Sample environment for powder diffraction

on example of high-resolution powder diffractometer SPODI

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Diffraction instrumentation at European neutron large scale facilities

Orphée
Laboratoire Léon Brillouin

- 3T2
- G4.1
- G6.1
- PHR-G44
- 5C1
- 5C2
- 6T2
- 7C2
- 6T1

HZB Helmholtz Zentrum Berlin

- E2
- E3
- E4
- E5
- E6
- E9
- E11
- EXED

NEUTRONS FOR SCIENCE

- D2B
- D20
- D4
- D1B
- D10
- D19
- D23
- OrientExpress
- D7
- CYCLOPS
- D3
- D9
- SALSA

ESS EUROPEAN SPALLATION SOURCE

- DREAM
- HEIMDAL
- MAGIC
- NMX
- BEER

PAUL SCHERRER INSTITUT
PSI

- HRPT
- DMC
- ZEBRA
- POLDI
- ORION

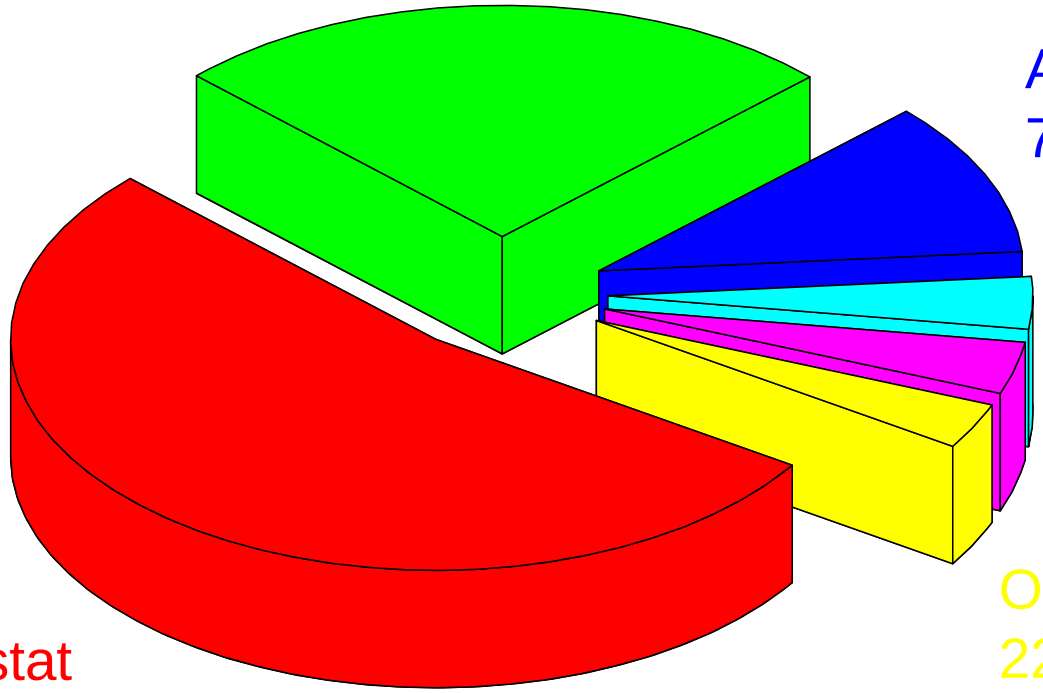
ISIS

- ENGIN-X
- GEM
- HRPD
- IMAT
- INES
- NIMROD
- OSIRIS
- PEARL
- POLARIS
- ROTAX
- SANDALS
- SXD
- WISH

MLZ
Heinz Maier-Leibnitz Zentrum

- SPODI
- STRESS-SPEC
- HEIDI
- POLI
- BIODIFF
- DNS
- RESI
- ERWIN
- SAPHIR
- POWTEX

Sample environment use at SPODI

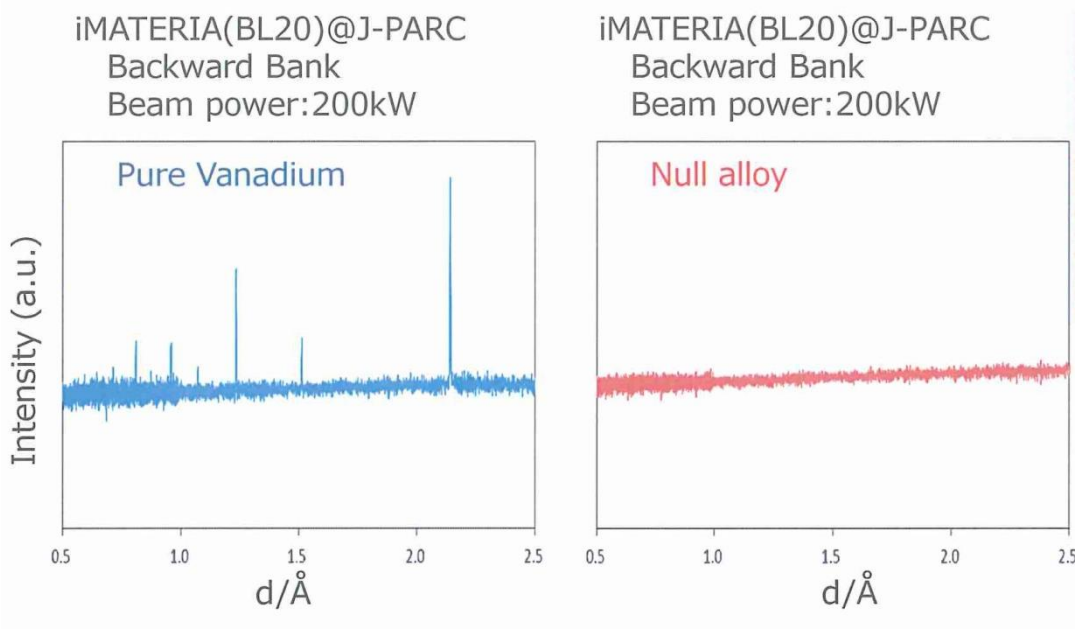


Ambient temperature – containers, cans etc.

Vanadium

© *MTI Albany*

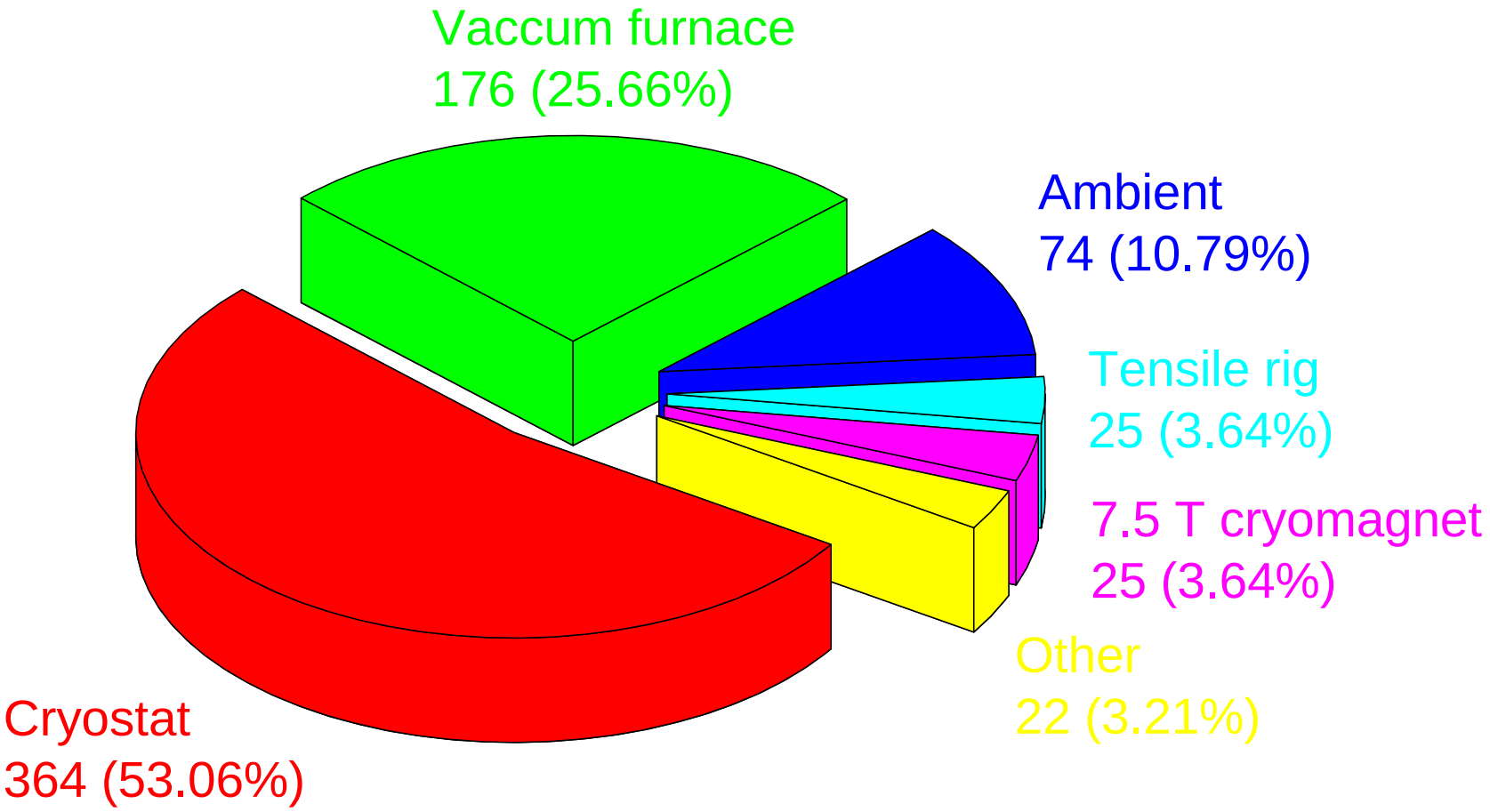
Typical diameters: 3-16 mm
Wall thickness: 100-150 µm
Price: 80-1000 $ per item



Null alloy and Titan-Zirconium

Neutron scattering lengths and cross sections							
Isotope	**conc**	**Coh b**	**Inc b**	**Coh xs**	**Inc xs**	**Scatt xs**	**Abs xs**
V	---	-0.3824	---	0.0184	5.08	5.1	5.08
Ni	---	10.3	---	13.3	5.2	18.5	4.49
Ti	---	-3.438	---	1.485	2.87	4.35	6.09
Zr	---	7.16	---	6.44	0.02	6.46	0.185

Sample environment use at SPODI

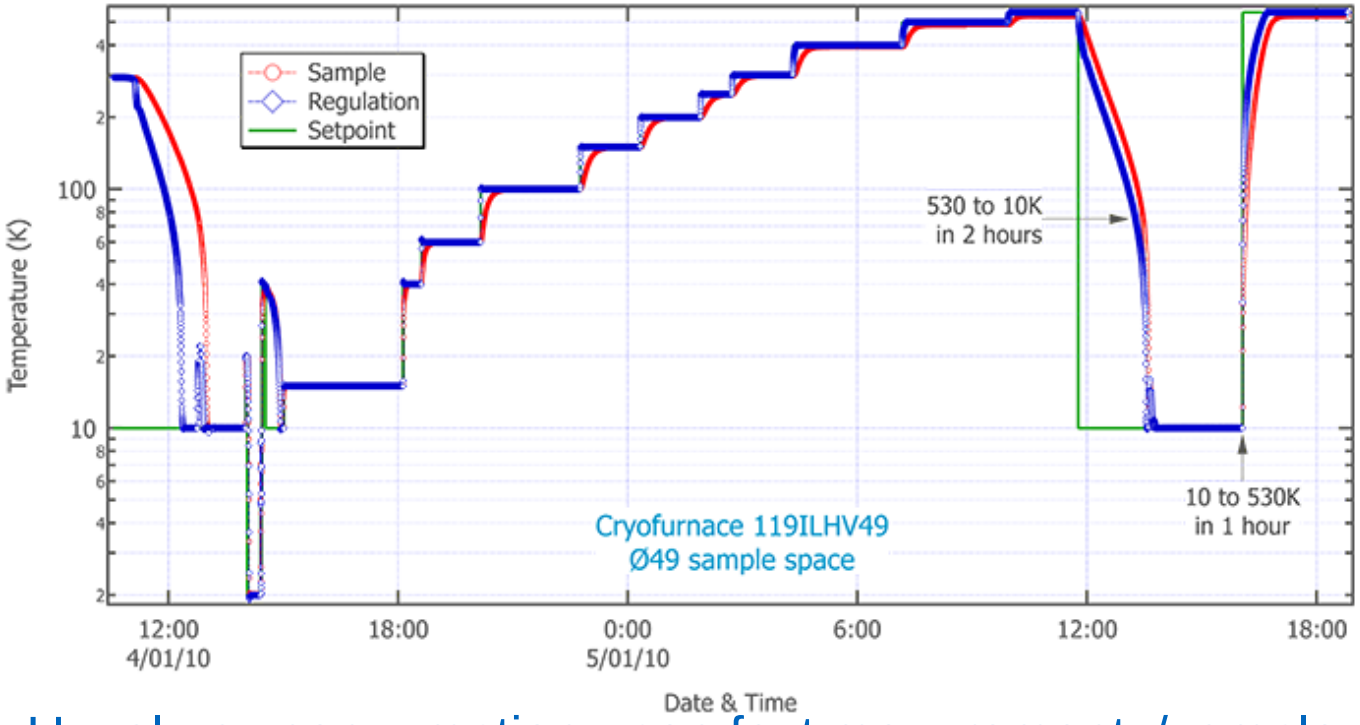


Ideal cryostat for neutron powder diffraction

(c) ISIS

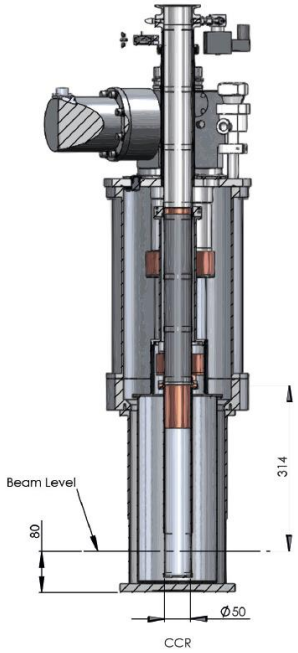
Development: 1975 @ ILL (cryofurnace 1984)
Commercialized by AS Scientific Products

Typical performance curve of the „orange“ cryofurnace



Usual gas consumption upon fast mesurements/sample change: 200-500 L/week

Closed-cycle refrigeration/Cryocooler



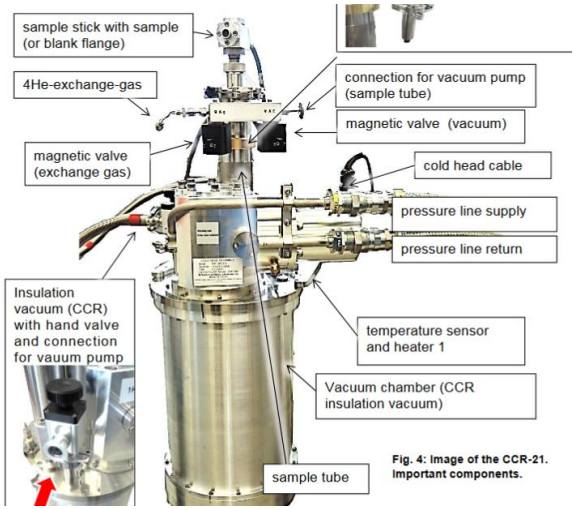
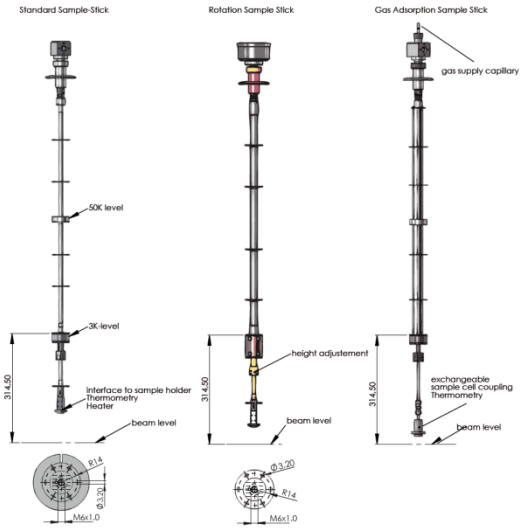
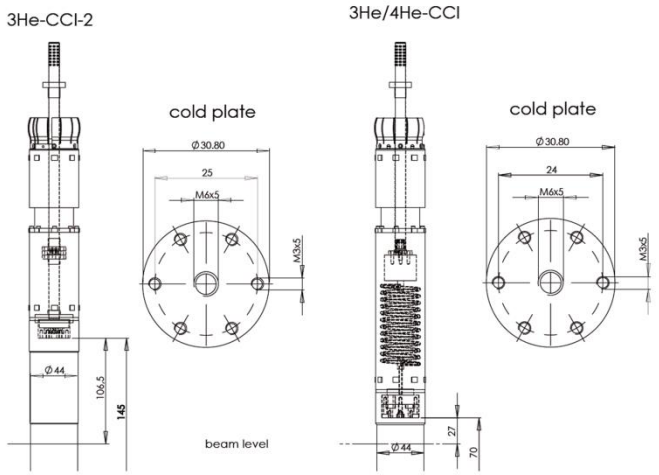


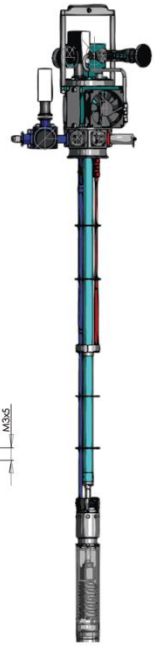
Fig. 4: Image of the CCR-21. Important components.

Temperature range (K), with exchange in sample gas	2,8 – 300 K
Cool down time of cryostat: 300 K – base temperature (h)	2,5 h
Cool down time for sample change with cryostat at base temperature (h)	< 1.5 h
Diameter of sample space (mm)	<50 or <80 mm
Height of sample space (beam window) (mm)	~75 mm

1.5 K cryostat and low temperature inserts

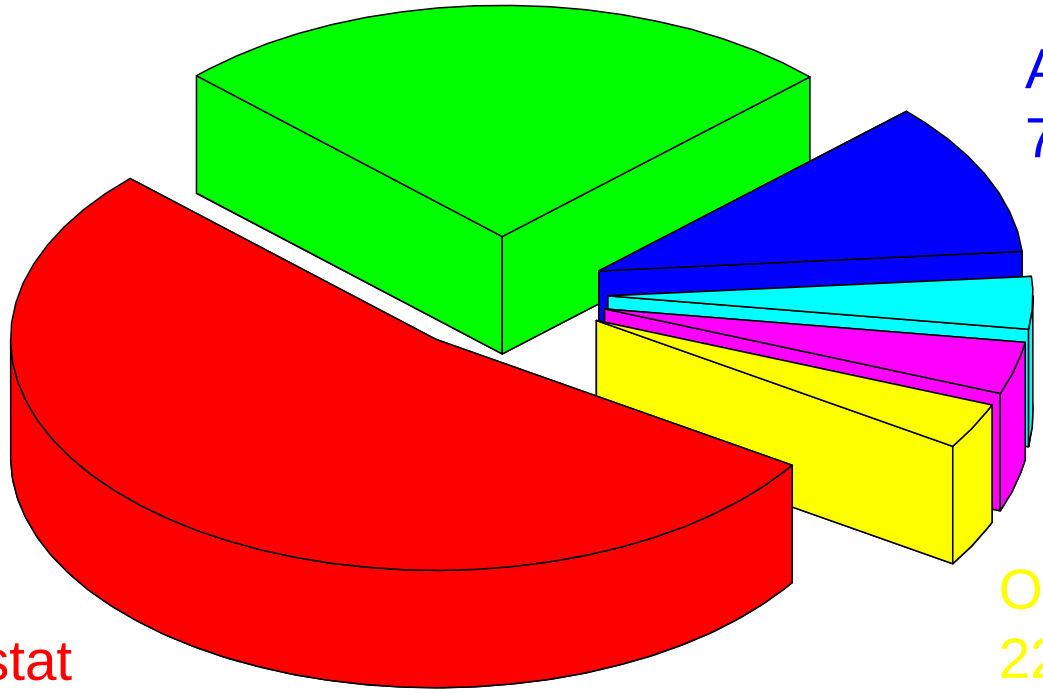






3He insert CCI – ^{3}He (K)	450 mK – 10 K
Dilution insert CCI – ^{3}He/^{4}He (K)	50 mK – 1 K
Extended temperature range (K) stick	<600 K
Rotation sample stick	Present
Gas adsorption stick	Present (10 bar)
High voltage stick	<2000 V
High pressure sample stick for gas pressure cells	4 kbar

Sample environment use at SPODI



ILL-type „blue“ furnace

Water cooled Al – body with Nb heating elements

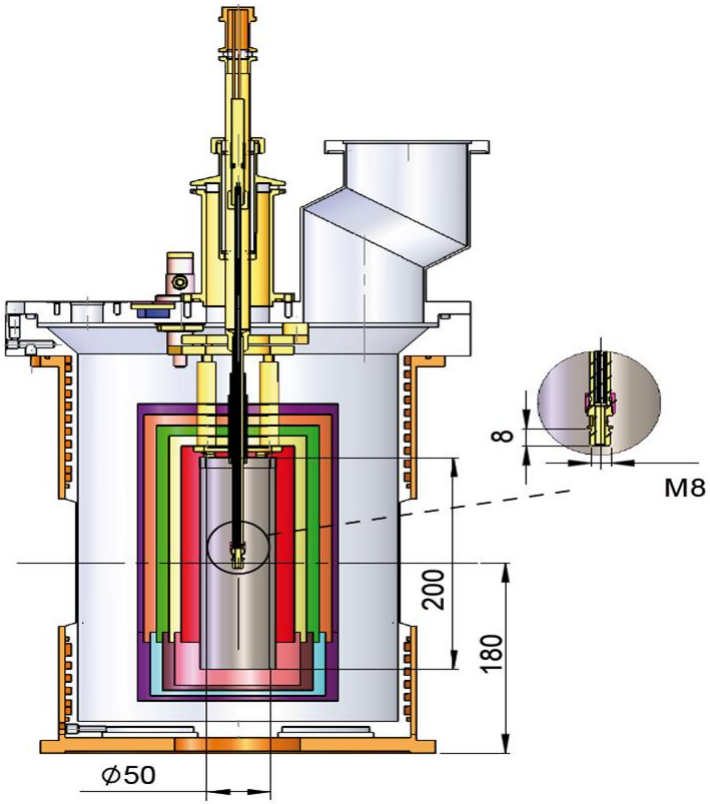
Temperature range: <1100 or <1800ºC
Sample dimensions: Ø 4.5 cm
10 cm high (max)

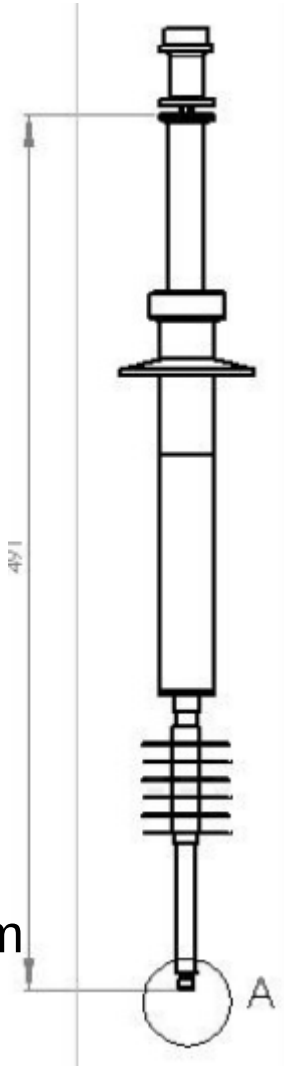
Heater input: 0-15 volts
0-400 Amps (max)
2000 VA

Vacuum: 10^{-5} - 10^{-6} mbar
inert gas atmosphere

Neutron access: 360º horizontal
±20º out of scattering plane.

High temperature vacuum furnace – FRM design

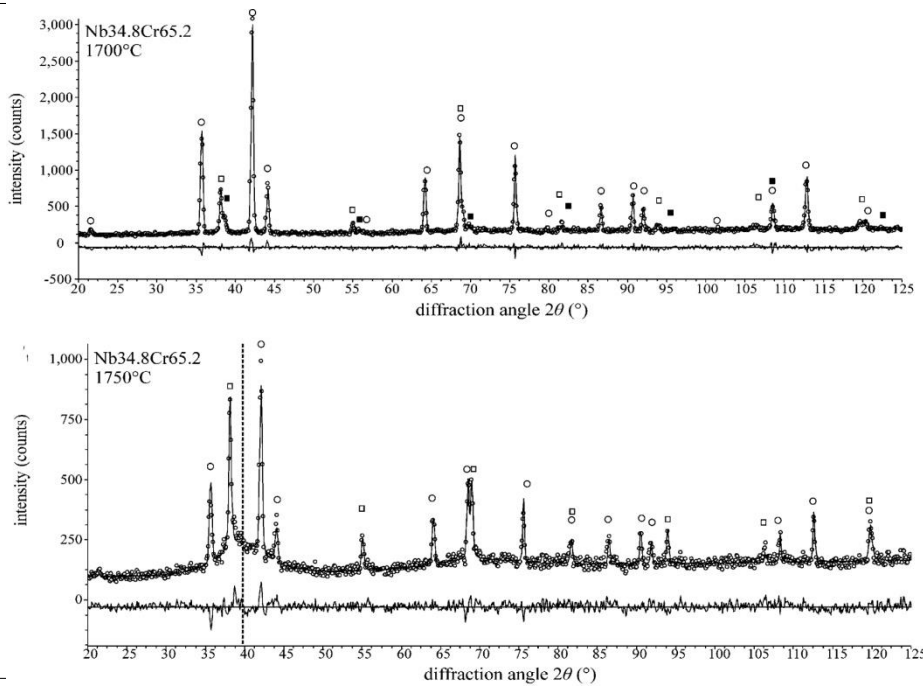




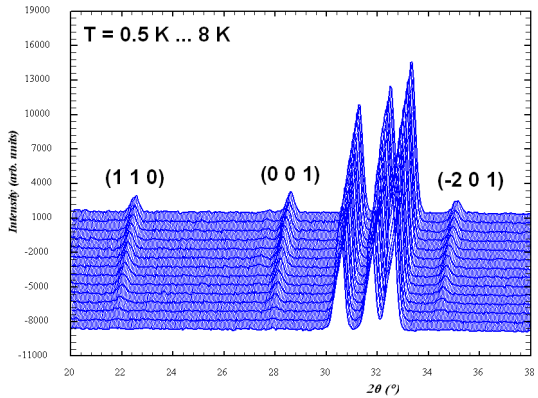
Temperature range RT - 1900°C
With exchange gas RT- 900°C
Diameter of sample space: 45 mm
Height of sample space: 100 mm

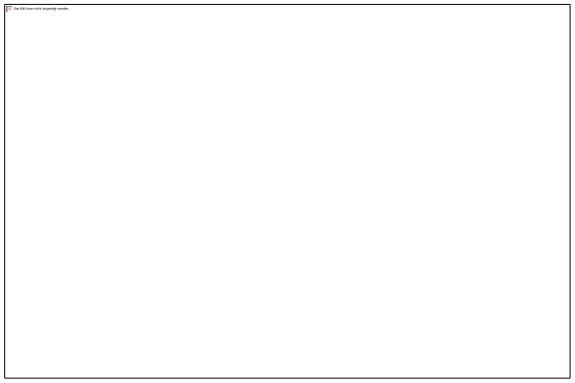
HT studies of $NbCr_2$ Laves phase

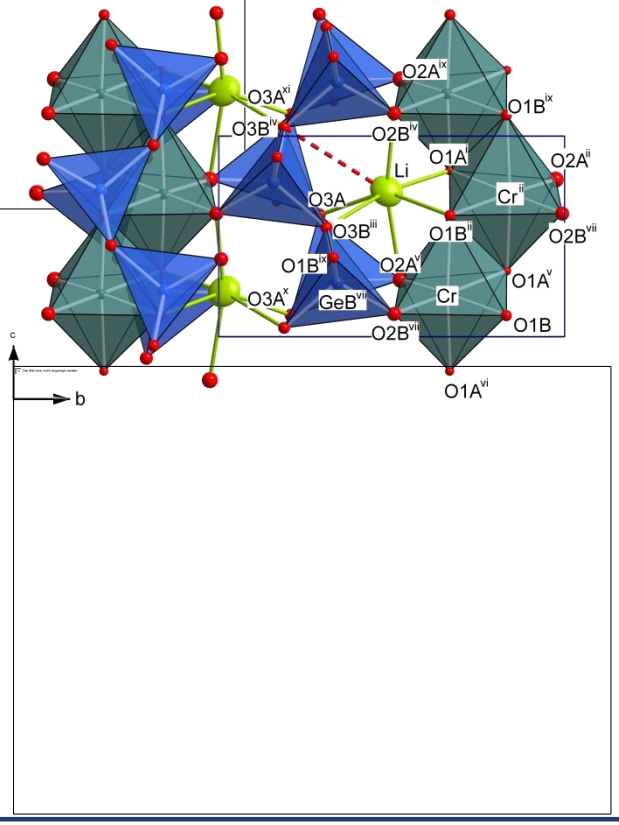
The polytypic phase transformations between the different Laves-phase structures C14 (hexagonal), C36 (di-hexagonal) and C15 (cubic) have been reported.



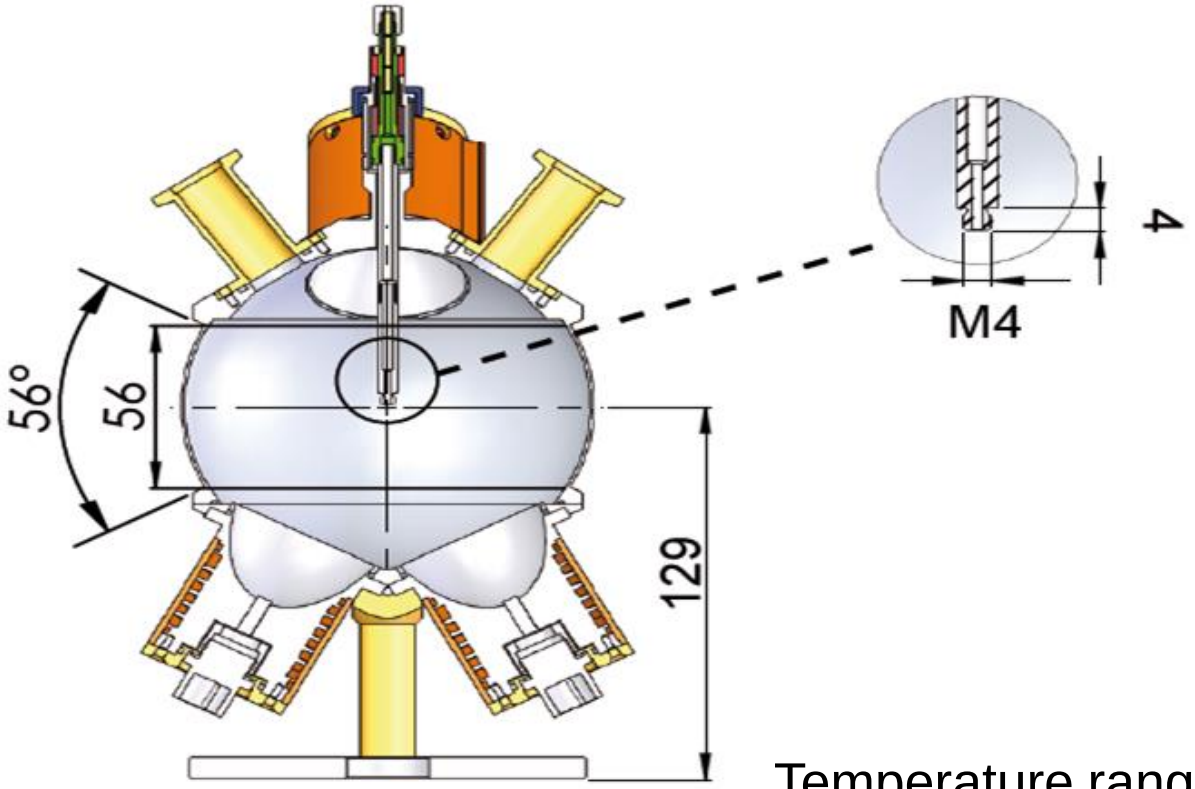
J. Aufrecht, A. Leineweber, A. Senyshyn, E.J. Mittemeijer, / Scripta Materialia 62 (2010) 227–230


T = 0.5 K ... 8 K
(1 1 0)
(0 0 1)
(-2 0 1)
Intensity (arb. units)
2θ (°)




O2A^ix
O3A^xi
O1B^ix
O3B^iv
O2B^iv
O1A^i
O2A^ii
Li
Cr^ii
O3A
O3B^iii
O1B^ii
O2B^vii
O1B^ix
O2A^v
O1A^v
O3A^x
GeB^vii
Cr
O2B^vii
O1B
O1A^vi
c
b

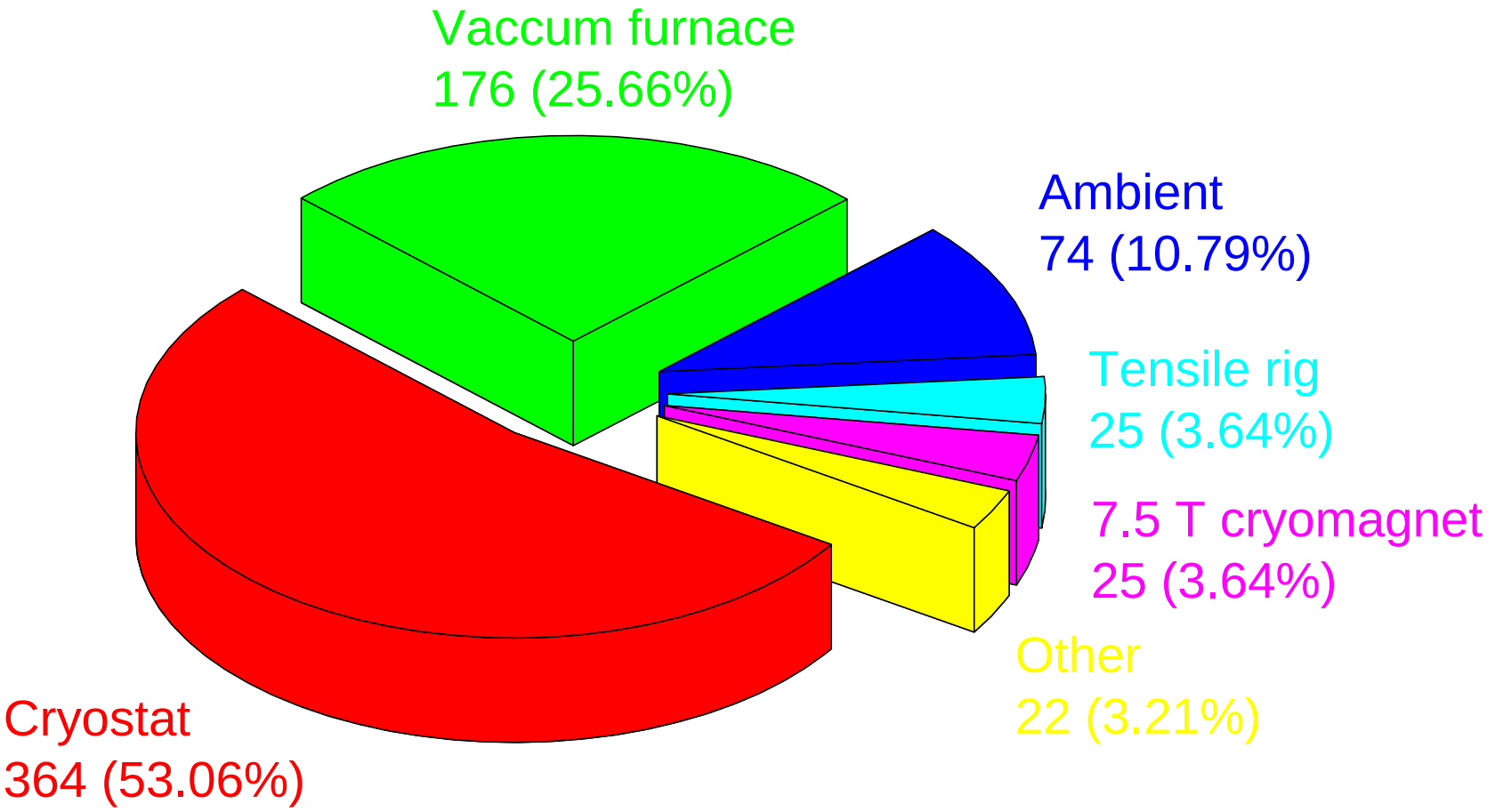
Infrared light furnace (IRF)



"The sample (a few mm³ in volume) is placed at the focus of four halide lamps. Measurements can be performed in vacuum or inert/exchange gas atmosphere. Studies on air lead to the partial oxidation of Al mirrors."

Temperature range in vacuum: RT–1200 C
Temperature range in exchange gas: RT–300C
Diameter of sample space: 10 mm
Total height of sample space: 15 mm

Sample environment use at SPODI



High pressure equipment for diffraction

Piston cylinder

Paris-Edinburgh

Diamond anvil cell

Pressure range	<0.5-2 GPa	<10-24 GPa	<94 Gpa*
Angle limitations	Low	Medium	Medium
Sample amount	Large	Medium	Low
Type of sample	Any	Any	Simple*

**According to SNAP@ORNL*

An instrument around sample environment

The multi anvil press SAPHIR at FRM II

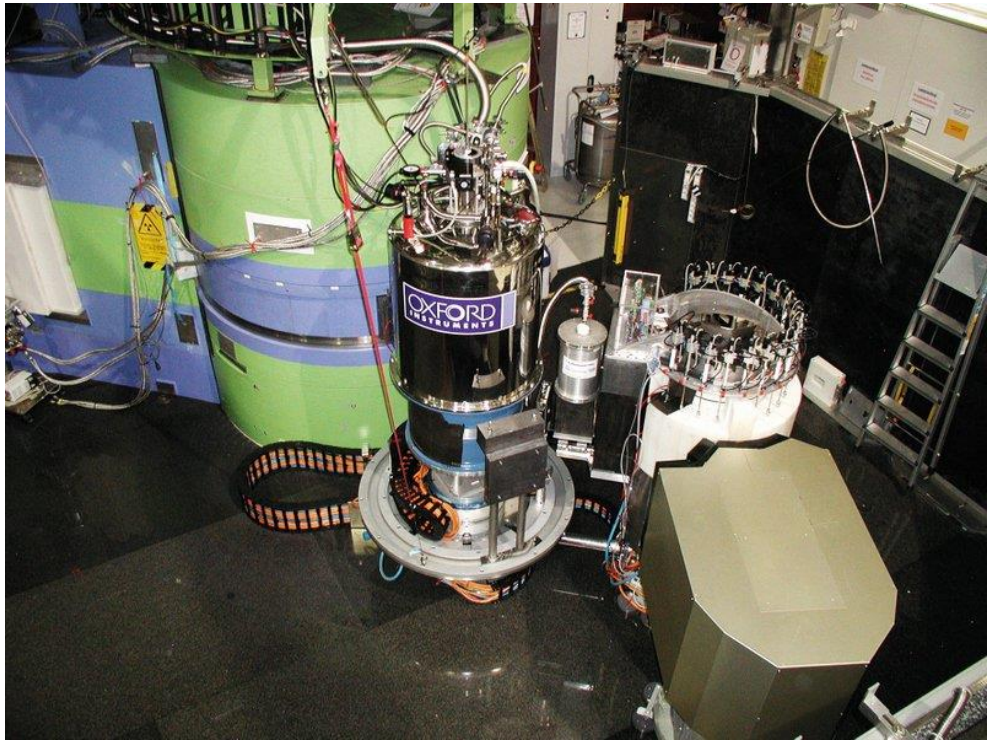
Pressing force <23.5 MN (2400 tons)
Nominal pressure: 15 (23) Gpa
Accessible temperatures: <2000C
Sample volume: >20 mm^3
Time-of-flight neutron diffraction
Neutron radiography: L/D>500

HFM/EXED - High Magnetic Field Facility for Neutron Scattering

Central field: >25 T
Opening angle: 30 deg
Field homogeneity: <0.5%
∅ 20 mm, height 20 mm
Operating current: 20 kA
Power Resistive insert: 4.4 MW

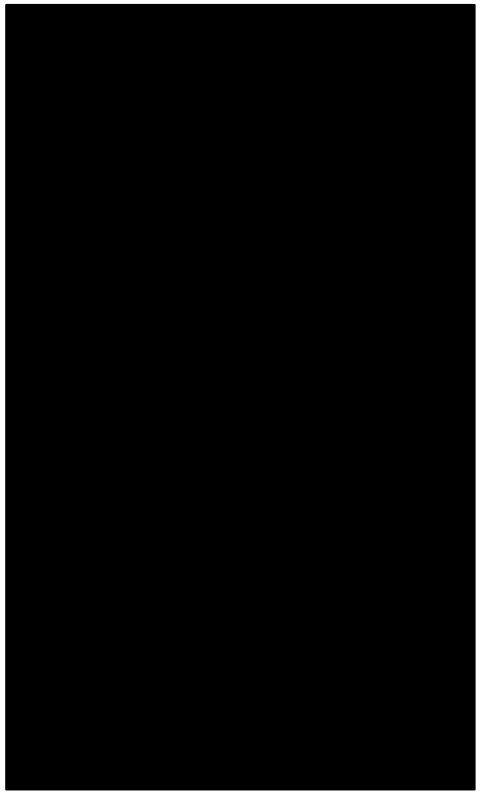
High magnetic fields

MAG-V-15T at PANDA spectrometer



Maximum magnetic field (T):
± 13.2 (14.5) (symm.)
Homogenity of the magnetic field:
<0.85 %
∅ 12 mm, height 20 mm
Bores: LT bore: Ø 20 mm
Beam window dimensions
vertical gap (mm): 20
scattering angle horizontal: 320˚
Cooling system: LHE
Additional sample environment available: dilution insert

High magnetic fields

	MAG-2.2T-HTS	MAG-V-7.5T	
			
Features	Max field 2.2 T No Al in a beam 150 deg. access	Max field 5.5 T RT bore 150 deg. access	Max field 8.0 T Compensated, LHe 160x35 deg. access

Pulsed magnetic fields for neutron diffraction

LNCMI
Laboratoire National des Champs Magnétiques Intenses - Toulouse

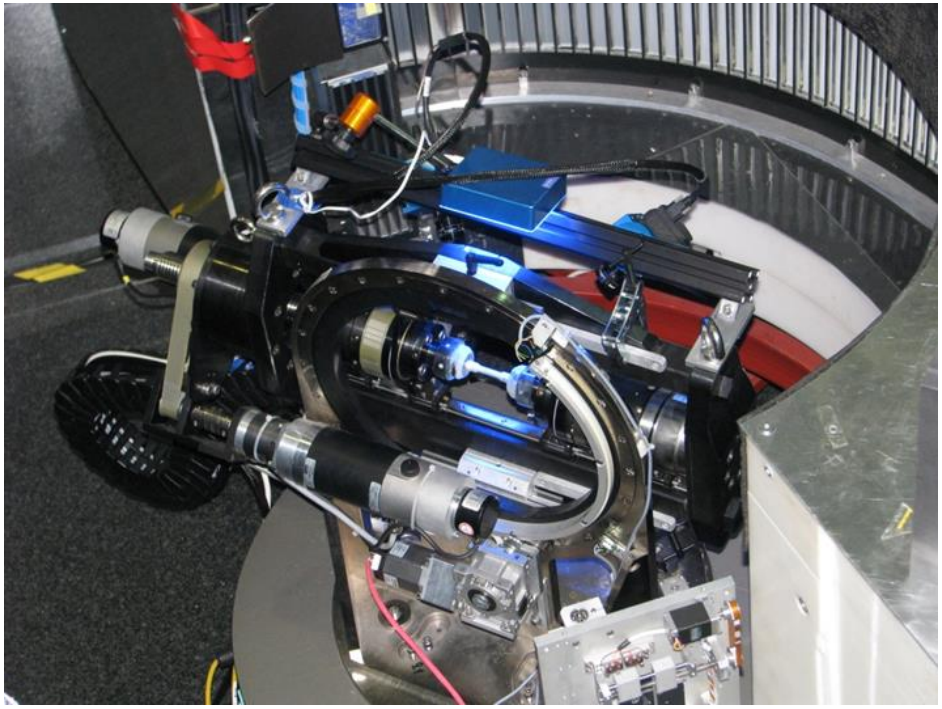
Photo of the 40 Tesla pulsed field magnet on IN22

Project Magfins funded by „ Agence National de la Recherche“

horizontal field up to 40 T with the 1.15 MJ transportable pulsed field power supply

Cryostat ILL: minimum temperature 2 K

Application of tensile rigs in diffraction



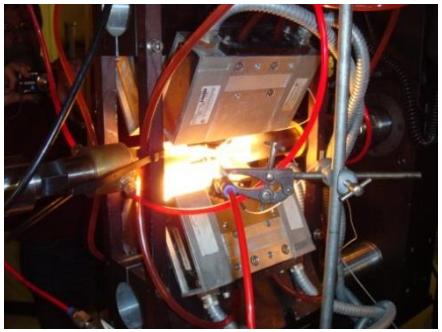
load axis: unique Eulerian cradle, i.e. movement with respect to incident beam

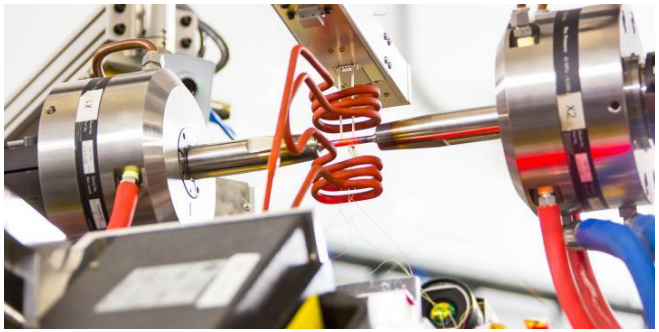
tensile/compressive stress: 50 kN
torsion :100 Nm

Camera based system for contact-free Extension recording

advantages of neutrons
for course grained samples

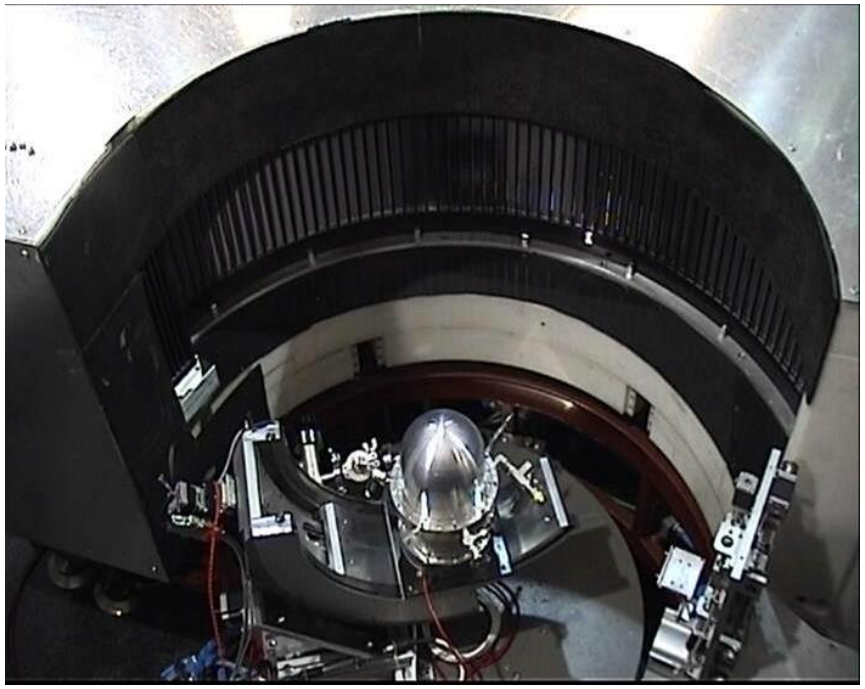
M. Hoelzel, W.M. Gan, M. Hofmann et al., Nucl. Instr. Meth. A 711 (2013)101-105

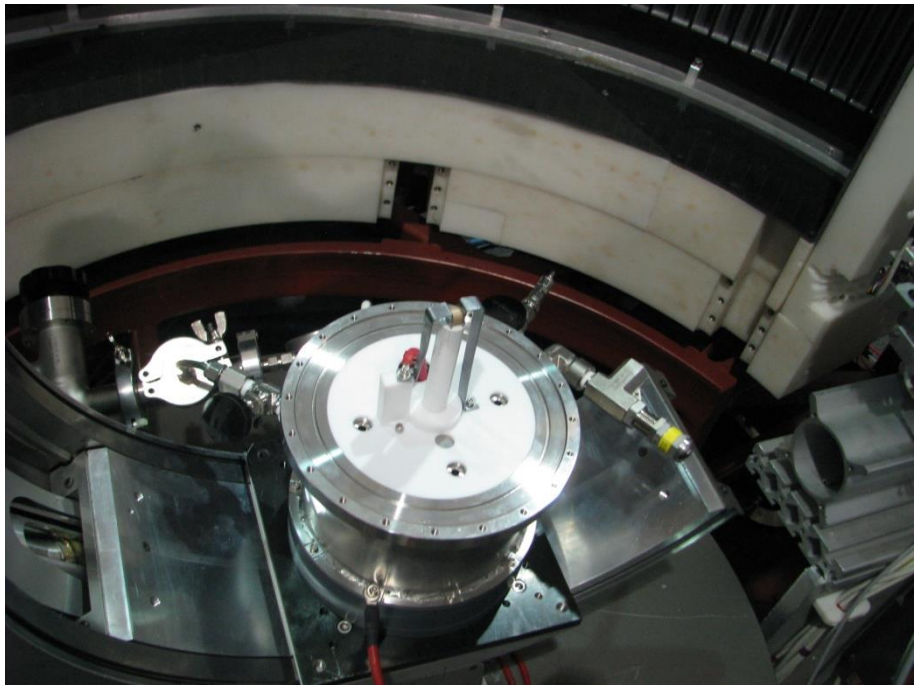




Use of resitive/inductive heating or radial furnaces may extend the T range to 1200 K

High electric fields



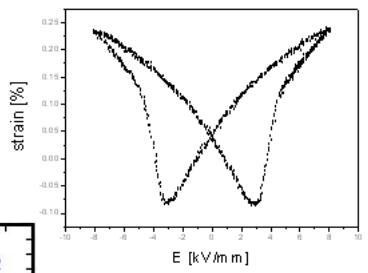


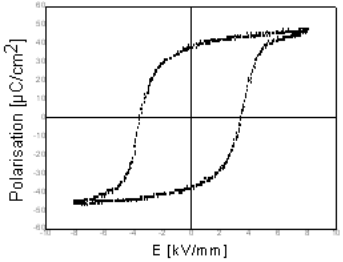
sample cell with SF_6 atmosphere
orientation of electric field with respect to incident beam

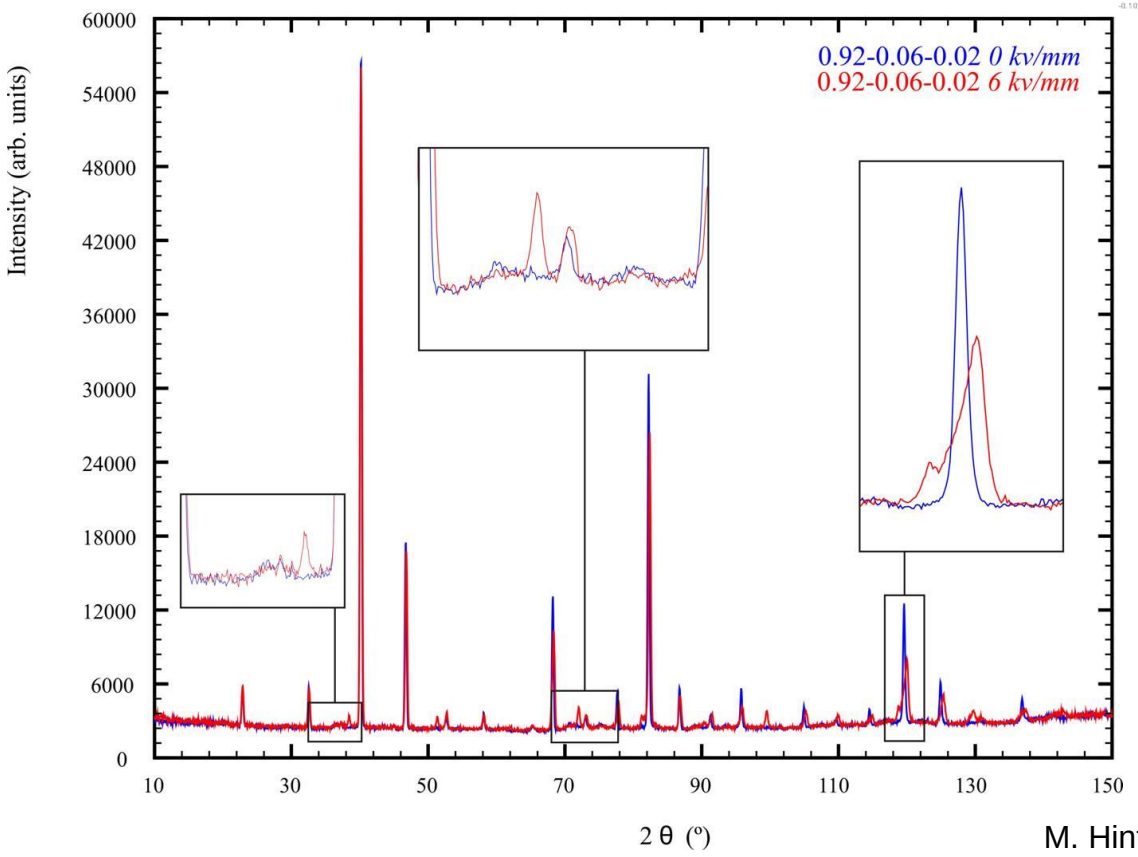
PZT bulk sample (7 mm): 2 kV/mm → 14 kV
BNT based bulk sample (6 mm): 6 kV/mm → 35 kV

Domain switching phenomena in modern ferroelectrics

$Bi_{1/2}Na_{1/2}TiO_3$-$BaTiO_3$-$K_{1/2}Na_{1/2}NbO_3$: 92-6-2
high reversible field-induced strain ≈ 0.45%
→ candidate for actuator applications







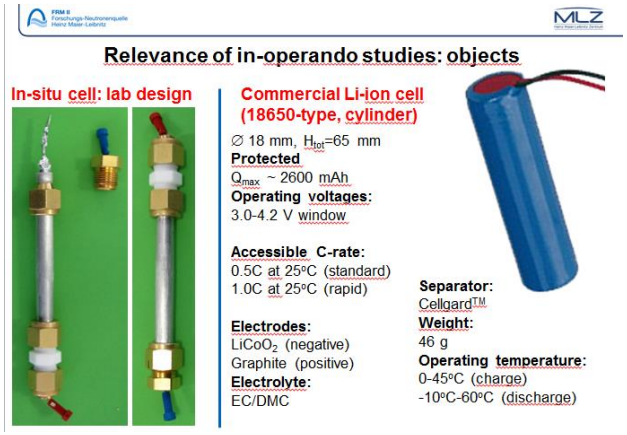
Superlattice reflections not visible by lab XRD and synchrotron

Electrical field induced tetragonal to rhombohedral phase transition - reason for high field-induced strain

M. Hinterstein et al., J. Appl. Cryst. 43, 1314-1321 (2010)

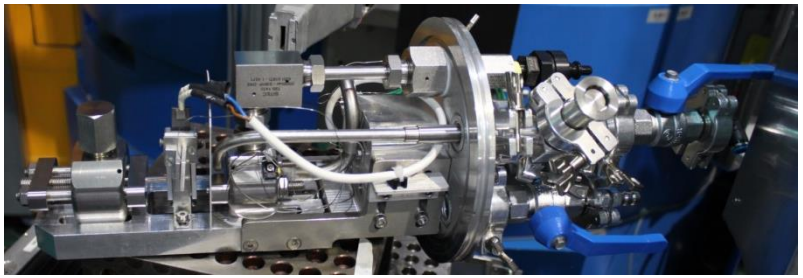
Diverse applications

Li-ion batteries

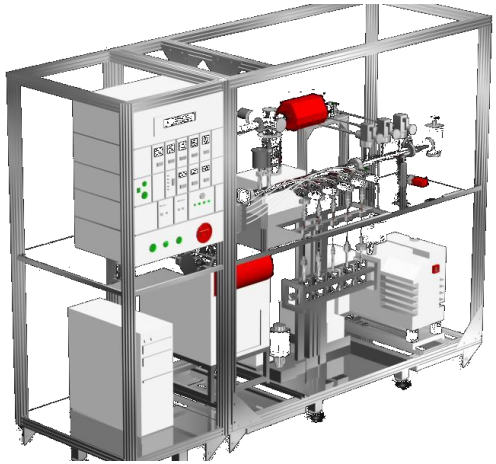


Hydrogen storage

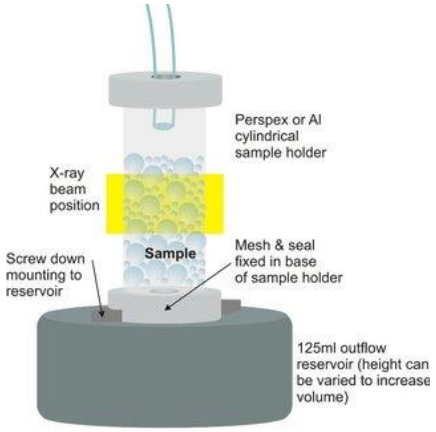
Chemisorption at 800C and 700 bar gas pressures (HZG)



Catalysis



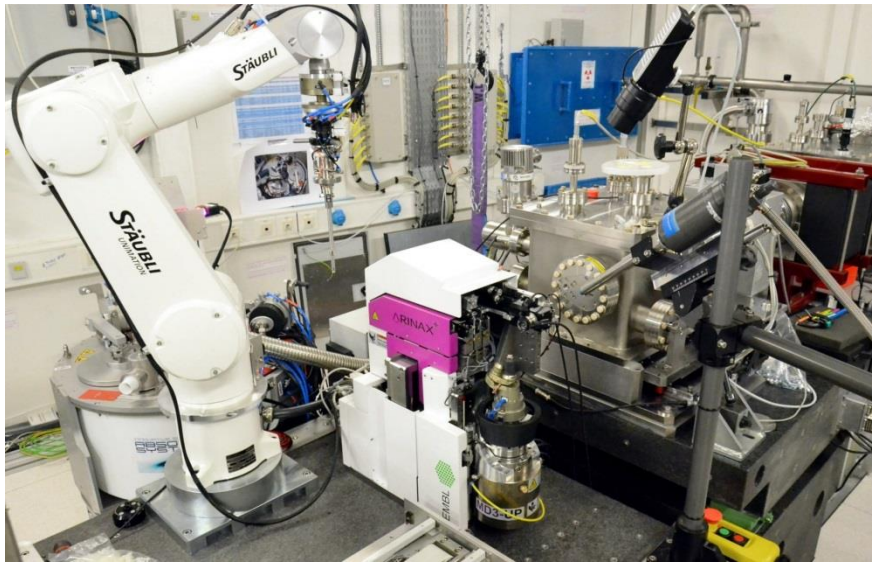
Ion exchange



Use of robotics in diffraction – sample change

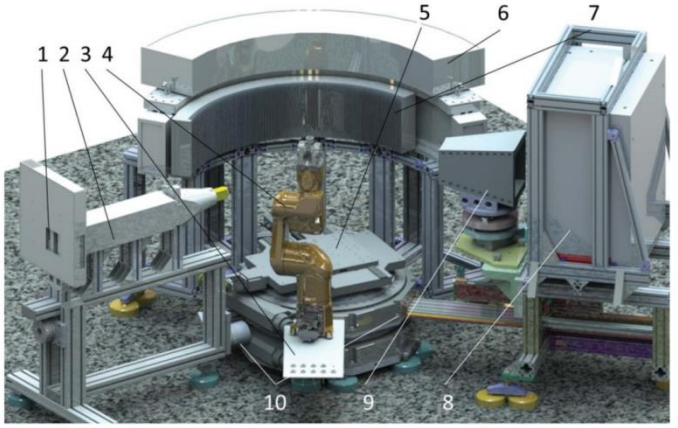
Serial crystallography

Exp. Layout of macromolecular beamline ID23@ESRF

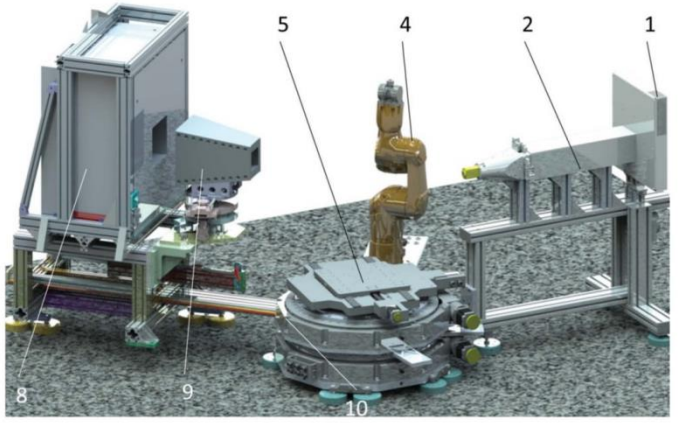


ECHIDNA

Robot at ERWIN

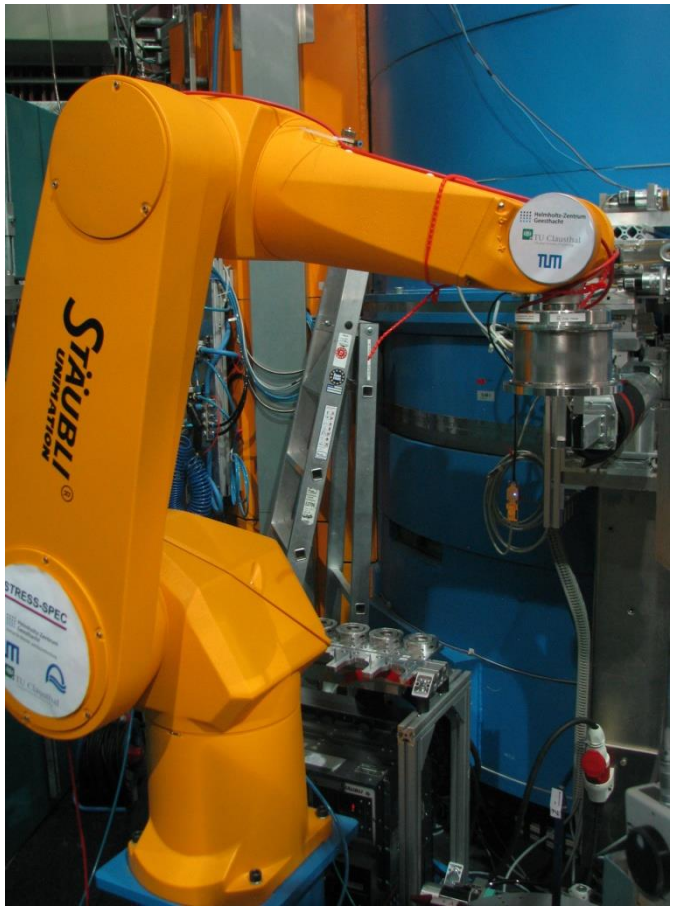


(a)

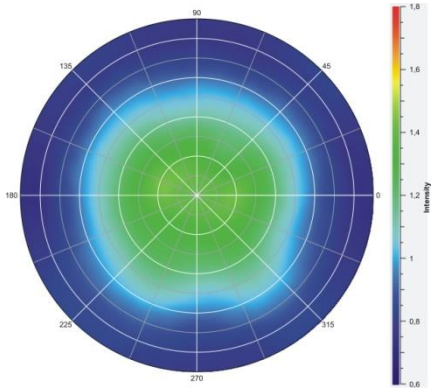


(b)

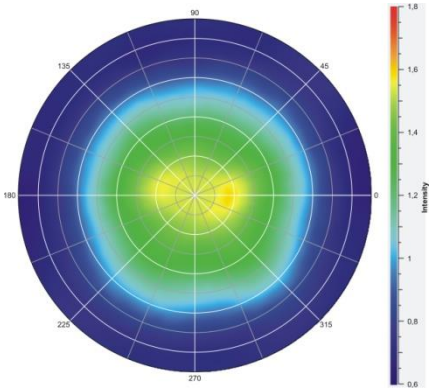
Domain orientation in actuator – pole figures under electric fields at STRESS-SPEC (FRM II)



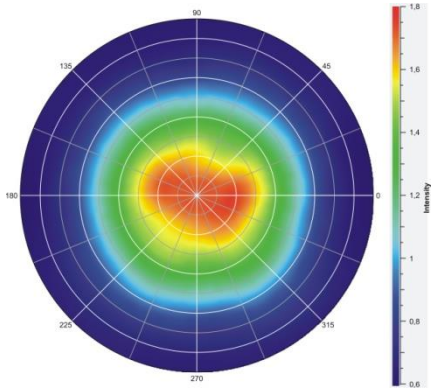
(002) pole figures



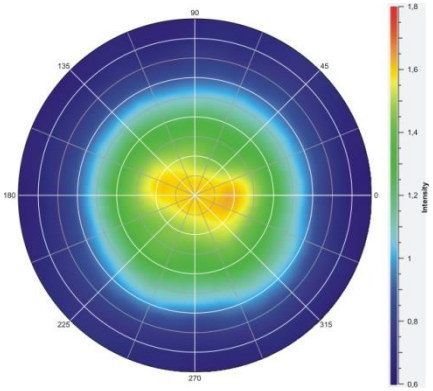
initial



60 V / 1 kV/mm (up)



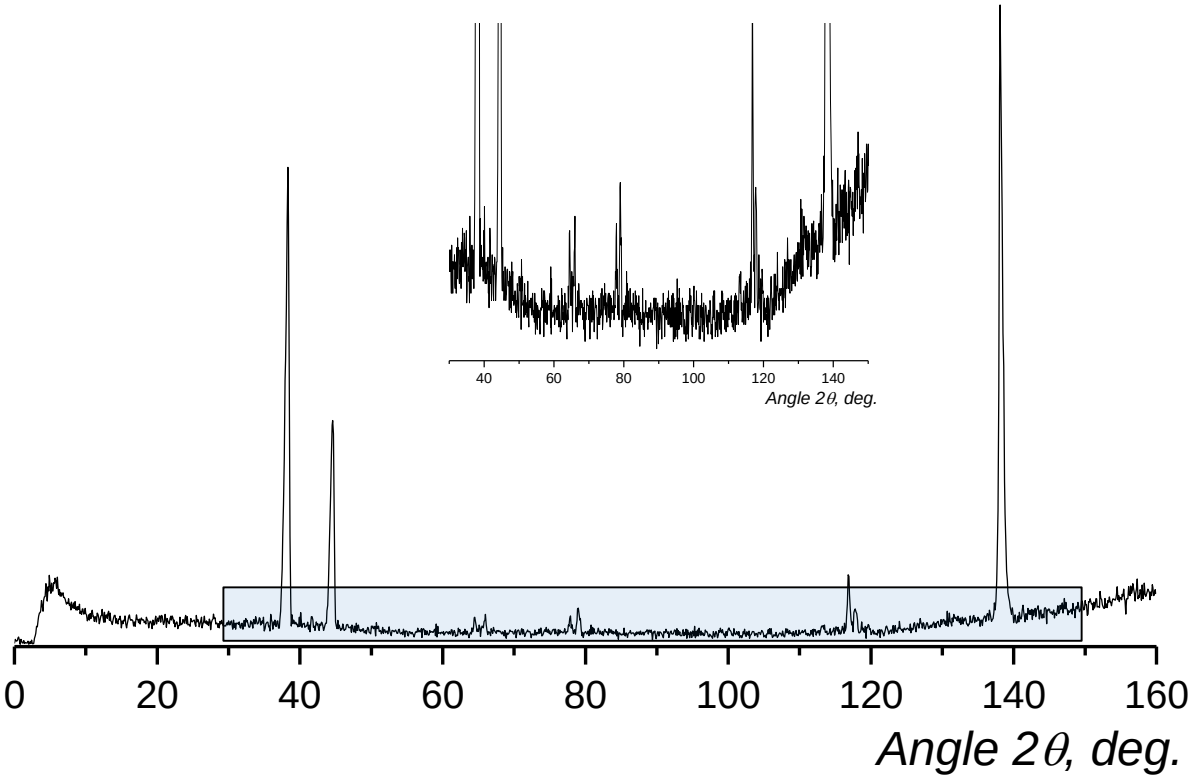
120 V / 2 kV/mm

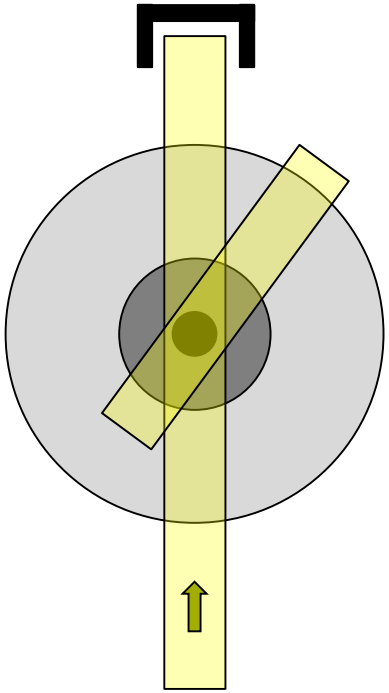


60 V / 1 kV/mm (down)

→*Pole figures reflect orientation of domains, forming a fibre texture*

Eliminating the diffraction contribution from the sample environment





Use of suitable window material: vanadium, sapphire etc.

Summary

- Diffraction techniques (especially powder diffraction) are highly demanding to the quality and quantity/„diversity“ sample environment

- Splitting of the sample environment to the „instrument“ relevant and „pool“ relevant equipment: working horses and racing horses

- Routine operation requires a robust instrumentation and well defined/stable experimental state and sample conditions

- Friendly use: ease of connection, installation, calibration and operation

- Dedicated personal/professional staff, sufficient lab space

Thank you.