Development of Sample Environment at CSNS

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Outlines

- User Service
- Low Temperature & Magnetic field
- High Temperature & High Pressure
- Soft Matter & Control System
- Goals for the next few years

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User Service

- More than 25 sets of SE equipment used online
- Temperature range of 0.3K - 1600℃, 10GPa pressure, 9T magnetic field and some coupled fields for users
- Not only used in CSNS, but also in other devices' experiments such as ANSTO and CMRR


CCR-06


CCR-04


ATC-01

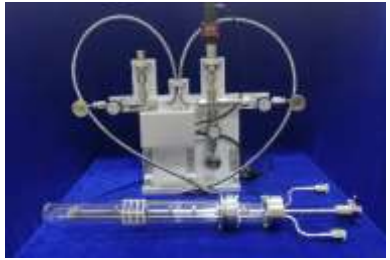

HOT-03


HOT-04


MAG-01


CPC-01


GP-01


Gas insert for furnace

SE list used in the beamlines

Main List		Equipment Name	Parameters	Beamline Used
1	CCR-02	Top-loading Cryostat	4.2-800K	GPPD
2	CCR-03		1.5-300K	GPPD, MPI
3	CCR-06		4.2-700K	GPPD, MPI
4	CCR-04		4.2-300K	GPPD
5	CCR-05	Bottom-loading Cryostat	5-500K	SANS, MPI, 9#BL
6	CCR-07		10-500K	SANS
7	ATC-01	Sample Changer for SANS	-30~130℃	SANS
8	ATC-02		25~300℃	SANS
9	HOT-01	Furnace	1600 ℃	20#BL
10	HOT-02		1000 ℃	GPPD, MPI
11	HOT-03		1230℃	SANS
12	HOT-04		1200℃	MPI
13	MAG-01	Magnet	9.0T, 0.3-325K	GPPD, MPI
14	MAG-02		2T@40mm	MR
15	MAG-03		5.0T, 2-700K	SANS
16	CPC-01	Clamp Cell	2.0GPa	MPI、ANSTO Pelican
17	CPC-02		1.0GPa	ANSTO Pelican
18	CPC-03		0.8GPa	MPI, GPPD
19	HPC-01	CSNS Cell	5.0GPa	CMRR HPND
20	GP-01	Gas Cell	20MPa	GPPD、SANS、MPI
21	Inserts	Gas Inserts × 4	0.1MPa	SANS、MPI
22	Rheo-01	Rheometer	50nNm~230mNm	SANS、VSANS

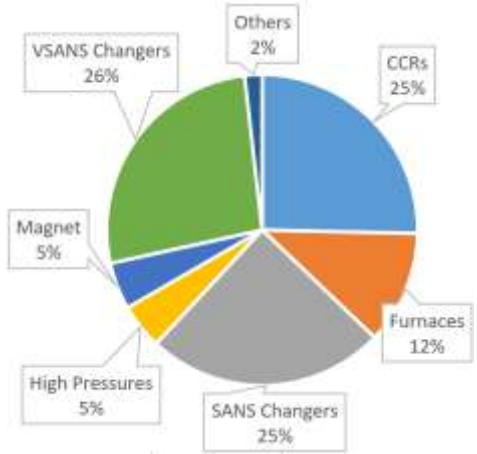
Subtotal of services in the past two cycles

SE Service time in the past year

Code	Equipment name	Beamline	Days	Sample-change times
CCR-03	CCR-toploading	MPI	15	12
CCR-05	CCR-bottomloading	MPI	8	16
CCR-06	CCR-toploading	GPPD	48	60
		MPI	65	90
CCR-07	CCR-bottomloading	SANS	4	1
HOT-02	Al Furnace	GPPD	8	10
HOT-03	SANS Furnace	SANS	22	45
HOT-04	V Furnace	MPI	35	52
ATC-01	auto Changer@oil bath	SANS	92	19
ATC-02	auto Changer@heater rod	SANS	45	17
ATC-03	auto Changer@diff. T.	VSANS	147	6
CPC-04	TiZr Cell@CCR06	MPI	8	8
CPC-04	TiZr Cell@Mag-01	GPPD	6	4
GI-01@HOT-03	Quartz Gas insert@furnace	SANS	1	2
GI-02@HOT-04	Quartz Gas insert@furnace	MPI	3	4
GI-03@HOT-04	V Gas insert@furnace	MPI	1	3
GP-03@CCR05	Gas Cell@CCR05	MPI	5	10
GP-03@CCR06	Gas Cell@CCR06	MPI	2	4
Mag-01	9T Magnet	GPPD	17	20
		MPI	2	3
Mag-02	5T Magnet	SANS	8	10
		Total	542	396

- more stable compared to the early stages
- 542 days and 396 times replacements for users
- Temperature related equipment used by users accounts for 88%.

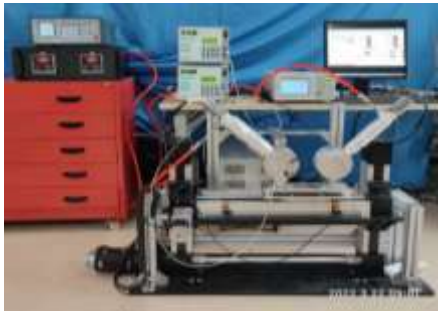
Classification statistics



Optimization and Development

- The necessary optimization, research and development work is being carried out. Some examples:

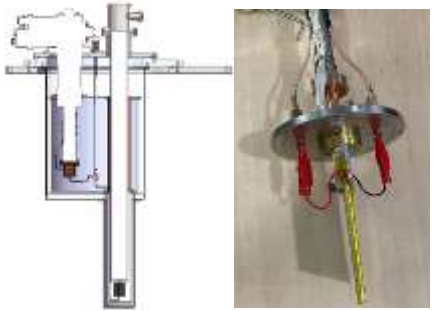
Temperature Jumper for VSANS/SANS



SANS/VSANS Rheometer commissioning

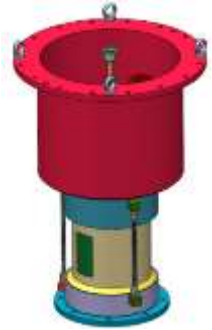
1.5K Cryostat, 4K CCR in independent R&D

Battery testing device with CCR

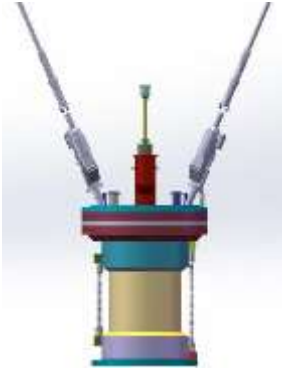


Auto sample changer with 1.5K~500K

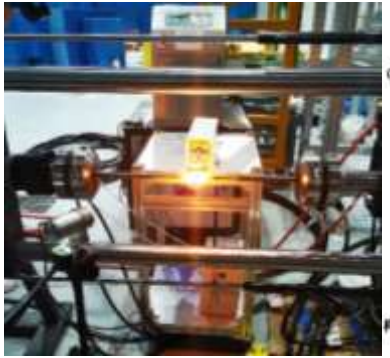
Furnace with lower background & better performance



Furnace with higher temperature ≥ 2000℃



Induction heating furnace for load-frame



7T magnet for inelastic scattering

5T magnet coupled with CCR for MR/SANS

Outlines

- User Service
- Low Temperature & Magnetic field
- High Temperature & High Pressure
- Soft Matter & Control System
- Goals for the next few years

Development and optimization of CCRs

CCR-01 CCR-02 CCR-03 CCR-04 CCR-05 CCR-06 CCR-07 CCR-08

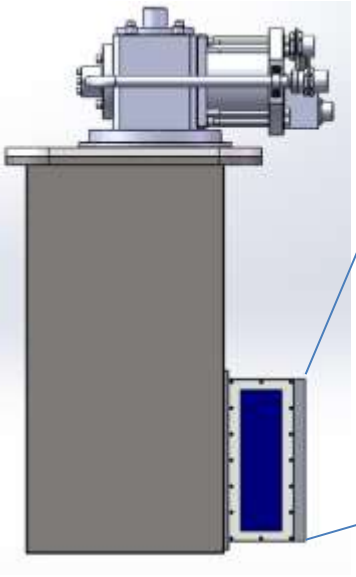
Parameters	2019	Right Now（2023）
Types	only 1, top-loading	3, top-loading, bottom-loading, multi-sample mode
Diameter of sample area	≤ 60mm	≥ 100mm
Temperature range	1.5 ~ 600 K	1.5K ~ 800 K
Window material	aluminum alloy	Al alloy, V, Single crystal sapphire
Types of instrument served	powder diffraction	powder diffraction, inelastic scattering, SANS

CCRs Development - Example 1

❑CCR for SANS/VSANS

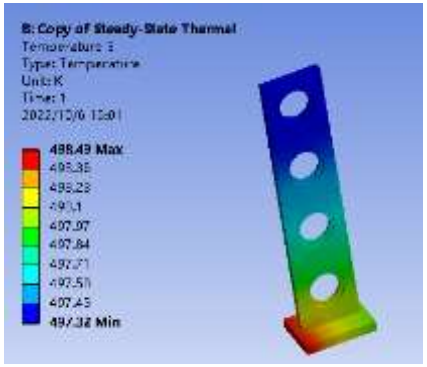
- Temperature range: 10~500K
- Based on the type of Bottom-Loading structure
- Sample numbers: 4
- combined with the multi-dimensional motion table of SANS/VSANS achieves precise control of sample position.

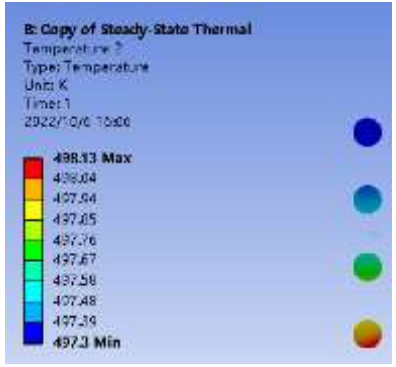
Design



Sample rack

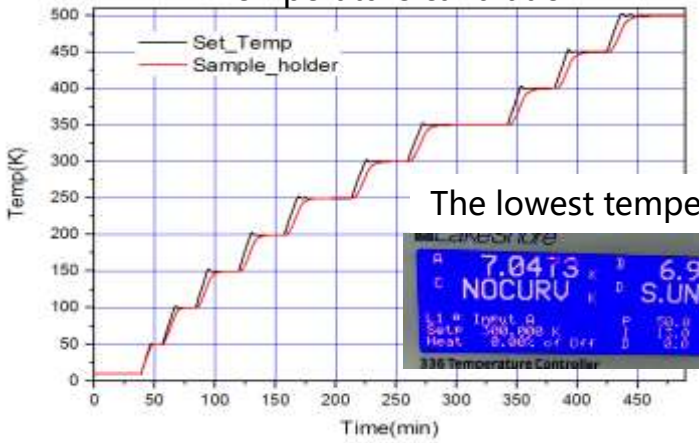
Photo





Temperature simulation: Sample rack, Samples

Temperature calibration



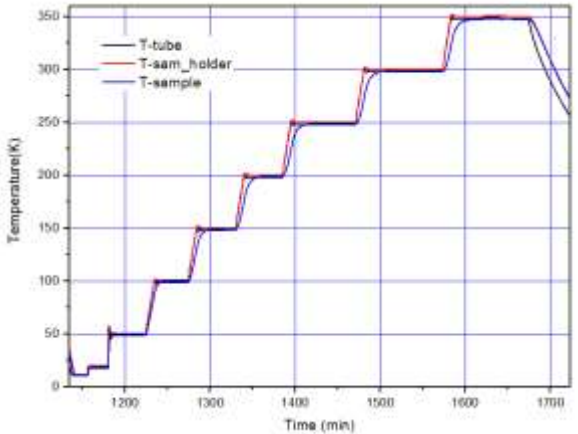
The lowest temperature

CCRs Development - Example 2

In situ charging/discharging CCR

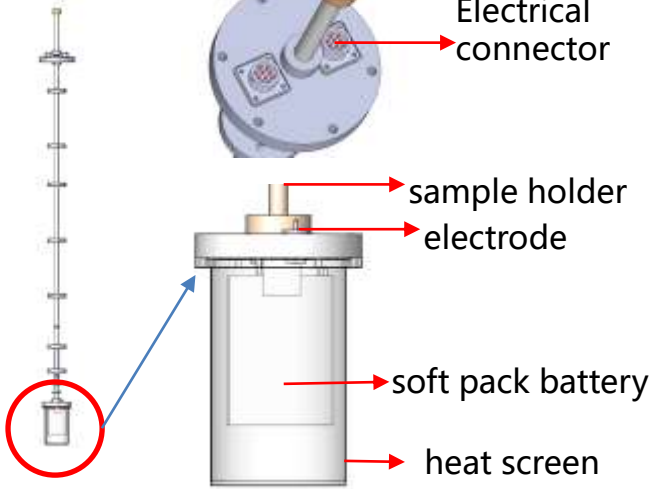
- Main application areas: battery research
- Temperature range: 5~800K （frequently-used 223~473 K）
- Charging/discharging voltage: 0~5 V, current: 0~5 A
- Ensuring accurate sample temperature:
  - low temperature dual temperature control
  - high temperature multi-layer heat screen
- Replaceable design of neutron beam window

Temperature calibration test



Design

Sample probe

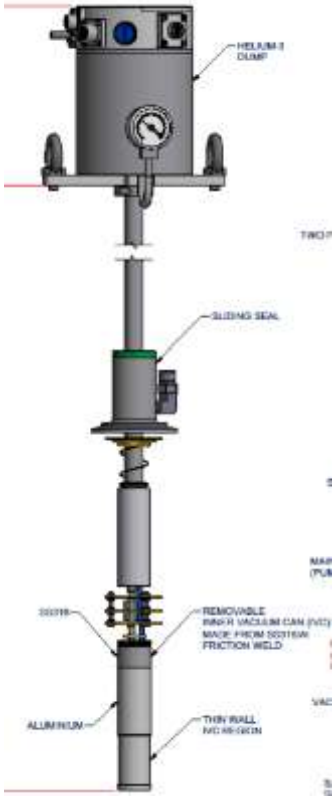


Sample Changer with Low Temperature

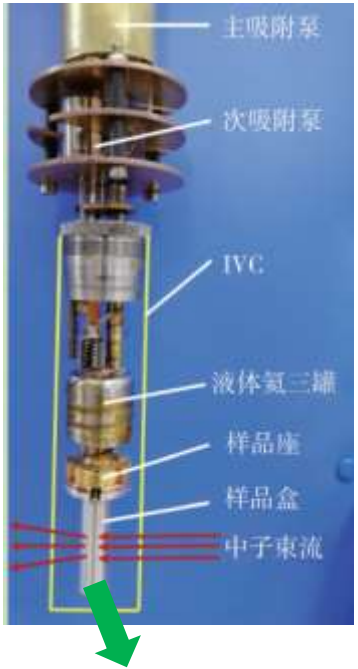
- Temperature range: 1.5 K – 500 K
- Number of samples: 6 （The untested sample is in the room temperature zone）
- Materials for neutron beam windows: TiZr

Temperature Control Optimization of He3 Insert

He^3 Inert



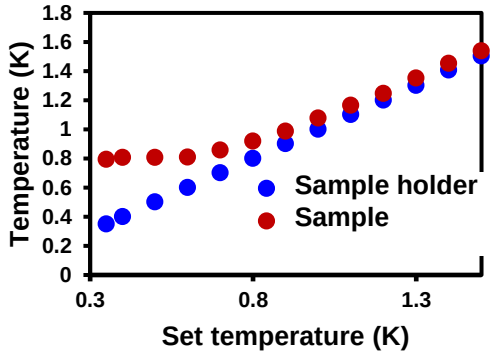
Picture



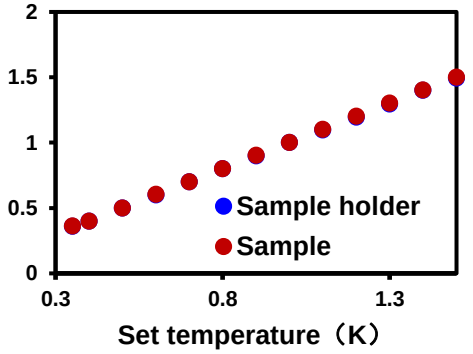
Sample actual temperature?

Helium film (overpressure filled)

0.1 MPa ^{4}He



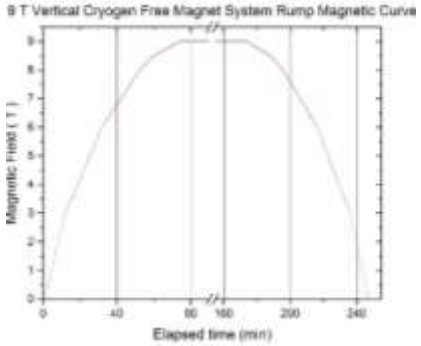
1 MPa ^{4}He

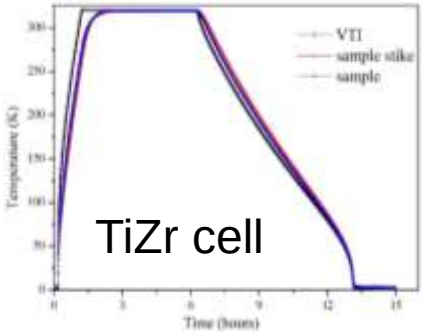


9 T vertical cryogen free magnet

Property	Value
Sample access	50 mm diameter
VTI temperature range	1.5 K - 320 K
Temperature accuracy	0.2% of set temperature
Central field range	9.0 Tesla
Nominal operating current	196.6 A
Central field uniformity	0.9% within Ø10mm x 20mm region 2.5% within Ø25mm x 30mm region

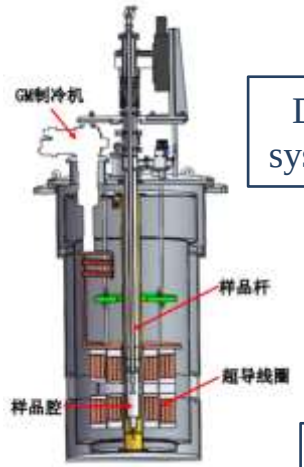
Property	Test value
Field range	0 – 9 T
Ramp time to full field	76 mins
VTI T range	1.5 – 320 K
Sample T range	2.5 – 320 K
Ramp time to lowest T	7.5 hours /3.5 h (manual)
Ramp time to maximum T	2.5 hours



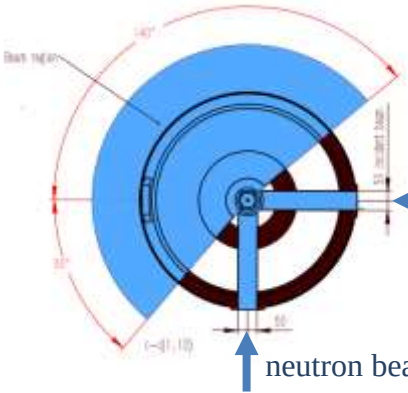


Asymmetric superconducting magnet for inelastic scattering

Maximum magnetic field: symmetric mode - 7 T; Asymmetric mode - 5 T; Lowest temperature - 1.5K



Dry system

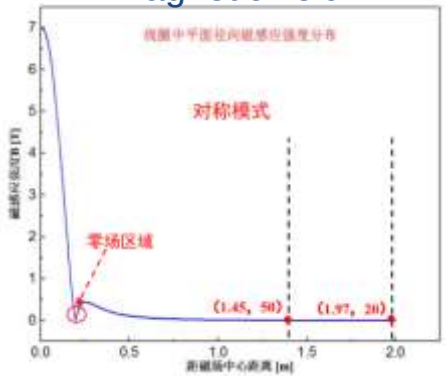


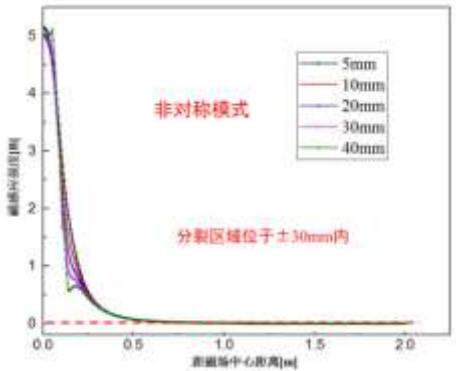
two neutron incidence channels

neutron beam

neutron beam

Magnetic field

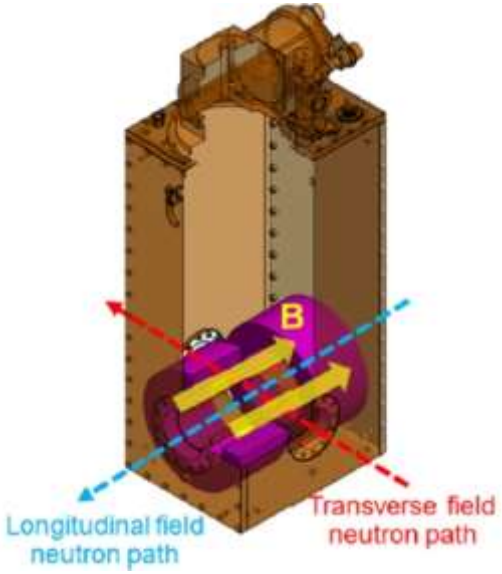




Magnet assembly

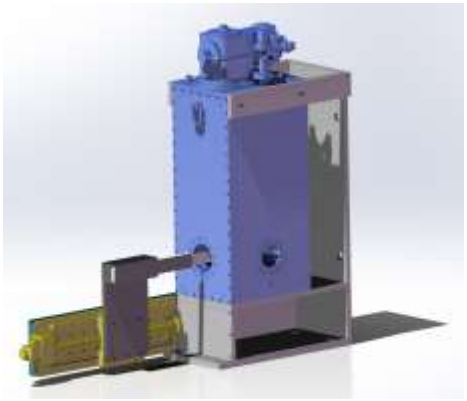
5T Magnet & 2K CCR System

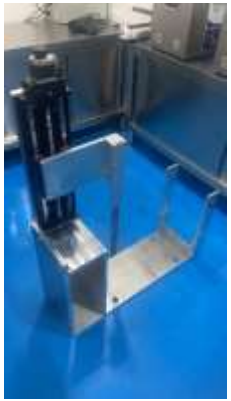
- The system contains a 5T SC magnet with room temperature bore and a 2-700 K cryostat, which can be applied in SANS, VSANS and MR neutron instruments.
- The 5T magnet can be coped with room temperature sample changer with up to 8 sample slots.
- The neutron window of the cryostat can be Aluminum or sapphire.



5T Magnet with RT bore

5T Magnet in SANS





Room Temperature Sample Changer

Outlines

- User Service
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Optimization of the Foil Heating Furnaces

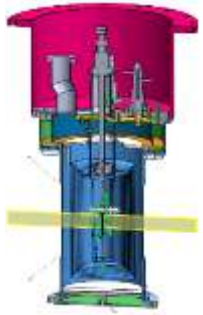
1) Optimization of temperature control performance

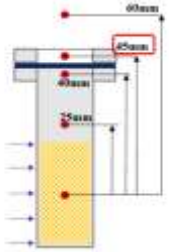

Contents lists available at ScienceDirect
Nuclear Inst. and Methods in Physics Research, A
journal homepage: www.elsevier.com/locate/nima

Full Length Article

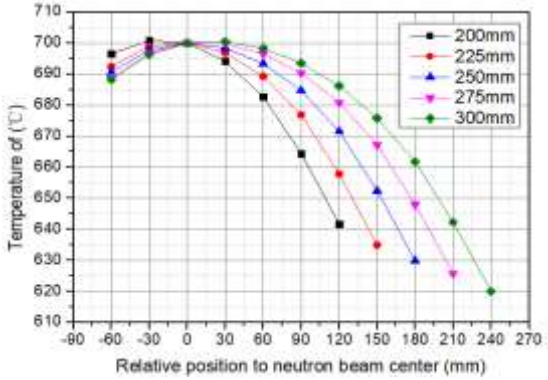
Experimental and numerical investigation the radiant heating element in neutron scattering furnace

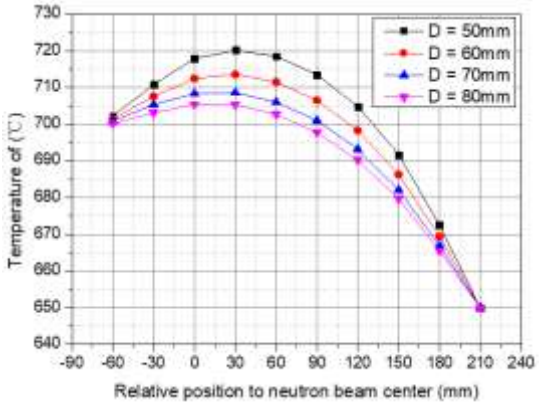
Haitao Hu, Chunchun Zhang, Mengjia Dou, Zhiqiang Huang, Yuan Sun, Fan Ye, Bao Yuan, Bo Bai, Hui Cheng, Shiyan Yang, Yufeng Duan, Xin Tong





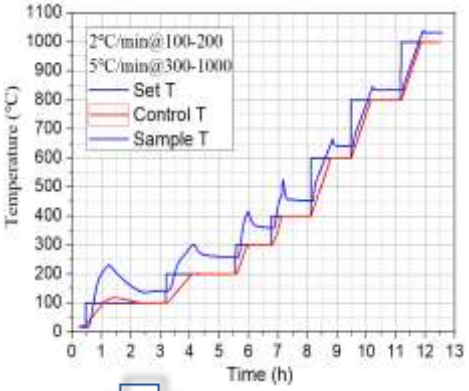
- ✓ Increasing the length or diameter of the heating element can broaden the uniform temperature zone of the sample.



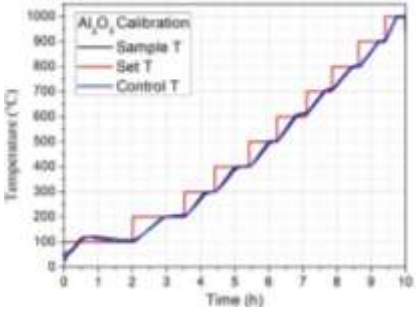


Temperature distribution of the heating element changes

- ✓ **Temperature Difference**
  - ☐ Initial state: ≈ 50℃, overshoot



  - ☐ optimization: ≤ 5℃, small overshoot



Optimization of the Foil Heating Furnaces

2) Optimization of the structure details

◆ For diffractometer

Neutron window: V, Ti Flange

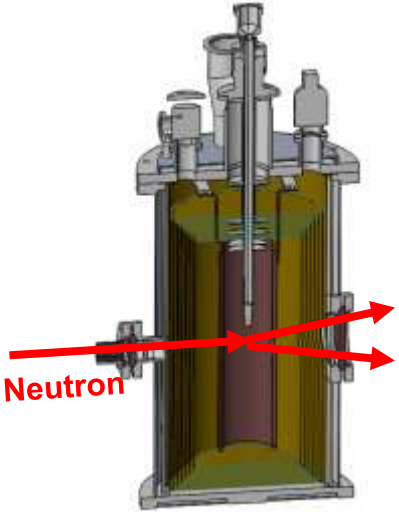
Water-cooled flange

Furnace body manufacturing

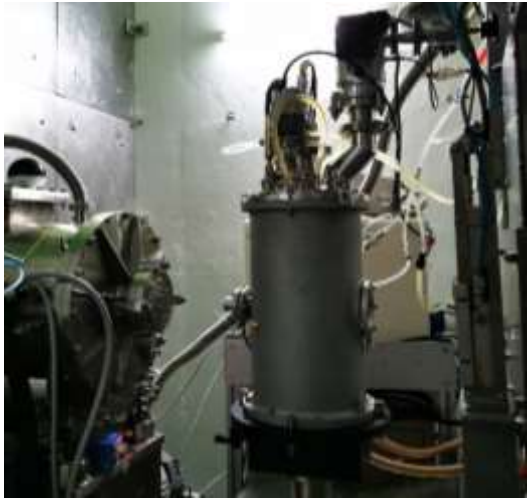
◆ For SANS/VSANS

Neutron window: Single crystal sapphire

Maximum scattering angle: 15°



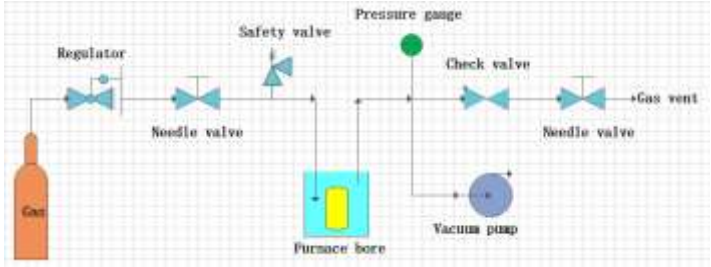
Tested in SANS



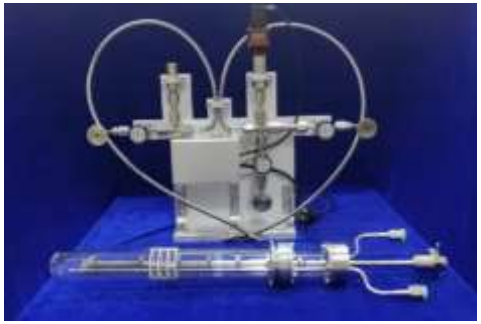
Position calibration：

Gas handling system (GHS) coupling with furnace

2) Optimization of the structure details

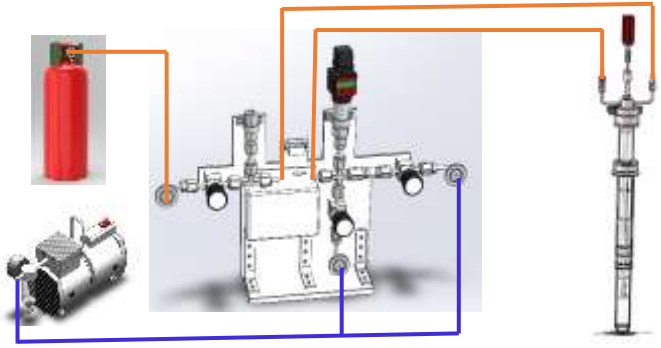


Flow chart of gas handling system

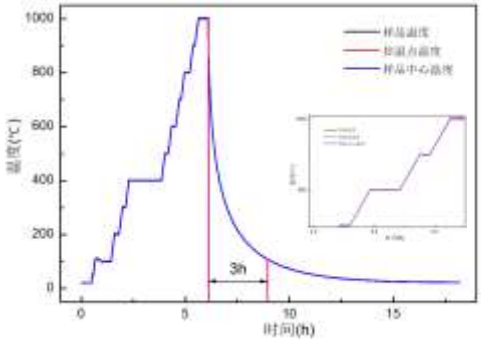


GHS for SANS

GHS @MPI,
ND 1000 ℃, PDF 800 ℃



Constitution of gas handling system

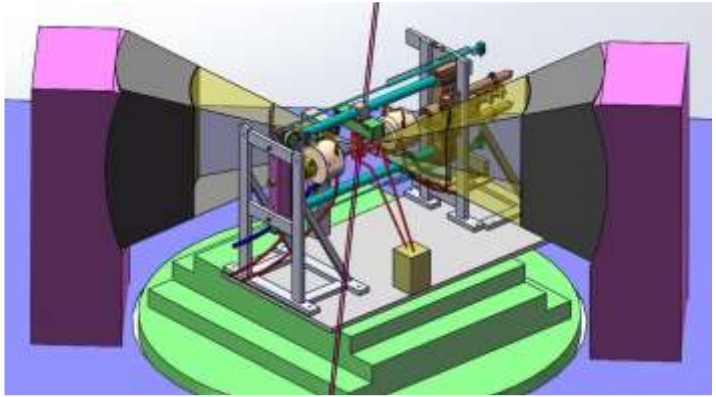


Temperature control test

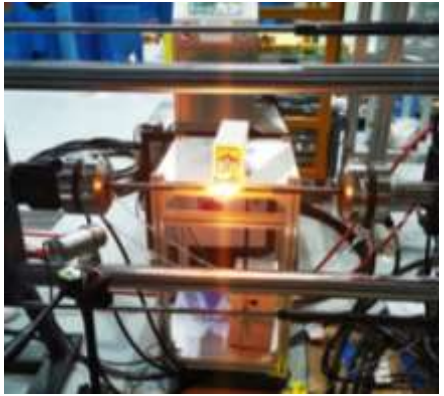
GHS @SANS, 1000 ℃

Inductive Heater for the stress load frame

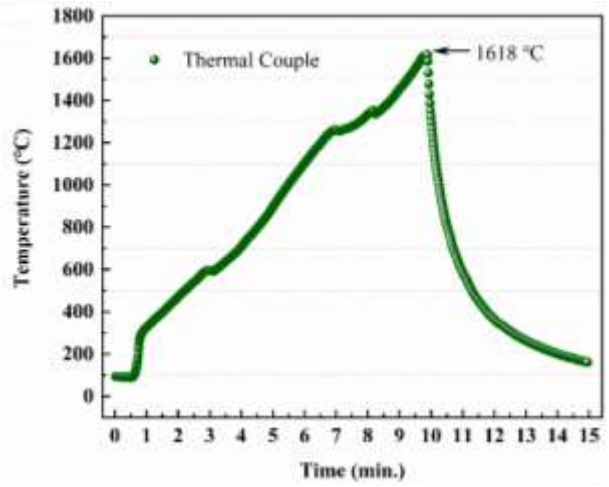
- inductive heater coupled with a stress load frame for EMD
- The maximum operating temperature of this heater prototype could reach 1600 ℃.
- The formal inductive heater is currently undergoing debugging.



Inductive Heating Stress Loading Furnace at EMD



prototype test

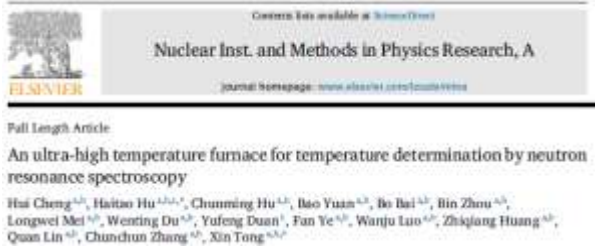


Maximum operating temperature exceeds 1600℃

Ultra High Temperature Furnace by Inductive Heating

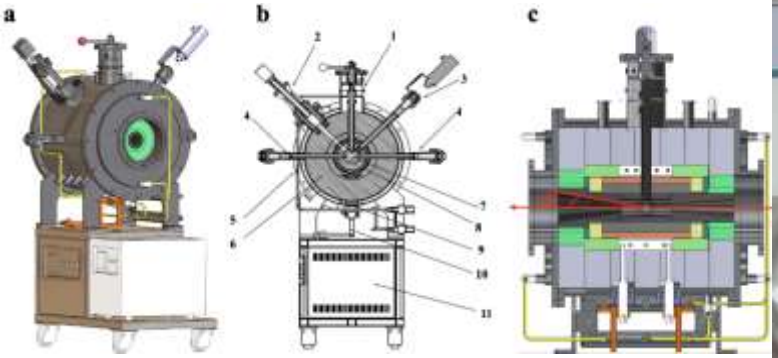
- This furnace is designed for internal temperature determination of materials by means of neutron resonance spectroscopy.
- It could be able to perform small angle neutron scattering and neutron imaging experiments in SANS, VSANS, and ERNI at CSNS.
- The maximum operating temperature of this device is over 2400 ℃.

Hui Cheng, et al. NIMA, 1049, 2023, 168072


Nuclear Inst. and Methods in Physics Research, A

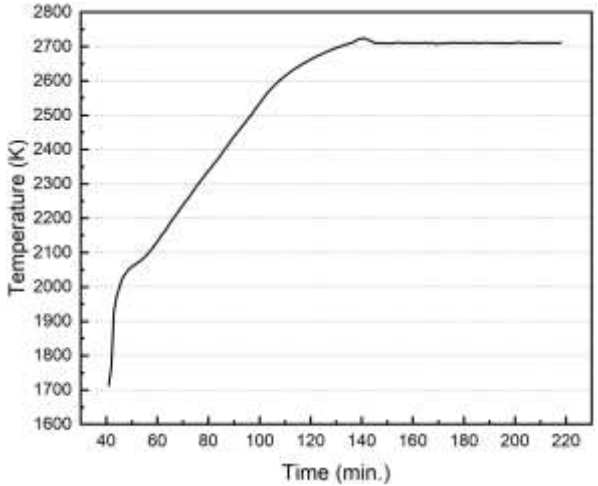
Full Length Article

An ultra-high temperature furnace for temperature determination by neutron resonance spectroscopy



The schematic representation of the inductive heating furnace

Overlook of the device



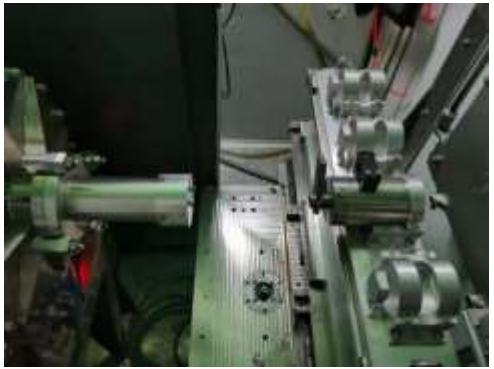
Maximum operating temperature exceeds 2400℃

High pressure cell for SANS

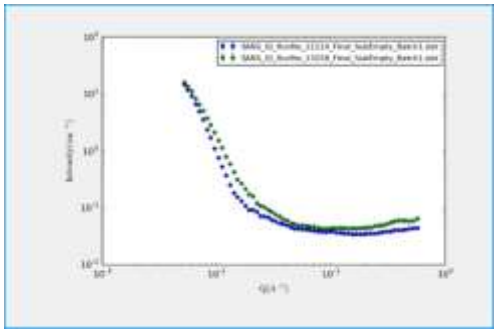
The high pressure cell has been used for shale rock research **Pressure limit： 100MPa**

SANS HP cell leakage test

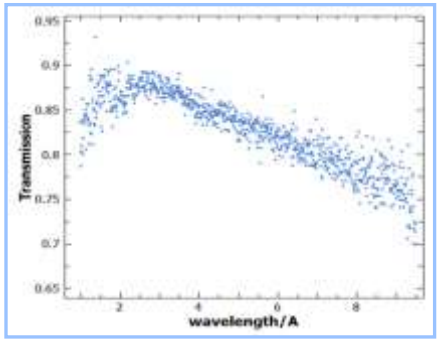
Sample preparation



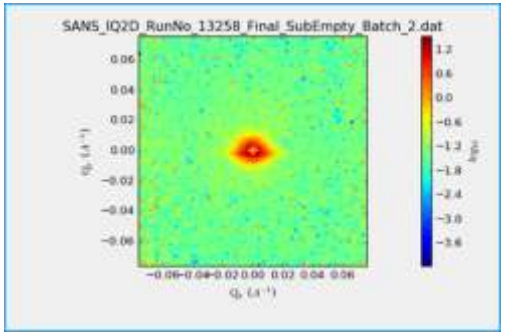
In-situ experiment @SANS



Background of empty cell VS air

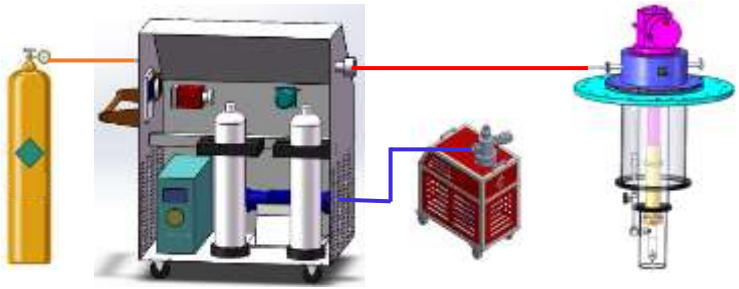


Neutron transmissivity ＞70%



Uniform scattering

Gas handling system (GHS) coupling with CCR



Gas handling system coupling with CCR



GHS + CCR06

Applications:

(1) Metal-Organic Frameworks;

(2) Methane hydrate;

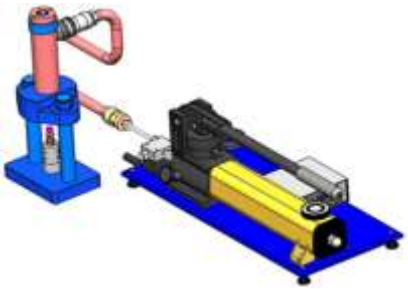
Gas types:

CD4 , C2D4, C3D6, C3D8

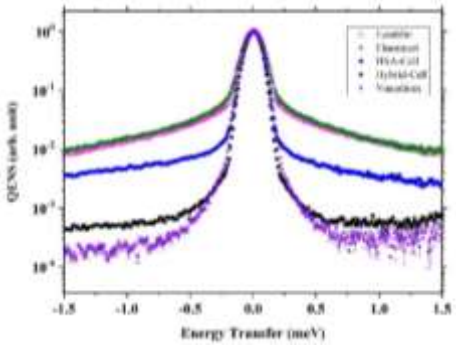


GHS + CCR05

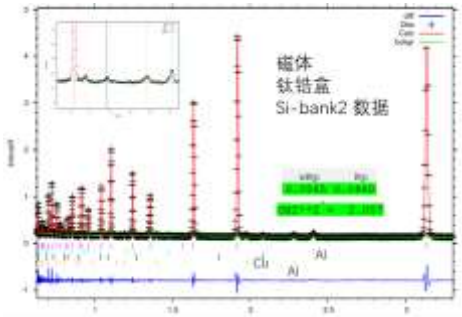
Clamp cell for diffraction and inelastic scattering



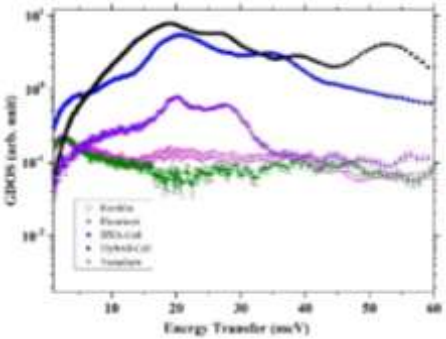
Clamp cell loading system



QENS @Pelican



The multi-field coupling extreme conditions have been preliminarily developed，HP+LT+M @ GPPD



GDOS @Pelican
EPJ Web of Conf., 2022

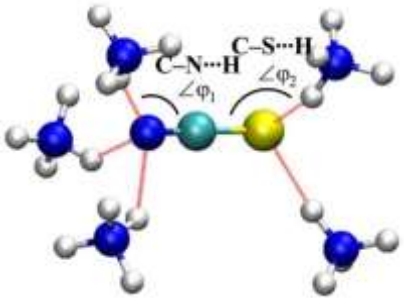
Application of clamp cell in inelastic scattering

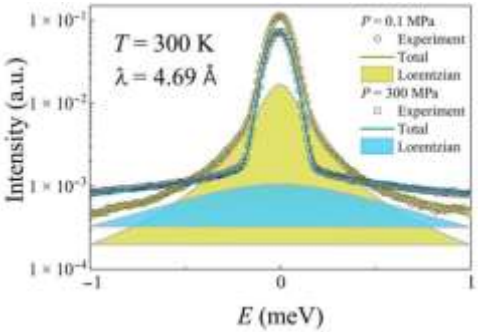
SCIENCE ADVANCES | RESEARCH ARTICLE

ELECTROCHEMISTRY

Thermal batteries based on inverse barocaloric effects

Zhe Zhang[1,2†], Kuo Li[3†], Shangchao Lin[4†*], Ruiqi Song[1], Dehong Yu[5], Yida Wang[3], Jingfan Wang[6], Shogo Kawaguchi[7], Zhao Zhang[1,2], Chenyang Yu[1,2], Xiaodong Li[8], Jie Chen[9], Lunhua He[9,10,11], Richard Mole[5], Bao Yuan[8,9], Qingyong Ren[8,9], Kun Qian[4], Zhuangli Cai[4], Jingui Yu[12], Mingchao Wang[13], Changying Zhao[4], Xin Tong[8,9], Zhidong Zhang[1,2*], Bing Li[1,2*]





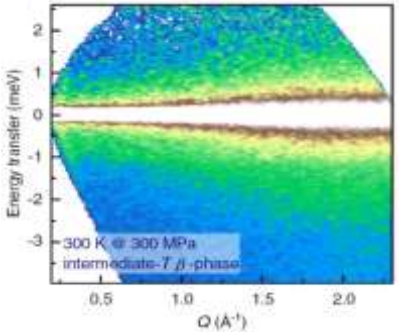
nature COMMUNICATIONS

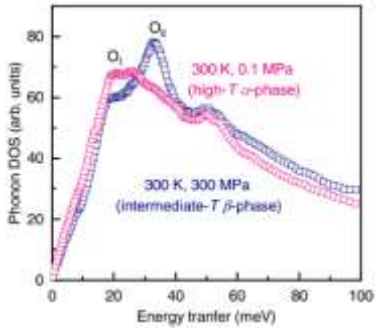
ARTICLE

https://doi.org/10.1038/s41467-022-29997-9 OPEN

Ultrasensitive barocaloric material for room-temperature solid-state refrigeration

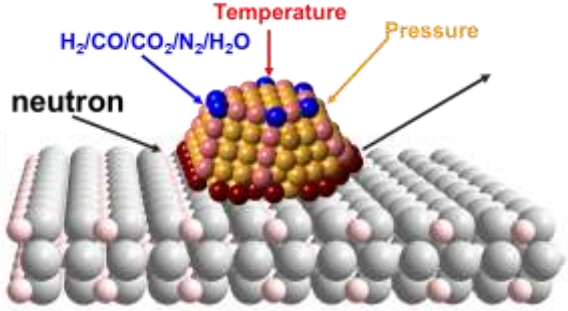
Qingyong Ren[1,2,6], Ji Qi[3,4,6], Dehong Yu[5], Zhe Zhang[3,4], Ruiqi Song[3], Wenli Song[1,2], Bao Yuan[1,2], Tianhao Wang[1,2], Weijun Ren[3], Zhidong Zhang[3,4], Xin Tong[1,2✉] & Bing Li[3,4✉]





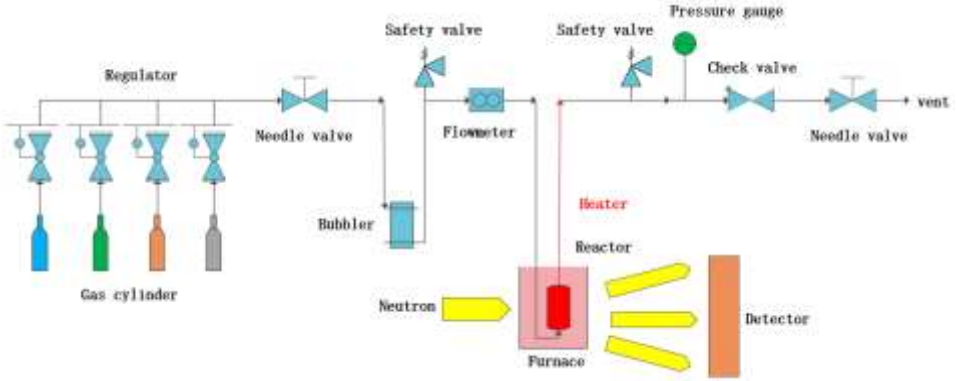
Development of the in situ catalytic SE

✓ In situ reaction - study the structure and evolution of catalysts

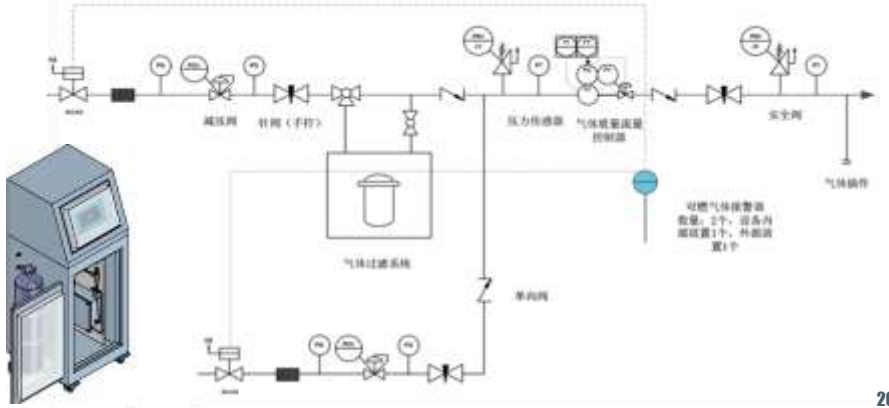


Requirements

- high temperature
- high pressure
- reaction atmospheres



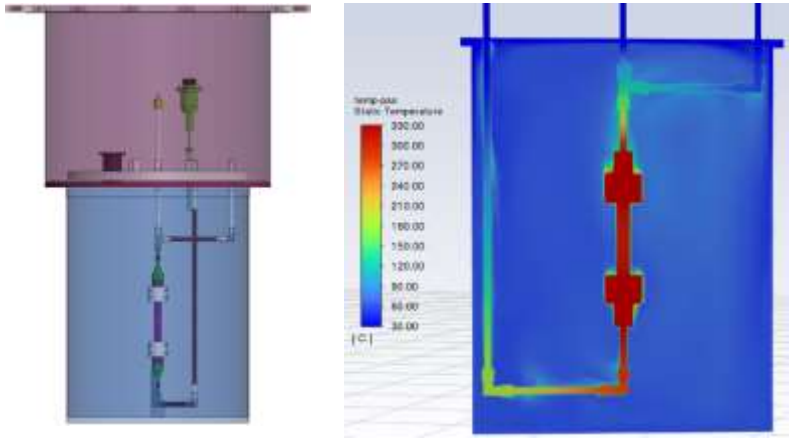
➢ **Gas-liquid supply systems**



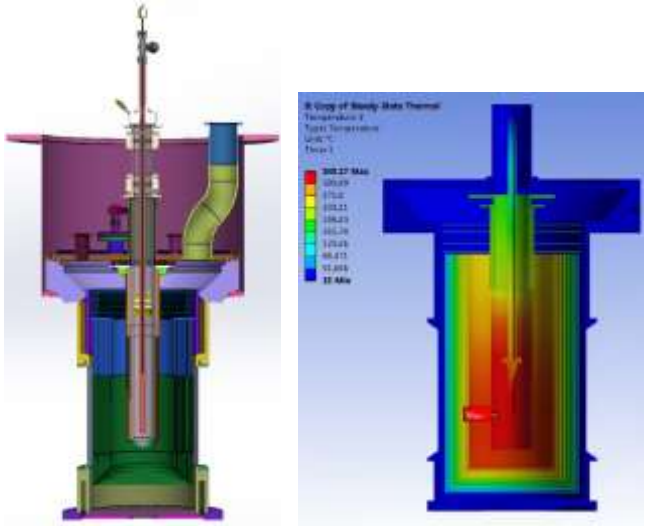
✓ Remote control and data transmission functions for hazardous gas functions

Development of the in situ catalytic SE

➢ In-situ catalytic reactor



High pressure reactor



High temperature reactor

- **High pressure, 0.1~5MPa**
- **Temperature, RT~300°C**

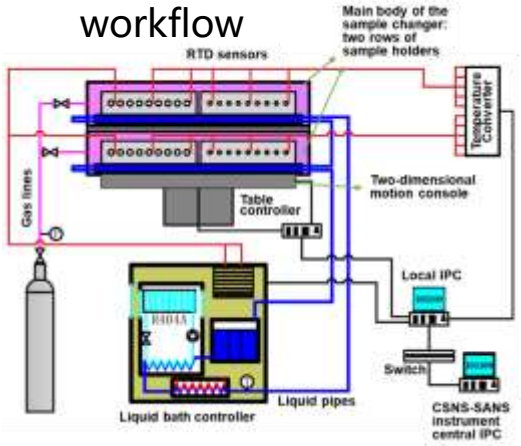
- **High temperature, RT~800°C,**
- **Pressure, 0.1~0.2MPa.**

Outlines

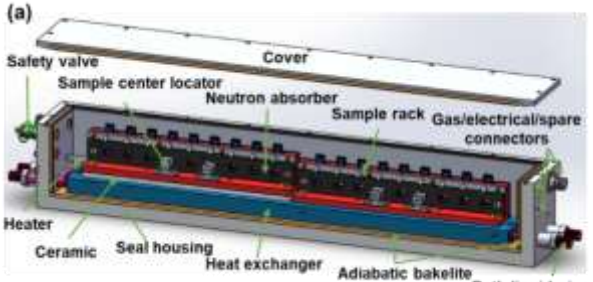
- User Service
- Low Temperature & Magnetic field
- High Temperature & High Pressure
- Soft Matter & Control System
- Goals for the next few years

Sample changer for SANS/VSANS

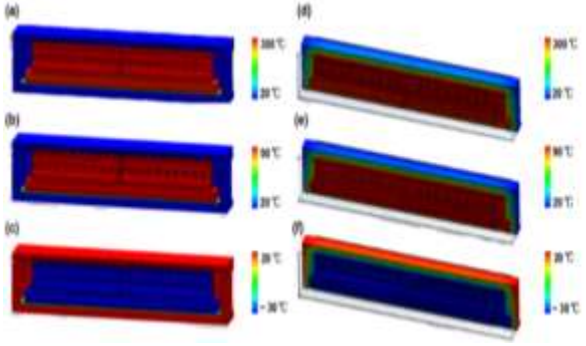
workflow



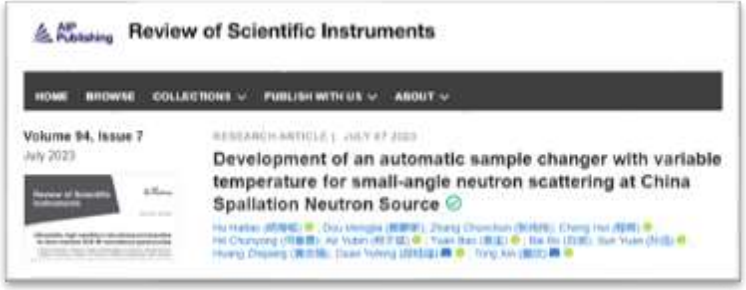
Design



Simulation Analysis



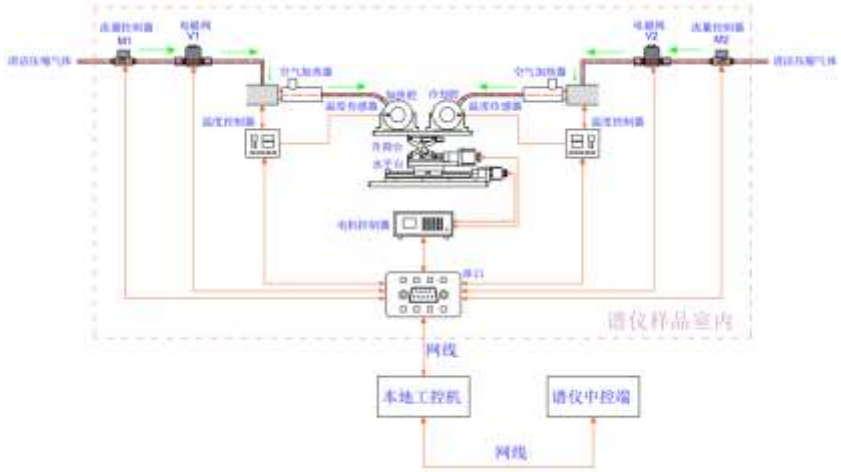
Temperature range: -30~300℃
Sample numbers: 36



@SANS

@VSANS

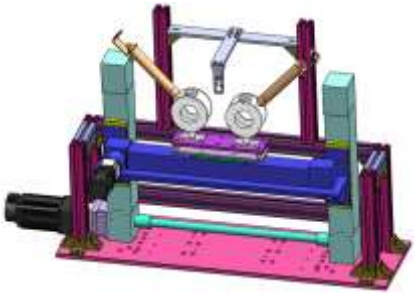
T-jump cell for SANS/VSANS



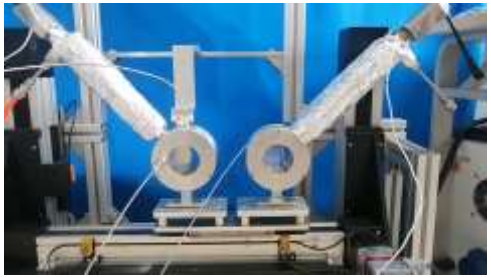
Schematic of T-jump cell system

Picture of the system

T-jump cell at VSANS



Model of T-jump cell



Video of T-jump cell

Main performance parameters

Parameters	Actual test value
Jump temperature difference	＞270 ℃
Motion switching time	＜5 s
Coupling control	Temperature-flow-motion

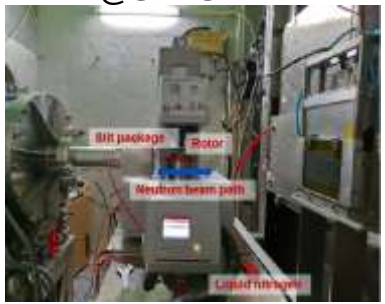
Rheometer for SANS/VSANS

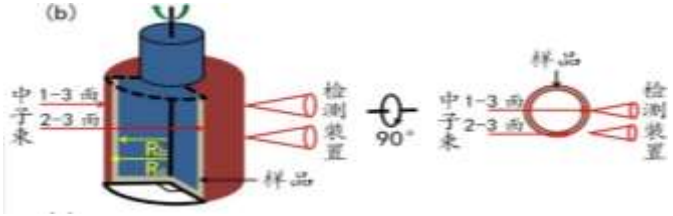
Main performance parameters

Test items	Value
Torque regime	50nNm ~230mNm
Temperature control regime	-40 ~200℃
Angular frequency regime	10^{-5} rad/s~628 rad/s
Rotation speed regime	10^{-7} min^{-1}~3000 min^{-1}

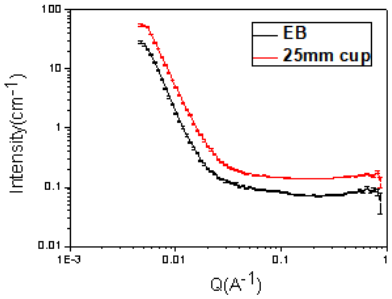
@VSANS

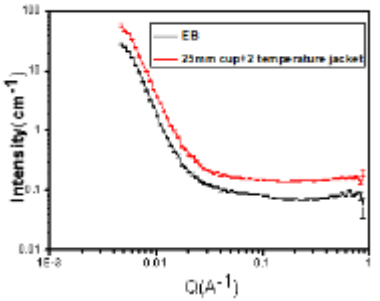
@SANS





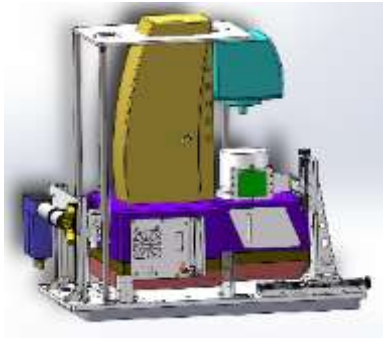
Primary: samples under stress in the 1-3 / 2-3 planes



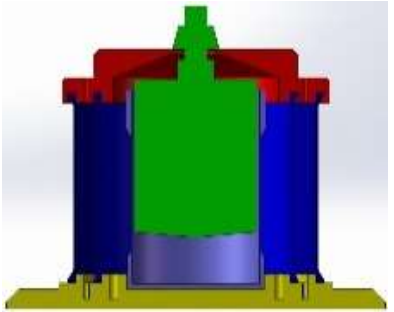


Scattering intensity from Ti cup and bob @ SANS

Next optimization



Neutron slit



Better cup

……

Framework design for the SE control system

The control interface standard between SE equipment and neutron instruments

SE Expert Interface

Neutron instruments

EPICS

SE Control System

Motor

LabVIEW

Modbus

Serial

Lake Shore

PLC

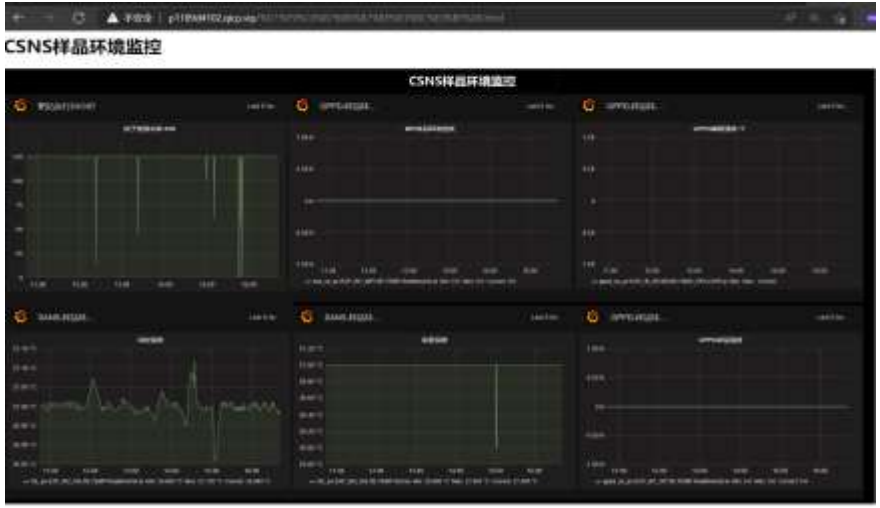
SE Device

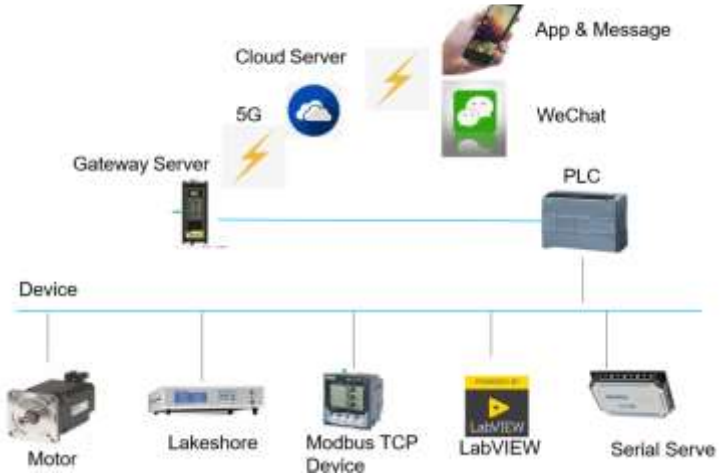
SE Remote Alarm System

- ✓ **Can monitor the SE on-line equipment parameters by the web page and mobile App;**
- ✓ **In case of an alarm, it can push notifications by short message or real-time communication software WeChat;**

Alarm Notice

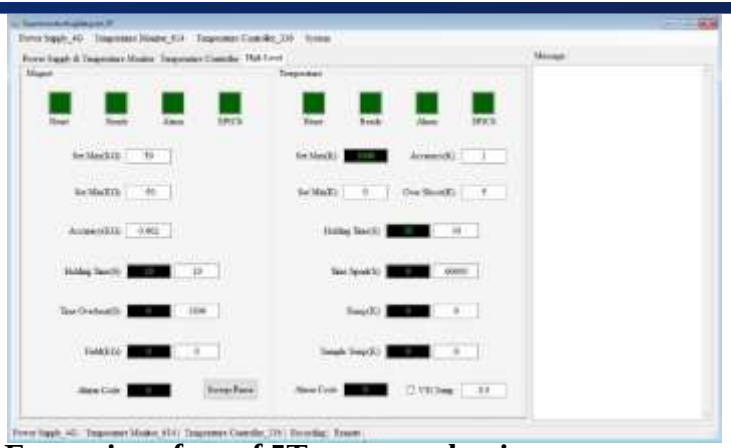
Monitoring through web page



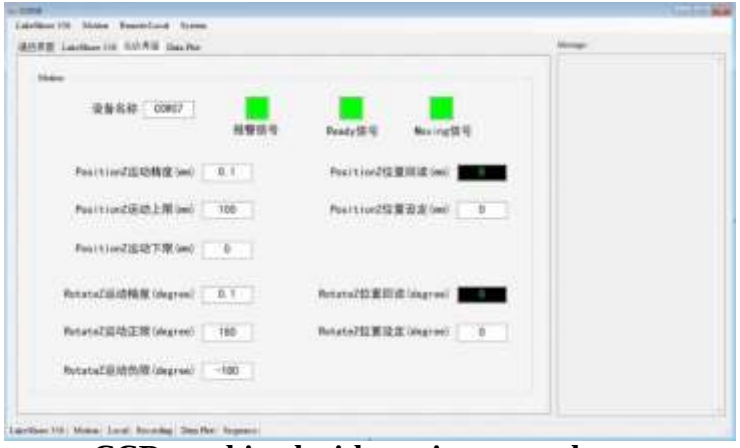


Control Program development

For Furnace （ with logical judgment function ）



Expert interface of 5T superconducting magnet



CCR combined with motion control

For Water bath equipment

Outlines

- User Service
- Low Temperature & Magnetic field
- High Temperature & High Pressure
- Soft Matter & Control System
- Goals for the next few years

SE Plan for CSNS-II and other needs

- Based on the current operation of CSNS and the demand for the newly built instruments in CSNS-II, the SE scheme has been determined (table 1).
- Based on some new scientific research and major scientific issues, other SE planning is required. Such as in-situ catalytic reactor, 14T magnet and 25mK DR.

Table 1. SE Equipment scheme for CSNS-II

Type	Specific type	Index	Quantity
Low Temp.	CCR、ULT-inserts (He3-insert、DR-insert)	0.04~800K	20
High Temp.	Traditional furnace、Laser furnace	RT~2000℃	4
High Pressure	GP, CPC, PE-cell, Gas adsorption analyzer	0~100 ton	6
Magnet	Electromagnets, Superconducting magnets	0~10T	3
Soft Matter & Special SE	Rheometer, Humidity controller, Gas adsorption tank, Auto changer, Gas insert, Electric field controller, etc	230mNm, 50kV/m, etc	23
Ancillary	Pumps, Chillers, Gas controller, IPC, PLC, etc	10E-5Pa, ±1kPa, etc	45

Low Temp.

CCRs ULT-inserts

High Temp.

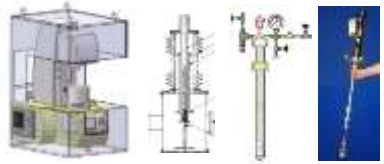
Furnace Laser Furnace

High Pressure

PE-cell CPC GP

Soft Matter & Special SE

Rheometer EFC Gas inserts



Humidity controller

Auto changer for Cryostat

Gas adsorption tank

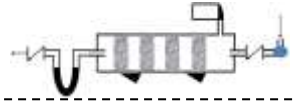
Auto changer insert

Other SE Planning

In-situ catalytic reactor

Gas/liquid mixer Reactor

Exhaust handler



14T Magnet 25mK DR

Magnet

Electromagnet 10T-magnet

Summary

- More than 25 sets of SE equipment operated online stably
- Provide experimental conditions such as temperature, magnetic field, pressure, softmatter and coupling fields
- The collaborative research and development with users on key technologies has progressed continuously.
- Further goal: expand the range of extreme conditions, improving equipment stability, reducing the background of equipment, and more intelligent

Type	Specific type	Current	Future
Low Temp.	CCRs, ULT-inserts (He3-insert, DR-insert) , etc	0.3 ~ 800K	**0.025 ~ 800K**
High Temp.	Traditional furnace, Induction furnace, etc	1600℃	**≥ 2000℃**
High Pressure	GP, CPC, PE-cell, Gas adsorption analyzer, etc	10GPa	**10GPa@50K**
Magnet	Electromagnets, Superconducting magnets, etc	9T	**14T**
Soft Matter & Special SE	Rheometer, Auto changer, Gas insert, Electric field controller, etc	50nNm ~ 230mNm, etc	**50nNm ~ 230mNm, 50kV/m, etc**

Thanks

Acknowledgement:

CSNS-SE Group, the Neutron Instrument Teams, and the International Society for Sample Environment