

Salt Irradiations at the Nuclear Research and Consultancy Group

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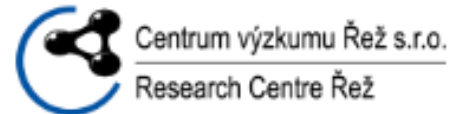
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Ensuring Nuclear Performance

Dutch program overview

- Sponsored by the Dutch Ministry of Economic Affairs as part of a broader Nuclear Energy R&D program.
- Collaborations with JRC, TU Delft and CV Rez
- Program objective: contribute to MSR technology development and realization
 - Obtain **operational experience** (salt handling, liquid fuel irradiation)
 - **Qualify materials (alloys, graphite) and fuels (fluoride, chloride)**
 - Study **fission product behavior** (normal and accident conditions)
 - Tackle **waste issues**
 - Work towards **Integral Demonstration**



High Flux Reactor Petten

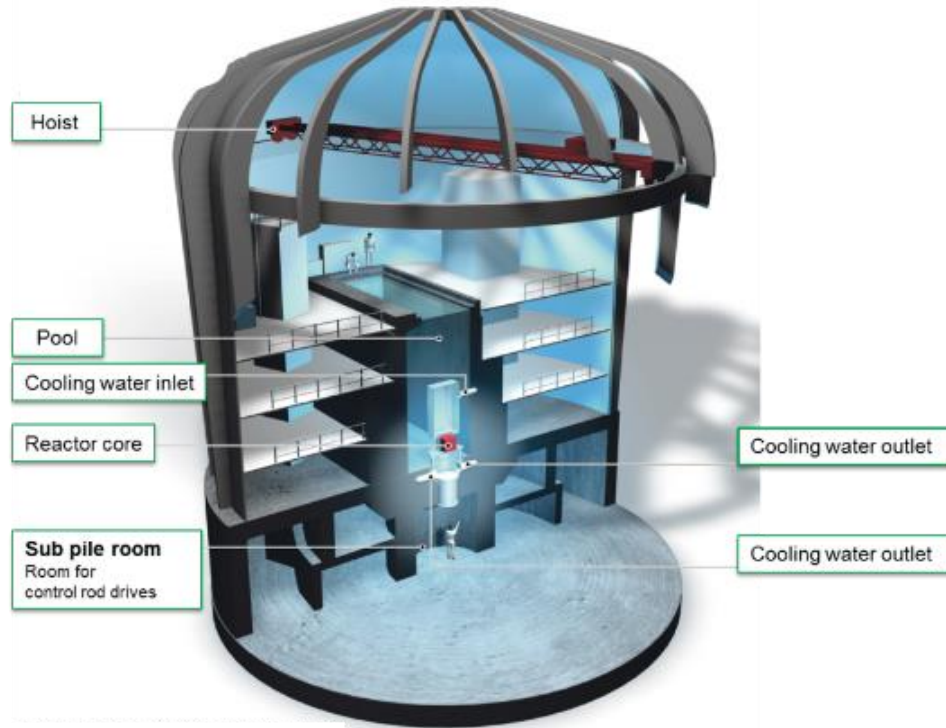
Nickel-based Alloy Irradiation & Mechanical Testing
(ENICKMA and ENICKMA-HTC)

In-pile Hastelloy N corrosion by fluoride fuel salt

LiF-ThF₄ irradiation in graphite crucibles & graphite
qualification

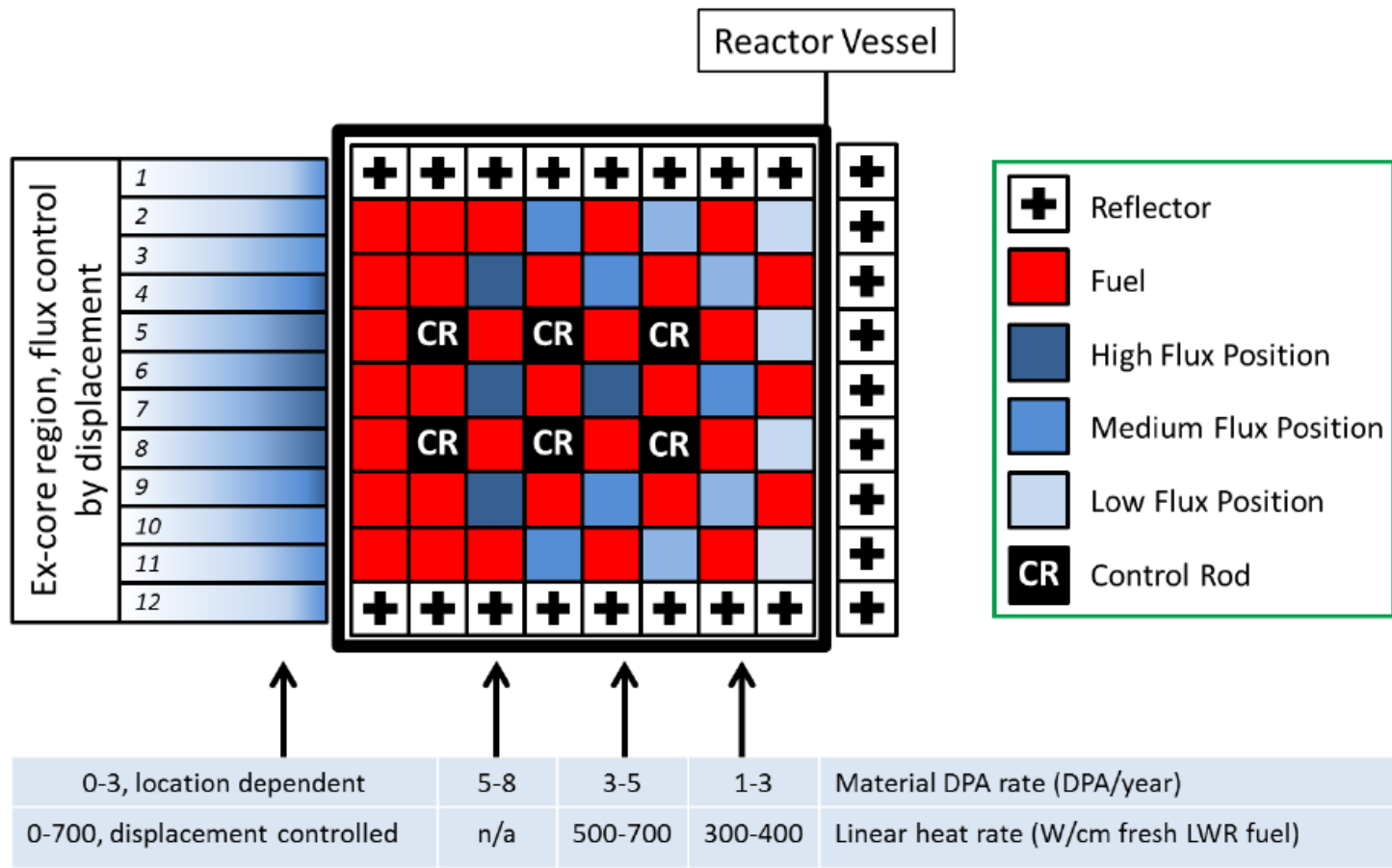


The High Flux Reactor



- High flux
- 45 MW thermal power
- Stable and constant flux profile in each irradiation position
- Main applications
 - Isotope production
 - Nuclear energy irradiation services
 - R&D
- 31 operation days per irradiation cycle, 9 cycles a year





The stable and constant flux profile in each irradiation position is a unique HFR feature

ENICKMA project

- **Goal:** Study material degradation behavior under neutron irradiation at high temperature + investigate underlying mechanism
- **Materials:** Nickel alloys with compositions close to Hastelloy N

ENICKMA materials

- Hastelloy N (Haynes)
- MONICR (CV Rez)
- HN80MTY (COMTES FHT)
- GH3535 (SINAP)
- Hastelloy 242 (Haynes)
- 316 L(N) (CEA)

HAYNES
International

COMTES FHT
Complete Technological Service - Forming, Heat Treatment



TU Delft
Technische Universiteit Delft

Centrum výzkumu Řež s.r.o.
Research Centre Řež

SINAP

Alloy	Weight % Main Alloying Elements											
	Al	Co	Cr	Fe	Mn	Mo	Nb	Ni	Si	Ti	V	C
Hastelloy N	0.29	0.078	7.10	3.60	0.46	17.10	0.070	Bal.	0.31	0.002	0.005	0.059
Hastelloy 242	0.17	0.026	8.00	1.16	0.26	25.80	<0.001	Bal.	<0.02	0.001	0.002	0.002



High-Temperature embrittlement

Helium embrittlement
Thermal embrittlement

Helium embrittlement in nickel based alloys

- (n, α) reactions in ^{10}B (fast burn-out) and $^{58,59}\text{Ni}$ (slow but steady)
- He-production from fast neutron (n, α) reactions

$$^{58}\text{Ni} + n_f \rightarrow ^{55}\text{Fe} + ^4\text{He}, \quad ^{60}\text{Ni} + n_f \rightarrow ^{57}\text{Fe} + ^4\text{He}$$
- For thermal neutrons

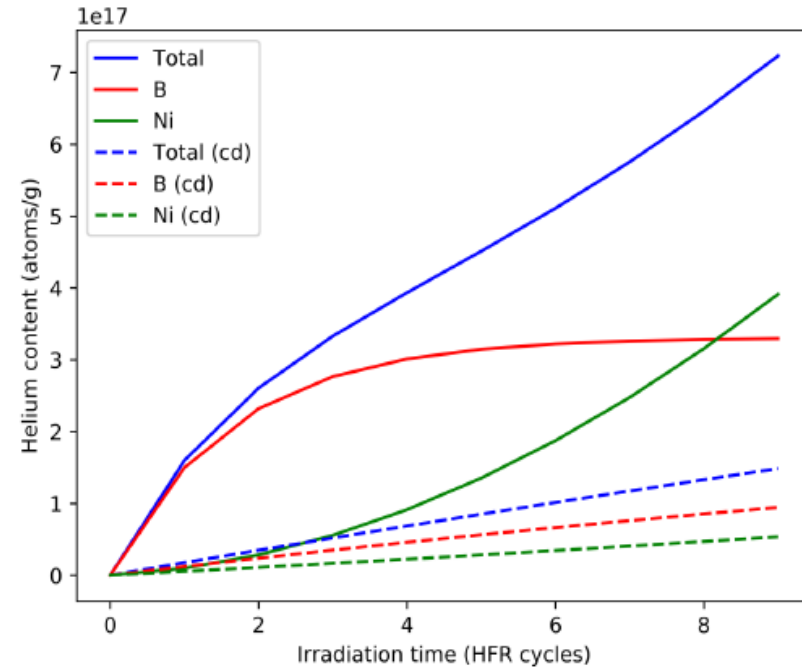
$$^{10}\text{B} + n \rightarrow ^7\text{Li} + ^4\text{He}$$

$$^{58}\text{Ni} + n \rightarrow ^{59}\text{Ni} + \gamma, \quad ^{59}\text{Ni} + n \rightarrow ^{56}\text{Fe} + ^4\text{He}$$

Target fluence: Up to $1\text{E}21$ n/cm² thermal,
 $3\text{E}21$ n/cm² fast (up to 50 appm helium, >1 dpa)

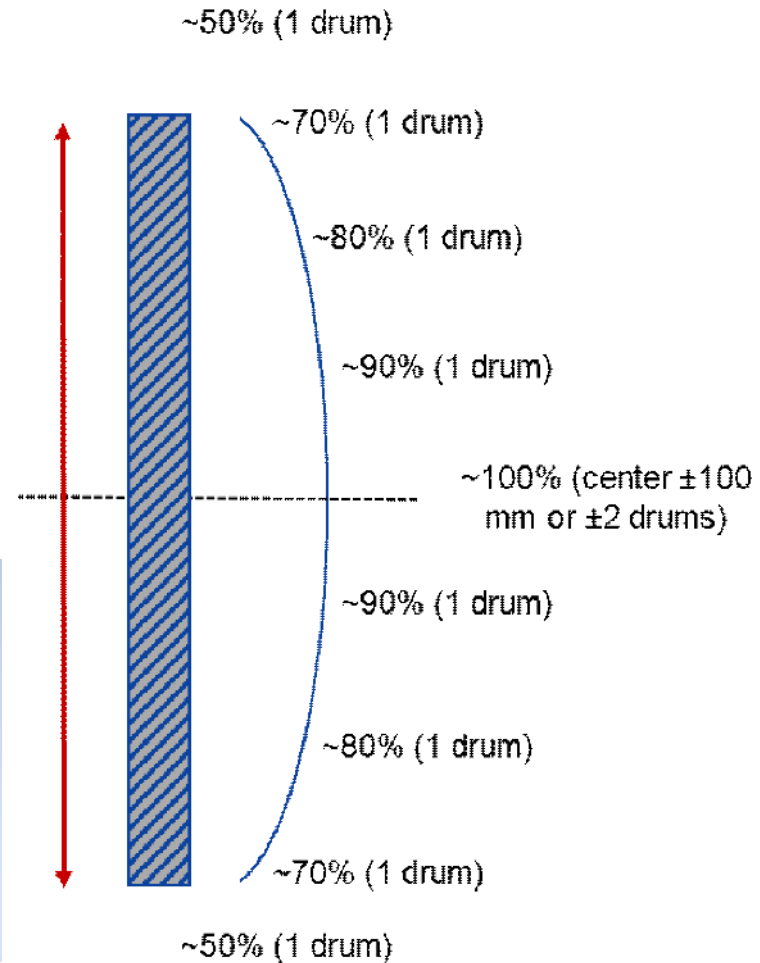
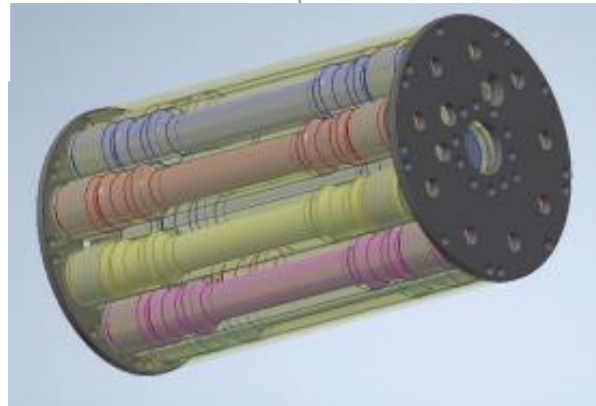
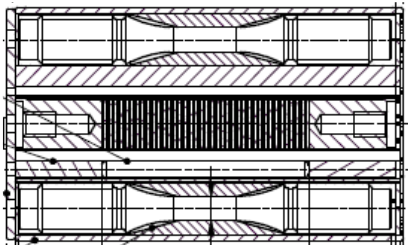
Sample temperatures 650 °C and 700/730 °C

- Oven anneal test at same temperatures for comparison with pure thermal embrittlement



ENICKMA irradiation capsule

- Welded capsule with inert gas plenum
- 10 drums each containing 10 specimens (tensile, LCF)
- Sample temperatures 650 and 700-730 °C
- Irradiation for 9 months (9 HFR cycles)



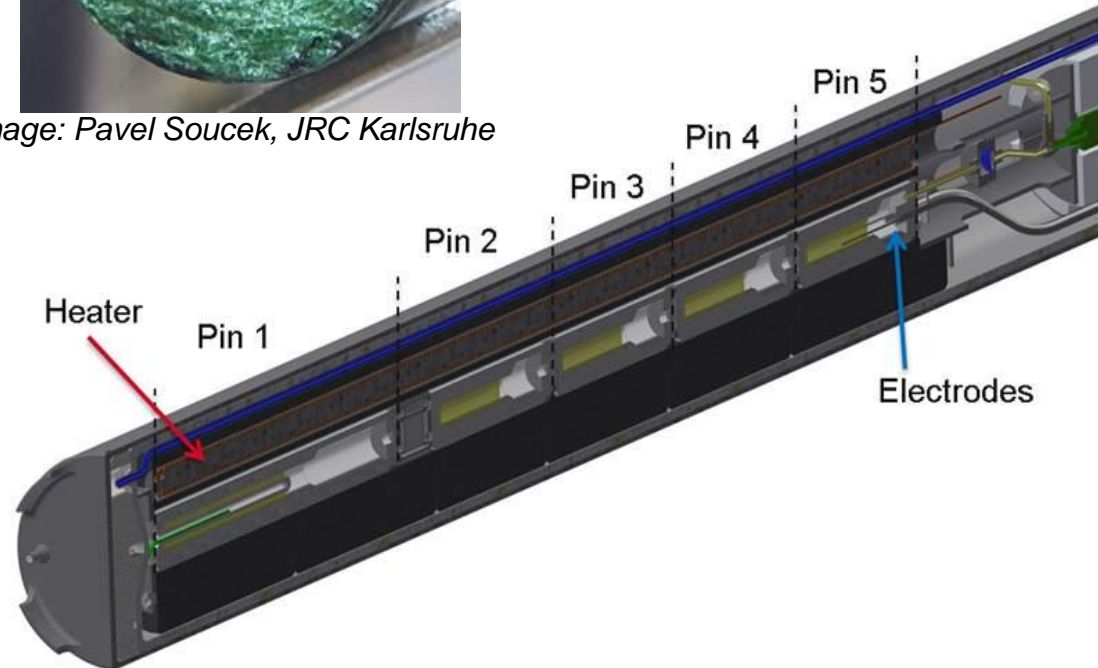
SALIENT-03

- Irradiation of 5 salt-filled capsules
- Investigates **in-pile corrosion of Hastelloy N** for different salt chemistry:
 - LiF-ThF₄-**UF₄**-PuF₃
 - LiF-ThF₄-**UF₄**-**UF₃**-PuF₃
(UF₄/UF₃ = 20-50)
 - LiF-ThF₄-**UF₄**-PuF₃ + CrF_x
- Additional focus on behavior/migration of non-soluble and volatile **fission products**
- Attempt to measure redox potential and FGR in-pile
- Use of **heaters** to avoid radiolysis during downtime (T ~150 °C)

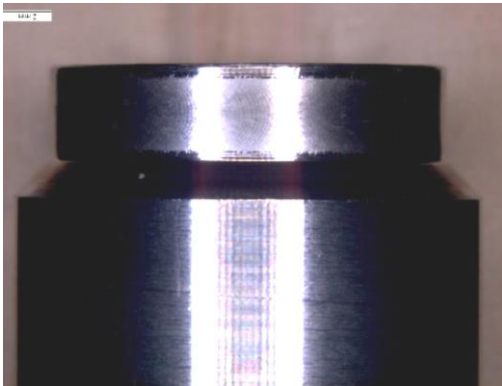


Image: Pavel Soucek, JRC Karlsruhe

Corrosive salt
Burn-up / fission products



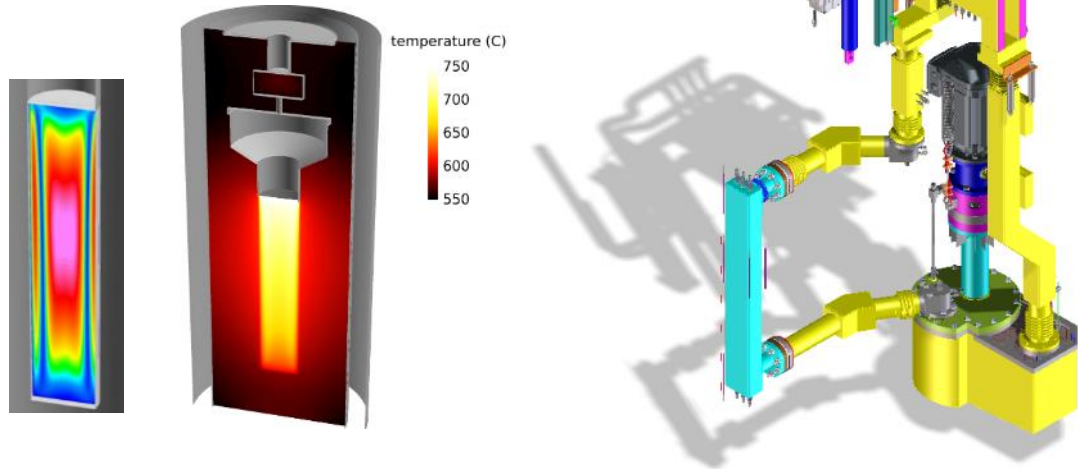
SALIENT-03 - Assembly



In-pile capsule corrosion tests vs. molten salt loops

- Convective flow at a rate of 1-2 cm/s creates an axial temperature gradient
- The axial gradient is of interest in relation to corrosion testing, creating conditions similar to convective flow loops.

	Capsule	Loop
Salt flow?	1-2 cm/second	meters/second
Temperature gradient	yes (less control)	yes (good control)
FP build-up	fast	slow
Volume to Area ratio	very low	low



Status and Outlook

ENICKMA

- Irradiation near completion (1 30-day cycle to go)
- 9-month oven anneal at 650 °C nearly completed
- **Next steps:** post-irradiation mechanical testing and microscopy (SEM/TEM)
- **Follow-up:** In-pile (tensile) creep experiment at 700 °C

SALIENT-03

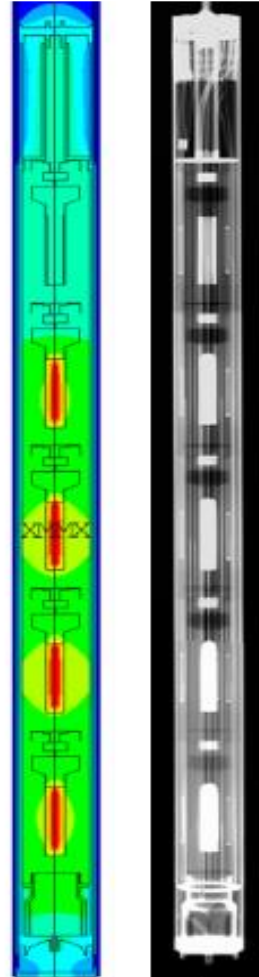
- Salt samples received from JRC Karlsruhe
- Fabrication & Assembly of the irradiation capsule ongoing
- Start of Irradiation expected Q1 2023

MIMOSA (EU HORIZON project)

- Similar studies in relation to chloride salt reactor

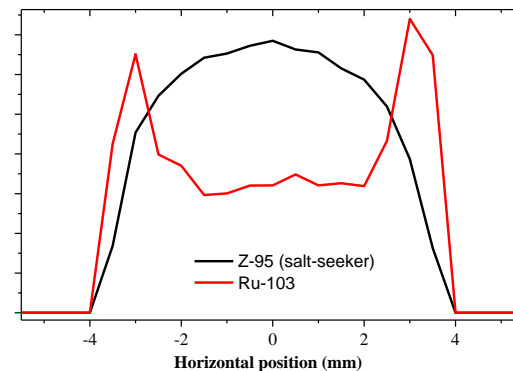
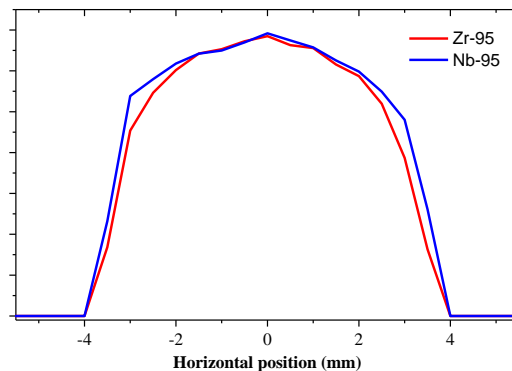
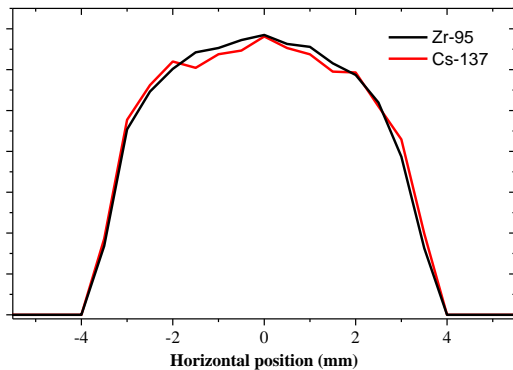
SALIENT-01

- Irradiation of ^{78}LiF - $^{22}\text{ThF}_4$ salt
- 5 open capsules fabricated from nuclear-grade graphite (4 loaded) in containment with He-Ne mixture
- Fuel power rises during irradiation due to production of U-233
- Fixed crucible temperature ($\sim 600\text{ }^\circ\text{C}$) actively maintained during irradiation

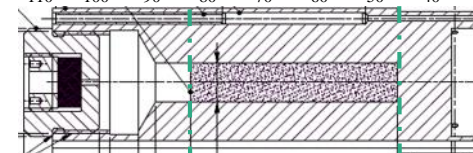
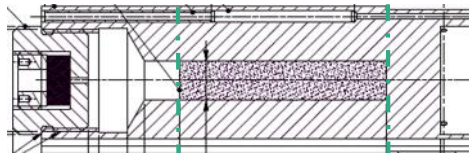
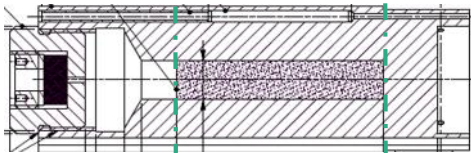
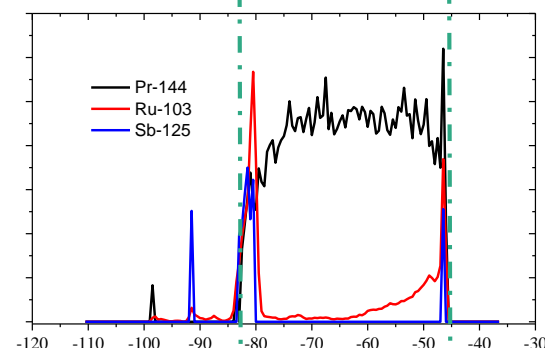
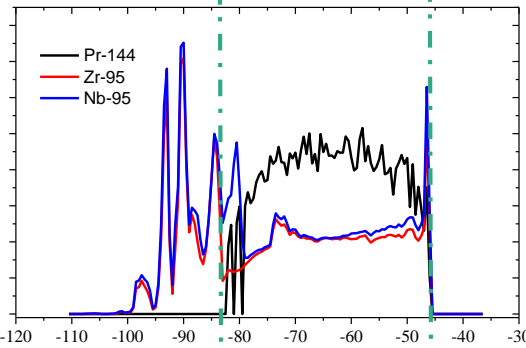
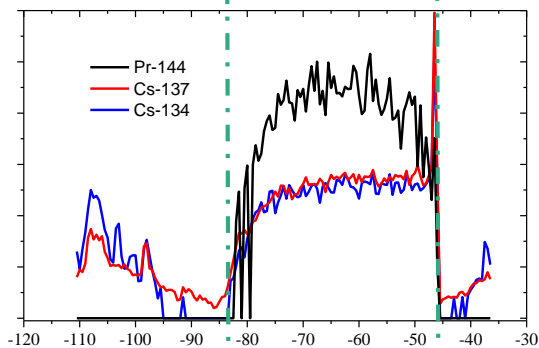


Results gamma spectroscopy

Radial scan
(ID crucible 7.6mm)

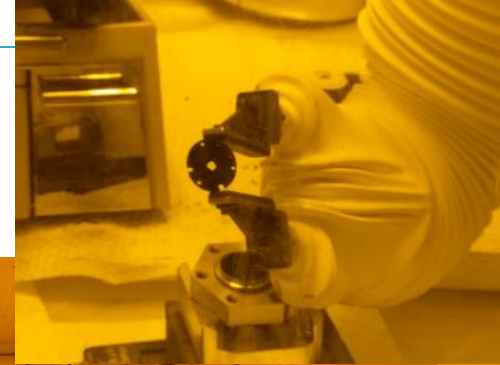
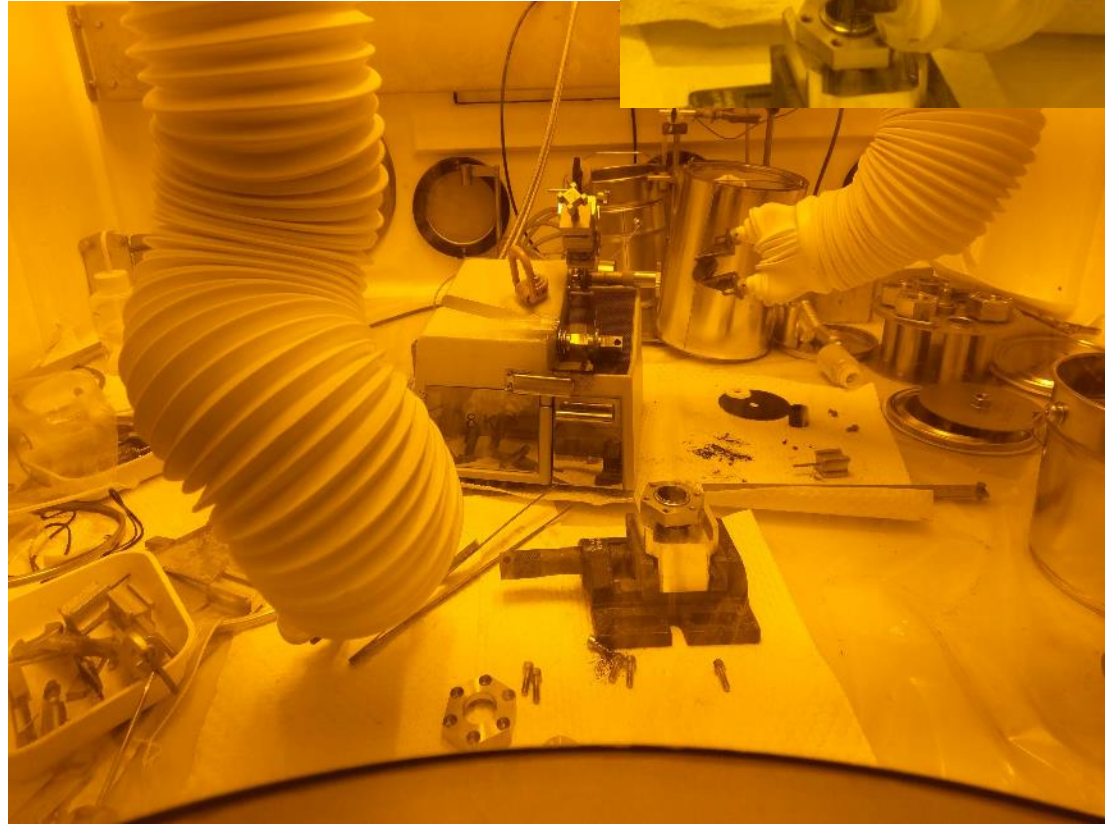


Axial scan
(L crucible 80mm)



Dismantling of graphite crucibles

- Alpha hot cell, nitrogen-flushed ($O_2 < 0.4\%$, $H_2O < 200$ ppm) for
 - dismantling
 - aqueous chemistry/sampling
 - ICP-MS / ICP-OES
 - Gamma spectrometric analysis
 - high-temperature oven testing (oven in test phase)
- Alpha hot cell, nitrogen-flushed ($O_2 \sim 1\%$) for electron microscopy (SEM/EDS/WDS/EBSD)



SALIENT-01 - Conclusions and next steps

Conclusions

- Successful irradiation of fuel salts in graphite crucibles
- First PIE results available give information on distribution and retention of fission elements

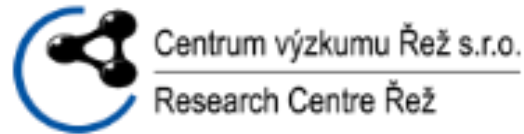
Next steps

- Continue preparation of samples for in-cell microscopy (light microscopy and SEM/EDS/WDS)
- Send samples to JRC Karlsruhe for TEM and Knudsen Cell tests

Acknowledgements



Ensuring Nuclear Performance



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