Cryogen-free cryostat with sample changer for fast automatic data collection

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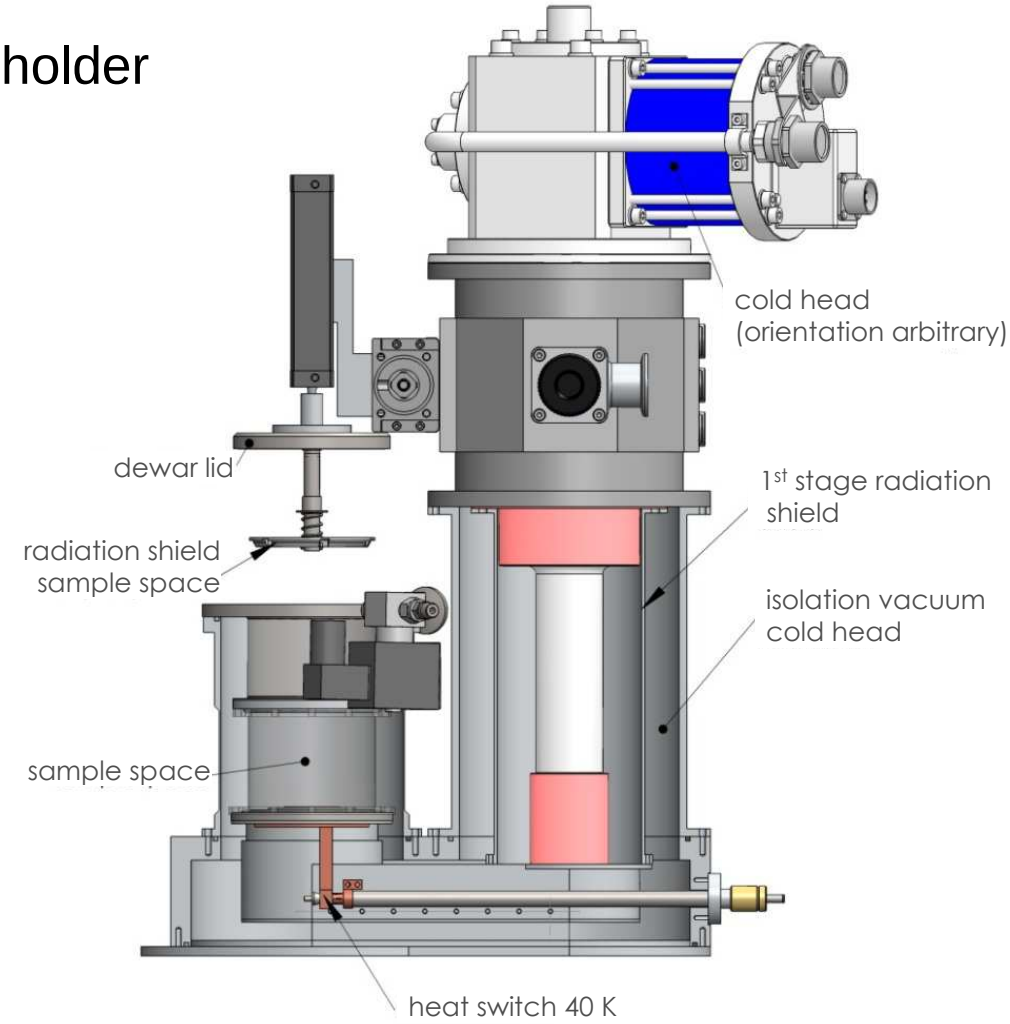
Compact Cryostat – Current topics

1. Thermally decoupling of sample holder
⇒Leakages whilst testing

2. Thermal connection of sample
⇒Research at other institutes
⇒Concepts

3. Icing inside the sample tube
⇒ Sample tube heating
⇒ Gas flow improvement



Thermally decoupling of sample desk

Thermal decoupling - Sample desk

Problem:

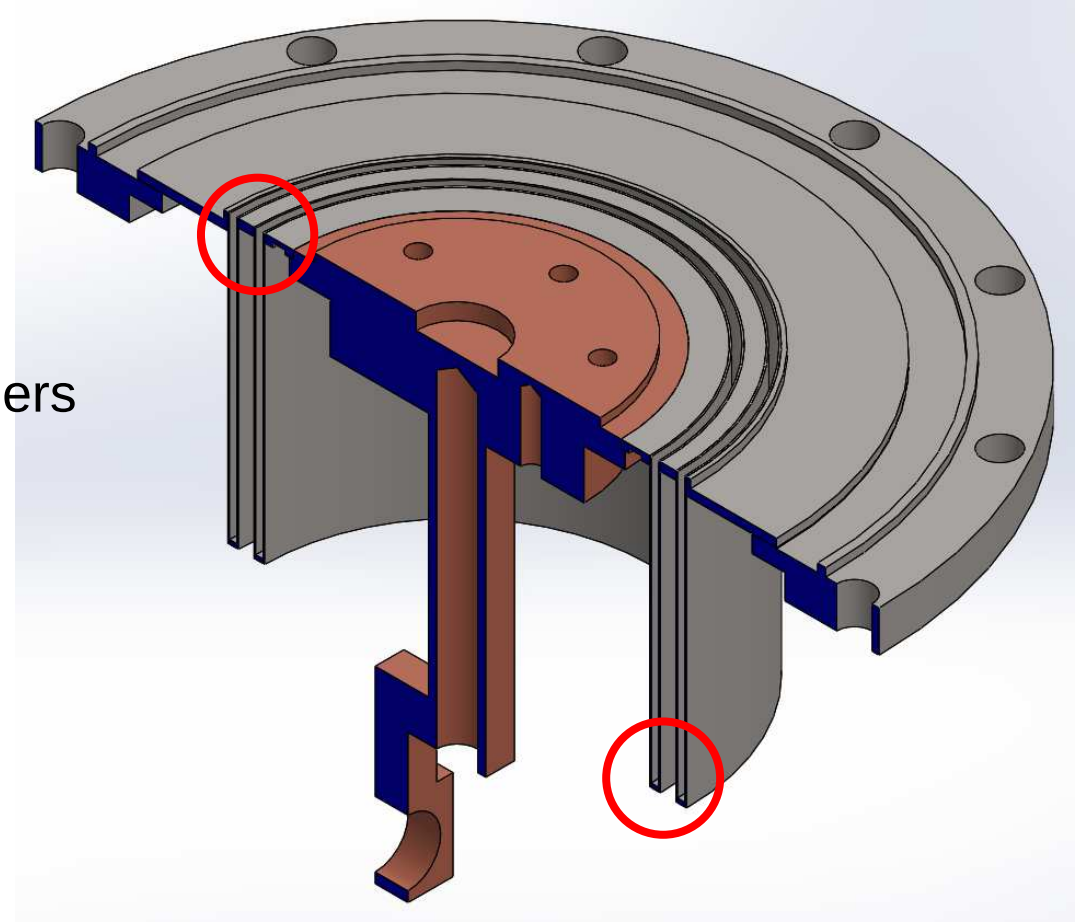
Structure too filigree

⇒New leakages whilst handling

New concept:

-Larger distance between cylinders

-Soldering at the cylinders



2nd topic

Thermal connection of sample

→ Research

Commercially available robots and sample exchange systems

Bruker (BruNo)

-Robot for X-ray crystallography

MarXperts (MARCSC)

-Automated sample changing robot for X-ray crystallography integrated in the marXperts multi-purpose goniometer

IRELEC (CATS)

-Complete sample exchange system using “Stäubli”-robot

NatX-ray (G-Rob)

-Robot-based system for crystallography (derived from CATS)

Existing automated sample changer at facilities

BCSB [BCSB Wiki]

Berkeley Lab – Advanced Light Source [Snell et al. 2004]

EMBL Grenoble Outstation (*SC3*) [Cipriani et al. 2006]

EMBL Hamburg Outstation [Pohl et al. 2004]

ESRF (upgraded *CATS*) [Jacquamet et al. 2009], [Fitch 2014]

J-PARC (*iMATERIA*) [Hoshikawa et al. 2010, 2014]

Max Planck Research Unit for Structural Molecular Biology [Karain et al. 2002]

Oxford Instruments Omicron NanoScience [Batey et al. 2014]

Rigaku in collaboration with Abbot Laboratories (*ACTOR*) [Muchmore et al. 2000]

SNS (FERNS and PAC) [Heroux 2014], [Rix et al. 2007], [Wenzel]

SPring-8 (*SPACE*) [Ueno et al. 2004], [Murakami et al. 2012]

SSRL (*SAM*) [Cohen et al. 2002, 2005]

Structural Biology Research Centre at the Photon Factory (*PAM*) [Hiraki et al. 2012/2013, 2013]

2nd topic

Thermal connection of sample

→ Concepts

Possible direct thermal connections

Screwing	→	Rotatory and/or vertical strain
Plugging / Bayonet	→	Vertical strains
Magnetic	→	Induction
Under pressure	→	Limited space, sealing
Clamping	→	Space?
	→	Piezo actuators

Thermal connection

Sample holder:

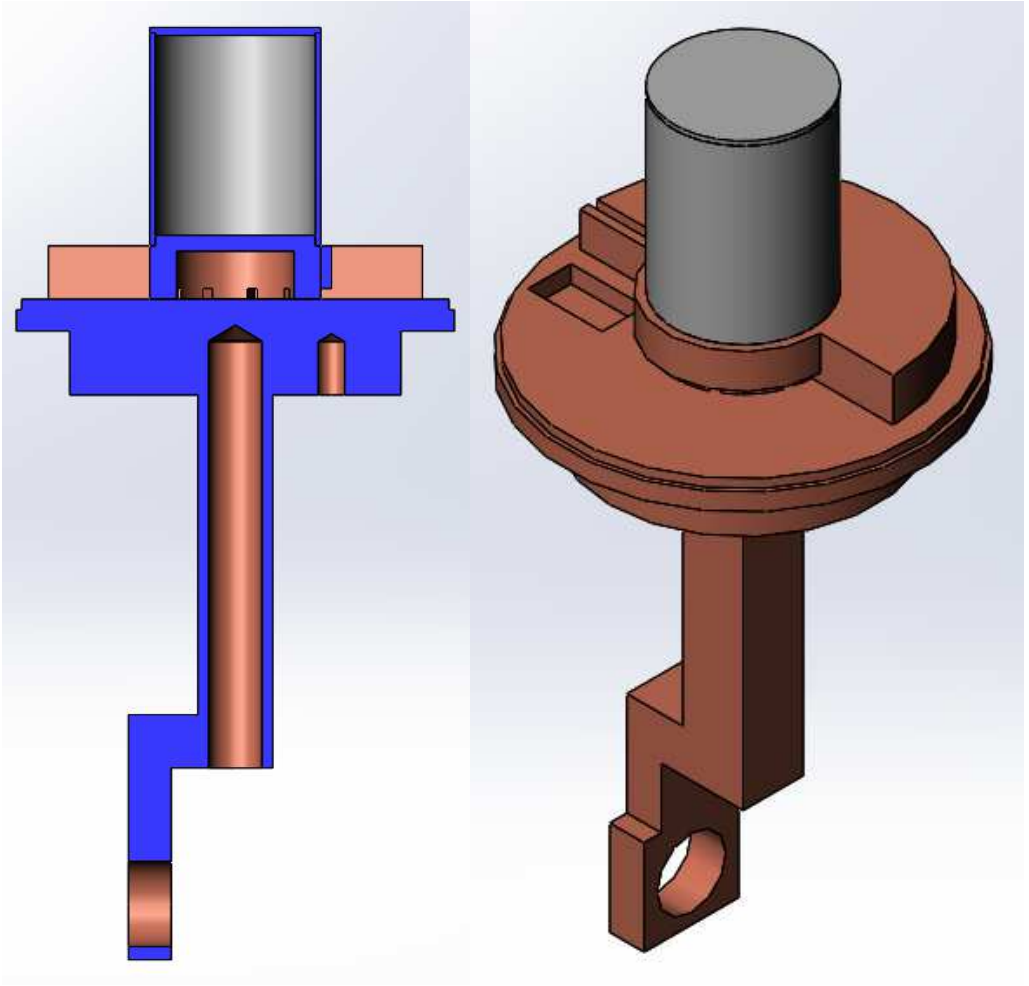
-Copper base

-Deep drawn container

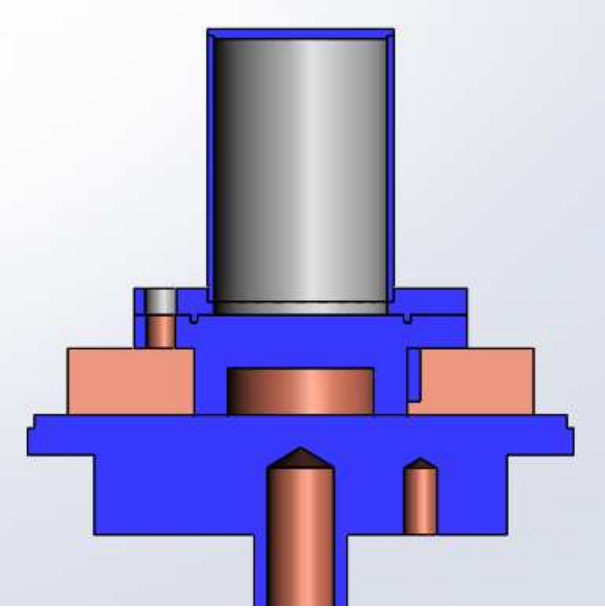
Clamping mechanism:

-Copper spring incorporated into sample desk

-Driven by piezo actuator



Sample holder sealing

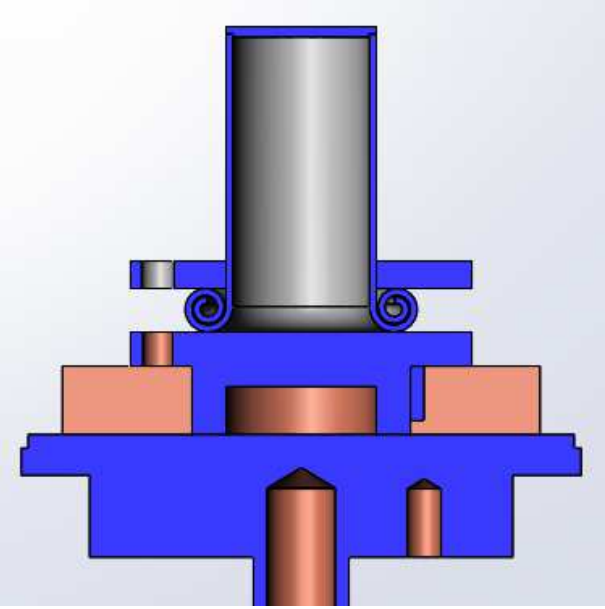


Indium

-Detachable

-Easy sample attachment

-Can loosen at low Temperatures

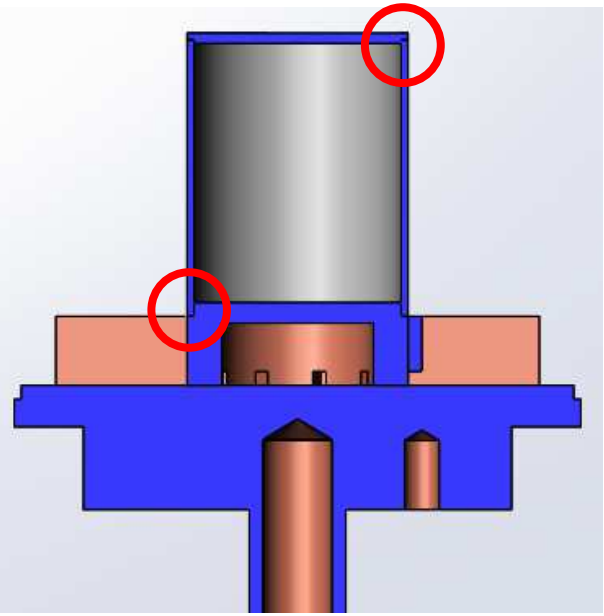


Spring

-Detachable

-Easy sample attachment

-No experiences yet



Orbital wielding

-Permanent connection

-Small size ratio

-Two welding seams necessary

Icing inside the sample tube

Sample tube heating

Thermal decoupling between sample desk and sample tube „too good“

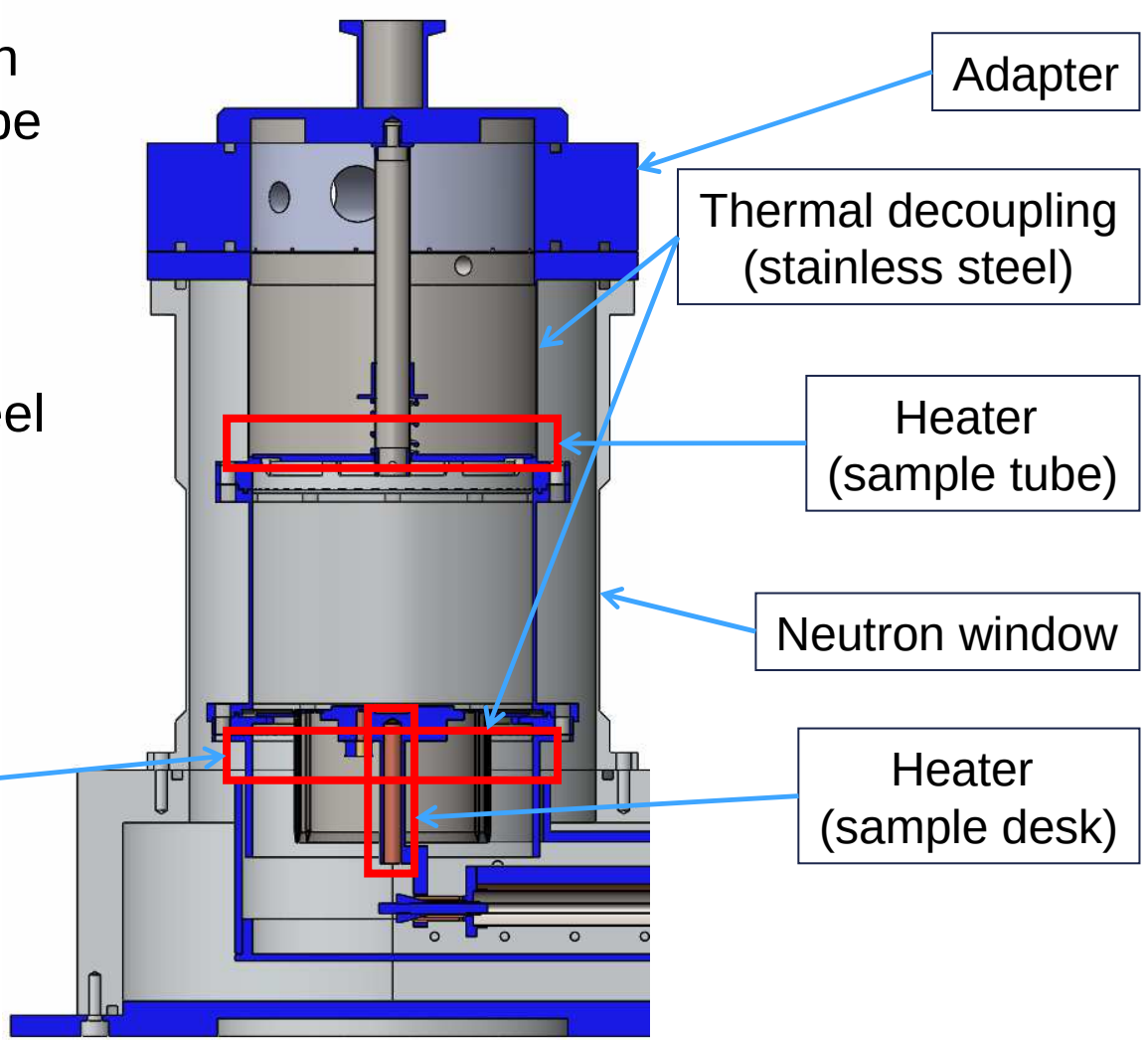
⇒Tube stays cold

Heater collar at stainless steel part

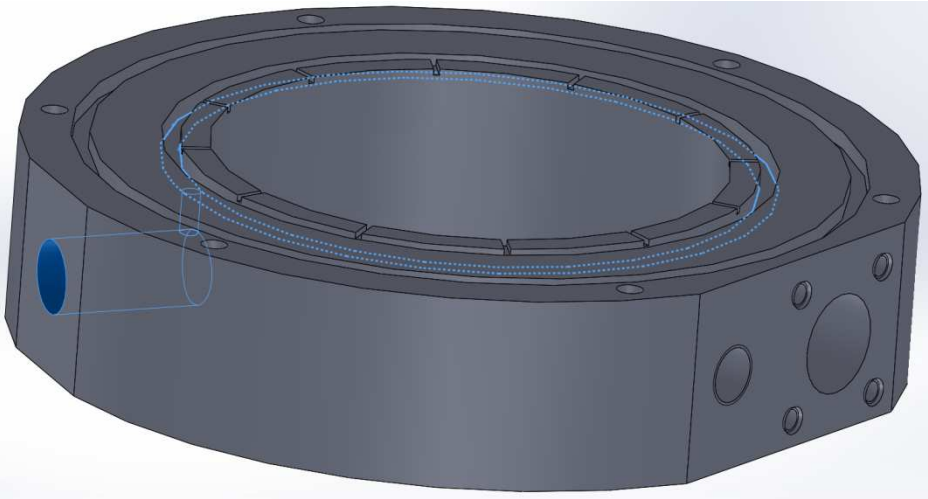
⇒No icing in heater region

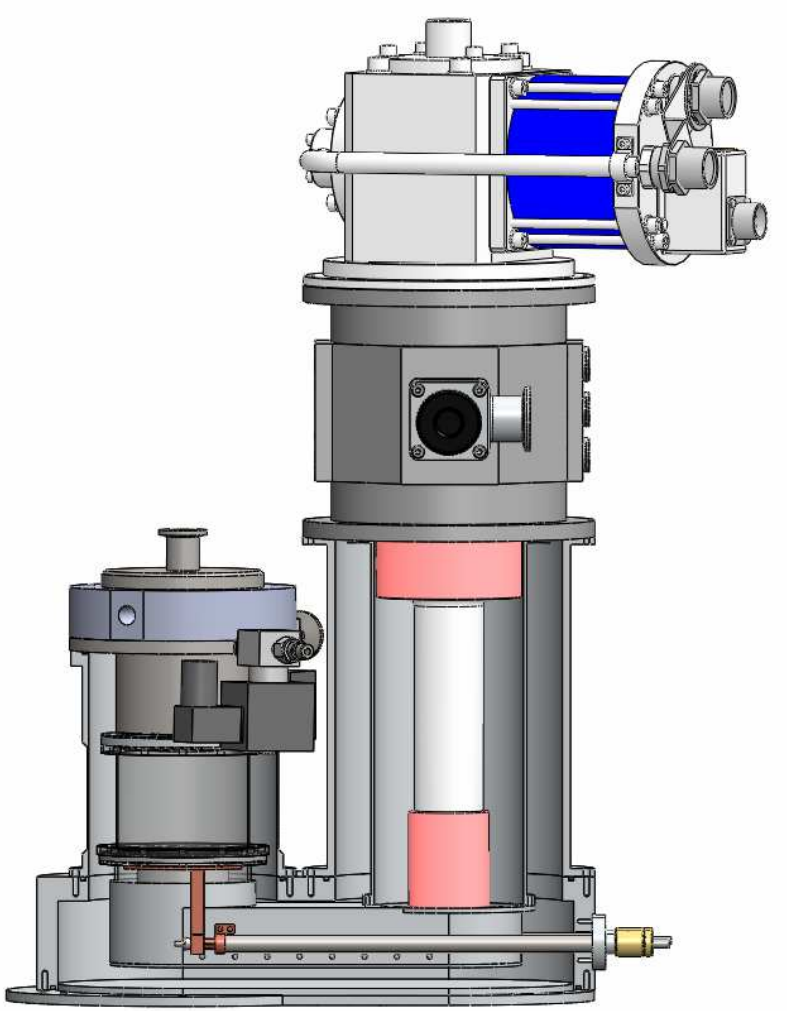
⇒Above and below still icing

⇒Relocation of heater necessary



Gas flow distribution





Gas inflow all around the sample tube

⇒Less fog inside the sample tube

⇒After a short time still icing inside the sample tube

Thank you for your attention!

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