

# Technical Meeting on Structural Materials for Heavy Liquid Metal Cooled Fast Reactors

IAEA Headquarters, Vienna, Austria

15–17 October 2019

## Statue of HLM technology and related materials research in China Institute of Atomic Energy

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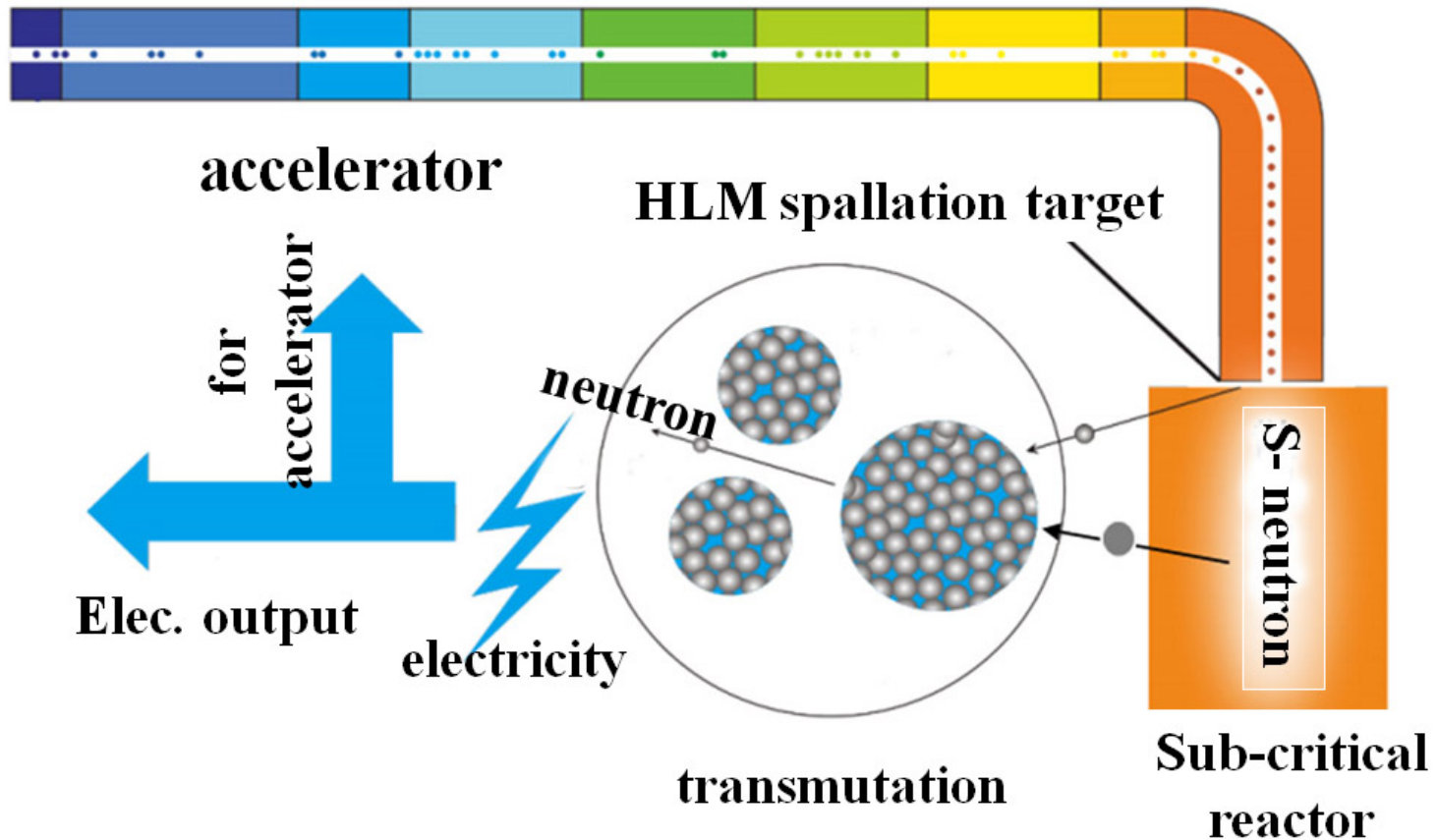
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☑ CiADS (China initiative ADS)

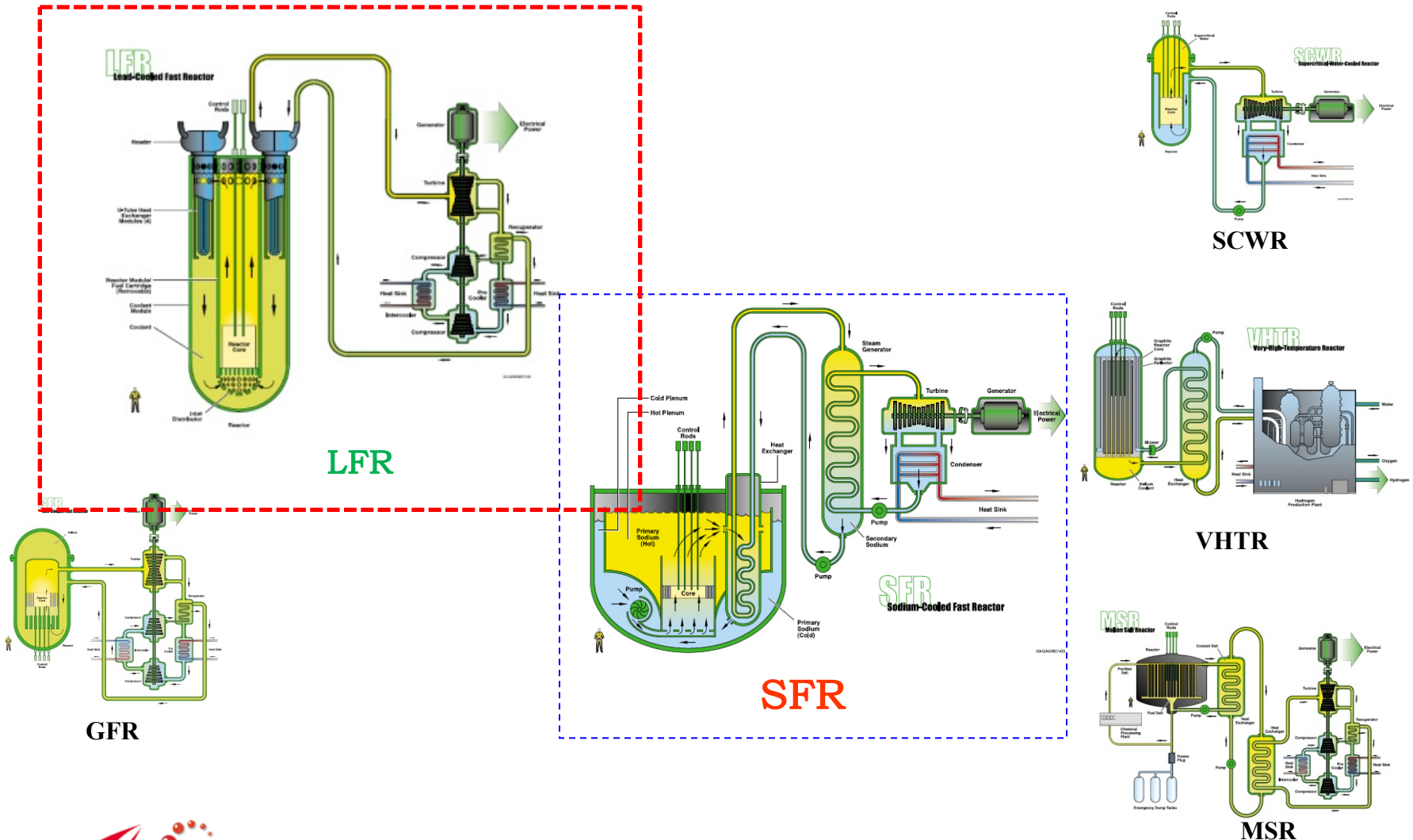


**High-energy proton** beam provided by high-current proton accelerator, bombarding **the heavy metal spallation target** to produce high **flux spallation neutron** to drive the **sub-critical reactor** to transmutation the long-lived (over 10,000 years) high-level radioactive waste into short-lived (less than 100 years) nuclear waste

# LBE-cooled Fast Reactor



## Innovative Nuclear Energy System-GIV Generation Reactor





# LBE related facilities in CIAE



National projects + Pilot project of CAS

Preparation technology of LBE



Compatibility of materials with LBE



Thermal-hydraulic characteristics of LLBE



OCS and oxygen sensor



fundamental research in physics



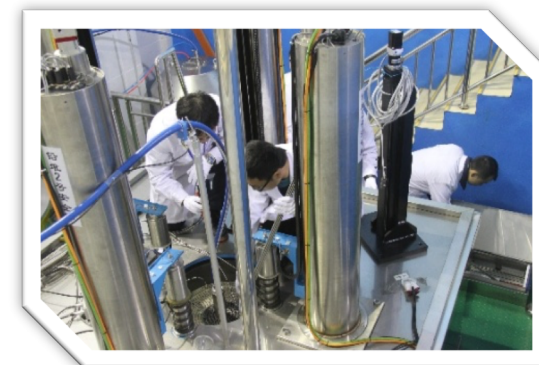
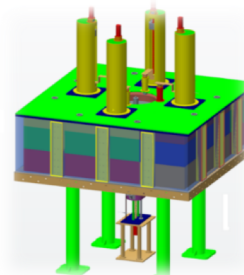
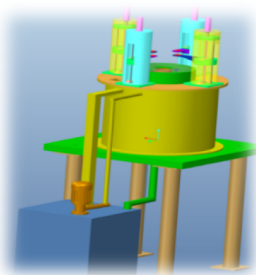
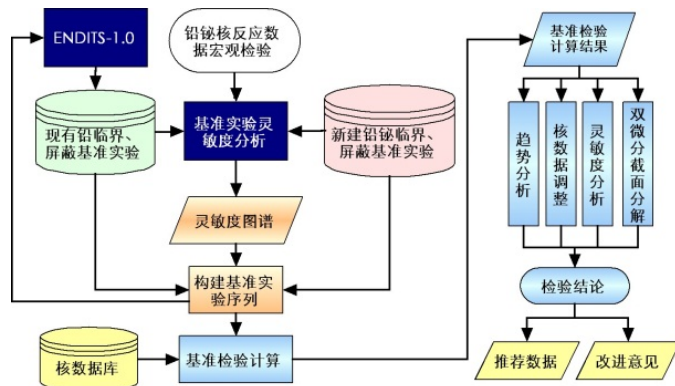
VENUS I



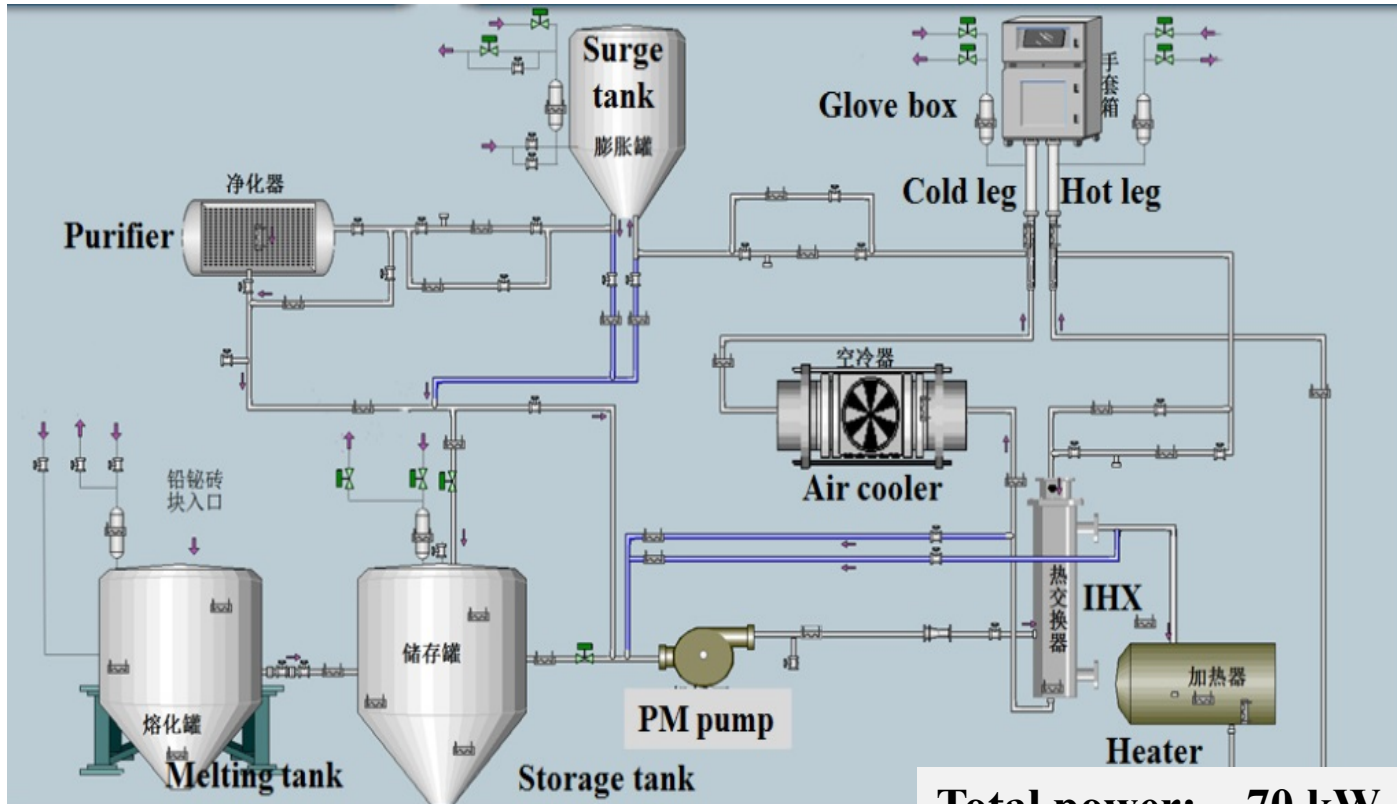
VENUS II



VENUS III



# Overview and parameters of LBE dynamic loop



Total power: ~ 70 kW

Total capacity: ~0.150m<sup>3</sup>

T of high temp. test section: 400°C~550°C

T of low temp. test section: 300°C~450°C

Maximum T difference  $\Delta T$ : 250 °C

LBE flow rate: 0.576 m<sup>3</sup>/h

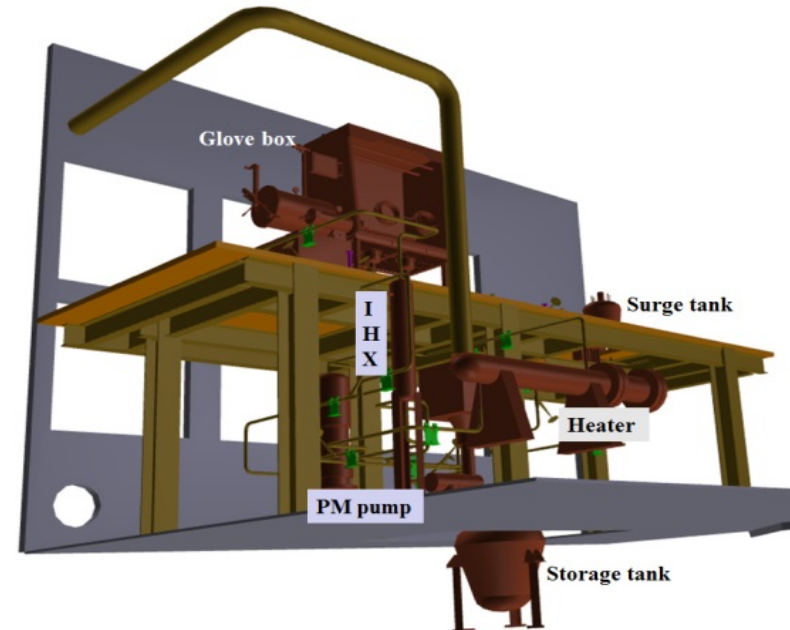
LBE speed on the sample surface: 1~5m/s

Pressure of system: 0.03~0.06MPa

LBE inventory: 1500Kg



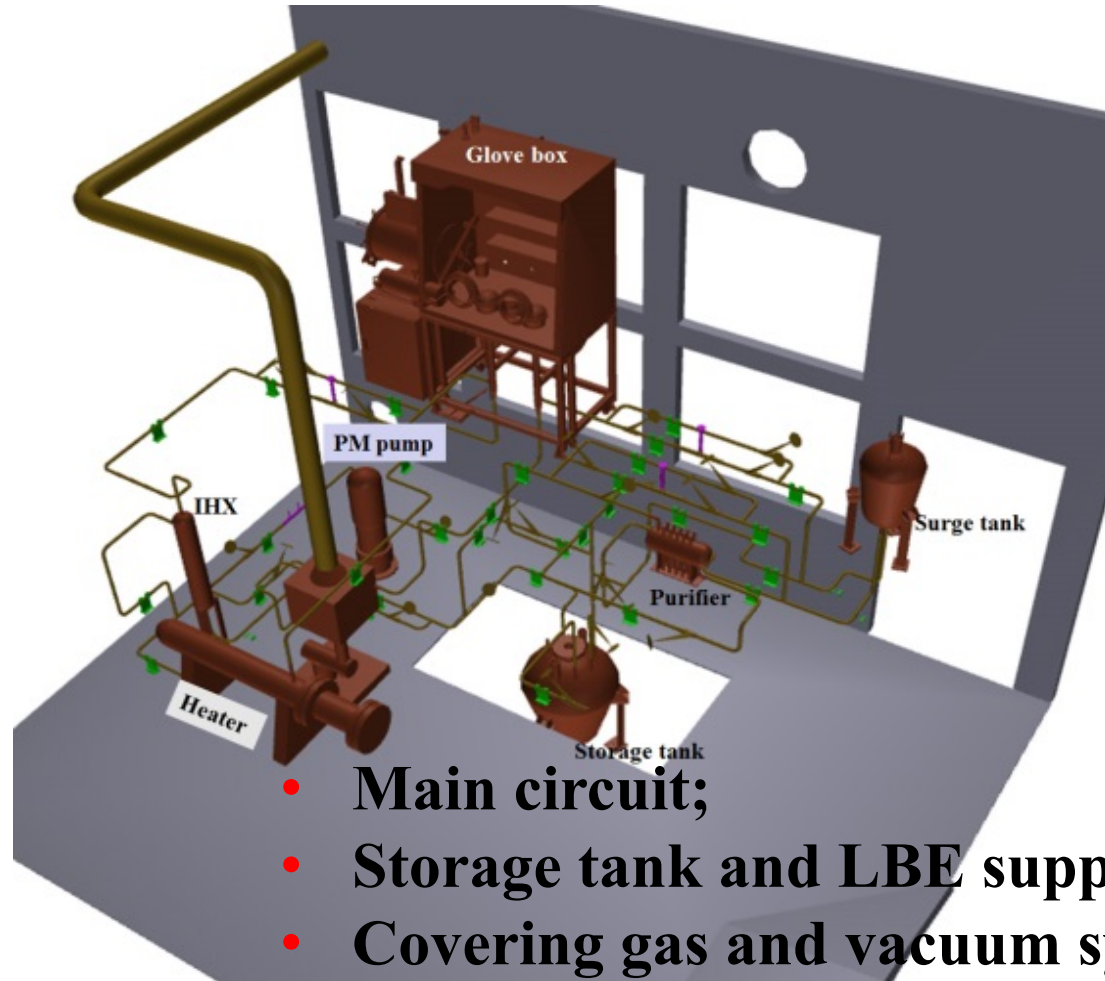
# Overview and parameters of LBE dynamic loop



# Main Equipment and Auxiliary System of Loop



The LBE dynamic corrosion test loop consists of as following:

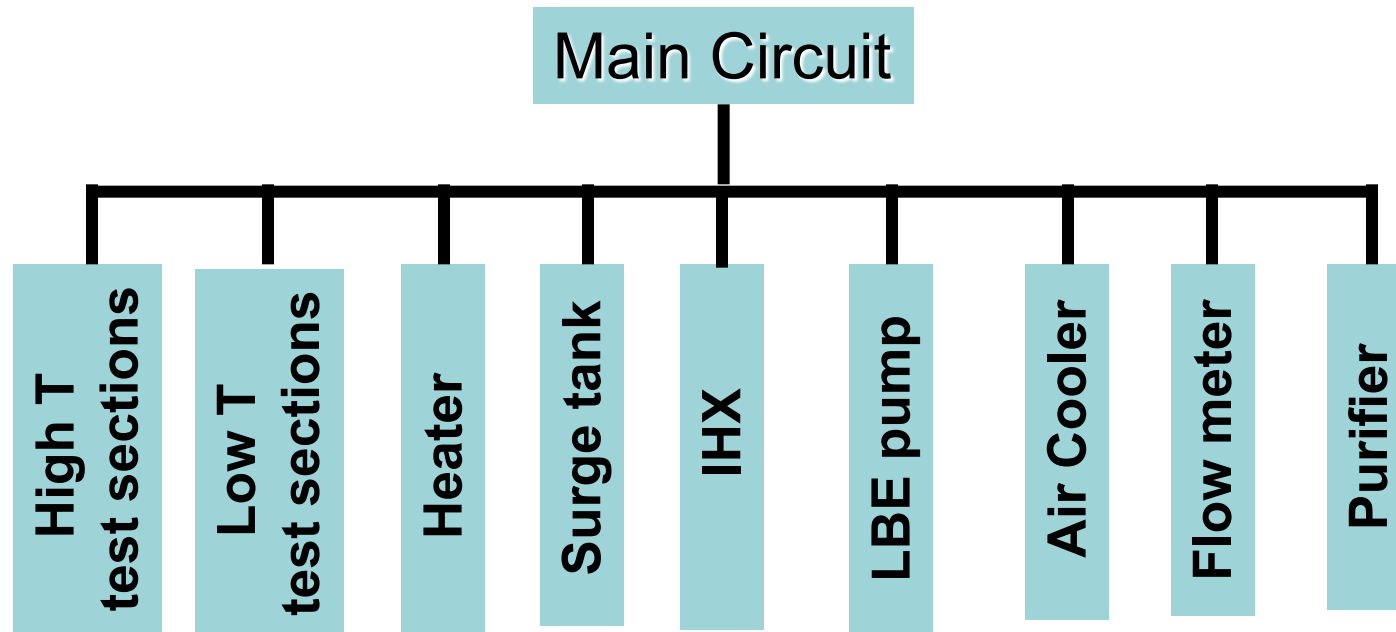


- Main circuit;
- Storage tank and LBE supply system;
- Covering gas and vacuum system;
- Sampling system;
- Instrumentation control system



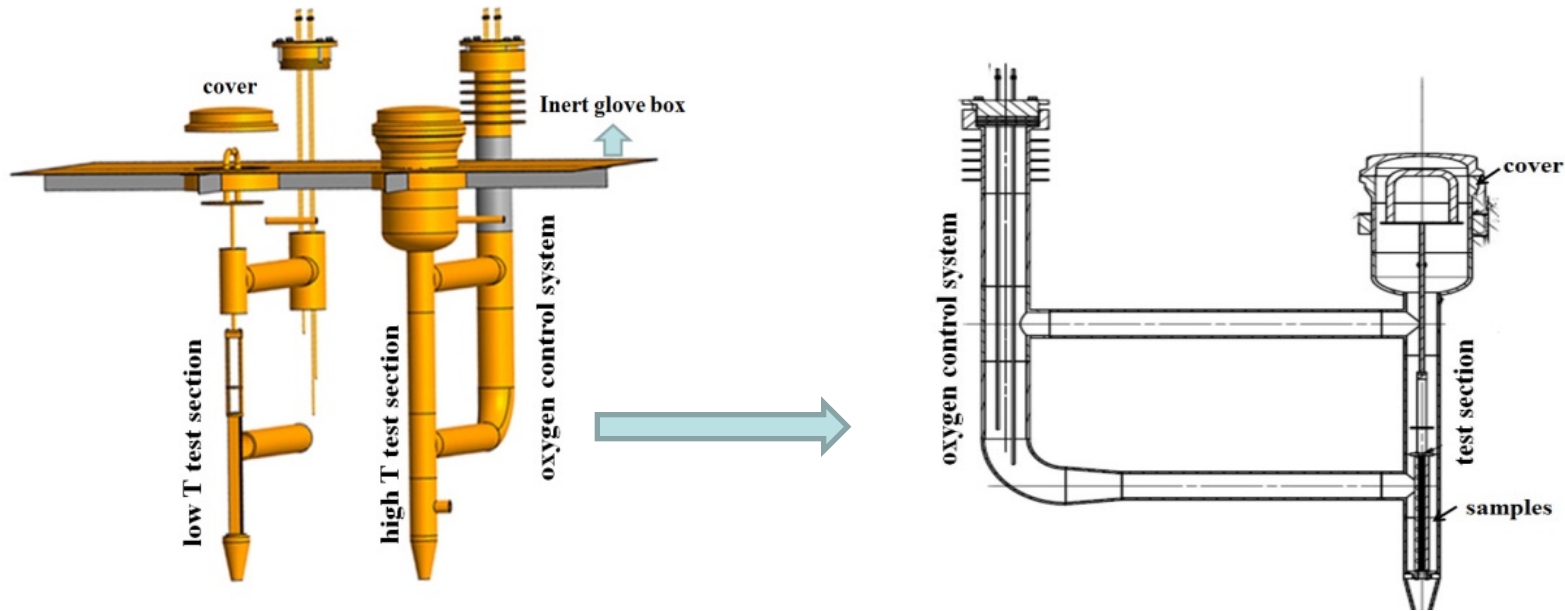
## ■ Main circuit system

### Composition of main circuit system





## □ High and low temperature test sections



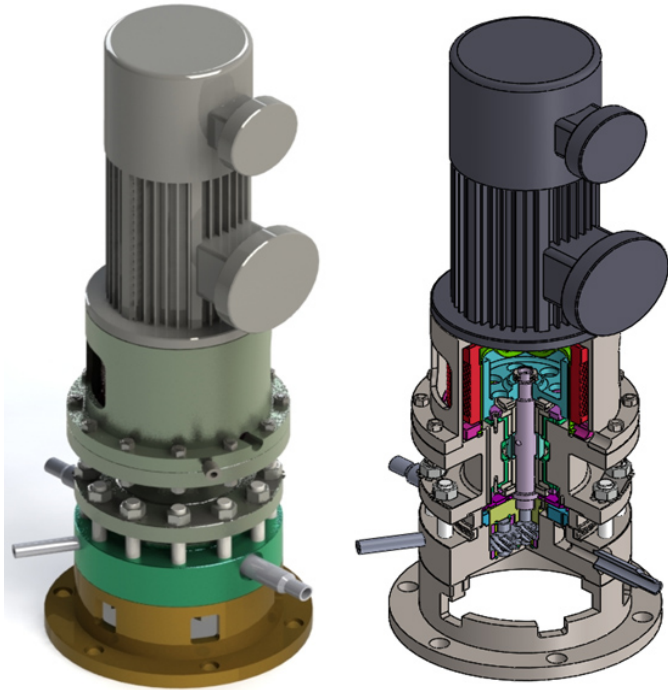
- It is made of 316 stainless steel pipes of  $\phi 50\text{mm} \times 3\text{mm}$ . The length of the test sample box is about 0.6m.



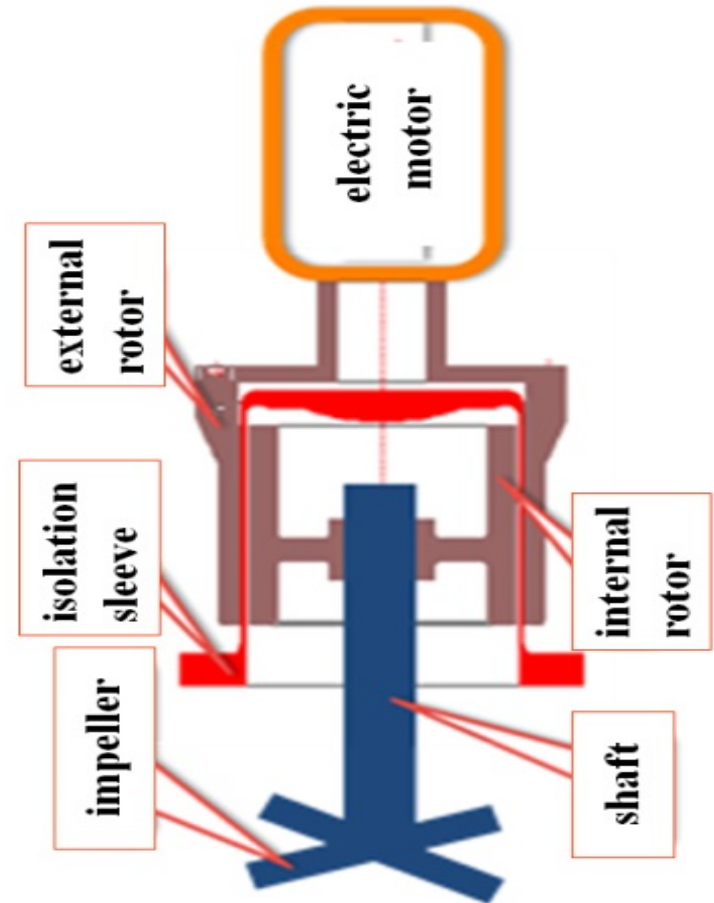
# Main Equipment and Auxiliary System of Loop



## □ LBE pump



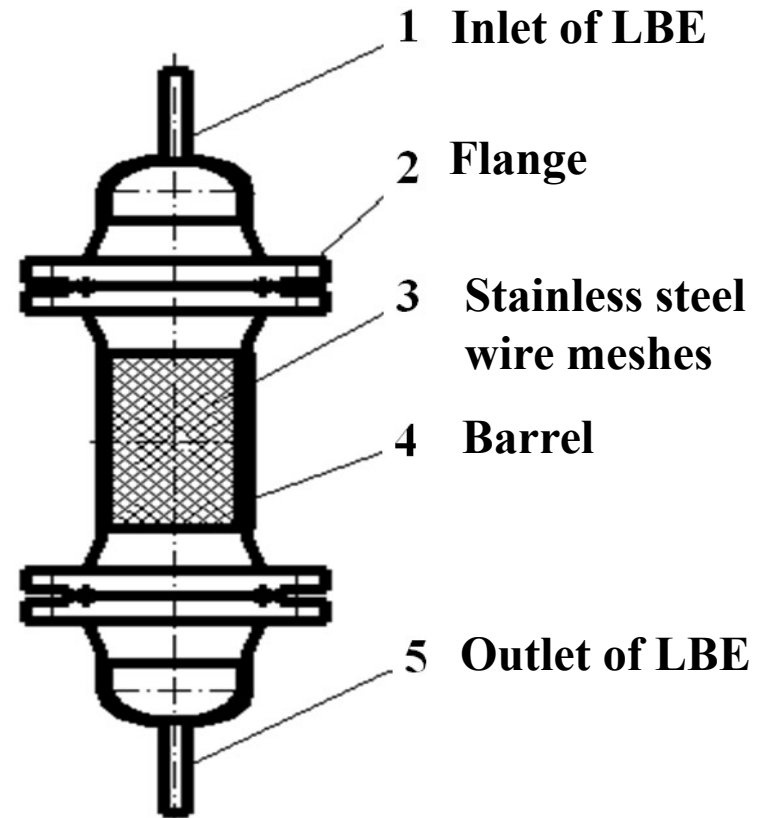
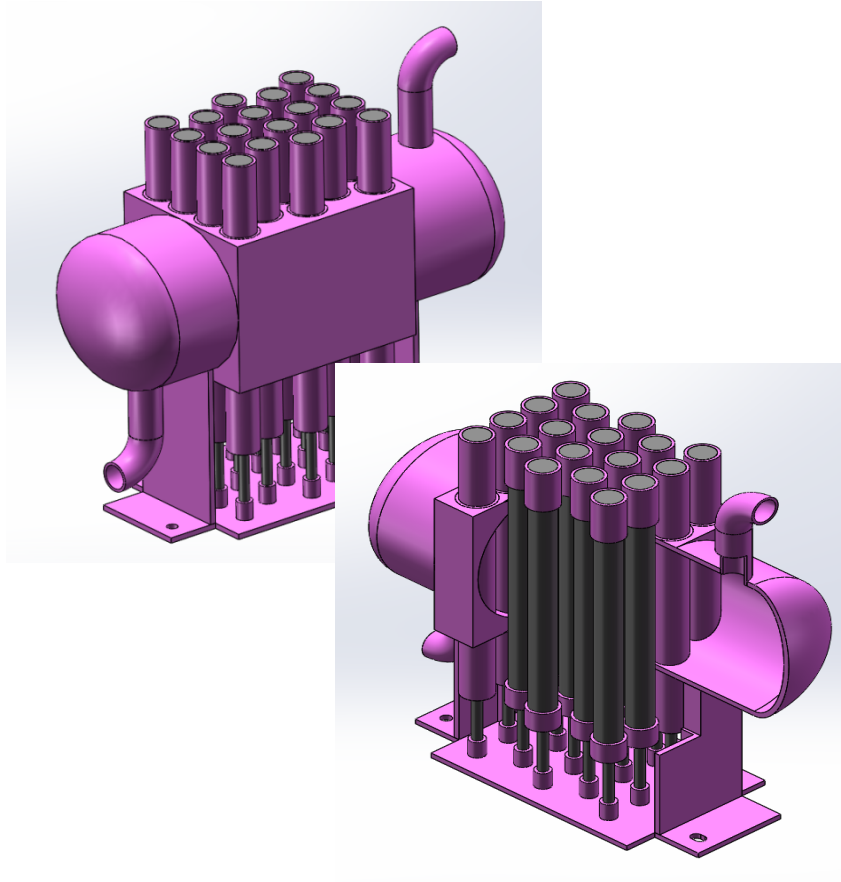
permanent magnet pump



- Inlet pressure: 0.4 MPa
- Outlet pressure: 1.2 MPa
- Flow rate: 0.1~ 0.6m<sup>3</sup>/h
- Pressure head: 1.2MPa
- Working temperature: 480°C



## □ Purifier (magnet trap)

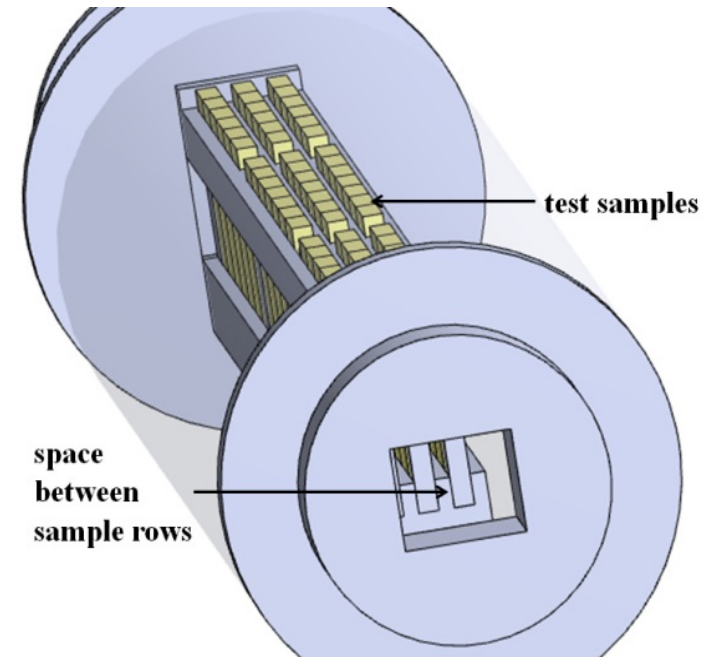
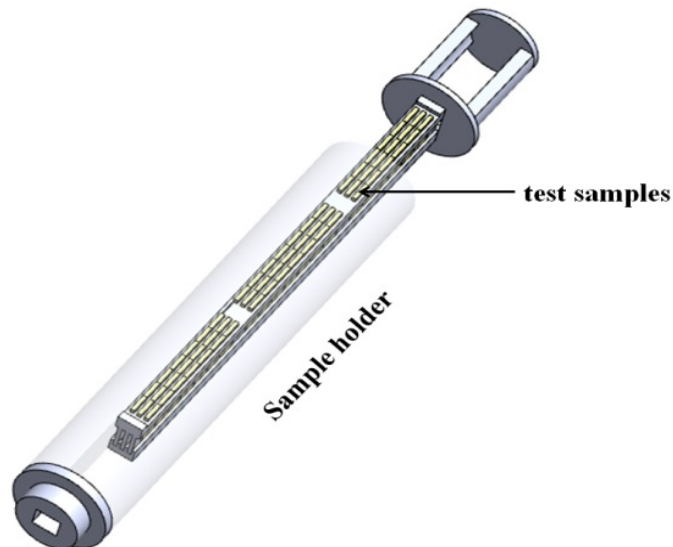


- The inner part of the purifier is filled with **stainless steel wire**, which occupied 60% of the volume,
- The purifier barrel is inserted in **AlFeCo** permanent magnet steel.



## ■ Auxiliary system

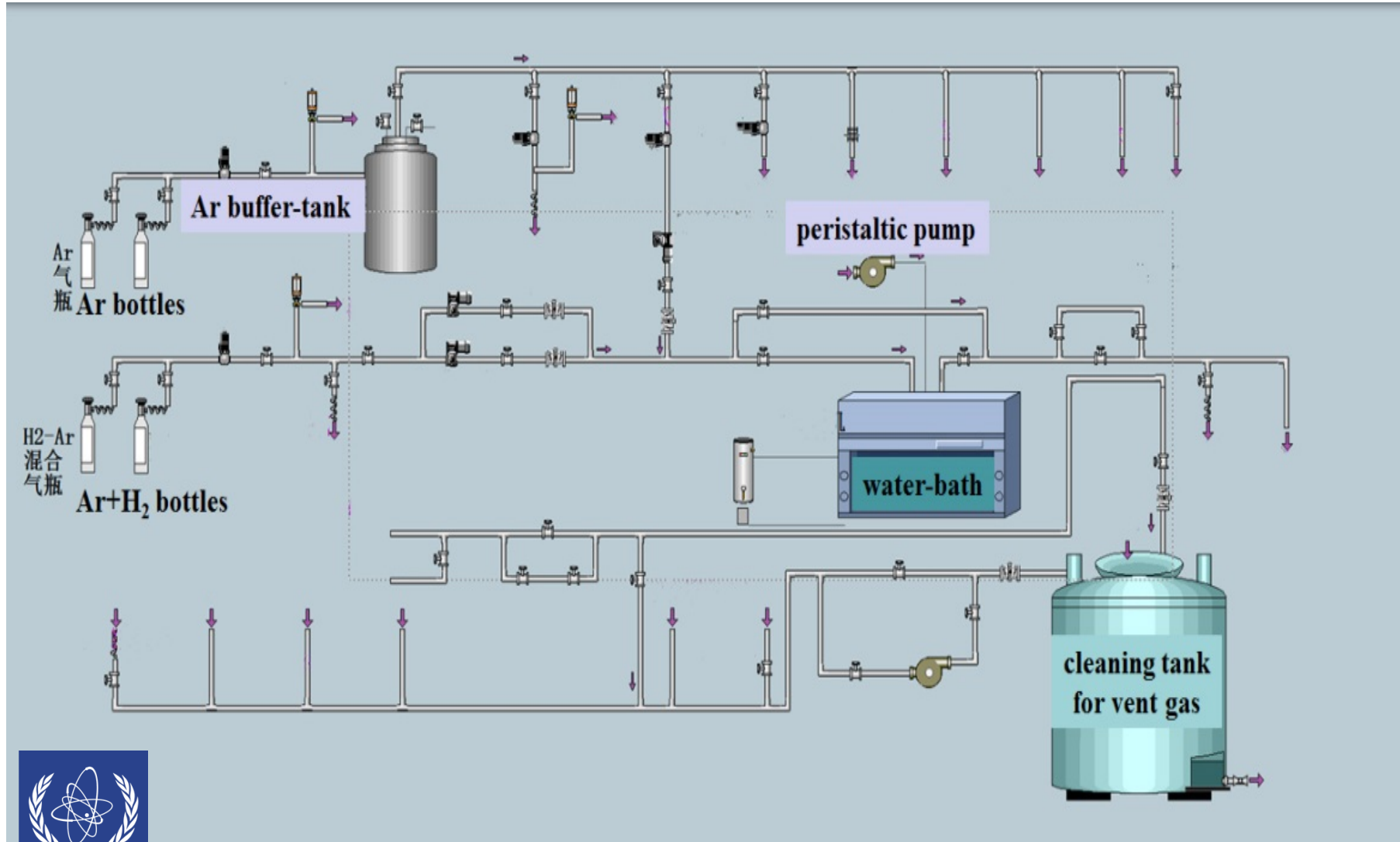
- Storage tank and LBE supply system
- Covering gas and vacuum system
- Sampling system
- Instrumentation control system



# Main Equipment and Auxiliary System of Loop



## Covering gas and vacuum system

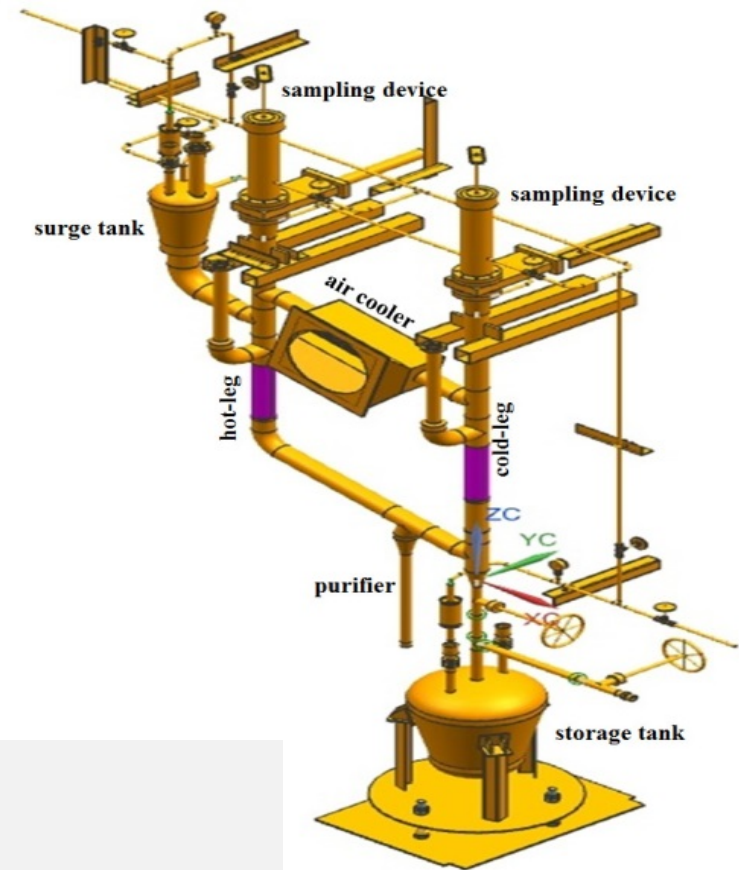
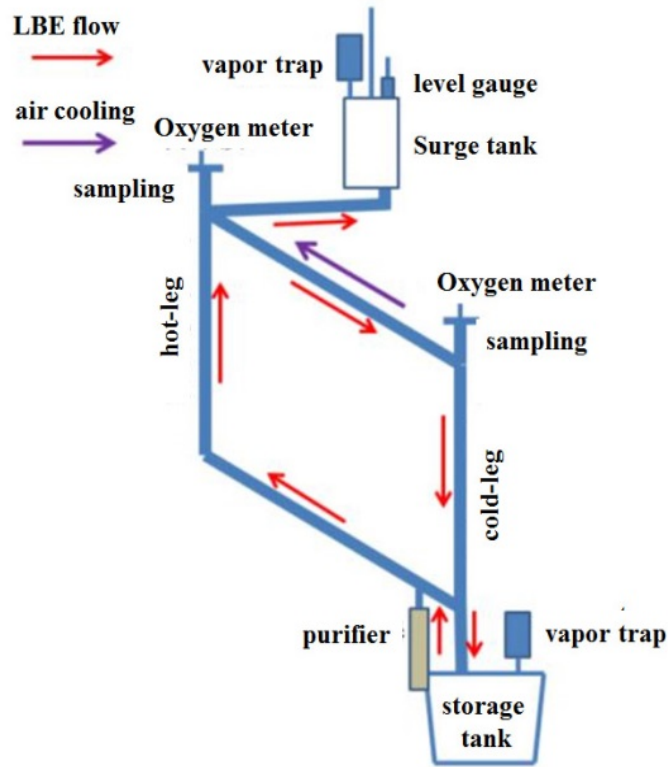


**□ At present, the loop has been installed and debugged, and operated safely and steadily for 168 hours under 550°C. The research work to be carried out on this circuit is as follows:**

- Corrosion behavior of austenitic stainless steel (including 316SS, 316LSS, 304SS and 15Cr-15NiTi SS) in LBE at high temperature and high velocity;**
- Corrosion properties of ferrite/martensitic steels (including T91, HT9, Si-added FM steel and Al-added FM steel) in LBE at high temperature and high velocity;**
- Corrosion behavior of coating materials in LBE at high temperature and high velocity**



# LBE THERMAL CONVECTION LOOP



## Main Parameters

- Temperature of hotleg: 400°C~550°C
- Temperature of coldleg: 300°C~400°C
- Maximum T difference  $\Delta T$ : 250°C
- LBE velocity on the sample surface: 0.03m/s~0.05m/s
- Pressure of system: 0.03~0.06MPa
- LBE inventory: 150 Kg
- Total power: 12 kW





# LBE THERMONVECTION LOOP

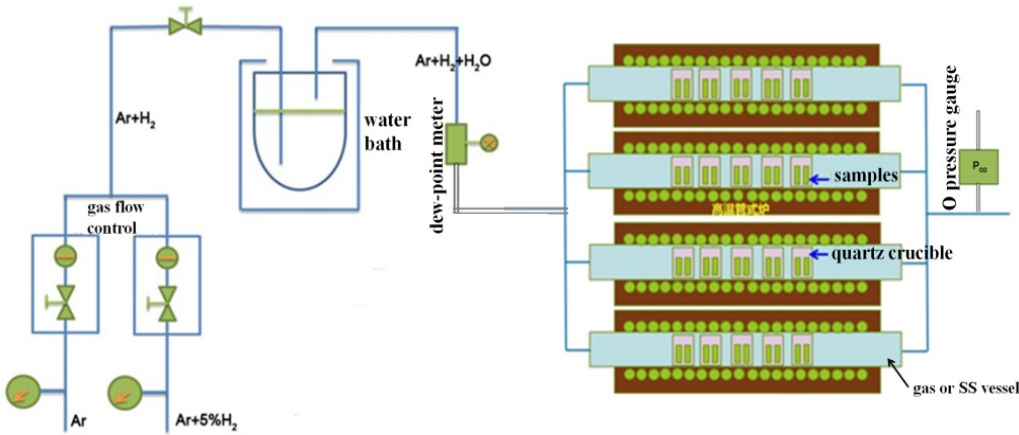


## □ Current studies performing in the loop

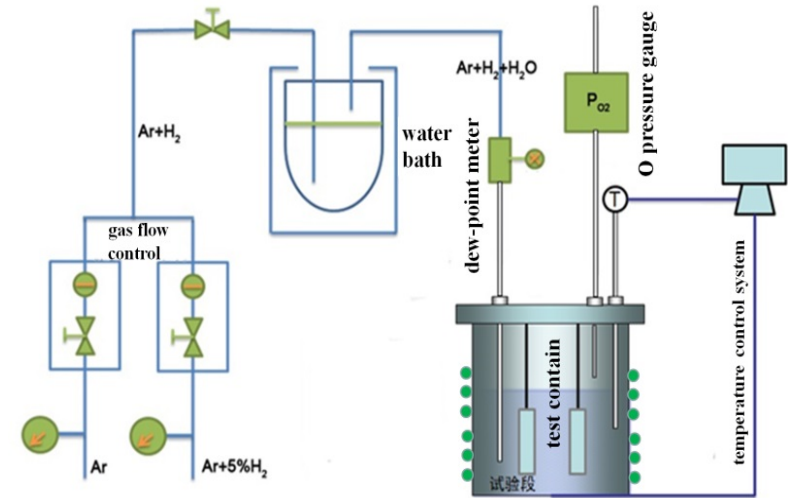
- Studies on the corrosion properties of candidate structural materials, including CN15-15, 316Ti, 316L, 304 stainless steel, FM steel T91, HT9, etc., in LBE for CiADS and LFR.
- Corrosion behavior of coating materials in LBE.
- R&D on high-entropy alloy with radiation resistance and LBE corrosion resistance.



# LBE STATIC CORROSION TEST APPARATUS



Type I

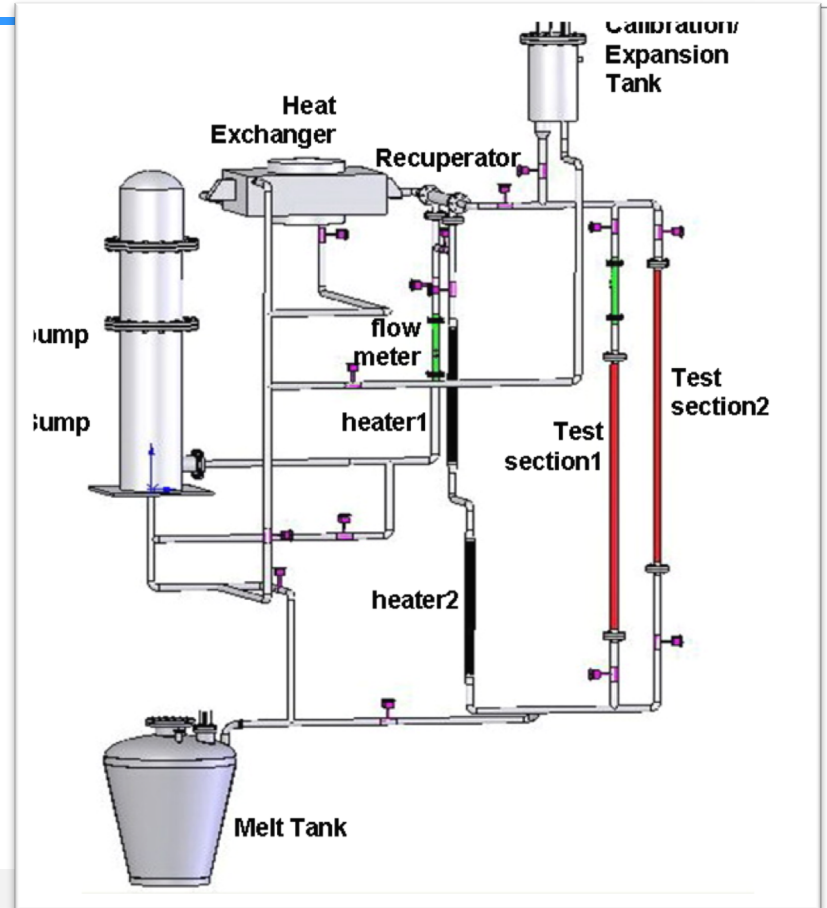


Type II

Maximum test temperature: 800°C  
Oxygen content in LBE: from saturated to  $1 \times 10^{-8}$  wt.%



# THERMAL HYDRAULIC LOOP



## Main Parameters :

- Max T of test section:: 550 °C
- nominal flow rate: 1 m/s ( max 3m/s )
- Number of test section: 2
- Max flow rate:: 6 m<sup>3</sup>/h
- Pressure: 0.3 MPa
- Height of test section : ~2m
- Height of test section : 5 - 6m



# LBE STATIC CORROSION TEST APPARATUS

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## □ Studies performed in the apparatus

- Corrosion properties of austenitic stainless steel (including 316SS, 316LSS, 304SS and 15Cr-15NiTi SS) in LBE at different oxygen contents;
- Corrosion behaviors of ferrite/martensitic steels (including T91, HT9, Si-added FM steel and Al-added FM steel) in LBE at different oxygen contents;
- Corrosion behavior of high entropy alloy coatings on austenitic steels in static LBE



# CORROSION TESTS IN LBE



## □ Specimens and test parameters

	C	Si	Mn	P	S	Cr	Ni	Mo	Ti	V	Nb	N	Fe
T91	0.1	0.4	0.4	<0.02	<0.01	9.0	0.1	1.0		0.2	0.08	0.05	Bal.
CN15	<0.08	0.3~	1.3~	<0.03	<0.01	15.5~	14~	1.9~	0.2~				Bal.
15		0.9	2.0			17.0	15.5	2.5	0.6				
316Ti	<0.08	<1.0	<2.0	<0.035	<0.03	18	12	2.0	0.7				Bal.
316L	<0.035	<1.0	<2.0	<0.045	<0.035	16-18.0	10- 14.0	2.0- 3.0					Bal.
304	<0.08	<1.0	<2.0	<0.045	<0.035	18-20.0	8-10.5						Bal.

Tests conditions and parameters are as follows:

- Sample size: 20mm \*10mm \*2mm
- Test temperatures were 450, 500, 550 and 600°C
- Test time was 1000h, 3000h and 6000h
- Oxygen content in LBE:  $10^{-6}$ - $10^{-7}$  wt.%



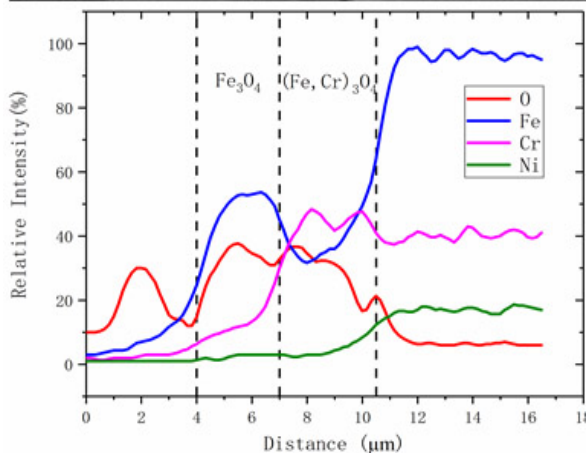
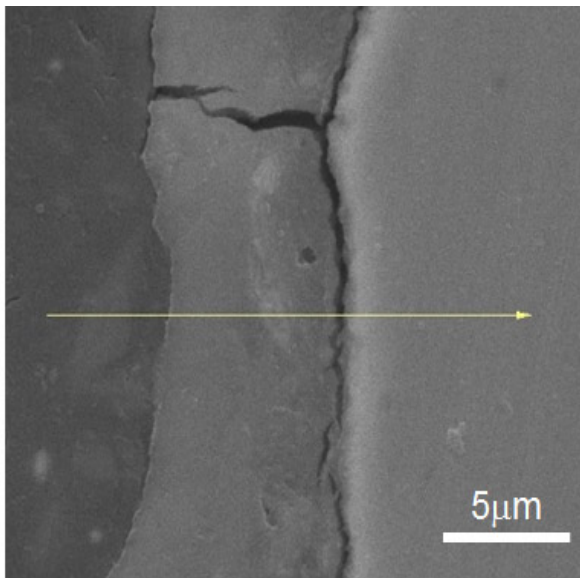


# CORROSION TESTS IN LBE



## Results (at 450°C)

3000hrs

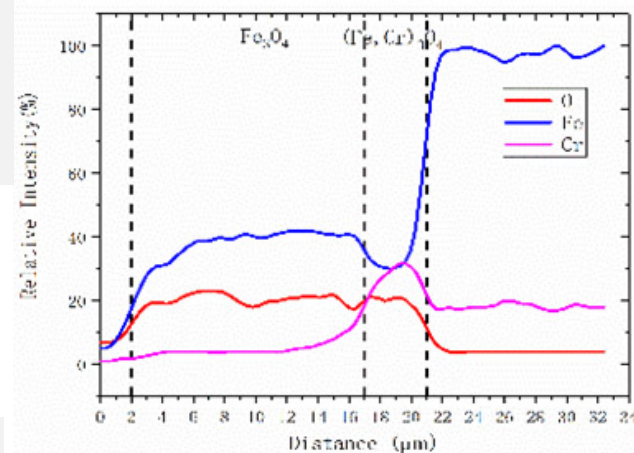
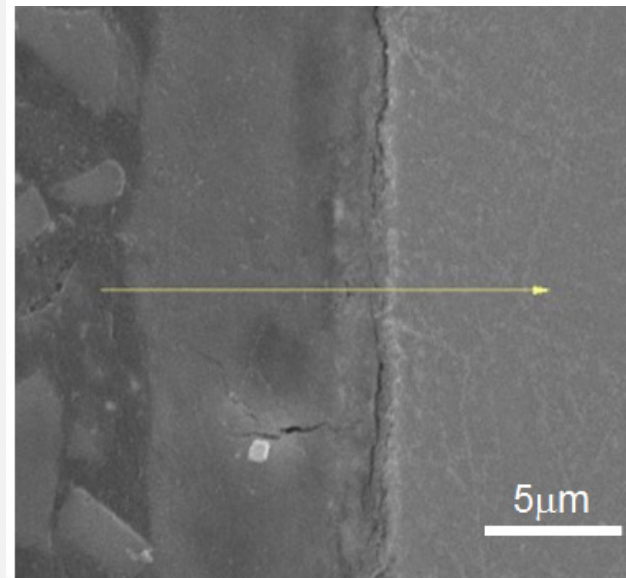


CN1515, 3000h

- the protective oxide layers can be formed on the surfaces for both CN1515 and T91;
- oxide on the surface has a double layer structure, the inner layer is Fe-Cr oxide, and the outer is iron oxide;
- The total thickness of the oxide layer is about 10-15µm, in which the Fe-Cr spinel layer is generally less than 5µm

The oxide layer has a protective effect.

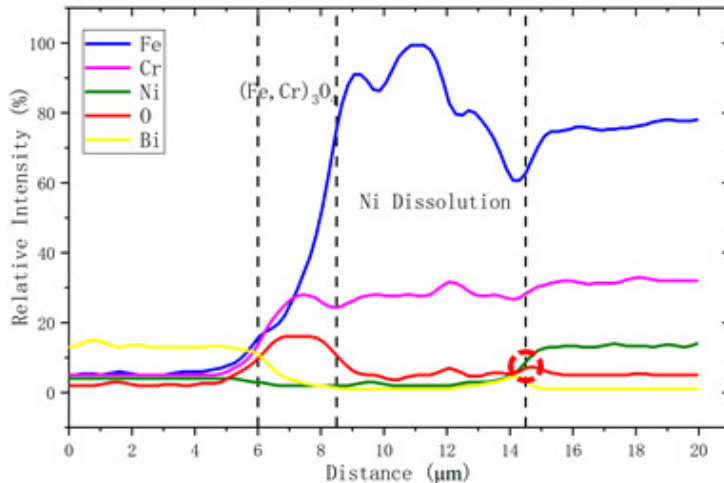
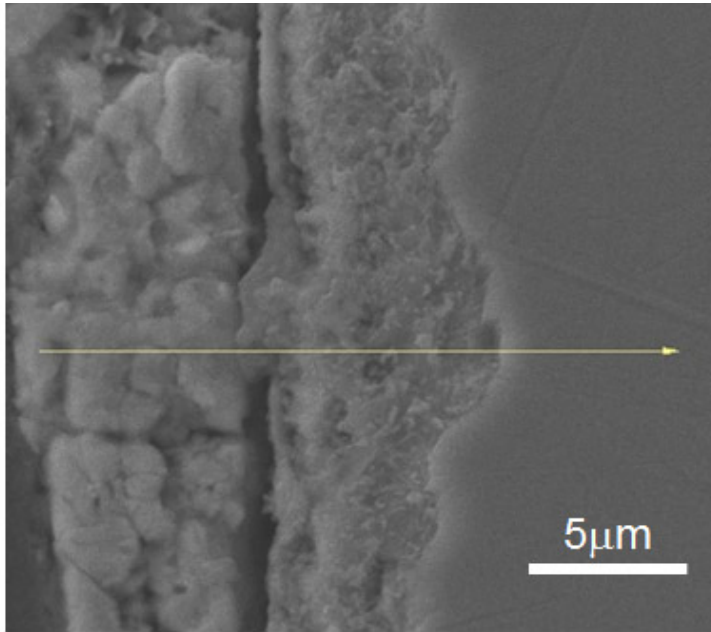
- **Austenitic SS** ✓
- **FM SS** ✓



T91, 3000h

# Results (at 450°C)

## Austenitic SS



450°C 6000h CN1515

### Austenitic SS

- a relatively complete Fe-Cr oxide layer on the surface of CN1515 austenitic stainless steel can still be observed, but lost its protective effect;
- dissolution of Ni and enrichment of Fe were detected in the matrix under the oxide layer

### FM

- Protective oxide layer formed on the surface about 1 μm;
- No lead or bismuth penetration

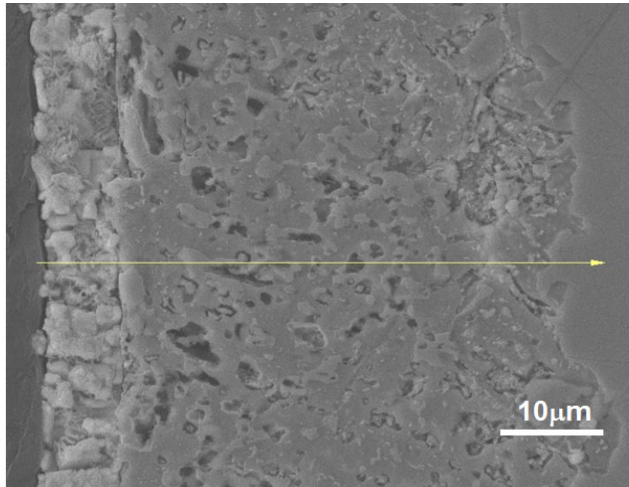
450°C

6000hrs



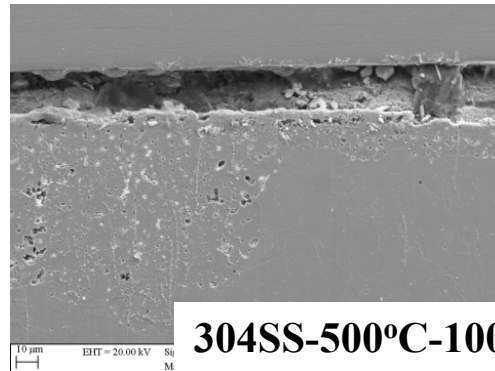
- Austenitic SS ×
- FM SS ✓

# Austenitic SS



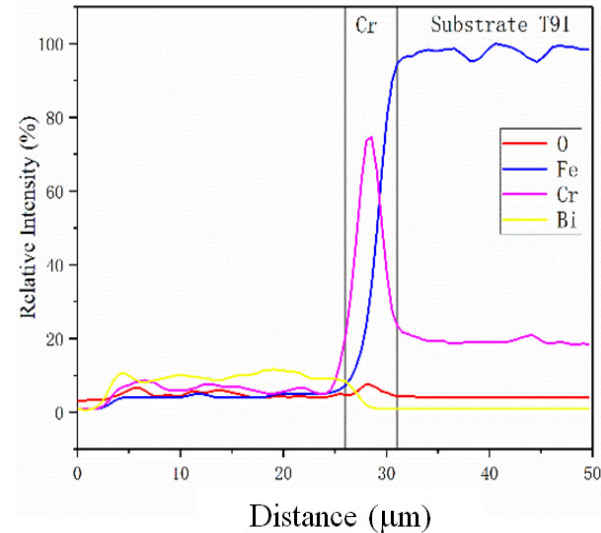
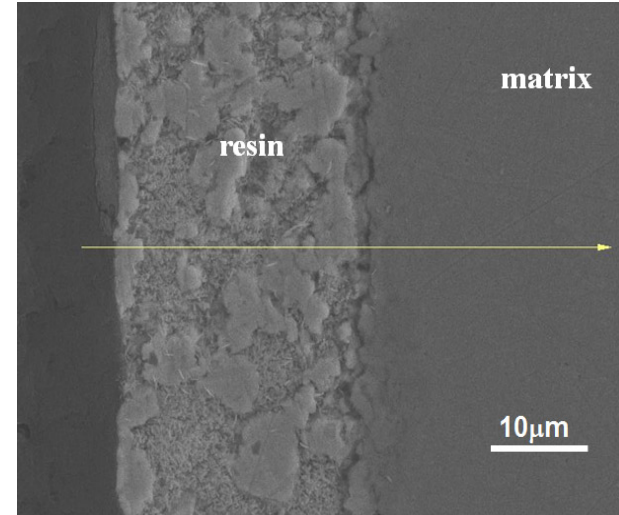
## Austenitic SS

- serious Ni dissolution was observed, and the corrosion depth varied greatly at different locations;
- a lot of cracks and voids in matrix were observed under discontinuous layers which resulting of a large amount of Pb and Bi penetration

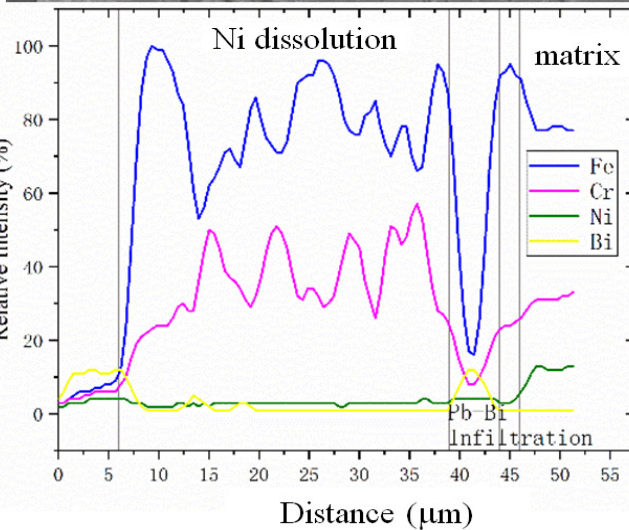


304SS-500°C-1000h

# FM SS



T91-500°C-6000h



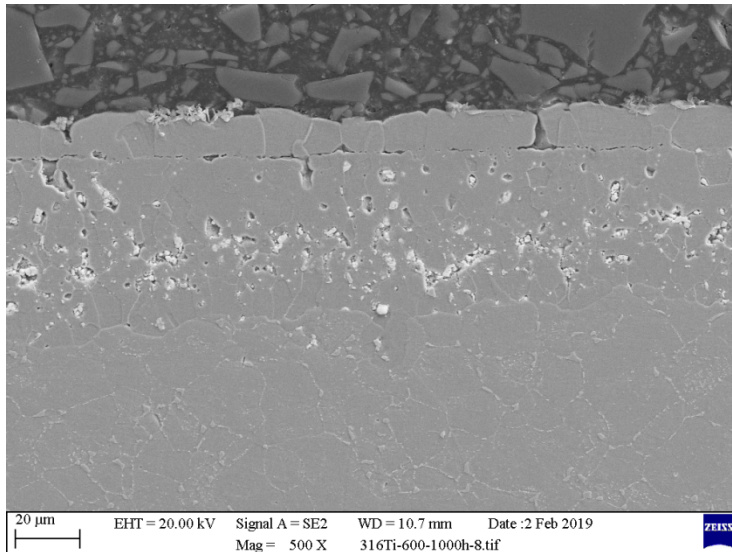
316Ti-500°C-6000h

# FM SS

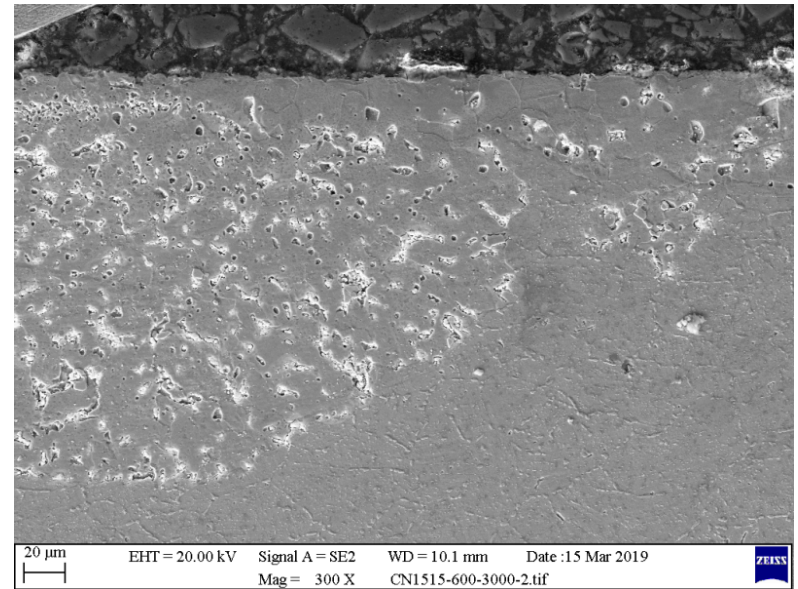
- slight dissolution of Fe and Cr occurred;
- there was a thin Cr-rich layer on the surface of the specimen, which may be a Cr<sub>2</sub>O<sub>3</sub> oxide.
- no lead and (or) bismuth penetration into the matrix

# Austenitic SS at 600°C

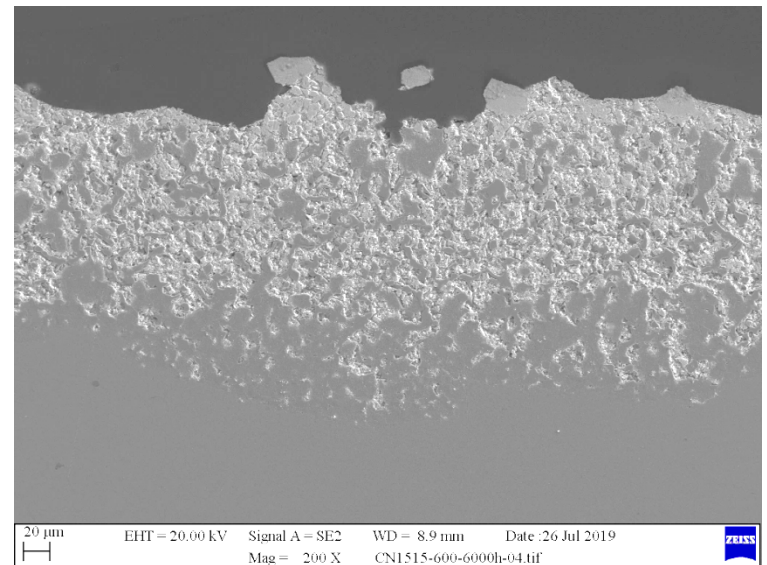
- **No protective oxide** layer was observed on the surface
- Serious **Ni dissolution** occurred **and lead or bismuth penetration** was obvious
- With the increasing of immersion time, the corrosion depth of specimens increased significantly



**CN1515-600-1000h 80µm**



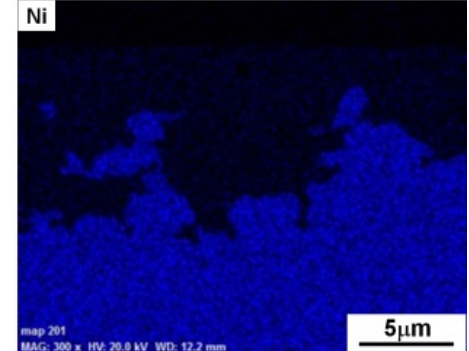
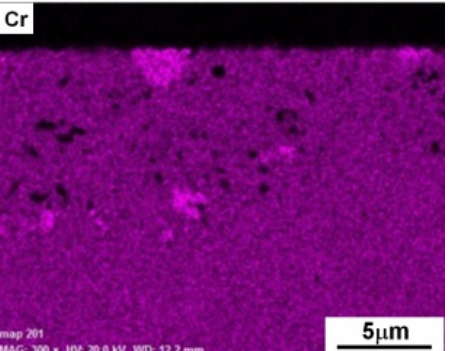
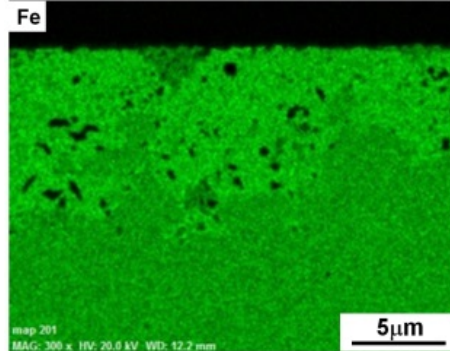
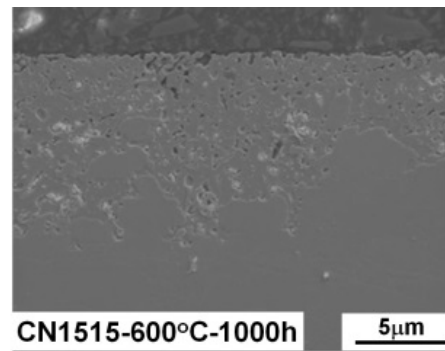
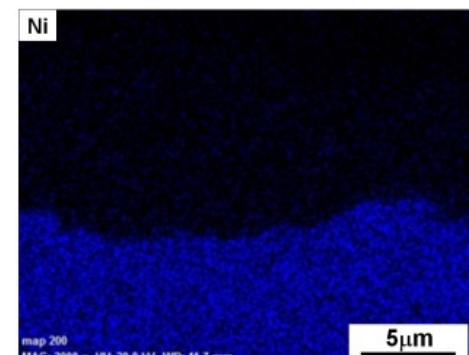
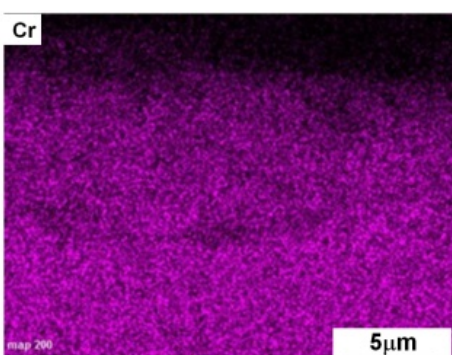
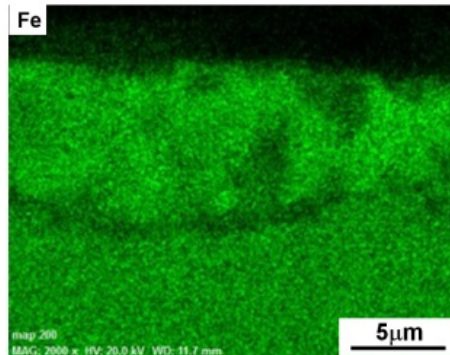
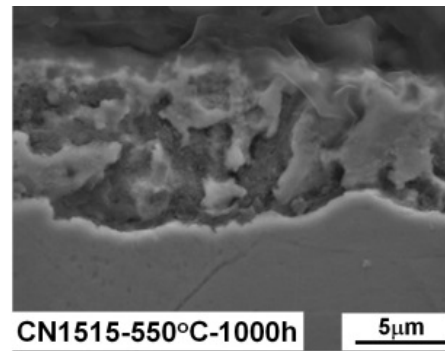
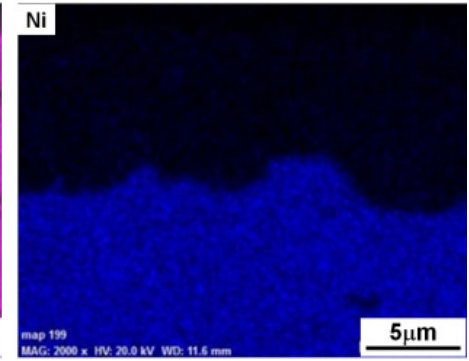
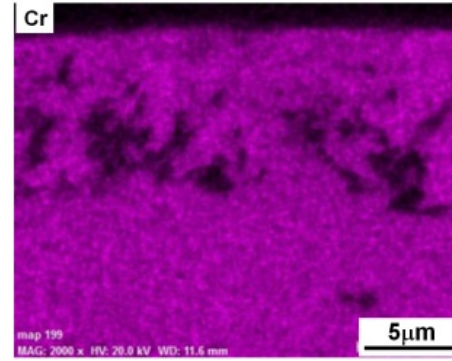
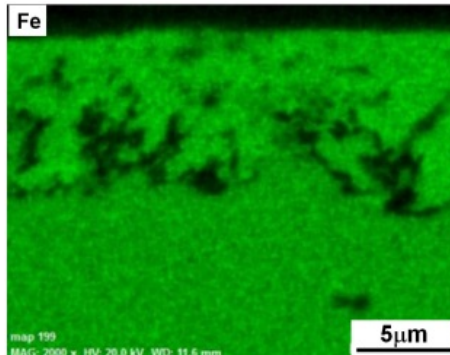
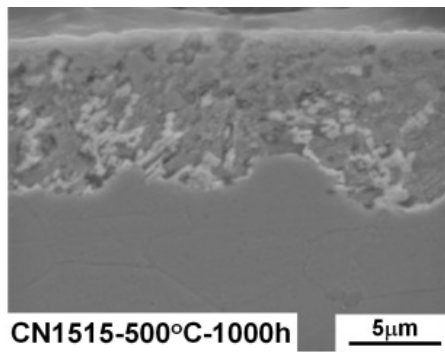
**CN1515-600-3000h 190µm**



**CN1515-600-6000h 234µm**



# Austenitic SS at different T

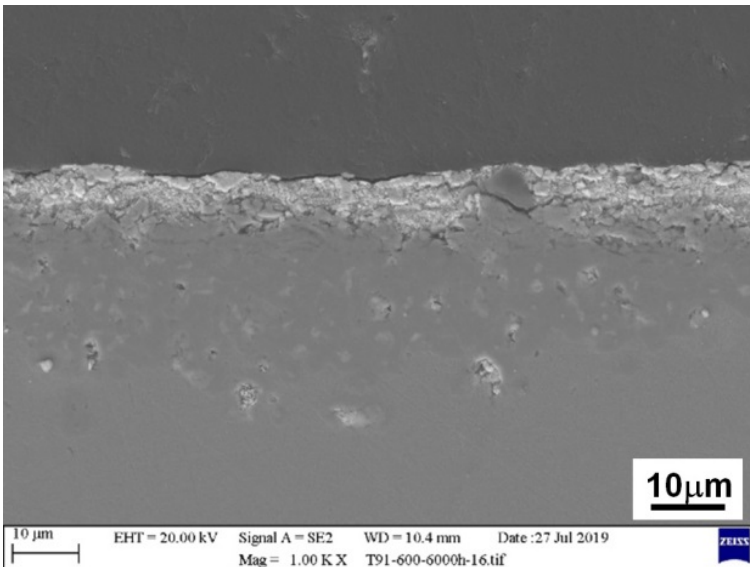


- the corrosion of the steel at 500, 550 and 600°C is mainly due to the diffusion and dissolution of Ni into the LBE

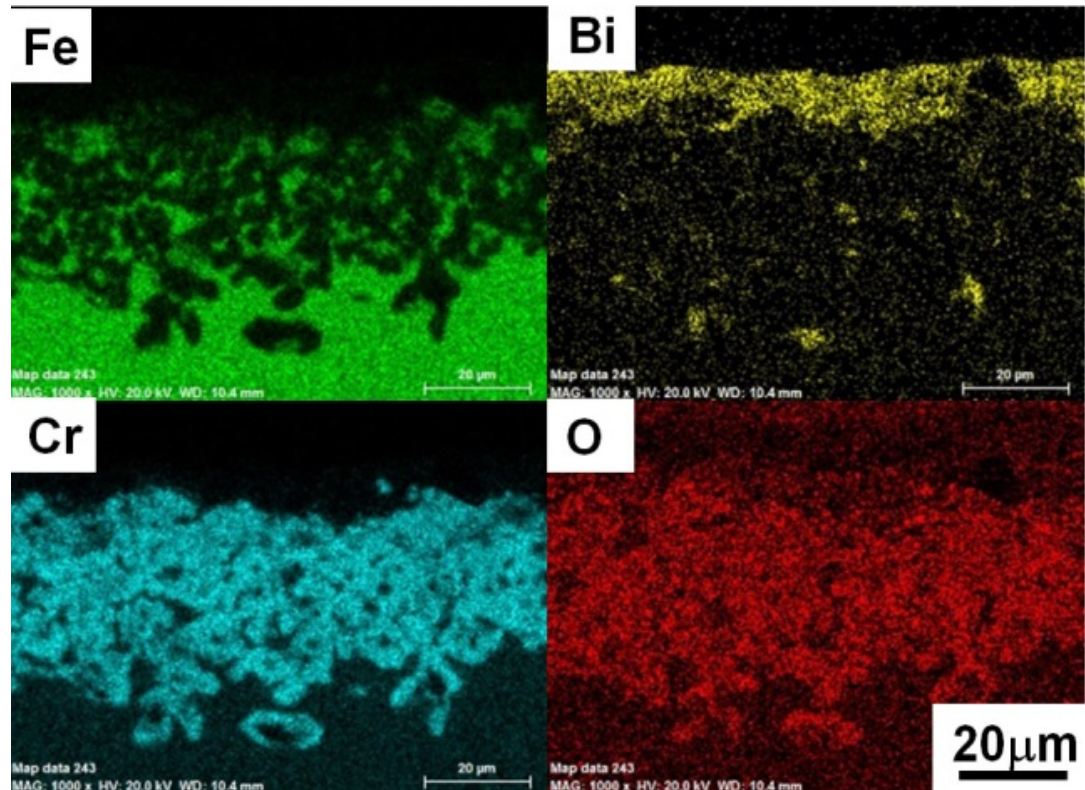
# FM SS at 600°C



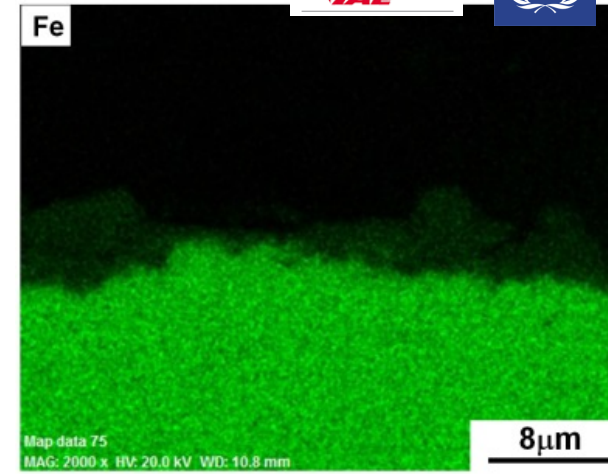
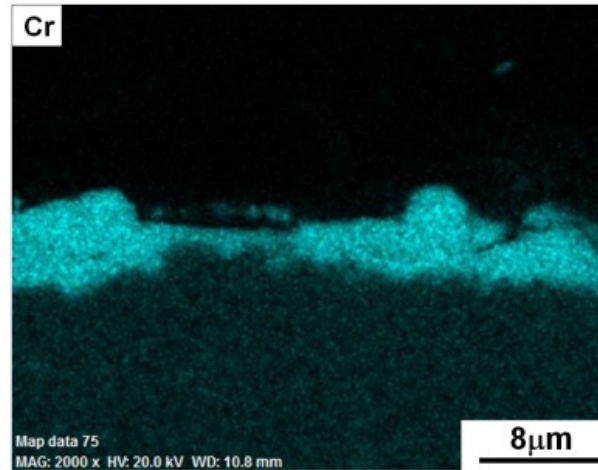
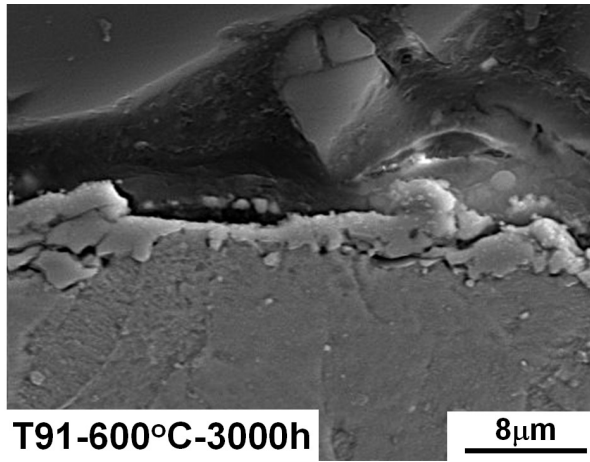
- dissolution occurred;
- penetration of lead and bismuth can be observed , but the depth were very small (could not be detected after 1000h and 3000h)
- maximum corrosion (penetration) depth is about 35μm



T91-600°C-6000h




# FM SS at 600°C



## FM results

- when T less than 450°C, a thin oxide layer about 1 μm formed which could prevent the dissolution of steel elements
- when T greater than 500°C, the steel began to dissolve locally, especially **iron dissolution**.
- the element dissolution was obvious at 600°C, accompanied by the penetration of Pb and Bi.

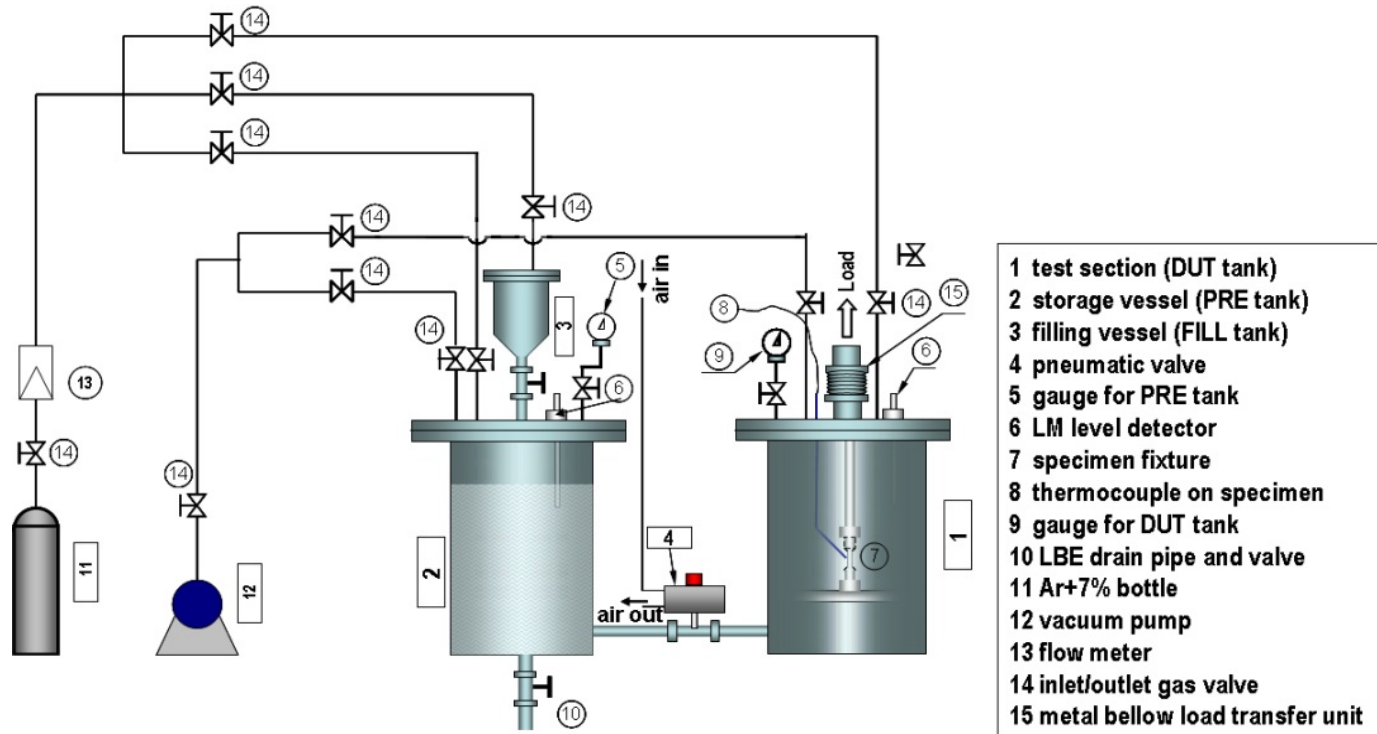
## SUMMARY

- 
- Austenitic steel could be used in LBE with T less than 450°C
  - Service temperature of FM steel in LBE suggested as not exceed to 550°C

# LME EFFECTS in LBE



## □ SSRT test



Tests conditions and parameters are as follows:

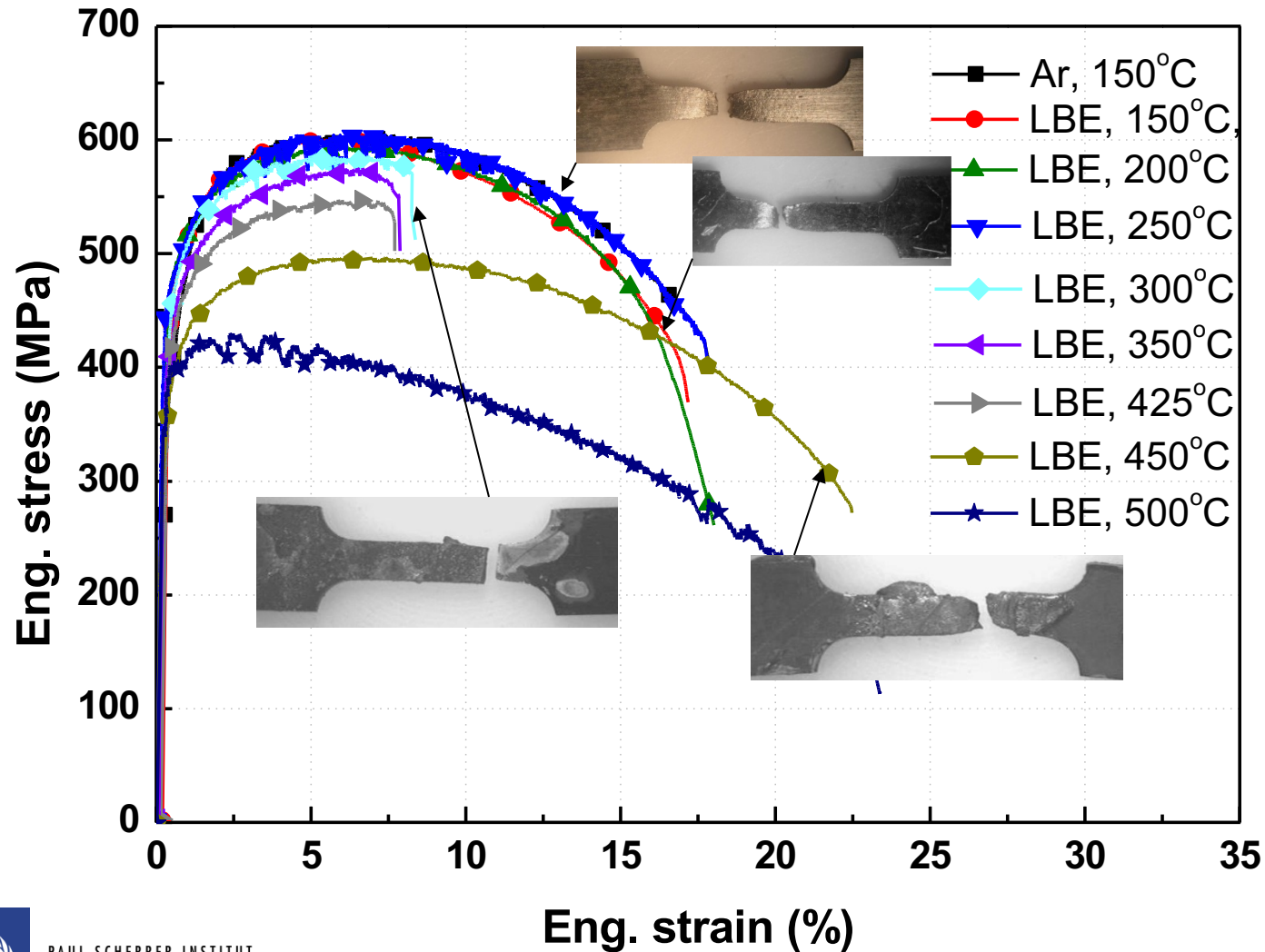
- Test temperatures: 150, 200, 250 , 300 , 350 , 400 , 425 , 450 and 500°C
- Test environments: in Ar and in LBE
- Strain rate:  $1 \times 10^{-5} \text{s}^{-1}$
- Oxygen content in LBE:  $10^{-6}$ - $10^{-7}$  wt.%



# LME EFFECTS in LBE



▲ LBE-induced embrittlement : 300 - 425°C



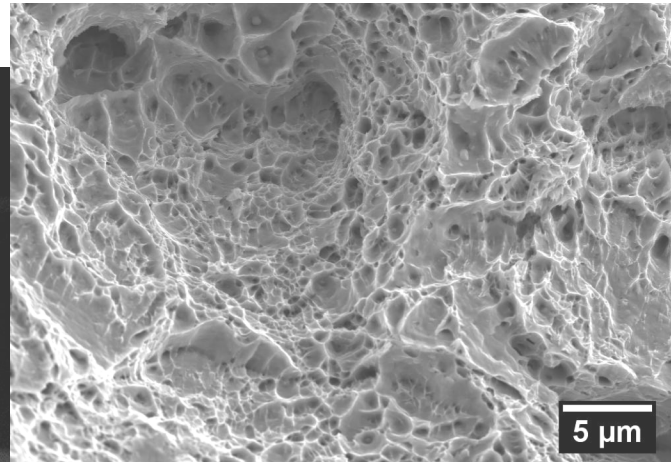
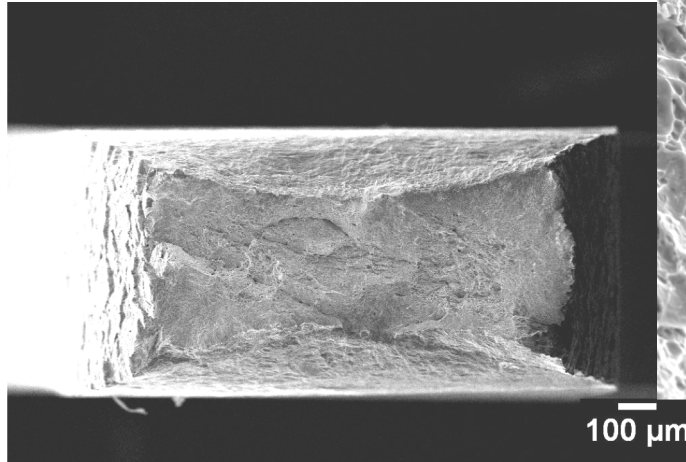
PAUL SCHERRER INSTITUT

PSI

# LME EFFECTS in LBE

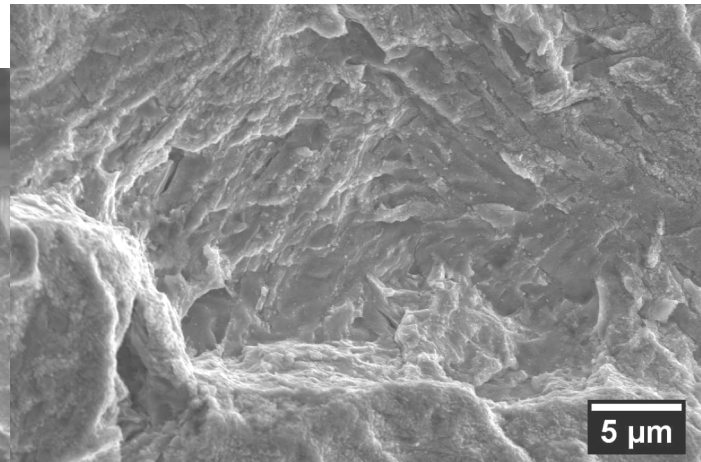
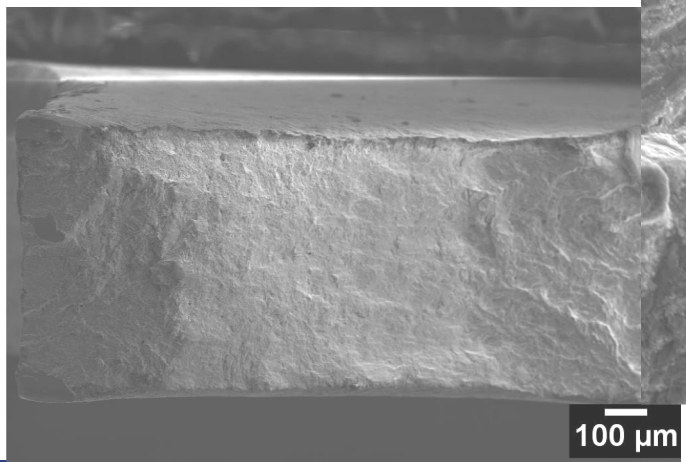


in Ar at 300°C



dimpled  
ductile  
fracture

in LBE at 300°C



transgranular  
cleavage  
brittle  
fracture



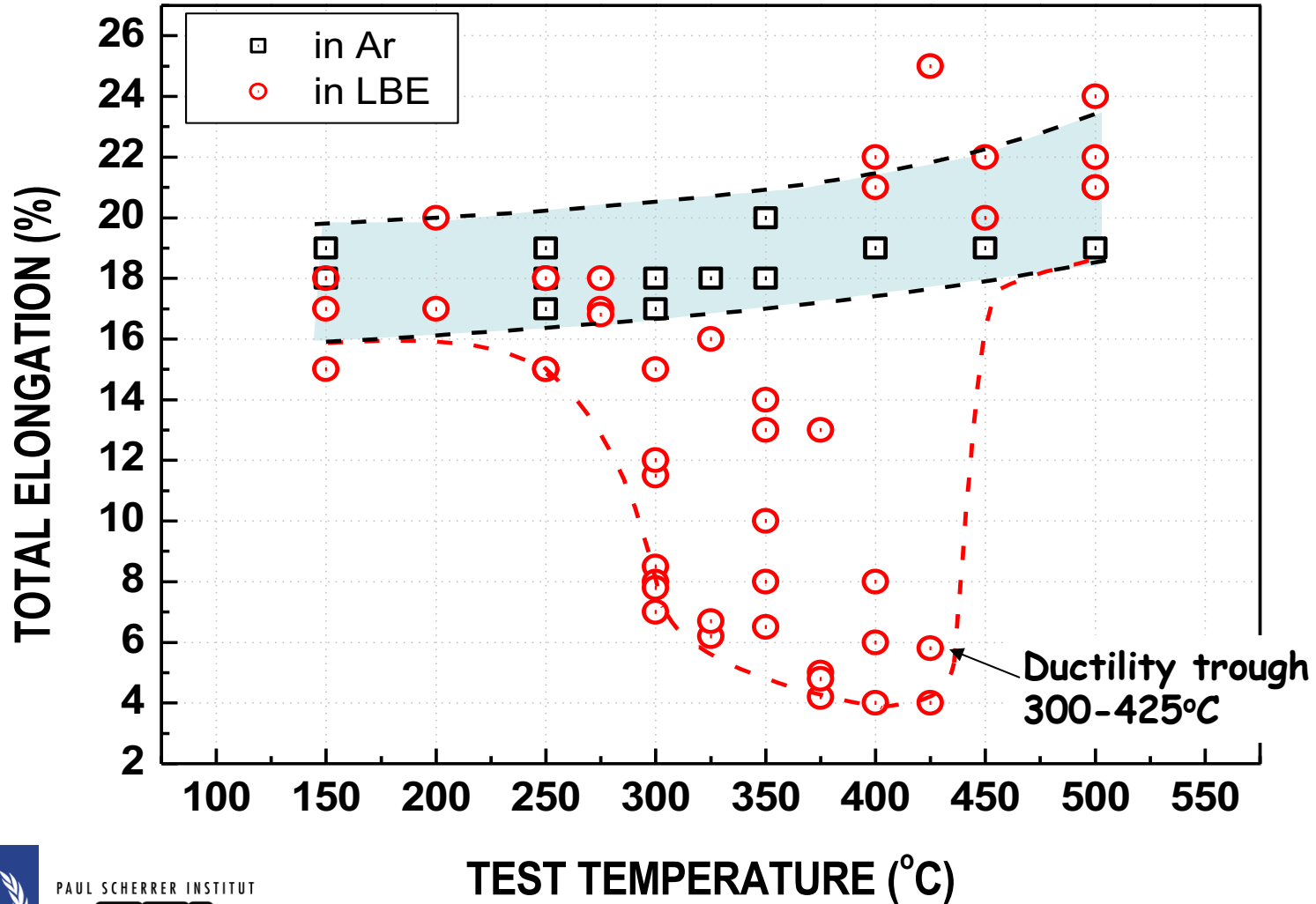
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PSI

# LME EFFECTS in LBE



▲ ductility trough: LME occurs in the temperature range of 300-425°C



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## Summary on SSRT

- **The T91 steel exhibits LBE embrittlement in the temperature range of 300 to 425 °C.**
- **High strength materials increase the sensitivity to LME (wider ductility trough, great degradation of ductility).**
- **The irradiated FM steels (T91 and F82H ) show irradiation induced hardening makes steel sensitive to LME**





# CONCLUSION

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- In order to meet the requirements of structural materials for CiADS and LBE-cold fast reactor, a series of LBE devices and platforms have been established for compatibility studies between LBE and structural materials and thermal hydraulic experiments.
- Preliminary test results show that austenitic stainless steel exhibits better corrosion resistance under 450°C in liquid LBE environment, while FM T91 steel exhibits slightly better corrosion resistance comparing with austenitic stainless steel, and it has good resistance to lead and bismuth corrosion below 550°C.
- The embrittlement effect induced by liquid metal, namely LME, should be considered in the application of T91 steel. Our experimental results give the "ductility trough" of T91 steel in LBE environment.



# CONCLUSION

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- With the improvement of design parameters of CiADS and LBE-cold fast reactor, the existing materials cannot meet the requirements of the system and equipment for their corrosion resistance. Therefore, the following studies are suggested:
- 1) R&D on new structural materials with resistant to LBE corrosion and neutron irradiation damage;
  - 2) R&D on anti-corrosion coating technology on structural materials with resistant to irradiation damage;
  - 3) Study on OCS of LBE for industrial application



# Technical Meeting on Structural Materials for Heavy Liquid Metal Cooled Fast Reactors

IAEA Headquarters, Vienna, Austria  
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## Thank you for your attention !

