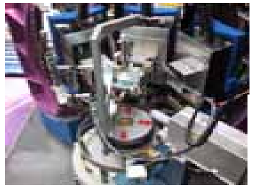
Diffraction Instrumentation at SINQ

- HRPT
  Thermal, High Resolution Powder Diffractometer

- DMC
  Cold, High Intensity Powder Diffractometer

- TriCS
  Thermal Single Crystal Diffractometer



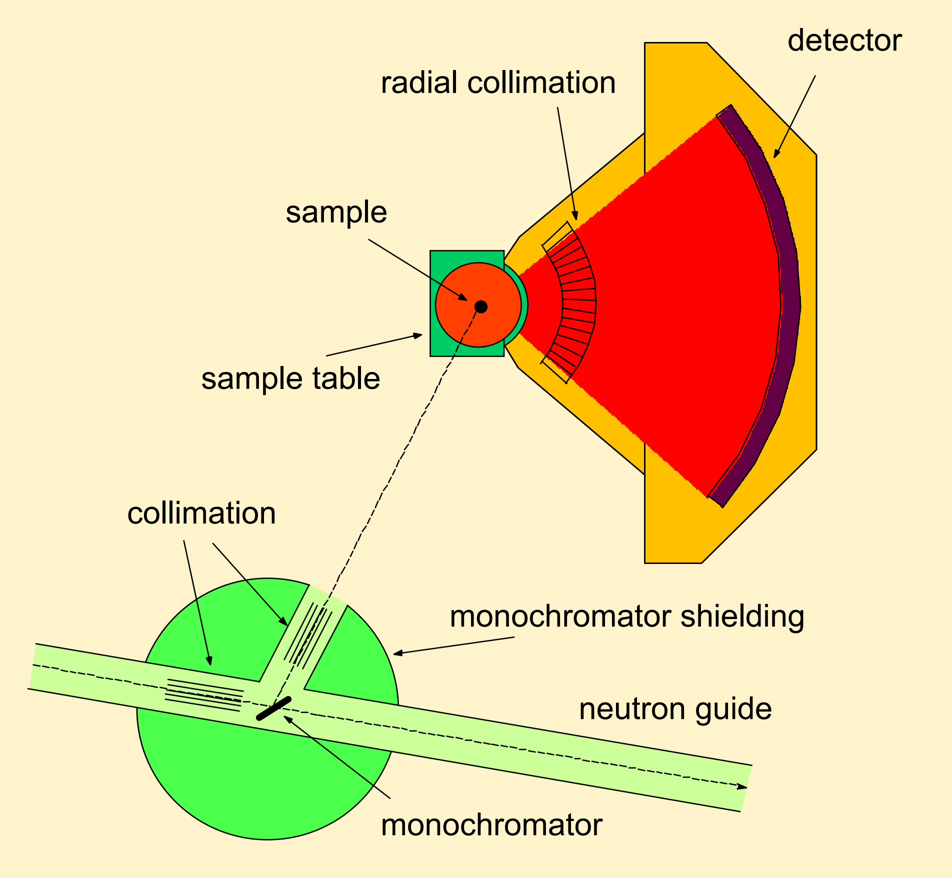
- Orion
  Test Diffractometer

- Projects (Panteher,DMC-2, Laue)

Update from Version 2005, work in progress

DMC - Cold Neutron Powder Diffractometer

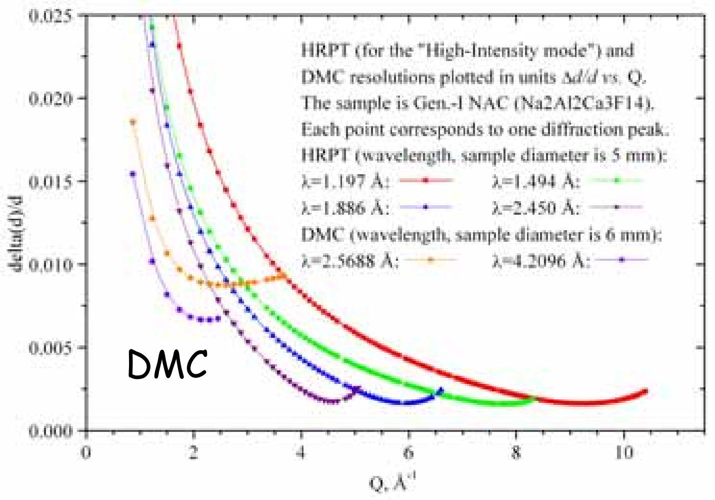
Lukas Keller, Aziz Daoud-Alladine



- position:
  guide hall, at the cold neutron guide RNR12
- supermirrorguide, m=2
- monochromators:
  PG 002, Ge hkk (optional)
  (vertically focusing)
- wavelengths:
  2.3 to 6 Å

Update from Version 2005, work in progress

DMC - Cold Neutron Powder Diffractometer



DMC: high resolution for low Q range

- detector:
  "banana" type multidetector, 400 detectors with angular separation of 0.2 resolution distribution is complementary to HRPT:
  efficiency: 44%@2.56Å, 61%@4.2Å

- oscillating radial collimator between sample and detector suppresses scattering from sample environment

New installation 2004:
Ge_{311} monochromator for higher resolution

Sample environment

- temperature range: 120 mK to 1400 K ($^3He/^4He$ dilution refrigerator, He cryostats, cooling machines, furnaces)
- field range: up to 4 Tesla (vertically), 1.8 Tesla (horizontally) at 1.5 to 300 K (cryomagnets)
- pressure range: up to 15 kbar at 2K ≤ 300 K (clamp pressure cells)

Update from Version 2005, work in progress

DMC - cold neutron powder diffractometer

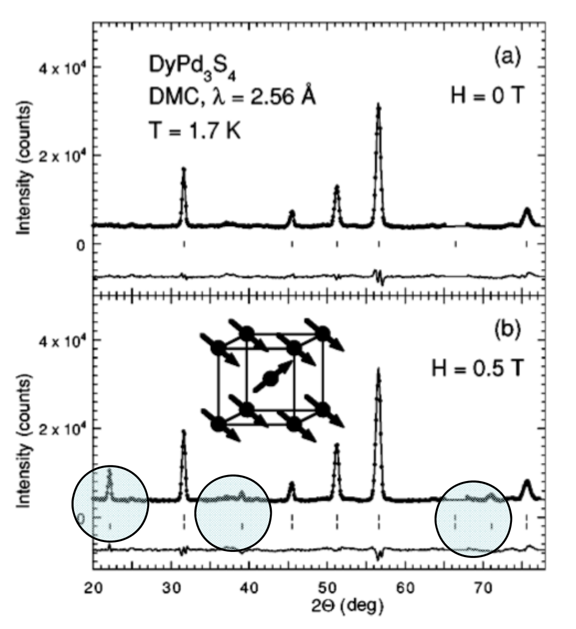
RAPID COMMUNICATIONS

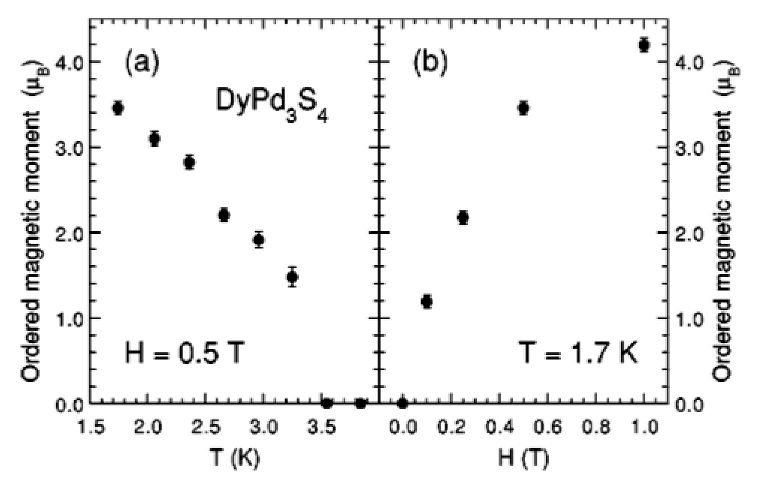
PHYSICAL REVIEW B **70**, 060407(R) (2004)

Quadrupolar and dipolar magnetic order in $DyPd_3S_4$: A neutron scattering and muon spin rotation and relaxation investigation

L. Keller,[1] V. Pomjakushin,[1] K. Conder,[1] and A. Schenck[2]
[1]*Laboratory for Neutron Scattering, ETH Zürich and PSI, CH-5232 Villigen PSI, Switzerland*
[2]*Institute for Particle Physics of ETH Zürich, CH-5232 Villigen PSI, Switzerland*

(Received 19 February 2004; published 30 August 2004)





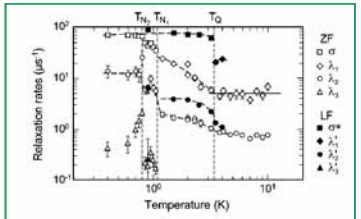


FIG. 1. Temperature dependencies of the muon spin relaxation rates. The zero (ZF) and longitudinal field (LF) experiments are shown by open and filled symbols, respectively. Anomalies at T_Q, T_{N1}, and T_{N2} are signatures of the quadrupolar and the magnetic ordering phenomena.

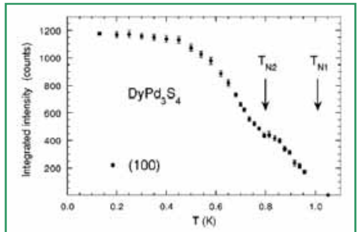


FIG. 6. Temperature dependence of the integrated intensity of the magnetic (100) peak, determined by neutron diffraction.

Canted anti-ferromagnetism
α = 91° at 1 K
α= 99° at 0.8K
system reacts to quadrupolar ordering

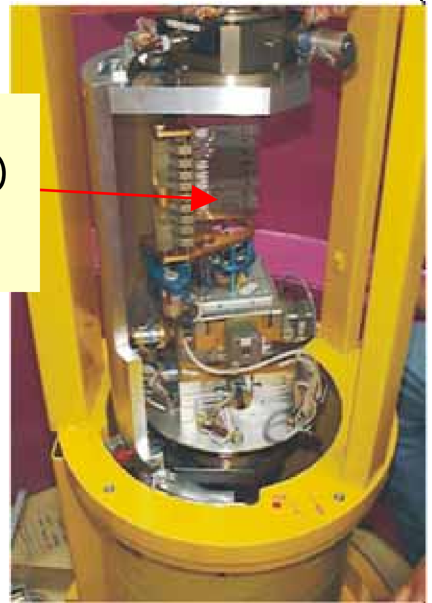
- first investigation of field induced magnetic order in the quadrupolar phase of $DyPd_3S_4$ and of its temperature and field dependence
- first investigation of spontaneous magnetic order at low temperature and its relation to the quadrupolar phase

Update from Version 2005, work in progress

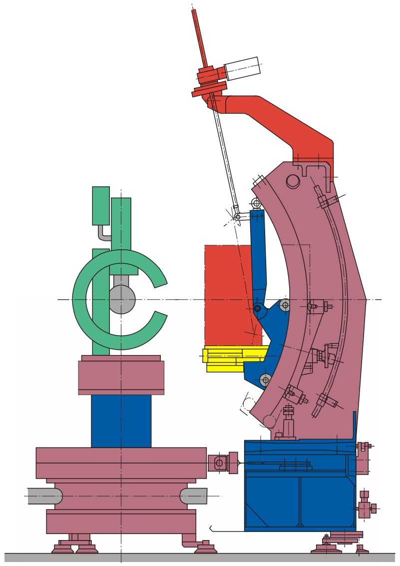
TriCS Single Crystal Diffractometer

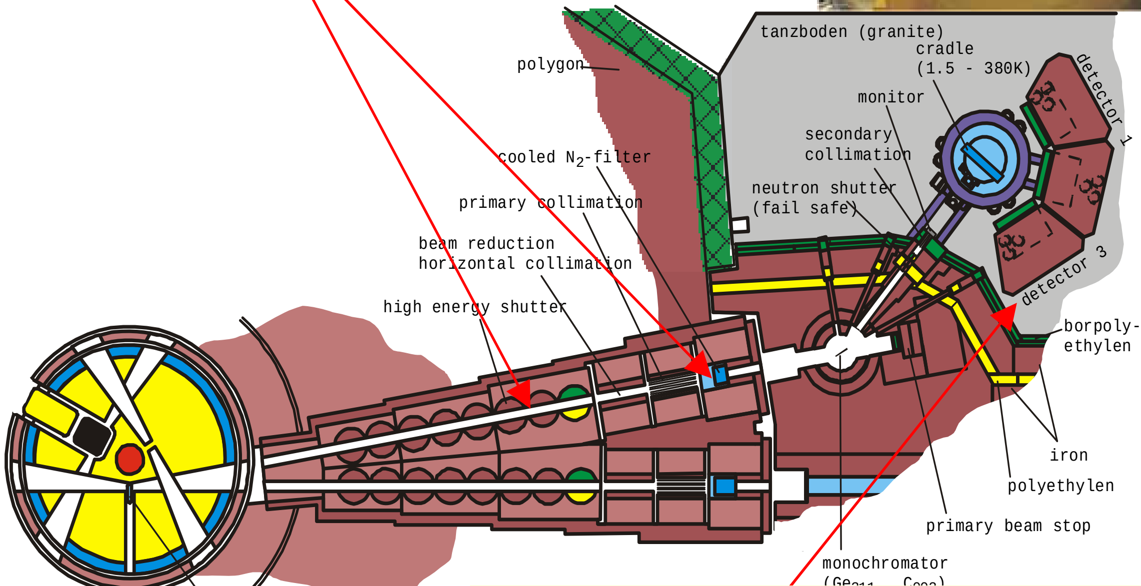
Jürg Schefer
Oksana Zaharko

Monochromator:
Ge_{311} 1.18 A (optimized for TriCS, 5·12.5cm^2,focusing)
C_{002}: 2.331 A (from Saphir, 5·4cm^2,non-focusing)



shutter
collimators
filter





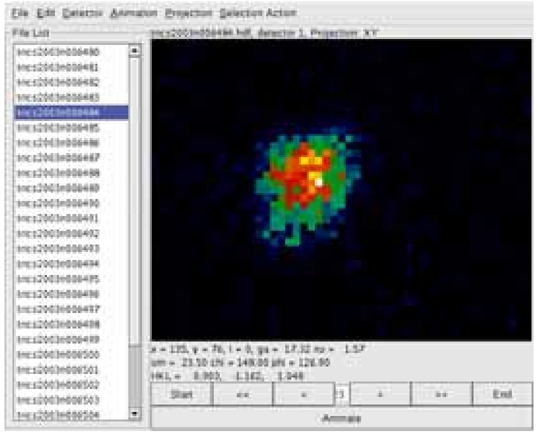
three 2D detectors,1 single detector

Kühlmashine
12-450 K

Update from Version 2005, work in progress

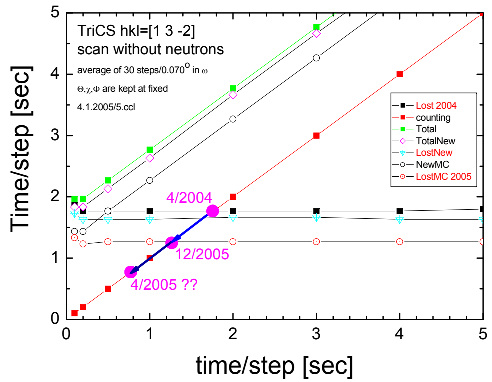
TriCS: Improvements 2004/5

1. New software package Cami4PSD



M. Könnecke, NUM Report 2004

2. Faster Scanning

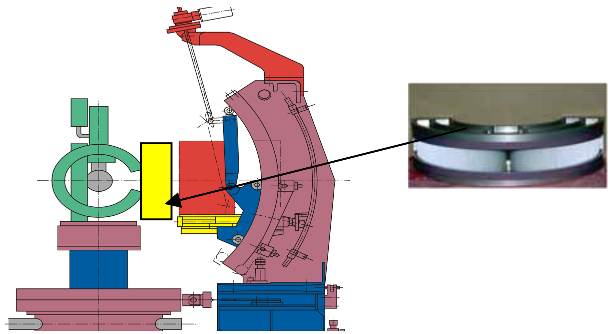


3. Closed Cylcle for 4 K

4K - 300K
Ordered 11/2004
Available Spring 2005

Old one: 12-450K
Still available

4. Radial Collimator



Update from Version 2005, work in progress

VOLUME 93, NUMBER 3 PHYSICAL REVIEW LETTERS week ending 16 JULY 2004

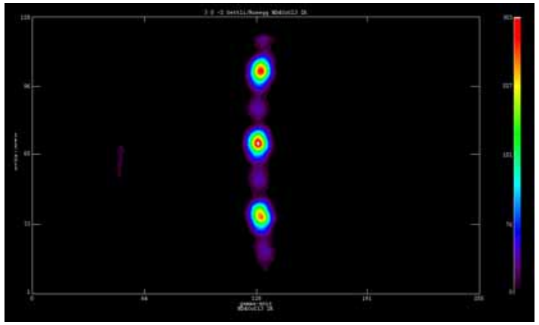
Neutron Scattering Study of the Field-Dependent Ground State and the Spin Dynamics in Spin-One-Half NH_4CuCl_3

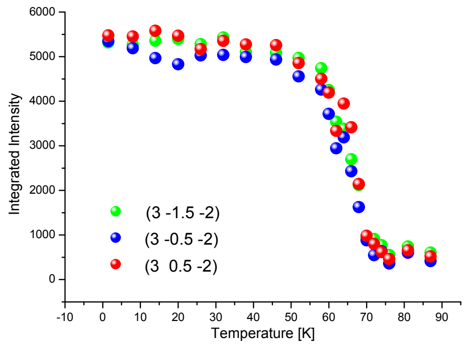
Ch. Rüegg,[1,*] M. Oettli,[1] J. Schefer,[1] O. Zaharko,[1] A. Furrer,[1] H. Tanaka,[2] K.W. Krämer,[3] H.-U. Güdel,[3] P. Vorderwisch,[4] K. Habicht,[4,5] T. Polinski,[4] and M. Meissner[4]

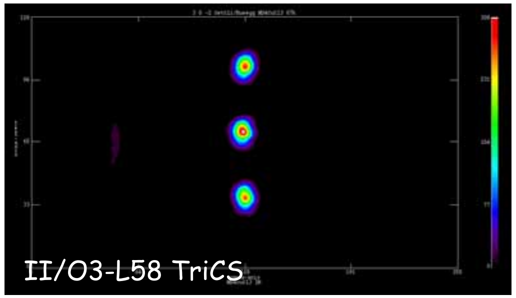
[1]Laboratory for Neutron Scattering, ETH Zurich and Paul Scherrer Institute, 5232 Villigen PSI, Switzerland
[2]Department of Physics, Tokyo Institute of Technology, 152-8551 Tokyo, Japan
[3]Department for Chemistry and Biochemistry, University of Berne, 3000 Bern 9, Switzerland
[4]BENSC, Hahn-Meitner Institute, 14109 Berlin, Germany
[5]Institute of Solid State Physics, Technical University of Darmstadt, 64289 Darmstadt, Germany

(Received 27 April 2004; published 16 July 2004)

Elastic and inelastic neutron scattering experiments have been performed on the dimer spin system NH_4CuCl_3, which shows plateaus in the magnetization curve at $m = 1/4$ and $m = 3/4$ of the saturation value. Two structural phase transitions at $T_1 \approx 156$ K and at $T_2 = 70$ K lead to a doubling of the crystallographic unit cell along the b direction and as a consequence a segregation into different dimer subsystems. Long-range magnetic ordering is reported below $T_N = 1.3$ K. The magnetic field dependence of the excitation spectrum identifies successive quantum phase transitions of the dimer subsystems as the driving mechanism for the unconventional magnetization process in agreement with a recent theoretical model.







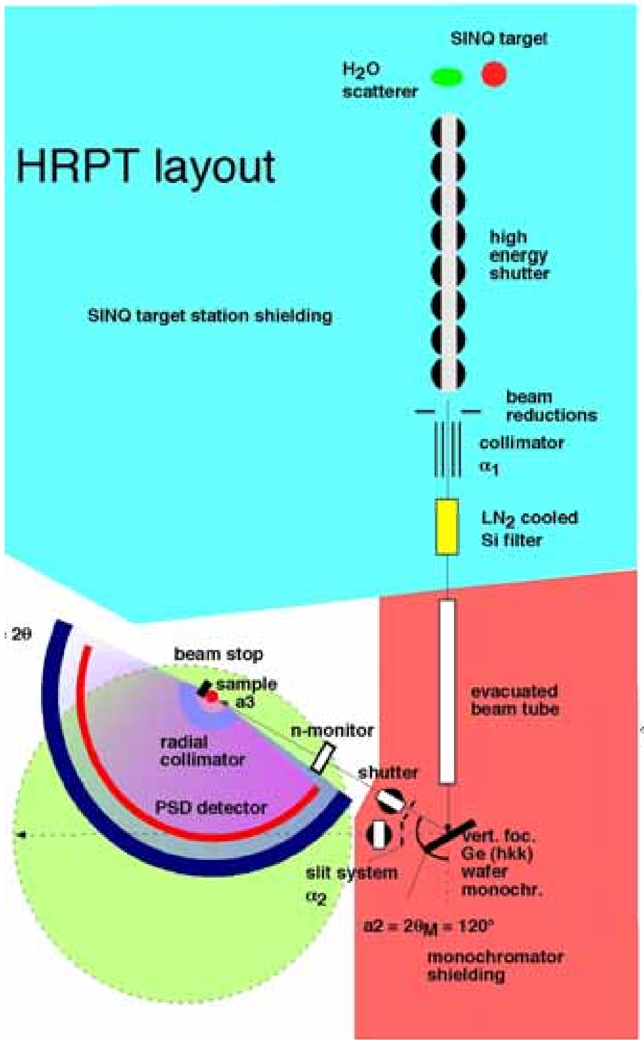
theory predictions:
segregation into different magnetic subsystems with fractions of 25%, 50% and 25% [Matsumoto et al., PRB **68** (2003) 18040]
Model needs doubling of the unit cell

Update from Version 2005, work in progress

TriCS measurement: **b is doubled**

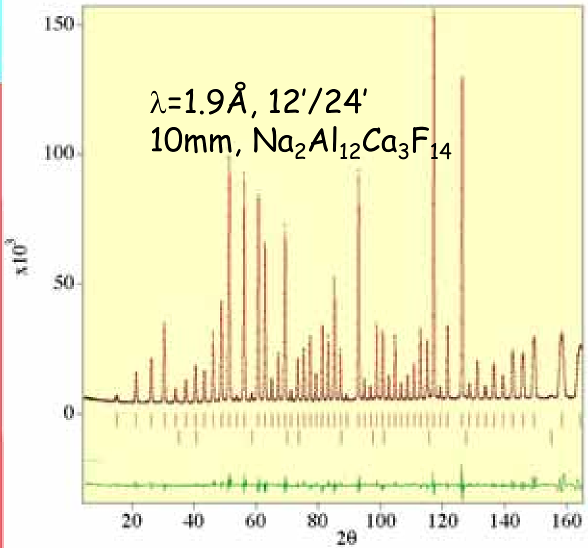
HRPT - High Resolution Powder Diffractometer for Thermal Neutrons

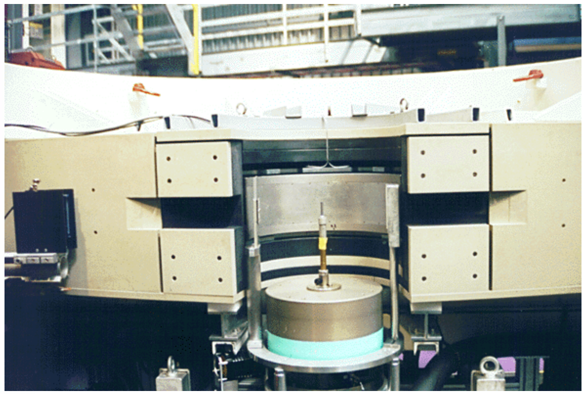
V.Pomjakushin, D.Sheptyakov



HRPT features

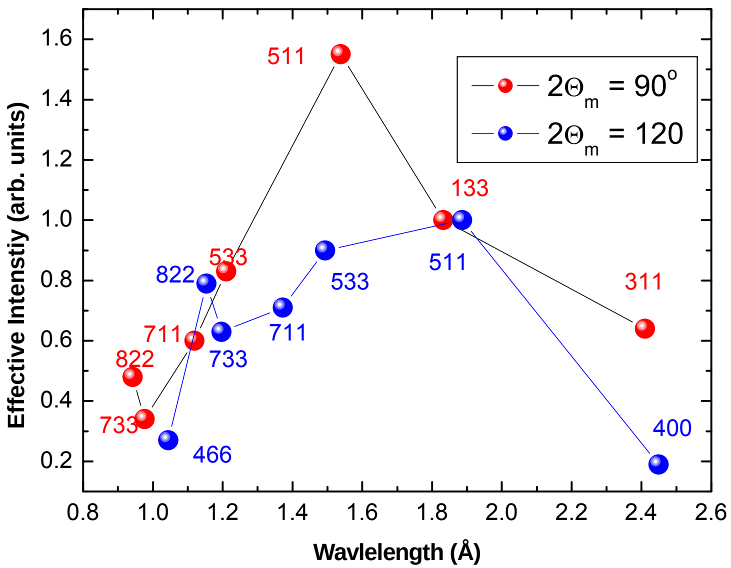
- Thermal neutrons (0.9-2.5) Å
- 2θ<165°→high Q ≤ 13 $Å^{-1}$
- High resolution δd/d =10^{-3}
- 1600-^{3}He detectors (70% efficiency @1.5Å) with angular separation 0.1°
- Flexible wavelength, resolution/intensity





HRPT - High Resolution Powder Diffractometer for Thermal Neutrons

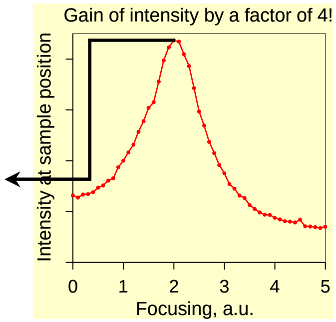
Flexible choice of wavelength



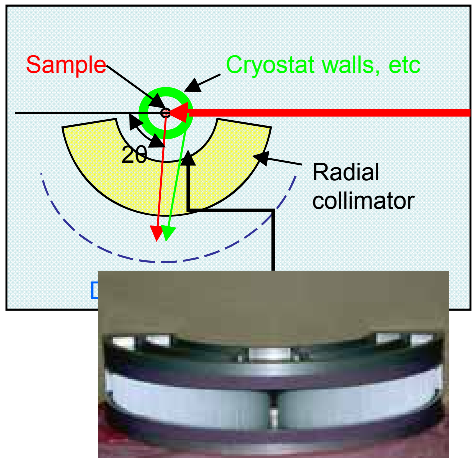
Focusing Monochromator

28.5cm high

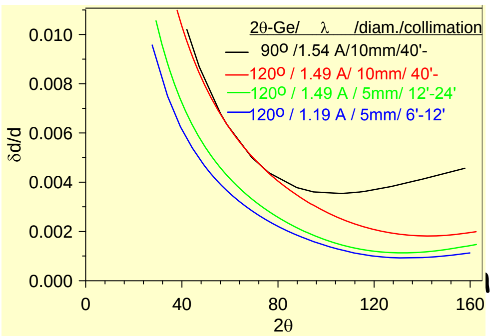
Mosaic fwhm 15'



Radial Collimator – clean pattern



… and resolution



2005, work in progress

HRPT - High Resolution Powder Diffractometer for Thermal Neutrons

Easy and safe access

The new working podest on top of HRPT

HRPT features

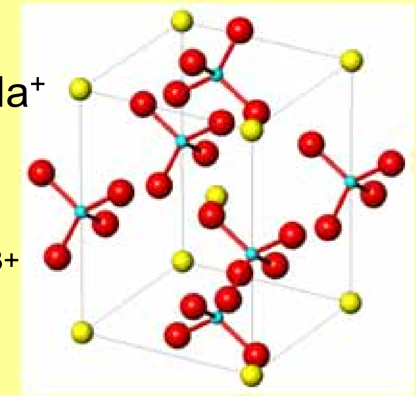
- Flexible choice of wavelength with vertically focusing wafer Ge monochromator 28.5cm high, total mosaic halfwidth 15'.
- Flexible resolution/intensity:
  - primary beam collimations 6', 12', 40'
  - slit system for secondary collimation < 40'
  - monochromator take-off-angle 90° and 120°
- Oscillating (1°) mylar-GdO radial collimator to eliminate Bragg peaks from sample environment such as from cryostat or furnace.
- Monochromatic beam shielding
- Sample environment
  - evacuated Al pot with oscillating closed-cycle He refrigerator.
  - zero matrix pressure cells (9, 15 kbar)
  - standard LNS sample environment: T=50mK—2100K, H=5T(vertical)
  - Podest for experimental infrastructure

Update from Version 2005, work in progress

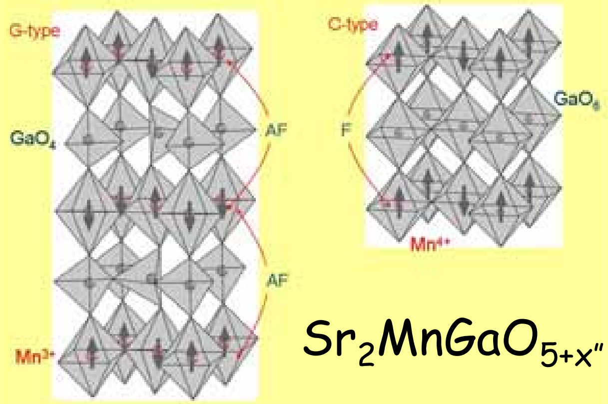
HRPT - High Resolution Powder Diffractometer for Thermal Neutrons

Applications

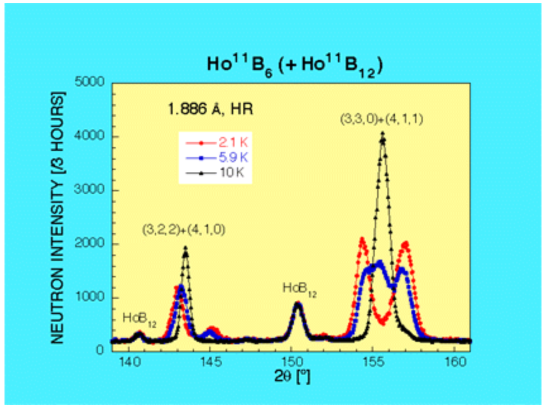
- Precise structure refinement complementary to X-rays
- Magnetic ordering phenomena complementary to DMC
- Detection of lattice distortions, defects, internal strains from (anisotropic) line broadening
- Phase analysis of new materials, direct structure solution from powder data
- Real time studies of chemical, structural, magnetic changes



$NaBD_4$",



Sr_2MnGaO_{5+x}",



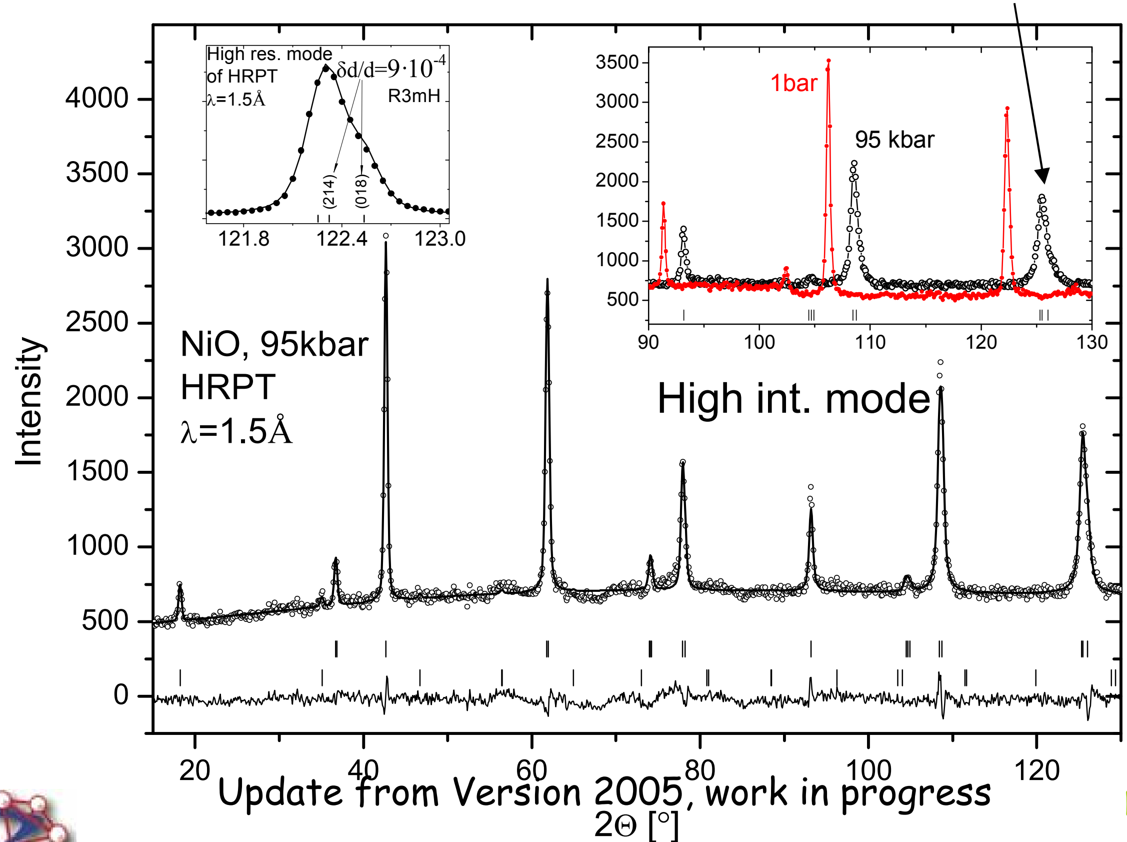
rsion 2005, work in progress

HRPT - High Resolution Powder Diffractometer for Thermal Neutrons

Lattice distortion and magnetic structure in NiO under high pressures (up to 130kbar) at HRPT

@ p=1bar: μ_{Ni}=1.73(9) μ_B, k =[½ ½ ½] in *Fm3m*
R3-m: a=2.9534(2)Å, α=60.061(2)°

Increased rhombohedral splitting at 95kbar



Update from Version 2005, work in progress

APPLIED PHYSICS LETTERS 86, 1 (2005)

Angle-dispersive neutron diffraction under high pressure to 10 GPa

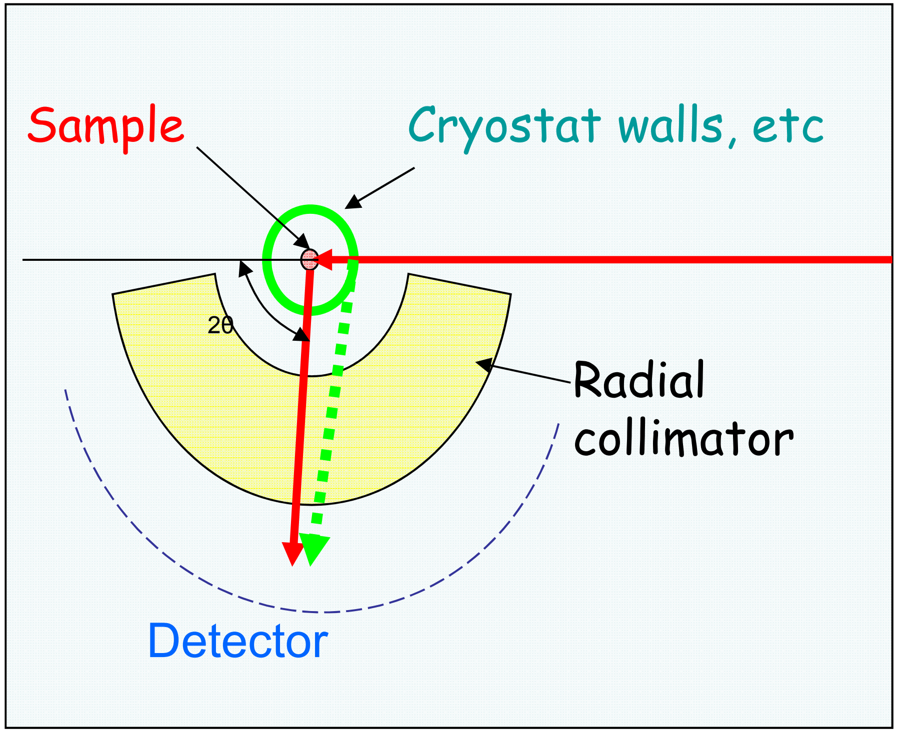
S. Klotz, Th. Strässle, G. Rousse, and G. Hamel
Physique des Milieux Condensés, Université P&M Curie B77, 4 Place Jussieu, 75252 Paris, France
V. Pomjakushin
Laboratory for Neutron Scattering, ETH Zürich and PSI, 5232 Villigen PSI, Switzerland

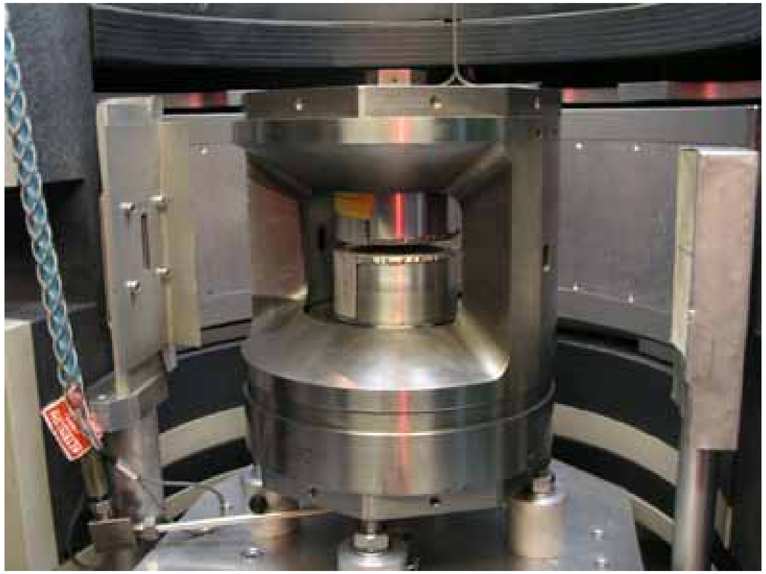
(Received 9 September 2004; accepted 29 November 2004)

We present a method which allows high-quality powder neutron diffraction patterns to be obtained under pressure by angle-dispersive diffraction to at least 10 GPa. This technique uses a new type of Paris–Edinburgh press in conjunction with sintered boron nitride anvils. As an example, we show NiO diffraction patterns obtained under purely hydrostatic pressures up to 10 GPa. These data were collected within a few hours, and are free from any contaminating signal from the pressure cell. High-resolution nuclear and magnetic structural information can be readily extracted by Rietveld refinements, without additional data correction. This technique will allow powder neutron diffraction at elevated pressures to become a standard tool on continuous neutron facilities. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1855419]

S. Klotz, Th. Strässle, G. Rousse, G. Hamel, V. Pomjakushin, *APL 2005.*

Improvements 2005:
High resolution radial collimator (ordered 11/2004)

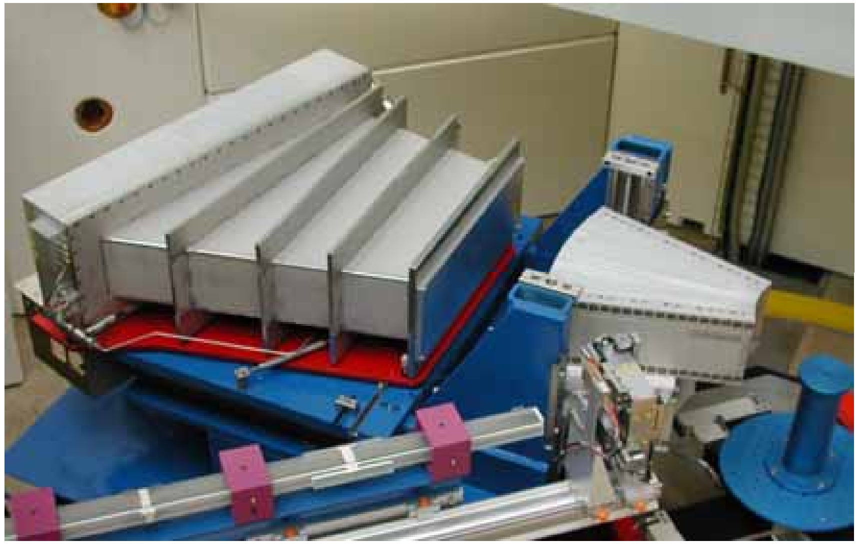


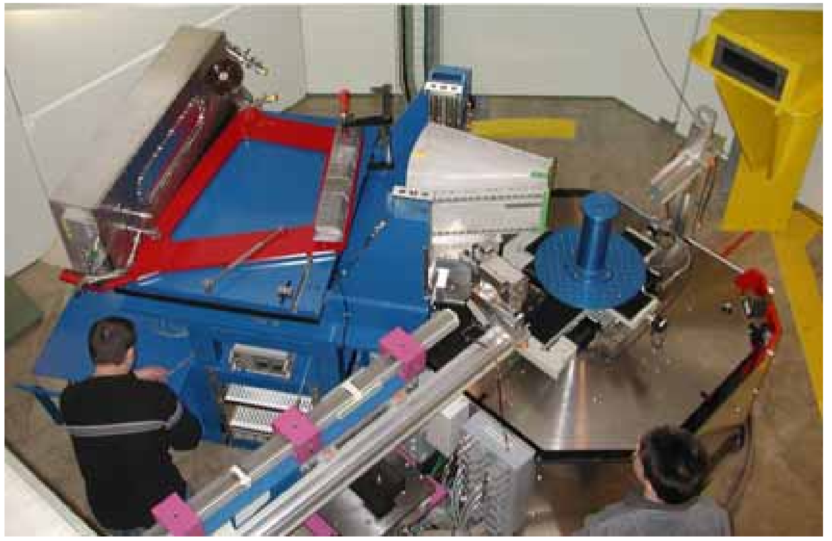


Update from Version 2005, work in progress

Stress Scanner: POLDI

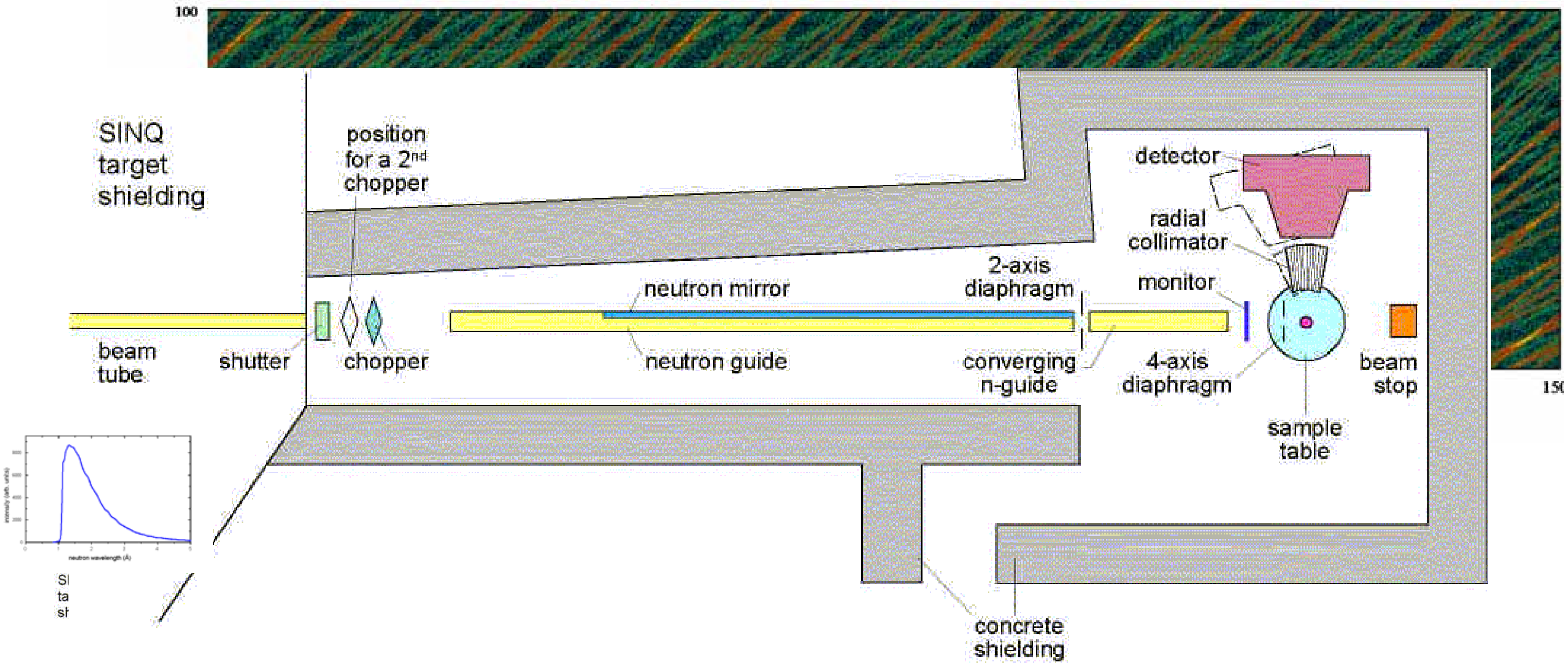
Uwe Stuhr, Mirco Grosse

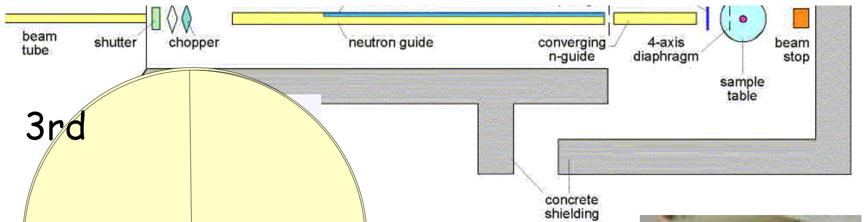




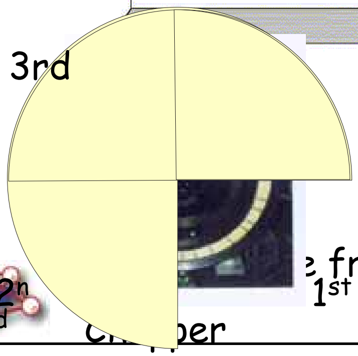
Update from Version 2005, work in progress

Layout of Strain Scanner POLDI





dθ (s_{tot} dθ)



…e from Version 20… …ogress

POLDI: Residual Stresses in Coatings of Turbine Blades

U. Stuhr (LNS), H.-P.Bossmann (ALSTOM AG, Baden)

NUM Report 2004

Modern gas turbines are running close to the theoretical efficiency limit of the Carnot-process

Only improvement: Higher gas temperature

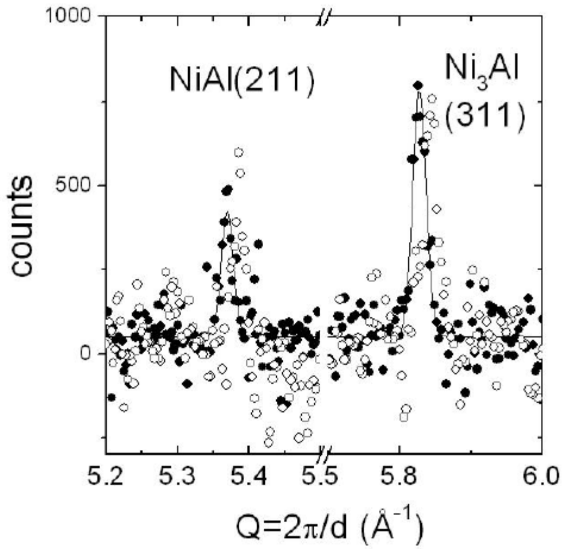
GT26, 281 MW

Coating protects inner part of turbine blade

- multi-phase Ni-Al alloy
  (~40%NiAl, ~40%Ni_3Al, ~20% andere)
  (complete surface, 0.3mm)
- Zr_2O_3 (very hot zones, additionaly 0.4mm)

Stress as a result of different expansion coefficients of the two layers

But stress limits lifetime of the turbine blade



• Parallel, ▫ perpendicular to surface
100 thermal cycles

Results: POLDI can observe the stress direction of the NiAl layer (NiAl, Ni_3Al), ~1000 10^{-9}

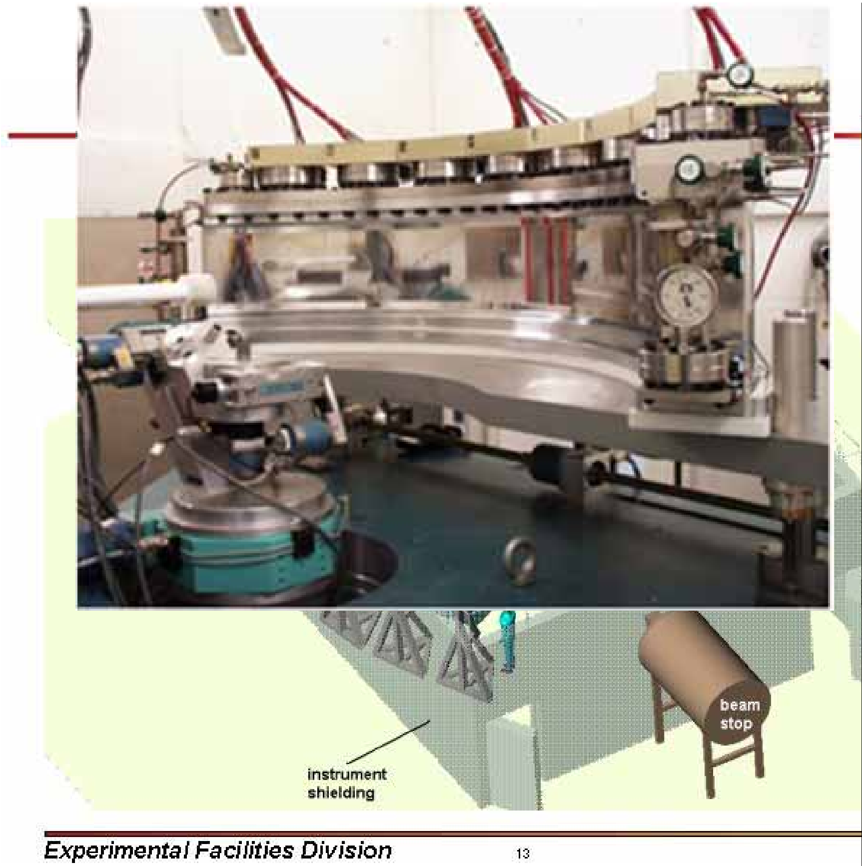
Future: Search maximum safe operation temperature measuring samples with different thermal history

Update from Version 2005, work in progress

Project: Two-Dimensional Banana Powder Diffractometer DMC^{160x300}

(Replacement of the 1983/4 built instrument)

L. Keller, J. Schefer (LNS)
M. Hildebrandt, N. Schlumpf (TEM), U. Filges, P. Keller, P. Allenspach (LDM)



Specifications

140-160° Banana-type, Radius=1500mm
300 mm high
resolution 2.5mm (2Θ) by 5-10mm (height)
10^5 counts/sec/wire
10^7 counts/sec/all
efficiency >90%@2.5Å
module production preferred

Gain to present DMC:
1-2 (2Θ) (145° compared to 80°)
3.3 height (300mm compared to 90mm)
Higher gas pressure, 3.6 bar He^3:
1.6 (@4.2Å) to 2.2 (@2.56Å)

Total gain **5-15** depending on problem and λ

Update from Version 2005, work in progress

Costs Estimation DMC-2 by comparison to POLDI

Poldi: 1-dim, 0.2 m²

Effective costs Poldi detector:
Gas-Box: 50 kFr
Hardware: 50 kFr
Electronic: 60 kFr

Commercial: e.g. HRPT
1.4 Mio without PSI upgrade done

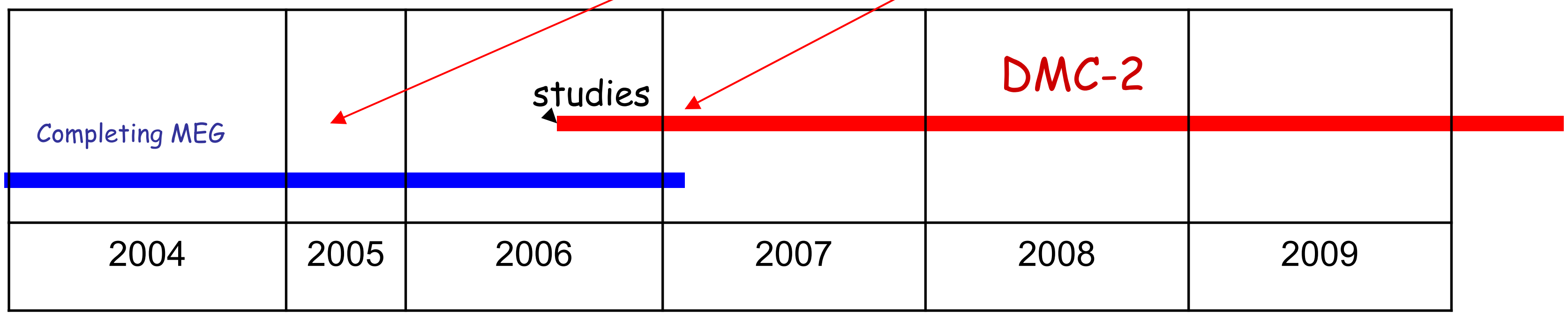
DMC-2
2-dim, 1.3m² = 6x area POLDI
+more complex due to 2-dimensional readout

approx. cost DMC⁺ Detector
Gas-Box: **300 kFr**
Detector: Hardware **400 kFr**
Electronic: **300 kFr**
Instrument: **550 kFr**.

Total 1.55MFr / 1Mio Detector only
(FOKO-Proposal, approved 3/2005)

Time Schedule

Start of Financing by NUM/PSI



Update from Version 2005, work in progress
(MEG=muon detector project)

Project: Laue/Quasi-Laue Diffractometer

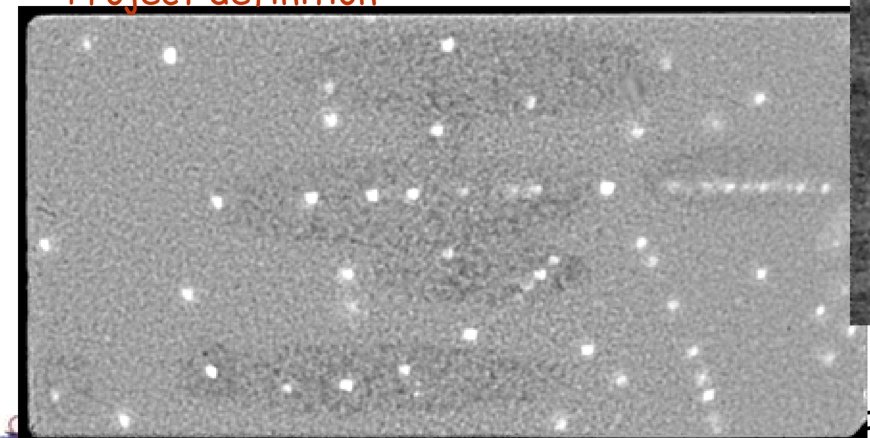
D. Sheptyakov, O. Zaharko and U. Stuhr
E. Lehmann, G. Frei (ASQ), P. Keller (LDM)

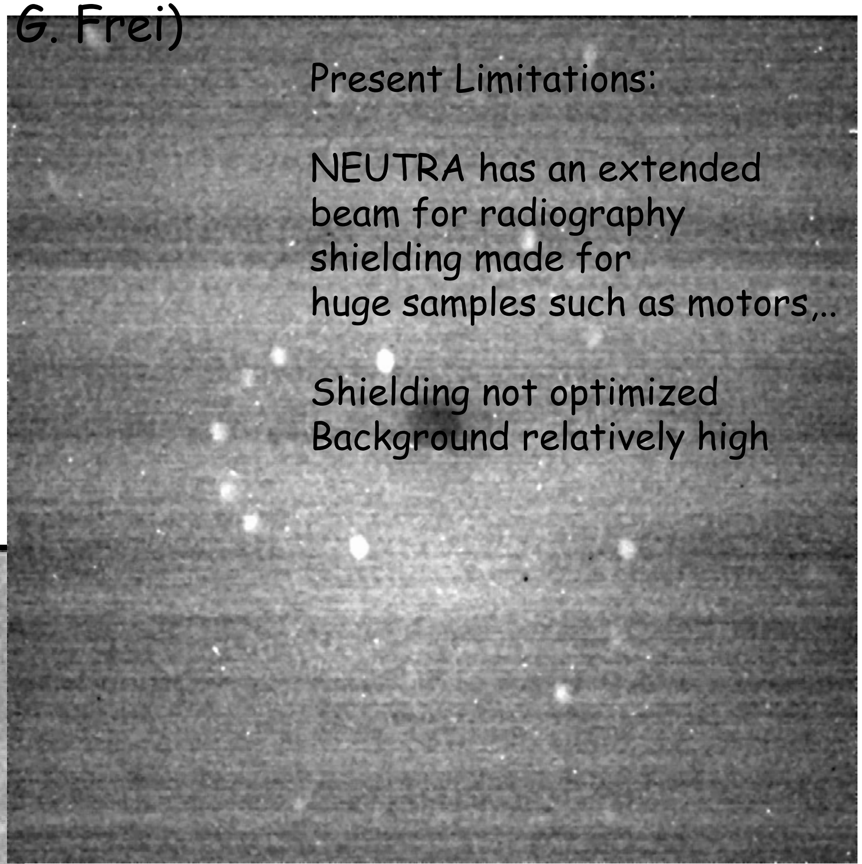
Test on NEUTRA (with E. Lehmann, G. Frei)

Crystal: (H. Ronnow)
$Yb_2Ti_2O_7$ diameter ~4 mm, height ~6 mm
neutron image plate
exposition time: 10 minutes
the 2Θ range ~5° to ~170° (horizontal)
~-36° to ~+36° (vertical)

2nd test on same sample:
CCD Camera
1 degree steps in omega
(sample table, Yb2Ti2O7 diameter ~4 mm, height ~6 mm)
1 minute exposure time / omega-step

Next steps in 2005:
Test on POLDI (Background optimization)
Project definition





Present Limitations:

NEUTRA has an extended beam for radiography shielding made for huge samples such as motors,..

Shielding not optimized
Background relatively high

progress

The Neutron Diffraction Group 2011

Vladimir
Pomjakushin

Lukas
Keller

Nadir Aliouane

Ph.D.:
Warren Wallace
Ravi Sura

Update from Version 2005, work in progress

Ende

Paris Edinburgh Pressure Cell

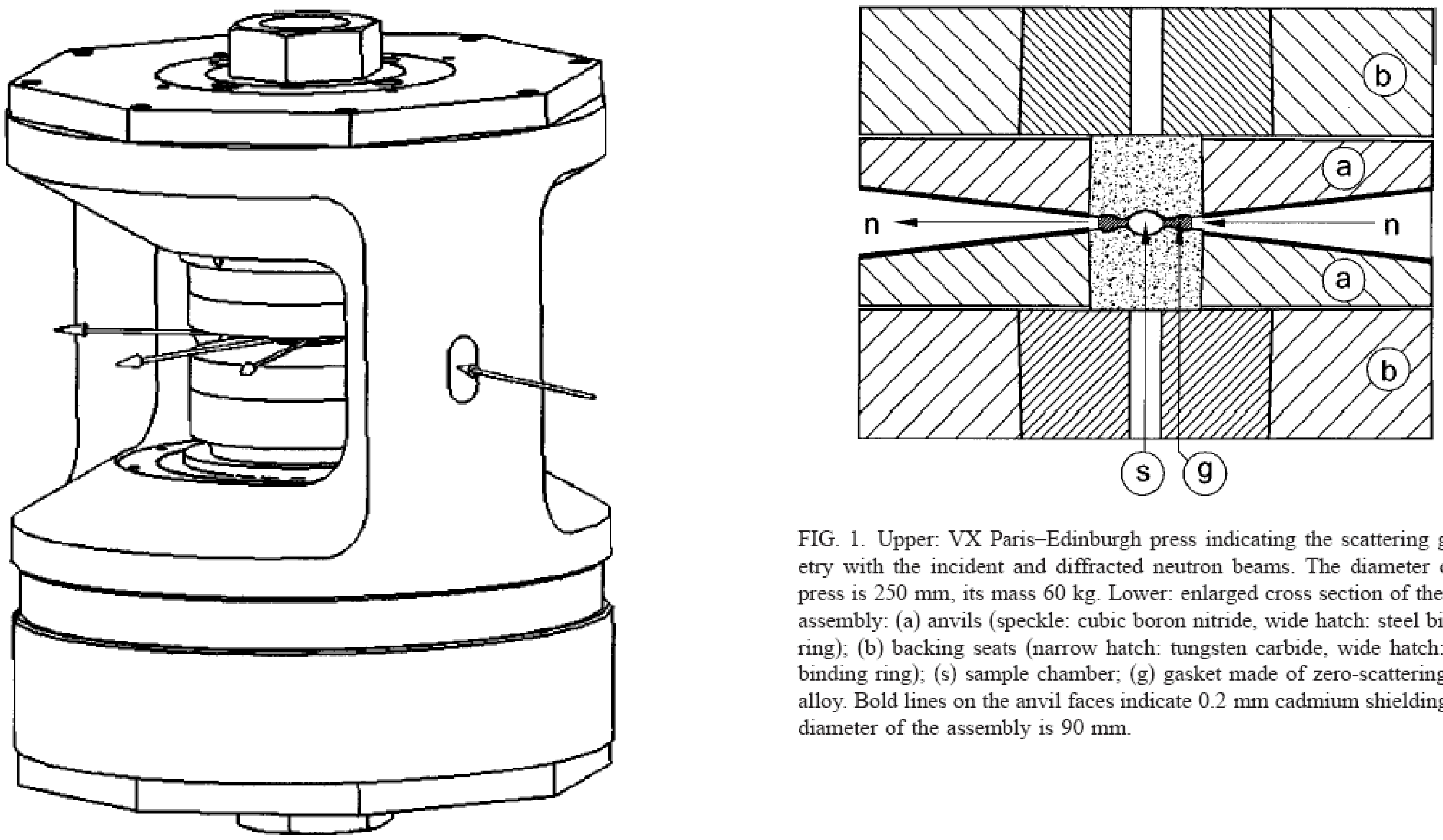


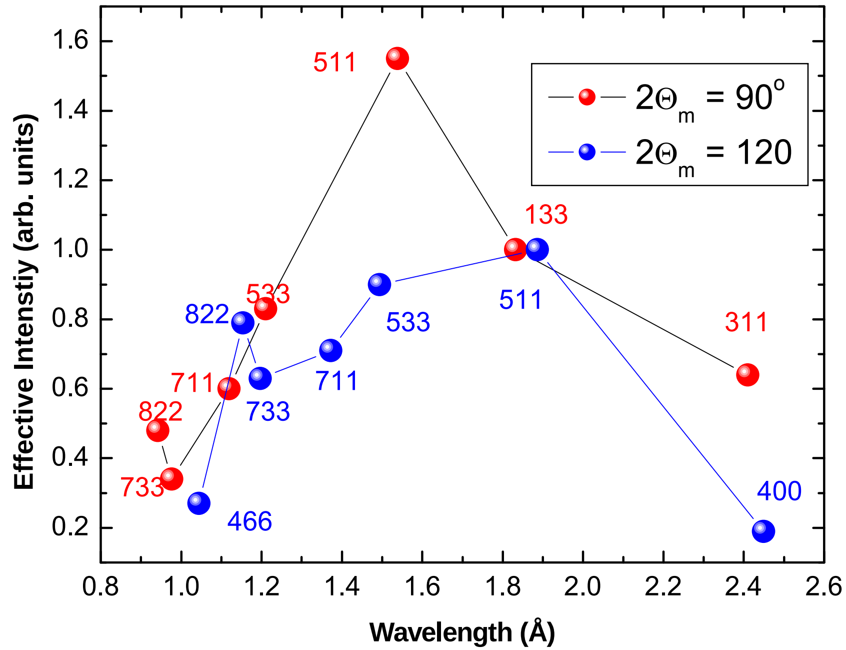
FIG. 1. Upper: VX Paris–Edinburgh press indicating the scattering geometry with the incident and diffracted neutron beams. The diameter of the press is 250 mm, its mass 60 kg. Lower: enlarged cross section of the anvil assembly: (a) anvils (speckle: cubic boron nitride, wide hatch: steel binding ring); (b) backing seats (narrow hatch: tungsten carbide, wide hatch: steel binding ring); (s) sample chamber; (g) gasket made of zero-scattering TiZr alloy. Bold lines on the anvil faces indicate 0.2 mm cadmium shielding. The diameter of the assembly is 90 mm.

~ 30-100 mm^3, up to 30 GPa

Update from Version 2005, work in progress

HRPT

	$2\theta_M=90°$		$2\theta_M = 120°$	
(hkk) Ge	λ, Å	Effective intensity	λ, Å	Effective intensity
311	2.4097[1]	0.64	2.953[1,2]	
400	1.9984[4,5]		2.449[1,3]	0.19
133	1.8324	1.00	2.246[1,2]	
511	1.5384	1.55	1.886	1.0
533	1.2183	0.83	1.494	0.90
711	1.1194	0.60	1.372	0.71
733	0.9763	0.34	1.197	0.63
822	0.9419	0.48	1.154	0.79
466			1.044	0.27



Update from Version 2005, work in progress

DMC-2

Simulations (U. Filges)

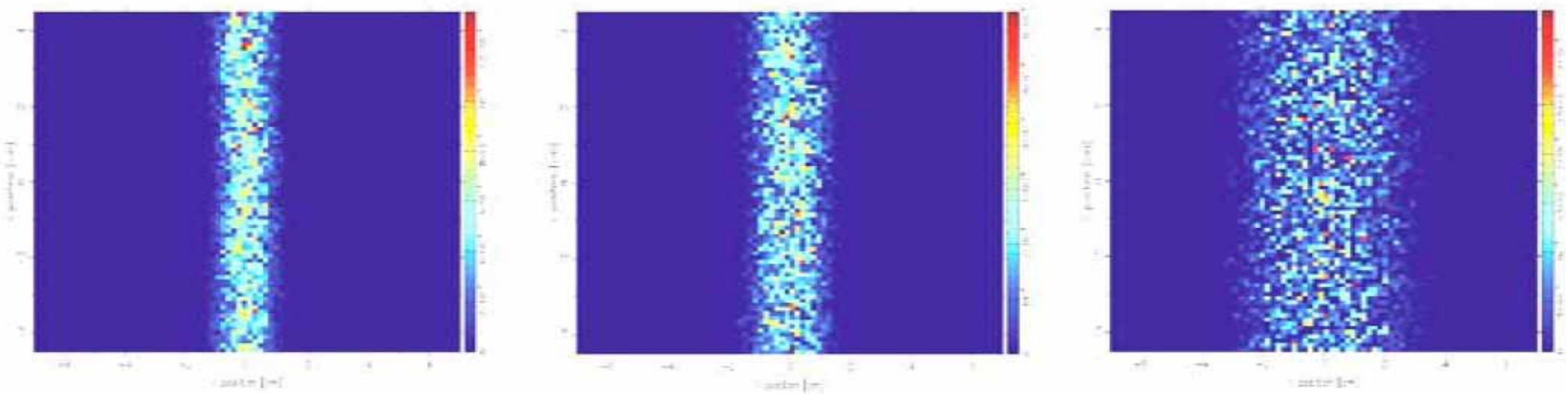


Figure 1: PSD images of the 9cm-detector for bragg reflections 35.73° , 51.42° and 90.2°

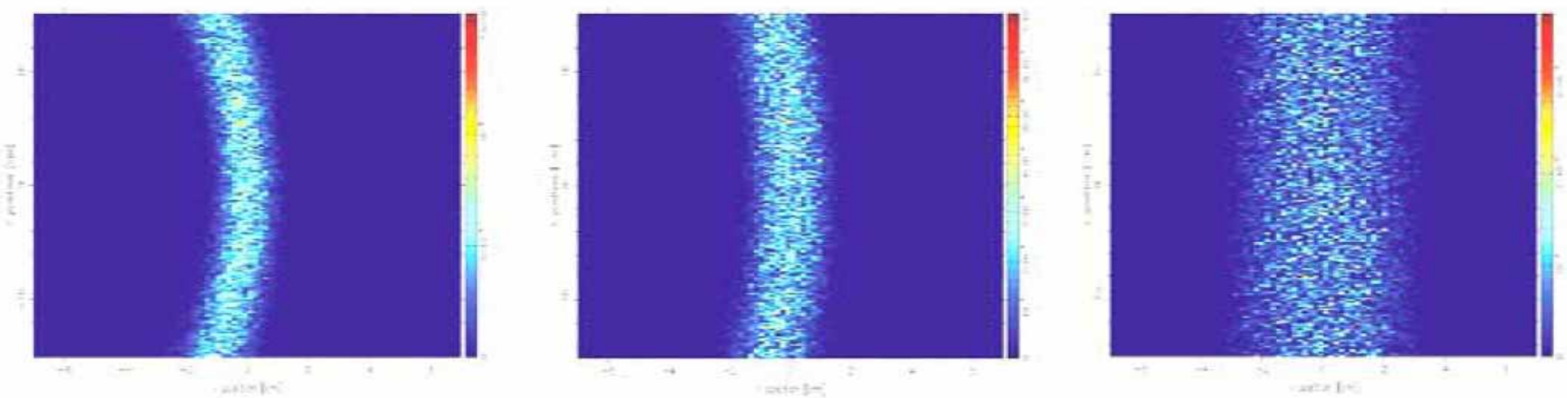
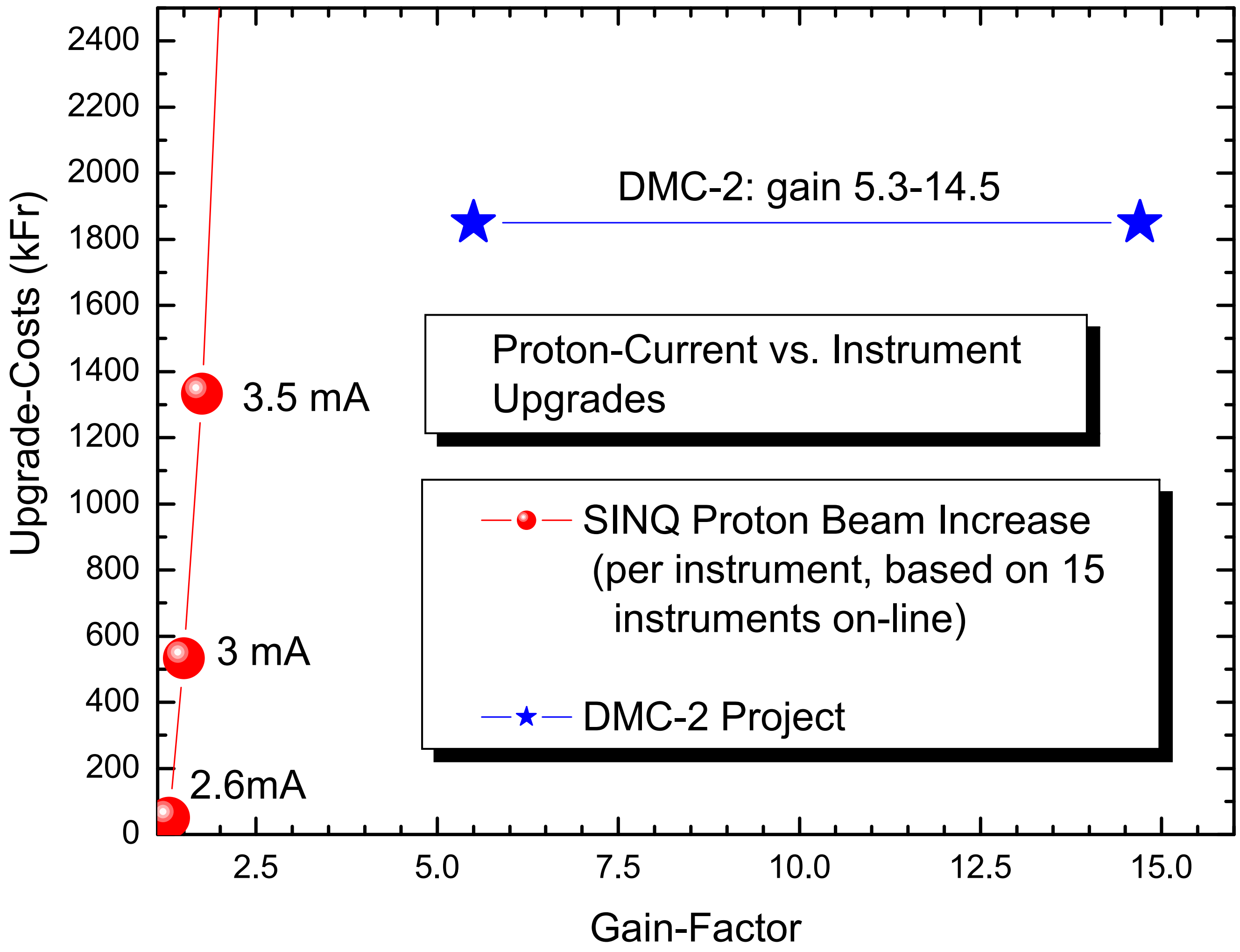


Figure 2: PSD images of the 30cm-detector for bragg reflections 35.73° , 51.42° and 90.2°

Update from Version 2005, work in progress

detector upgrade vs. higher proton current



Update from Version 2005, work in progress

Quadrupolar and dipolar magnetic order in $DyPd_3S_4$

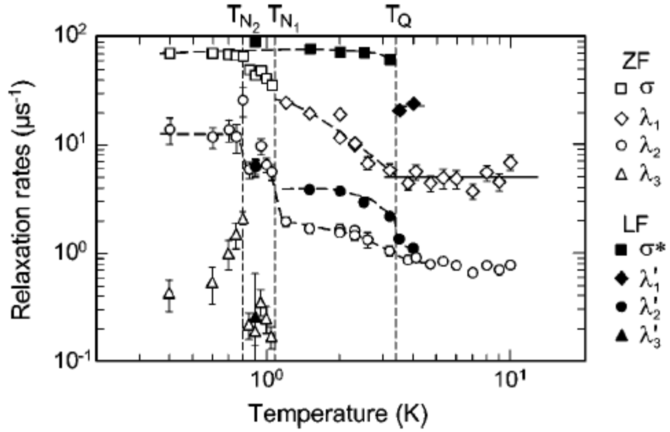


FIG. 1. Temperature dependencies of the muon spin relaxation rates. The zero (ZF) and longitudinal field (LF) experiments are shown by open and filled symbols, respectively. Anomalies at T_Q, T_{N1}, and T_{N2} are signatures of the quadrupolar and the magnetic ordering phenomena.

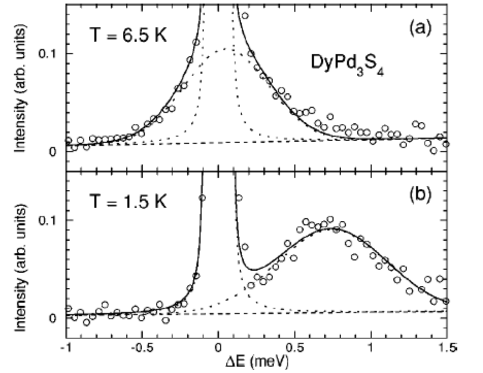
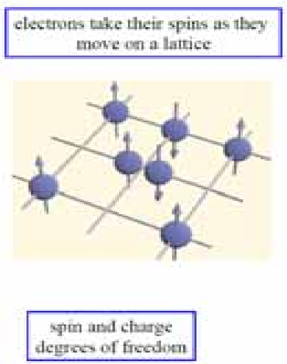
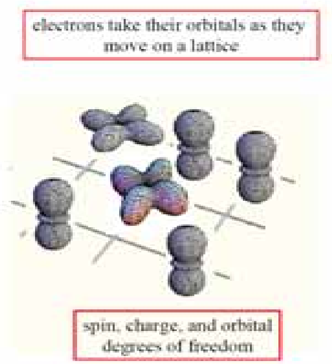
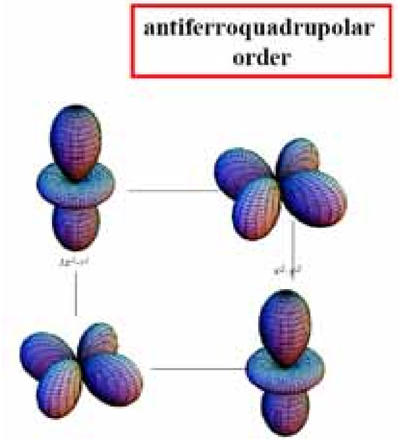


FIG. 2. Inelastic neutron scattering spectra of $DyPd_3S_4$ at (a) $T>T_Q$ and (b) $T<T_Q$.







Update from Version 2005, work in progress