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
DMC - Cold Neutron Powder Diffractometer

- 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:
Ge311 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)
DMC - cold neutron powder diffractometer

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

Quadrupolar and dipolar magnetic order in DyPd$_3$S$_4$: A neutron scattering and muon spin rotation and relaxation investigation

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(Received 19 February 2004; published 30 August 2004)

- first investigation of field induced magnetic order in the quadrupolar phase of DyPd$_3$S$_4$ and of its temperature and field dependence
- first investigation of spontaneous magnetic order at low temperature and its relation to the quadrupolar phase

Canted anti-ferromagnetism
\[\alpha = 91^\circ\] at 1 K
\[\alpha = 99^\circ\] at 0.8 K
system reacts to quadrupolar ordering
TriCS Single Crystal Diffractometer

Jürg Schefer
Oksana Zaharko

Monochromator:
$\text{Ge}_{311}$ 1.18 Å (optimized for TriCS, 5·12.5cm$^2$, focusing)

$C_{002}$: 2.331 Å (from Saphir, 5·4cm$^2$, non-focusing)

Update from Version 2005, work in progress

Neutron Diffraction Group PSI - SINQ
TriCS: Improvements 2004/5

1. New software package Cami4PSD

2. Faster Scanning

3. Closed Cycle for 4 K

4. Radial Collimator

M. Könnecke, NUM Report 2004

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

Old one: 12-450K
Still available

Update from Version 2005, work in progress
Elastic and inelastic neutron scattering experiments have been performed on the dimer spin system NH$_4$CuCl$_3$, which shows plateaus in the magnetization curve at \( m = 1/4 \) and \( m = 3/4 \) of the saturation value. Two structural phase transitions at \( T_1 \approx 156 \text{ K} \) and at \( T_2 = 70 \text{ K} \) 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 \( T_N = 1.3 \text{ K} \). 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**

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**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
- $2\theta < 165^\circ$ → high Q $\leq 13$ Å$^{-1}$
- High resolution $\delta d/d = 10^{-3}$
- 1600 $^3$He detectors (70% efficiency @ 1.5 Å) with angular separation 0.1°
- Flexible wavelength, resolution/intensity

**HRPT layout**

- SINO target
- $^3$He scatterer
- High energy shutter
- Beam reductions collimator $\alpha_1$
- LN$_2$ COoled Si filter
- Beam stop, sample $\alpha_2$
- Evacuated beam tube
- PSD detector
- Slt system $\alpha_3$
- Vert. foc. Ge (Bragg) water monochr.
- $\lambda = 1.9$ Å, 12' / 24'
- 10mm, $\text{Na}_2\text{Al}_{12}\text{Ca}_3\text{F}_{14}$
HRPT - High Resolution Powder Diffractometer for Thermal Neutrons

Flexible choice of wavelength

- 2θ_m = 90°
- 2θ_m = 120°

Focusing Monochromator

28.5cm high
Mosaic fwhm 15'

Radial Collimator - clean pattern

... and resolution

Effective Intensity (arb. units) vs. Wavelength (Å)

Gain of intensity by a factor of 4!

Flexible choice of wavelength

Flexible choice of wavelength

Flexible choice of wavelength
**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

**Easy and safe access**

The new working podest on top of HRPT
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

\[
\begin{align*}
\text{Na}^+ & \quad \text{D}^- & \quad \text{B}^{3+} \\
\text{NaBD}_4^- & \quad \text{Sr}_2\text{MnGaO}_{5+x}^+ & \quad \text{Ho}^{11}\text{B}_6 (\text{+ Ho}^{11}\text{B}_{12})
\end{align*}
\]

Version 2005, work in progress

Neutron Diffraction Group PSI - SINQ
Lattice distortion and magnetic structure in NiO under high pressures (up to 130 kbar) at HRPT

@ p=1bar: $\mu_{\text{Ni}}=1.73(9)$ $\mu_{\text{B}}$, k = [1/2 1/2 1/2] in Fm3m

$R3m$: $a=2.9534(2)\text{Å}$, $\alpha=60.061(2)\text{°}$

High int. mode

Increased rhombohedral splitting at 95 kbar

High res. mode of HRPT $\lambda=1.5\text{Å}$

$\delta d/d=9\cdot10^{-4}$

95 kbar: 121.8, 122.4, 123.0

Internal mode

Intensity

HRPT - High Resolution Powder Diffractometer for Thermal Neutrons

Neutron Diffraction Group PSI - SINQ
Improvements 2005:
High resolution radial collimator (ordered 11/2004)
Stress Scanner: POLDI

Uwe Stuhr, Mirco Grosse

Update from Version 2005, work in progress
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%Ni₃Al, ~20% andere)
  (complete surface, 0.3mm)
- Zr₂O₃ (very hot zones, additionally 0.4mm)

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

But stress limits lifetime of the turbine blade

Results: POLDI can observe the stress direction of the NiAl layer (NiAl, Ni₃Al), ~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^{160\times300}
(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 \(\lambda\)
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

Completing MEG | studies | DMC-2
---|---|---
2004 | 2005 | 2006 | 2007 | 2008 | 2009

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)

2nd test on same sample:

Crystal: (H. Ronnow) Yb$_2$Ti$_2$O$_7$ diameter ~4 mm, height ~6 mm

- 1 degree steps in omega
- Neutron image plate
- Exposition time: 10 minutes

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...

- Background relatively high
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.
HRPT

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<th>Effective intensity</th>
<th>$\lambda$, Å</th>
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Update from Version 2005, work in progress

Neutron Diffraction Group PSI - SINQ
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

Neutron Diffraction Group PSI - SINQ
detector upgrade vs. higher proton current

Update from Version 2005, work in progress
Quadrupolar and dipolar magnetic order in DyPd$_3$S$_4$

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$_3$S$_4$ at (a) $T>T_Q$ and (b) $T<T_Q$.

Update from Version 2005, work in progress