We have input from:

- PGAA, NePoMuc, Hereon (Solis), Powder diffractometers, Stress-Spec, AdvMat-group, Tof-Tof, Imaging

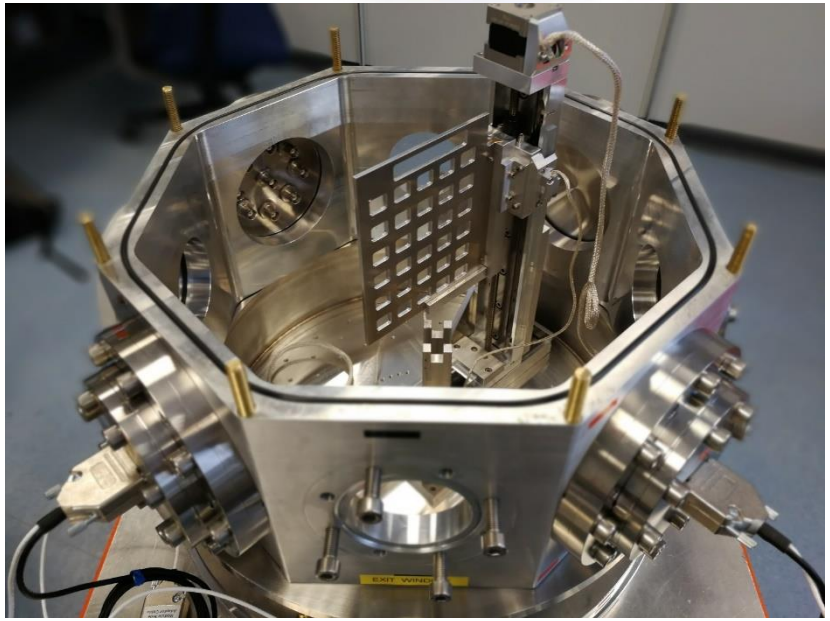
- A large number of instrument specific SE exists

- In addition, a large variety of SE needs is evident where the SE group would need to be involved, besides the supply and maintenance of standard equipment.

  (1) HT equipment – control rack, measurements at different atmospheres
  (2) Transport boxes for sensitive materials (i.e. batteries), glove box in E-Halle
  (3) Advanced LT equipment (ADR cryostats) - including sample changer
  (4) Load rig upgrades – support here for LT/HT measurements
  (5) High pressure equipment (gas pressure cells) + infrastructure
  (6) Humidity measurements or environment?

PGAA and NDP Sample Environment Needs

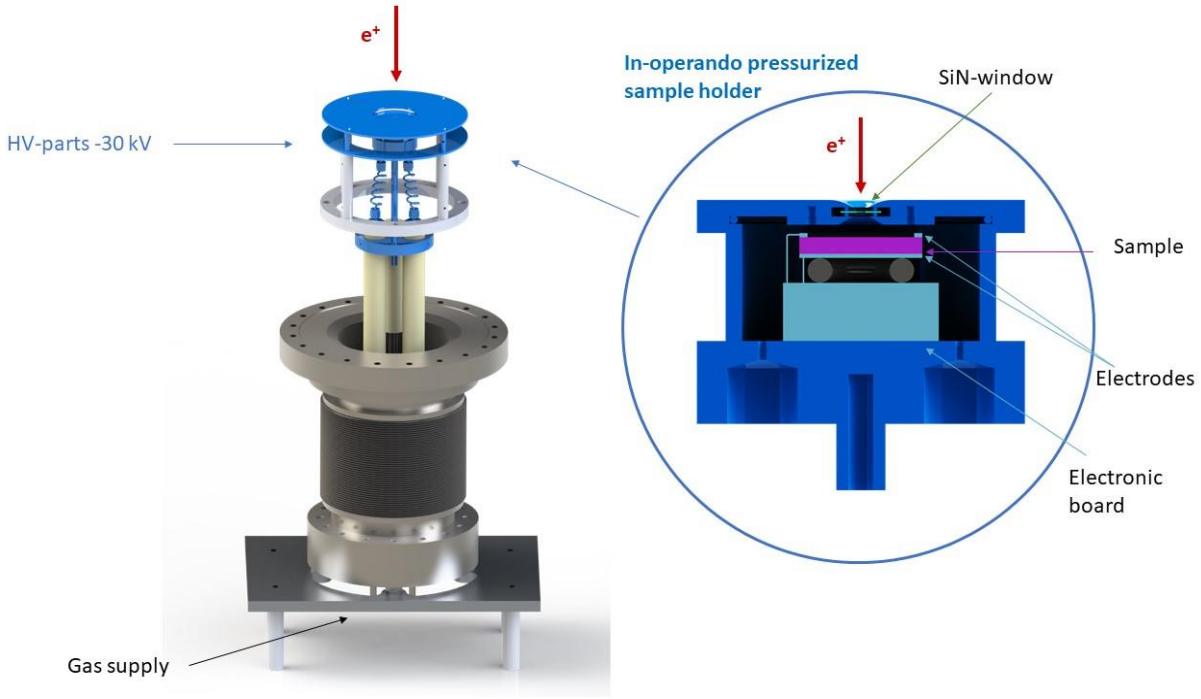
- PGAA needs only vacuum (<0.01mbar), it is available
- NDP needs, they are available at PGAA
  - Vacuum (<10^{-5} mbar) or He atmosphere in the chmaber
  - Cooler (Jubalo, <-20°C)
  - Portable potentiostate
- NDP needs, maybe interesting for other instrumenst, too
  - Evacuated or argon-filled transfer boxes (more of them) to transport the sensitive (flammable) samples (typically Li-ion batteries) between the sample preparation lab and the NDP measurement.
    - It should be compatible with other instruments, too.
    - The chamber needs to be modified to dock this box and to move the sample to sample position



NEPOMUC – CDBS Sample Environment Needs

- Positron beam experiments require ultra-high vacuum (< 10^{-7} mbar)
- Sample needs to be at high voltage (up to -30 kV)

- Sample environment (planned, partially realized)
  - High T → upto 1300 K
  - Low T → down to ~10 K
  - Higher gas atmosphere at sample upto 2 bar
  - Humid environment at sample
  - Application of electric fields / various potentials



- Sample transfer
  - Evacuated or argon-filled transfer boxes (more of them) to transport the sensitive (flammable) samples

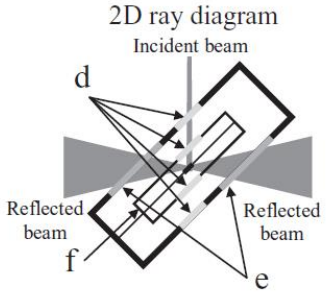
Cryogenic load frame

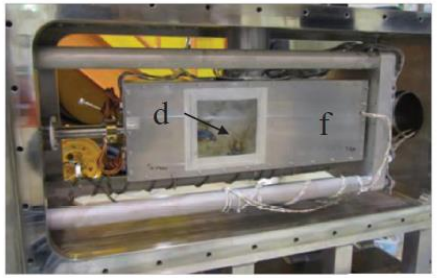
In situ mechanical behavior at cryogenic temperatures
Applications: cryogenic texture processing of zirconium nuclear alloys, strain sensitivity of superconducting magnet wires, cryogenic structural steels and low temperature shape memory alloys for space applications materials

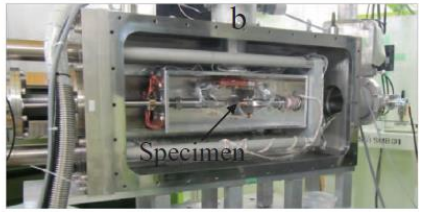
Neutron Diffraction @STRESS-SPEC
+ compatible with SANS @SANS1
+ compatible with imaging for Bragg edge imaging and nGI

Temperatures below 75 K
Applied load up to 50 kN
+ rotation??

@JParck

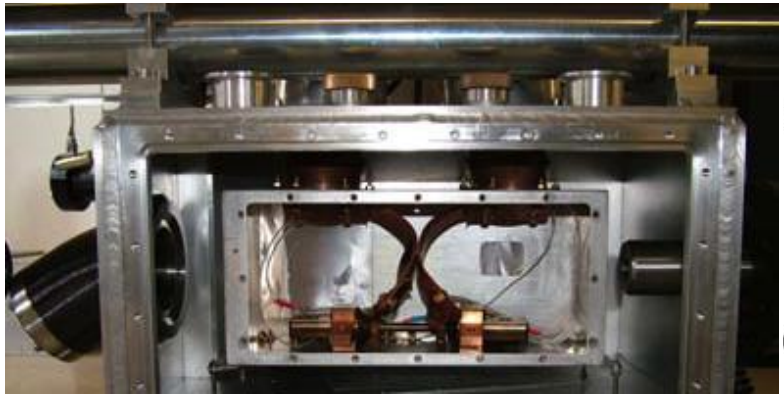


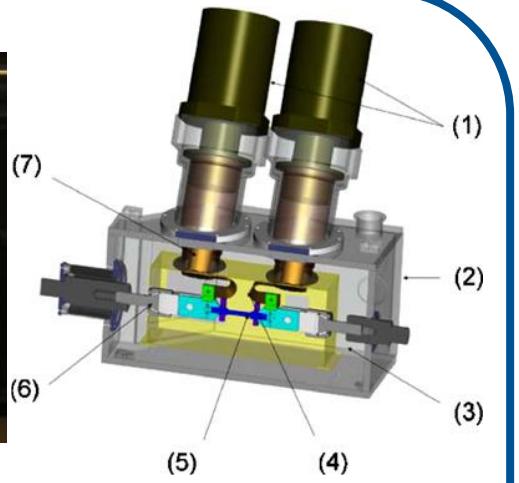




(a) vacuum vessel, (b) GM refrigerator, (c) loading driver, (d) vanadium windows, (e) aluminum windows, and (f) thermal shield.

@ISIS



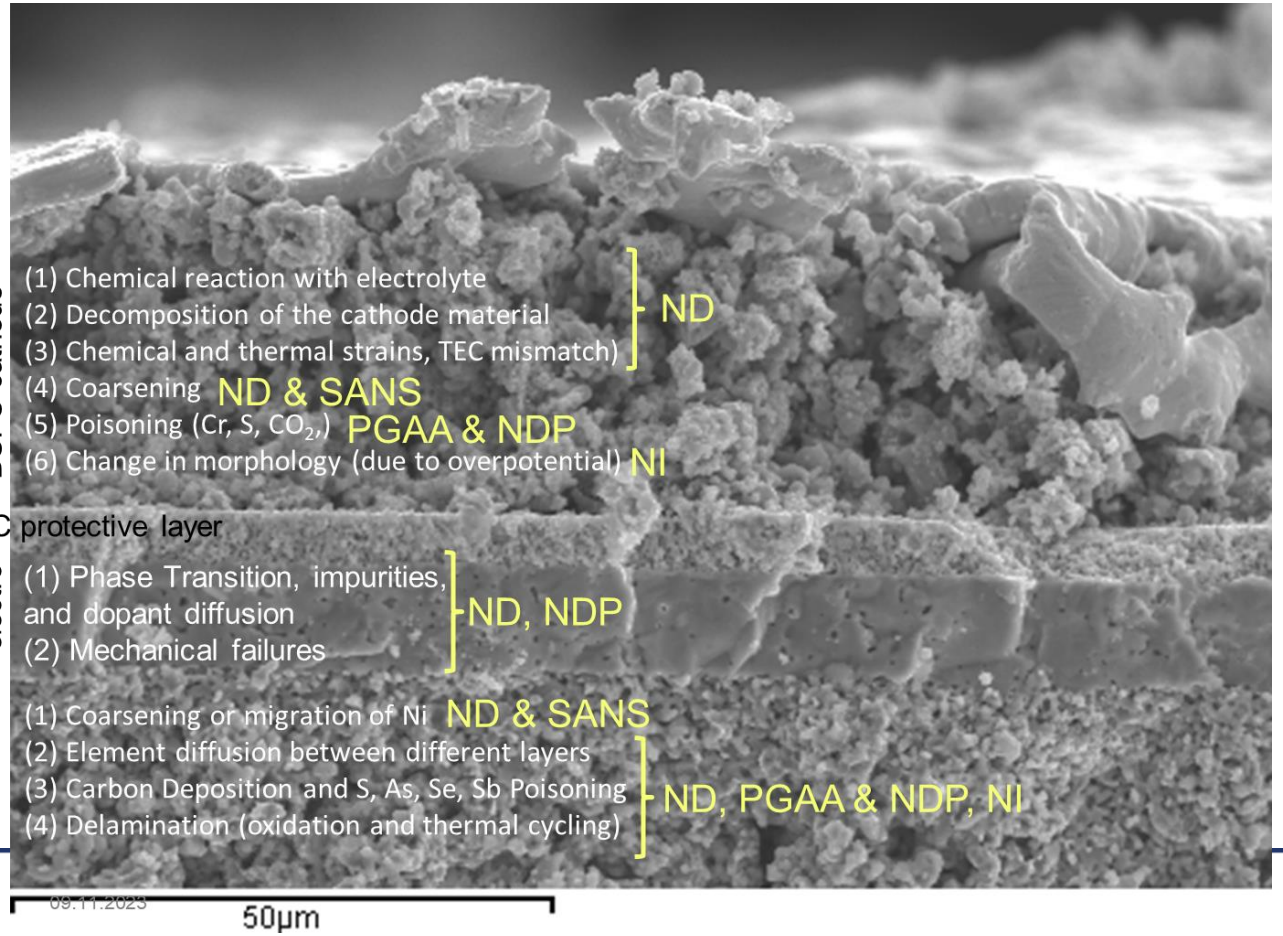


(1) two Leybold 1245 CCRs; (2) an outer vacuum vessel with aluminium windows to provide access for the incident and scattered neutrons; (3) the infrared radiation shield which is connected by thermal links (7) to the first stages of the CCRs; (4) the thermal links connecting the CCR second stages to the rig jaws; (5) the test sample; and (6) the 50 kN hydraulic stress rig.

Electrochemical cell

In situ electrochemical characterization
Applications: electrodes for high temperature fuel cells and electrolzyers, gas separation membranes and electrochemical reactors

Neutron Diffraction @STRESS-SPEC
+ compatible with SANS @SANS1?
+ compatible with imaging → tomography (+ rotation)



Temperatures up to 500 ºC or more
Differemt atmospheres: O_2, air, H_2, CO...
wires

Diffraction: phase transformations and chemical stability, chemical and thermal strains at real operation condictions, proton diffusion…

SANS: nanocatalyst exsolution and nanocatalyst coarsening

Imaging: water / hydrogen / Li distribution

Sample Environment for Powder Diffraction

Sample environment already in use for SPODI

- CCR cryostats (SPODI equipment, modified FRM II standard)
- High-temperature furnace (SPODI equipment, FRM II standard)
- Automatic sample changer (SPODI equipment)
- Potentiostats for batteries (SPODI equipment)
- Tensile rig (SPODI equipment)
- Electric field setup (SPODI equipment)
- 5.5 T magnet (from FRM II pool)

Operation of ERWIN and FIREPOD besides SPODI will enhance the demand for sample environment

→ Common pool for three diffractometers

→ Supply of standard sample environment equipment

→ Maintainance of standard sample environment equipment

Projects with sample environment group

- Completion of the cryogenic sample changer for CCR
- Use of robotic arms on cryostats - automated sample stick exchange
- High temperature measurements at different atmospheres, including gas flow rates using an exchange sample stick

Requirements for sample environment group

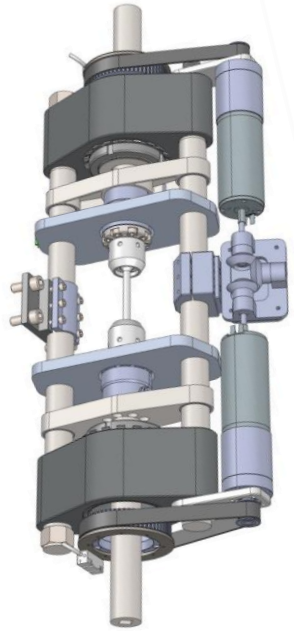
- Maintenance of cryostats
- Maintenance of high-temperature furnaces
- Support in the "automated sample change at low temperatures" projects
- Establishment of ADR cryostats and support to enable automated measurements
- Support in the project "High temperature measurements at different atmospheres“
- Support in the use of magnets

Sample environment used already at STRESS-SPEC

- High temperature furnaces (up to 1550° C)
- *Mirror furnace (own devices)*
- *Load rigs (own devices)*
- Cryostats (occasionally)
- Adapted devices for batteries (occasionally)

Projects/Sample environment needs

- Laser furnace as replacement of mirror furnace
- Common HT control rack – **SE/Instrument control groups were involved from the start**
  - Modular design to control different types of HT equipment
  - Would be useful to have also for all other HT applications/instruments
- Light weight load frame (50 kN) for robot
- Additional Heating and cooling capacities for load rigs

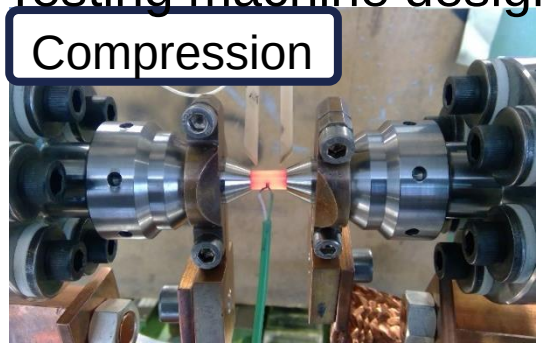


Light weight tensile rig
(< 30kg)

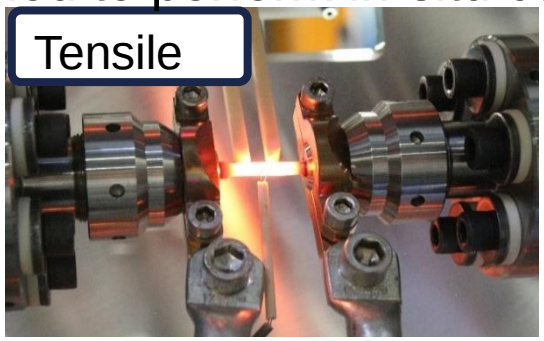
Laser furnace for 100kN testing machine

Testing machine designed to perform in-situ compression, tensile and fatigue experiments

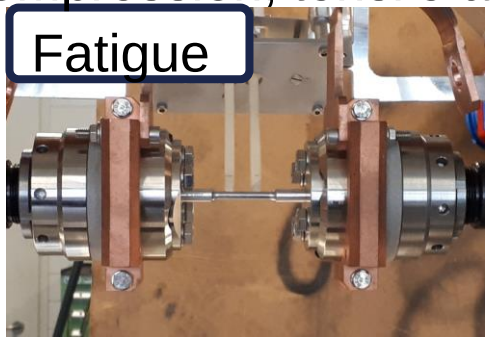
Compression



Tensile



Fatigue



Sample environment for:

- STRESS SPEC
- SANS-1
- ANTARES

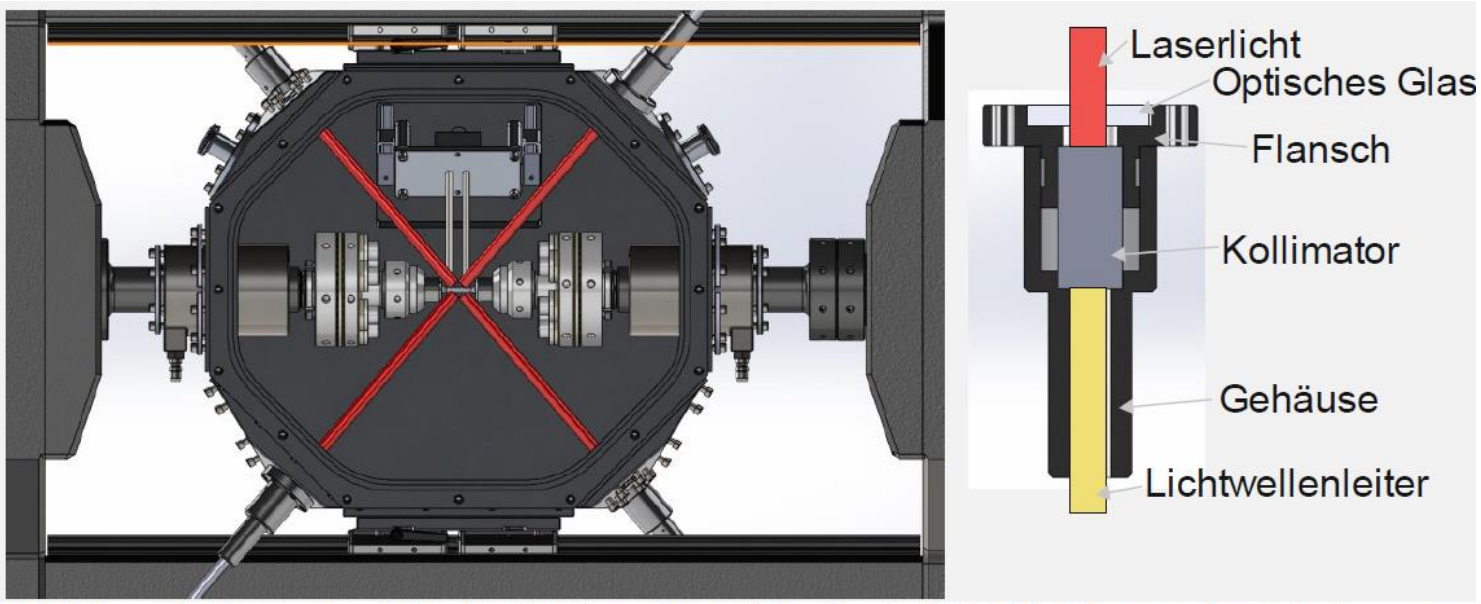


Abbildung 1: Gesamtansicht der Prüfmaschine (oben) mit CAD-Zeichnung der Ofenkammer mit Strahlenoptik sowie der Kollimatorbefestigung (unten links bzw. rechts).

For samples with high conductivity that cannot be brought to high temperatures with conventional resistance heating.
Four infrared lasers with 70 W each provide the power to heat common samples up to 1200 °C.

-> Control rack for laser heating has to be built

Electrochemistry infrastructure at FRM II

- Sample preparation:
  - Ar Glovebox with mg scale, pouch cell sealer, coin cell crimper, spin coater
  - Büchi Vacuum oven(s) for pre drying of cell parts, electrodes, separators
  - Electrochemical cells for transmission/flexion geometry (SANS/GISANS), 2 and 3 electrode versions
  - Transfer port for vacuum system on Glovebox
- Experiment:
  - Potentiostats with TTL connection to beamline
  - Cell holders (reusable cells, pouch cells) for sample stages
  - Vacuum suitcase and transfer system on PGAA for inert sample loading
  - Heating system (<120°C) for Electrochemical cells (relevant for polymer batteries)

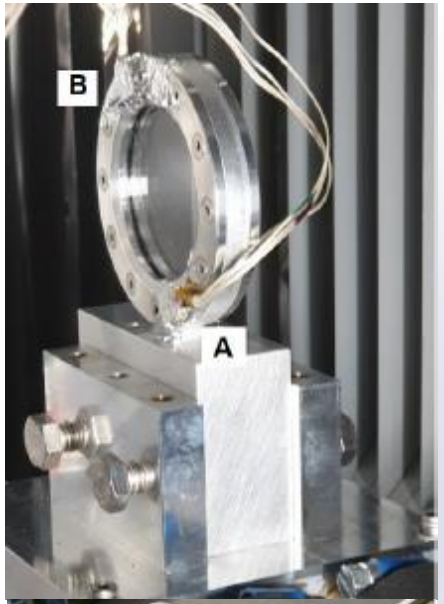
Target Beamlines:
SANS-1
PGAA
Stress Spec
SPODI
ERWIN?

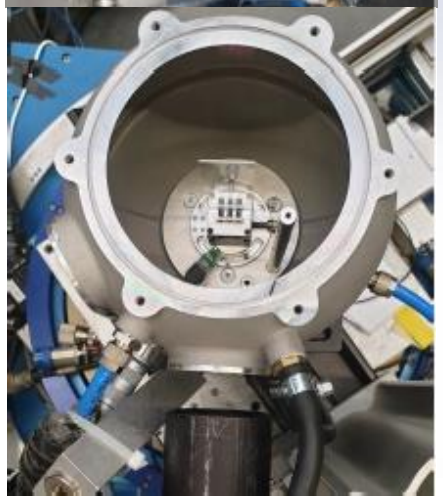
TOFTOF

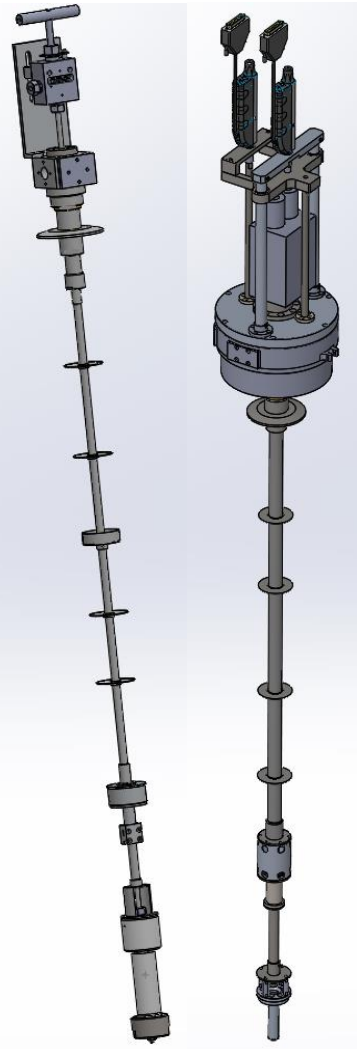
- Requirements for the sample environment group

  Maintenance and support of already existing sample environments:

  - Cryostat (CCR-17)
  - Dilution unit
  - High temperature furnace
  - 2.2 T magnet
- Actual projects:
  - Raman spectroscopy in situ with neutrons
    - Optical cell (10 – 100 °C), available, not tested with neutrons
    - Sample stick, CCR-17, under construction
  - High pressure
    - Up to 200 MPa (10 – 100°C), available
    - Up to 500 MPa, CCR-17, under construction
  - Humidity measurements (membranes and thin films)
    - Up to now humidity generator from E13 will be used
    - Humidity chambers for TOFTOF, LaDiff, and SANS / RefSANS (E13) available
    - Possibility to combine with Raman for TOFTOF and LaDiff







TOFTOF

- Future projects:
  - Humidity measurements
    - Measurement of different kind of humidities (H_2O, D_2O, Ethanol, …) and there mixtures and logging them with NICOS
    - Design of new humidity generator:
      - Controlling over NICOS
      - Designed for transportation
  - High pressure:
    - Using the motor-drive pressure pump from sample environment group at TOFTOF with NICOS

- Scientific areas:

  Material science, batteries, soft matter, magnetism

Sample Environment at ANTARES

General requirement: **Short distance between sample and detector** for high spatial resolution

- Most pool sample environment not optimized for neutron imaging (too bulky)
- This will probably not change…

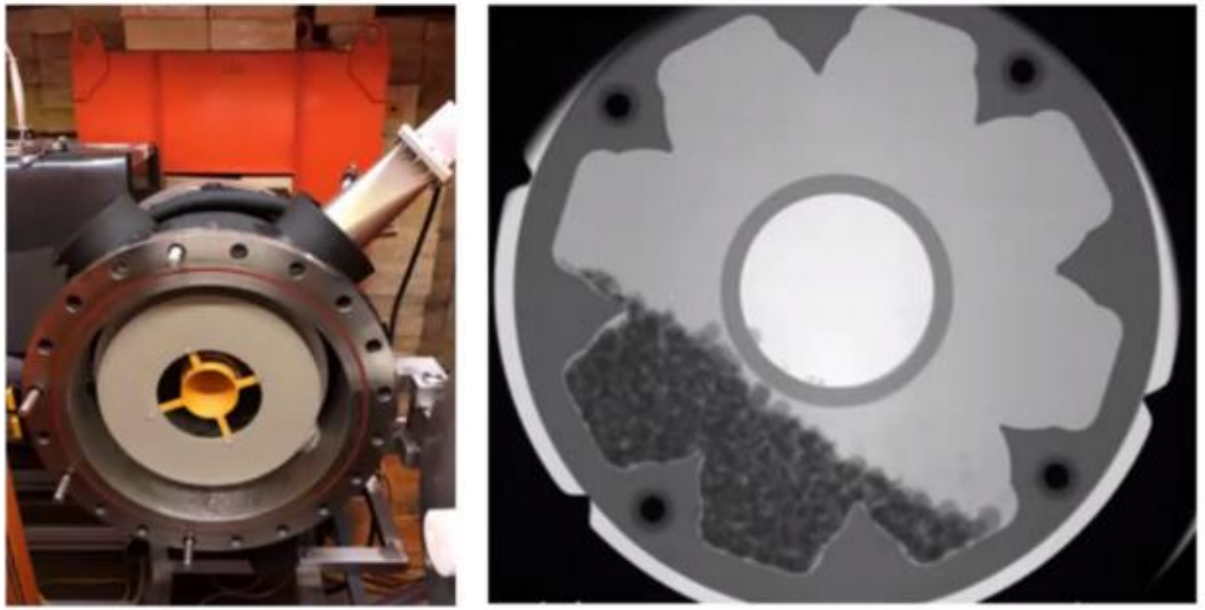
Existing *dedicated* sample environment at ANTARES:

- Bottom-loading ^{3}He cryostat (500mK)
- CCR with 50mm sample tube diameter (4K)
- CC from PUMA (4K)
- Custom vacuum furnace for H-diffusion in metal, magnetism, etc. (up to 400°C, no dedicated control box, need to use control box of „battery furnace“)
- TUM Tensile rig designed in collaboration with M. Hofmann, R. Gilles, P. Jüttner
- Helmholtz magnet (0.4T, normal conducting) for polarized neutrons + nGI

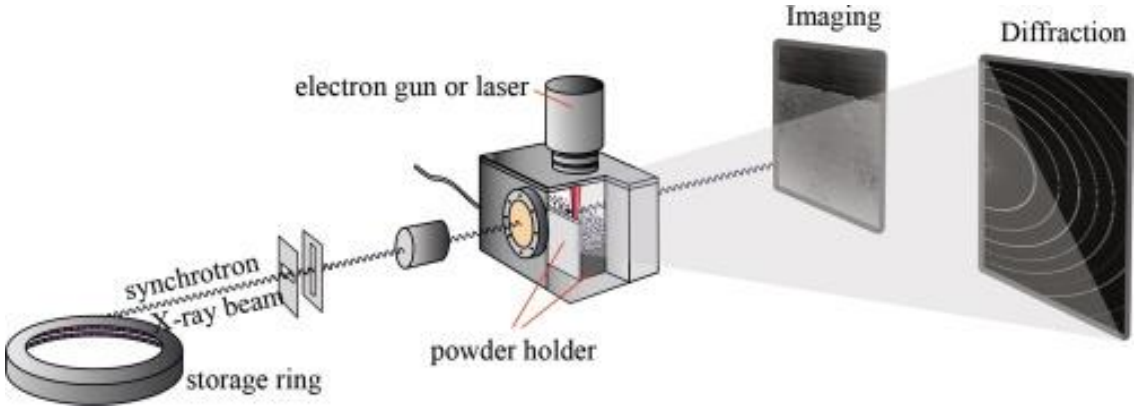
→ We are well-equipped but mostly independent of pool equipment

Future Plans for SE at ANTARES

- Setup for in-situ microwave freeze-drying experiments (designed by M. Hilmer, TUM Weihenstephan)
- Upgrade tensile rig for tomography?
- In-situ setup for additive manufacturing by SLM:
  - In-situ study of texture evolution during printing
  - Defect evolution as function of printing parameters
  - No clear plans yet, but MLZ should try to address this field
  - Joint development for diffraction, SANS, imaging?



Freeze-drying setup test at BNC, Hungary



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