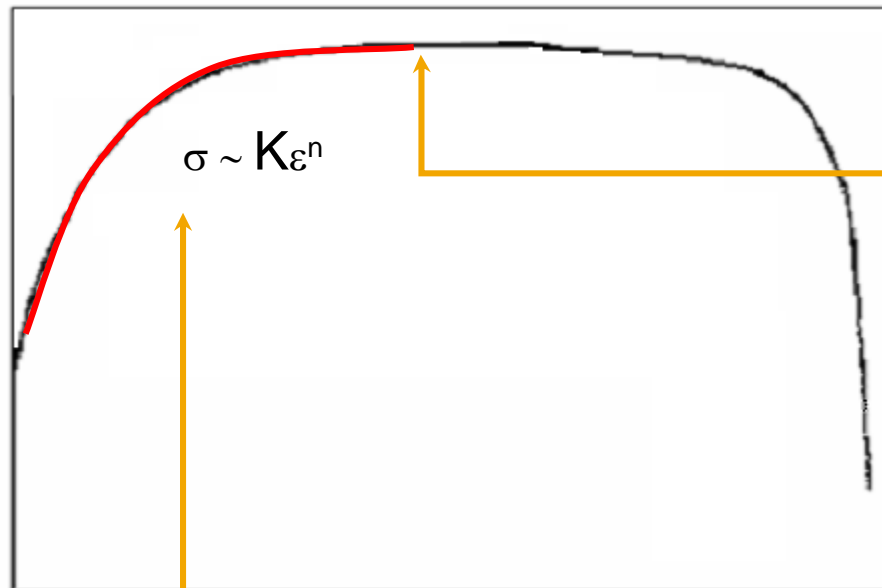


Mechanical properties \Batch	A	B
RRR	~ 310	~ 320
Yield Strength $\sigma_{0.2}$ (MPa)	<b>66</b>	150
Tensile Strength $\sigma_m$ (MPa)	180	183
Elongation A (%)	59	40
Strain Hardening Coef. n	<b>0.31</b>	0.10
Hardness Hv	<b>56</b>	65
Grain size (ASTM)		
- core	<b>4</b>	5
- surface	<b>4</b>	6
Forming Aptitude	GOOD	BAD

ASTM #6 : 45  $\mu\text{m}$   
 ASTM #5 : 64  $\mu\text{m}$   
 ASTM #4 : 90  $\mu\text{m}$   
 ASTM #3 : 125  $\mu\text{m}$

# TRACTION CURVE AND FORMING

- Plastic deformation description @ mono axial deformation
- Not very accurate for elastic data (estimation only)
- Not very accurate for bi-axial deformation (estimation only)



*Uniform elongation stops there !*

*Total elongation not very relevant !*

*Strain hardening coefficient :  
formability ↑ if n ↑*

*Bi axial deformation :  $\epsilon_{max}$  varies  
like  $4/11(2n+1)$*

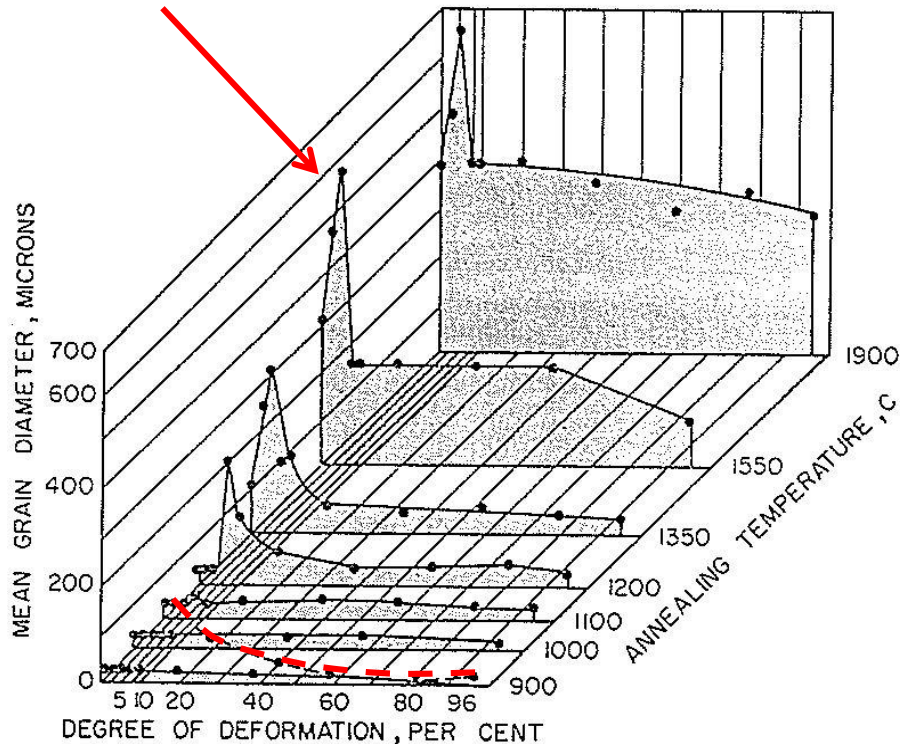
Metal	n
softened steel	0.15 - 0.25
austenitic steel 18-10	0.4 - 0.5
aluminum	0.07 - 0.27
copper	0.3 - 0.47
zinc	0.1
nickel	0.6
Nb	n
RRR 270 $\epsilon \sim 50\%$	0.075
RRR 270 recrystallized	0.287
NbUHP	0.45
Nb+ 80 Wppm O	0.45
Nb+ 230 Wppm O	0.45
Nb+ 330 Wppm	0.45

**RECRYSTALLIZATION:  
DETERMINES THE QUALITY OF THE  
MATERIAL  
(SUPPLIER SURVEY MANDATORY)**



## Critical deformation:

only high deformation leads to small grain recrystallization



Commercial Nb  
RRR ~ 50-100

## Deformation > 65% =>

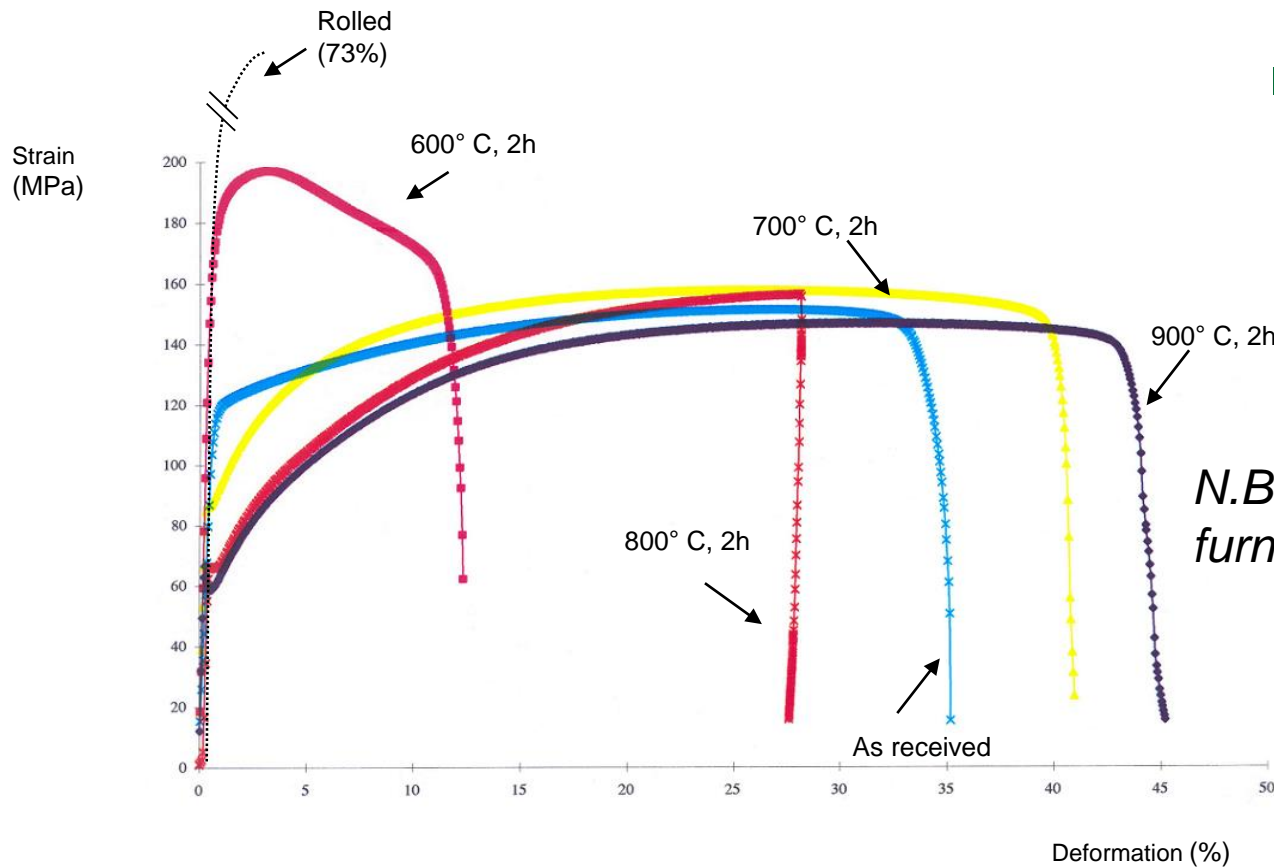
- uniform nucleation
- small grains
- if purity  $\uparrow$ ,  $T_{\text{recryst}} \downarrow$ 
  - RRR  $\leq 100 \Rightarrow T_{\text{recryst}} \geq 900 \text{ C}$
  - RRR 300  $\Rightarrow T_{\text{recryst}} \sim 800 \text{ C}$
  - RRR 400  $\Rightarrow T_{\text{recryst}} \sim 750 \text{ C} ?$
- Large grain material
  - Recrystal<sup>n</sup> into smaller grains

## Recommended : 2h, 800 C :

- Removes cold work
- Also removes H

*N.B. careful monitoring of furnace temp.!*

# STRAIN-STRESS CURVES => FORMABILITY



■ Formability : you need recrystallized material !

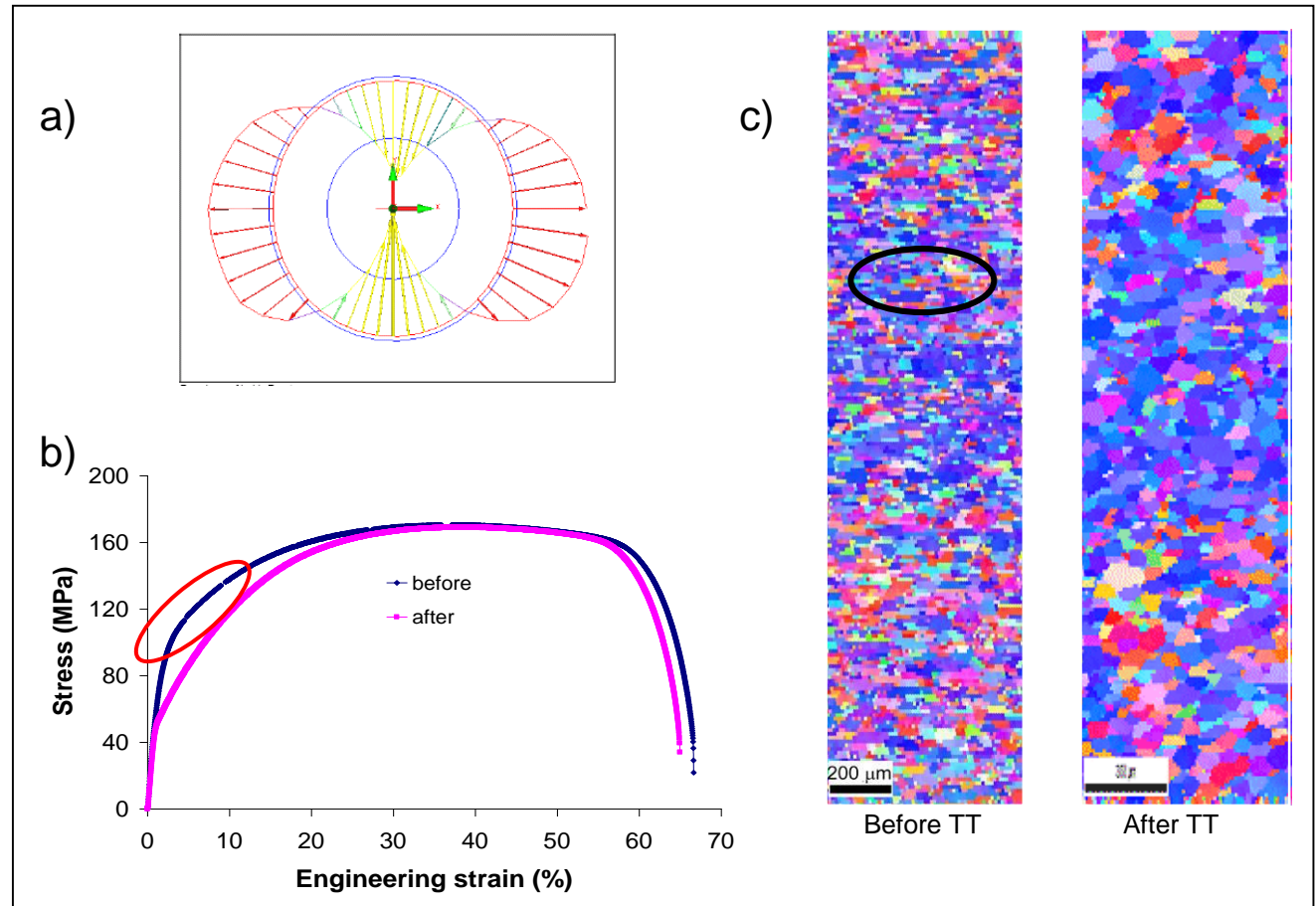
*N.B. careful monitoring of furnace temp.!*

**Mechanical resistance : you also need recrystallized material !**  
*( it is the only way to know which are the mechanical properties !)*

# EXAMPLE OF INDUSTRIAL PRODUCTION'S PROBLEM

- Too stringent grain size specification (30  $\mu\text{m}$ )
- Incomplete recrystallization in order to comply to specifications
- => forming problems !

QA issues: better have less stringent specifications, but check you meet all requirements.  
Here grain size was met but not "90% recrystallized"



[Fermilab, ~2006]



If specifications are met, then most of the big defects come out :

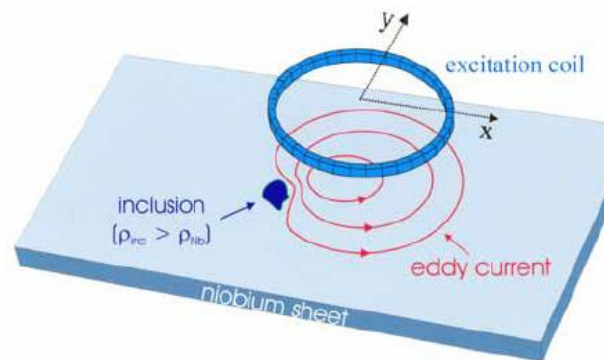
- Inclusion: dust embedded during deep drawing
- Welding void or strain
- Strain corrosion, chemical residue



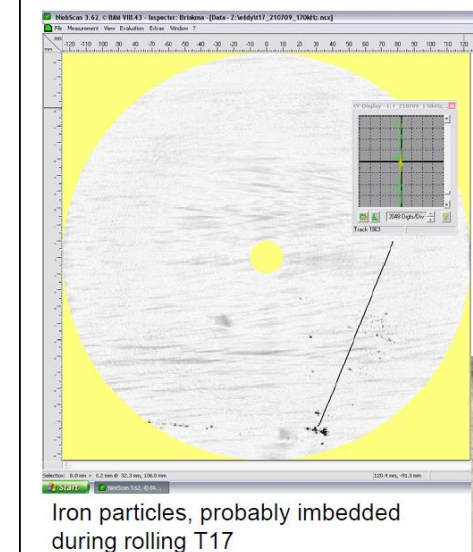
Search for clusters in Nb sheets.  
Eddy current system.



DESY eddy current scanning apparatus for niobium discs. 100% Nb sheets for TTF scanned and sorted out. **Feed back to Nb producer was very important**



Principle of eddy current measurement



# **MECHANICAL RESISTANCE & COLD BEHAVIOR**

Official values:

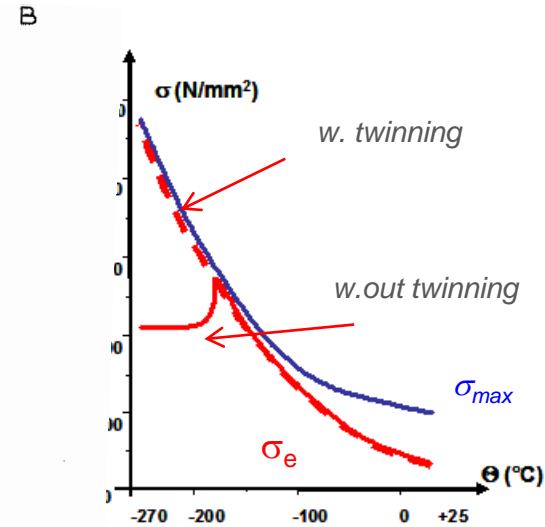
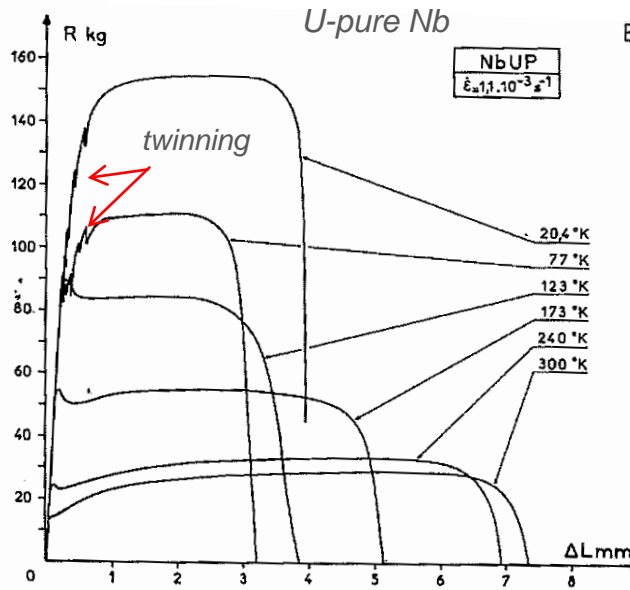
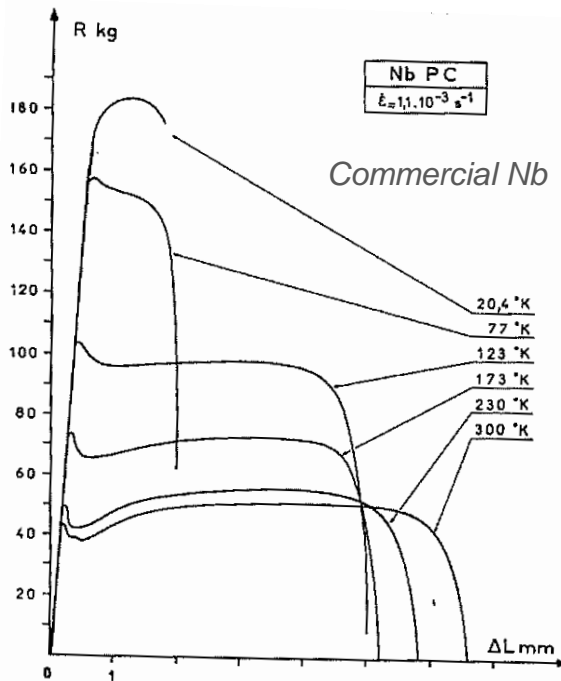
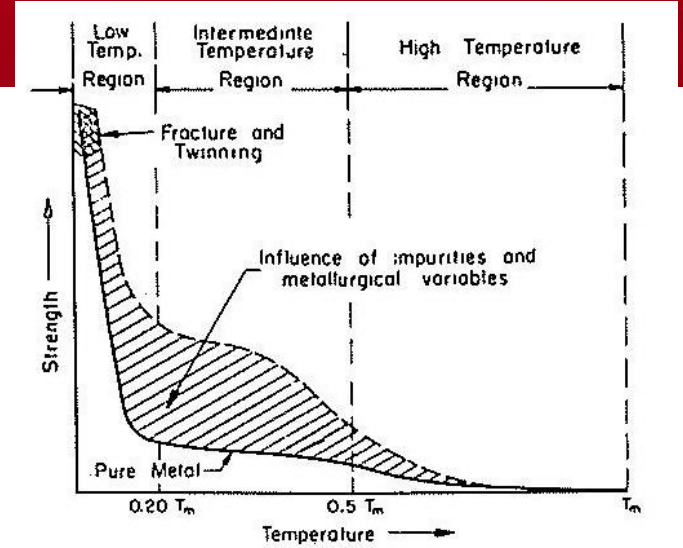
- Young modulus **E = 104.9 GPa**
  - Intrinsic
  - Depends on crystalline orientation(=> mean value)
  - Might be an issue in case of large grain material
  - Is somewhat higher @ low temperature ( $\Delta \sim 10-15\%$ )
  - Not accurately measured in traction tests ( $\Delta \sim 10-15\%$ )
- Poisson coefficient  **$\nu = 0.397$**
- Y.S. depends on the crystalline state (cold working) + T

**Mechanical resistance : you also need recrystallized material ! \***  
*(it is the only way to know what are the mechanical properties !)*

*\* Exception : flange material : forged Nb or NbTi (to increase hardness)*  
*NbTi : SC, but high  $R_S$  : make sure it does not see field*

# LOW TEMPERATURE BEHAVIOR

- Y.S. ↑↑ when  $T_p \downarrow$
- ductility ↓
- ! @  $T_p < 20 \text{ K} \Rightarrow \text{Nb}$  is brittle : no plastic deformation
- ! During cooling procedure

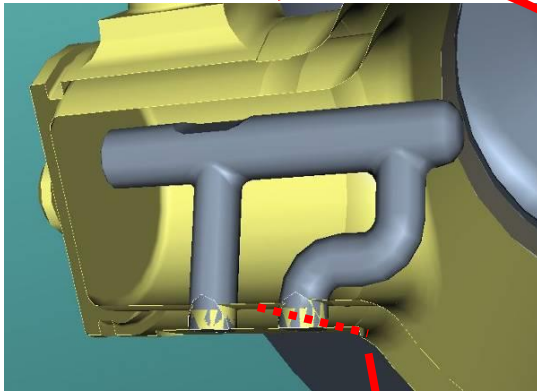


[J. F. Fries, PhD, Paris XI, 1972]

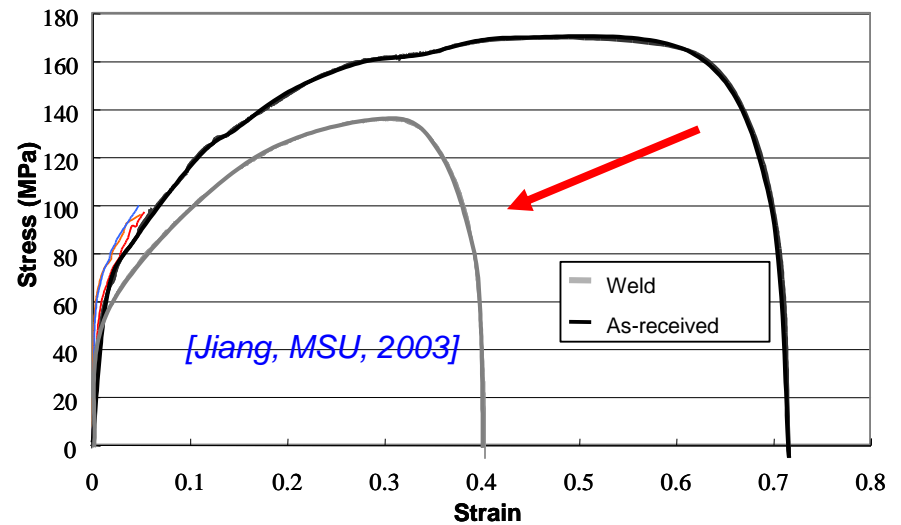
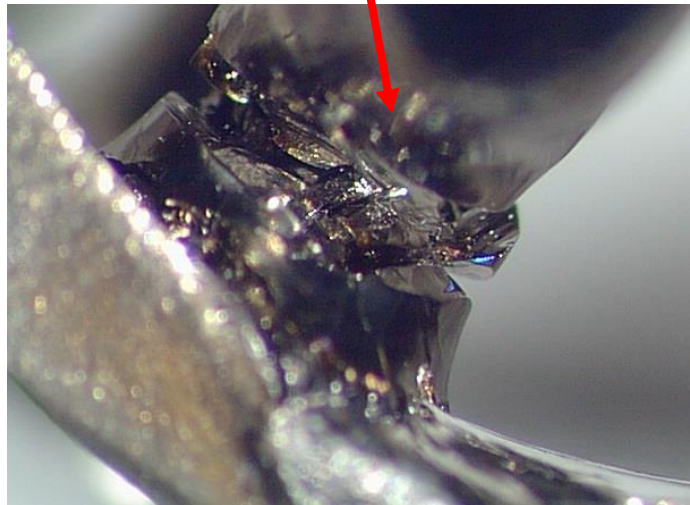


# ISSUES WITH WELDING

[Fermilab, ~ 2007]



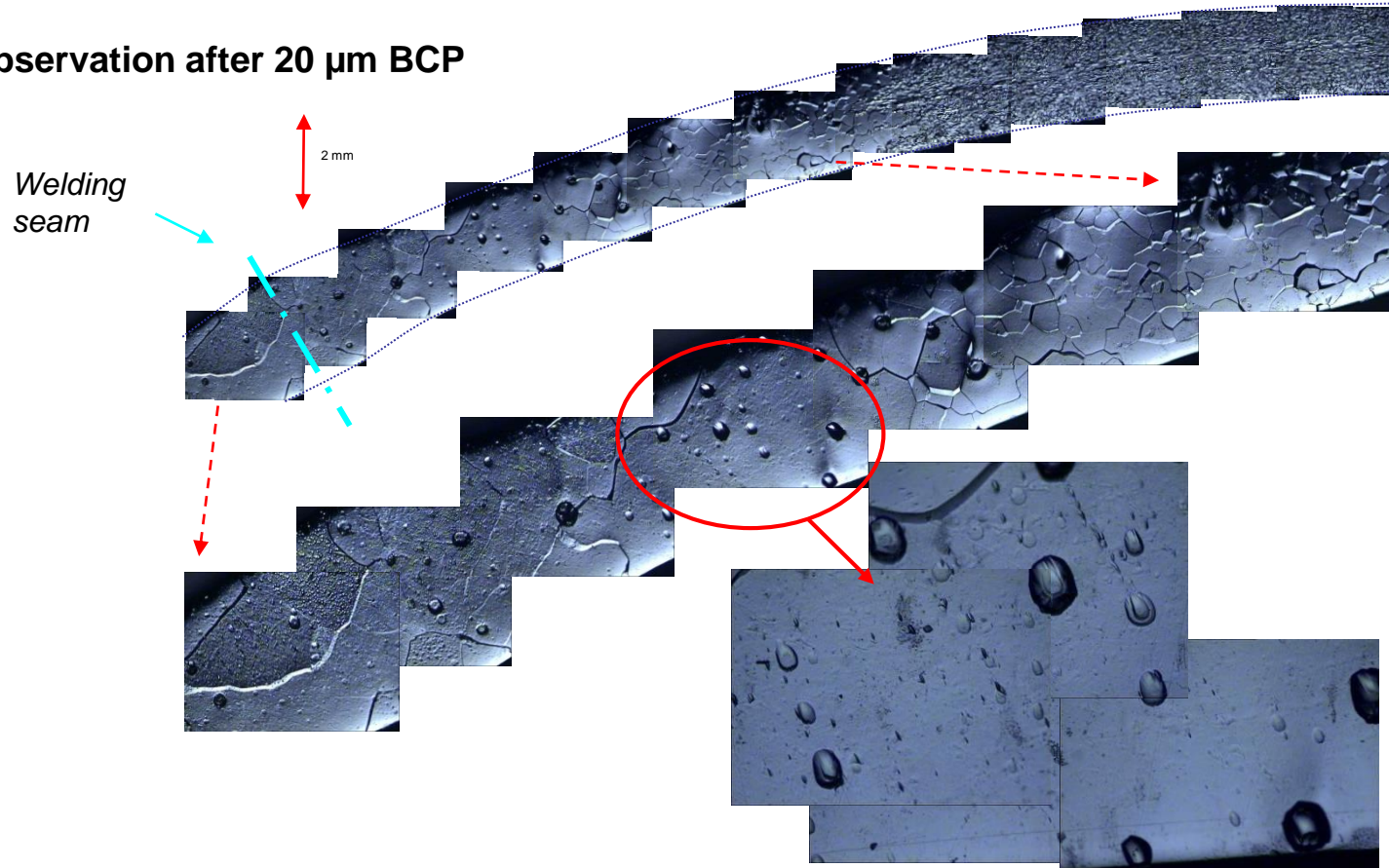
- 3.9 GHz HOM coupler after cold test
- welding + brittle transition (~15 K) ?



■ Careful exploration of the cold properties of welded is absolutely necessary

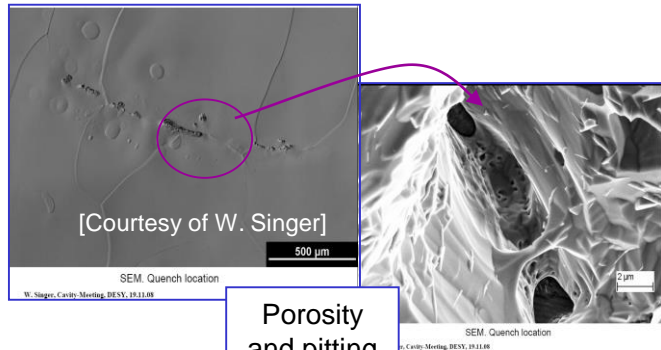


## Optical observation after 20 $\mu\text{m}$ BCP



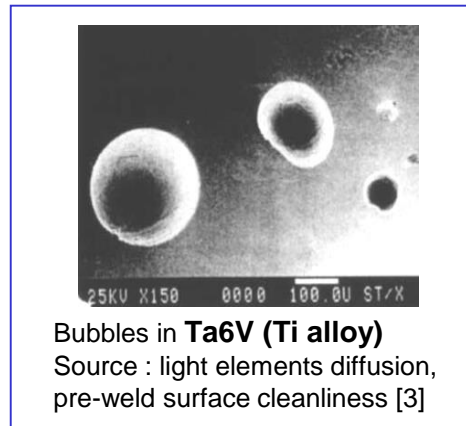
- Deep etching pits (aligned with crystallographic direction ?) are found in the heat affected area.
- Careful exploration of the remaining stress due to welding is also necessary (i.e. with orientation imaging)

# PITTING AND VOIDS IN HAZ: A GENERAL FEATURE OF BE WELDING

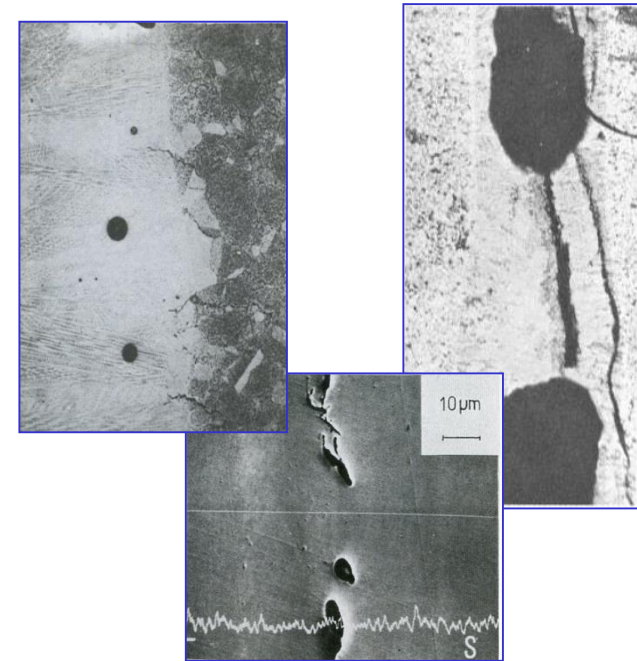


[Courtesy of W. Singer]

Porosity and pitting in Nb



Bubbles in **Ta6V (Ti alloy)**  
Source : light elements diffusion, pre-weld surface cleanliness [3]

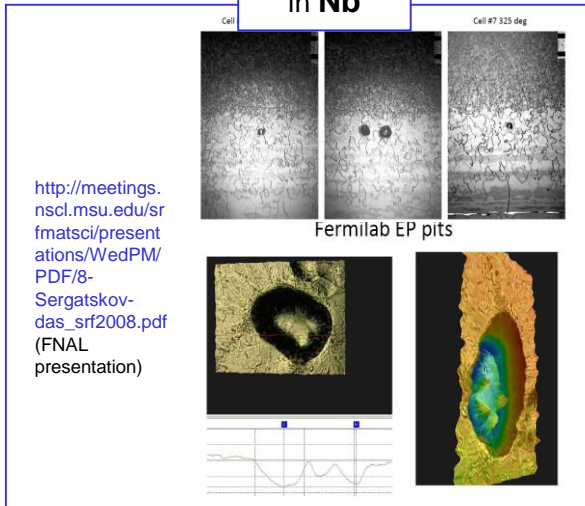


Porosity and/or cracks in **Steel** [5]

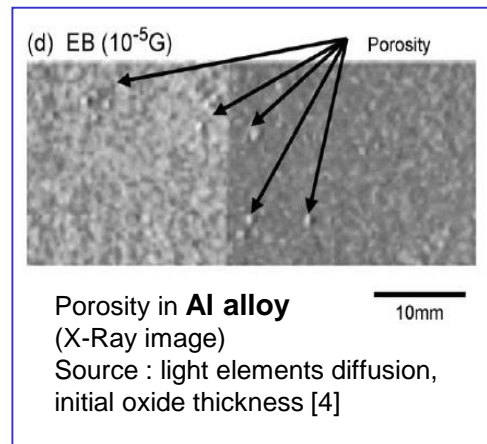
Possible sources :

- welding speed
- pre- and post heating
- position of the focus point
- light elements diffusion

Welding speed and pre- and post heating influence on void development also observed in **Ti and Ta** [6]



Fermitlab EP pits



Porosity in **Al alloy**  
(X-Ray image)  
Source : light elements diffusion, initial oxide thickness [4]

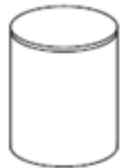
## ■ 2 main sources of pits

- Voids due to light elements during cool down of the melt
- Residual thermal strain => pit/stress corrosion during etching

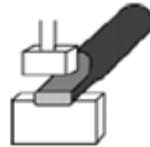
[Antoine, SRF 2009]

# LARGE GRAIN NIOBIUM

# cea TYPICAL SHEET PREPARATION



Mother material



Forging



Cutting

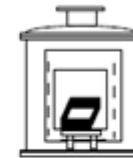
15-20 steps



Pressing



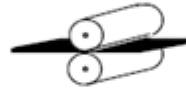
Milling



Annealing



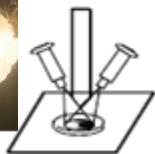
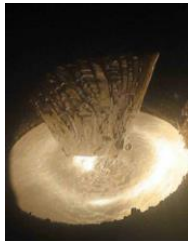
1st EB melting



Rolling



Leveling



2nd, 3rd etc. EB melting



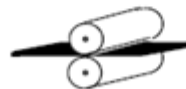
Annealing



Polishing



Separate from base plate



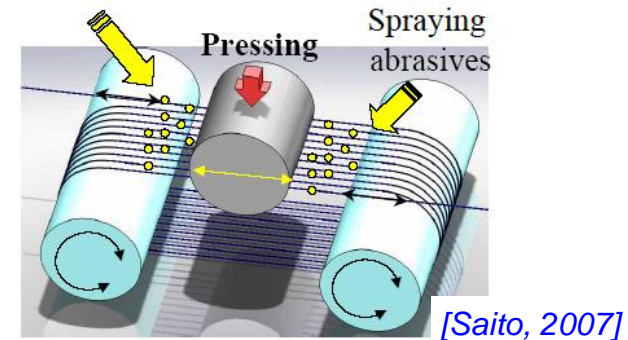
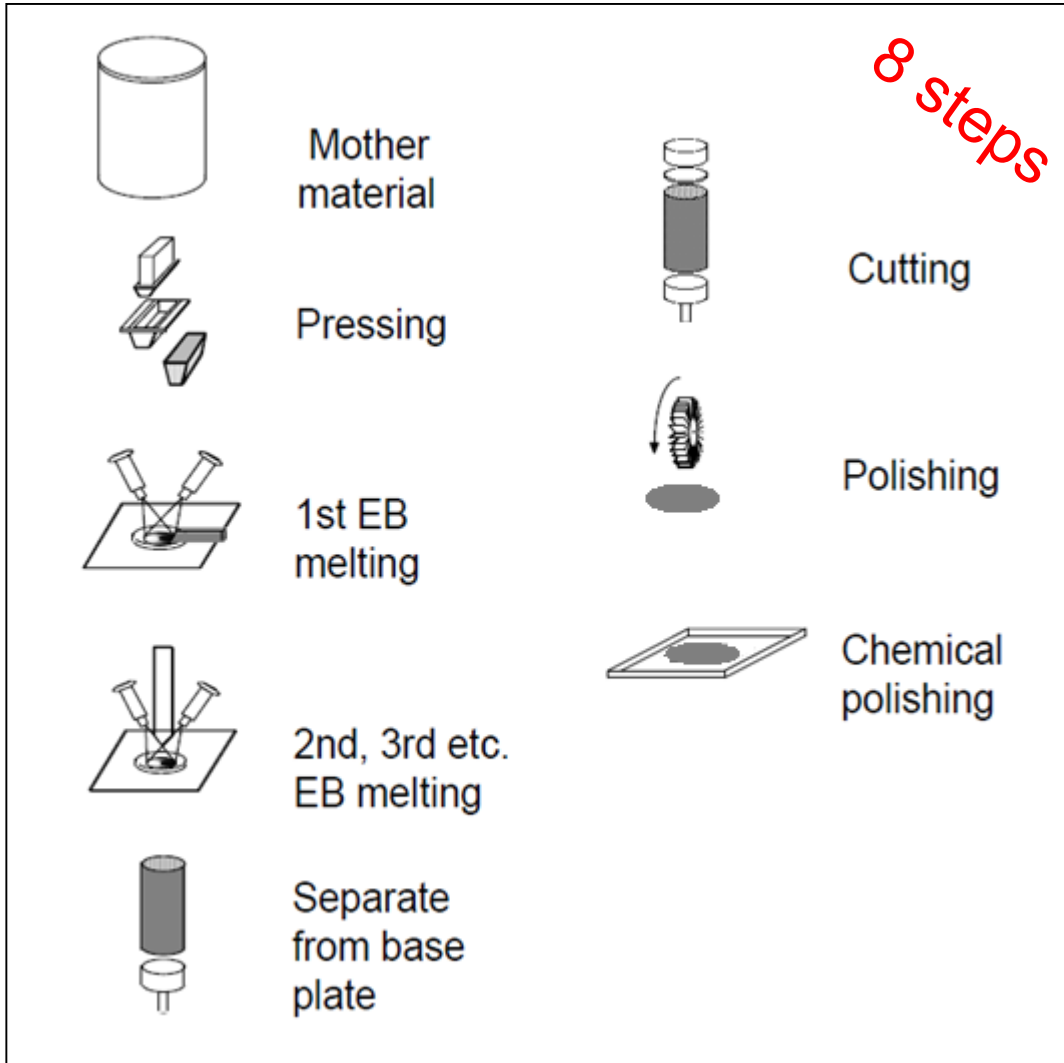
Rolling

(2 or 3 times)



Chemical polishing

# LARGE GRAIN DISK PREPARATION



But....



**Forming of polycrystals w. ≠ orientations**

- => deformation differ for ≠ grains (same phenomena as “orange peel” for smaller grains)
- ∃ textures more favorable

Polishing is needed after forming



# LARGE GRAIN FORMING

## 4. Mechanical Properties: Tensile Test; Bulging Test

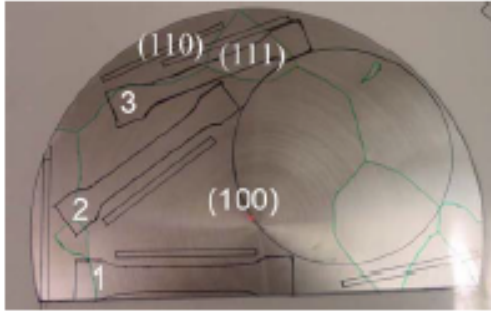


Figure 4. Orientation of used large grain niobium and samples cut for tensile and bulging test

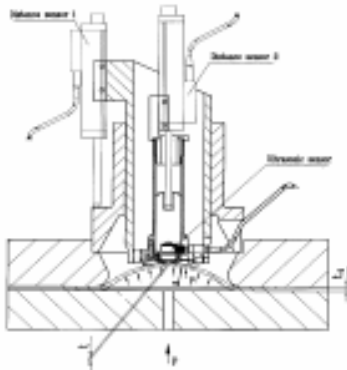


Figure 6. Scheme of the bulging device

[Singer, 2008]

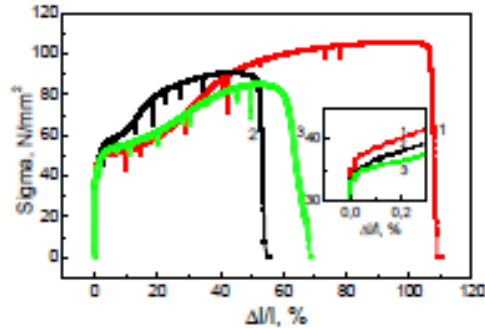


Figure 5. Elongation tests results for 3 single crystal samples with different orientations.

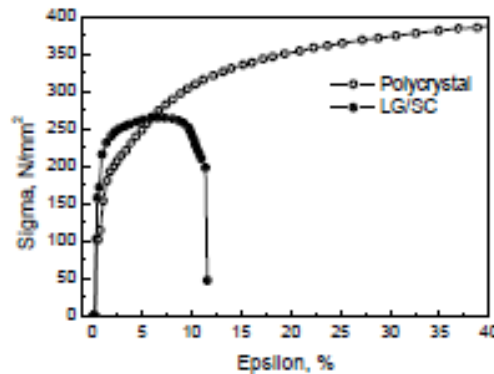


Figure 7. Biaxial bulging test results on large grain Nb sample. Curve for polycrystalline sample shown as reference



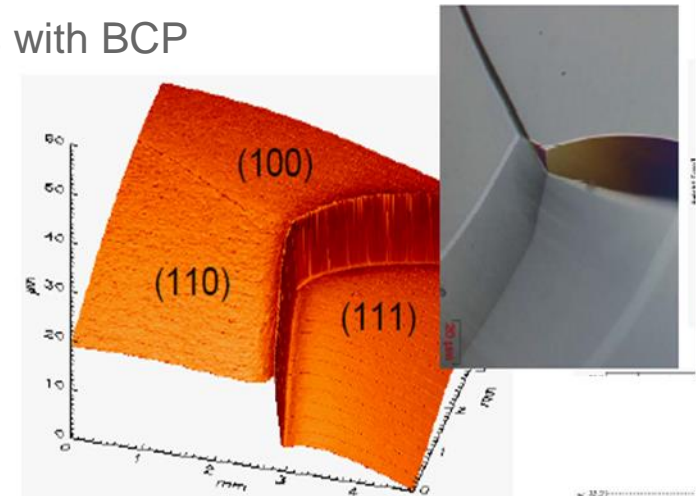
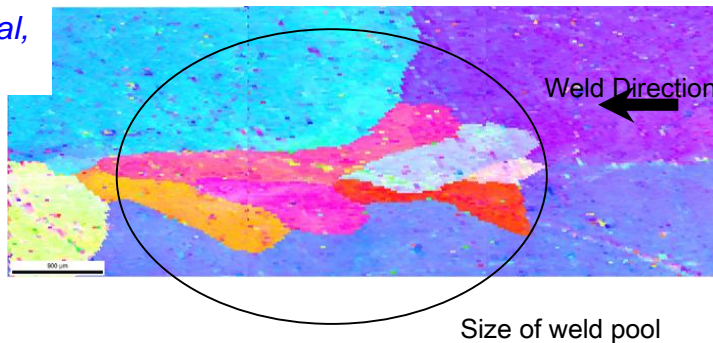
- Non uniform forming
- asymmetric deformation
- risk of tearing (irises)
- risk of holes during welding
- larges steps @ GB

**=> a lot of forming failure  
=> a whole new industrial  
process need to be developed**

## ■ Recrystallization @ welding

- Grain surface very flat:  $R_q \sim$  some 10 of nm (depends on orientation and/or pitting)
  - Steps at GB : some 10 to 100  $\mu\text{m}$  !!!, very sharp
  - Recrystallization into smaller grains @ welds
- => Severe field enhancement => high quench risks with BCP

[H. Jiang et al,  
MSU]



## ■ Note: seamless cavities (monocrystalline, hydroformed, spinned...)

- No welding seam => no recrystallization => no sharp edges @ equator
- Reach consistently 38-45 MV/m with BCP or EP indifferently

(See [http://www.helmholtz-berlin.de/media/media/spezial/events/srf2009/Tutorials/w\\_singer\\_material\\_fabrication\\_and\\_qna.pdf](http://www.helmholtz-berlin.de/media/media/spezial/events/srf2009/Tutorials/w_singer_material_fabrication_and_qna.pdf))

- RF performances : ~ same as smaller grain cavities
  - Medium Q ~ a little better for EP cavities
- Savings for (very) large Nb sheet production (small elliptical cavities only)
  - Less fabrication steps
  - Ingot => disks : no losses of material in the corners
  - High purity material with intrinsically good crystalline quality

But not fitted for  $\varnothing > 30$  cm (typical ingot  $\varnothing$ )
- Increased costs and delay for Cavity forming
  - More fabrication steps, higher failure risks
  - No special basic R&D needed, only development by industry => no benefit for the lab
  - Industrial production is not yet mastered in Europe => long delays
  - Transfer to (European !) industry could be interesting in view of long term project (e.g. ILC)

**Not fitted for a short term project like ESS !**



# **SURFACE STATE REMINDERS ON CHEMICAL STATES**

## 1) BCP (Buffered Chemical Polishing):

### Composition

- ~ 2 vol. of  $\text{H}_3\text{PO}_4$  (buffer, very viscous)
- ~1 vol. of  $\text{HNO}_3$  (oxidant, transforms  $\text{Nb}^0$  into  $\text{Nb}^{5+}$ )
- ~ 1 vol. of  $\text{HF}$  (complexant of  $\text{Nb}^{5+}$ , dissolves the oxide layer formed by  $\text{HNO}_3$ )

*Variation of composition allows to adjust the etching rate*

### Pros

- Easy to handle, middle stirring is necessary
- Fast etching rate
- Very reproducible

### Cons

- It is not “polishing”, it is “etching” : all crystalline defects are preferentially attacked (etching pits, etching figures)
- Grains with various orientation are not etched at the same rate => roughness !
- Except a few cases,  $E_{acc}^{max} \sim 25\text{-}30 \text{ MV/m}$

### Caution :

- Don't process at T higher than  $25^\circ \text{C}$ 
  - Risk of runaway
  - Hydrogen loading is higher

## 2) EP (Electropolishing):

[http://ilc-dms.fnal.gov/Members/tajima/References/Antoine\\_EP\\_tutorial\\_01JUN2006.ppt/file\\_view](http://ilc-dms.fnal.gov/Members/tajima/References/Antoine_EP_tutorial_01JUN2006.ppt/file_view)

### Composition

- ~ 9 vol. of  $H_2SO_4$  (buffer, very viscous)
- ~ 1 vol. of HF (complexant of  $Nb^{5+}$ , dissolves the oxide layer formed due to the high potential applied to  $Nb^0$ )

Variation of composition allows to adjust the etching rate

### Pros (when idealistic conditions, i.e. viscous layer present)

- It is really “polishing” => soft surface, no sensitive to crystallographic defects.
- Should not be sensitive to the cathode-anode distance
- Gives (not always!) the best ever  $E_{acc}^{max} \sim 40-45$  MV/m (TESLA shape)

### Cons

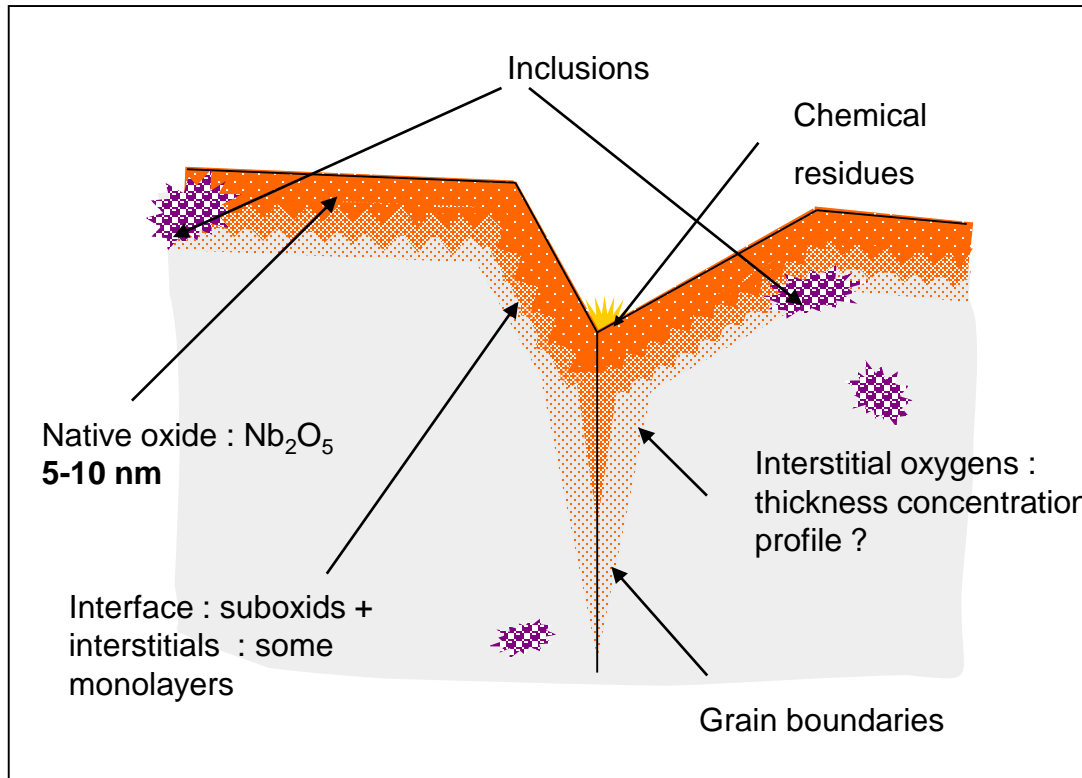
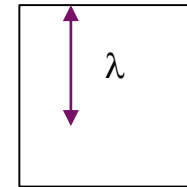
- Idealistic conditions are not possible to reach in most of our processing conditions
- Very sensitive to stirring condition, temperature, aging of the mixture
- Not very reproducible
- Safety issues (acid mixture sensitive to water,  $H_2$  evolution...)

### Caution :

- If  $T \uparrow$  : etching rate  $\uparrow$  but pitting risk  $\uparrow$ , H loading  $\uparrow$ , HF evolution  $\uparrow$
- If  $V \uparrow$  : etching rate  $\uparrow$  but pitting risk  $\uparrow$ , S generation  $\uparrow$ , sensitivity to Cathode/Anode distance  $\uparrow$

# SURFACE POLISHING?

- Real Niobium is far from ideal “textbook” superconductor



## ■ Surface

- scratches, contamination, dust particles
- roughness

## ■ Oxide

- thickness~ 5nm, depends on orientation and previous process
- mainly  $Nb_2O_{5-x}$ , with ppb impurities content ( $PO_x$ ,  $SO_x...$ )
- one layer NbO @ interface
- decompose into suboxides upon baking (UHV)

## ■ First 10 nm of Nb

- Distorted (lattice mismatch)
- A lot of interstitial atoms. Mainly H, O (At% to 10s of At%), also F, C, P... i.e. surface segregation & chem residue
- Higher imp content for EP (O, C) !

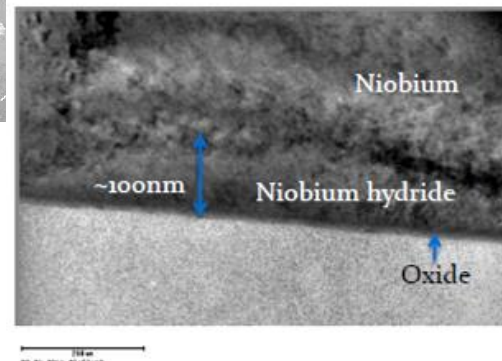
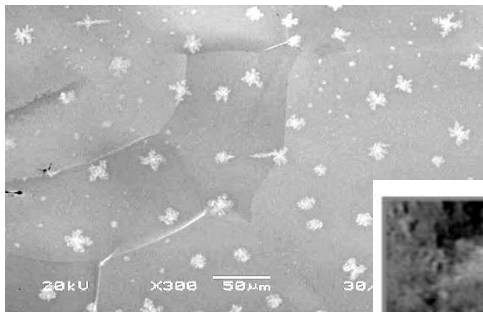
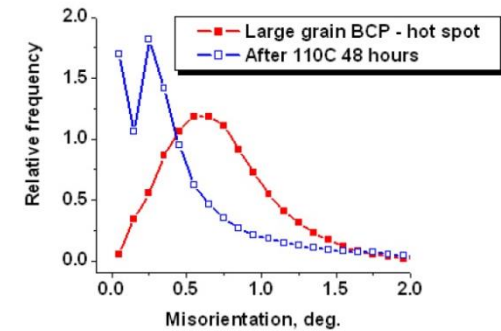
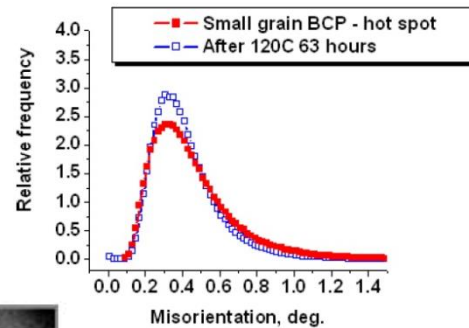
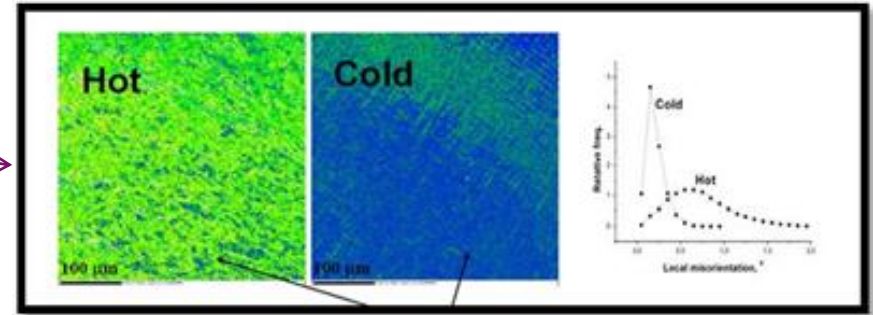
# **SURFACE STATE DAMAGE**

## Hot spots in cavities are correlated with :

- Early vortex penetration (see [Grasselino] and [Ciovati])
- High misorientation (i.e. high density of dislocation)
- High density of hydrides precipitates (Cottrell clouds)
- Recover partially upon baking, except for small grains cavities

## Hydrides

- SC, low  $H_{C1}$
- Symptom or reason for early vortex penetration ?

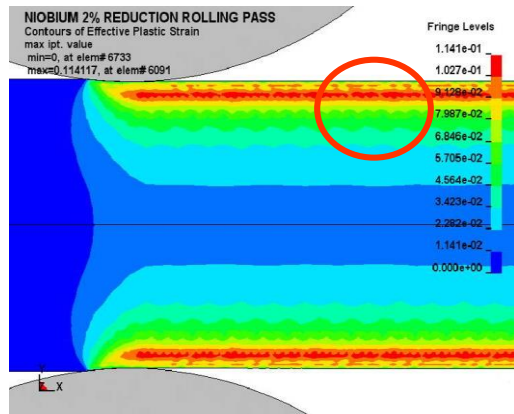
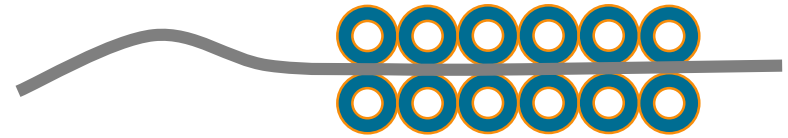


[A. Romanenko, Cornell , FNAL]

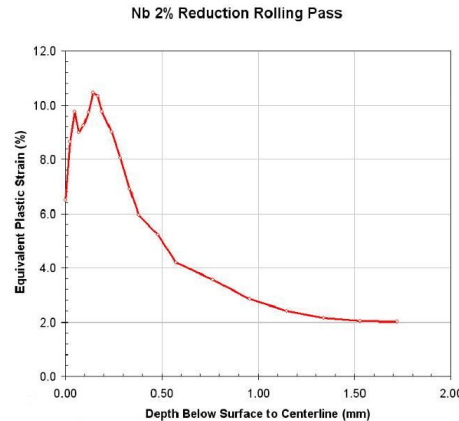
# WHY SURFACE POLISHING?

## => DAMAGE LAYER

After rolling sheets undergo a skin pass for planarity



[R. Crook et al, Black Laboratory]



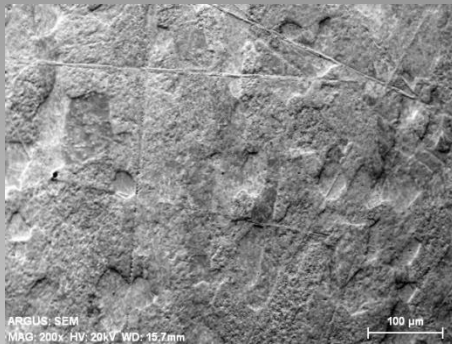
Finite element simulation of 2% reduction of 3.5 mm sheet with 1 cm diameter rolls (Courtesy Non-Linear Engineering, L.L.C.). Strain is concentrated in the near-surface region (~300 μm). Localized strain exceeds the average by a factor of 5

- Damage layer = deformed grains + high density of dislocations + (foreign atoms)
- Rolling leaves a damage layer ~2-300 μm with a texture resistant to recrystallization, i.e. same order of magnitude than the necessary etching of material.
- Further damage (dislocations !) brought by deep drawing and thermal strain during welding
- Interesting trails :
  - look at remaining stress after forming/welding,
  - chemical mechanical polishing

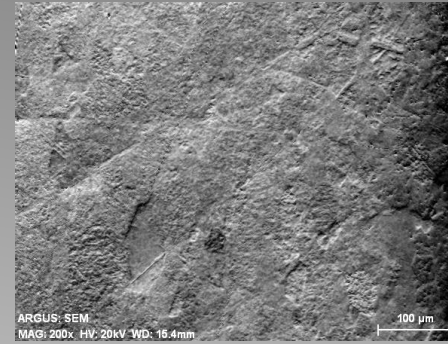


# DAMAGE LAYER

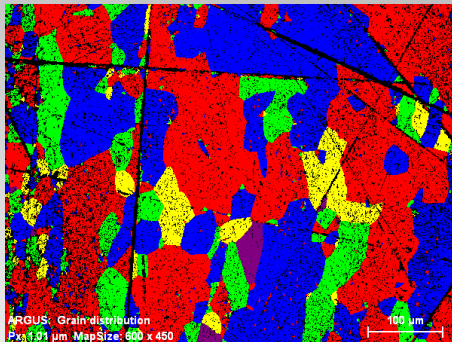
## Analysis of a cut of Nb Sheet (MC polishing)



Bulk **SEM**

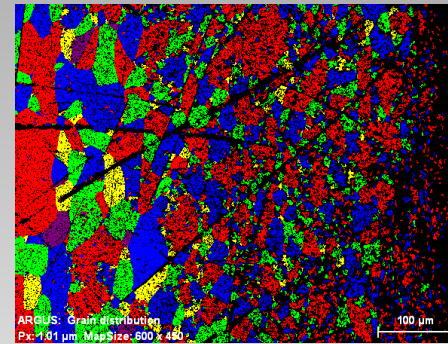


Surface



## EBSD

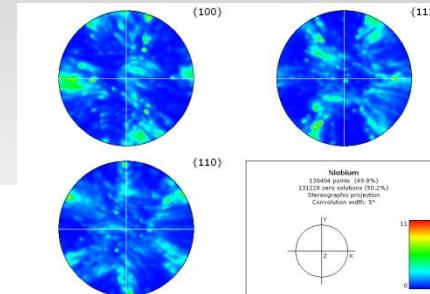
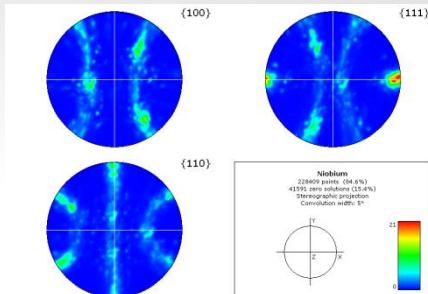
Bulk : Grains are large, slightly elongated,  
NB small dark spot uniformly distributed  
source = MC polishing damage ( $\ll 100$  nm)



Surface: Grains are small, distorted,  
A lot of large, dark spot close to the surface :  
grain are so distorted => cannot refract e- any more

## Pole figures

Bulk : clear and clean cubic pole figure :  
Sheet is textured as expected for rolled material

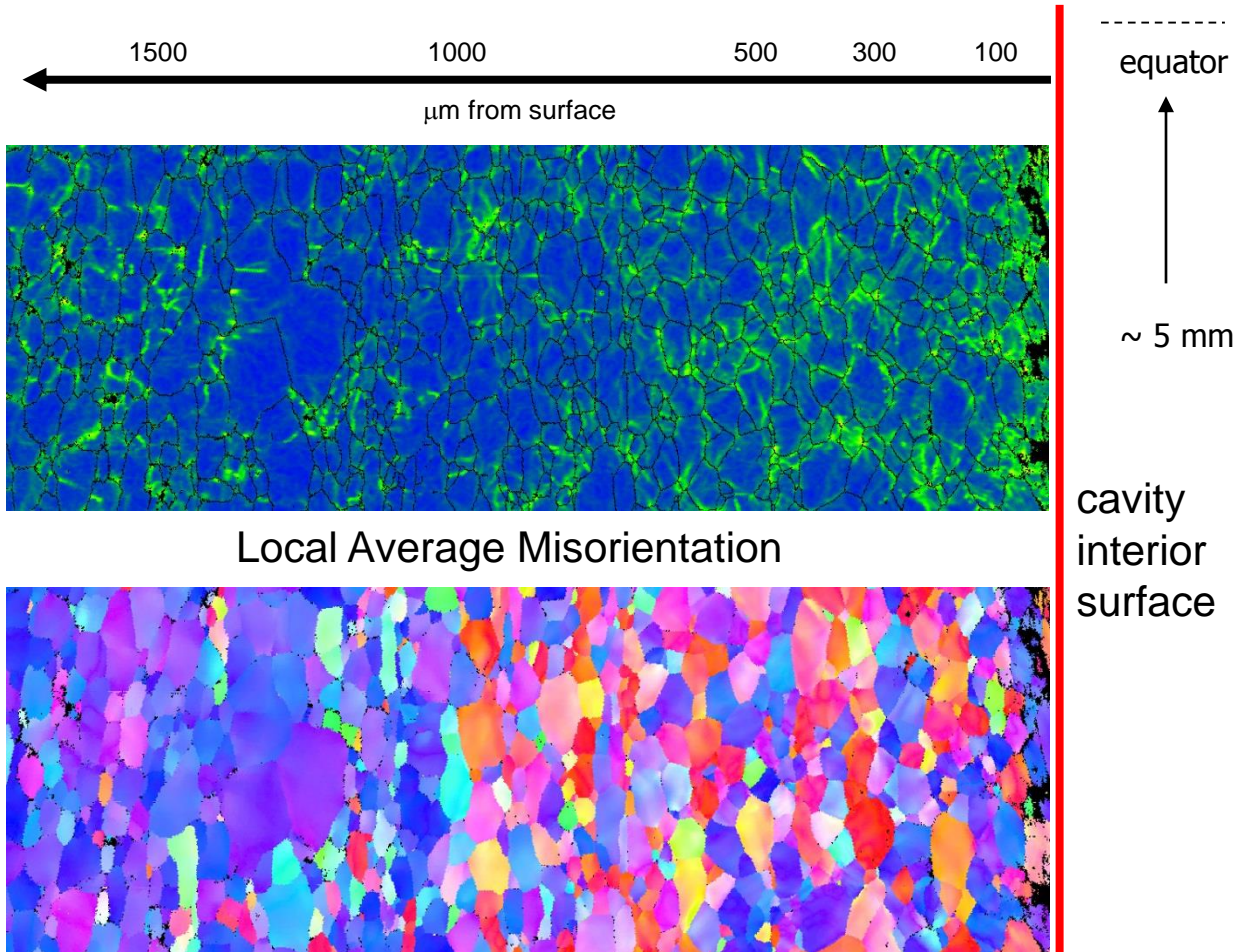


Surface: blurred pole figure :  
many distortions,  
different orientations



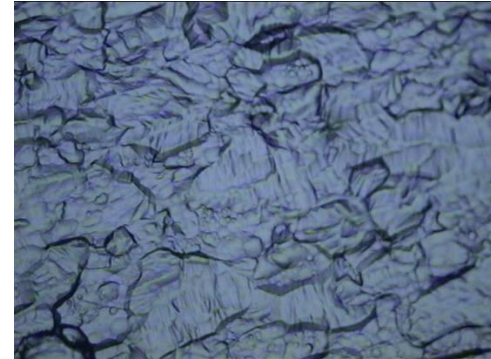
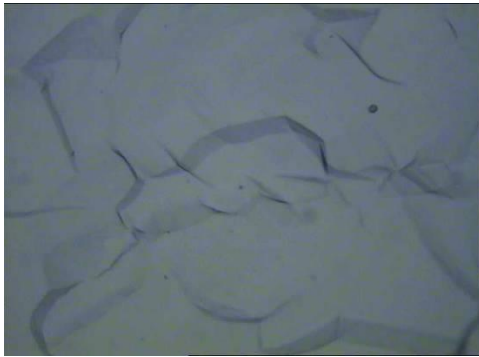
# DEEP DRAWING : ORIENTATION IMAGING

Half cell cutout  
(just after deep drawing,  
before welding)



[R. Crook et al, Black Laboratory]

## ■ Mechanical-chemical polishing (metallographic technique)



*Same Nb sheet, after 20µm BCP; left after MC polishing, right as received*

## ■ Electrochemical etching (BCP/EP) needed to remove damage layer

- Very long process
  - Not well adapted for the coarse, thick etching (EP: aging of the bath, BCP: roughness)
  - Still necessary to produce a surface without mechanical damage
- => Try to reduce it to a minimum time

# CENTRIFUGAL BARREL POLISHING

Developed @ FNAL

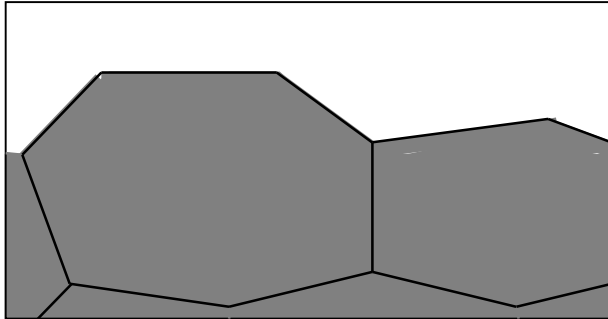
Cooper - SRF 2011



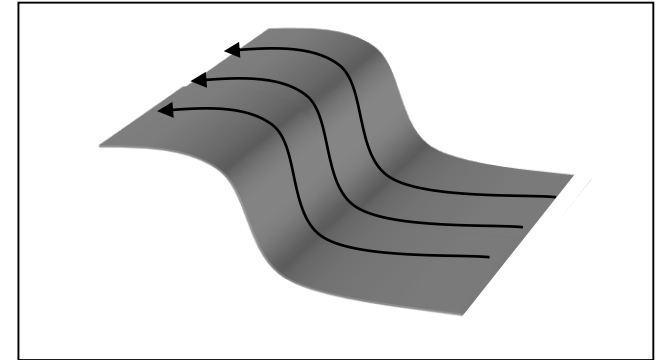
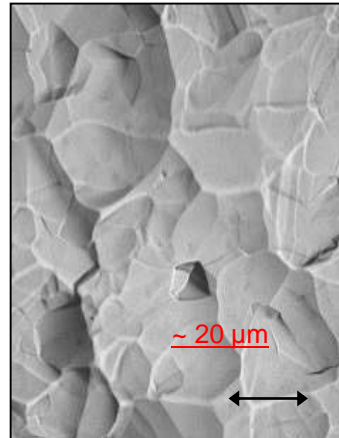
# **SURFACE STATE ROUGHNESS**



# CHEMICAL ETCHING VS ELECTROPOLISHING : SURFACE MORPHOLOGY EFFECT ON QUENCH



Grains do not etch @ the same rate  
=> relief

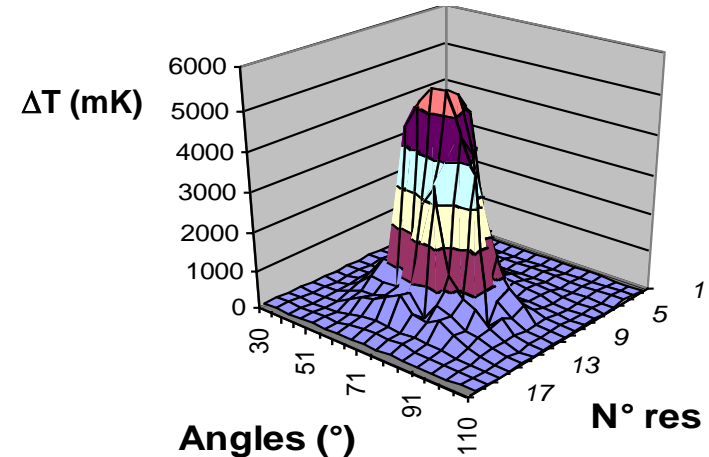
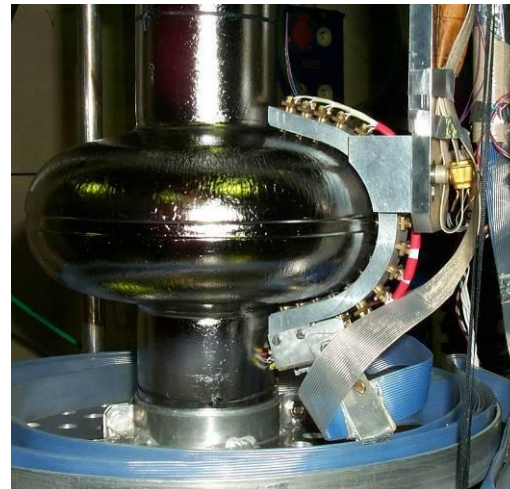


Curvature of magnetic field lines =>  
local  $\uparrow$  of  $H_{\perp}$

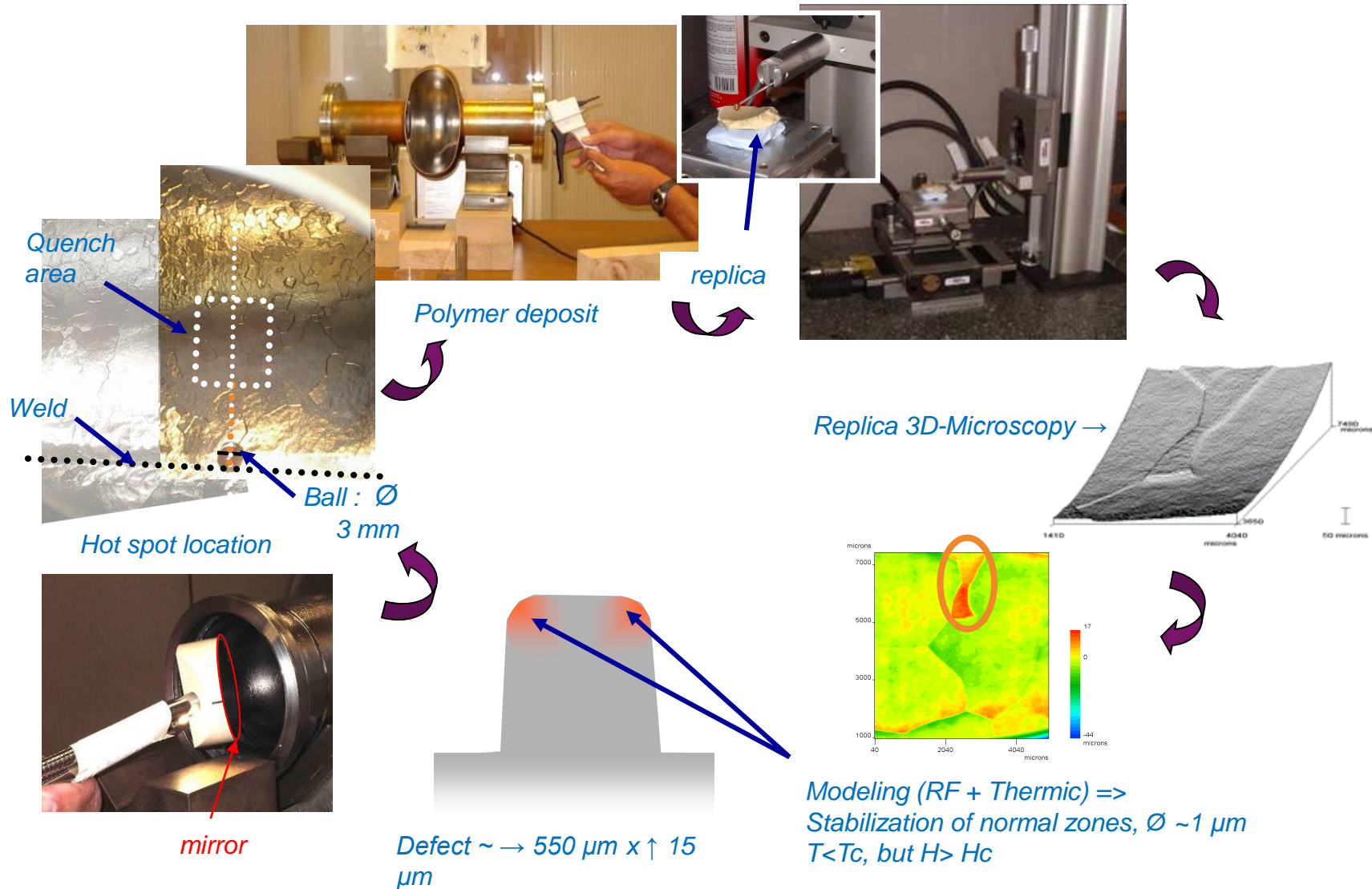
Local  $\uparrow$  of the magnetic field => local normal state transition => Quench ?

Quench location:

superfluid He  
Temperature maps

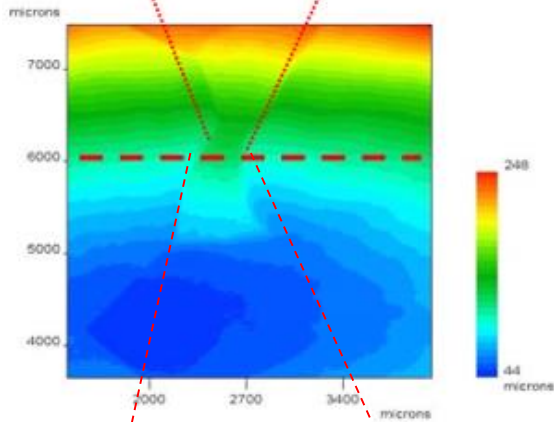
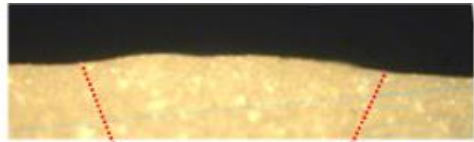


# MORPHOLOGY : REPLICA AND FIELD MODELING

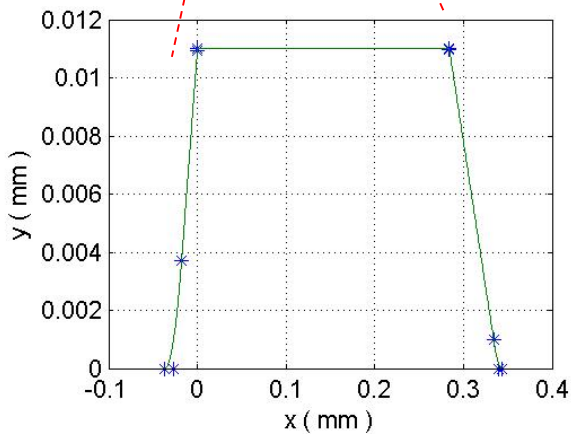
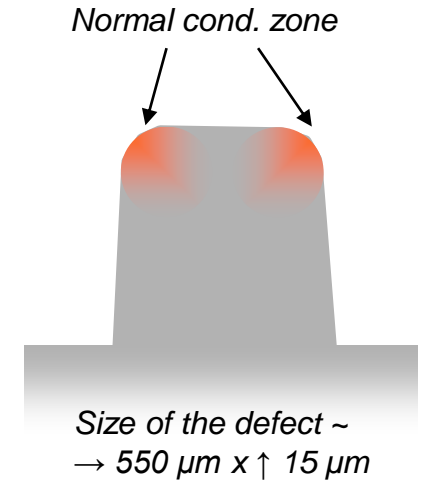
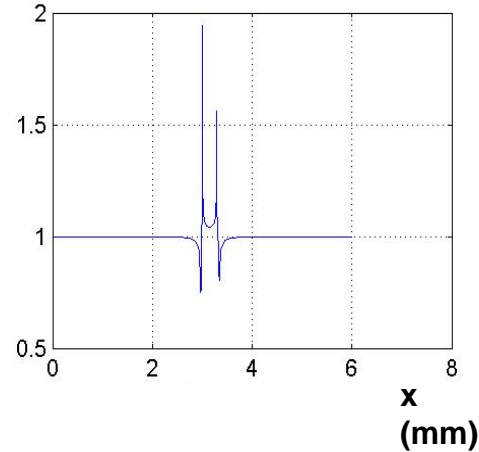




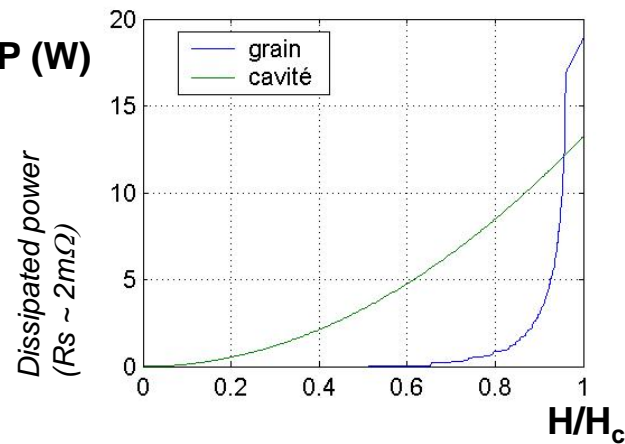
# REPLICA @ THE QUENCH SITE



$H/\langle H \rangle$



$P$  (W)



- Thermal behavior :
- Edges thermally stabilized until  $T \sim 5.35$  K and  $W \sim 142$  mW
  - $T < 9.2$  K but  $H > H_c$
  - When  $W \sim 143$  mW => **Quench !**

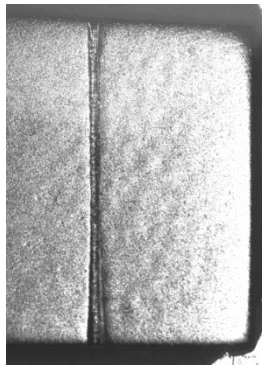
# MORPHOLOGY EFFECT: FIELD ENHANCEMENT

[A. Polyanskii et al, FNAL/FSU]

Single crystal with notch on the surface :

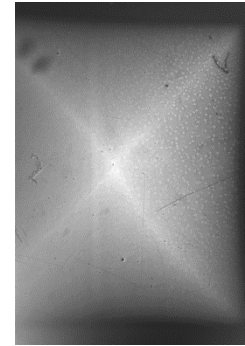
$H // \text{ surface}$

0.5 mm  $\leftrightarrow$

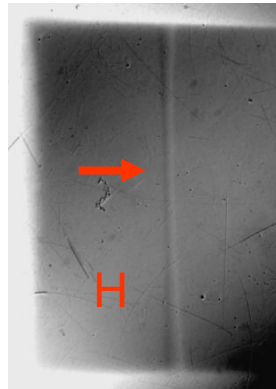


H

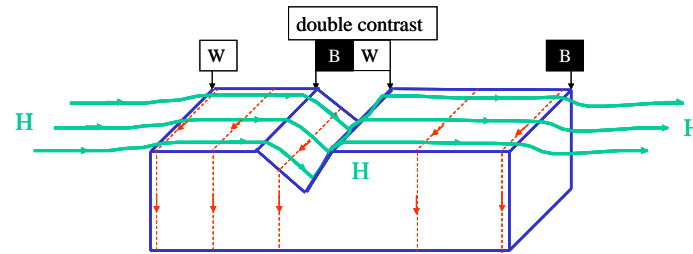
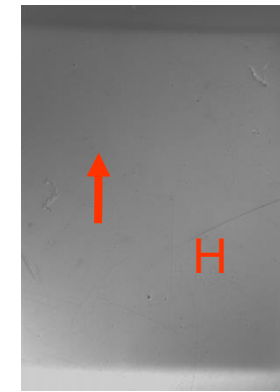
H=80 mT T=7K



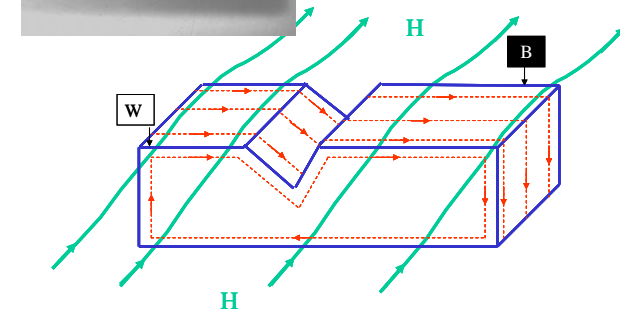
$H \perp \text{ surface}$ : notch has small impact on flux distribution even at higher T



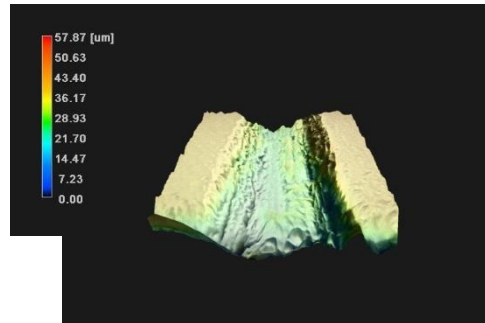
T=5.6K



MO contrast is double at the groove, when in-plane field perpendicular to groove



No MO contrast at the groove, when in-plane field parallel to groove



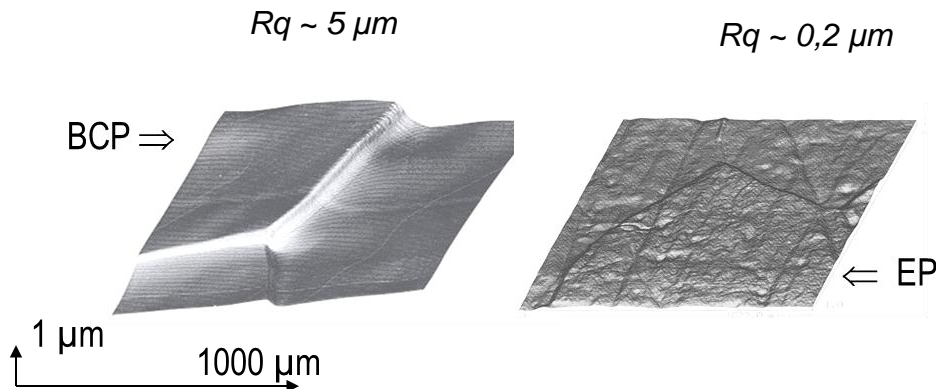
# MORPHOLOGY : EVALUATION OF ROUGHNESS

- Roughness parameter is not sufficient to evaluate field enhancement behavior



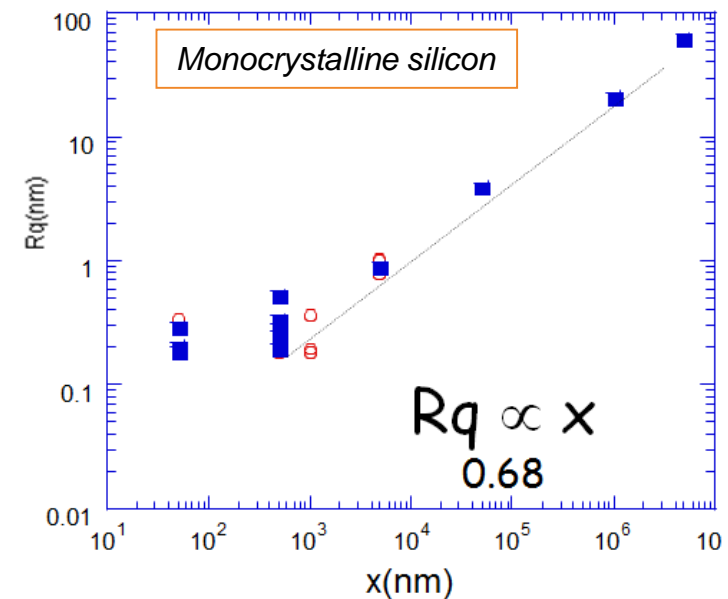
Same height distribution,  $\neq$  RF behavior

- Roughness measurement depends on observation scale



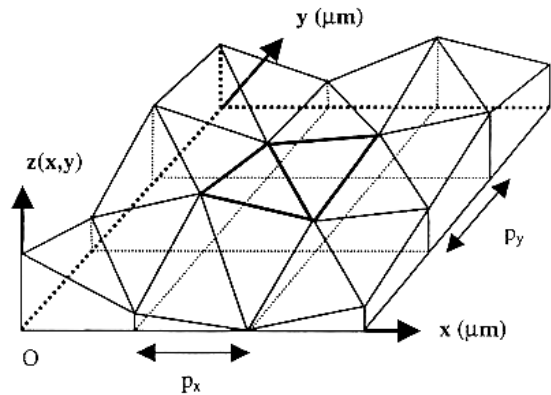
*Topological approaches can give a better evaluation of the surface*

[Amrit 2004]



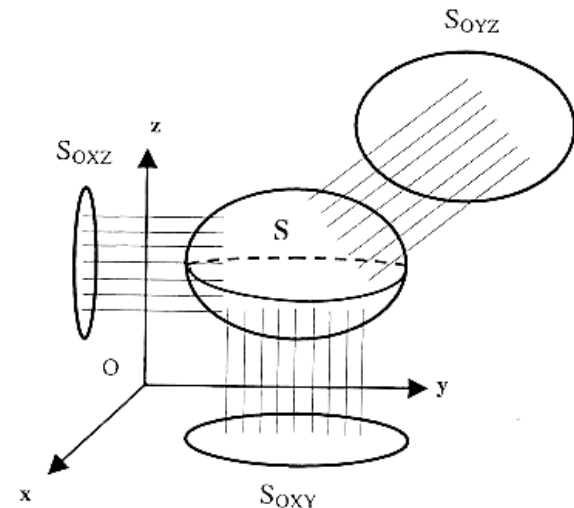
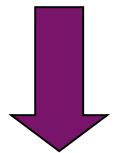
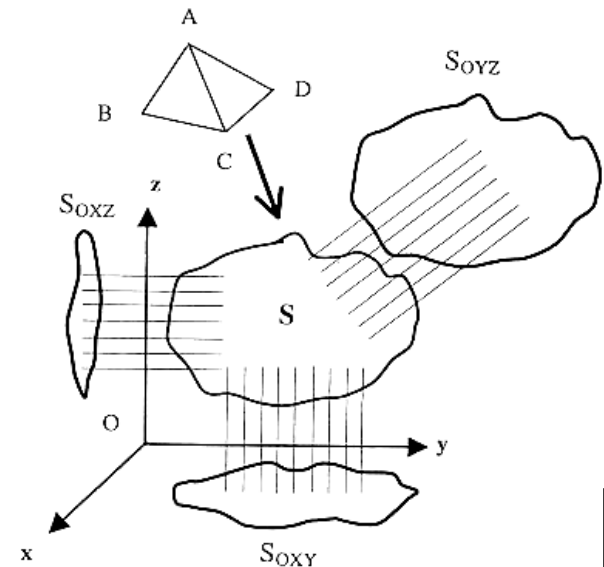
*Fractal approach:  
=> Scale independent roughness !!!*

# A TOPOLOGICAL USEFUL TOOL : CONFORMATIONAL EQUIVALENT ELLIPSOIDS

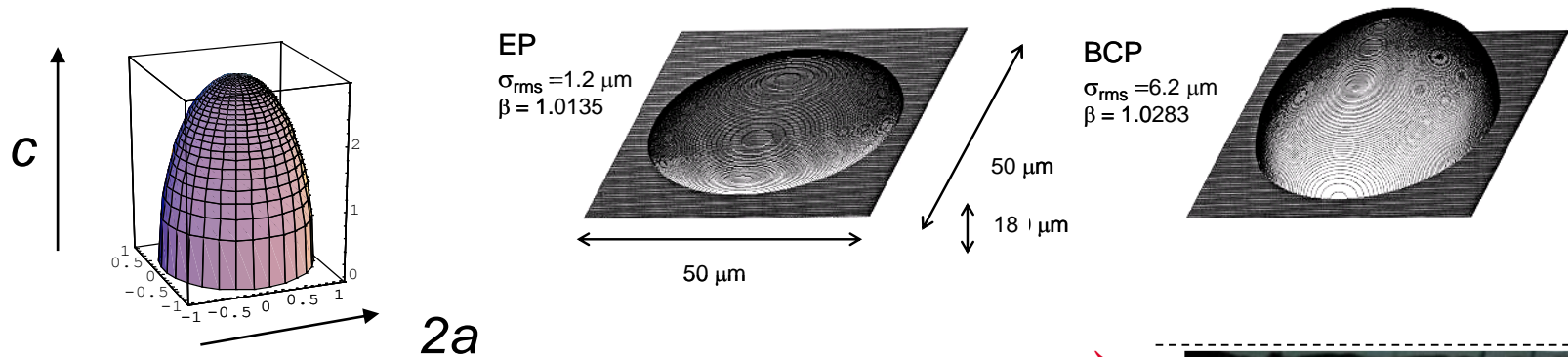


1. Decomposition of a sampled surface into elementary segments (mode) or elementary micro-triangles (3D mode).

- Can model 1! step or give a medium value / 1 surface.
  - Surface characterization
  - Access to 3D model
- Ellipsoid demagnetization factor easy to calculate

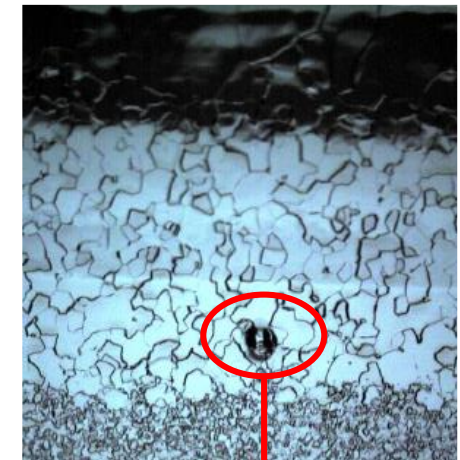


# CONFORMAL EQUIVALENT ELLIPSOIDS AND DEMAGNETIZATION FACTOR



Weld →

Parameter	Small grain material	Annealed, away from the weld	Thermally affected zone @ weld	Mean value for EP material	Defect, close to weld* C ~ 50 $\mu\text{m}$ 2A ~ 200 $\mu\text{m}$
	BCP : chemical etching			Electropolishing	
$\Phi$ grains	70 $\mu\text{m}$	1-2 mm	0,5-1 cm	1 mm => 1 cm	-
Ra	1-2 $\mu\text{m}$	4-8 $\mu\text{m}$	40-80 $\mu\text{m}$	~ 1 $\mu\text{m}$	-
C	~ 300	~ 90-100	~ 350	~ 70	50
$\beta=1/(D)$	1,065	1,028	1,4	1,018	1,9 !!! *



Chemical etching  
=> premature quench @ weld

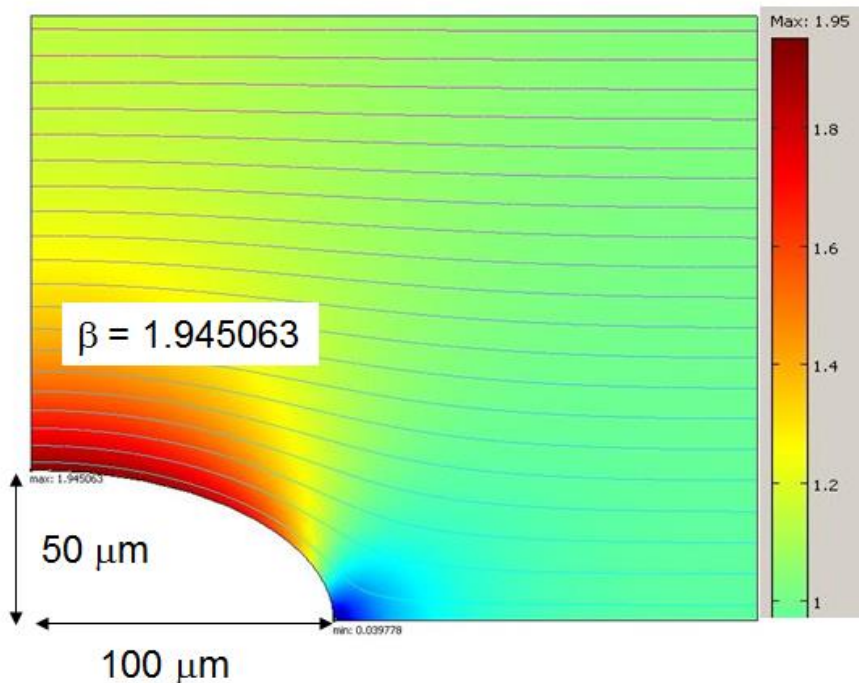
FNAL cavity  
quench @ ~15  
MV/m



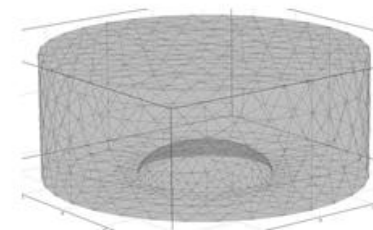
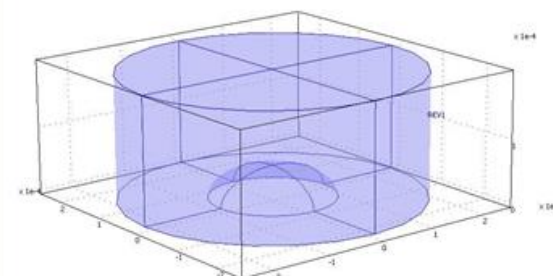
[INSEPOV, NOREM, ANL

# Field enhancement of Ellipsoid via a FE simulation

Surface: Electric field, norm [V/m] Contour: Electric potential [V] Subdomain marker: Electric field, norm [V/m]



3d-ellipsoid

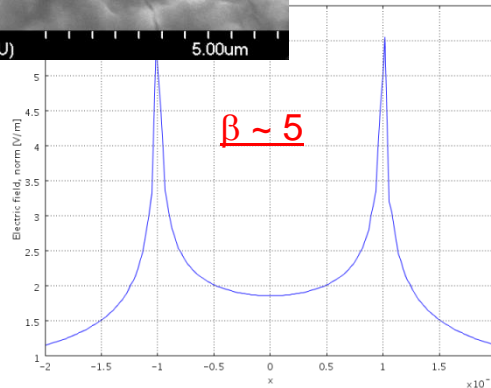
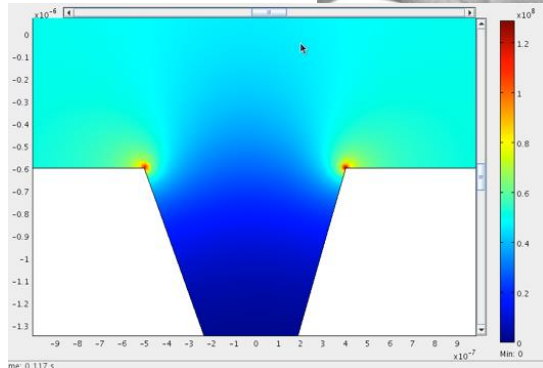
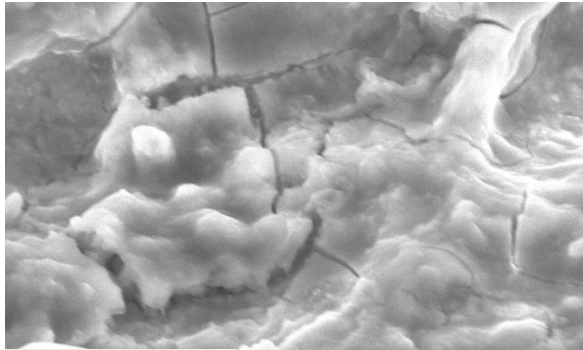


3d-mesh

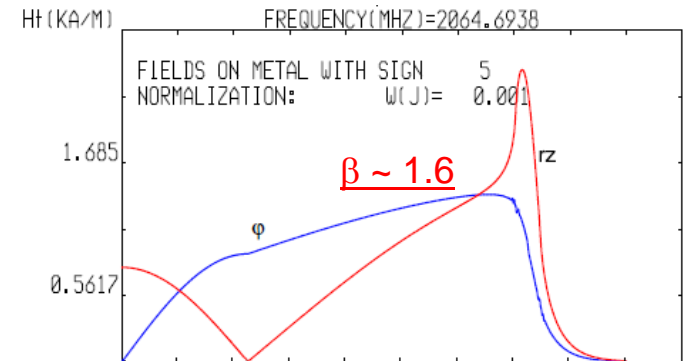
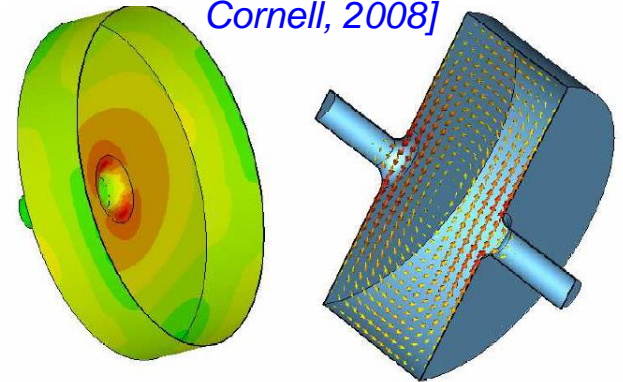


# OTHER 2D-3D MODELING OF HOLES AND PITS

[Norem, ANL 2011]



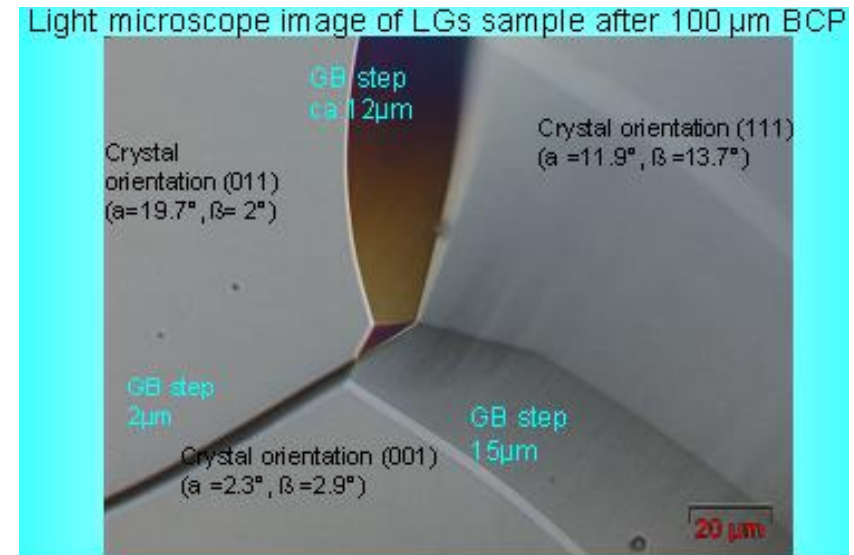
[Shemelin, Cornell, 2008]



- $\beta$  does not depend on the depth
- $\beta$  depends a lot on curvature radius
- $\beta \sim 1-10$  (rather less than 2 for small defect) => magnetic field sensitive, no field emission !

[http://flash.desy.de/sites2009/site\\_vuvfel/content/e403/e1644/e2271/e2272/infoboxContent2358/TTC-Report2008-07.pdf](http://flash.desy.de/sites2009/site_vuvfel/content/e403/e1644/e2271/e2272/infoboxContent2358/TTC-Report2008-07.pdf)

- Topography induces local magnetic field enhancement;
  - Edge curvature: more important than height
  - Pits ~ bumps
- This effect is important on macroscopic defects
  - Welding defects
  - Thermally affected zone
  - Large grains cavities
- Modeling, RF + thermal => quench
- Prevention:
  - => Electropolishing, CBP ...
  - Or ... Avoid welding !
    - Hydroforming
    - Monocrystalline cavities (no large grain !)



[W. Singer, DESY]

## Recommendations

- Do not ask for too stringent specification
- Check the delivered materials meet ALL of them
- Follow closely what is done during ALL fabrication steps
- Sensitive steps :
  - Cleanliness of industrial workshops
  - Welding : pre-cleaning, vacuum, cooldown
  - Surface preparation: enough etching required
- R&D needed
  - Properties, especially cold properties of welded parts
  - Quench location for large cavities (e.g. 2<sup>nd</sup> sound)
  - ....

Acts like soft copper or lead

Machining : tendency to gall, to seize => special attention to tool angles and lubrication.

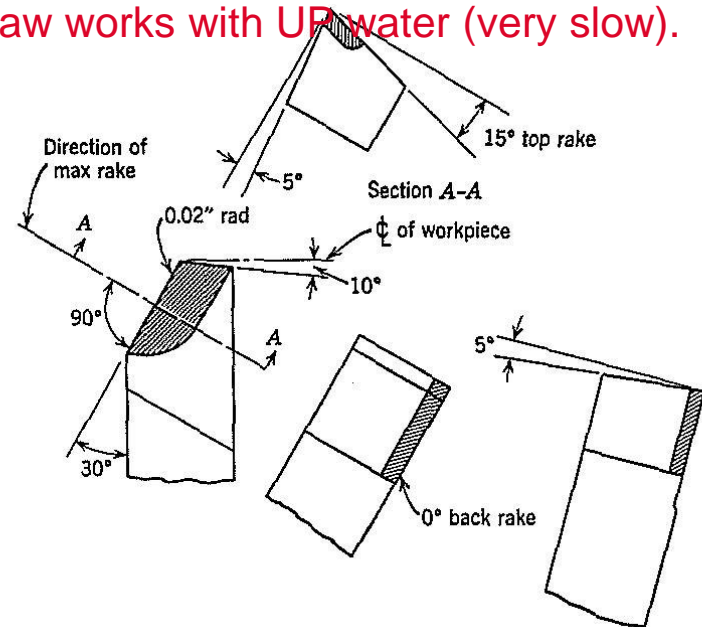
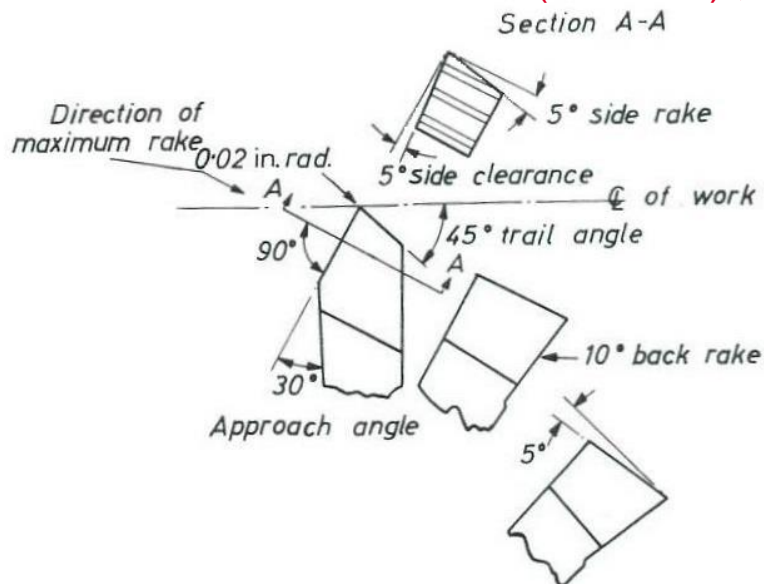
High speed recommended.

Steel rather than carbide.

Tools must be very sharp

High pressure forming operations: tendency to stick to tooling during operation=> specific lubricant and die material : brass, bronze ; (Be-Cu or steel also)

NB ethanol has been tried (lubricant) ; diamond saw works with UP water (very slow).



# THANK YOU FOR YOUR ATTENTION

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Commissariat à l'énergie atomique et aux énergies alternatives  
Centre de Saclay | 91191 Gif-sur-Yvette Cedex  
T. +33 (0)1 69 08 73 28 | F. +33 (0)1 69 08 64 42

Etablissement public à caractère industriel et commercial | RCS Paris B 775 685 019

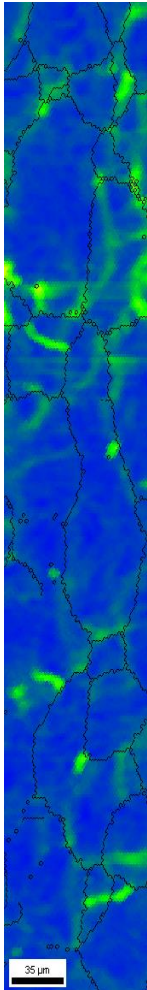
DSM  
Irfu  
SACM  
LIDC2

**SPARES**

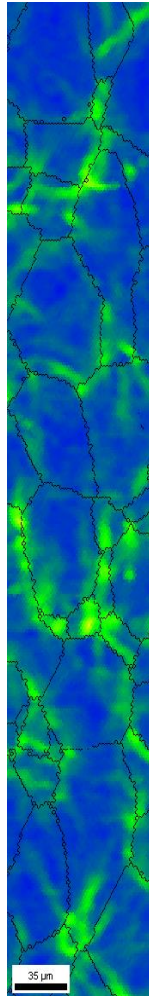


# STRAIN AT CAVITY SURFACE

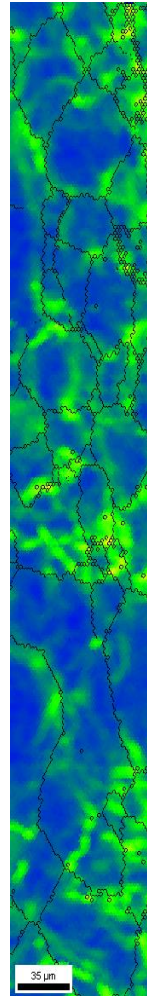
1000  $\mu\text{m}$



500  $\mu\text{m}$

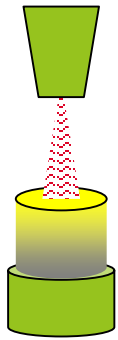


100  $\mu\text{m}$  cavity interior surface

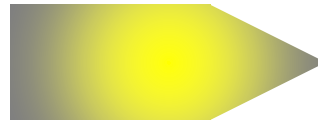


Strain diminishes,  
*especially in the grain interiors*  
as a function of  
distance from  
the cavity surface  
towards the  
sheet mid-plane

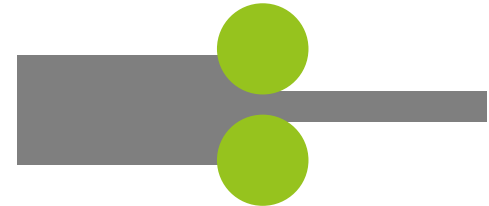
# LARGE GRAIN : PREPARATION STEPS



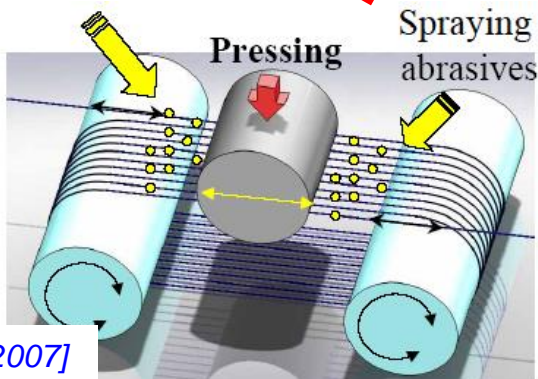
- EB melting
  - > 4 => high RRR
  - slow cooling => large grains



- Hot forging (air)
  - necessary to feed rolling machine
  - ! RRR↓↓↓



- Cold rolling + recovering
  - necessary to get small grains (formability, Y.S.)
  - small grain ↓ "orange peel"



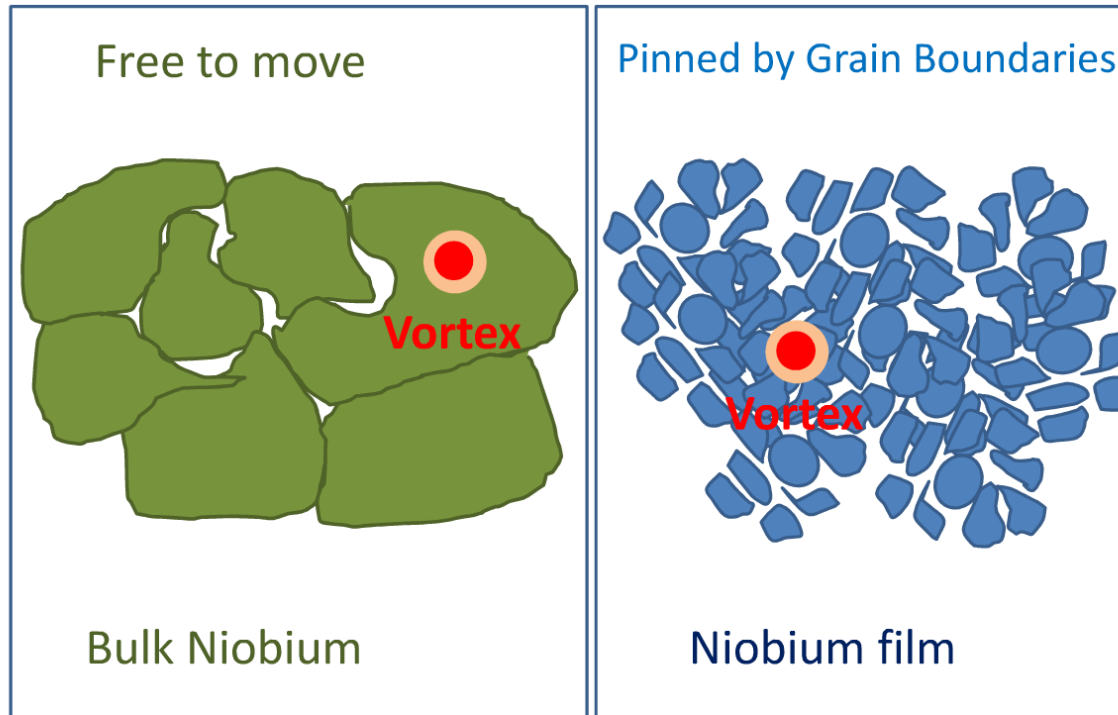
## Forming of polycrystals w. ≠ orientations

- => deformation differ for ≠ grains (same phenomena as "orange peel" for smaller grains)
- ∃ textures more favorable

[Saito, 2007]

# BULK VS THIN FILM

- Bulk Niobium: grains  $\varnothing > \sim$  mm, sensitive to earth magn. Field (trapped flux)
- Niobium deposited onto copper ( $\sim 1-5 \mu\text{m}$  thick) :  $\varnothing < \sim 100$  nm, nearly insensitive to trapped flux



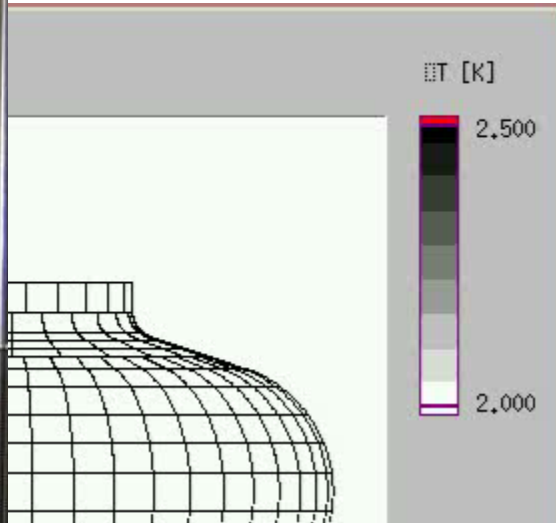
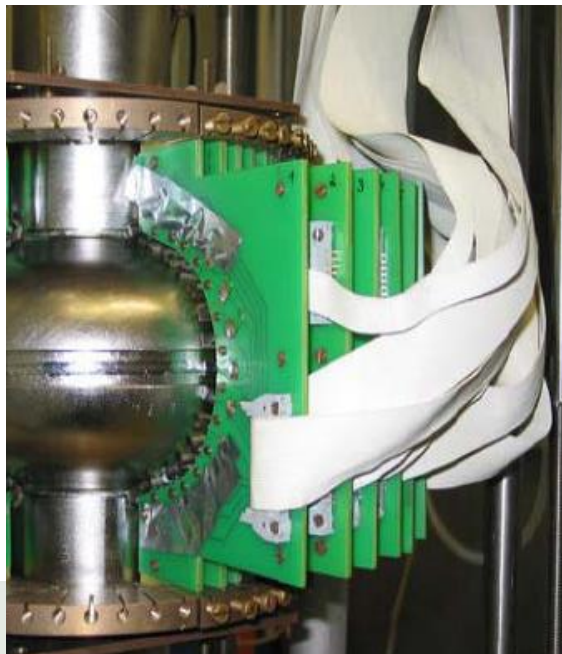
[Palmieri, 2010]

Low depinning frequency  $\omega_0$

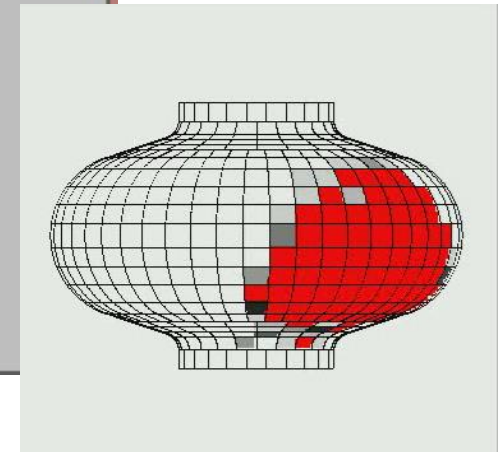
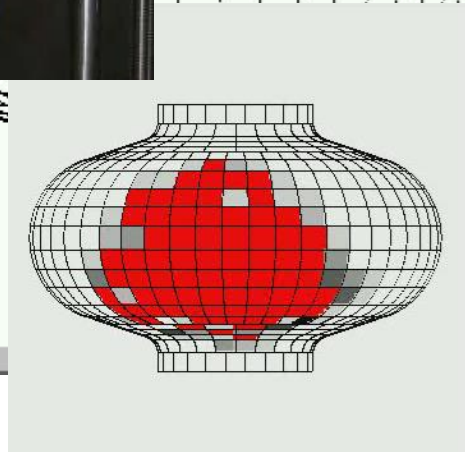
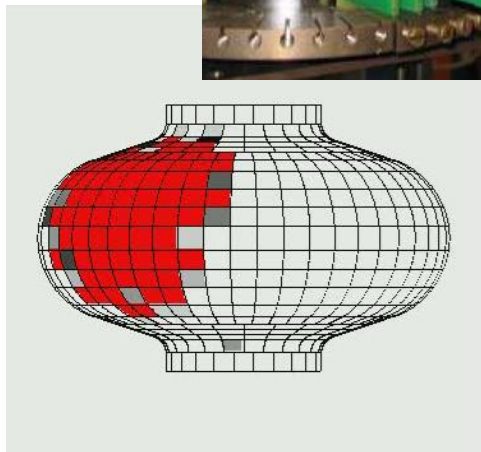
High depinning frequency  $\omega_0$

<http://www.slideshare.net/thinfilmworkshop/palmieri-rf-losses-trapped-flux>

# BULK NB ULTIMATE LIMITS : NOT FAR FROM HERE !



*Cavité 1DE3 :  
EP @ Saclay  
T- map @ DESY  
Film : courtoisie  
A. Gössel +  
D. Reschke  
(DESY,  
Début 2008)*



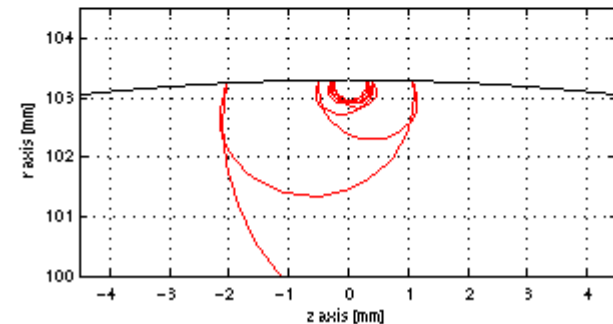
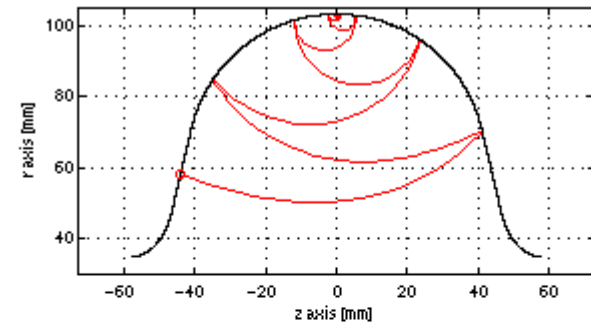
*The hot spot is not localized : the material is ~ equivalent at each location => not limited /local defect, but by material properties ?*

Due to resonant electron emission  
(secondary emission)

It is influenced by adsorbed layers...

But the main ways to overcome it is:

- changing the cavity's geometry...
- RF processing



[http://www.rni.helsinki.fi/research/em/EM\\_multipacting.html](http://www.rni.helsinki.fi/research/em/EM_multipacting.html)