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RESI: Thermal neutron single crystal diffractometer

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Abstract: The diffractometer RESI, which is operated by the Department für Geo- und Umweltwissenschaften Sektion Kristallographie, Ludwig-Maximilians-Universität München and the Technische Universität München, is designed for high q-resolution, low background and best flux usage allowing optimum measurements of weak diffraction phenomena in a large portion of the reciprocal space on single crystalline samples.

1 Typical Applications

Structure analysis with thermal neutrons (λ = 0.8 Å to 2 Å) is complementary to structure analysis with X-rays. The measurement possibilities provided by this instrument are crucial for many scientific questions:

- **Structure analysis, bonding theory, electron densities:** Due to the interaction with atomic cores and the diffraction angle independence of the atomic form factor, it is possible to measure Bragg scattering up to high diffraction angles.
- **Real crystals** and compounds of interest for material science are often not perfectly ordered. The elucidation of these real structures requires the analysis of the corresponding diffuse scattering. The diffuse scattering - off the Bragg reflections - is normally differentially weak and distributed continually (anisotropic) in the reciprocal space.
- **Partially crystalline** compounds, like fibre structures, show a specific scattering, which is highly anisotropic and continously distributed in the reciprocal space. Therefore, diffractometers with area detectors like RESI are best suited for this kind of problems.
- **Structural phase transitions** can be accompanied by continuous reflection shifting.

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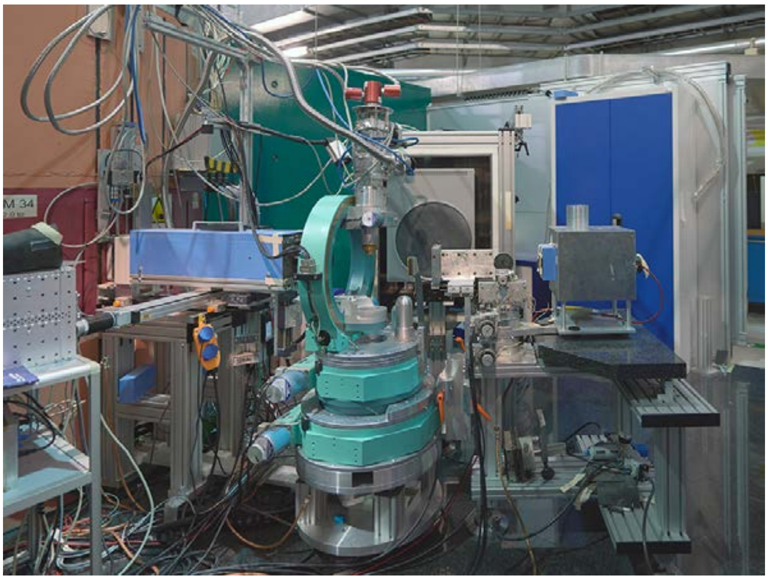

Figure 1: Instrument RESI with focusing guide (left), Eulerian cradle (middle, front), area detector (middle, back) and single counter (right); (Copyright by W. Schürmann, TUM).

- **Modulated structures** show satellite reflections at "incommensurable" positions. Both areas require analysis of large portions of the reciprocal space.
- A new class of **aperiodic crystals** ("quasi crystals") show dense, but discrete reflex patterns, where more than 90% of the reflexes are very weak. Additionally, due to the fact that quasi crystals often contain two or more transition metals (which are almost isoelectronic), neutrons offer much higher contrast than X-ray methods.
- **Twinned crystals and multi-domain/multi-phase crystals** are often difficult to measure on single-counter instruments. The area detector at RESI allows for easy detection and in many cases separation of reflections in such systems.

The advantages of the high-resolution area detector can be utilised best, if the reciprocal space is not too empty. That means, that RESI is optimal for cells of ca. 1000 Å^3 to ca. 20000 Å^3. Typical crystal sizes range from 5 mm^3 to 25 mm^3.

2 Sample Environment

Dedicated sample environment of RESI:

- Oxford Cryosystems Cryostream 700
  temperature range 100 K - 400 K
  consumption 20 l L-N_2/d
- Oxford Instruments Helijet
  temperature range 15 K - 100 K
  consumption 2 l L-He / h
  sample size 1 x 1 x 1 mm^3 max.

Standard sample environment usable with RESI:

- Closed-cycle cryostat CC, 2.5 K – 300 K
- Closed-cycle cryostat CCR, 3 K – 100 K
  using ^{3}He insert, 500 mK – 4 K
  using ^{3}He/^{4}He dilution, 50 mK – 1 K
- Vacuum furnace, 340 K – 2100 K
- Mirror furnace, RT – 1250 K

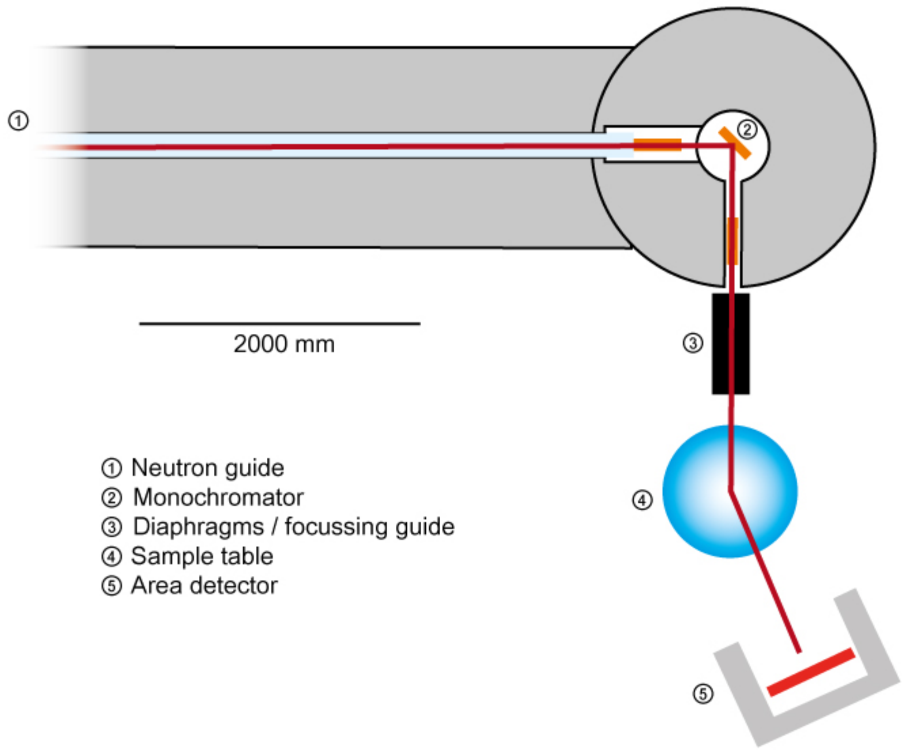

Figure 2: Schematic drawing of RESI.

3 Technical Data

3.1 Primary beam

- Beam tube SR-8b
- Neutron guide
  Length: 12 m, focussing vertical / horizontal
  section: 70 x 40 mm → 60 x 30 mm
- Coatings: m = 3 top/bottom; m = 1 side

3.2 Monochromators

Vertically focussing lamella type, fixed take-off 90°

- Cu-422, 20' mosaic, 1 Å: $2 \cdot 10^6$ n cm^{-2} s^{-1}
- Ge-511, 25' mosaic (deformed wafer stack) 1.5 Å: $6 \cdot 10^6$ n cm^{-2} s^{-1}

3.3 Secondary neutron guide

Vertically focussing ellipitical guide-in-guide

- Length: 1 m
- Focus 400 mm after guide exit
- Coating: m = 5

3.4 Available goniometers

- Kappa-Goniometer:
  Bruker-Nonius Mach3 carrying capacity: max 100 g
- Eulerian cradle Huber 420:
  higher carrying capacity, e.g. for closed-cycle cryostat
- Huber 2-circle goniometer:
  with tilting head highest carrying capacity, e.g. for CCR with ^{3}He insert

3.5 Available detectorss

- MAR345 image plate detector:
  345 mm diameter, N-sensitive image plate
- Single counter ^{3}He with optional analyzer for pure elastic scattering