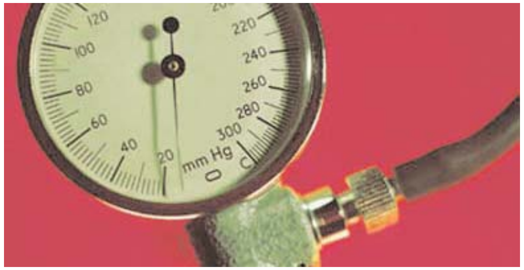
Sample Environment JRA

General Assembly

Rome 2011

High Pressure



The goal is to

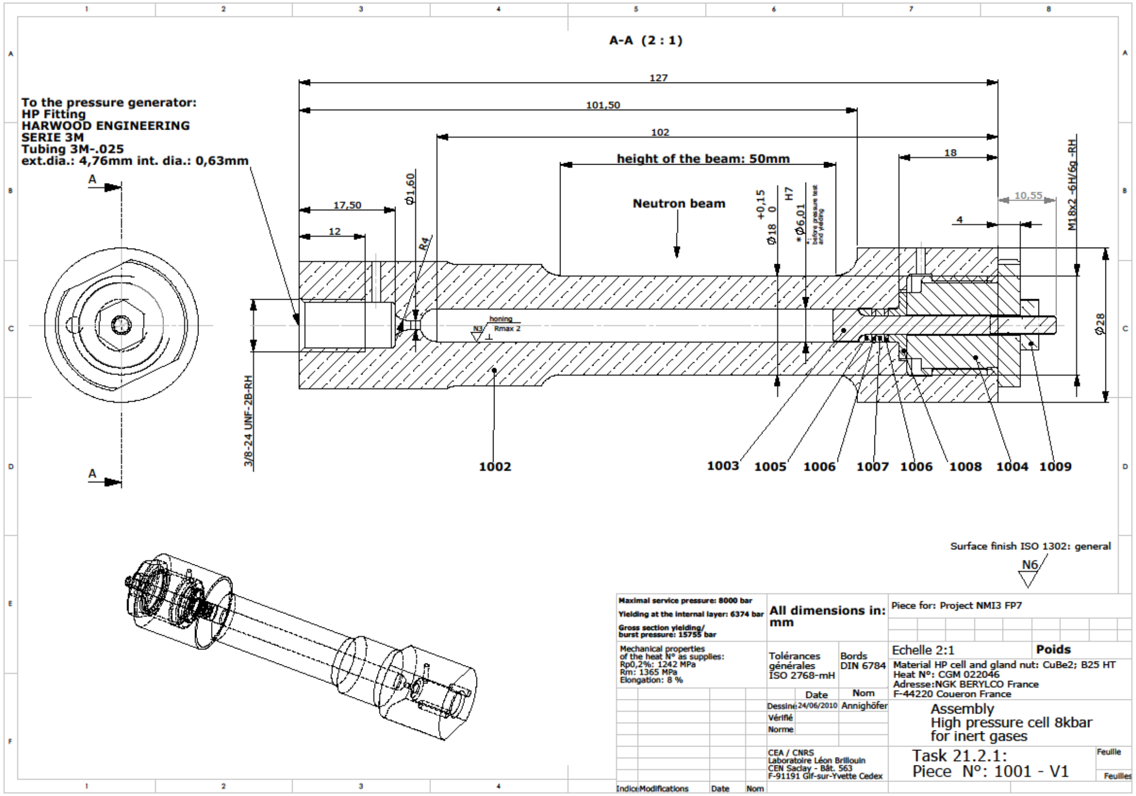
- produce a series of cells to increase the available pressure envelope both for inert and hydrogen gas

- the infrastructure to provide support to the user programmes

Cells 8kbar inert gas cell

Design features

- Inner diameter: 6 mm
- Outer diameter: 18 mm
- Beam height: 50 mm
- Material: CuBe2, B25 HT



Prototypes tested at ISIS

Calculated burst pressure: 15.7 kbar
Yielding at the internal layer: 6.3 kbar

Cell ②: applied pressure 12.7kbar → heavy yielding

Cell ① and ③: applied pressure 9.2kbar → ready for use at 8kbar

Cells now in use at HZB and LLB

Design plan review for cell up to 10 kbar at 300k

Infrastructure

Automated 10 kbar gas handling system @ LLB and ISIS

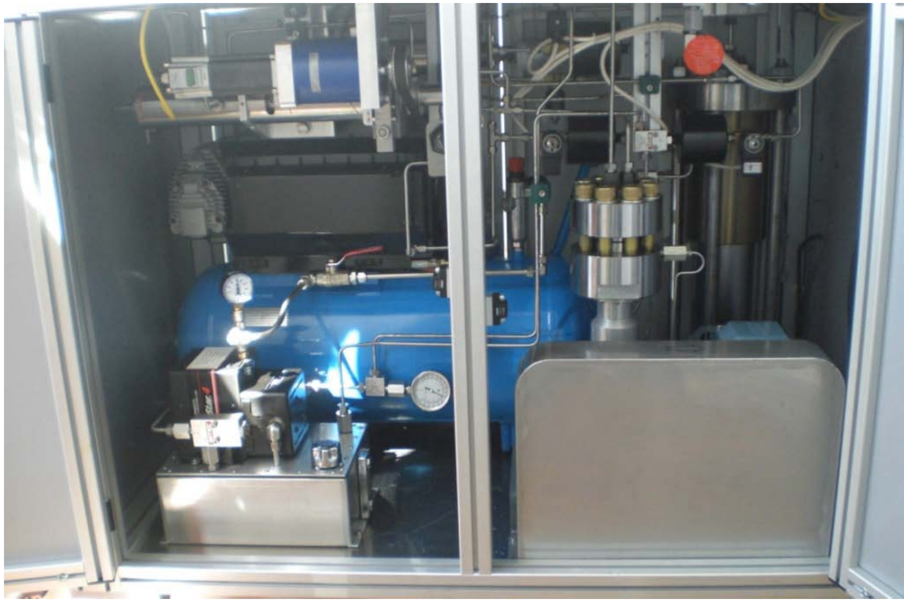
LLB - Upgrade of existing 10kbar manual system

Developments:

- Provide smooth ramping up and down
- Ensure *P* stability while warming and cooling, or in case of minor leakage.
- Automated pressure changing

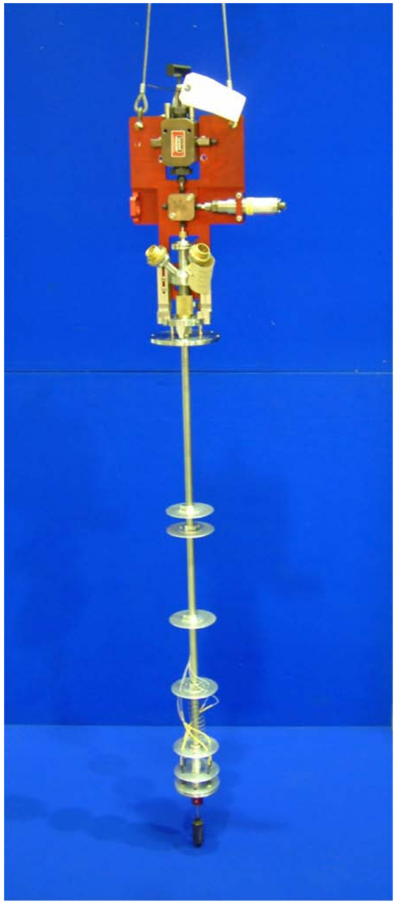
ISIS - commissioning of new fully automated system

- Transducer accuracy of 0.3%
- Full scale and pressure changes in steps of 40 bar



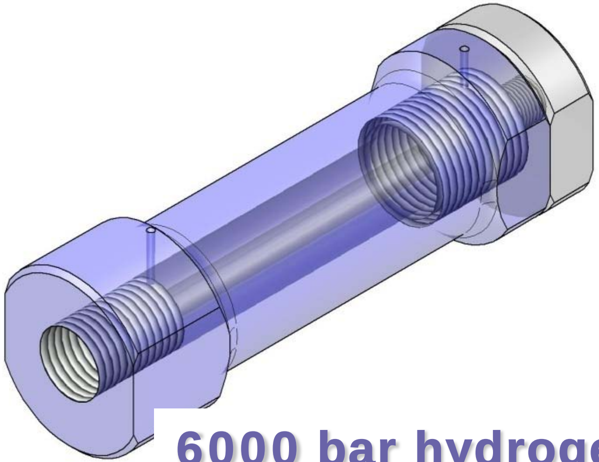
10 kbar H_2 Intensifier and gas handling system @ ISIS

Difficulties in getting manufacturers to construct a automated 10kbar system for hydrogen – this will now be constructed at ISIS – components purchased assembly has started.

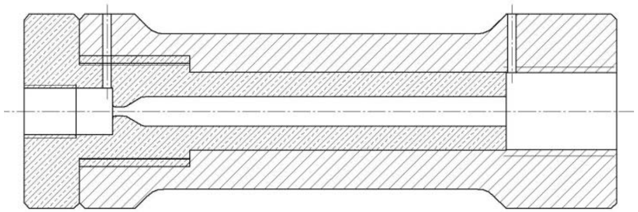


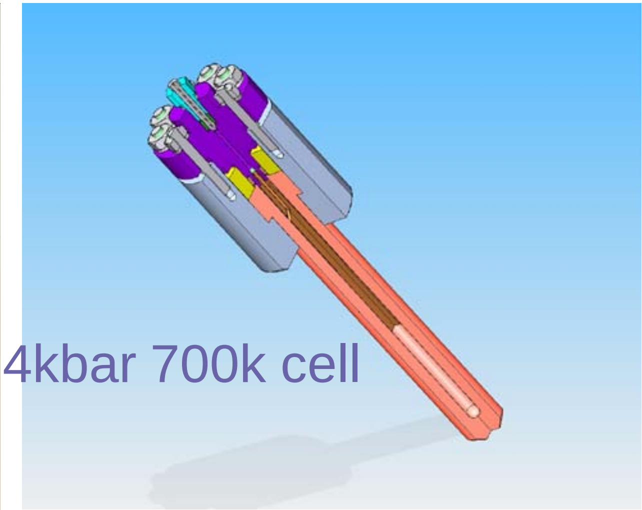
We have discovered the only 10kbar hydrogen valve on the market doesn't work

Cells for hydrogen

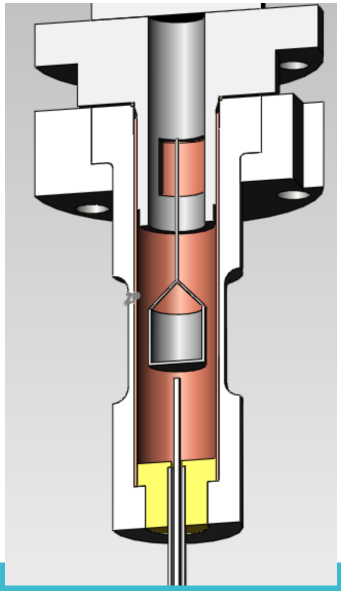


6000 bar hydrogen gas – 300k to 4 K





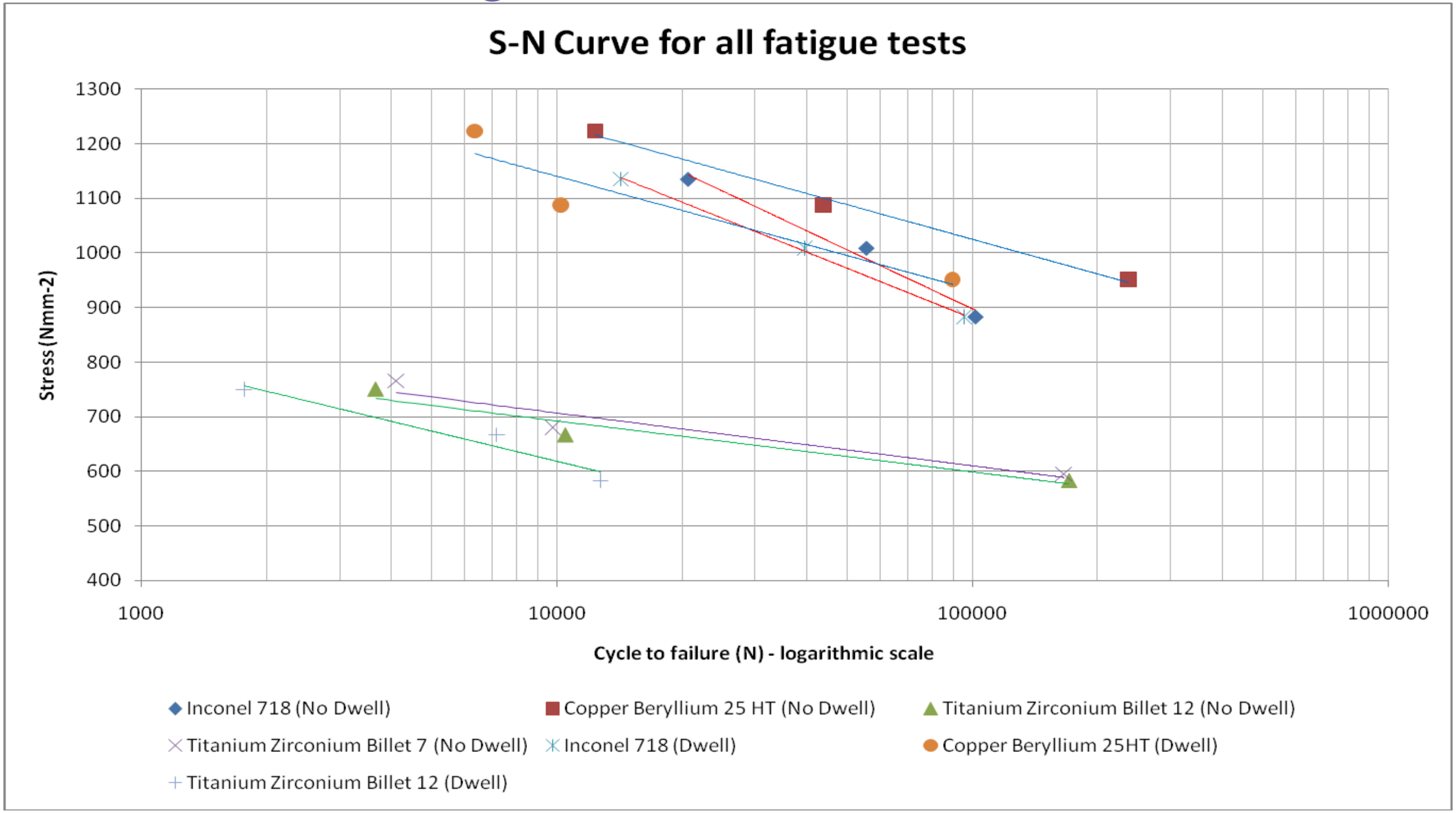
4.4kbar 700k cell



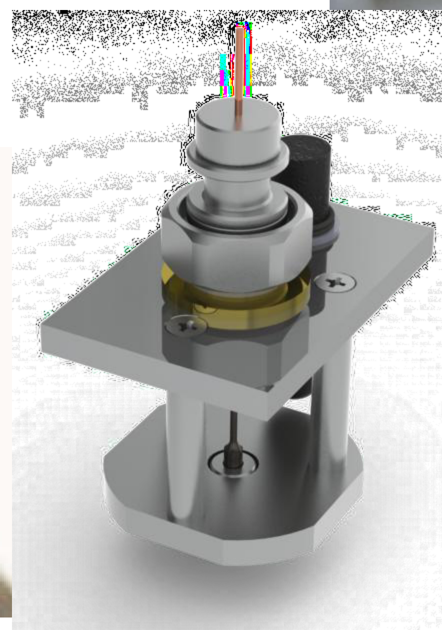
New cell for gas sorption system @ HZB

Material – beryllium copper inner, Ti/Zr outer
Nominal ratio of OD/ID = 28.0/7.0 = 4.0
Wall Thickness = 3.5mm BeCu, 7.0mm Ti/Zr
δ=0.02/0.04mm

Materials investigation



Work done in collaboration with Hugh MacGillivray Imperial College

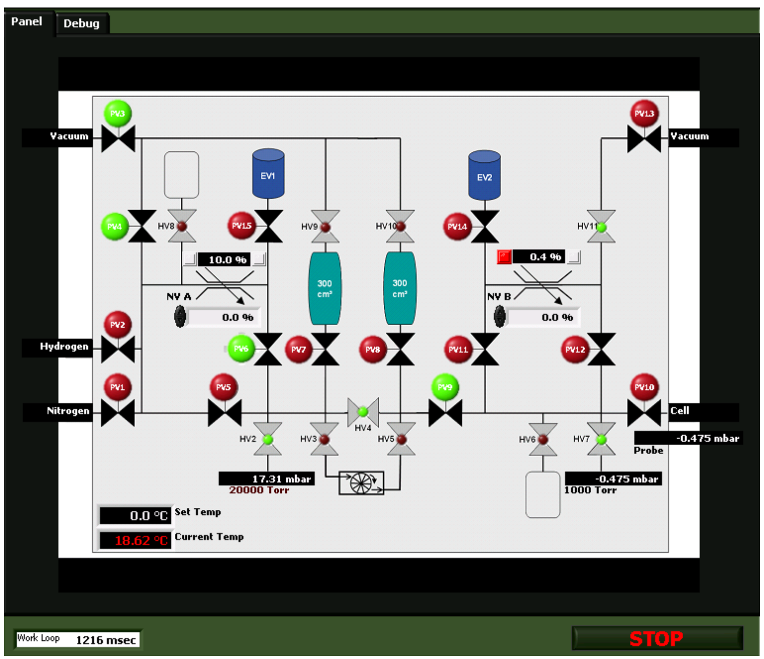


Work done in collaboration with Hugh MacGillivray Imperial College

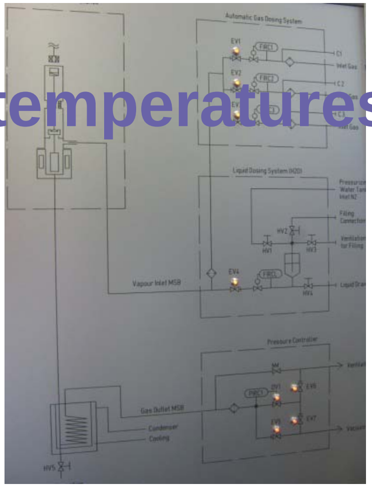
Gas sorption systems

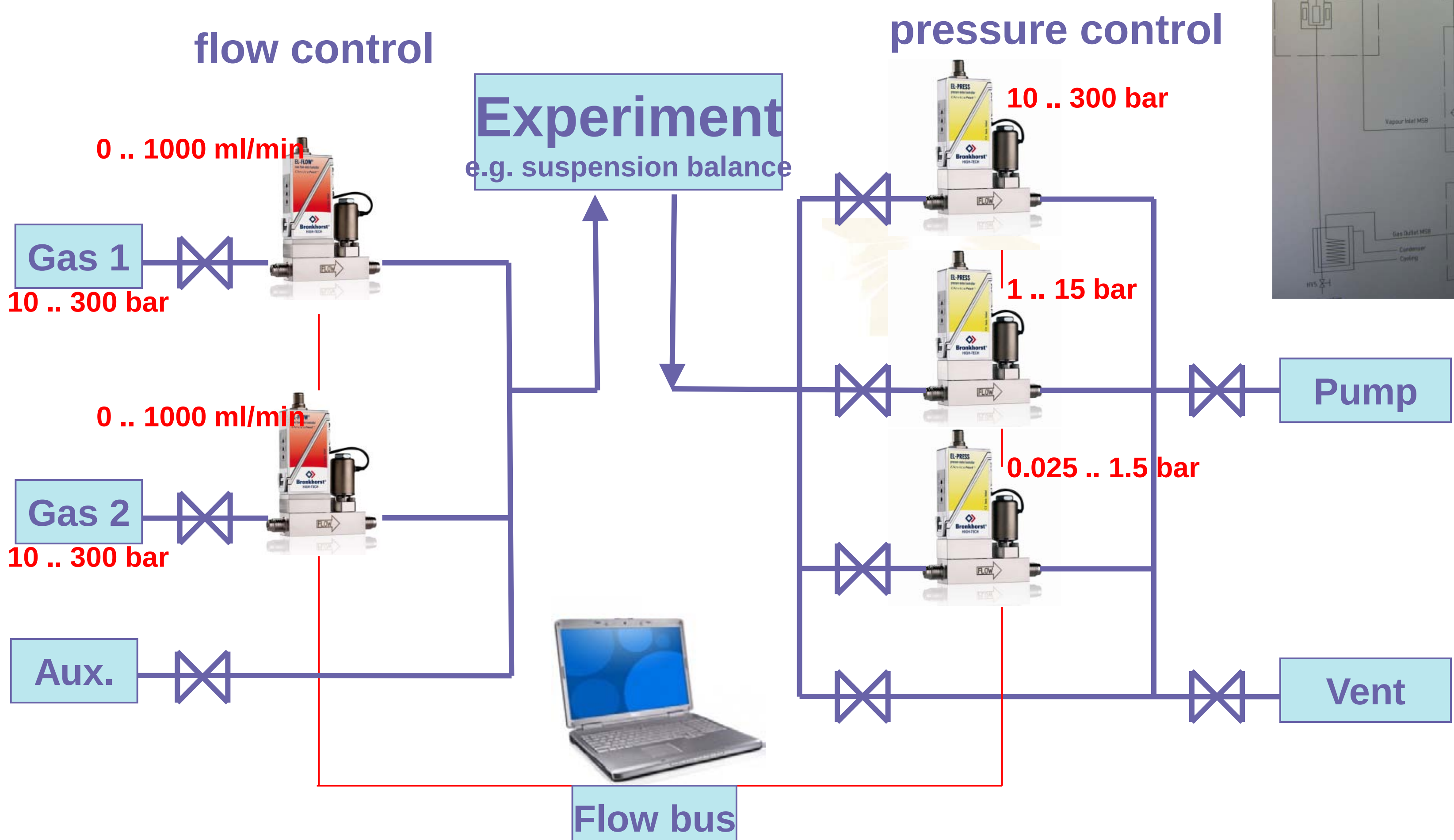
Gas handling system 300 bar at 500 K

- construction and assembly finished
- remote control operation panel established in LabView
- automated dosing software in test phase



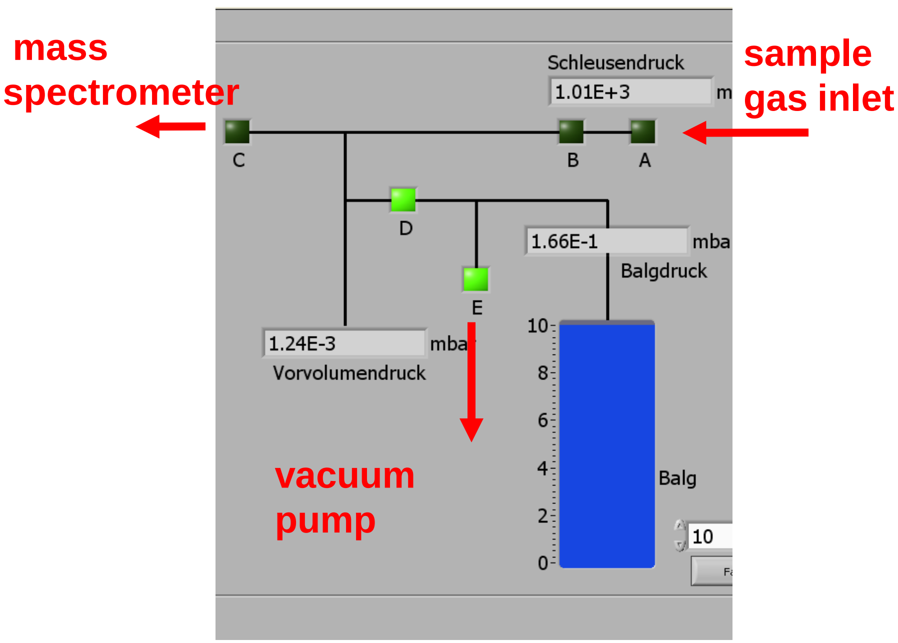
Gas handling system 300 bar – continuous flow improved pressure and flow control at ambient temperatures

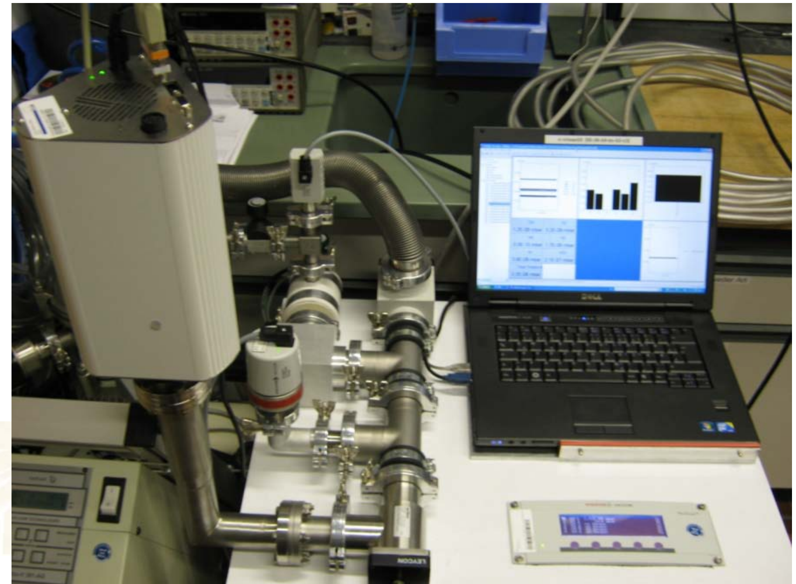




Residual gas analysis station

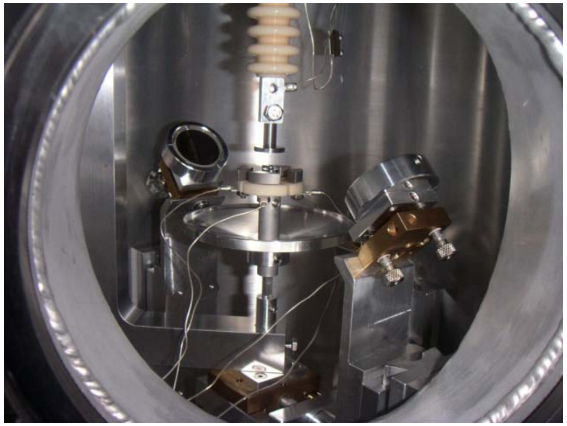
- extended by static gas analysis option, which allows automated analyzing very small gas probes





High Temperature furnaces

Electrostatic levitation



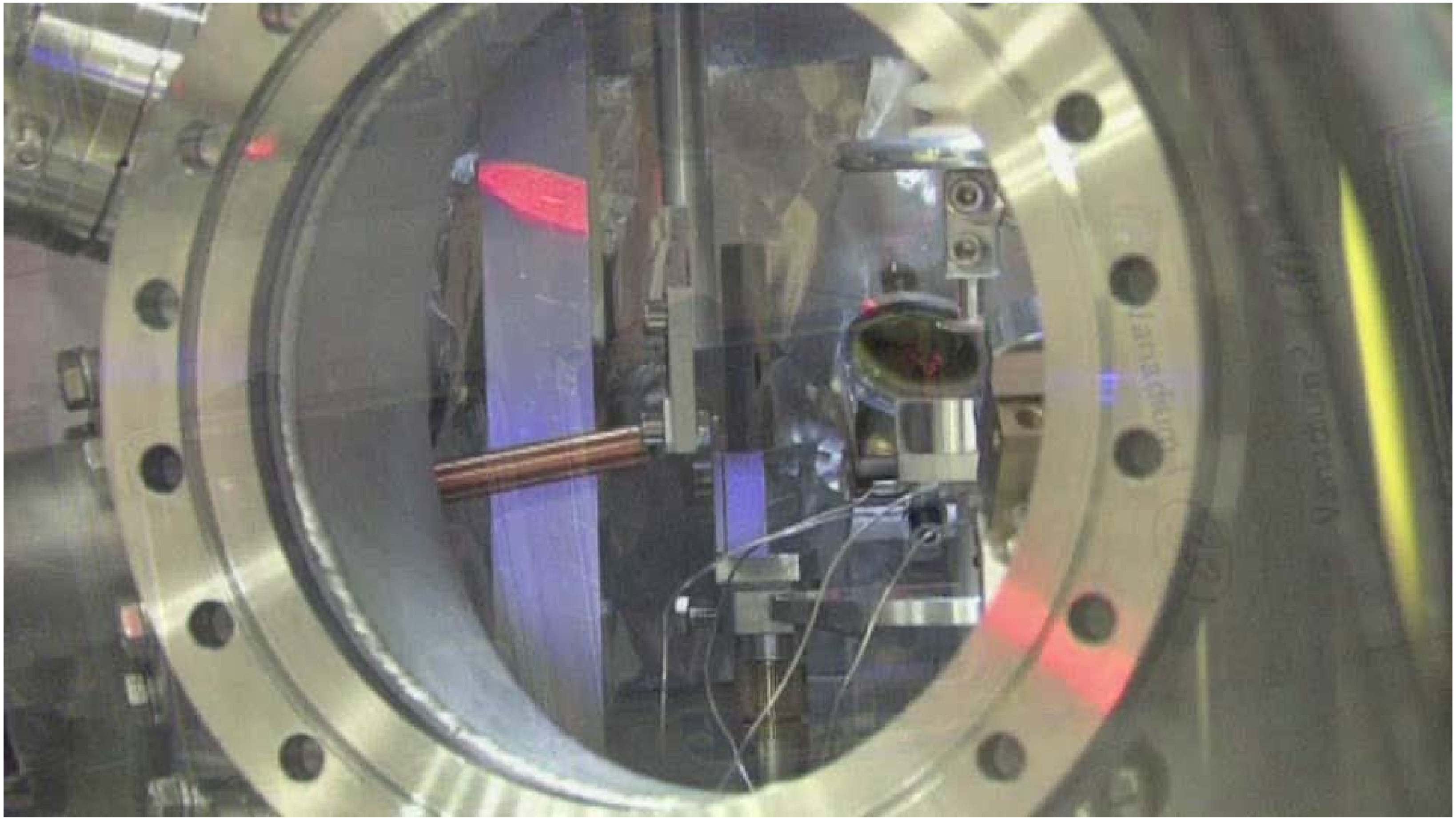
Experiments in 2010

•FRM II: TOFTOF Ni self-diffusion coefficient in chemically highly reactiveZr64Ni36 as a function of temperature up to an undercooling of 167 K below the melting point
•ILL: high flux diffractometer D20. With a neutron wavelength of 0.94 °A the total structure factor between 0.5°A−1and 12.3 °A−1was measured and the quality was improved significantly

Conclusion:
System successful in operation,
• Compact setup (fits to TOFTOF chamber)
• "Ease of use"
• Sample diameter up to 6,5 mm (0,5g -1g)
• Levitation of ceramic samples reveal problems

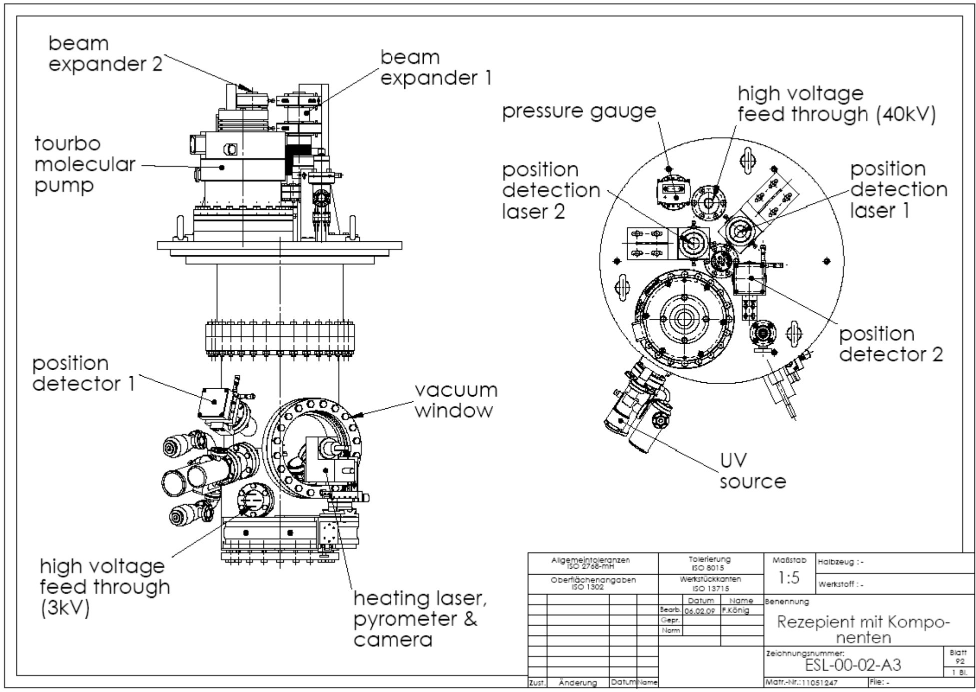
T. Kordel at al, accepted for PRB
Forschungsneutronenquelle

D20 Experiment



Ongoing developments:

- Laser preheater without levitation
- Cleaning of conductive sample in the ESL -removal of organic material from the surface, removal of dissolved gas and other contamination of the bulk material - enhanced reliability of the fusing process, processing of new sample systems, reduced fusing time
- Ceramic samples: charging of the sample by thermionic emission. No need of coating or doping



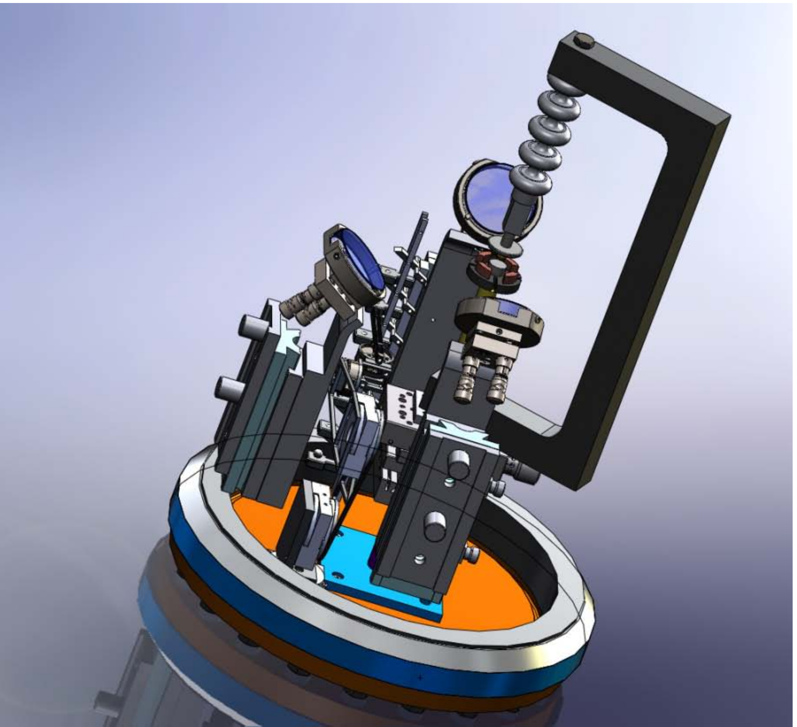
2nd laser heater:

Due to the rapid heating of the sample temperature gradients occur in samples with increasing diameter.

Below 1300K these gradients amount to ± 10K referred to the mean temperature.

With increasing temperature the gradients increase up to 50 k and more.

A 2nd laser will therefore be installed in the rear of the vessel

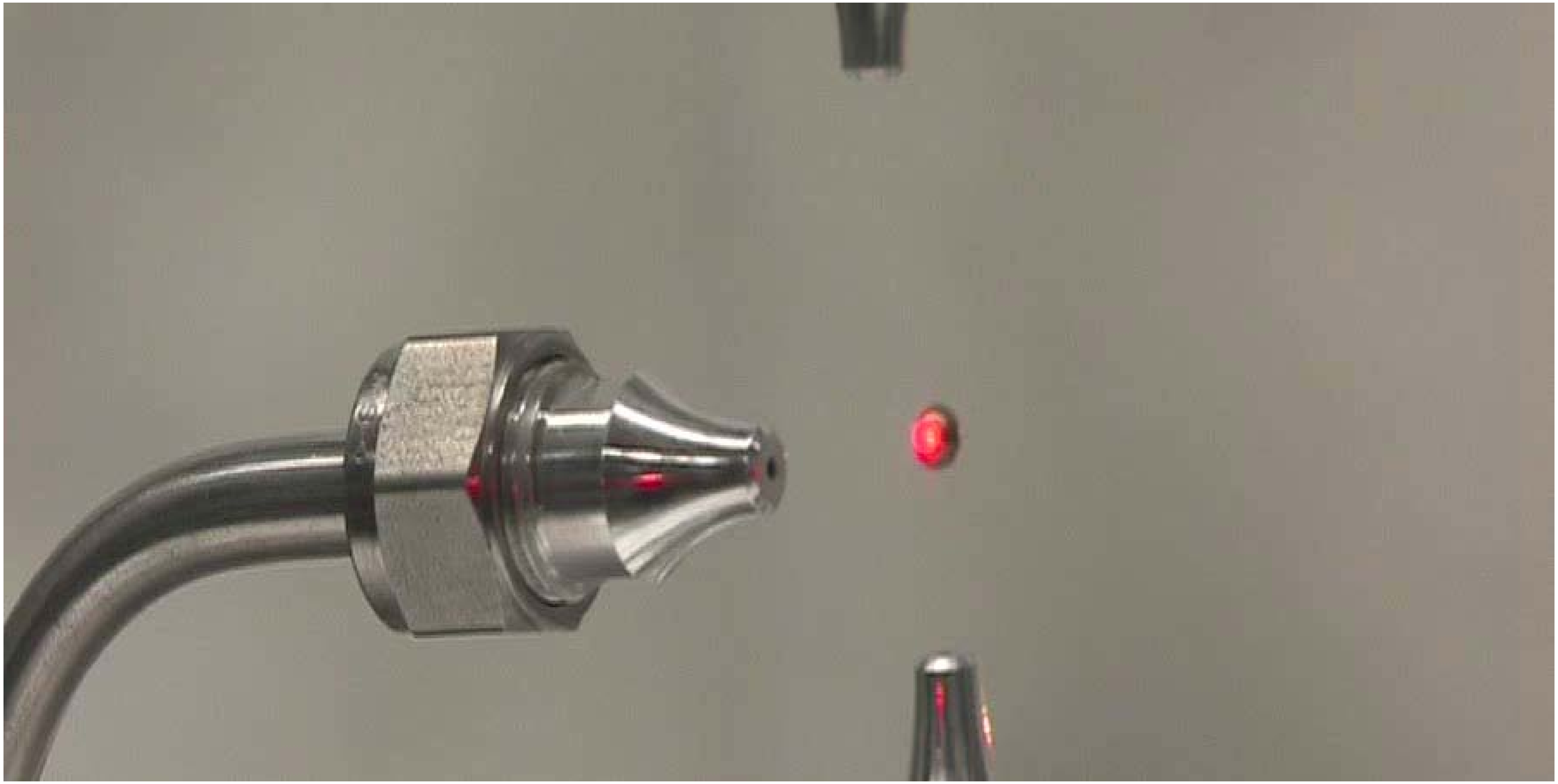


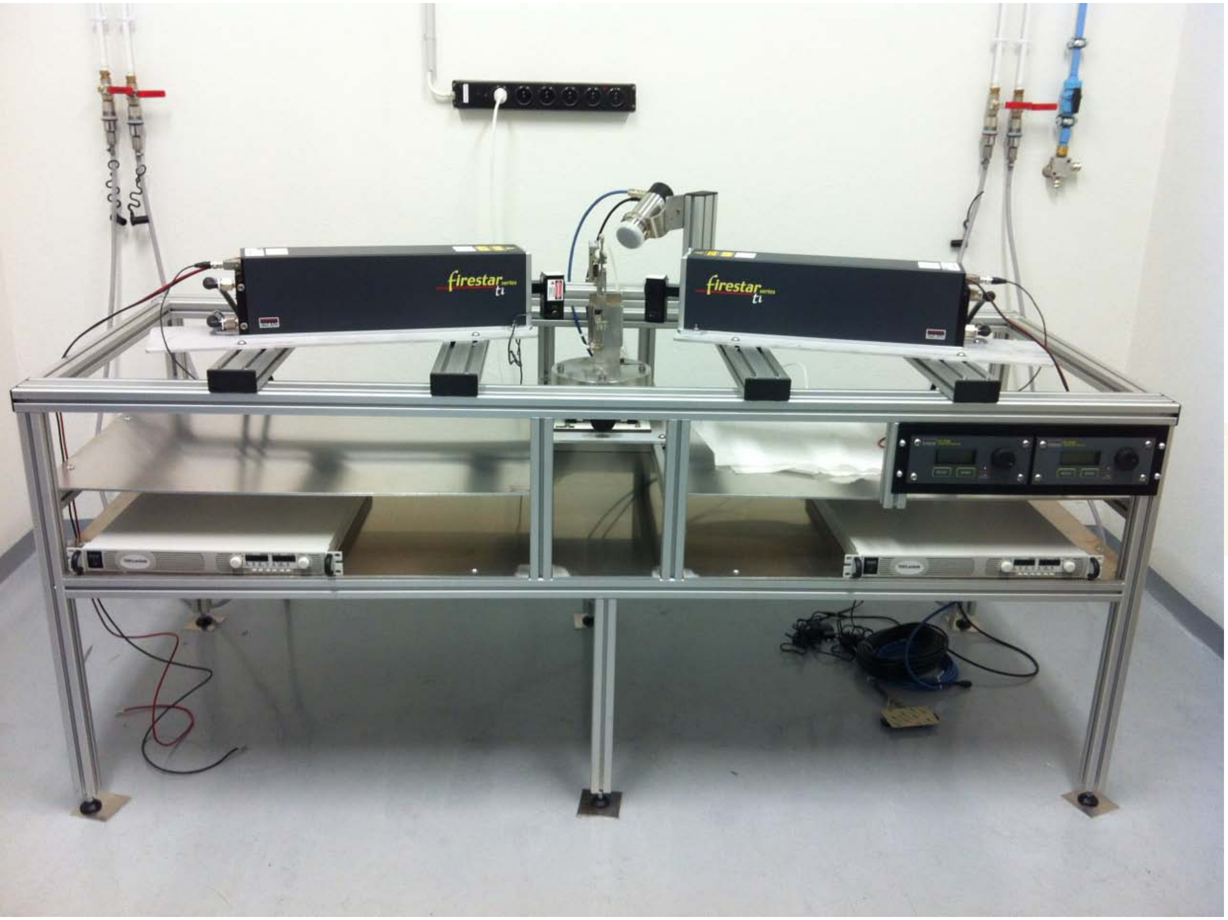
Sample changer

- Manipulation of several samples without breaking the vacuum
- Must fit in the restricted inner space of the NESL
- Use for preheater setup
- Sample storage rack easy to load and change (outside the ESL)
- Samples on stock must be protected from vapor deposition
- Recovery of samples kicked out the levitation for reuse and clearance

2009

2011

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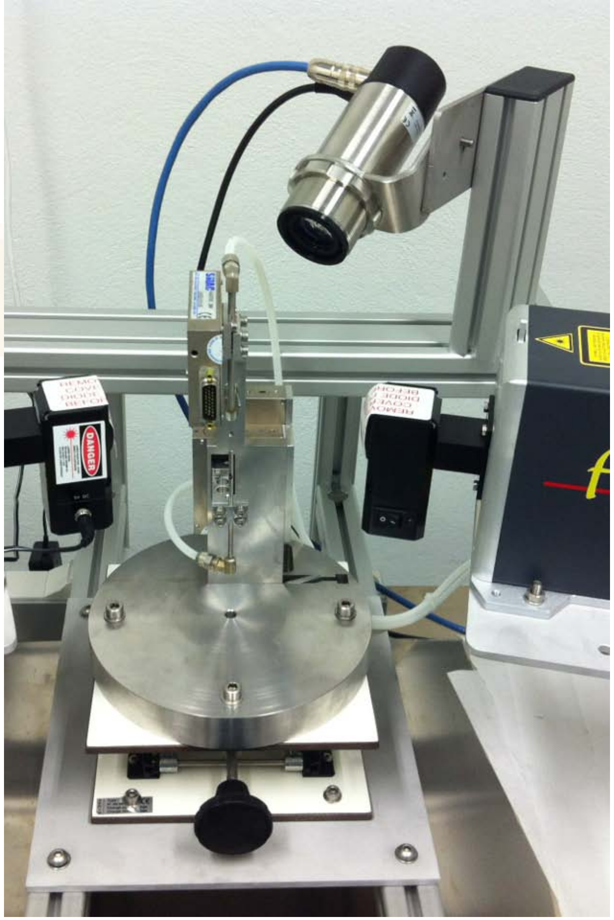
DANGER