A high temperature furnace for in-situ SANS and diffraction measurements on steel samples

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Introduction

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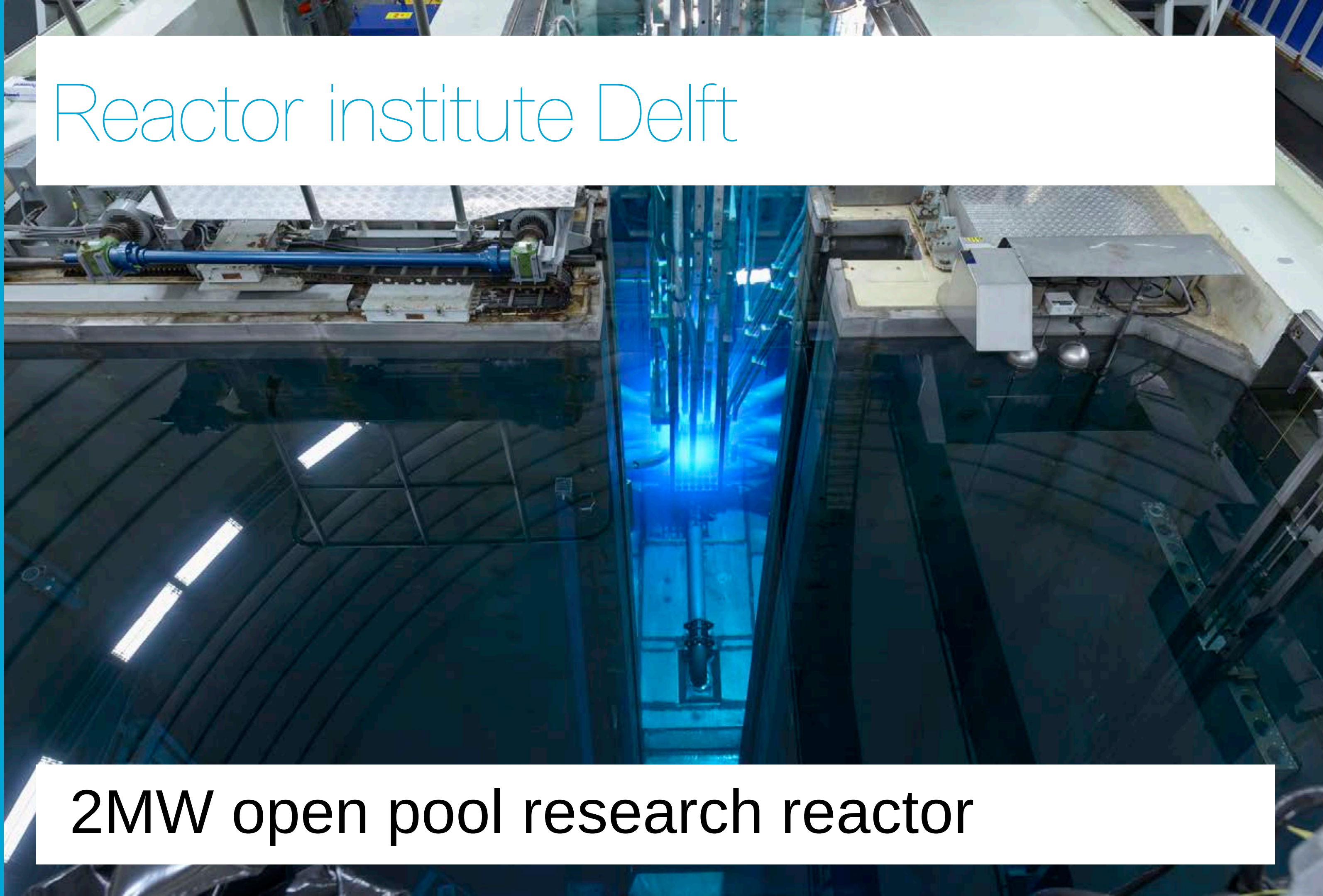
Reactor institute Delft



2MW open pool research reactor

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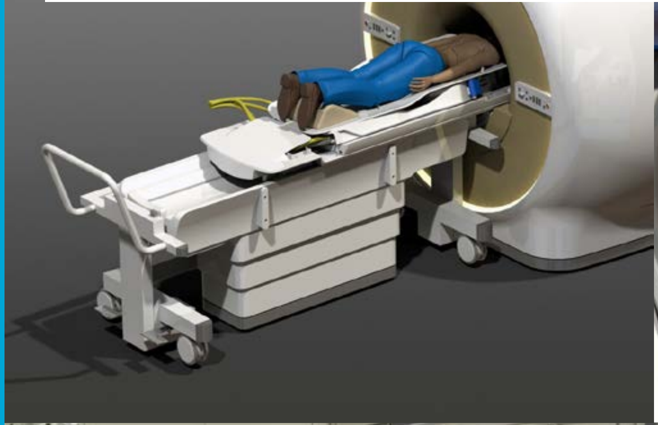
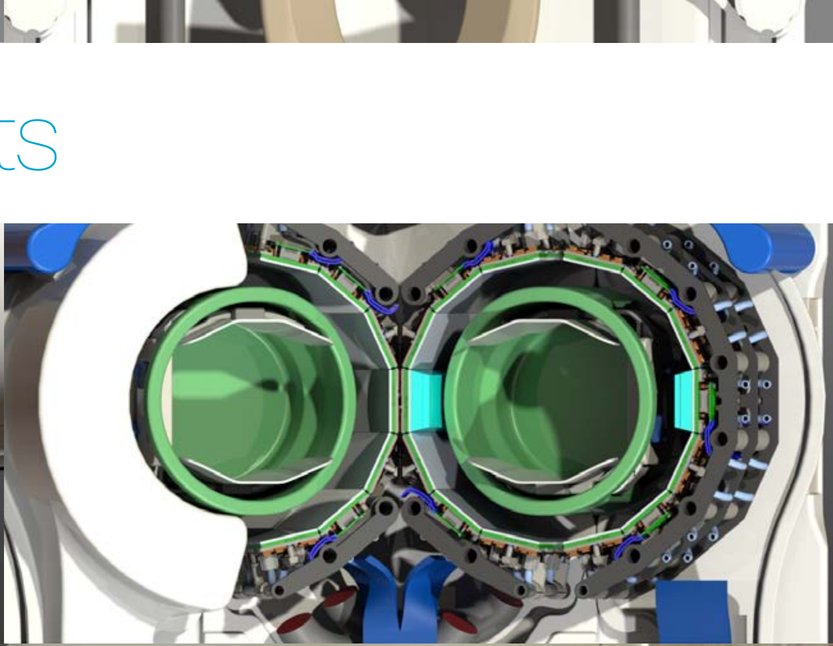
DEMO - Projects





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A high temperature furnace for in-situ SANS and diffraction measurements on steel samples

Scientific case

Goal

The goal of the research is to get **fundamental insight** into the role of **individual chemical elements** on the precipitation and phase transformation kinetics, which is deemed essential for the development of **NANO-steels** with **reduced amounts of alloying elements without compromising properties**.

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NANOsteels

- Ferrite with addition of Niobium, Molybdenum, Titanium and Vanadium
- Single-phase, ferrite matrix in combination with nano-meter-sized precipitates
- Used in automotive industry for resource efficiency and weight reduction

Research methods

- Electron probe micro analysis
- Atom probe tomography
- Dilatometry
- SANS
- **SANS and ND**

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Goal SANS and ND

Study in-situ and simultaneous the precipitation kinetics (with SANS) and the phase transformation kinetics

(with ND) in NANOsteels.

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What is needed

- SANS and ND instrument
- Sample environment

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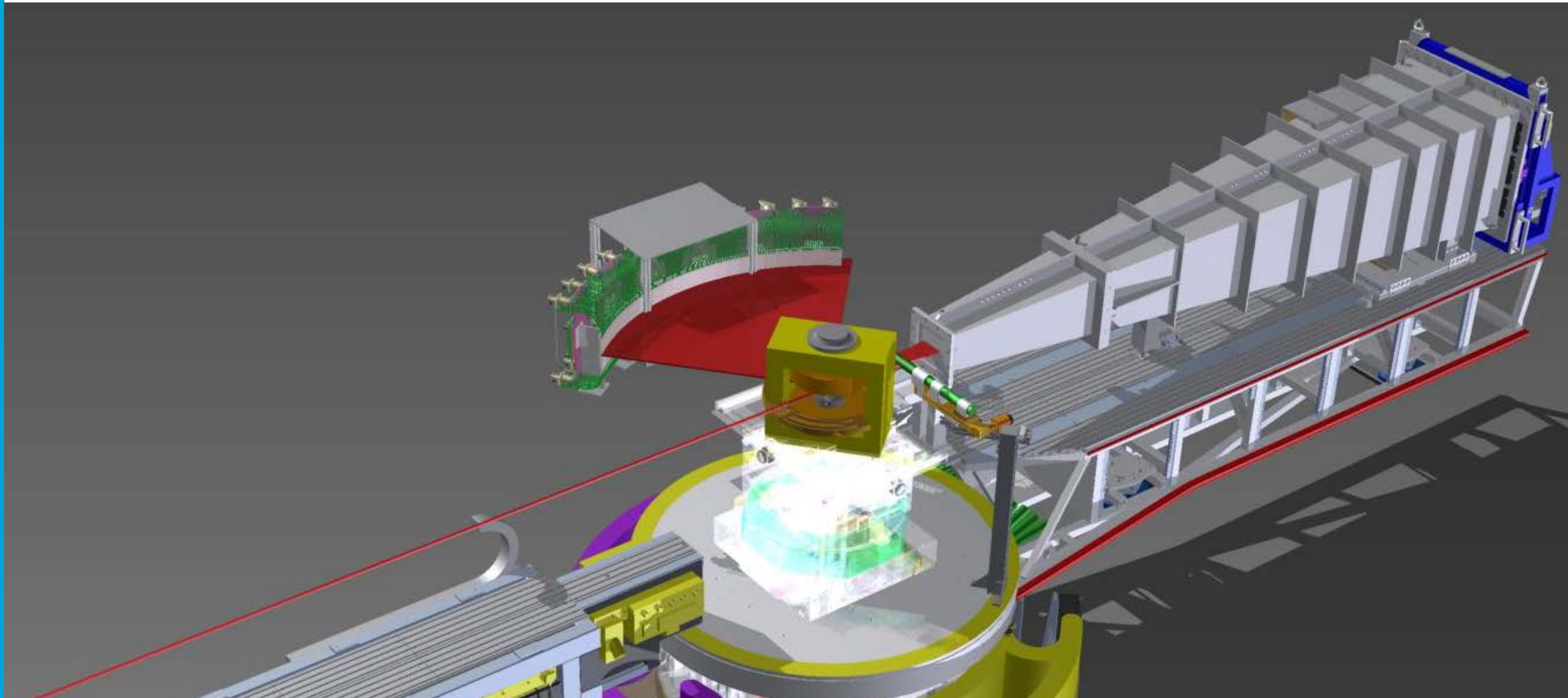
SANS and ND instrument

- Developed within LARMOR at ISIS



Sample environment

- High temperature and fast cooling
  - Heat treatment while doing measurements
- Magnetic field
  - Homogeneous over the sample for magnetic contrast
- Rotation
  - Reduce texture effects

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Furnace requirements

Sample

- Sample 1mm thick

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SANS and ND

- Incoming beam size: 8x8mm
- SANS beam: -4° - 4°
- Diffracted beam: 49.5° - 76.5°

- Neutron transparant windows, no percipates growth due to temperature threatment

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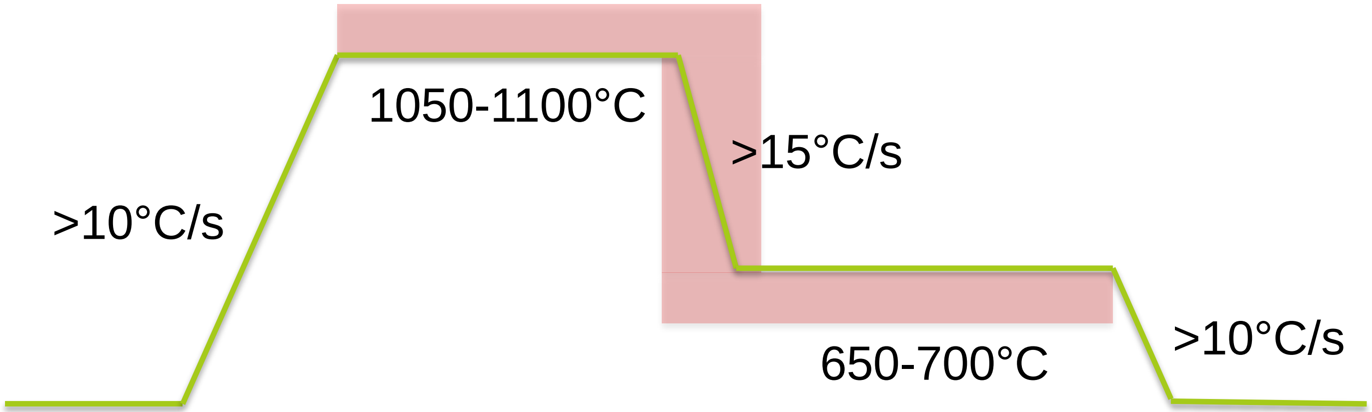
Temperature

- Heat treatment:



- Temperature gradient
  - 0.2°C/mm

Magnetic field

- GMW magnet
  - Pole shoes 75mm
  - Pole shoe gap 44mm for 1.67T

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Rotation

- -15 to 60°

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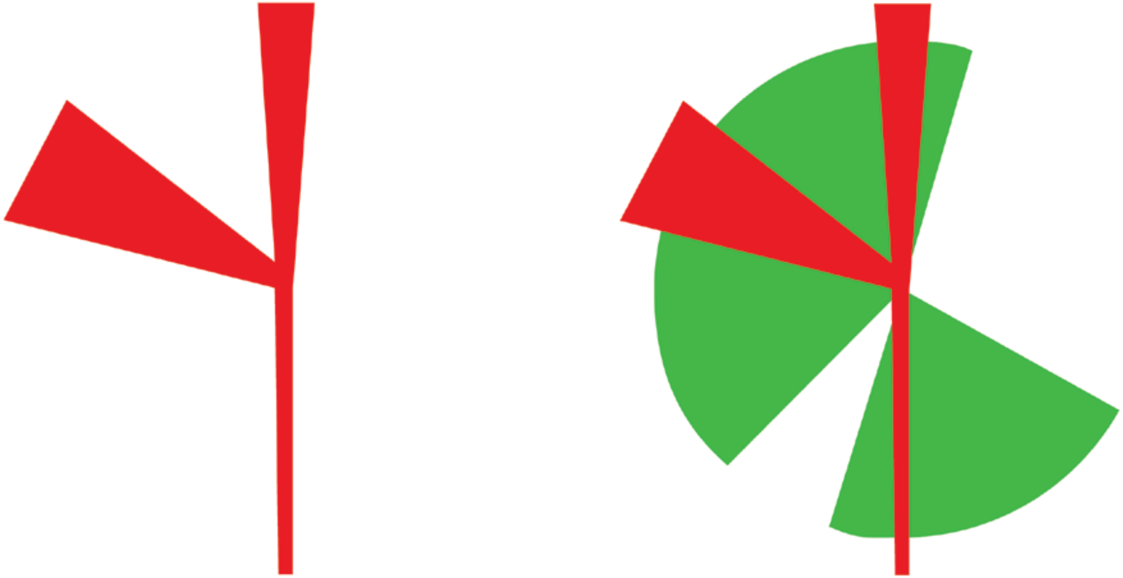
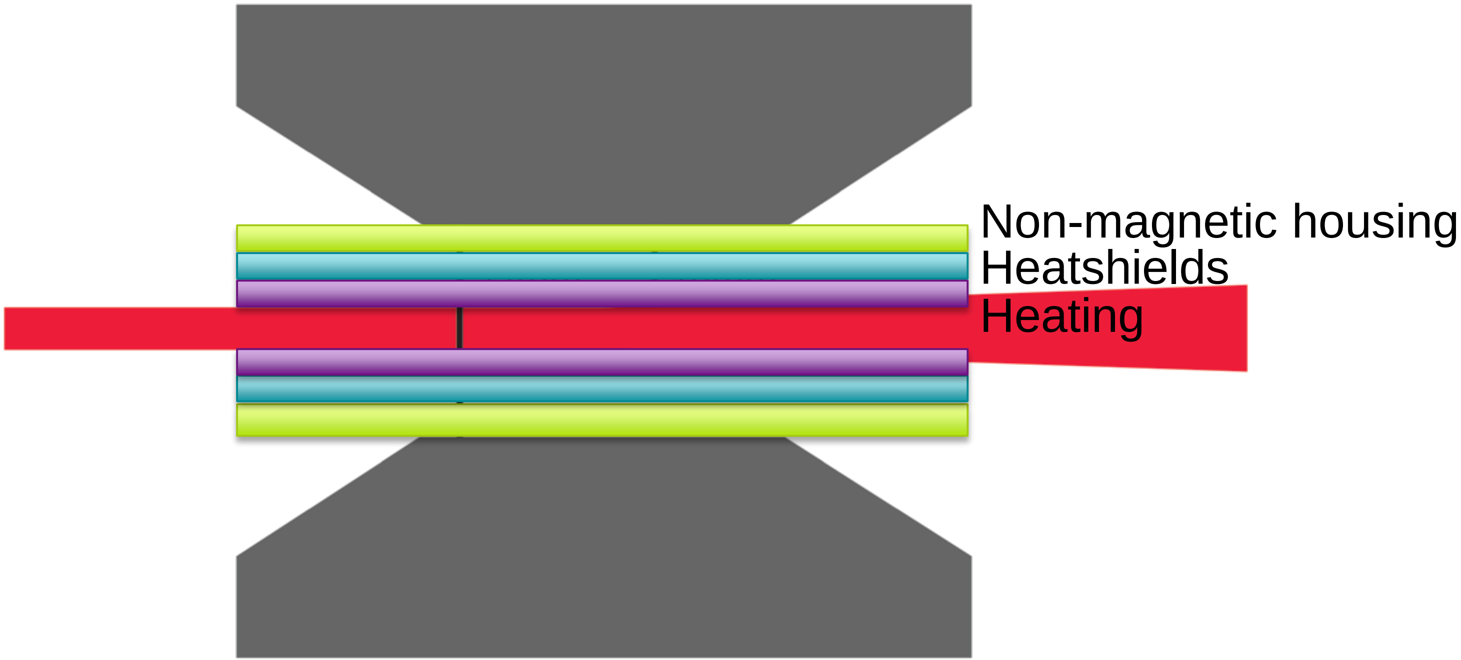
Dimensional constraints

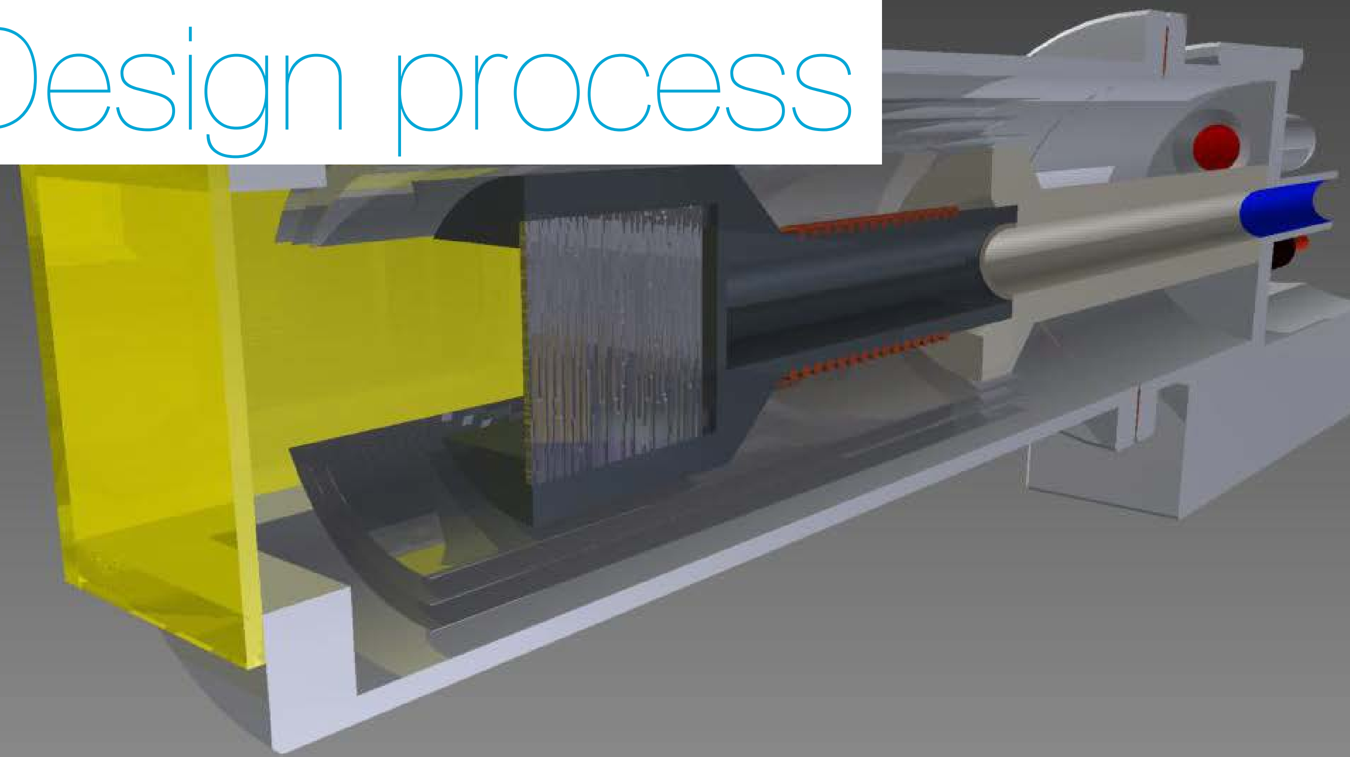
- -15 to 60°



Dimensional constraints



Design process



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Types of heating

- RF
- Laser
- Resistance
- Hot finger
- Radiation
- …

Heating wire

- Platinum
  - Melting point 1700°C
  - Flexible
  - Expensive
- Molybdenum
  - Melting point: 2 620 °C
  - Recrystallization Temperature: 600°C
- Tungsten
  - Recrystallization Temperature: 1300°C

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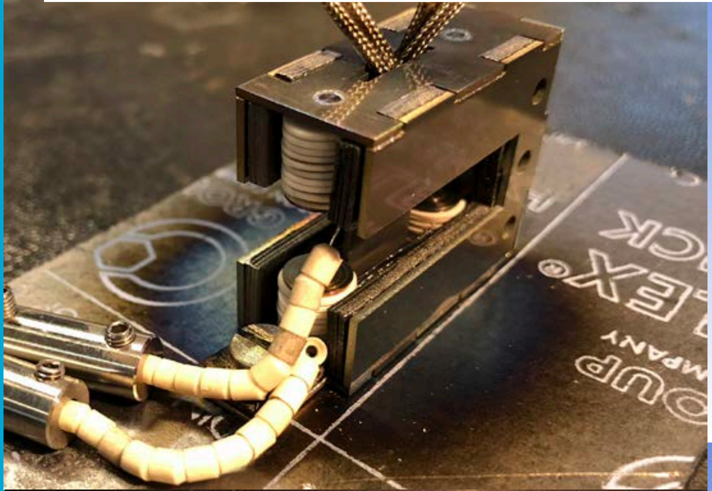
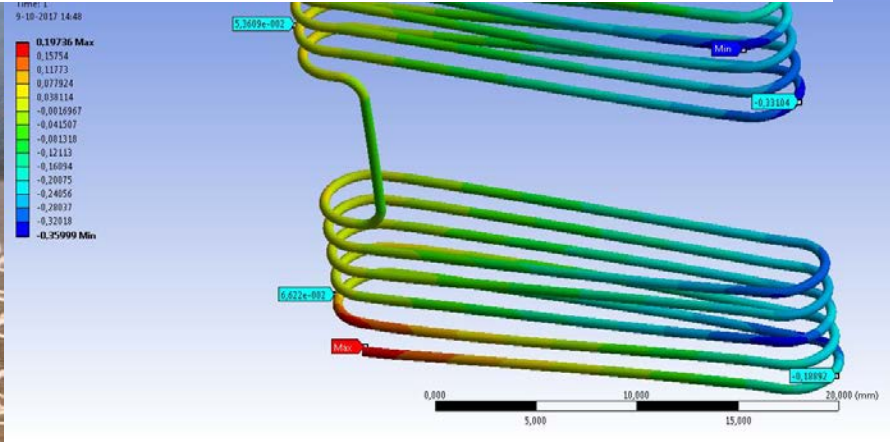
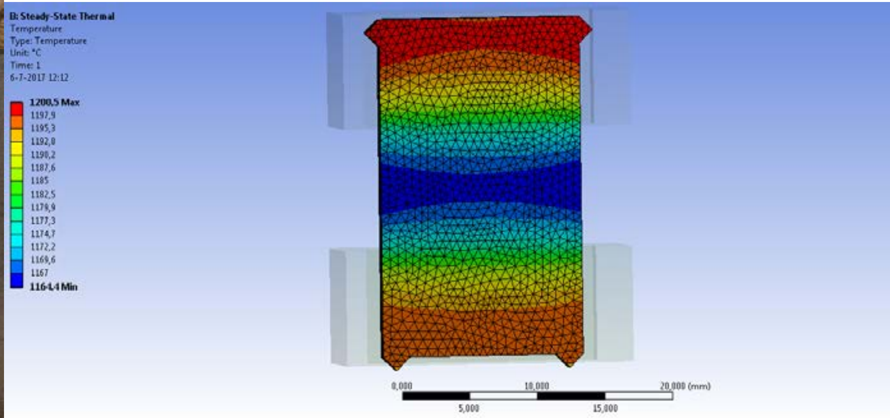
Experiments and FEM







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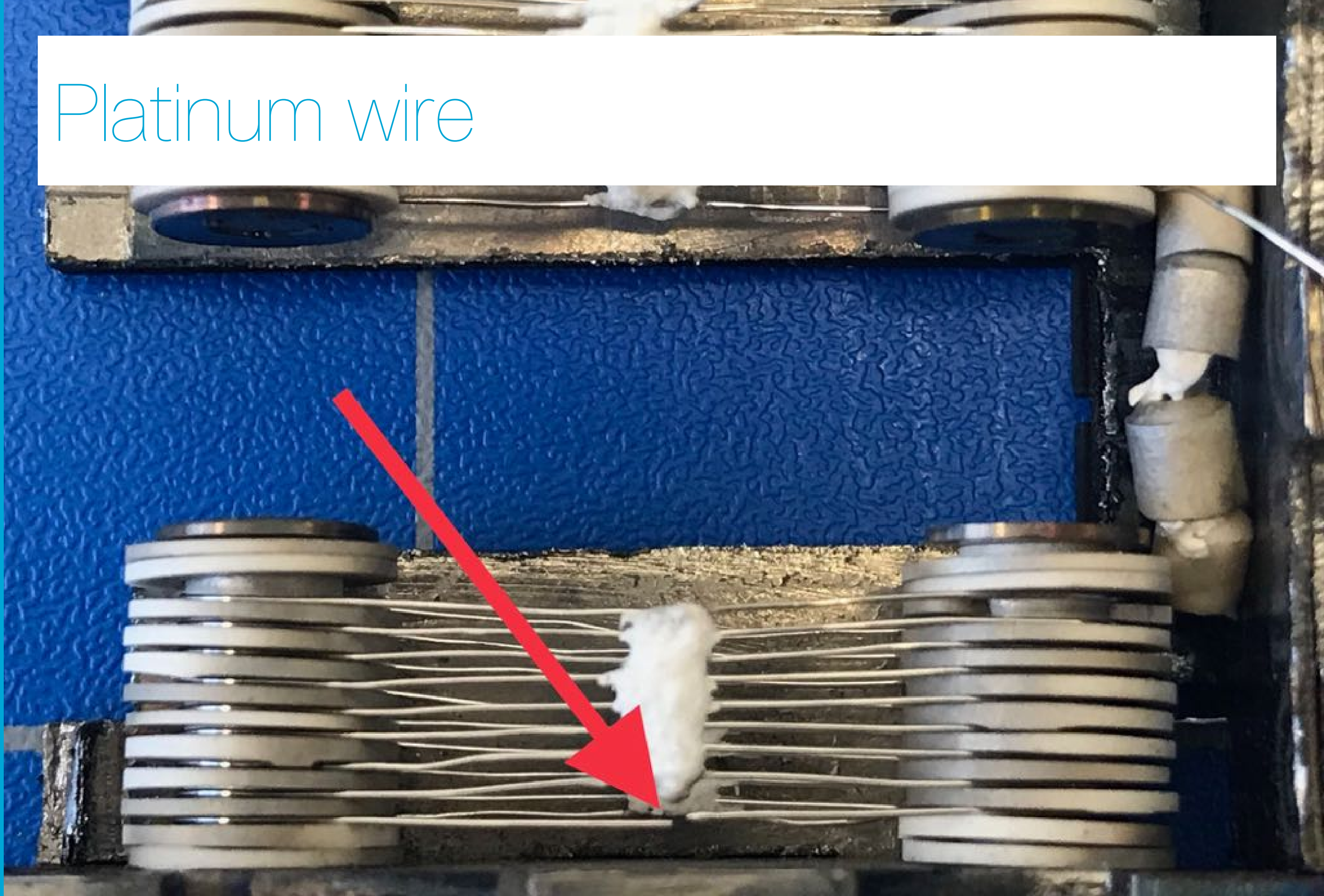
Platinum wire



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Heating wire

- ~~Platinum~~
- ~~Molybdenum~~
- ~~Tungsten~~
- Molybdenum-Lanthanum
  - Recrystallization Temperature: 1700°C
  - Cheap

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Prototyping

Prototyping

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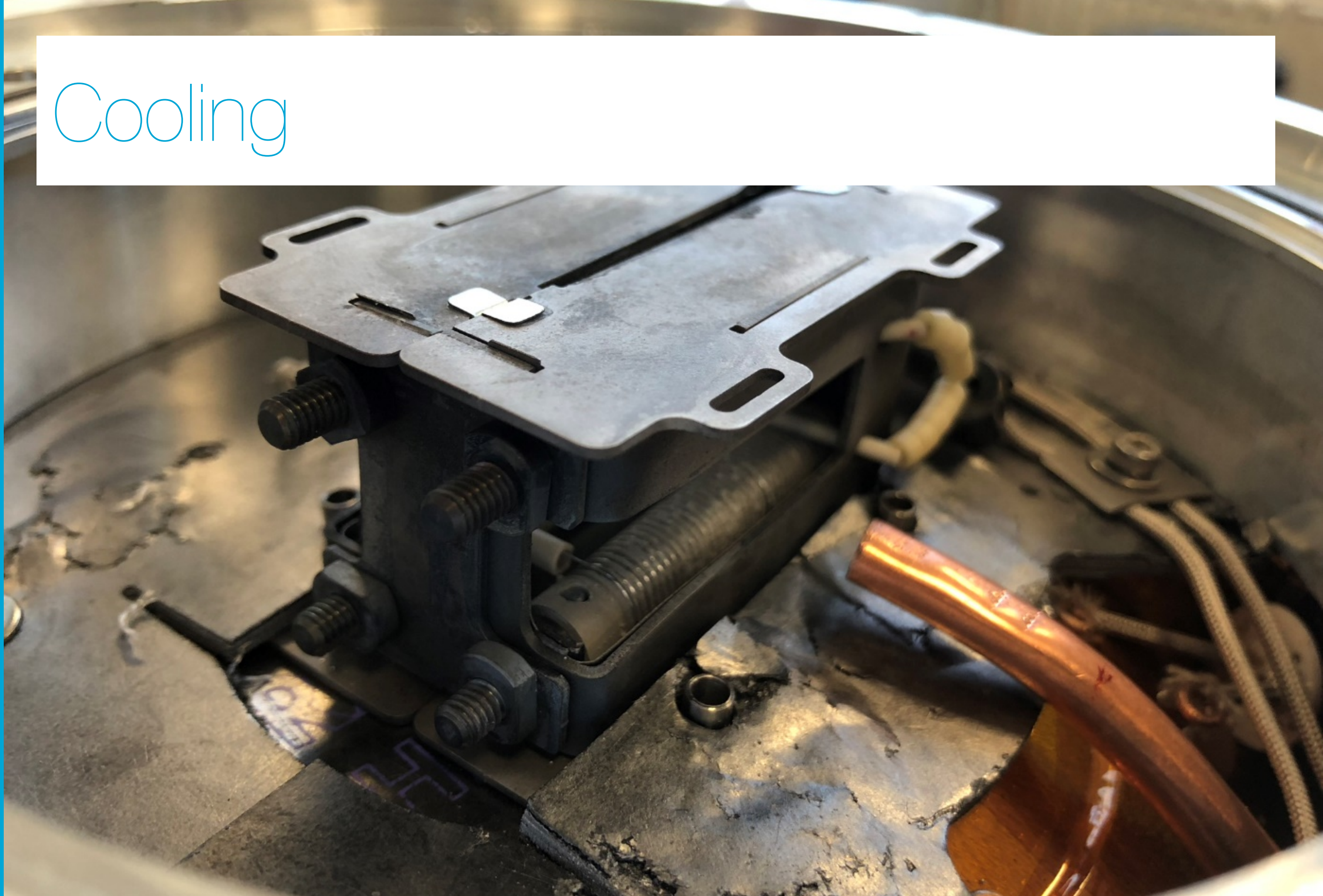
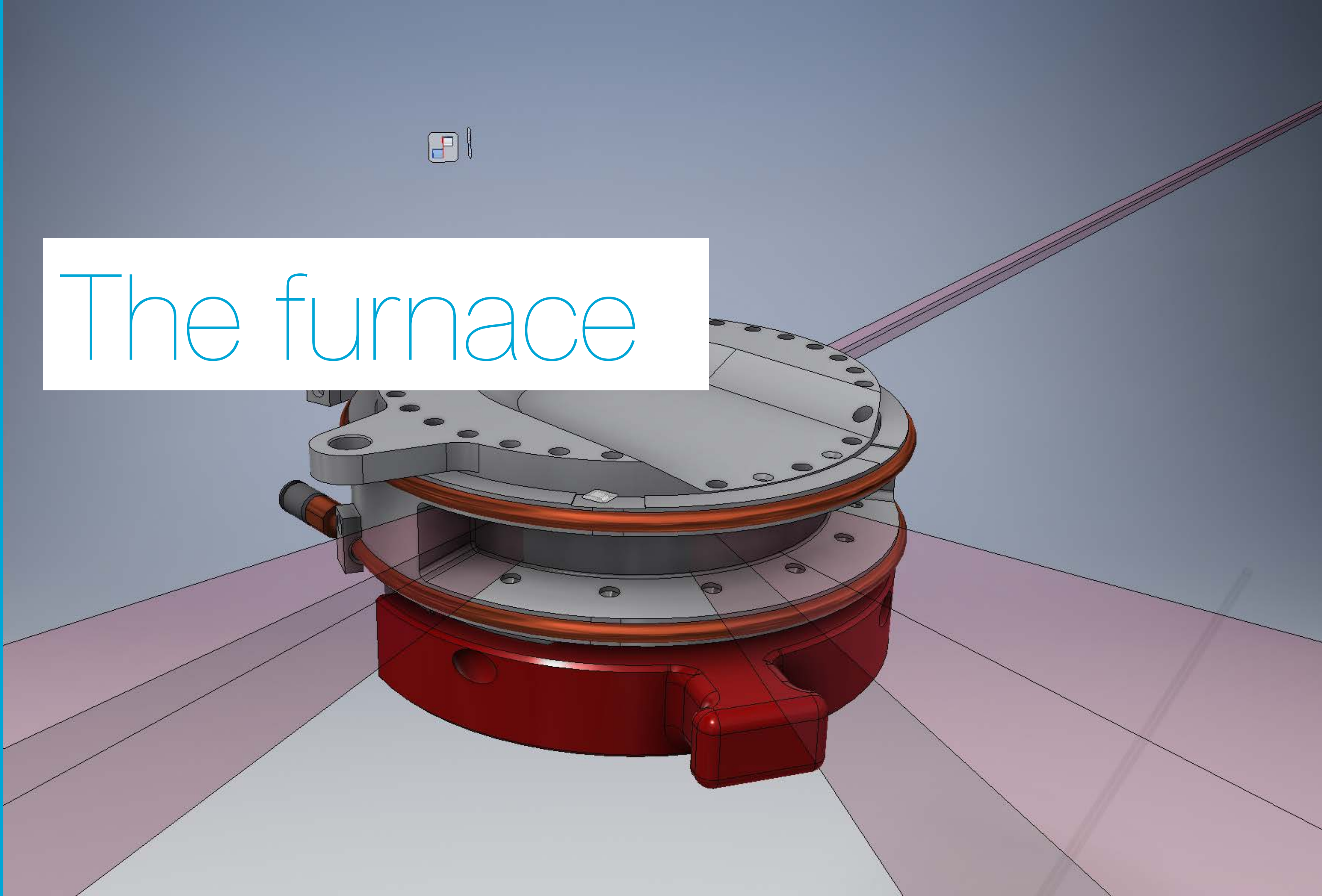
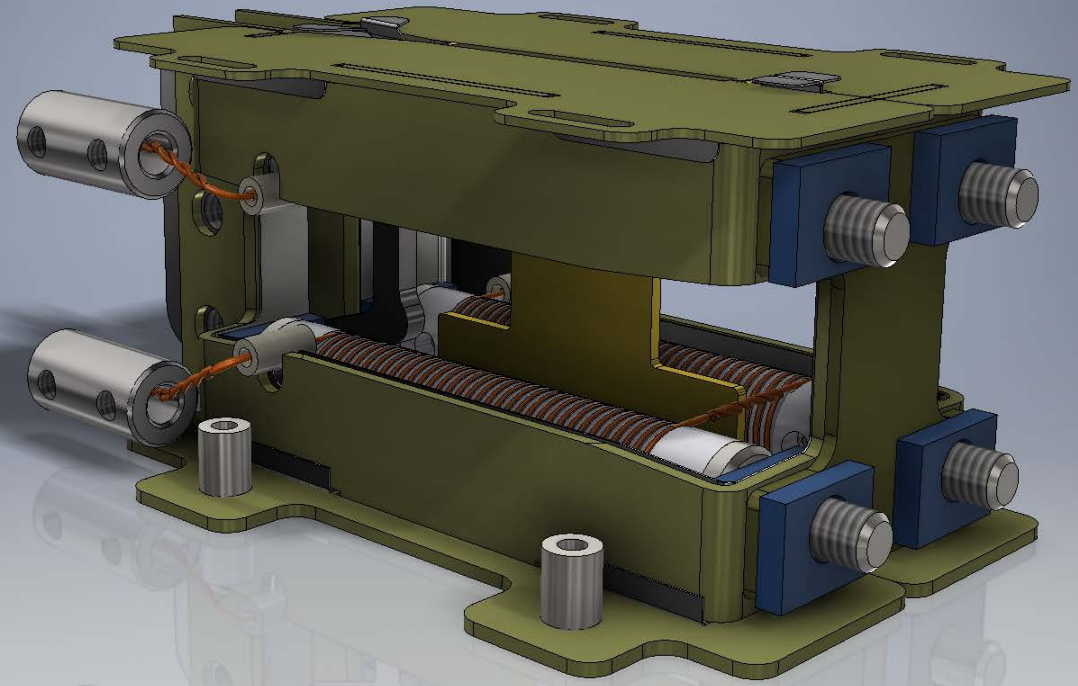
Cooling



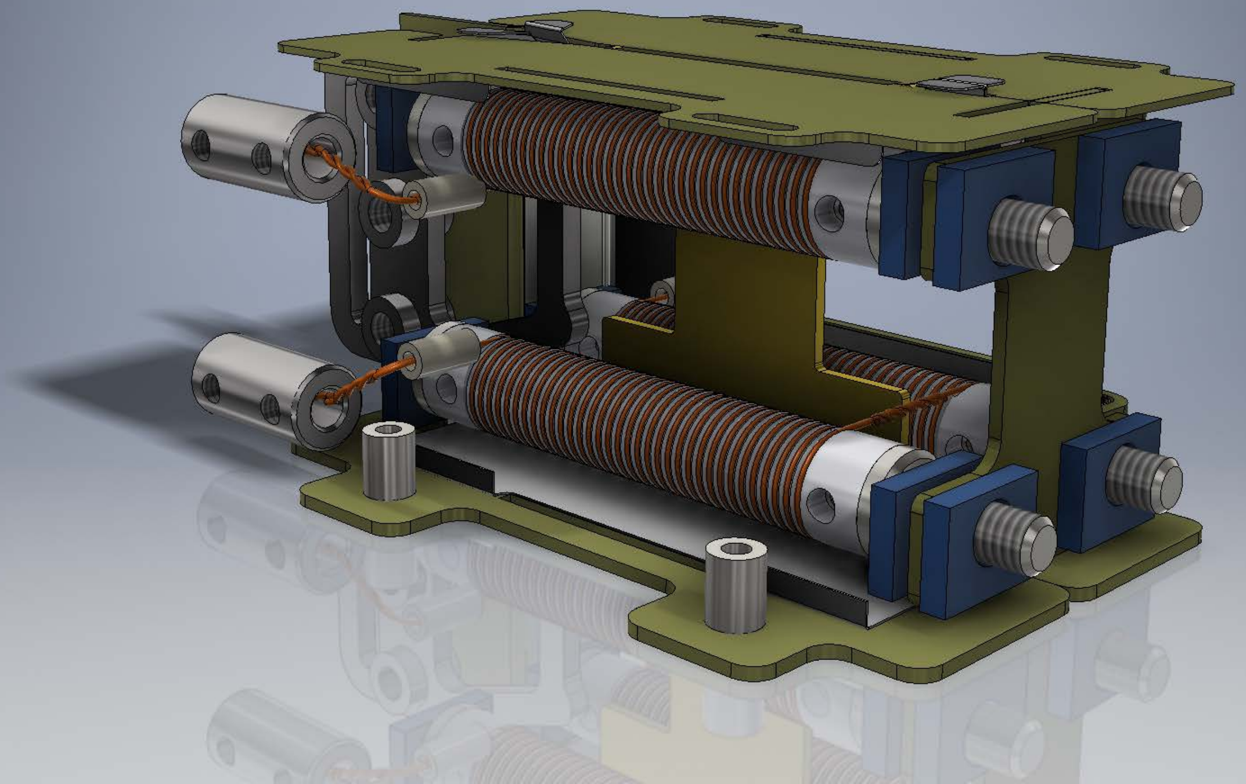
The furnace



Heating coil



Heating coil



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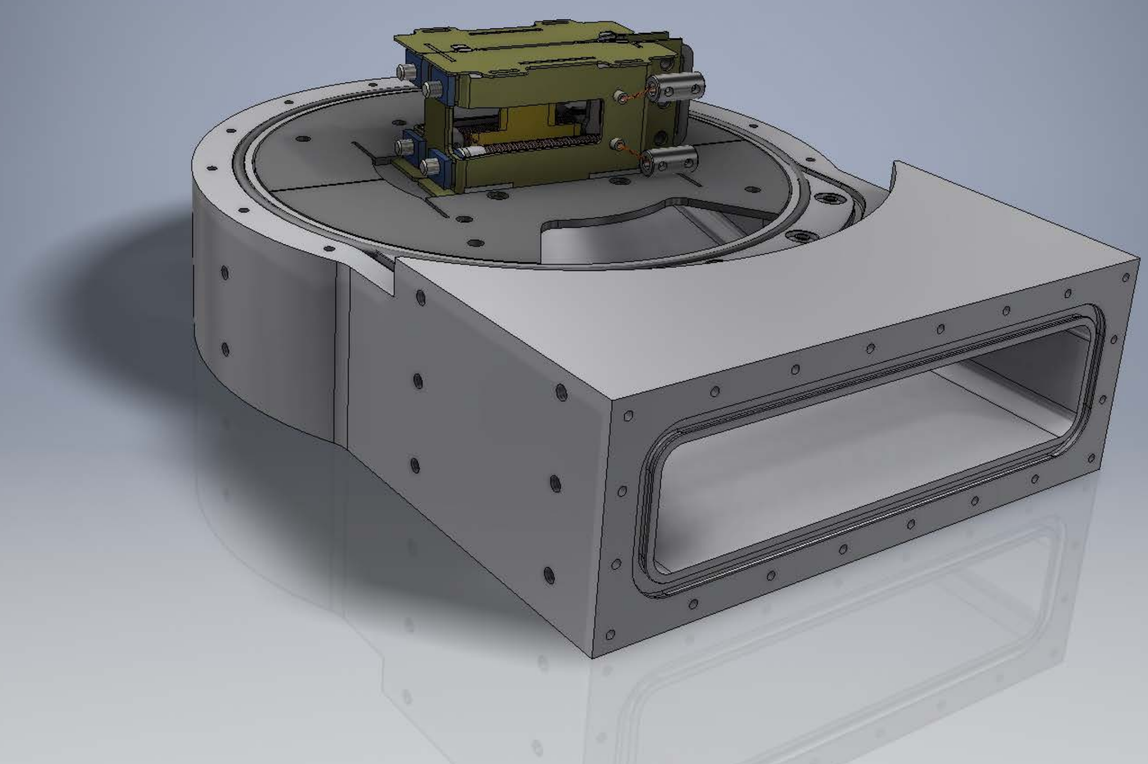
Base



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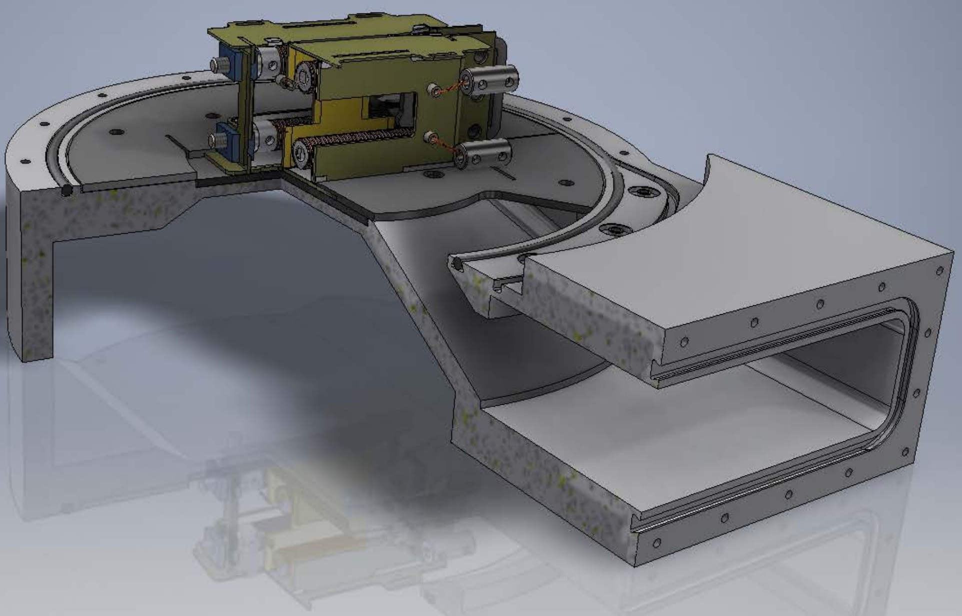
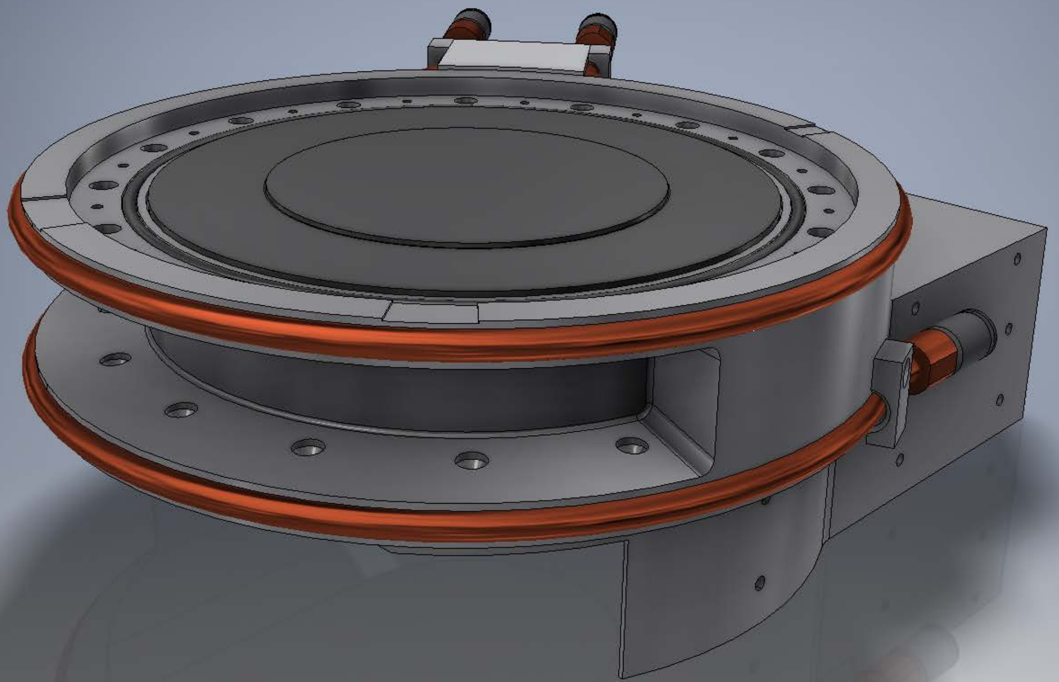
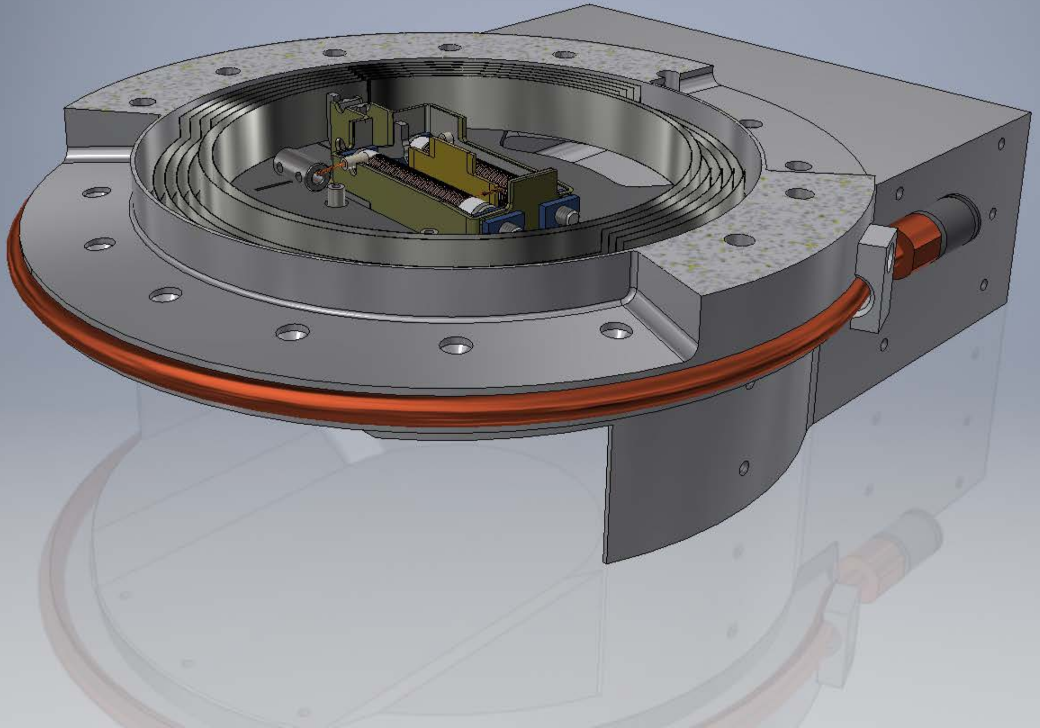
Base



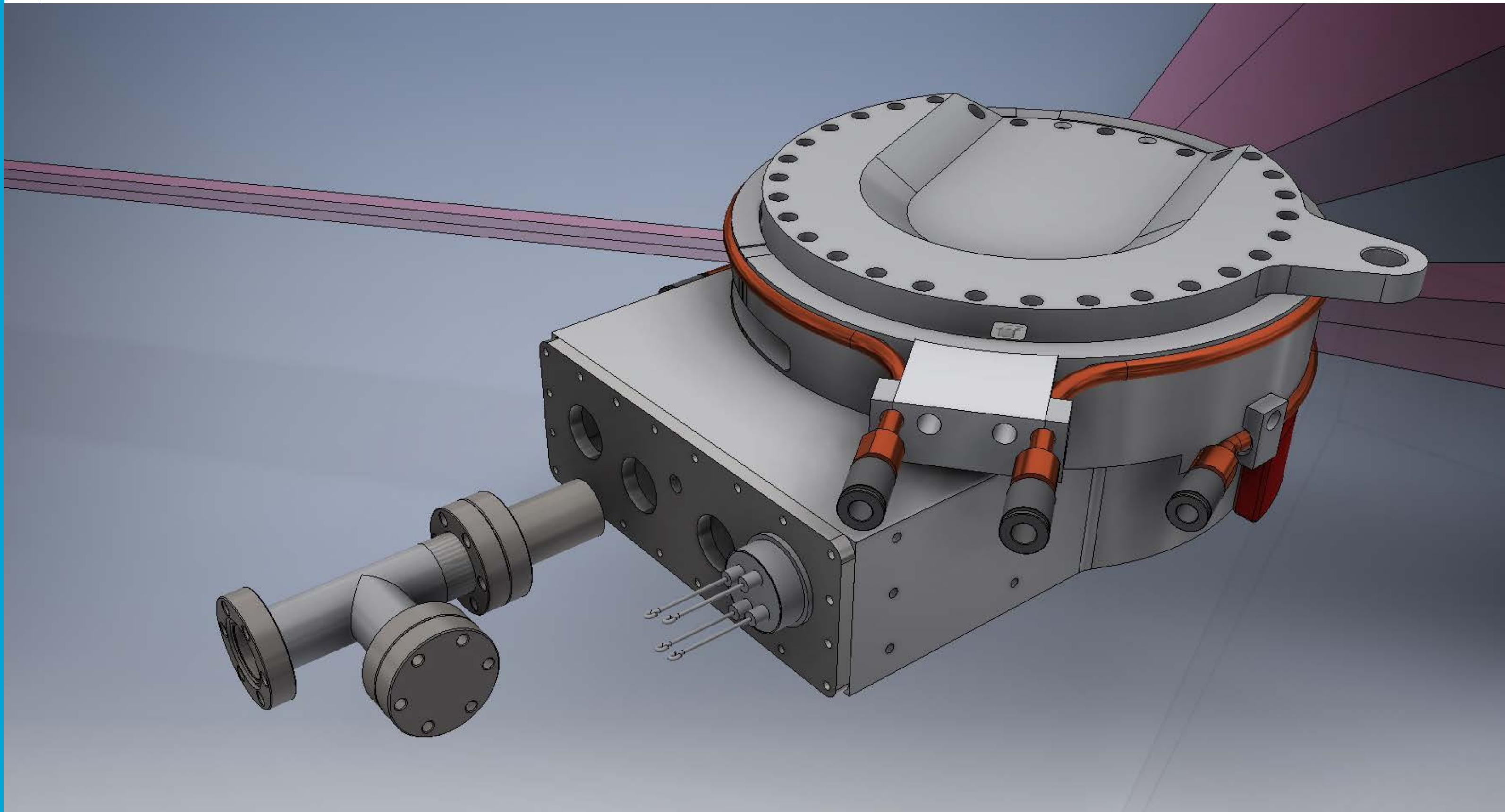
Window section and heatshields



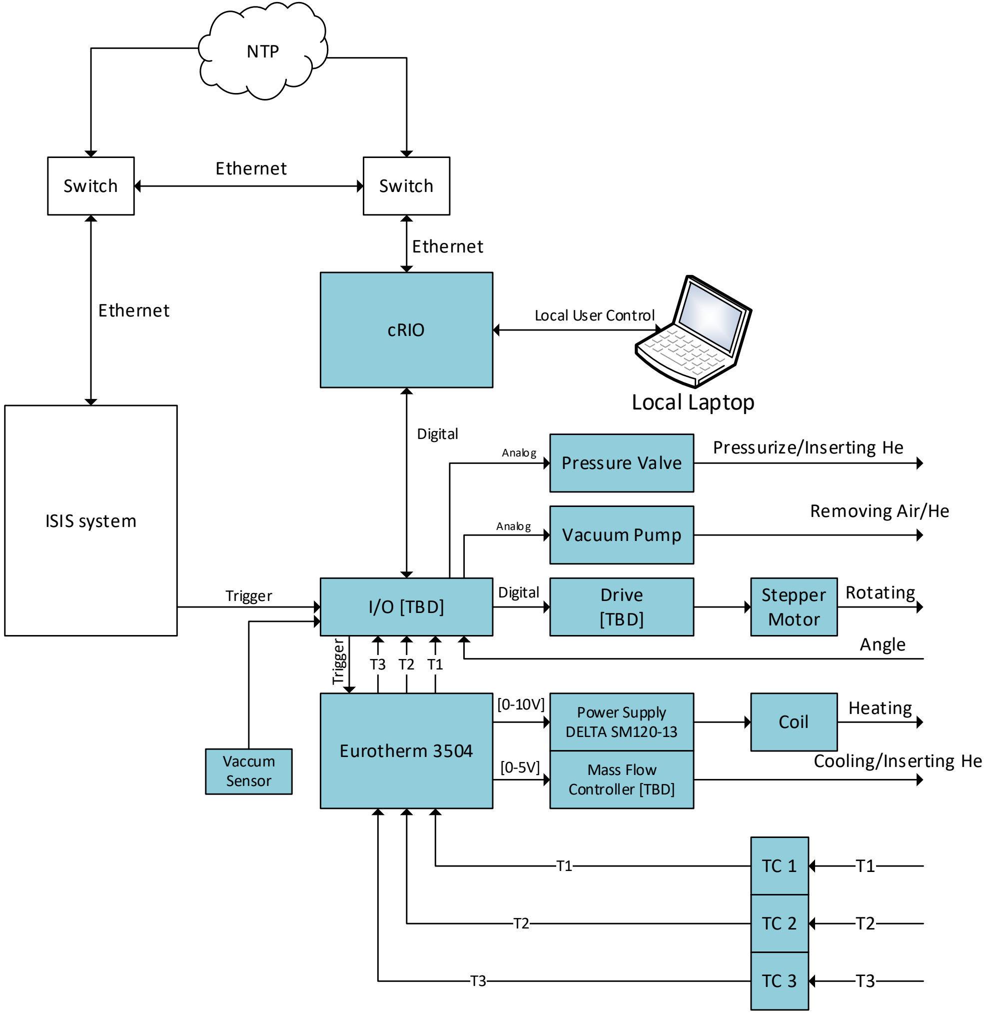
Window section and heatshields

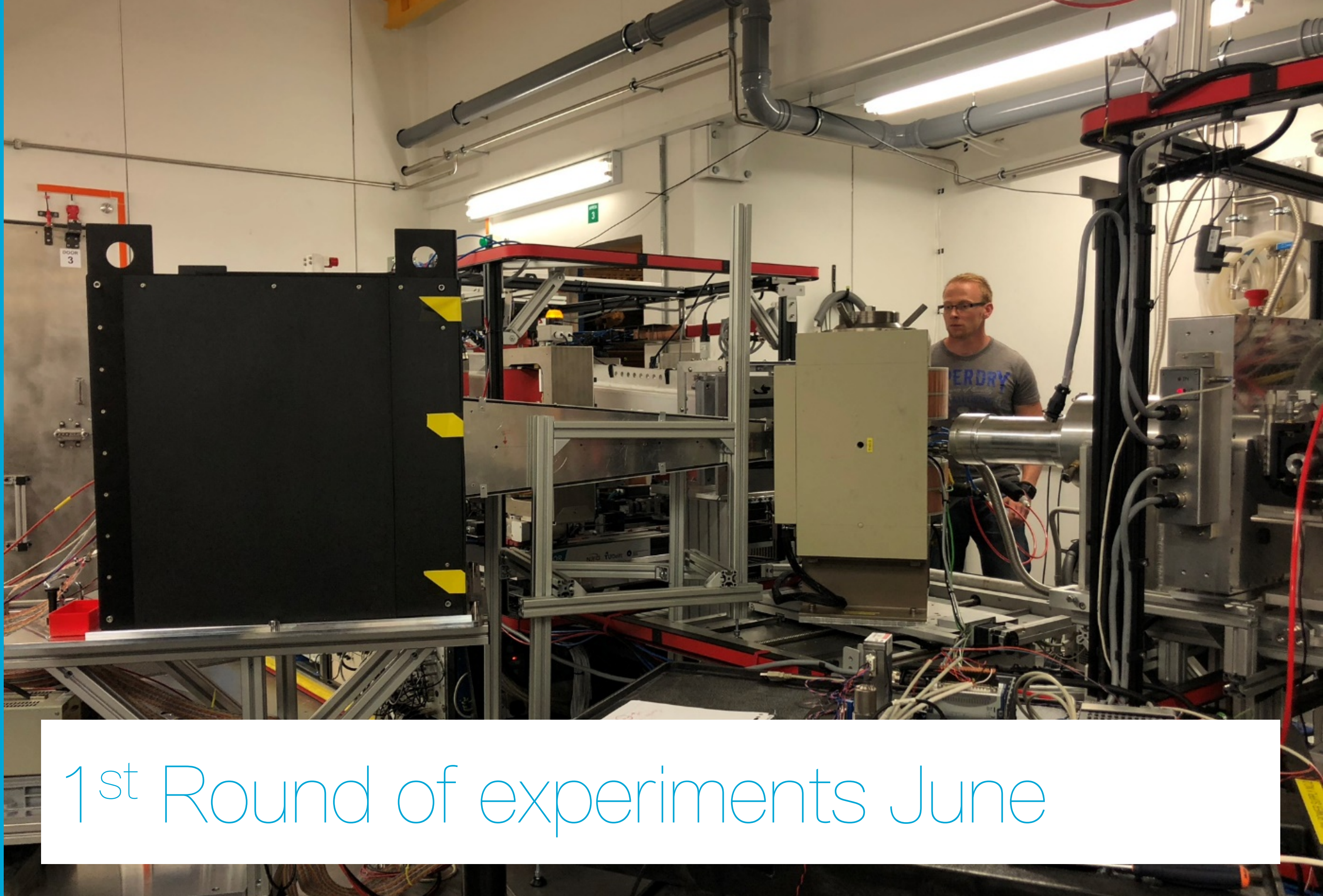


Lid and connections



Electronics





1^{st} Round of experiments June

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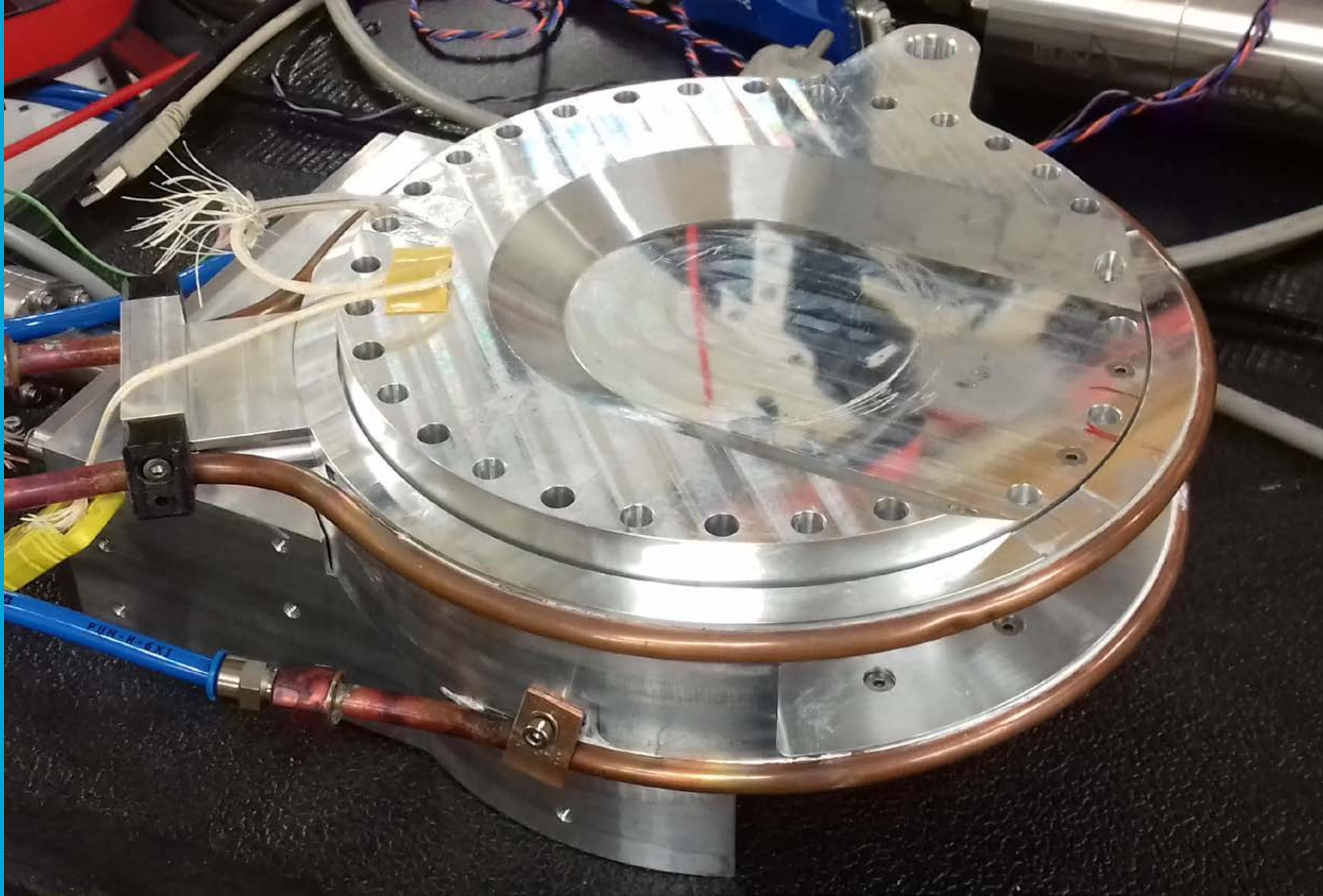

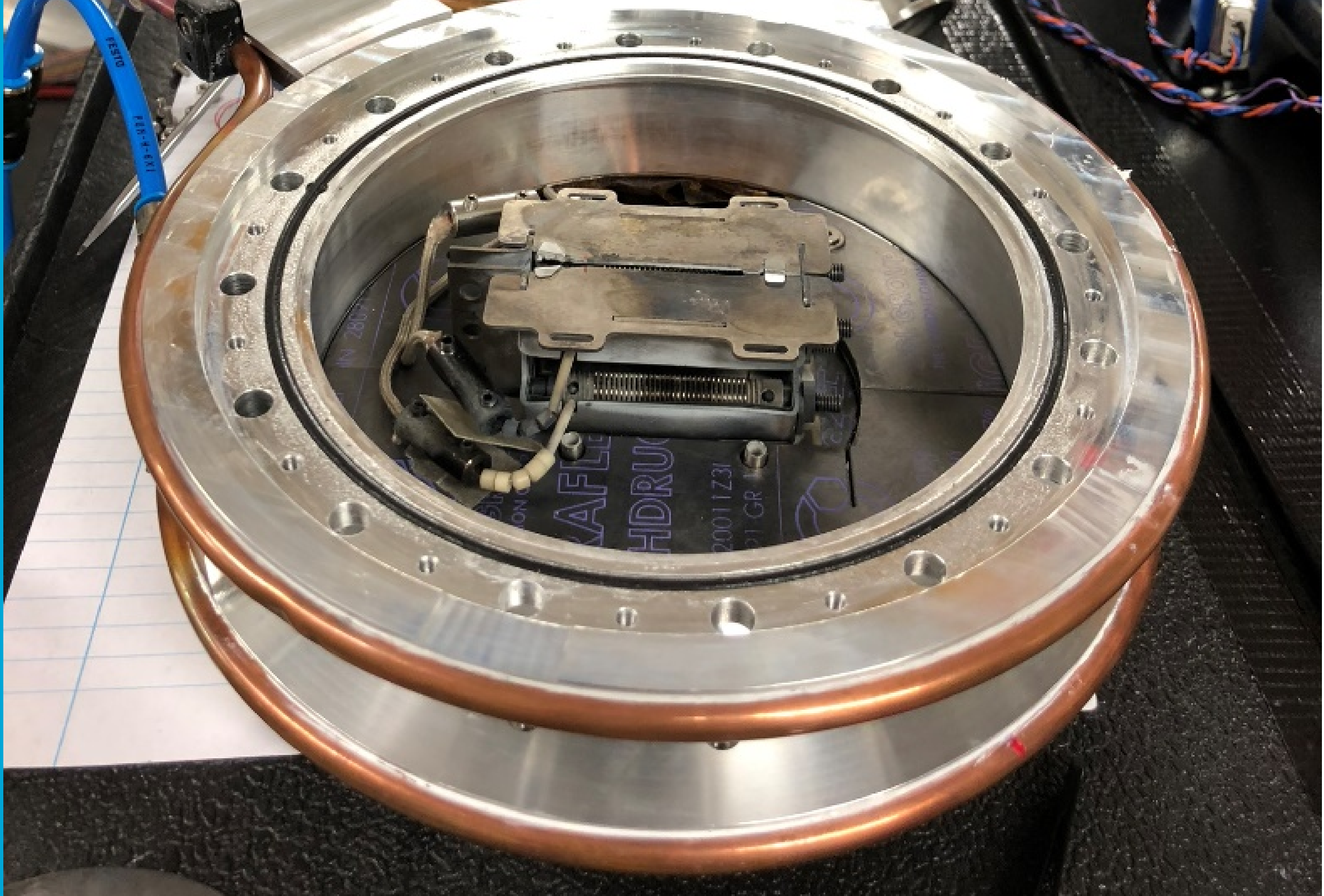

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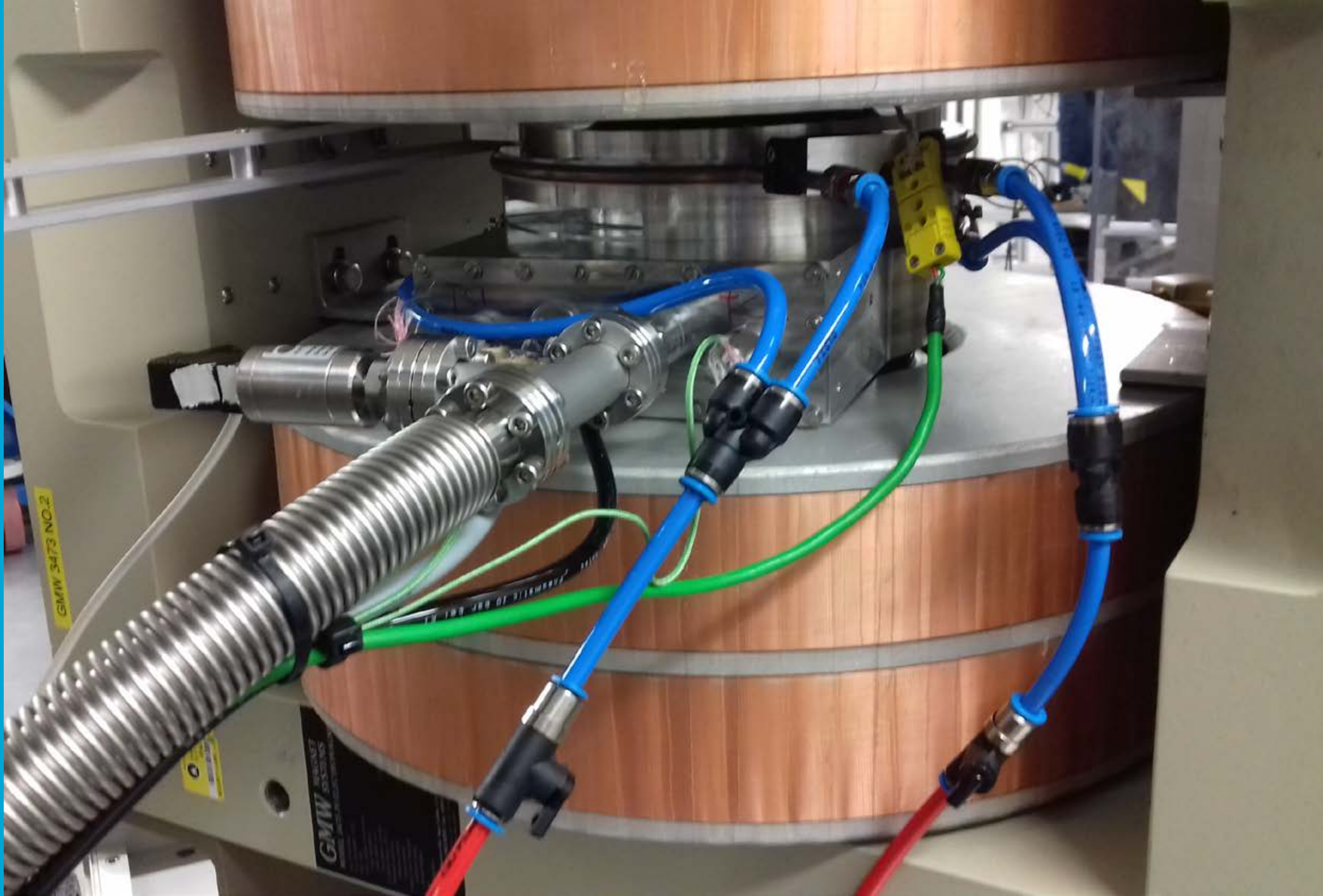

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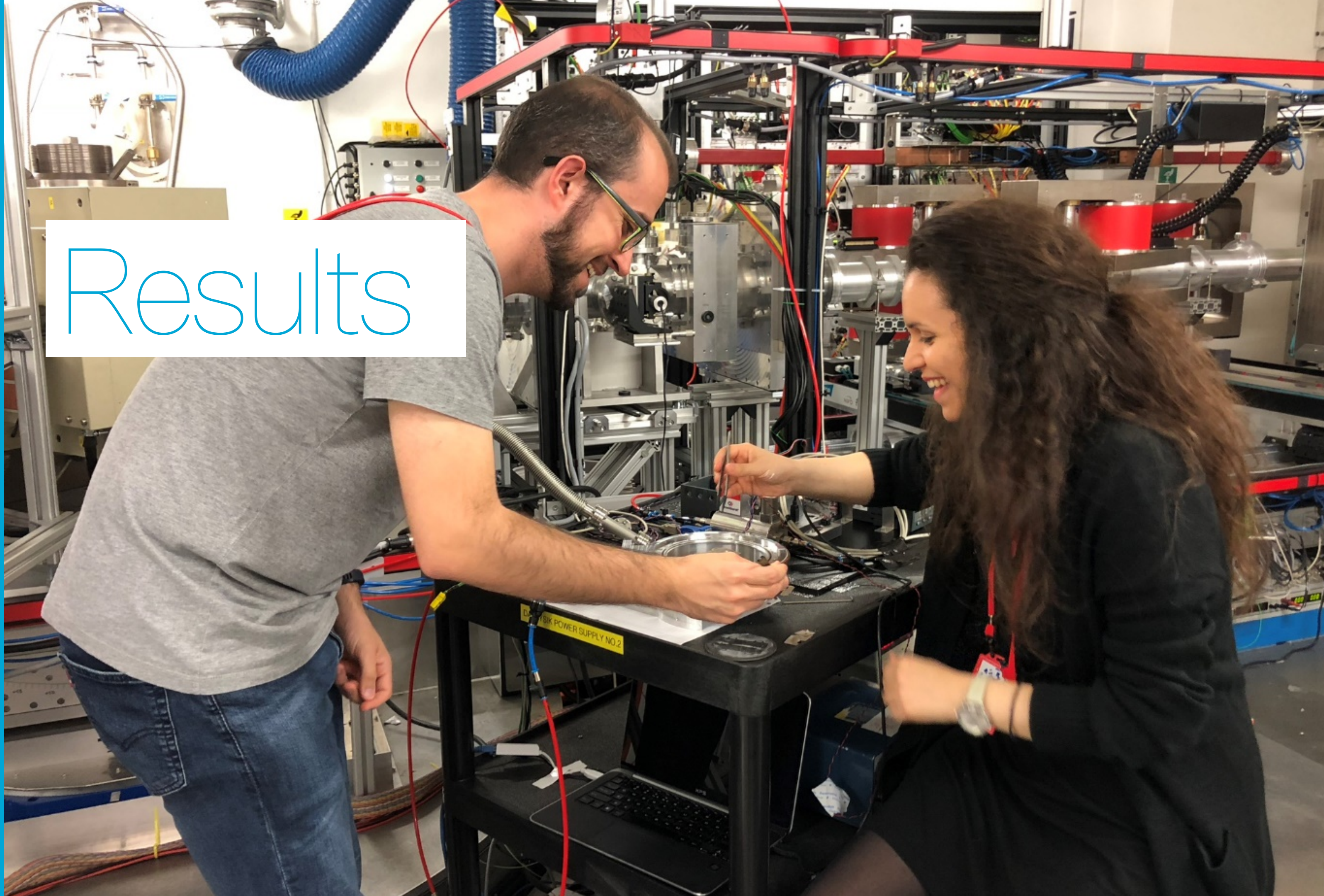
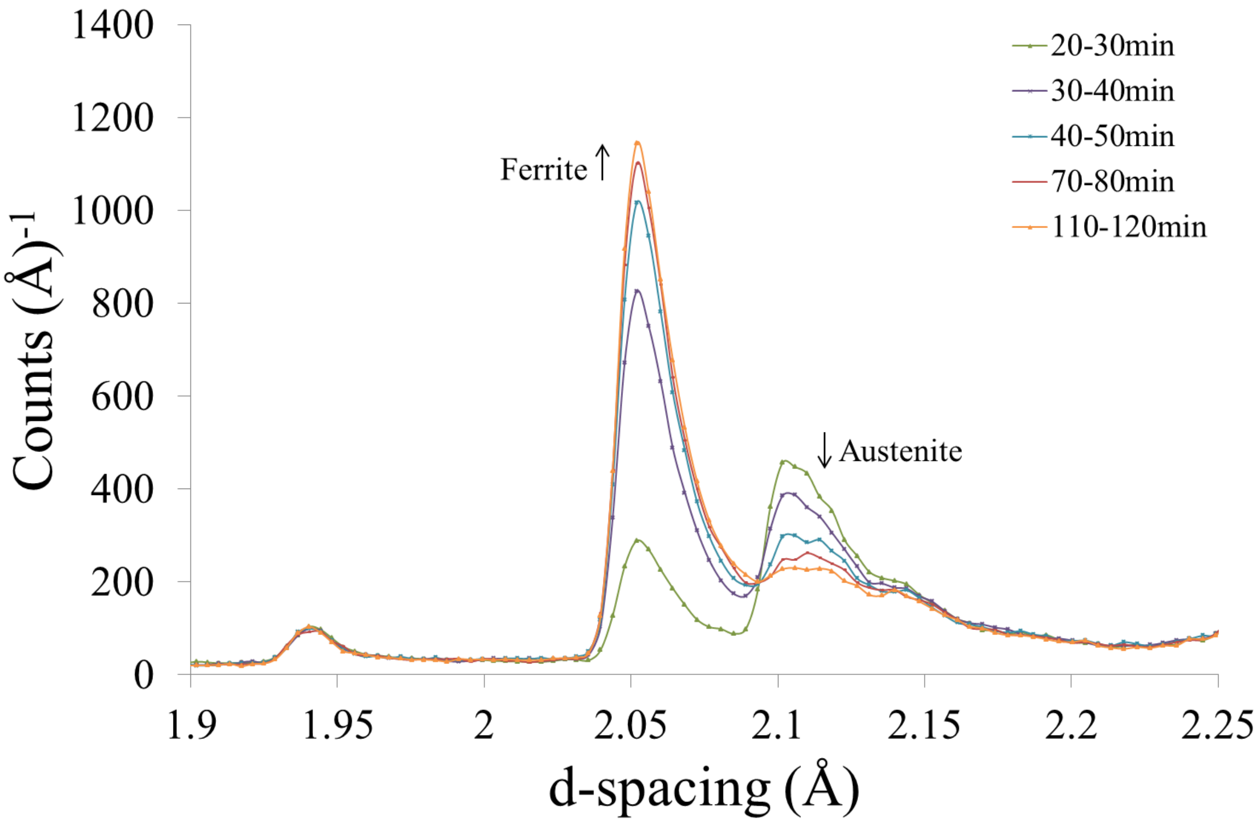
Heat treatment

Results



In-situ ND



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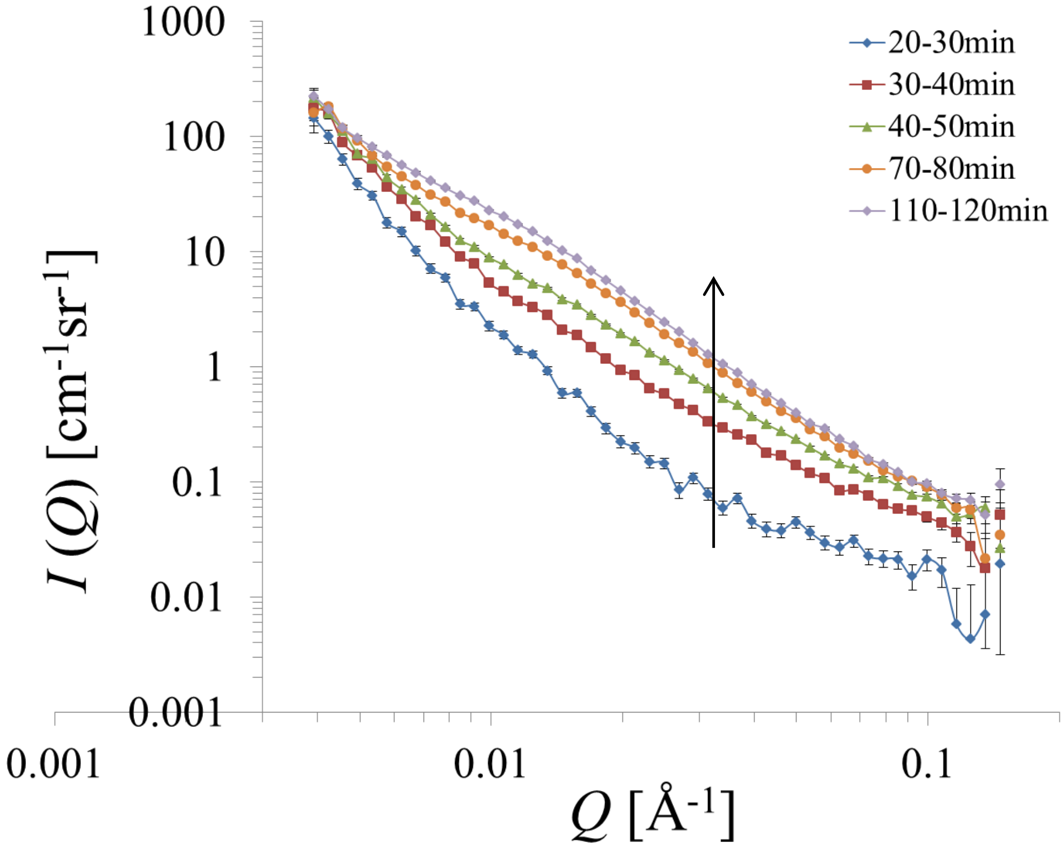
In-situ SANS



1000
100
10
1
0.1
0.01
0.001
I (Q) [cm-1sr-1]
0.001
0.01
0.1
Q [Å-1]
20-30min
30-40min
40-50min
70-80min
110-120min

Follow-up

Furnace improvements

- More stable temperature control system
- Improve electronics
  - Automation
  - Safety
- Better vacuum to prevent corrosion
- Neutron shielding
- 0 - 45° Rotation

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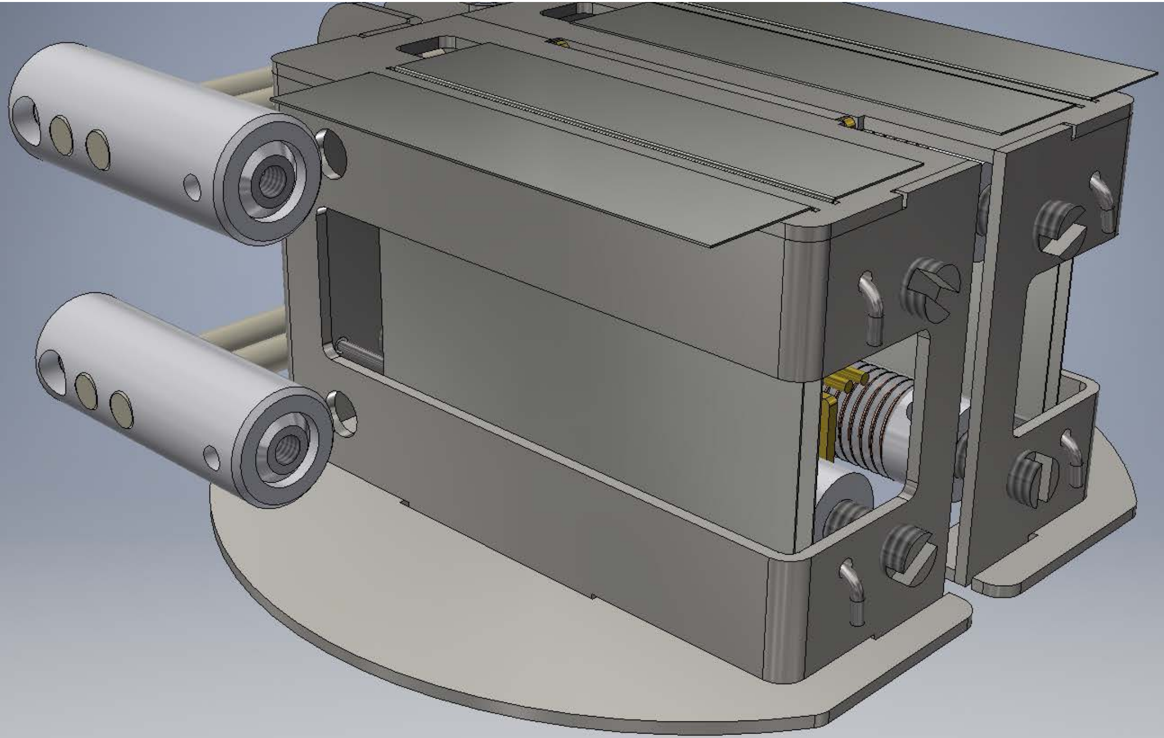
Heating coil improvements



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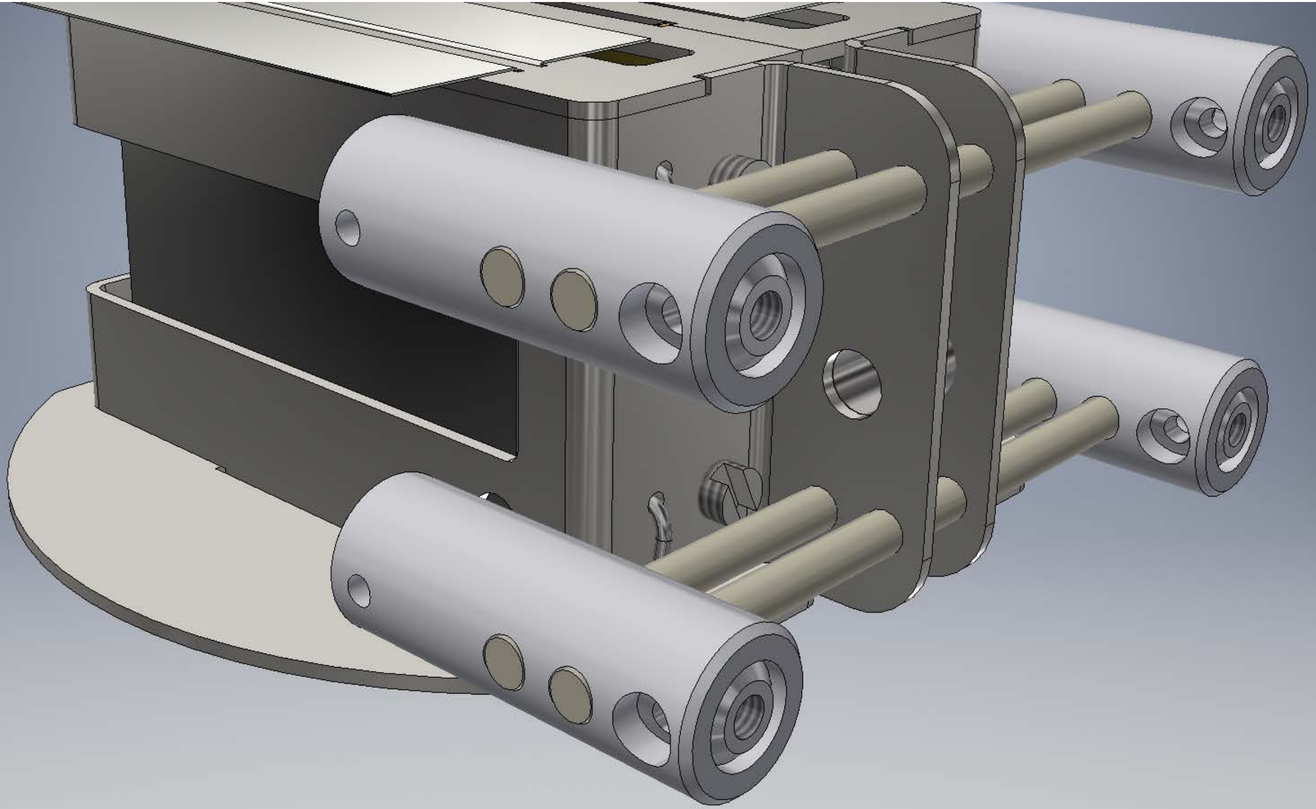
Heating coil improvements



Questions?

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