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Pulsed magnetic fields at the ESRF

First results

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Outline

- Motivations
- Experimental set-up
  - Pulsed field & cryogenics (LNCMP)
  - ESRF beamline and detector
- Integration of both techniques
- First results on $TbVO_4$
- Future developments and applications

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Motivations

LNCMP
LABORATOIRE NATIONAL DES CHAMPS MAGNETIQUES PULSES

- Magnetic field is a thermodynamic variable of fundamental importance
  It is one of the important external parameters, such as *T*, *P* and *E*, which control phase transitions.

- **Studies in high magnetic fields** of systems with quantum correlations effects are of fundamental interest
  - **- to lift the degeneracy of degenerate quantum states (Zeeman effect)**
  - **- to select quantum states**
  - **- to break up of strong correlations between electrons (magnetism, superconductivity)**

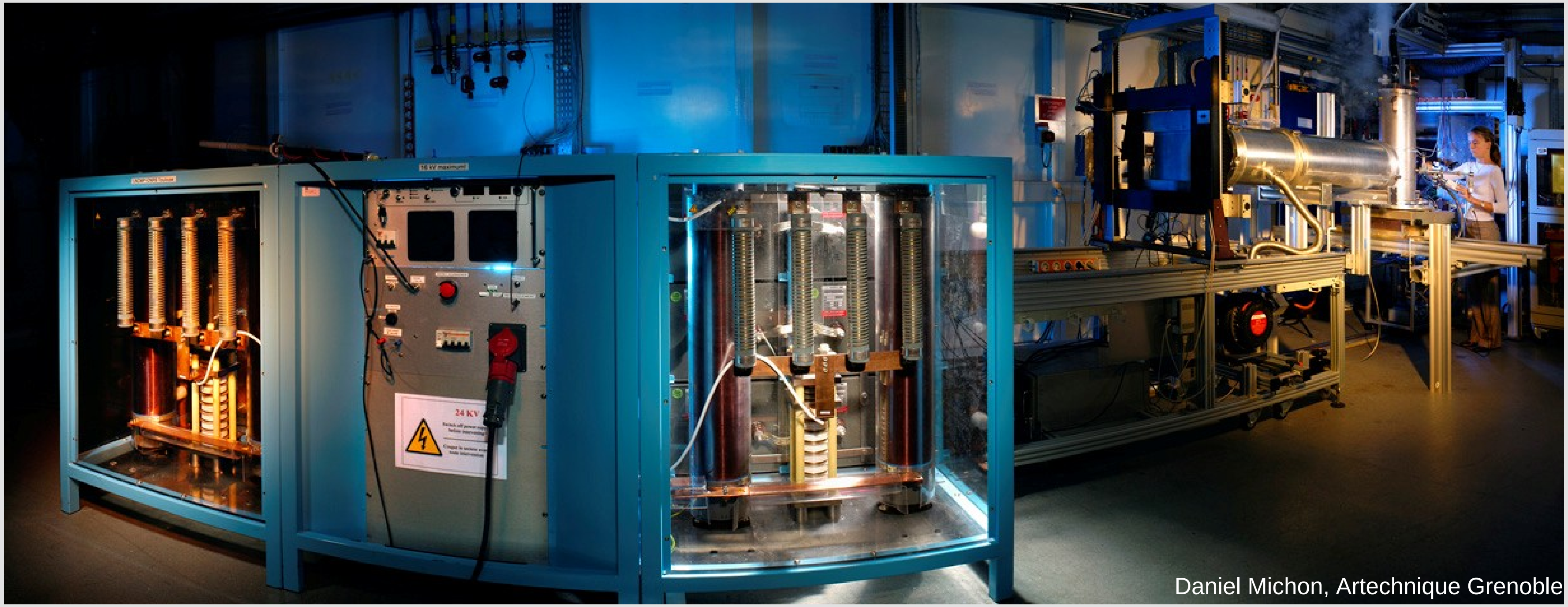
- All of the current high field measuring techniques are macroscopic (M, χ, r, V_H, C_v, v_s,…)…
  …Until recently, there was no direct information on the structural properties above 15 T. Whereas we know (from low field measurements) that often field-induced phase transition do have a structural component.
- Sound velocity v_s and dilatometry at high fields also indicate structural effects.

➡ Combination of X-ray techniques with High Magnetic Fields

The Pulsed Field generator

Mobility of pulsed fields equipment

The Pulsed Field generator

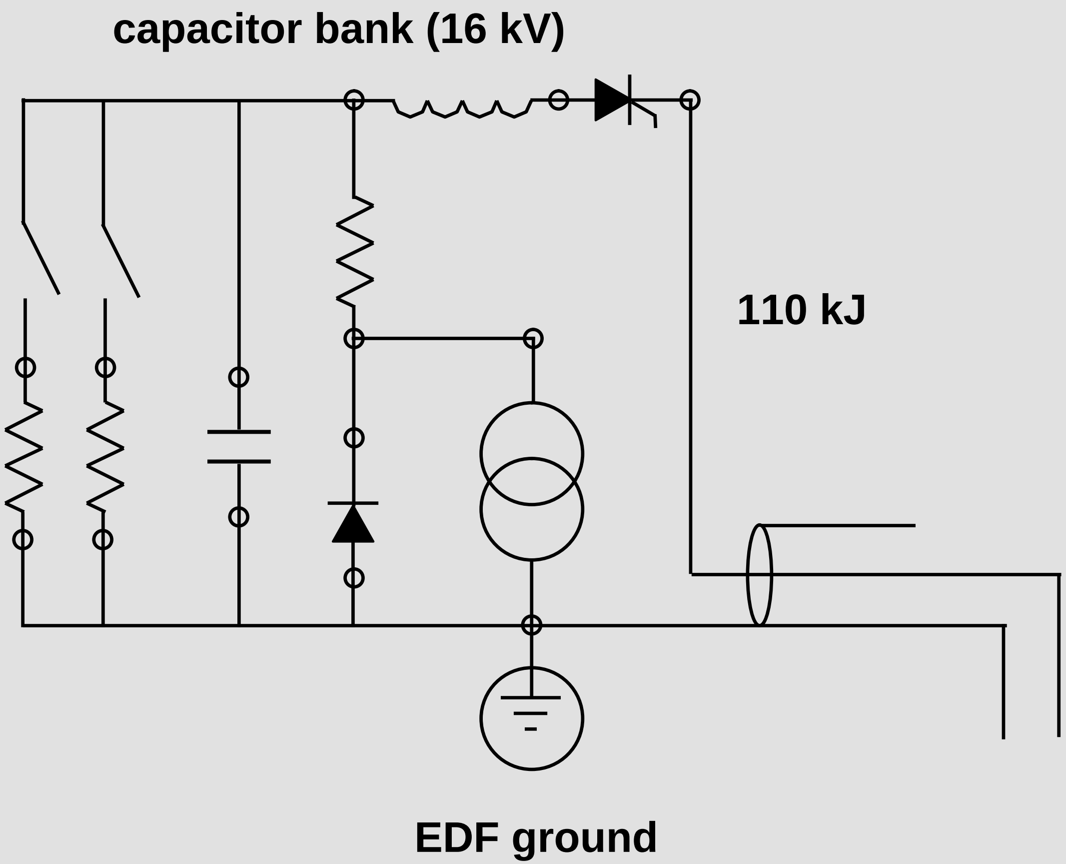


Daniel Michon, Artechnique Grenoble

Generator (LNCMP capacitor bank)

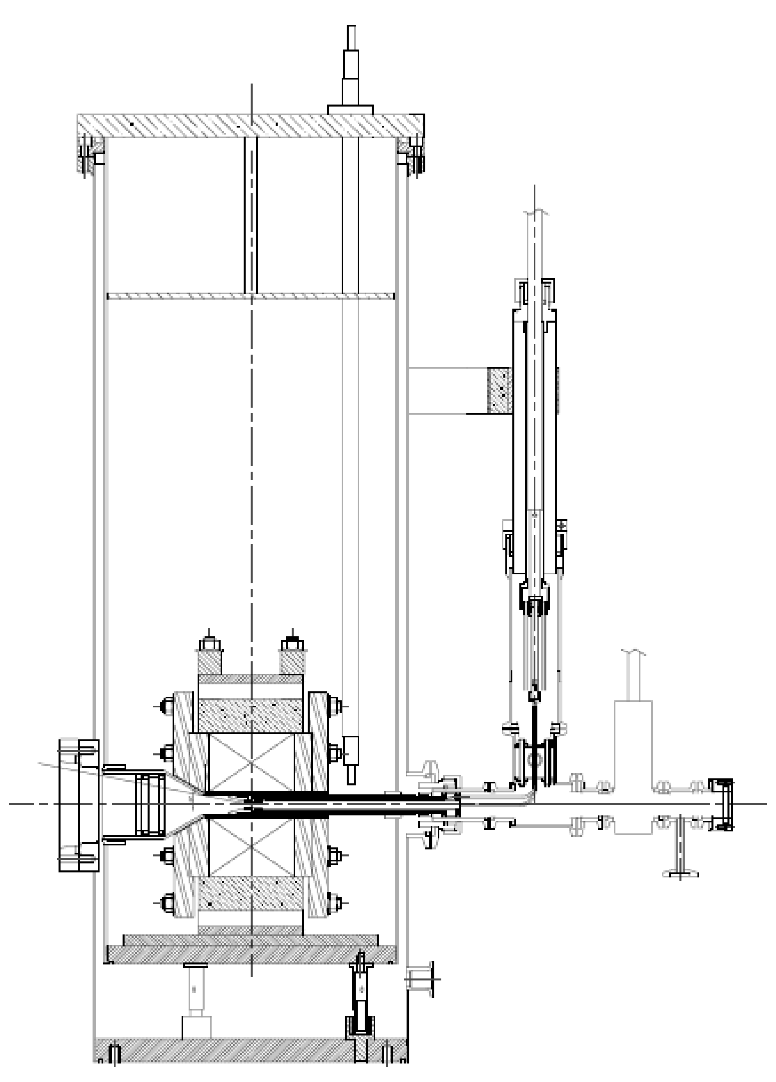
- Three subunits:
  2 storage units contain: capacitors, crowbar diodes and resistors, and current limiters.
  Control unit houses: charger, thyristor stack, dump resistors and relays, I-V monitors.
- 130 kJ. 16 kV
- The combined weight of the generator is 2800kg and dimensions are 1.25 x 1.3 x 2.85 m^3.

The LNCMP capacitor bank



- Max energy: 130 kJ
- V_{max} = 16 kV
- C= 1mF (12 x 0.083 mF)
- $R_{crowbar}$ = 225 mΩ
- L_{prot} = 0.25 mH (17.5 mΩ)
- I_{short} = 48 kA (0.8ms)
- Optically triggered thyristor switch
- R_{dump1} = 1 kΩ
- R_{dump2} = 1 kΩ

Pulsed field & Cryogenics



High field magnet (LNCMP)

- coil is wound of Glidcop wire
- bore of 20 mm Ø, external Ø = 124 mm, height = 74 mm
- L(T=77K)= 8.63 mH, R(T=77K)= 80 mΩ
- coil is horizontally mounted
- Beam axis was along the bore of the magnet (Faraday geometry)
- magnet immersed in liquid nitrogen

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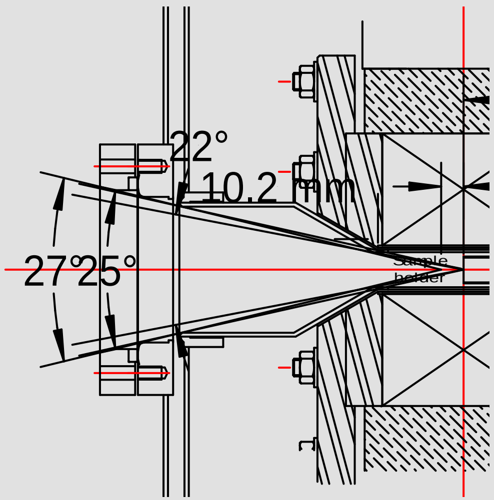
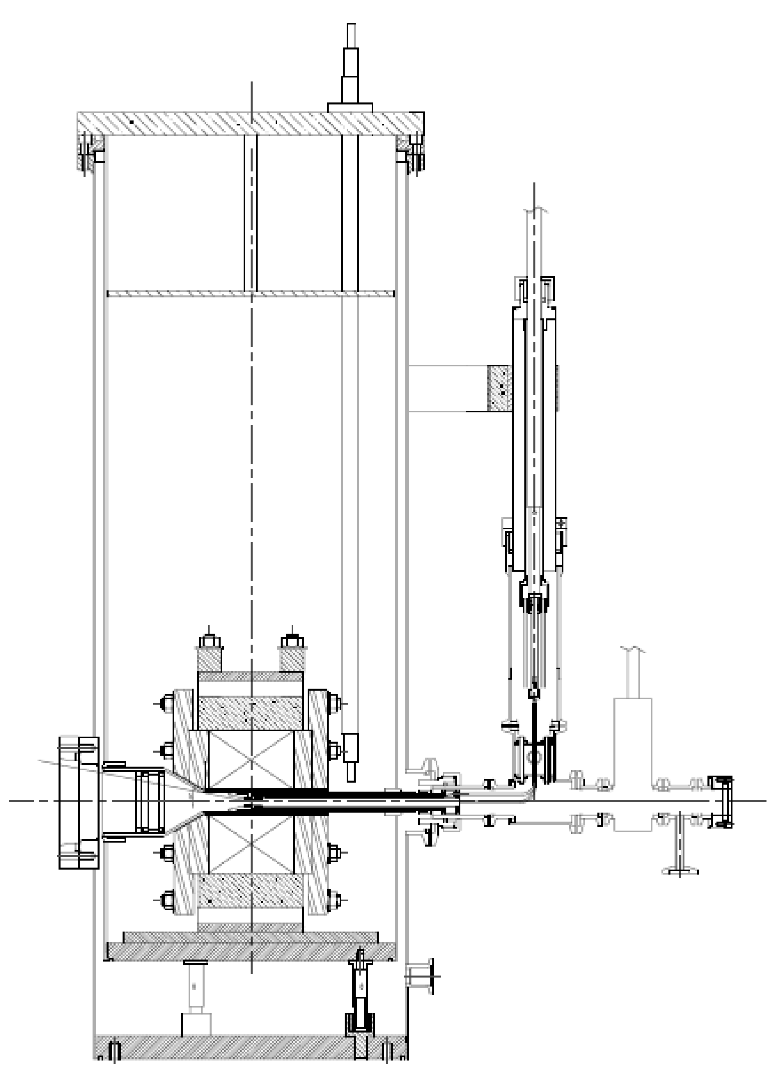
Cryogenics

Sample cryostat (LNCMP)

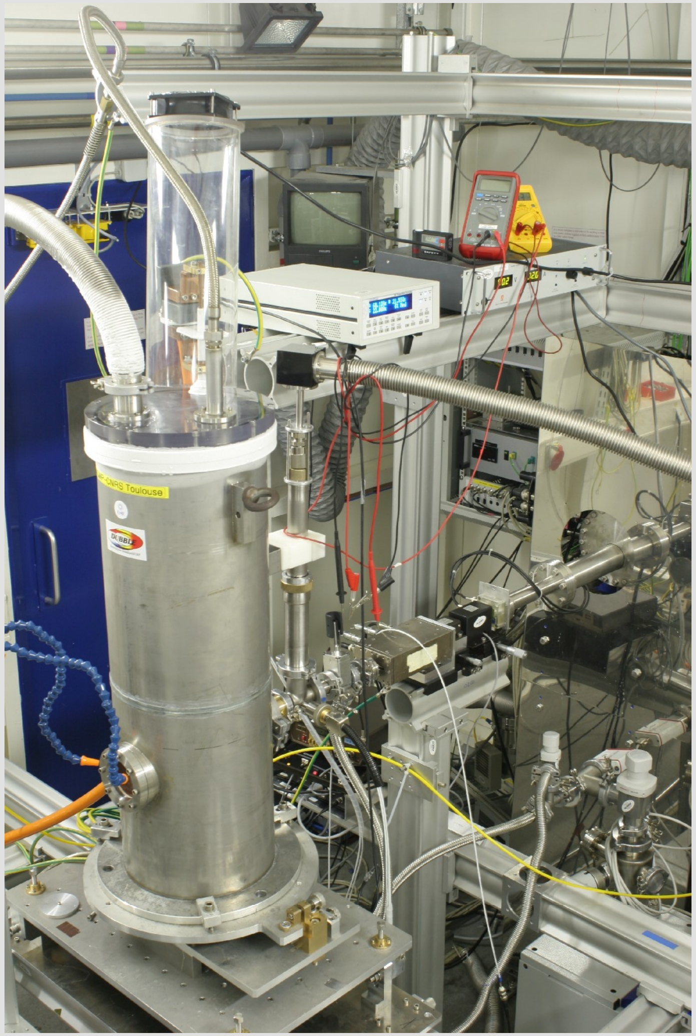
- He flow system
- ~7 K to 300 K
- PID heater control
- T-sensors (2)
- dB/dt sensor (Field)
- Fits inside magnet
- allows bundle to pass
- sample top-loading !

Pulsed field & Cryogenics

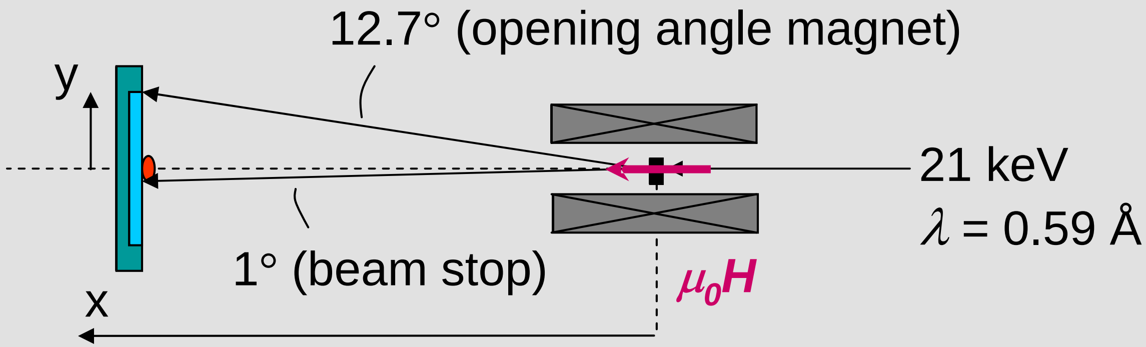




Integration of Synchrotron and Pulsed Fields



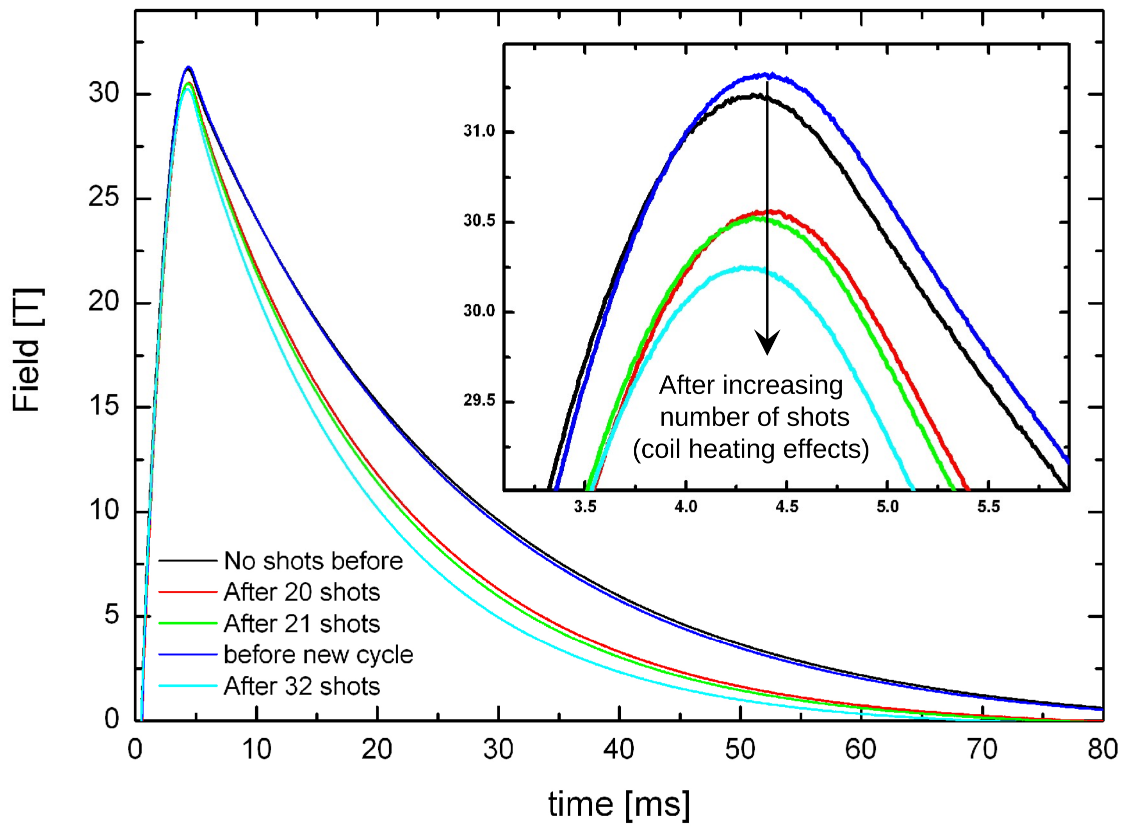
- Limited opening angle cryostat/magnet : ~13°



$$2d\sin\theta = \lambda$$
$$d = 2\pi/Q$$

⇨ Restriction of the available Q-space to 2.4 $Å^{-1}$

The pulsed magnetic field

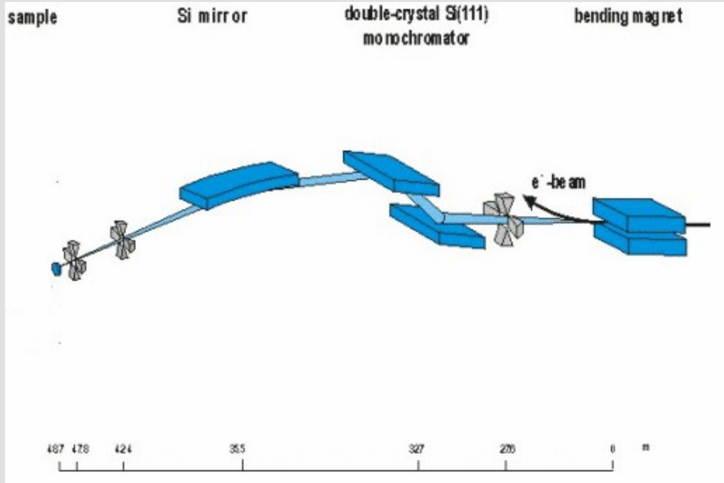


Magnetic field **up to 30 T** (110 kJ. 16 kV)

ESRF beamline and detector

DUBBLE CRG-Beamline (BM26B, ESRF)

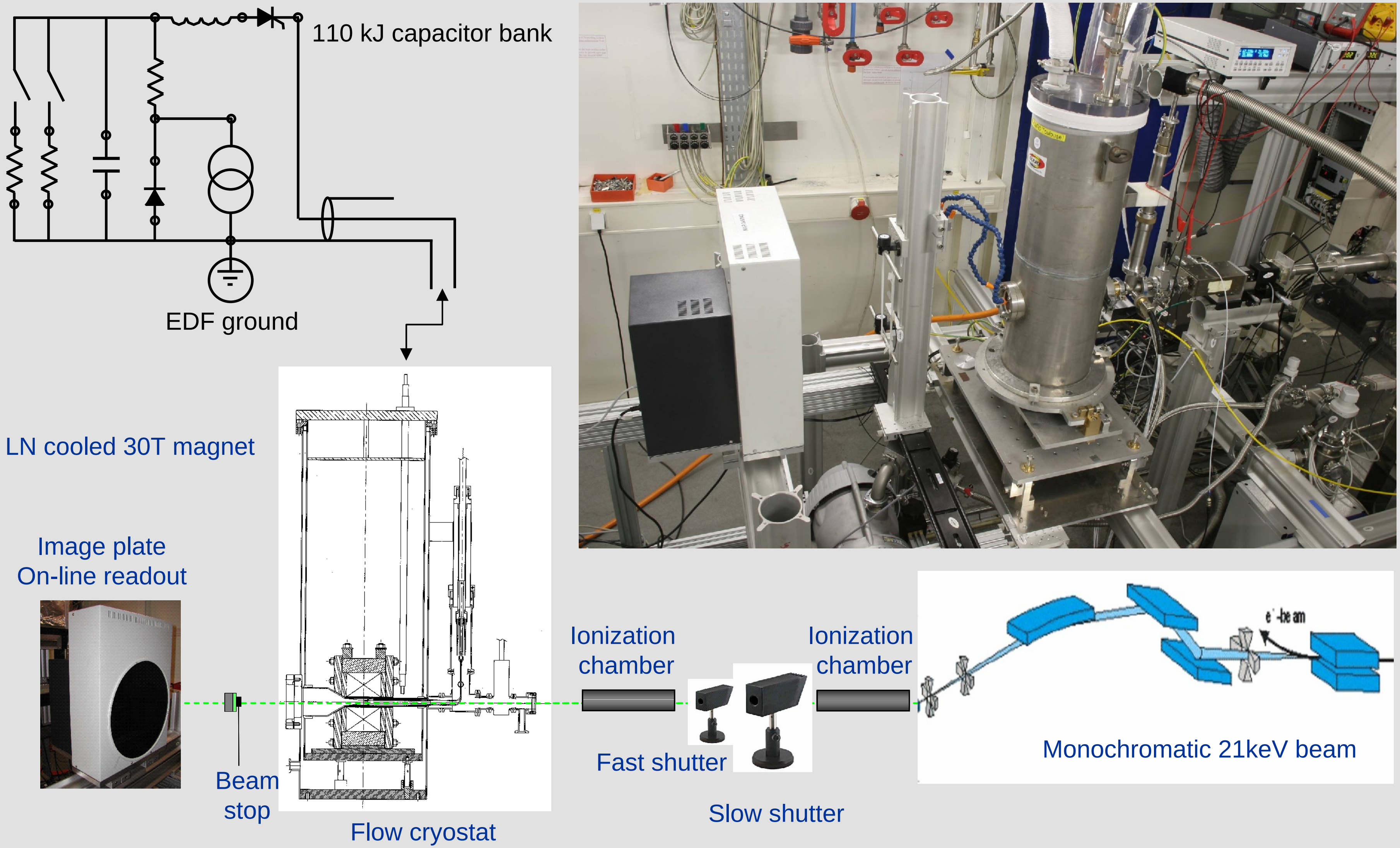
- Photons emitted from the 0.8T bending magnet
- Monochromatized by a Si(111) double crystal monochromator tuned to 21 keV (λ = 0.59 Å)



ESRF detector

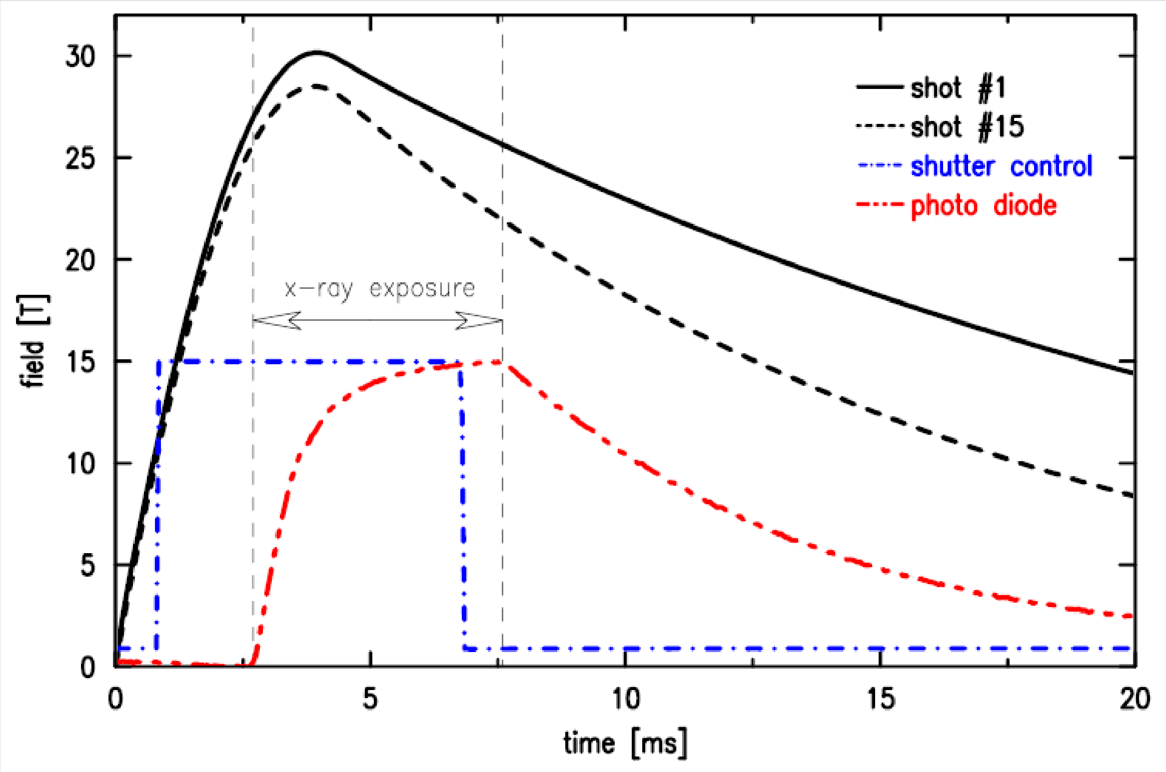
- on-line image plate (MAR345)
- resolution of the plate : 100 μm/pixels
- detector area: 345 mm x 345 mm
- Not time resolved !

Full experimental set-up



Data acquisition synchronization

- The magnetic field pulse and the X-ray shutters are synchronized by a common trigger
- Open/close beam after predefined delays
- Average magnetic field during the exposure is within 90% of its maximum value
- Time frames = 4.9ms
- Accumulation of series of pulses in order to obtain enough diffraction counts in the detector



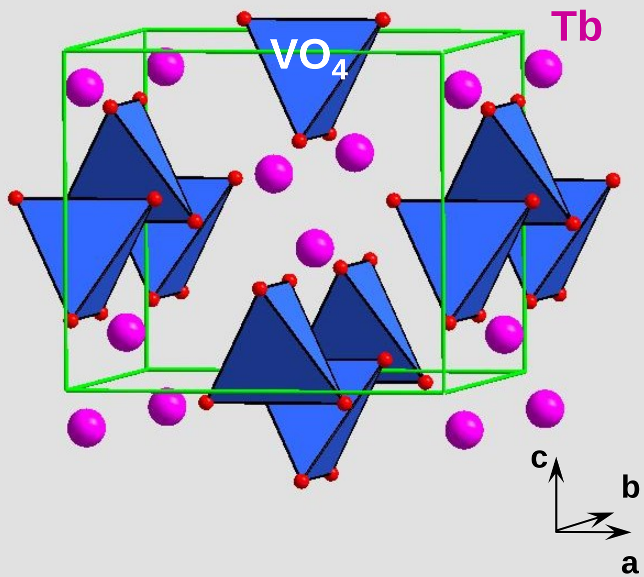
Data collection at one field :

- **30T** : 15 pulses in 15 min
- 45 min of cool down time } 1H00
- Real exposure time:

  ~ 15 x 5ms = 75ms

The Jahn-Teller compound $TbVO_4$

$TbVO_4$ ($REXO_4$)



- T_{JT} = 33.1 K
  Cooperative Jahn-Teller transition :
  Ordering of the quadrupolar moments of the RE ions

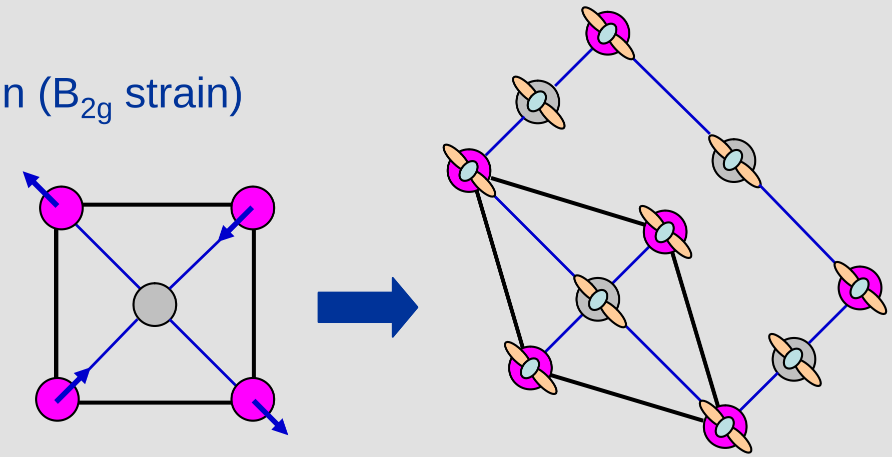
- T > 33.1 K
  Tetragonal zircon structure $I4_1/amd$ (D_{4h}^{19})

  a = 7.1841(3) Å
  c = 6.3310(4) Å

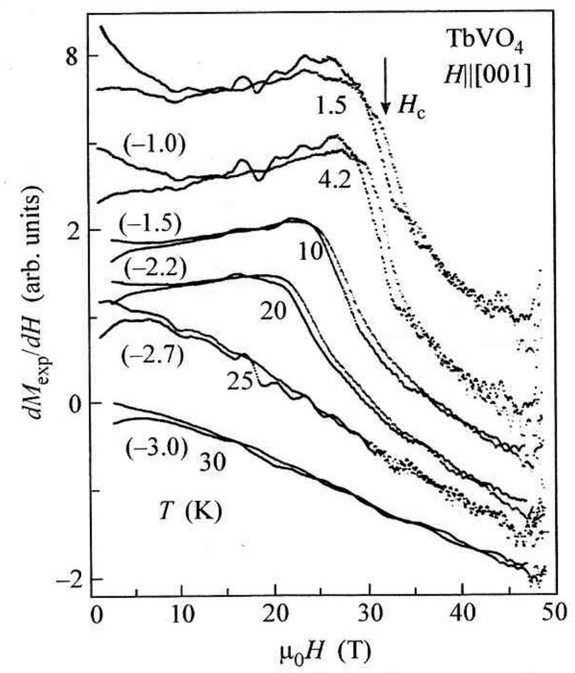
  Site symmetry of Tb^{3+}, $4f^8$: (D_{2d})

- T < 33.1K
  Crystal deformation along the [110] direction (B_{2g} strain)
  Orthorhombic structure Fddd (D_{2h}^{24})

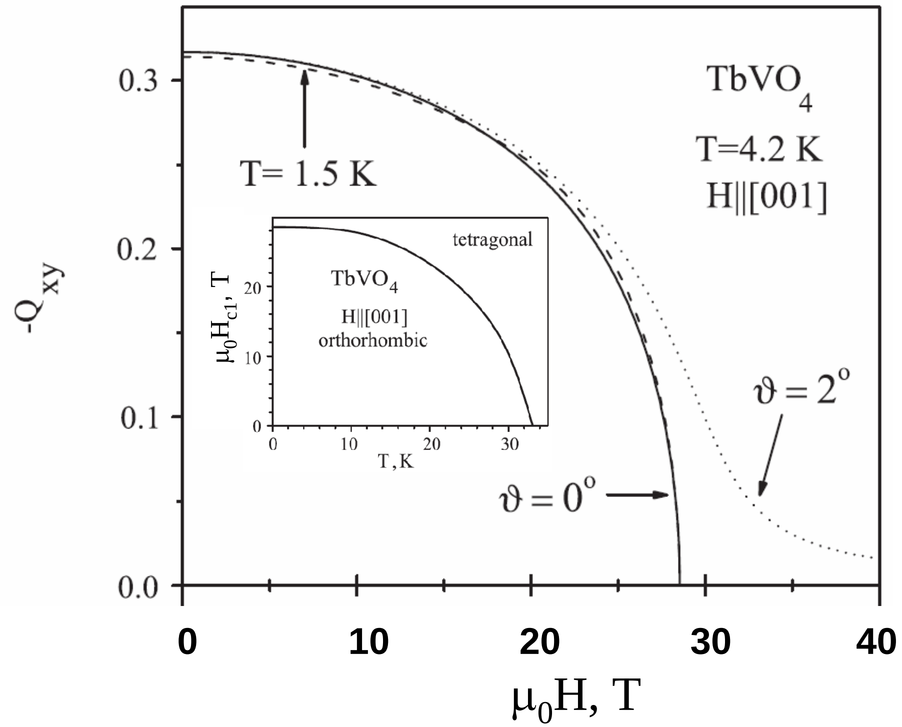
  a = 10.239(2) Å
  b = 10.029(2) Å
  c = 6.315(1) Å



The Jahn-Teller compound $TbVO_4$



Z.A. Kazei *et al.*, *JETP Letters* 82 (2005) 609-612



Demidov *et al.*, *Physica B* 363 (2005) 245-251

Effect of a strong magnetic field *B* // [001]

- Prediction of the destruction of the quadrupolar ordering

⟷ Suppression of the Jahn-Teller state of $TbVO_4$ by a field of 29 T

⇒ Inducing symmetry change from orthorhombic to tetragonal

Sample preparation

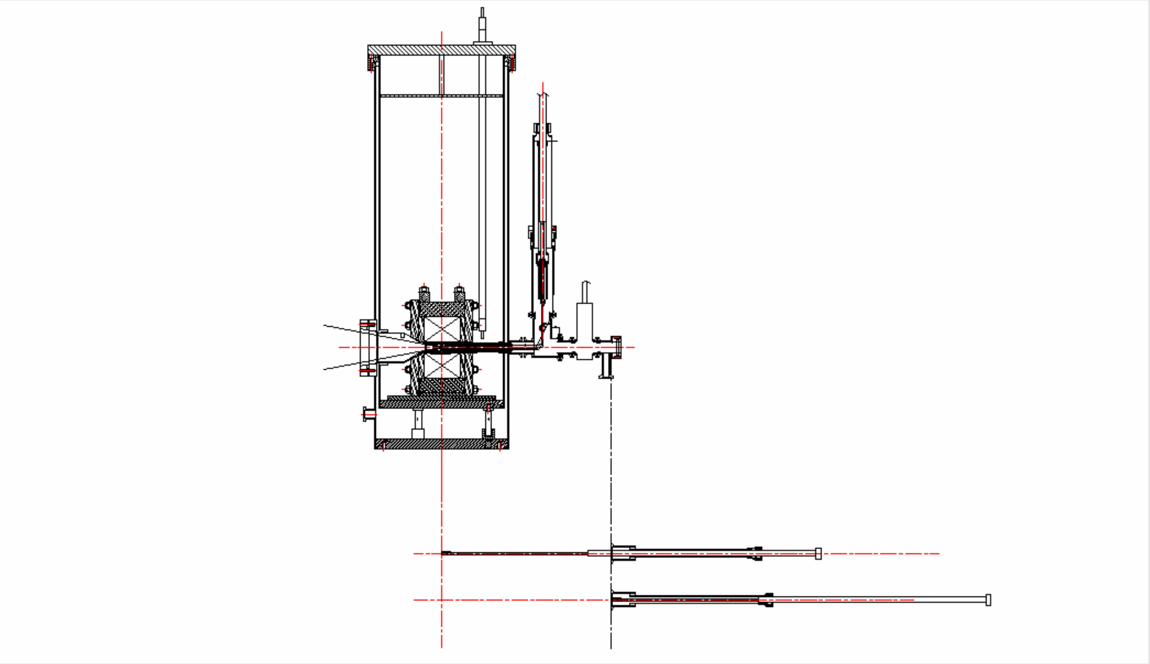
Flux-grown single crystals of $TbVO_4$ (P.C. Canfield, Ames Lab.), ground into a fine powder

- Random distribution of grain orientations

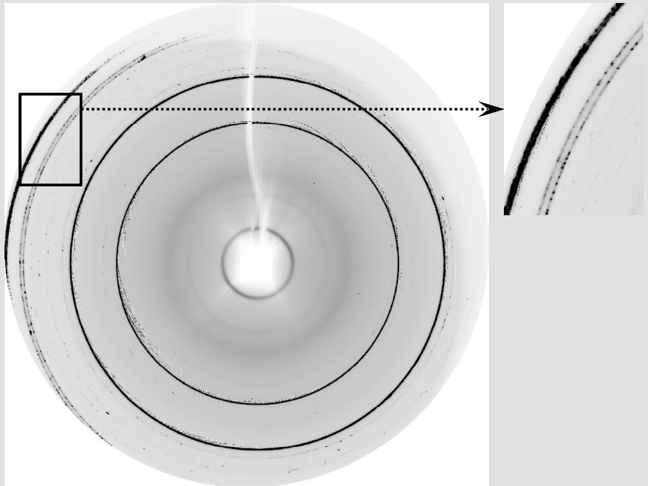
Embedded in a polymer matrix (polyvinylpyrrolidone)

- Reduce movement of the powder grains
- Improve thermal contact

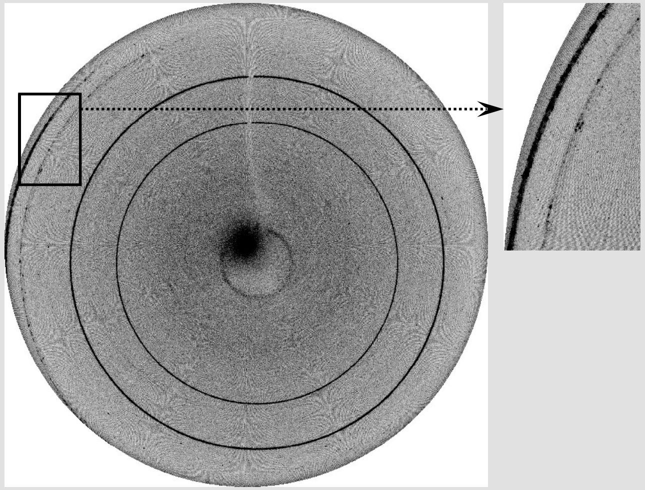
Thickness of the pellets adjusted to limit X-ray absorption (pellets of 4 mm Ø)



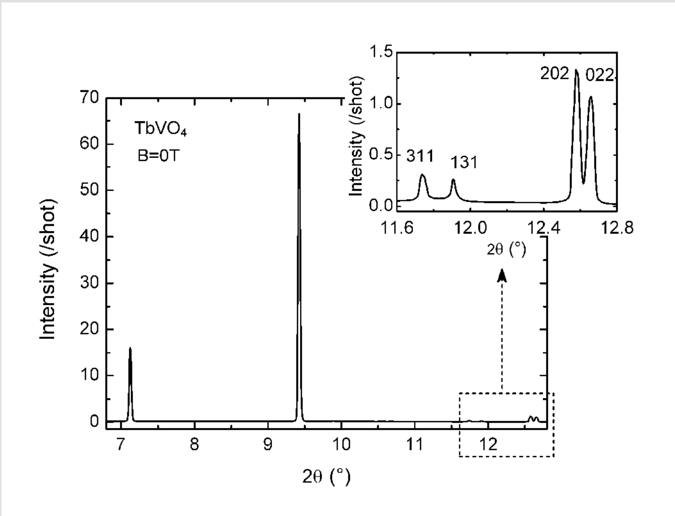
Pulsed field diagrams

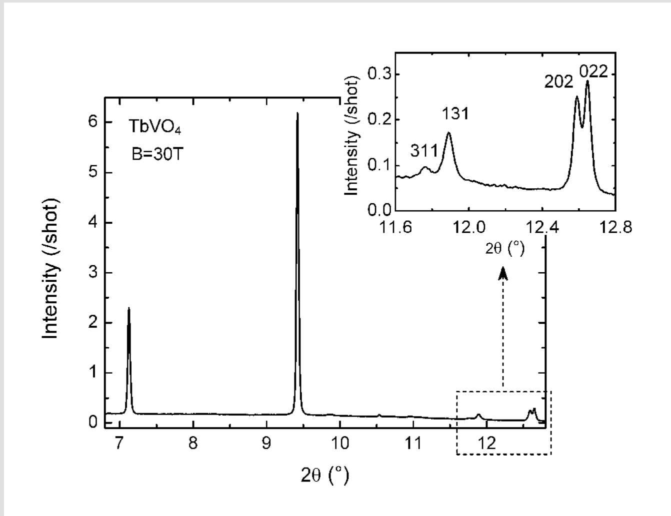


B = 0 T, *T* = 7.5 K, exposure time : 60s



B = 30 T, *T* = 7.5 K, exposure time : ~75ms

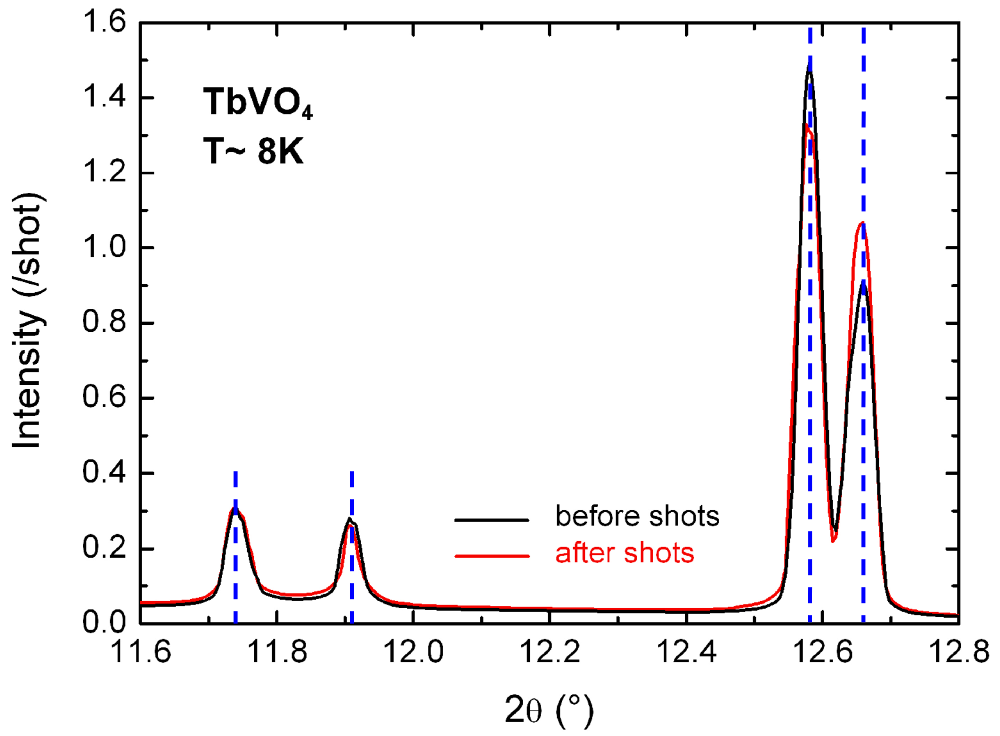




Data normalized to one shot, i.e. 5ms

Reproducibility

- Control of change in distribution by **zero field measurement** before and after applying series of *B* = 30 T magnetic field shots:

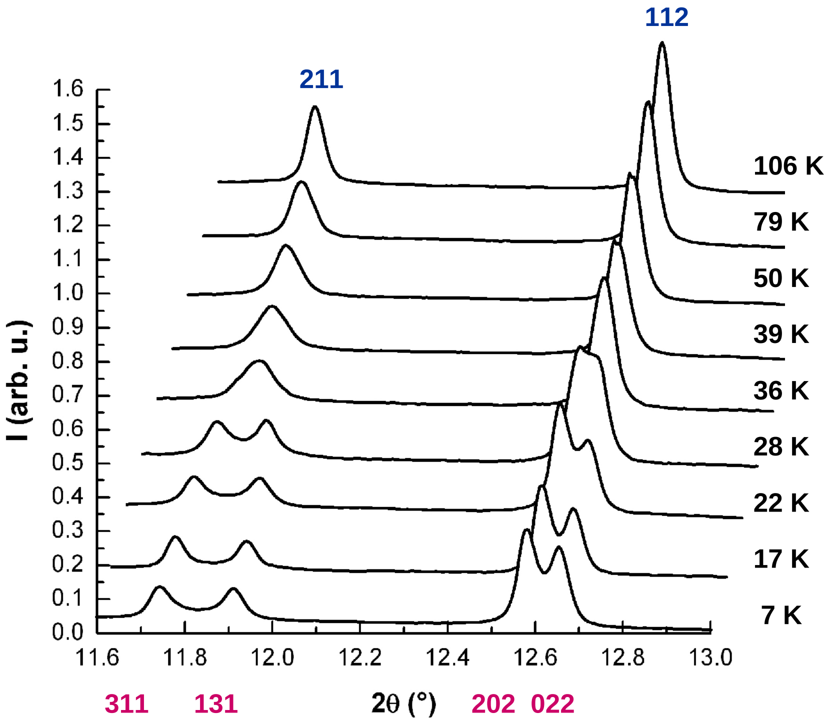


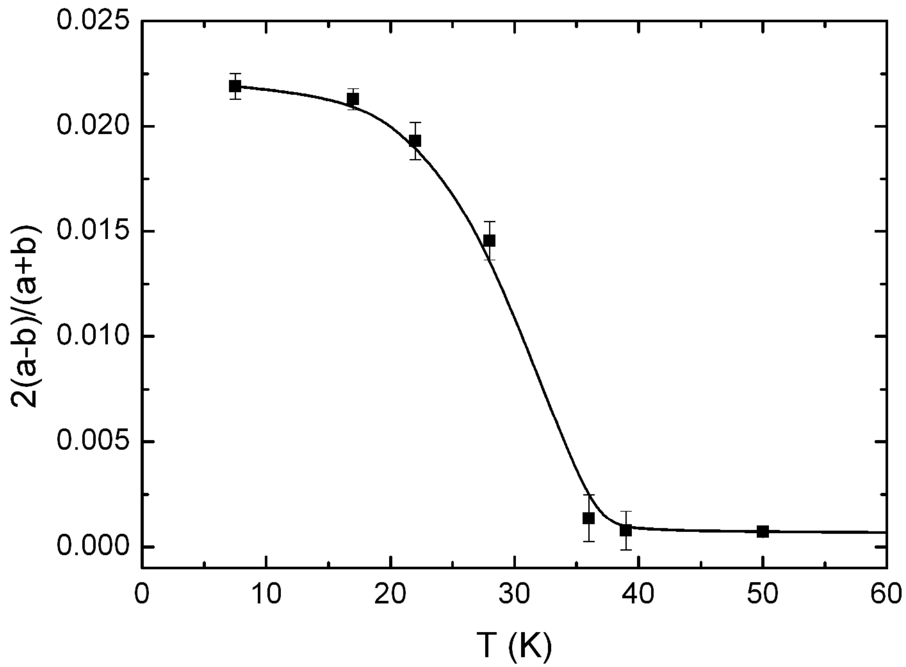
- No shift of diffraction peaks
- Small variation in peak amplitude

Structural changes with temperature
The Jahn-Teller transition in $TbVO_4$

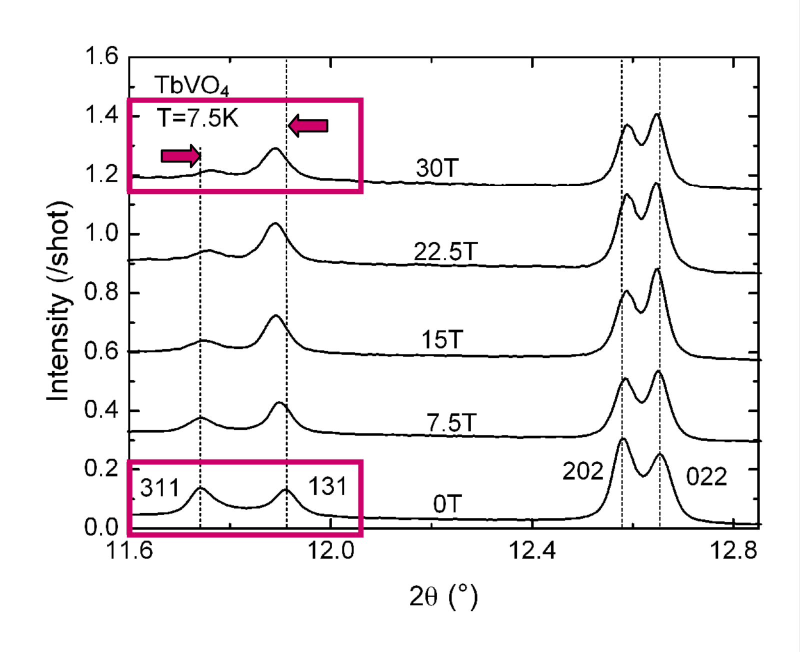
Temperature dependence of diffraction patterns

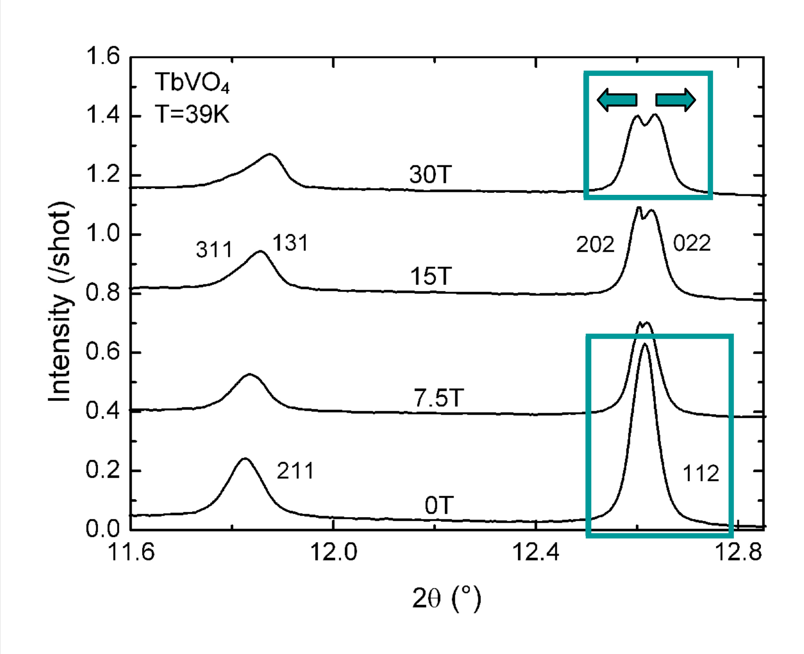
Orthorhombic distortion





Field induced structural changes





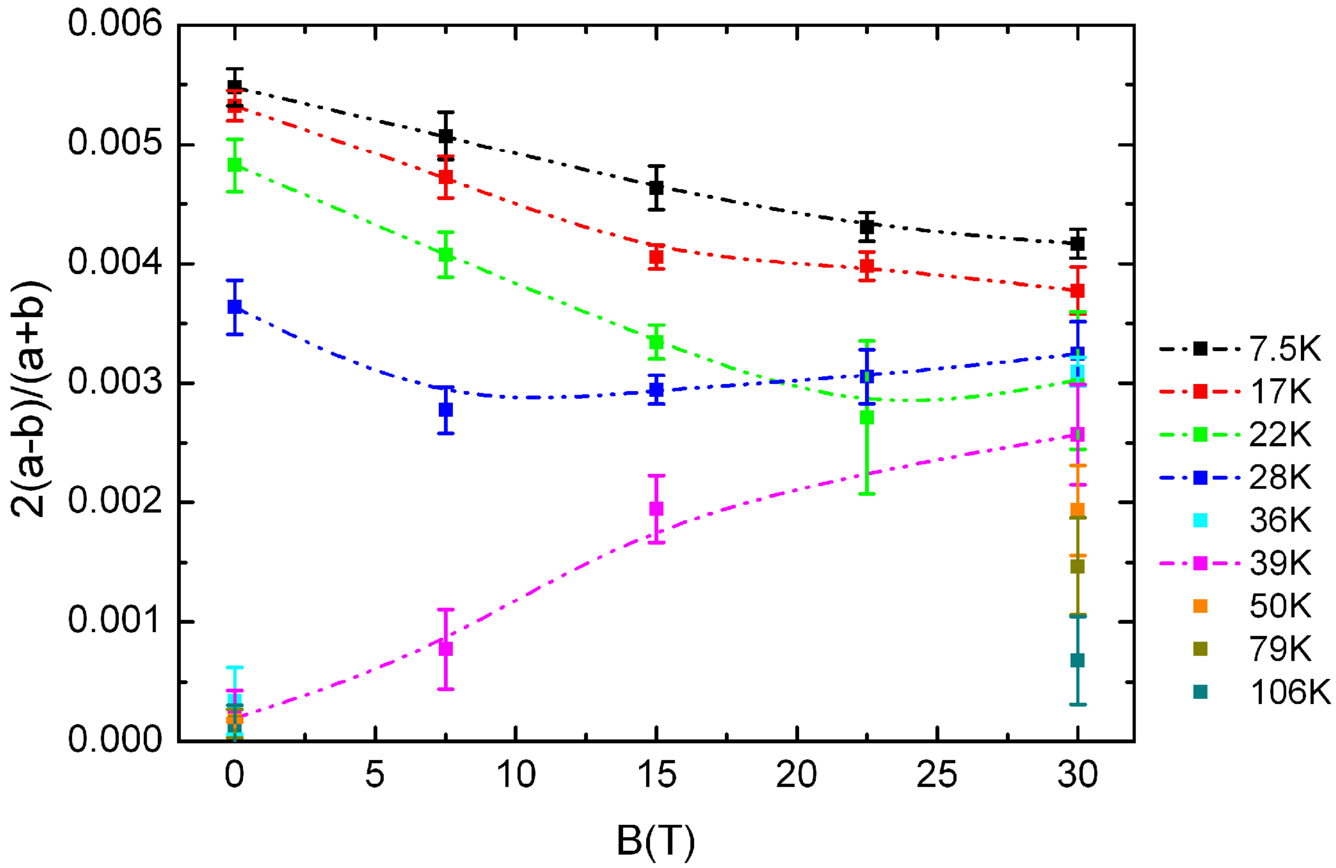
- ***B* = 0T, *T* = 7.5 K → Orthorhombic**
  (311) + (131)
  (202) + (022)
- ***B* ≠ 0T, *T* = 7.5 K**
  Preferential population of domains
  In-plane magneto-crystalline anisotropy
  Splitting decreases with increasing ***B***

  → ***B* decreases the ortho. distortion**

- ***B* = 0T, *T* = 39K → Tetragonal**
  (211)
  (112)
- ***B* ≠ 0T, *T* = 39K**
  Splitting appears with ***B***
  Preferential domain population

  → ***B* induces the ortho. distortion**

Field induced structural changes



Orthorhombic distortion as a function of applied field

The Jahn-Teller compound $TbVO_4$

Quantitative analysis of the data

- Theory:
  ***B* // [001]** suppress the distortion
  ***B* // [110]** enhance the effect

- $TbVO_4$: sample with large magneto-crystalline anisotropy
  The *H-T* phase diagram depends on: - the magnitude of ***B***
  - the orientation of ***B*** relative to the symmetry axes

- X-ray powder diffraction:
  Direct measure of the Jahn-Teller distortion (lattice parameters, symmetry changes)
  Random distribution of the powder grains with respect to the external field
  ⟶ in some grains the JT-effect is enhanced
  ⟶ in others it is suppressed

This had to be taken into account in the data analysis

Future technological developments

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Final goal of the project

Provide the highest magnetic fields possible within the constraints imposed by the synchrotron x-ray beamline

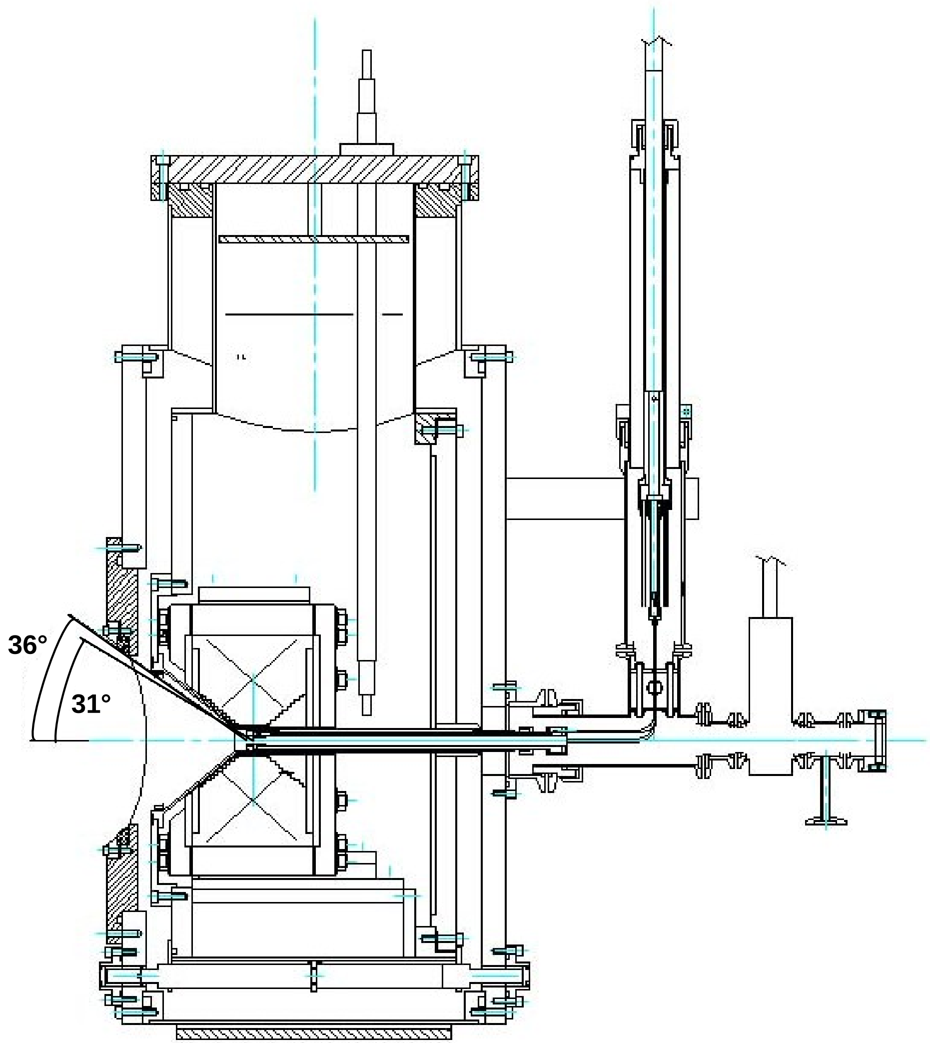
Technological developments

- Higher fields, up to 40 T
- New design of the magnet coil

  1) Increase the opening angle of the coil in the Faraday geometry (*B* // beam)

  2) Split pair coil (*B* $\perp$ beam): wider optical access angles
- Lower temperatures with better temperature control
- Automation of the operation mode
- Longer field pulses to facilitate the synchronization with shutters and detection electronics
- Fast x-ray detector (frame rate of 1 kHz or better)

Pulsed field and cryogenics: new design



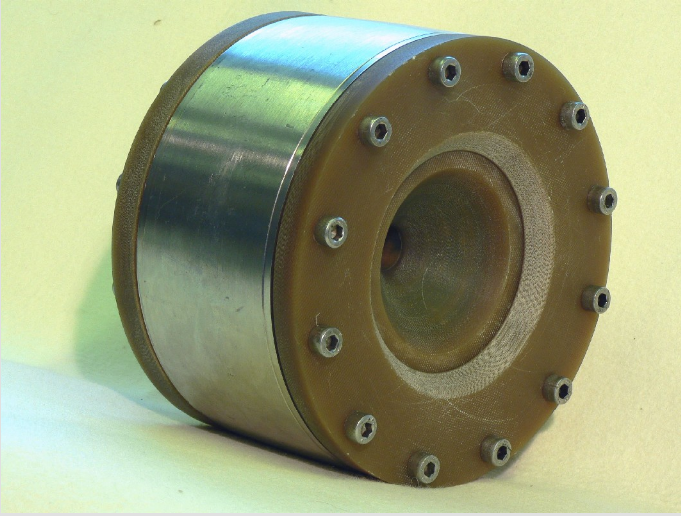
New magnet coil

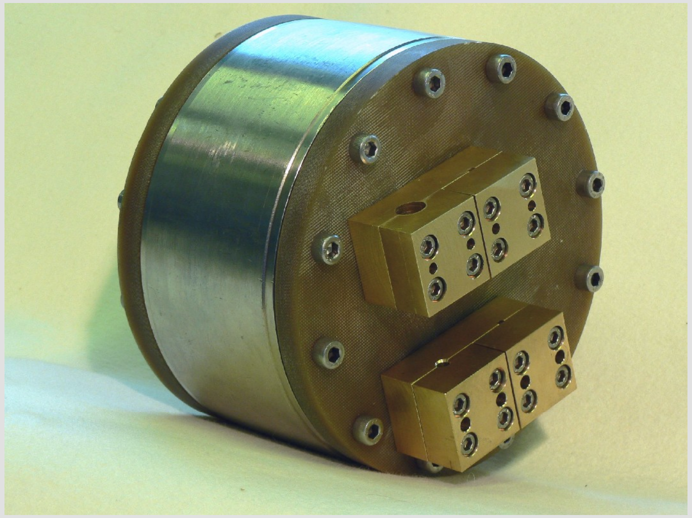
- coil is wound of CuNbTi wire
- bore of 20 mm Ø, external Ø = 130 mm, height = 80 mm
- R(T=77K) = 60 mΩ
- Opening angle of the coil: 40°
- coil is horizontally mounted
- ***B*** // beam (Faraday geometry)
- magnet immersed in liquid nitrogen

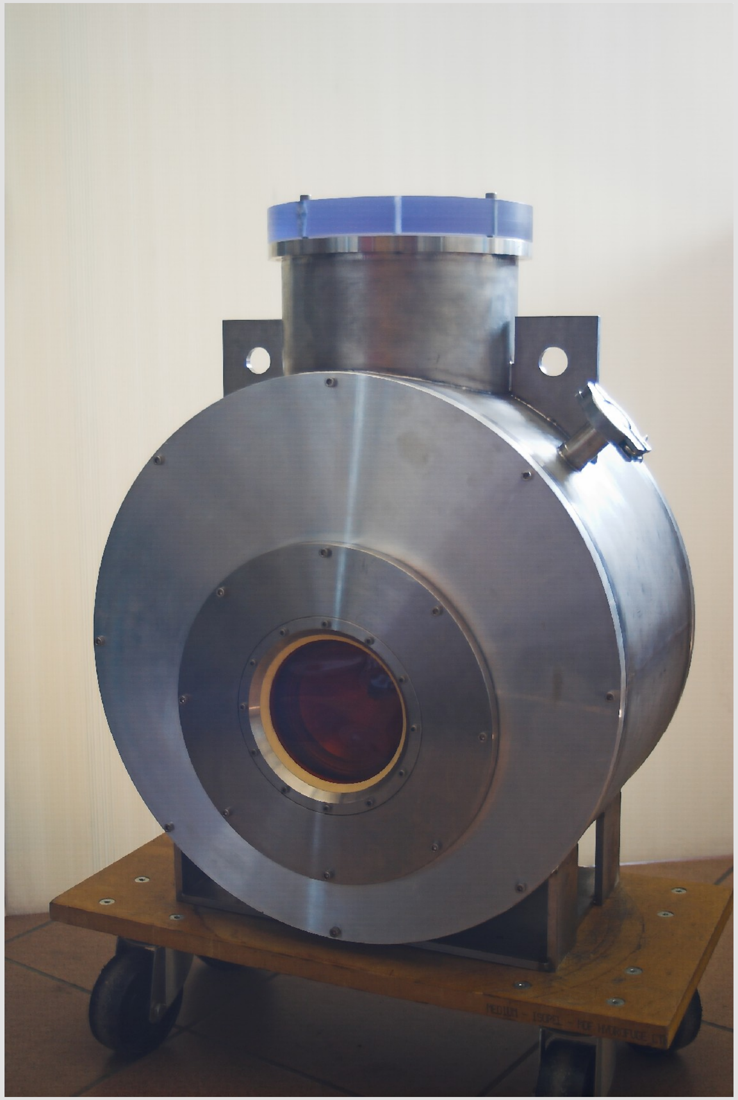
New N_2 cryostat

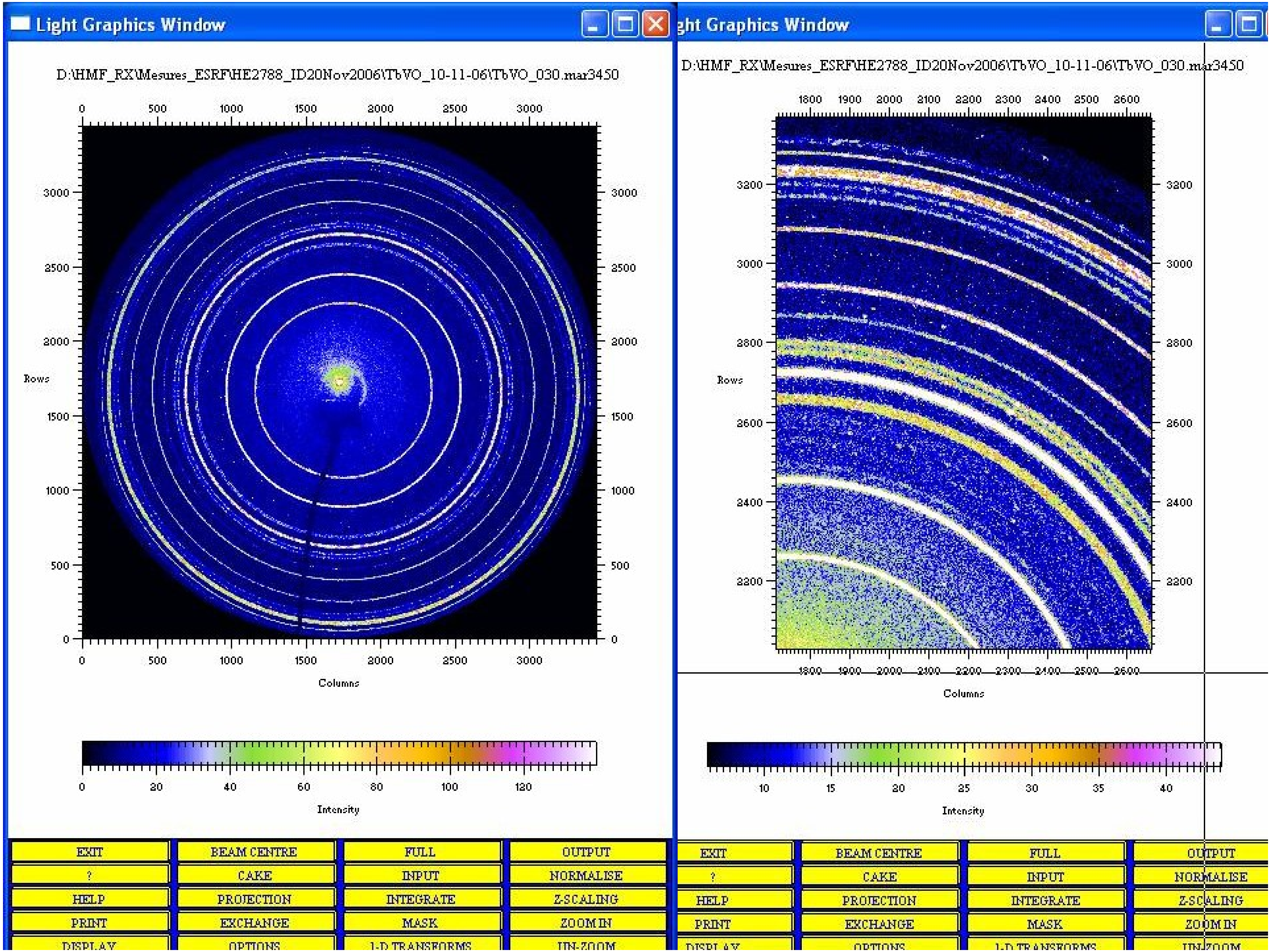
- 2 Kapton foils (120 μm thickness)
- Optical access angles: up to 31°/36°

Pulsed field and cryogenics: new design









Light Graphics Window
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CAKE
INPUT
NORMALISE
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PROJECTION
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MASK
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Rows
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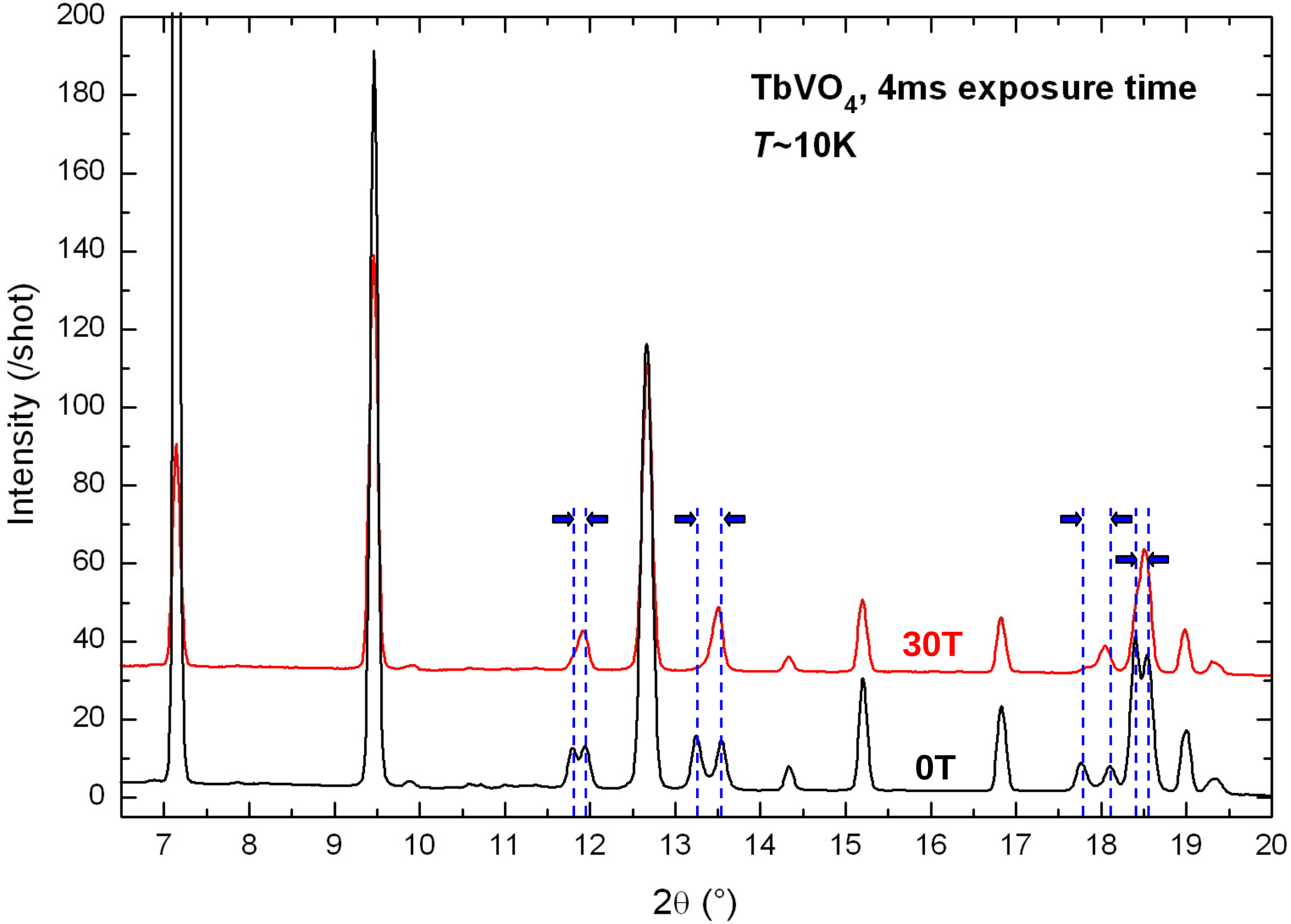
Diffraction pattern on $TbVO_4$



Future developments and applications

Future applications

- Mobile experimental device, can be used on a non-dedicated beamline (ESRF, SOLEIL)
- Other X-ray techniques: Spectroscopy (EXAFS, XMCD)
  Powder and single crystal diffraction (Laue Diffraction)
- Field induced magneto-structural transitions: magneto-caloric compounds, manganites, multiferroïcs, frustrated systems, superconductors, metamagnetic transitions, etc …

New beamline at the ESRF: ID06 will become operational at the end of 2007

ANR project: SysMAF (2006-2008)
Diffraction et Spectroscopie des rayons X synchrotron sous champ magnétique intense

→ **Post-doctoral position available at the LNCMP**

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Acknowledgements

- NWO/FWO Vlaanderen and ESRF : beamtime allocation
- DUBBLE CRG staff : F. Meneau, D. Detollenaere, J. Jacobs
- ESRF Safety group
- P. Van der Linden : cryogenics support
- ESRF detector pool and T. Buslaps (ESRF, ID15 beamline) : X-ray detector
- P.C. Canfield (Ames Laboratory, Ames, IA) : $TbVO_4$ samples

Thank you for your attention !