Large Grain/Single Crystal R&D at DESY

Presented by W.Singer

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Outlook

- LG: Fabrication and some results
- SC: Fabrication and some results
- Material investigation

Status of DESY LG/SC R&D program

Material of the company	RRR	No./Type	Fabrication by	Fabrication Procedure	Status, September 2006
		1-cell cavity			
Heraeus/LG	500	1AC3	ACCEL	Deep drawing + EB welding	Tested after EP, tested after BCP
Heraeus/LG	500	1AC4	ACCEL	Deep drawing + EB welding	Tested after EP
Heraeus/LG	500	1AC5	ACCEL	Spinning + EB welding	Tested after EP and BCP
CBMM/SC	200	1AC6	ACCEL	Spinning + EB welding	Tested after BCP and EP
Heraeus/LG	340	1AC7	ACCEL	Deep drawing + EB welding	In EP treatment
Heraeus/SC	300	1AC8	ACCEL	Deep drawing + EB welding	In BCP treatment
Heraeus/LG	300	1DE20	DESY	Deep drawing + EB welding	In fabrication
Heraeus/LG	300	1DE21	DESY	Deep drawing + EB welding	In fabrication
Ningxia/LG	400	1DE22	DESY	Deep drawing + EB welding	In fabrication
CBMM/LG	250	1DE25	DESY	Deep drawing + EB welding	In fabrication
NPC/LG	240	1DE26	DESY	Deep drawing + EB welding	In fabrication
		9- cell cavity			
Heraeus/LG	340	AC114	ACCEL	Deep drawing + EB welding	Tested after BCP
Heraeus/LG	370	AC113	ACCEL	Deep drawing + EB welding	In BCP treatment
Heraeus/LG	500	AC112	ACCEL	Deep drawing + EB welding	In BCP treatment

Large Grain: Several 1-cell and three 9-cell cavities fabricated (ACCEL)

The surface is more shiny after BCP. The steps at grain boundaries are more pronounced as in polycrystalline material.

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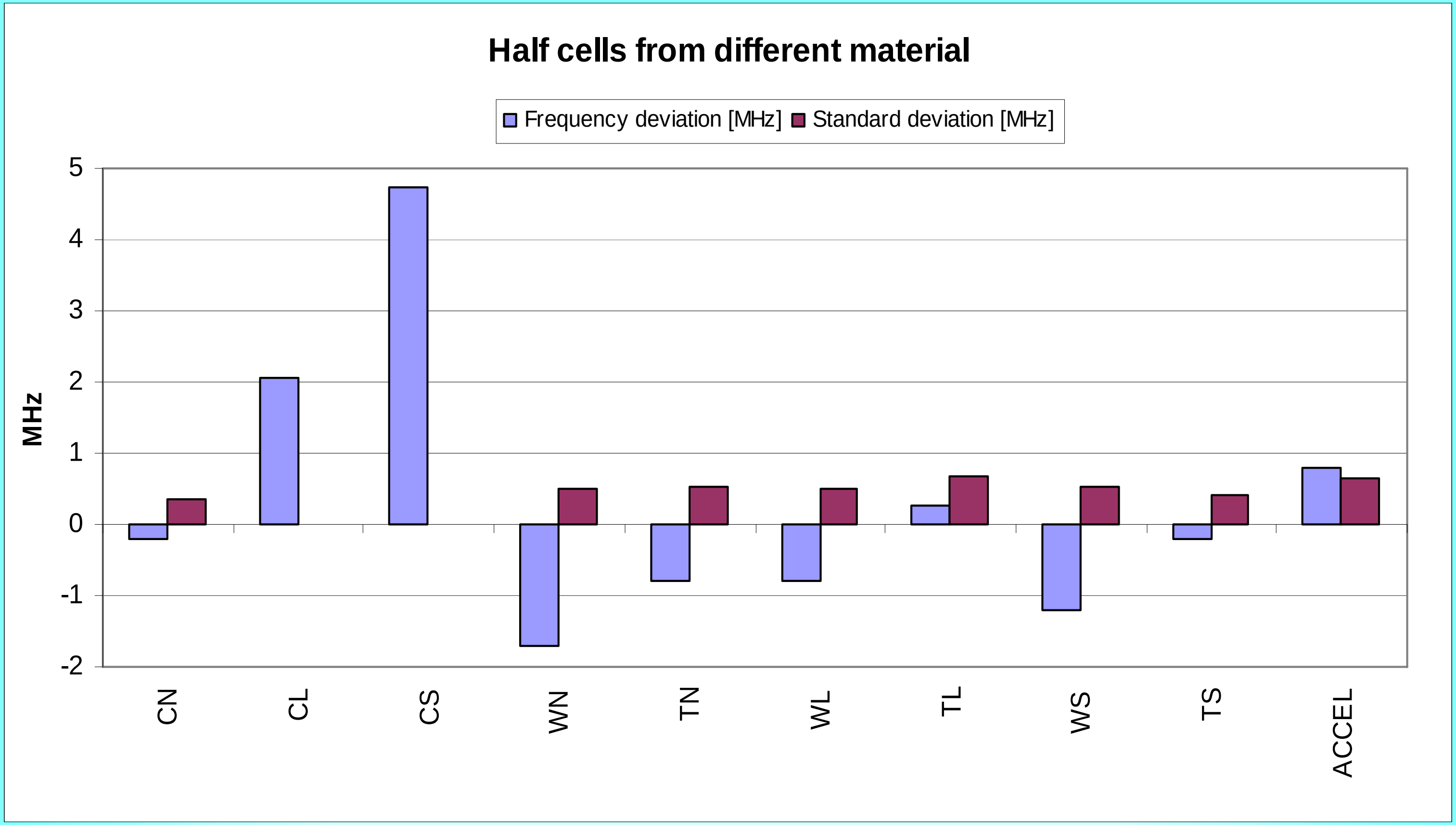
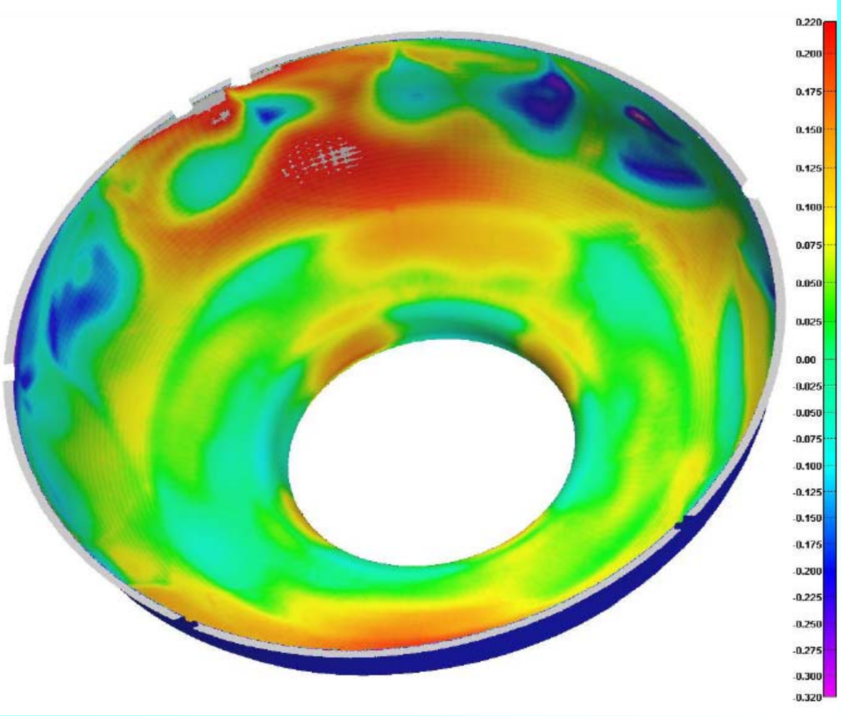
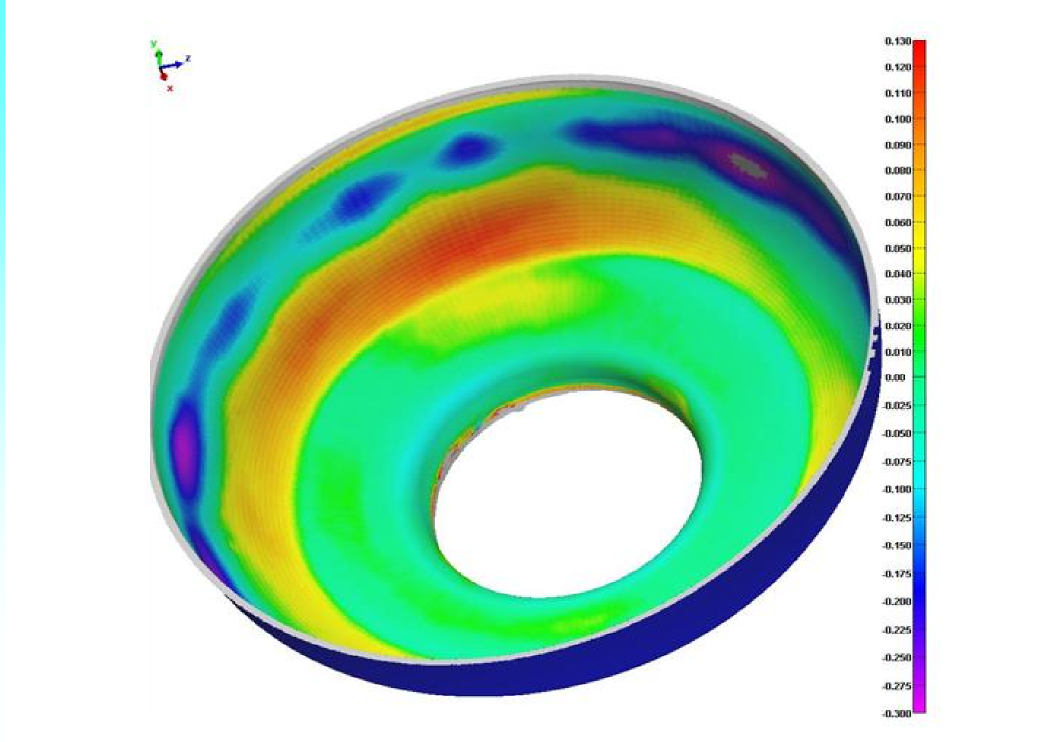
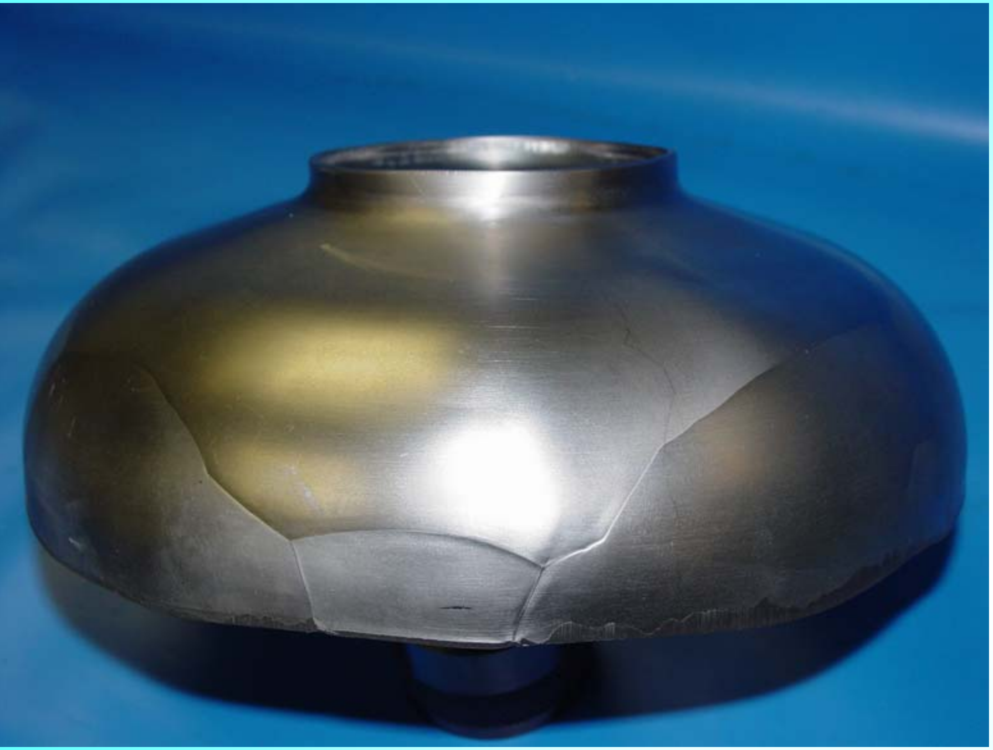


Frequency measurement of 6 end half cells (L and S) and 48 middle half cells (N) for cavities AC112-114. C - large crystal, W - Wah Chang, T - Tokyo Denkai. The shape conformity of half cells from large grain material is lower as of conventional fine grain (could be improved by correction of the tools), the uniformity of the half cells from large grain material is better.



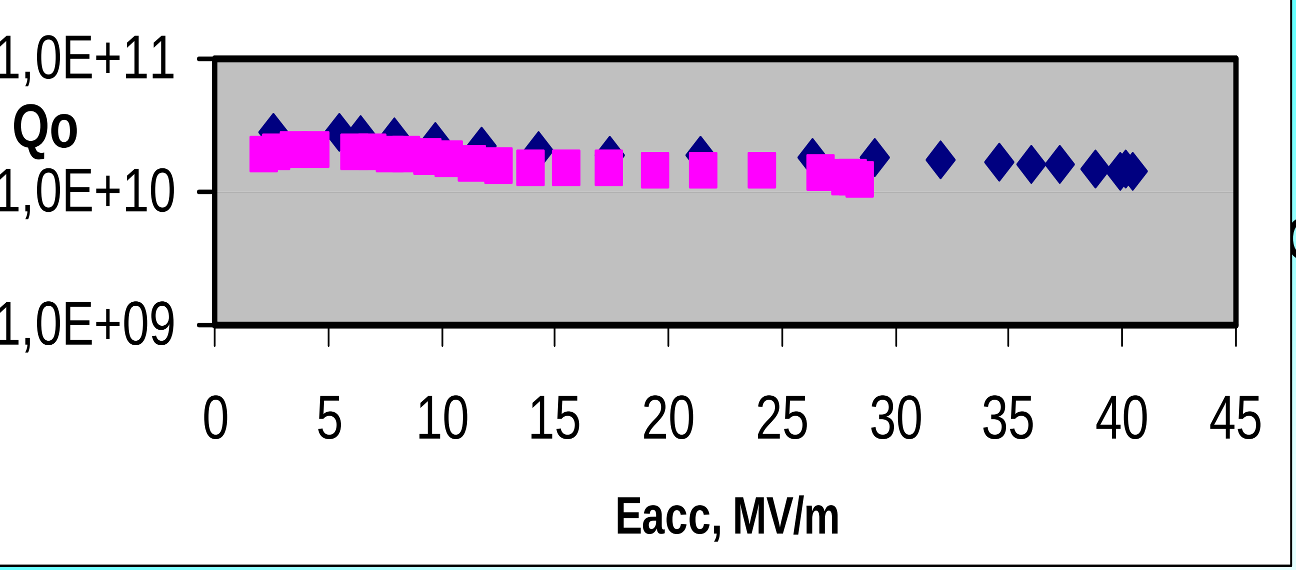


3D Image of the optical measurement of the shape on large grain half cell (left; realized accuracy +0,22 / -0,32 mm) in comparison with a fine grain half cell (right; realized accuracy +0,13 / -0,30 mm). The large grains are fractionally pronounced. The variation of the large grain half cell shape is somewhat larger

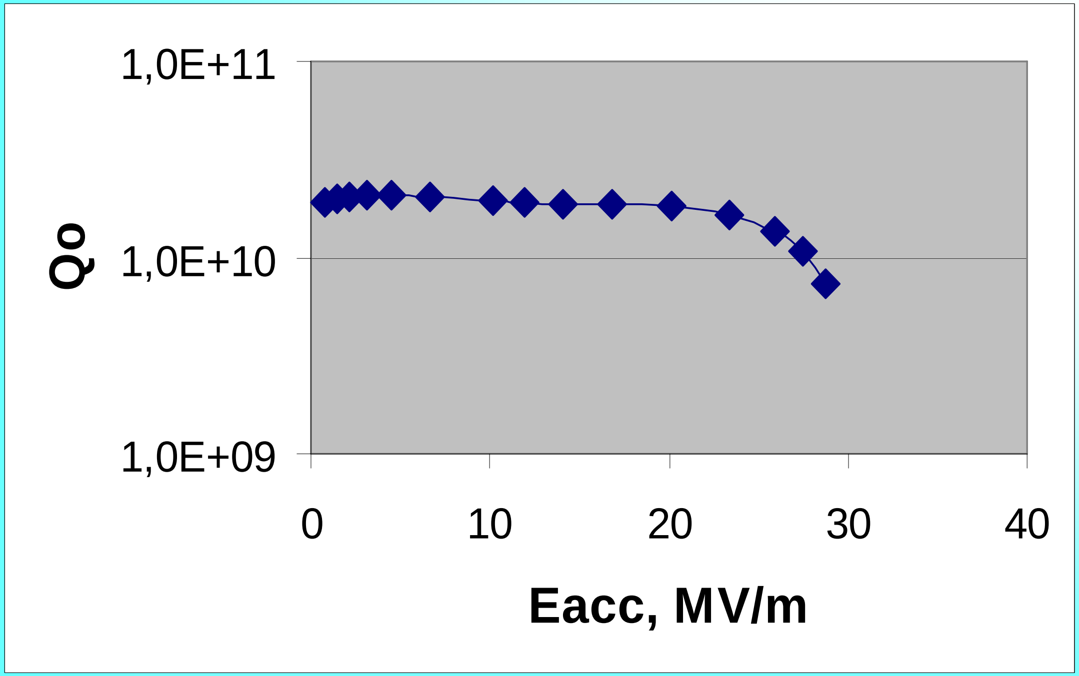


Deep drawn half cell of HERAEUS large grain niobium; Large single crystal at centre, no problems on iris area

Deep drawn half cells of Ningxia LG Nb; necking of the wall thickness at iris on grain boundaries



Q(Eacc) curve of the large grain single cell cavity 1AC3 after EP and BCP treatment



First Q(Eacc) curve of the large grain nine cell cavity AC114 after BCP treatment

LG

• Fabrication of single cell and multi cell cavities from large grain niobium by deep drawing and electron beam welding is feasible. Two aspects for fabrication are important: very desirable is to have only one large grain ca. 150 mm in the centre; desirable is to have only few large grain at the equator

• Accelerating gradient on the level of best fine grain cavities are achievable. A gradient up to 41 MV/m at $Q_0 = 1.4 \cdot 10^{10}$ (TB = 2K) was measured after electropolishing. Performance of ca. 29 MV/m was achieved on the nine cell cavity after only BCP treatment.

Grain boundaries GBs contribute to reduction of the cavity performance:

- responsible for magnetic field enhancement (steps on GBs after BCP)
- make easier the penetration of external magnetic field (GBs are planar weak links with reduced critical current density)
- additional RF resistance due to vortices penetrating along the grain boundary (reduce the quality factor Qo)
- make easier the hydrogen absorption and diffusion
- gathered impurities (reduced RRR)
- reduce the thermal conductivity at low temperatures (reduced phonon contribution)
- possibly make worse the baking (oxides and impurities in grain boundaries)
- possibly make worse high pressure water rinsing (enhance the surface roughness)

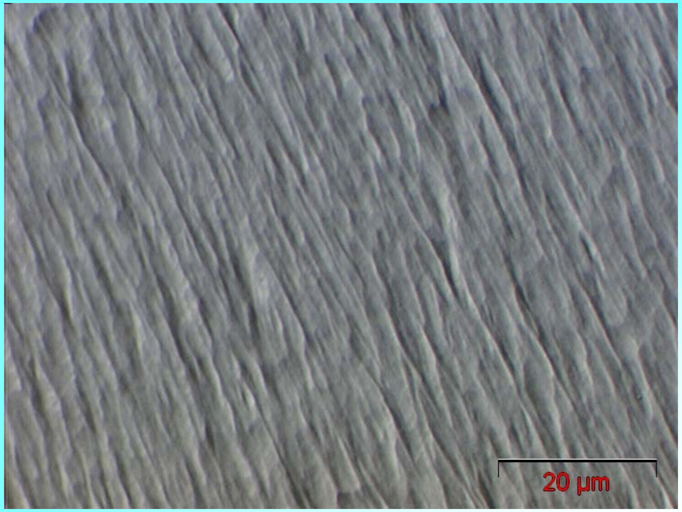
Single Crystal

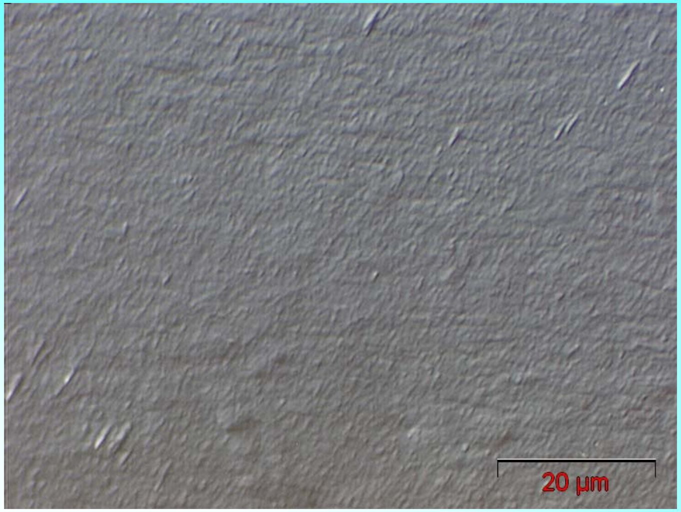
Fabrication of TESLA shape single crystal single cell cavities was proposed. Two single crystal single cell cavities 1AC6 (spinning + EB welding) and 1AC8 (deep drawing +EB welding) are produced

Following aspects have been investigated and taken into consideration during cavity fabrication

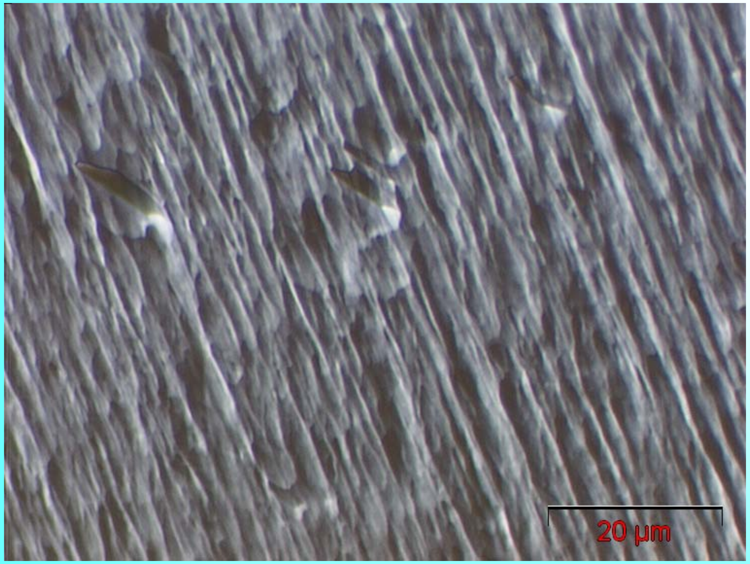
- Definite enlargement of the discs diameter is possible without destroying the single crystal structure in an existing state.
- Appropriate heat treatment will not destroy the deformed single crystal
- The single crystals keep the crystallographic structure and the orientations after deep drawing and annealing at 800°C
- Two single crystals will grow together by EB welding, if the crystal orientations is taken into account.

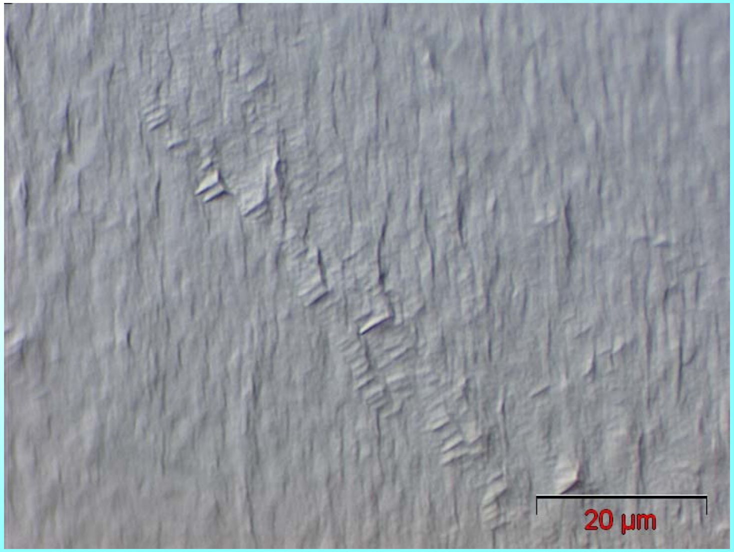
What deformation degree can withstand the SC?



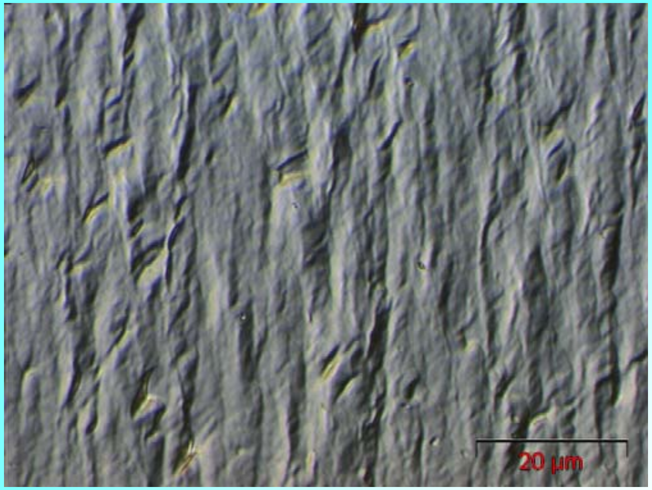


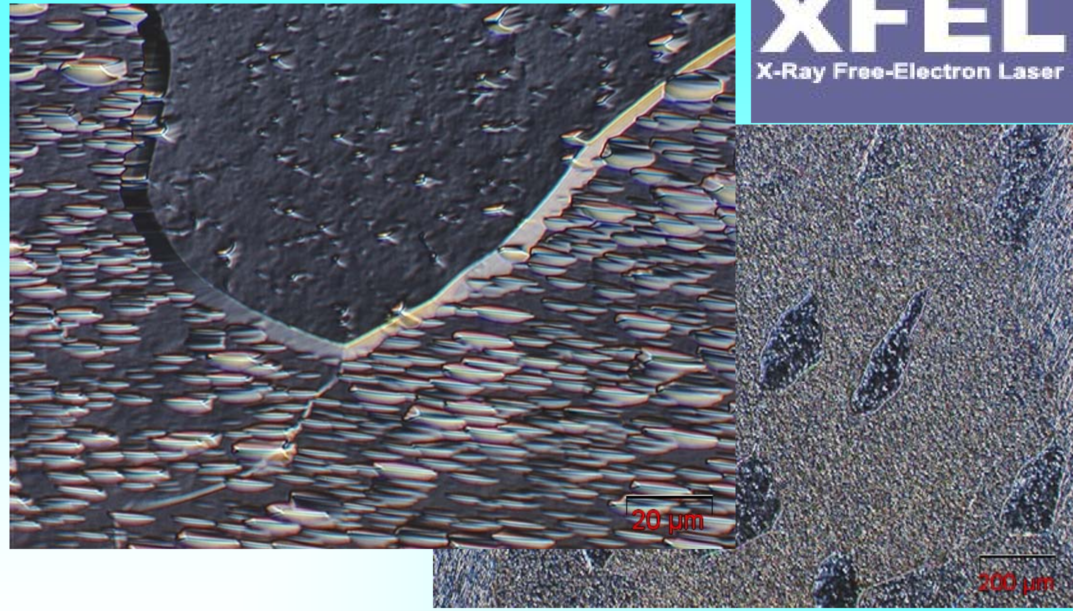
Nb single crystal after deformation degree of 60% (left) and annealing (right)



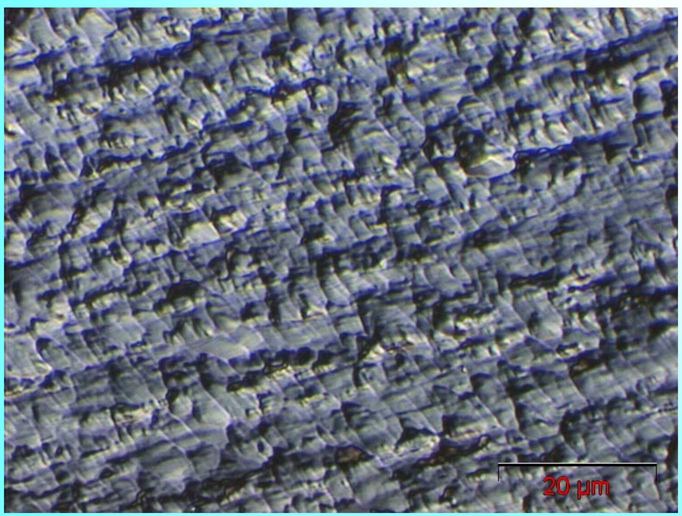


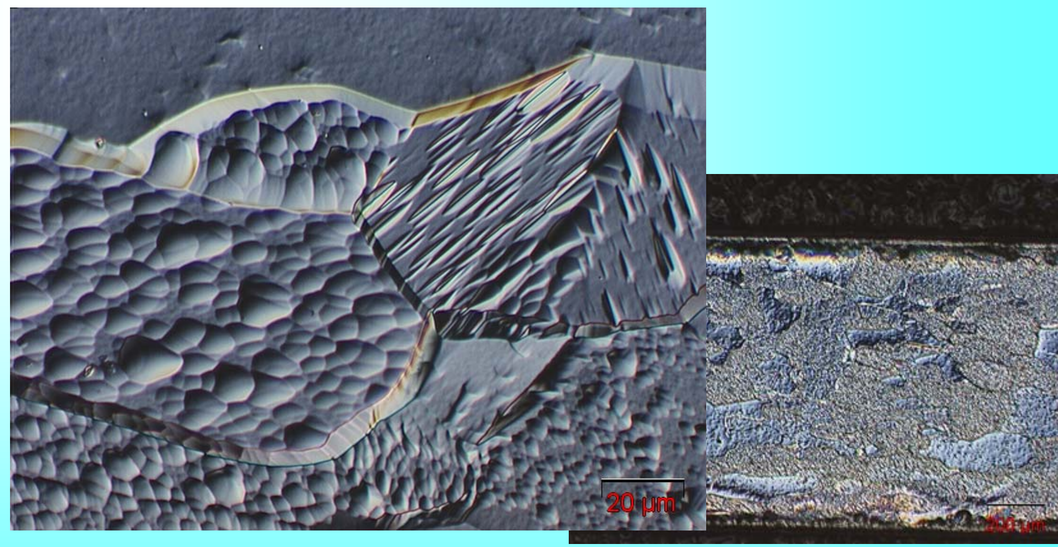
Nb single crystal after deformation degree of 70% (left) and annealing (right)



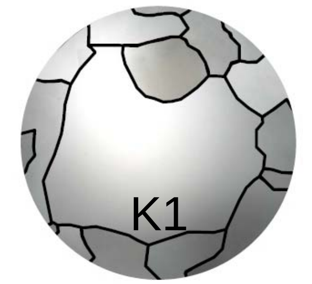


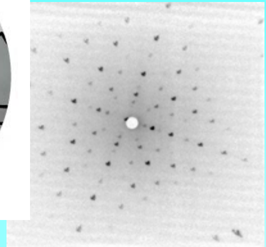
Nb single crystal after deformation degree of 80% (left) and additional annealing (right)





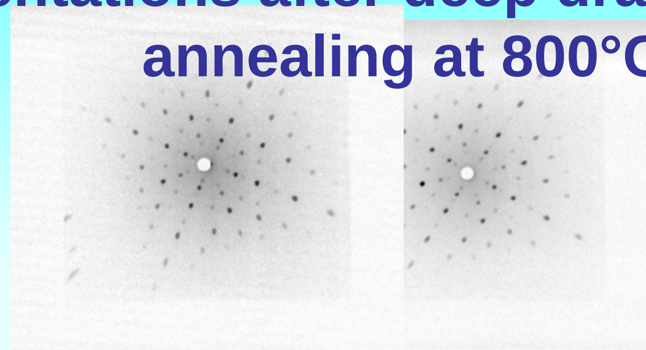
Nb single crystal after deformation degree of 90% (left) and annealing (right)



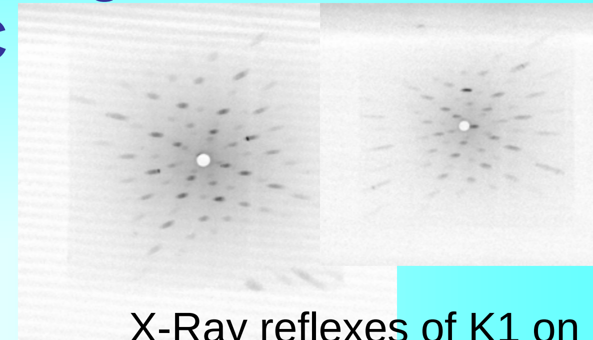


Single crystals keep the crystallographic structure and the orientations after deep drawing and annealing at 800°C

X-Ray reflexes of the central crystal K1 in the flat disc. Orientation (100)



X-Ray reflexes of K1 on position 1 before (above) and after annealing at 800°C, 2hs

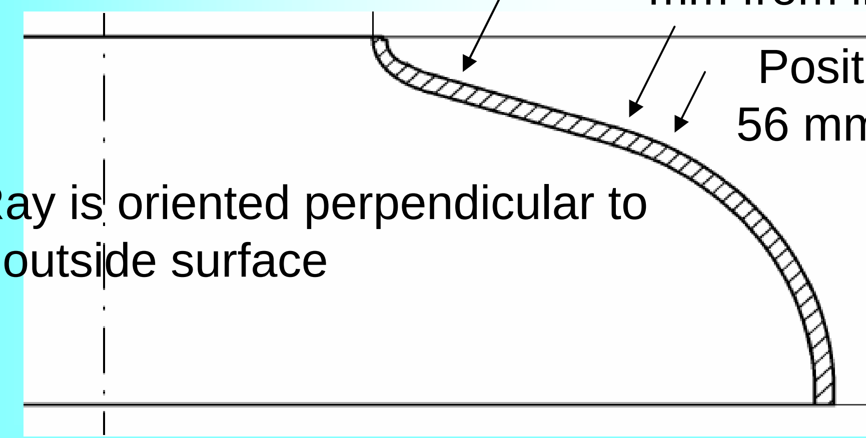


X-Ray reflexes of K1 on position 2 before (above) and after annealing at 800°C, 2hs

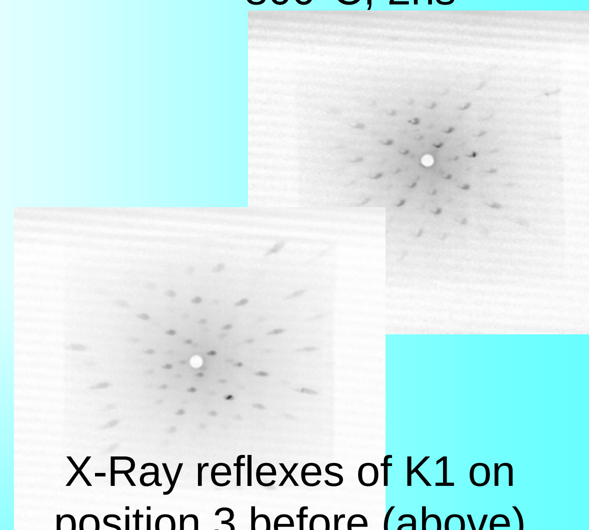
Position 1 ca. 20 mm from iris

Position 2 ca. 50 mm from iris

Position 3 ca. 56 mm from iris



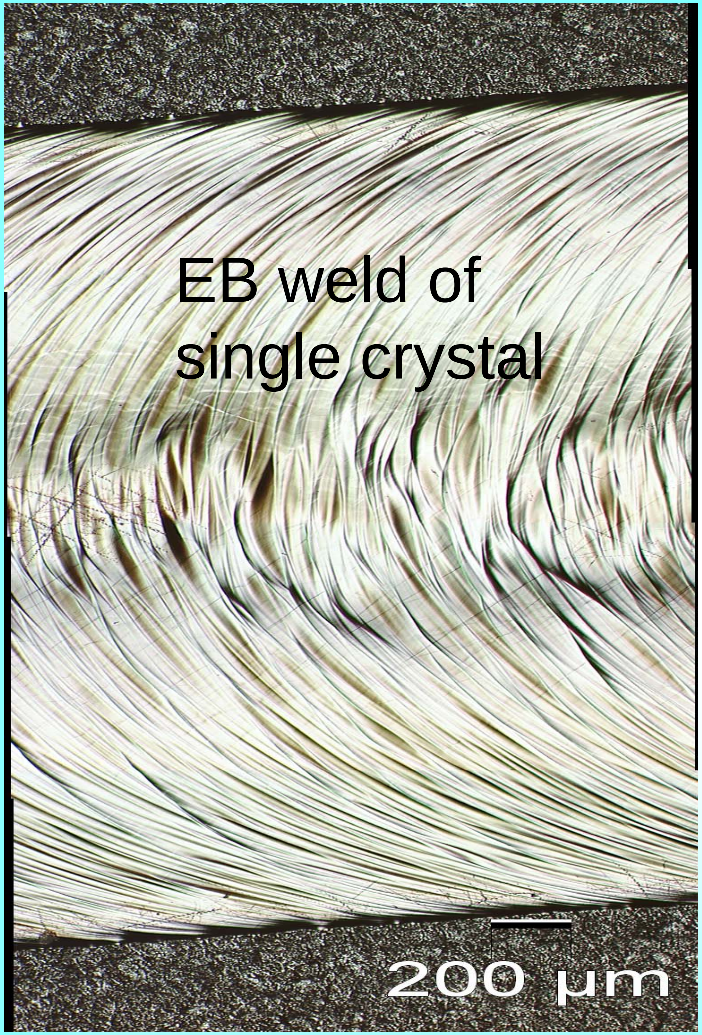
X-Ray is oriented perpendicular to the outside surface

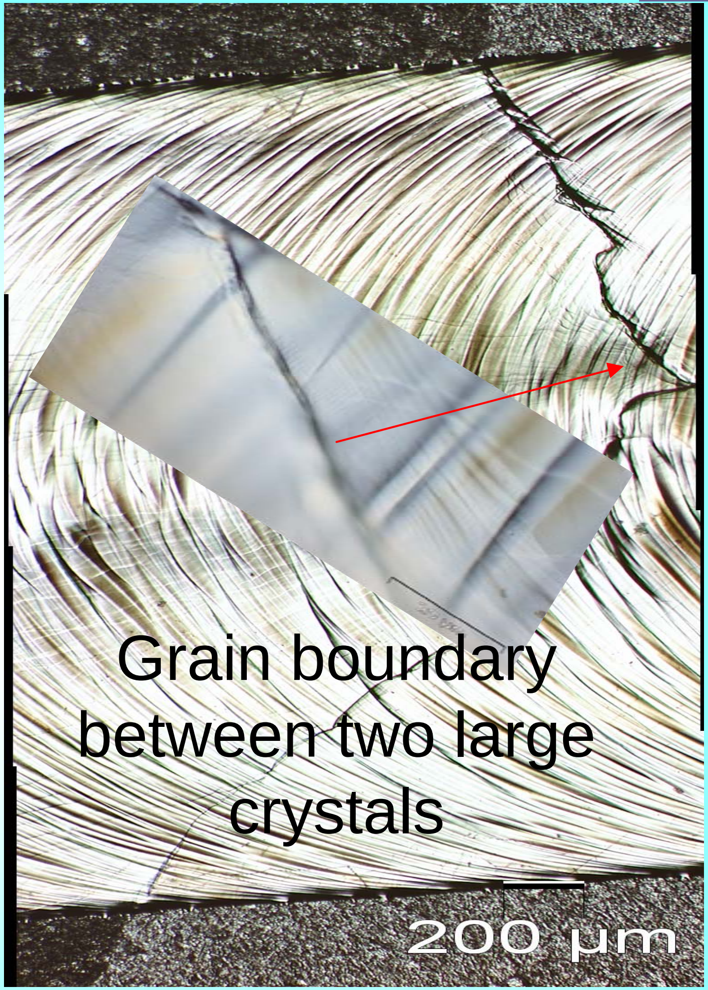


X-Ray reflexes of K1 on position 3 before (above) and after annealing at 800°C, 2hs

Determination of orientations of M. Spiwek (HASYLAB)

Electron beam welding





It seems that no new grains appear in the EB welding area, but the grain boundary remains

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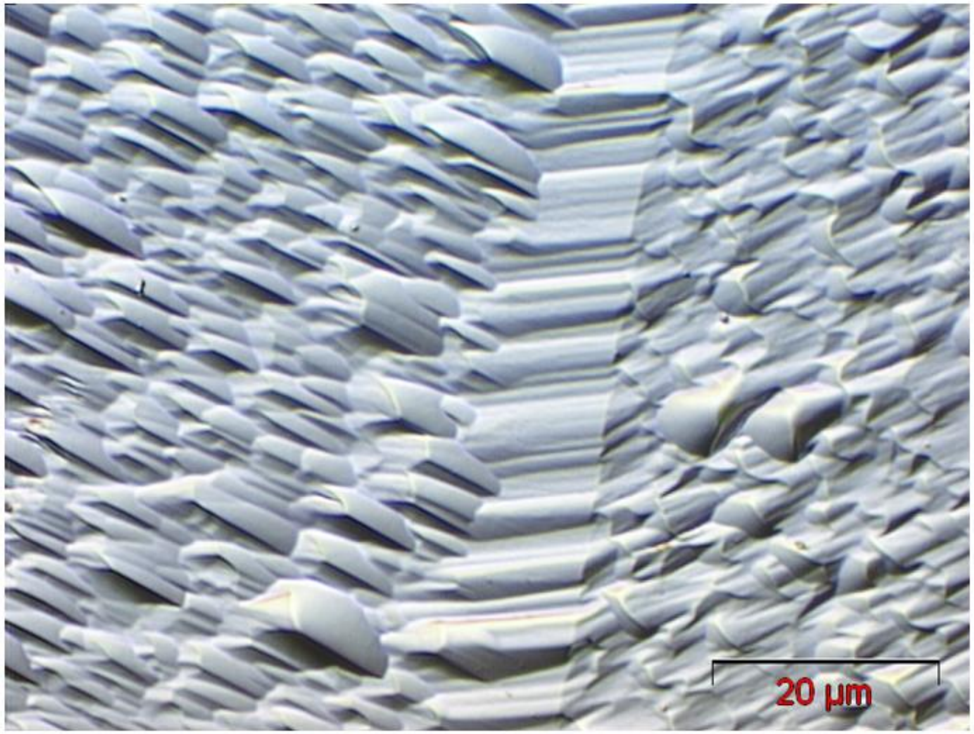
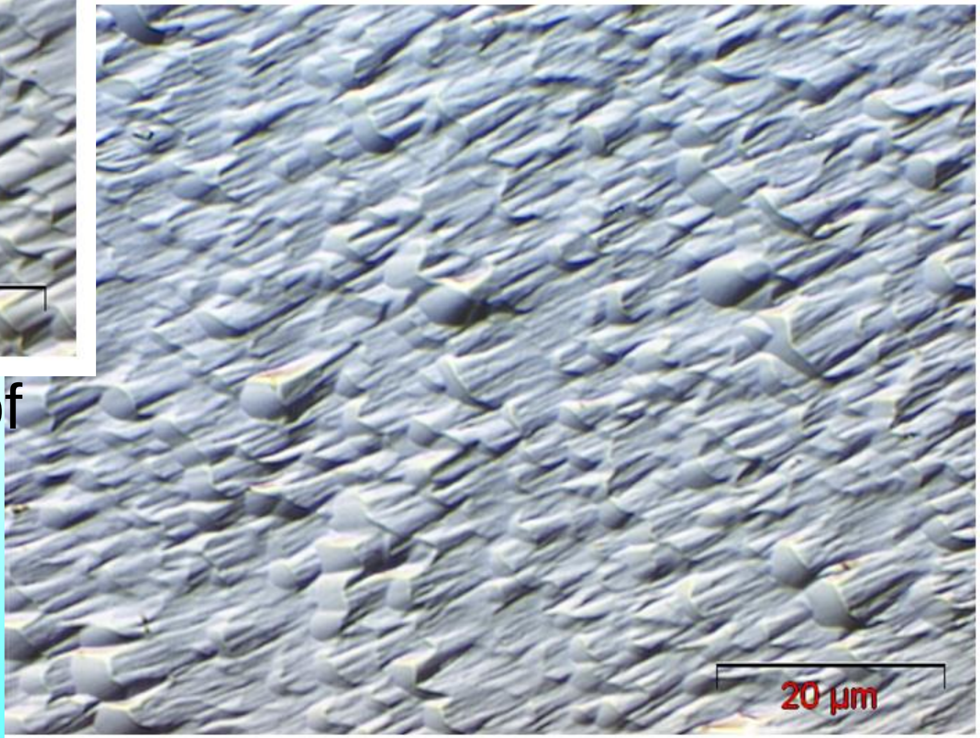
In the appropriate EB welding the single crystals can grow together

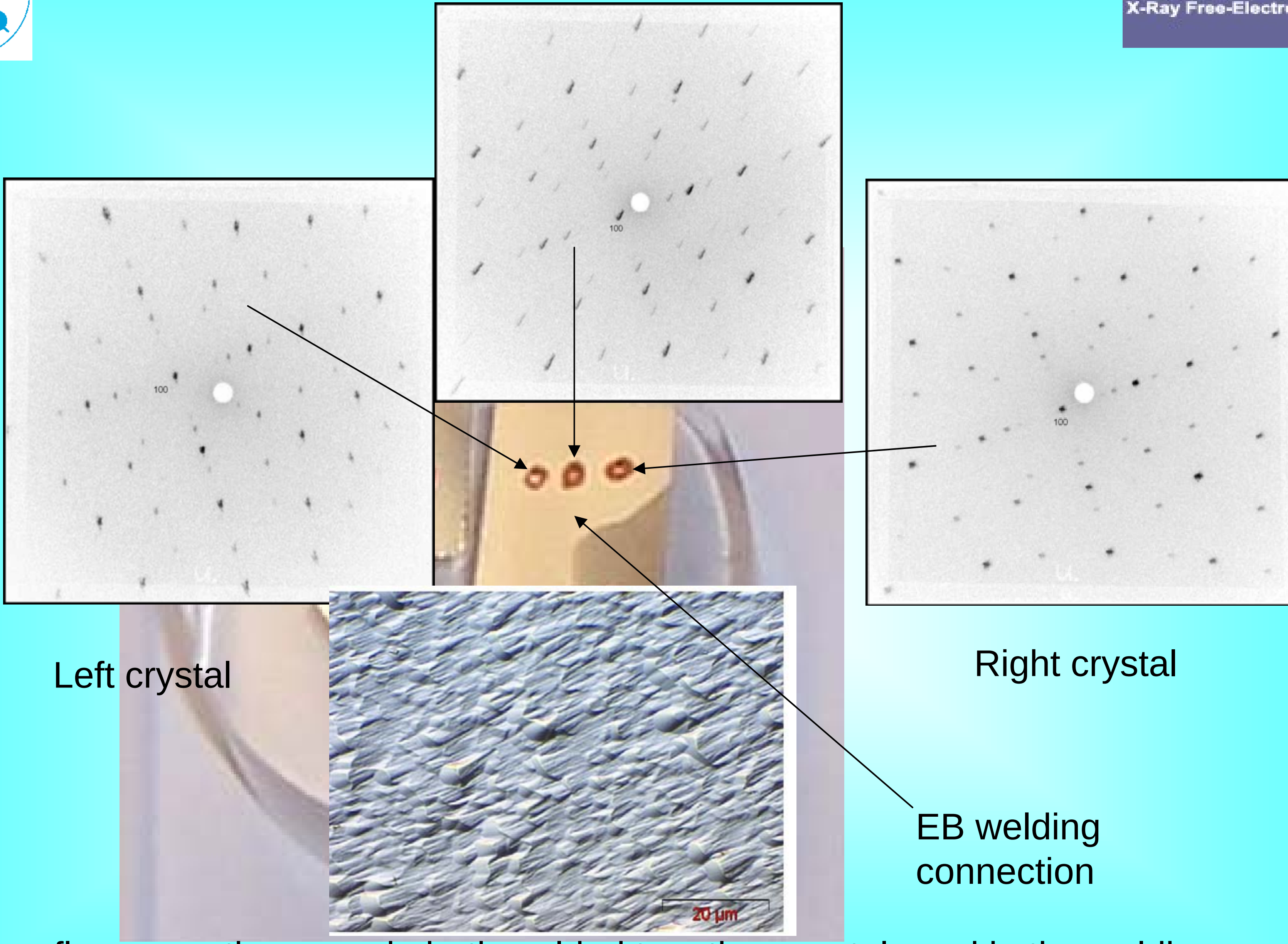


Left: Electron beam welding connection of two single crystals without regarding of crystals orientation (the grain boundary is pronounced)



Right: EB welding connection of two single crystals after assembling considering the crystal orientation (the grain boundary is absent)

EB welding seam



X-Ray reflexes are the same in both welded together crystals and in the welding seam

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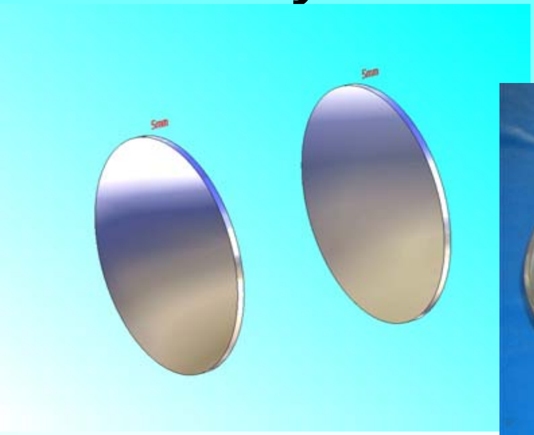
Single crystal cavity fabrication

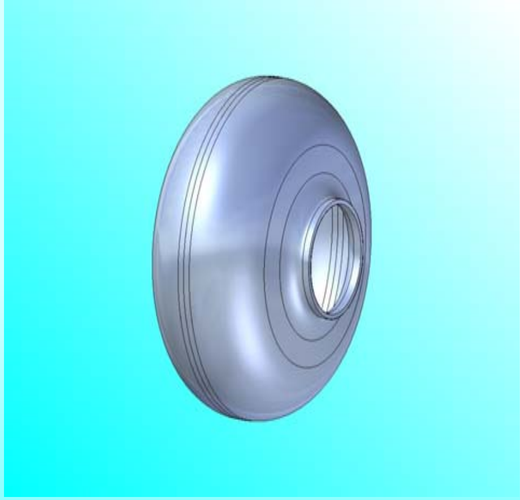


1. Take out central single crystal of definite thickness

2-3.Cutting through the disc and increasing of diameter by special rolling

Single crystals after deep drawing at ACCEL

4. Deep drawing



5. EB welding considering the crystal orientation

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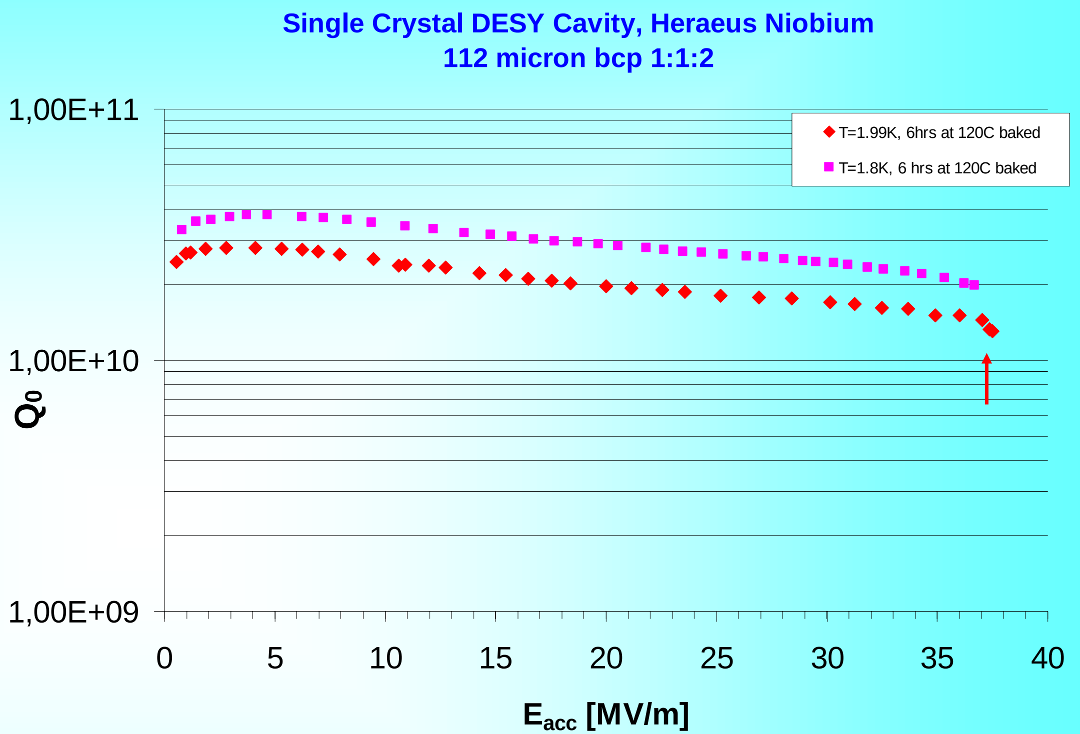
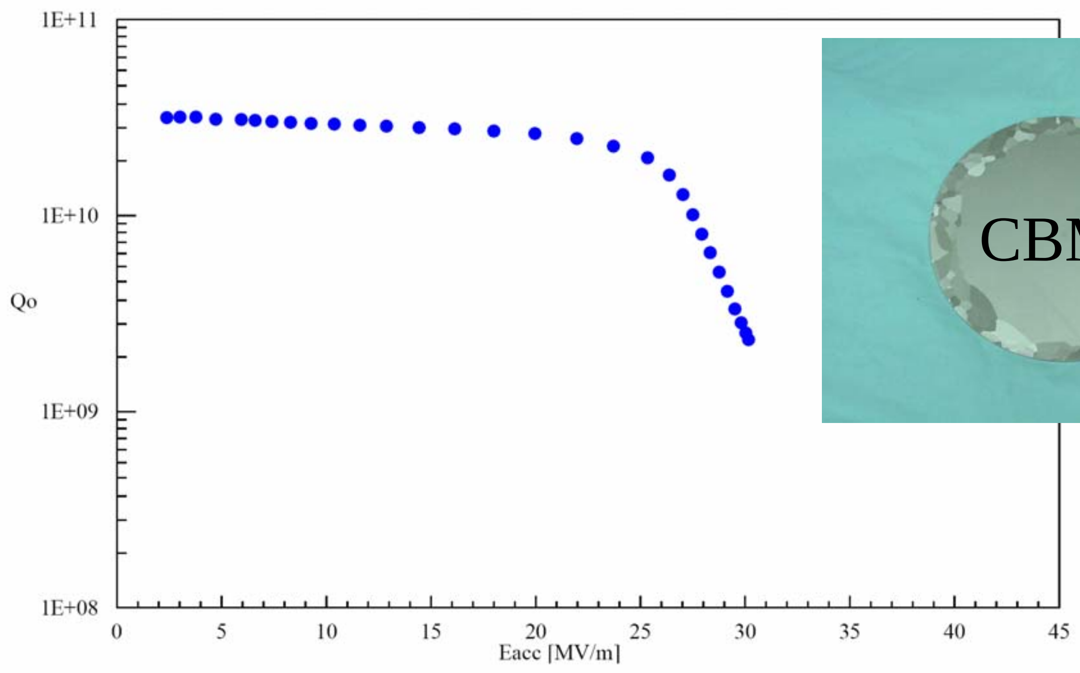
DESY single crystal cavity 1AC8 build from Heraeus disc by rolling at RWTH, deep drawing and EB welding at ACCEL



Q(Eacc) curve after only 112 µm BCP and in situ baking 120°C for 6 hrs.

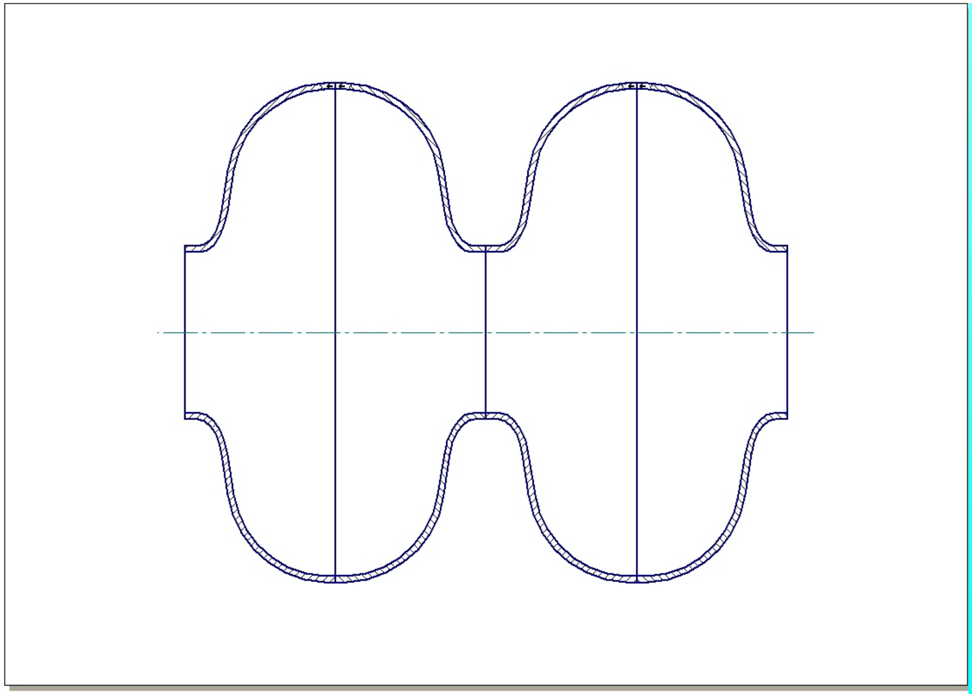
Preparation and RF tests of P.Kneisel, JLab

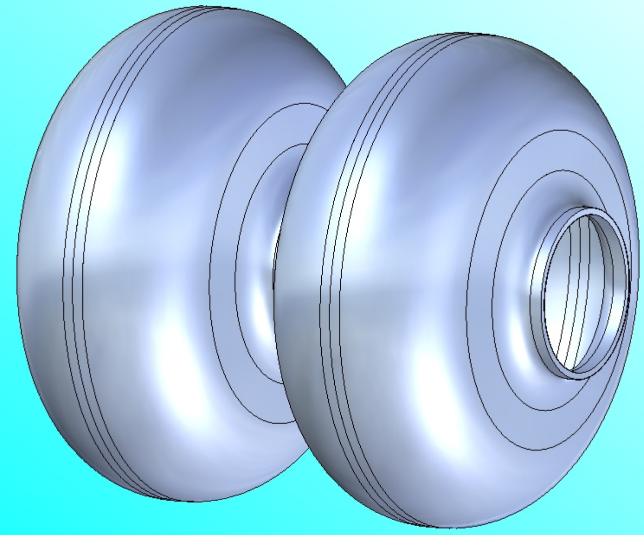


DESY single crystal cavity 1AC6; CBMM material, ca. 250 µm BCP, 100 µm EP at Henkel. X-Ray started at 24 MV/m. Limited by power

Single cell cavity 1AC6 fabricated by ACCEL (spinning and EB welding) from CBMM single crystal of diameter ca. 200 mm

SC. It works. The proposed method can be extended on fabrication of multi cell cavities.



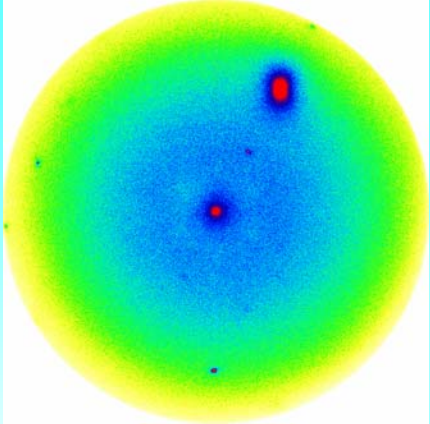


Summary for on LG/SC cavities (best result, 1.8K)

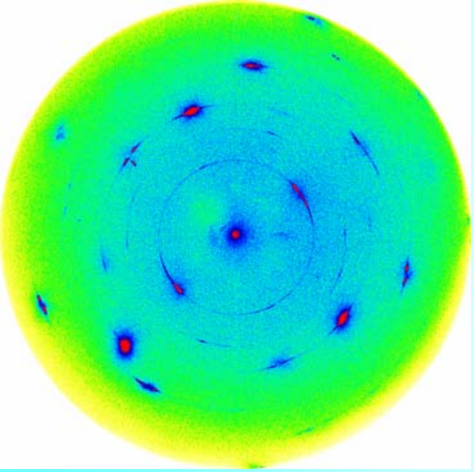
Material of the company	No./Type	Treatment	Eacc, MV/m	Qo at Eacc=23.5	Limitation
Heraeus/large grain	1AC3/single cell	190µm **EP**, 800°C 2h, 120°C 48h, HPR	41.2	3.2E+10	Quench at equator
Heraeus/large grain	1AC4/single cell	190µm **EP**, 800°C 2h, 128°C 48h, HPR	38.5	2.3E+10	Quench at equator
Heraeus/large grain	1AC5/single cell	275 µm **EP**+**BCP**, 800°C 2h, 135°C 48h, HPR	29.7	2.0E+10	Quench, not equator
CBMM/single crystal	1AC6/single cell	360µm **BCP**+**EP**, 750°C, HPR	30.1	2.3E+10	Field Emission
Heraeus/large grain	1AC7/single cell	220µm **BCP**, 800°C 2h, 120°C 48h, HPR	25.3	3.0E+10	Quench
Heraeus/single crystal	1AC8/single cell	112µm **BCP**, 120°C for 6 hrs	37.5	2.9E+10	Quench
Heraeus/large grain	AC114/nine cell	100µm **BCP**, 800°C, 20µm BCP, HPR	28.7	2.1E+10	Quench probably FE induced

Material Investigation:

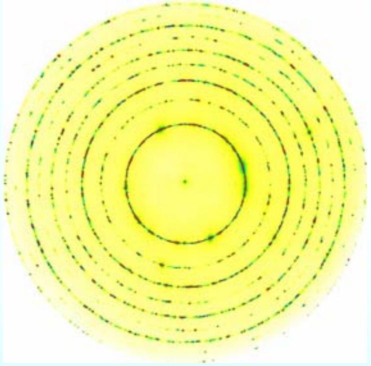
Crystal characterization (H.-G. Brokmeier curried out on BW5 of DESY HASYLAB for X-ray experiments)



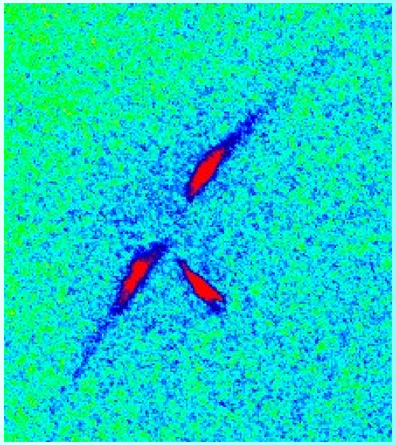
Real single crystal (sharp reflexes)



Not real single crystal



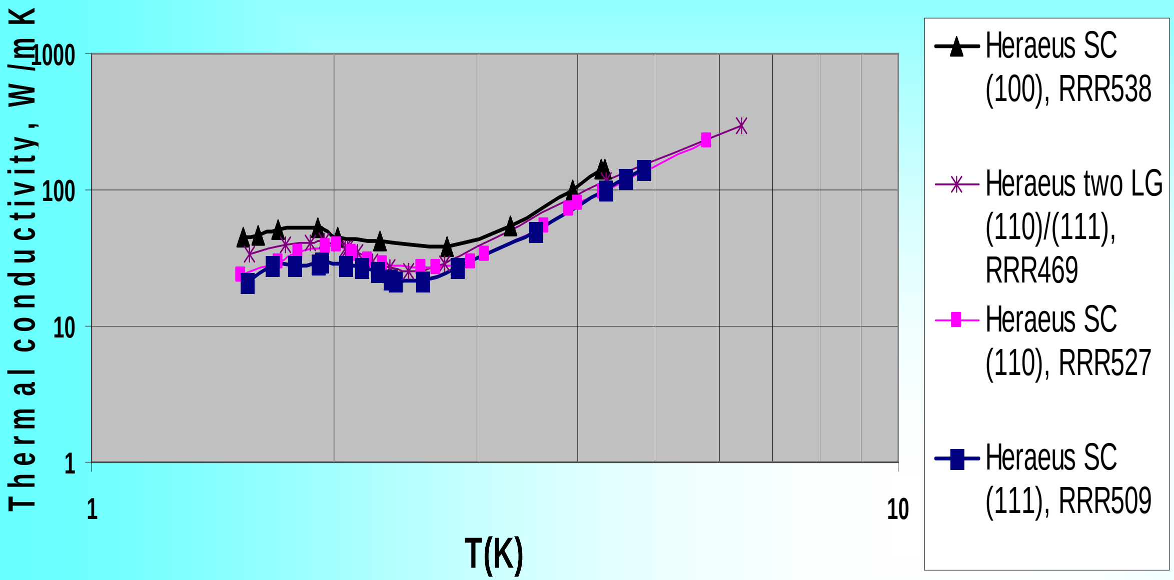
Powder X-Ray reflexes

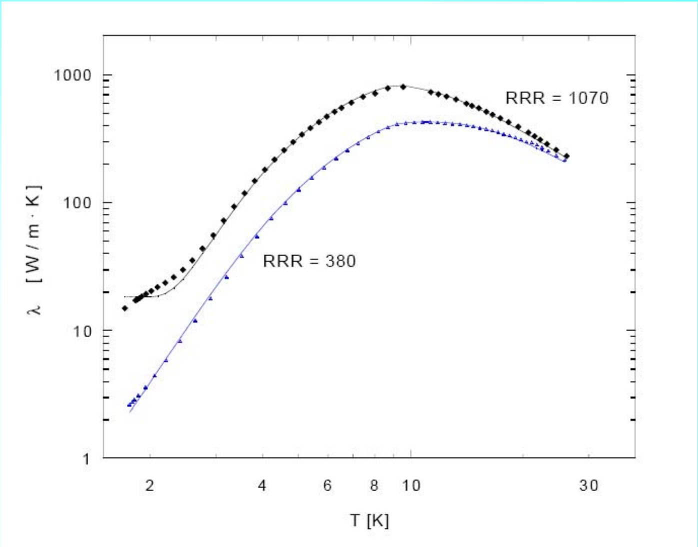


Signal splitting

Signal splitting of reflexes (indication for several single crystals) and Debye Scherrer rings (indication for the powder or small crystals material) have been observed in addition to the SC signal in some LG.

Thermal conductivity

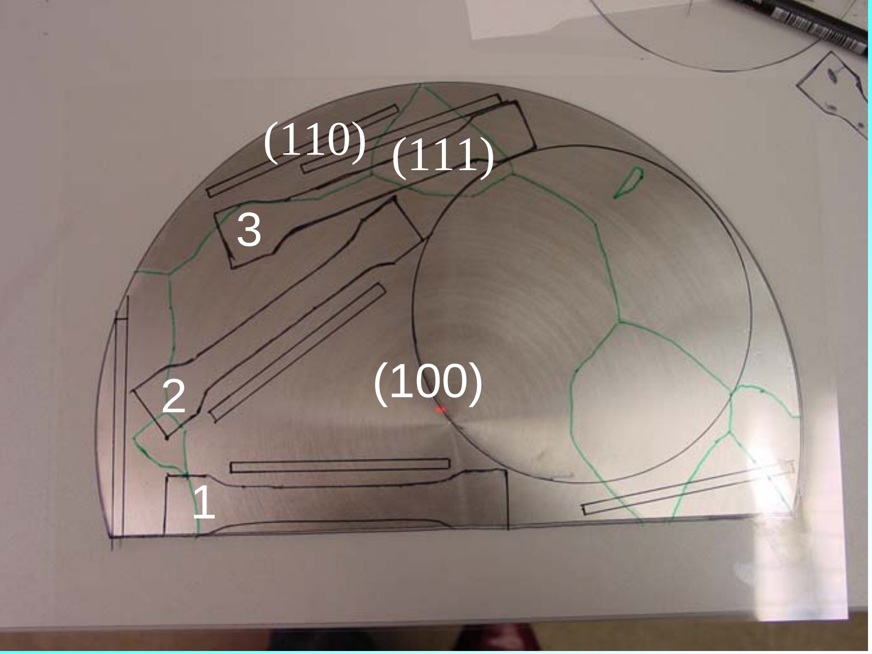


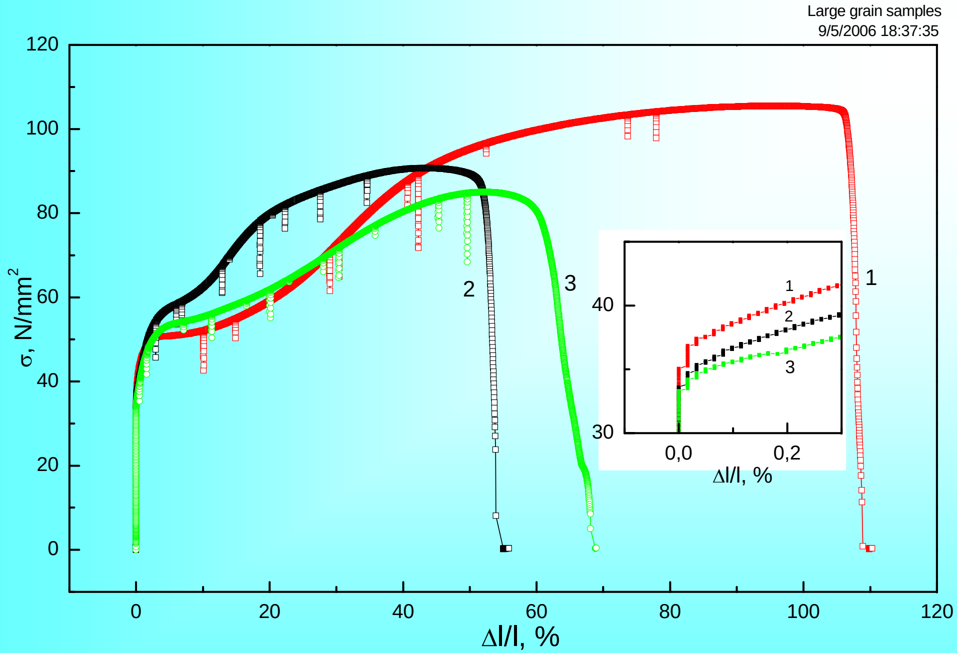


Thermal conductivity of HERAEUS single crystal (100), HERAEUS single crystal (110), HERAEUS single crystal (111), HERAEUS large grain (two grains (110) and (111)) at low temperatures. Phonon peak is clearly pronounced

Thermal conductivity of TTF polycrystalline niobium (Th. Schilcher etc.)

$$\lambda(T,RRR,G) = R(y)\cdot\left[\frac{\rho_{295K}}{L\cdot RRR\cdot T} + a\cdot T^2\right]^{-1} + \left[\frac{1}{D\cdot\exp(y)\cdot T^2} + \frac{1}{B\cdot G\cdot T^3}\right]^{-1}$$





Mechanical properties: Tensile test

Percentage elongation after fracture for LG is rater high despite of grain boundaries.

Percentage elongation after fracture for SCs depends significantly on the load direction

Strain-stress curve of LG/SC niobium

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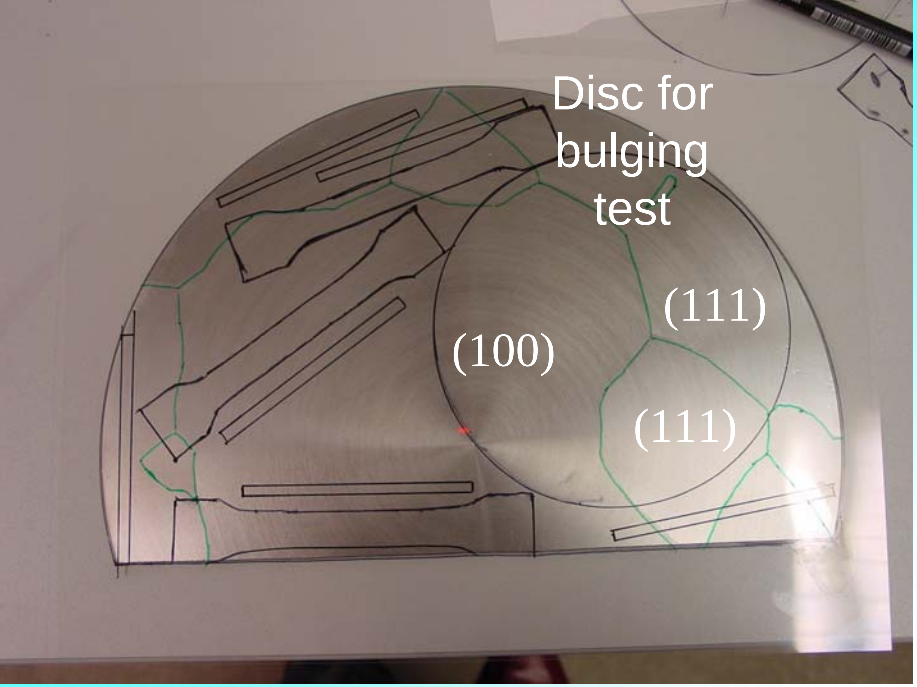

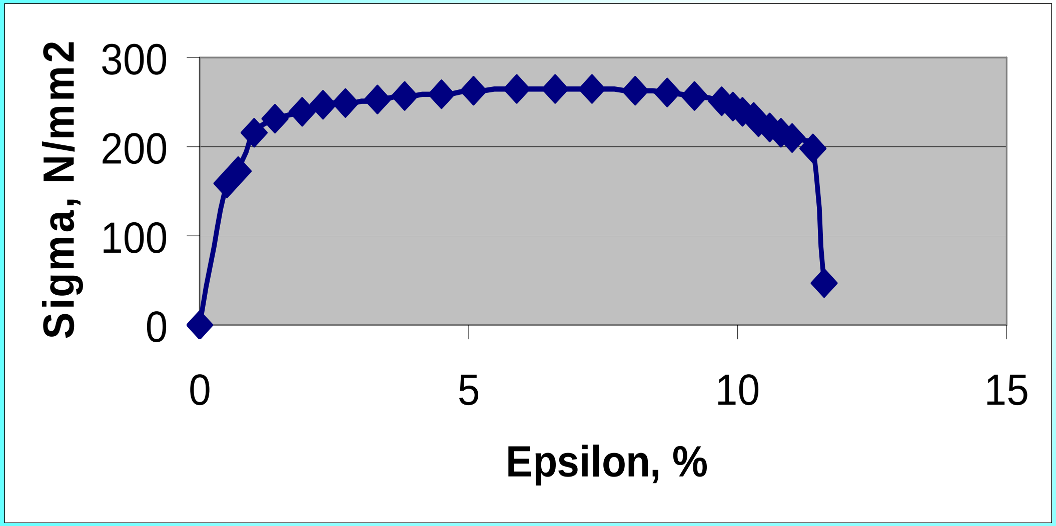

Bulging test on large grain niobium.

Mechanical properties: Bulging test

Percentage elongation after fracture by two dimensional deformation for LG is low (<15%). The rupture takes place close to grain boundary

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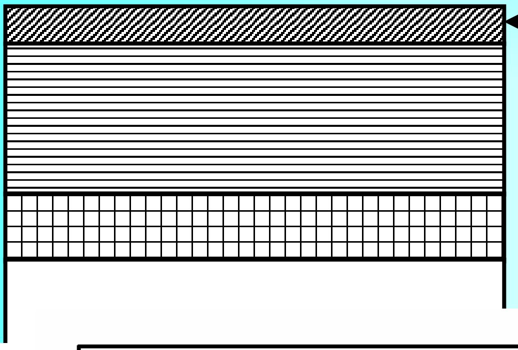
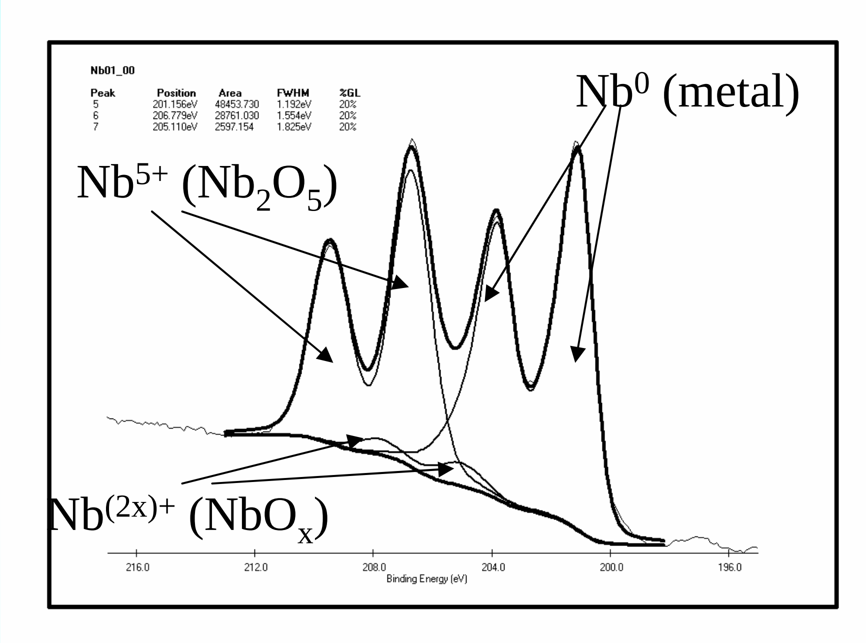
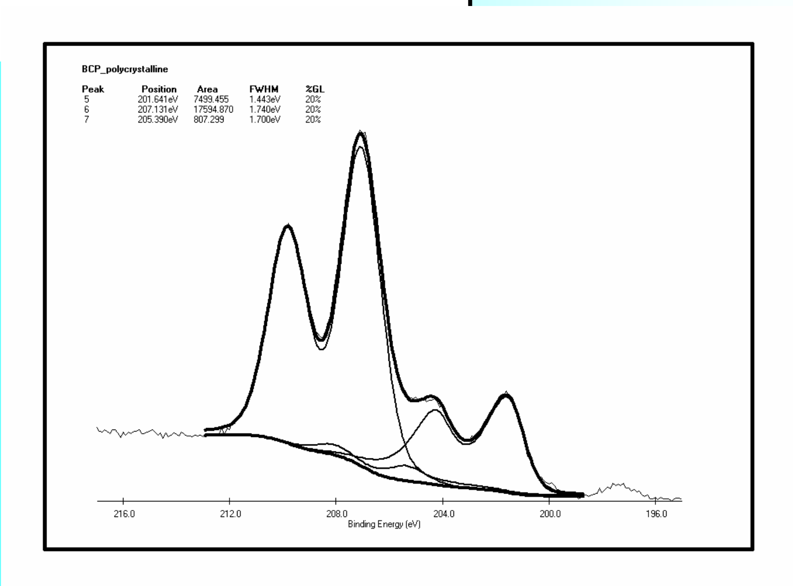
XPS on single crystal with different crystal orientation (preliminary results)

K.Kowalski, A.Bernasik (SSL, Krakow)





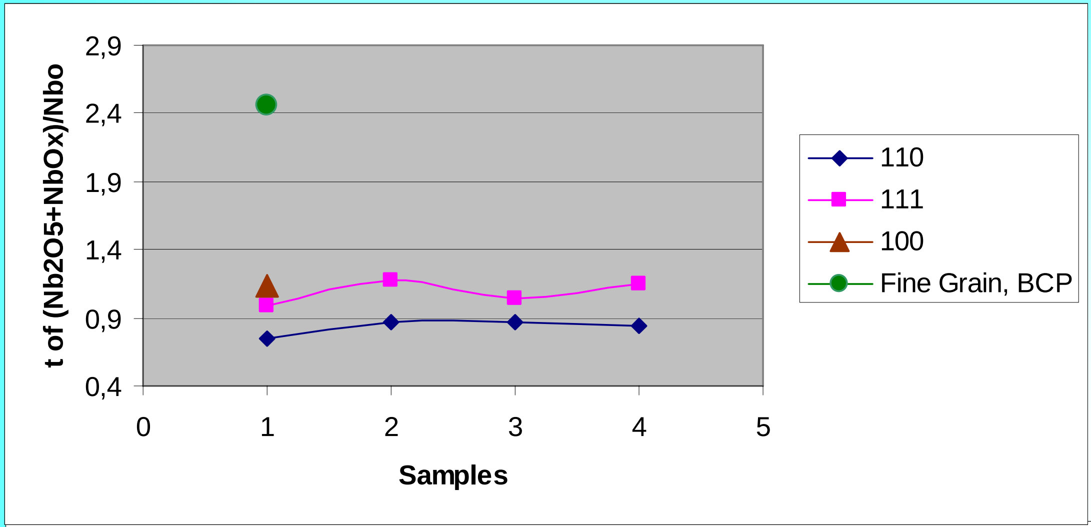
XPS spectrum for BCP single crystal Nb



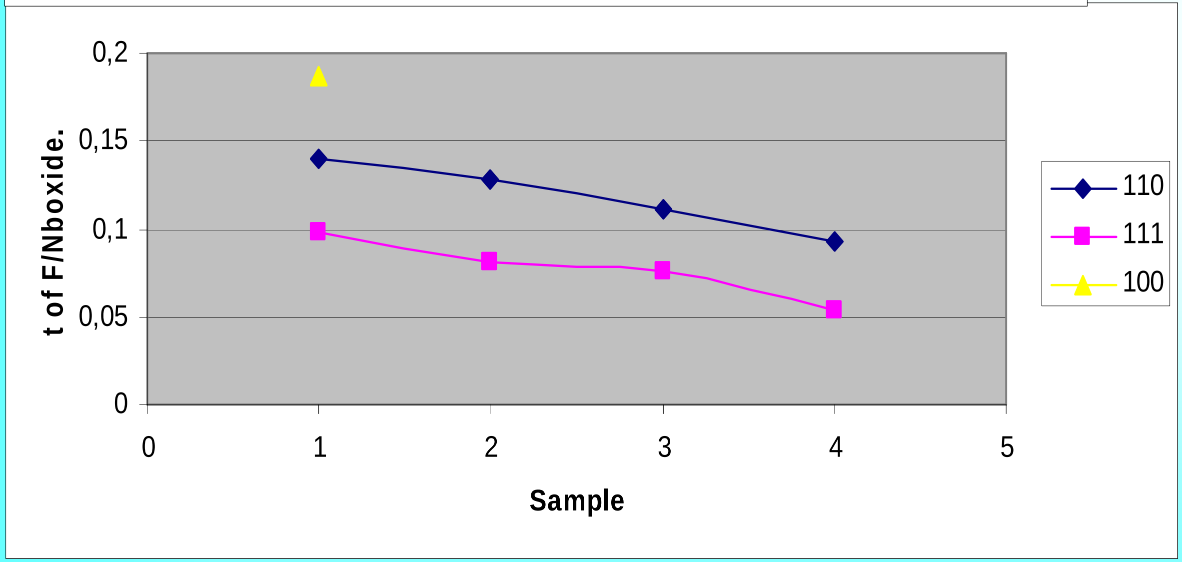
XPS spectrum for BCP polycrystalline Nb

The oxide layer is thicker in polycrystalline Nb compare to single crystal

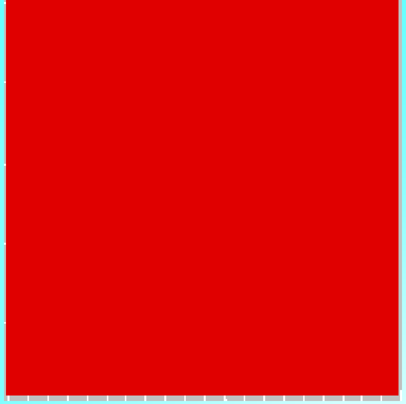
XPS on single crystal

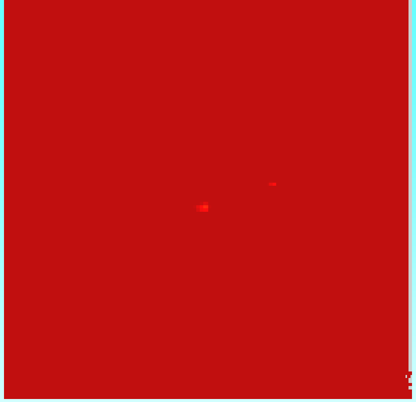


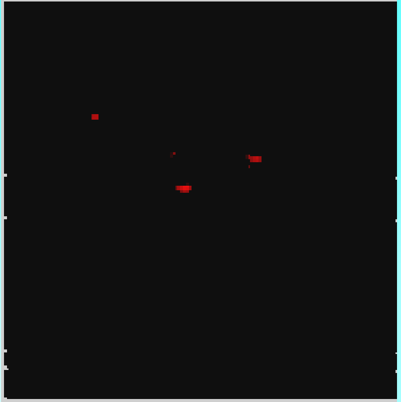
The thickness of the oxide layer is smaller in single crystals compare to fine grain sheet and depends on the crystal orientation of substrate bulk niobium



Surface contamination by fluorine depends on the crystal orientation of substrate bulk niobium

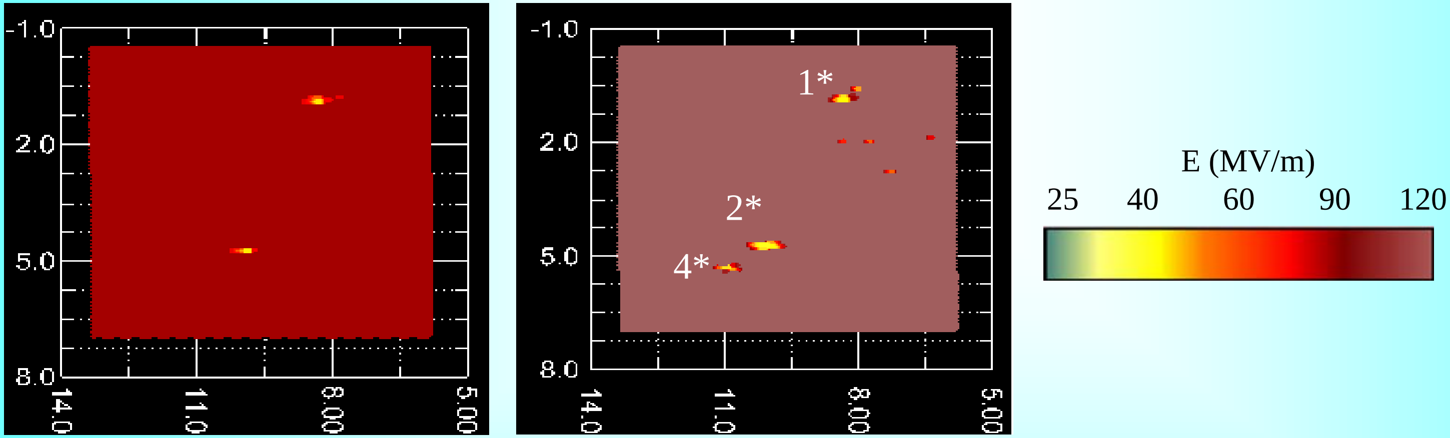






E = 120 MV/m, 0 emitters E = 150 MV/m, 2 emitters E = 200 MV/m, 4 emitters

FE scans on single crystal Nb sample after 30 µm BCP.



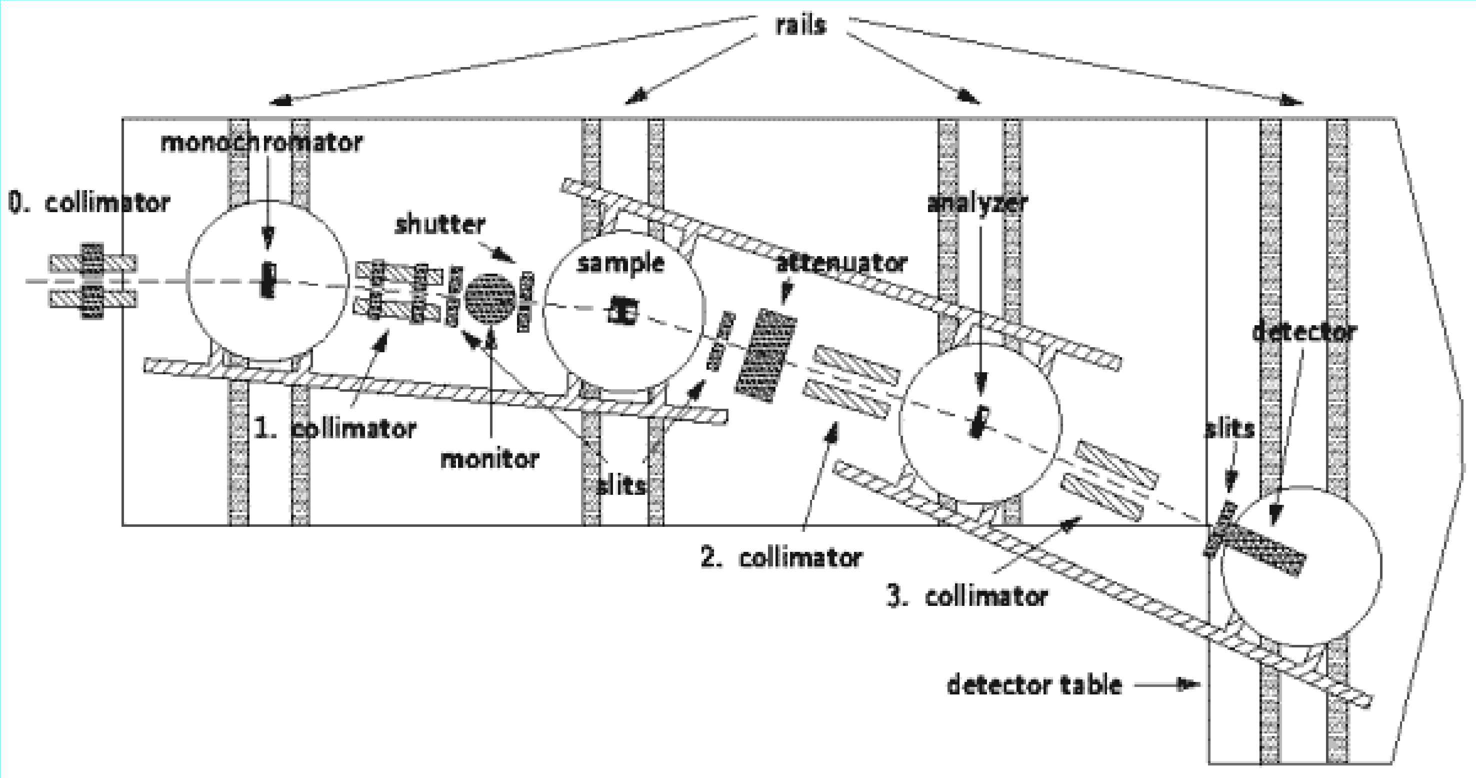
Example of similar FE scans on fine grain EP Nb sample. (left) E = 90 MV/m, 3 emitters (right) E = 120 MV/m, 8 emitters

Field Emission Scanning: A.Dangwal, G.Mueller (Wuppertal)

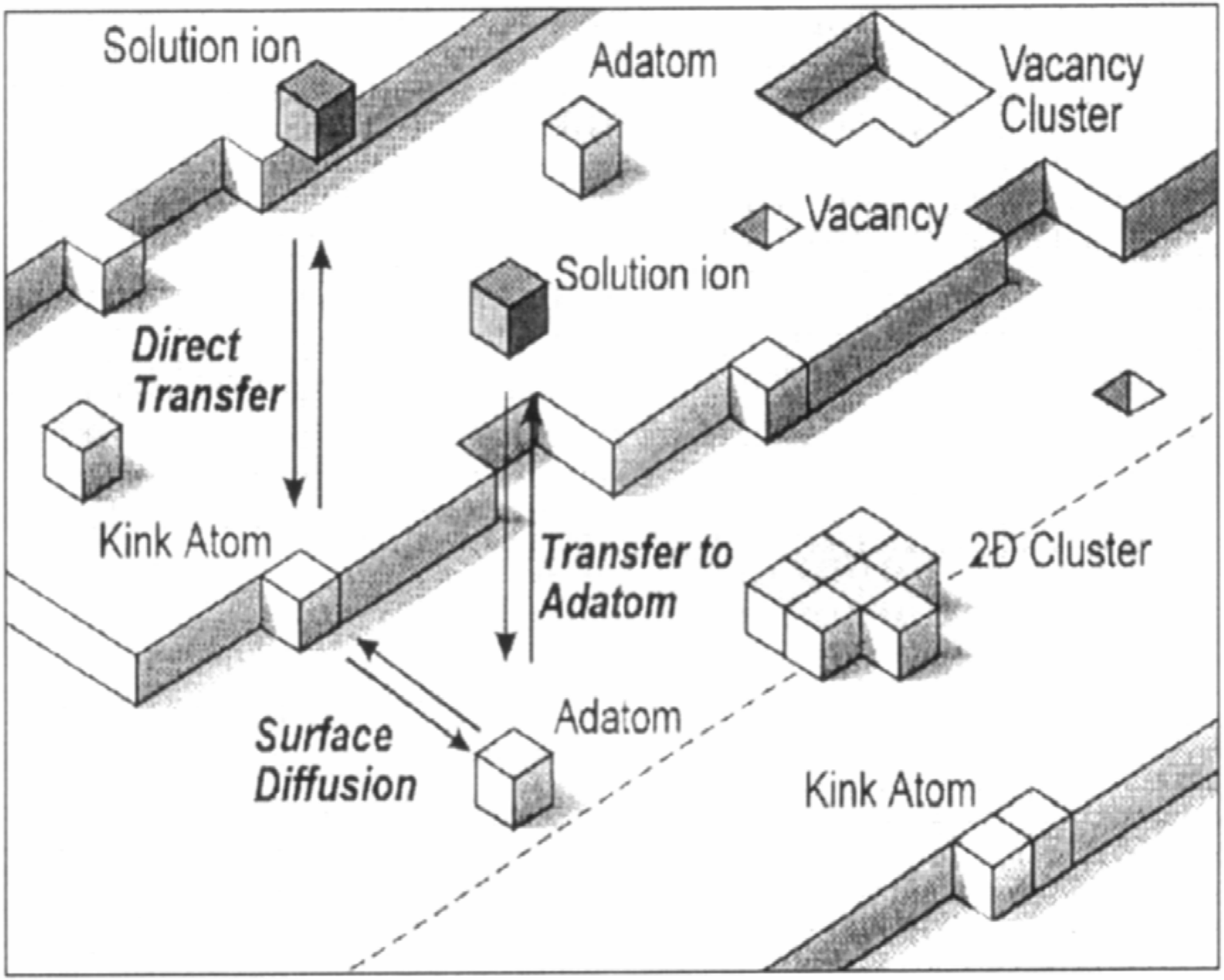
Surface quality of the BCP treated SC is better as of EP treated polycrystalline Nb

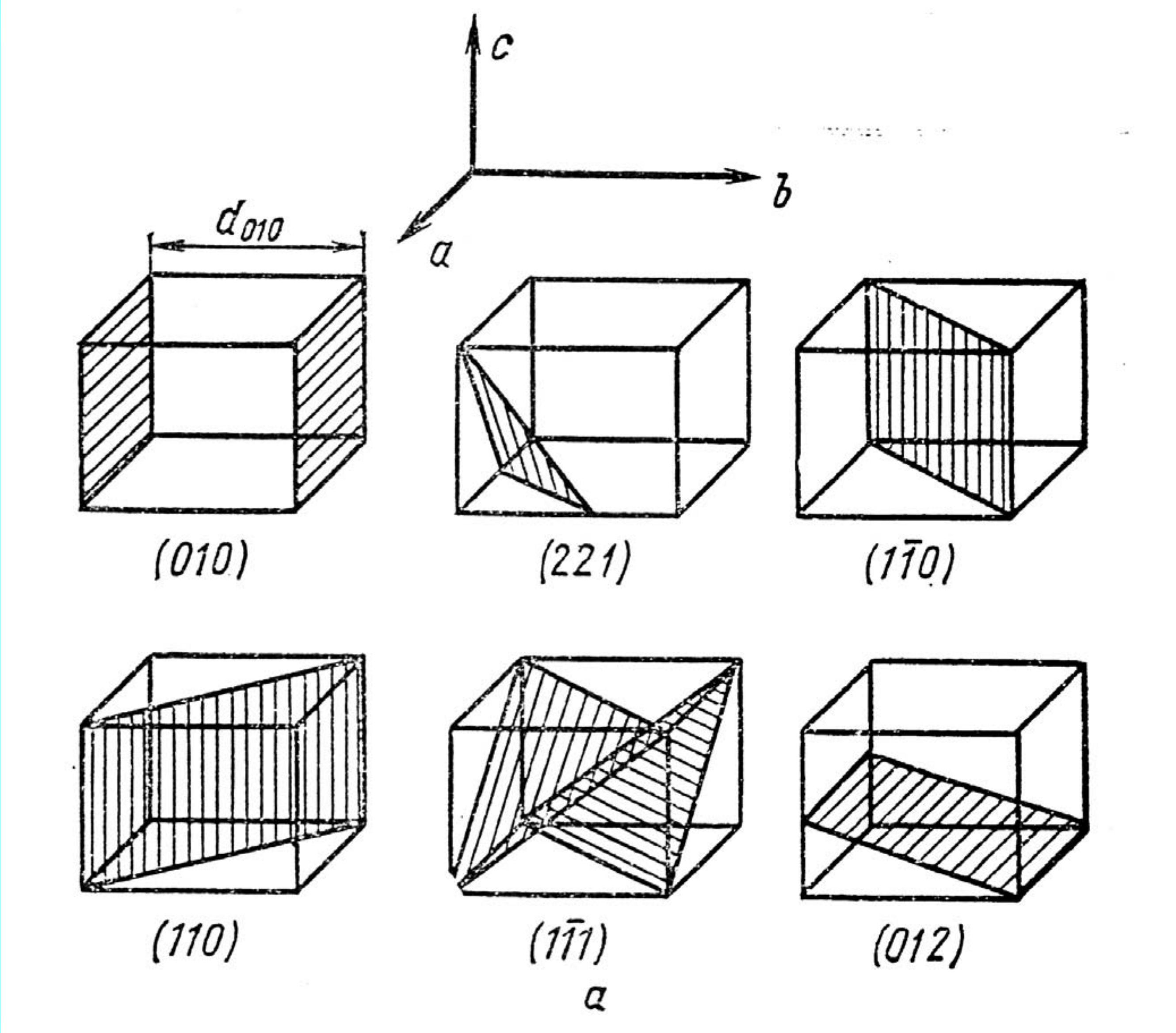
A lot of aspects of LG and SC have to be understand

- How to produce SCs of required dimensions ?
- What is the best crystal orientation for the best cavity performance (Hc1, Hc2, Hc3 dependence on crystal orientation)?
- Is the baking mechanism for SCs and LG the same as for polycrystalline material?
- Are the SCs surfaces oxides different compare to polycrystalline Nb and depend on the crystal orientation of the niobium substrate?
- Is higher onset of field emission for LG and SCs caused only by smooth surface or the mechanism is more sophisticated?
- What is the difference between EP treated and BCP treated LG material?
- Why the one dimensional tensile test on LG demonstrates high elongation, but in the two dimensional bulging test the elongation is much smaller?
- What are the exact conditions allowing to connect two SCs in one SC by EB welding and where is the limitation?
- What maximal deformation degree can tolerate the SCs and what is the optimal heat treatment for SCs?



BW5 of DESY HASYLAB for X-ray experiments


Solution ion
Adatom
Vacancy
Cluster
Vacancy
Solution ion
Direct
Transfer
Kink Atom
Transfer to
Adatom
2D Cluster
Surface
Diffusion
Adatom
Kink Atom


c
b
a
d_{010}
(010)
(221)
$(1\bar{1}0)$
(110)
$(1\bar{1}1)$
(012)
a