

# Tensile testing of sub-sized T91 steel specimens in liquid lead

Z. Száraz, K. Tuček, R. Novotný, K.-F. Nilsson

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Joint Research Centre



# Joint Research Centre (JRC)

- The Joint Research Centre is the **European Commission's science and knowledge service**
- Mission of JRC is to support EU policies with independent evidence throughout the whole policy cycle
- JRC hosts specialist laboratories and unique research facilities and is home to more than **2000 scientists** working to support **EU policy** and **EU Member States**
- The JRC has ten Directorates and is located across five EU Member States (Belgium, Germany, Italy, the Netherlands, and Spain)
- The JRC's nuclear work is conducted by the **Directorate G for Nuclear Safety and Security** and funded by the Euratom Research and Training Programme. JRC pursues research, knowledge management and training activities with an emphasis on nuclear safety and security



## **Specialist laboratories and research facilities at JRC, incl.**

High Flux Reactor (HFR) for fuel and material behaviour studies under irradiation

Actinide User Laboratory for handling of transuranium materials

AMALIA laboratory for studies of ageing of materials in LWR environments

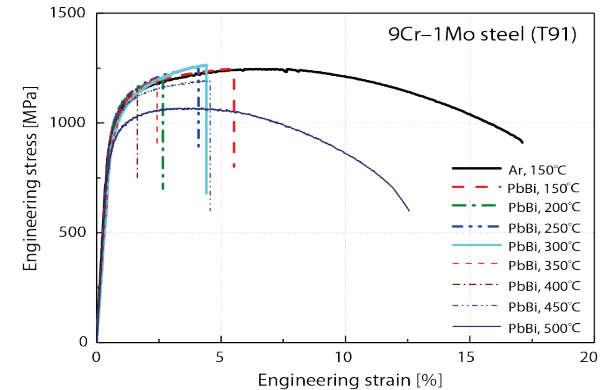
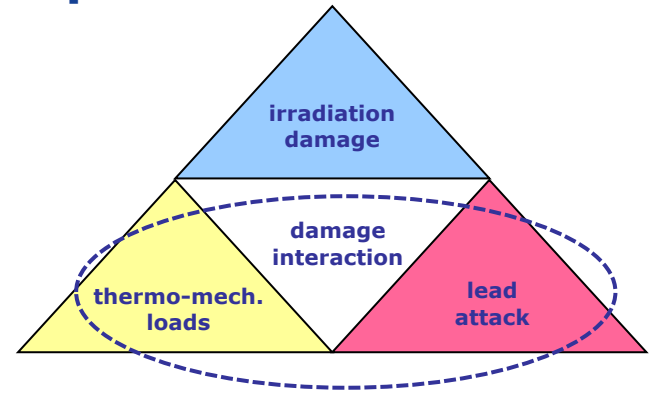
Mechanical Performance Assessment laboratory for pre-normative research

High Performance Computing clusters for physics-based/multi-scale modelling



# New LILLA facility for material testing in liquid lead

- **Objective:** contribute to feasibility and safety assessment of future nuclear power systems
  - **Safety**
  - **Sustainability** (uranium utilization & nuclear waste management)
  - **Economy**
- The LILLA (LIquid Lead LABoratory) facility allows conducting **qualification tests** of candidate structural materials for **Generation-IV** lead-cooled fast reactors (**LFR**) in temperatures up to **650°C**
- The LILLA facility is designed to study liquid metal embrittlement (LME) phenomena and perform mechanical tests in liquid lead with well-controllable parameters, in particular temperature, load, and oxygen content in **lead**
- Tests of the reliability of lead chemistry control systems and related components and instrumentation are also possible



B. Long, Z. Tong, F. Gröschel, Y. Dai, *Journal of Nuclear Materials*, 377 (2008) p. 219-224

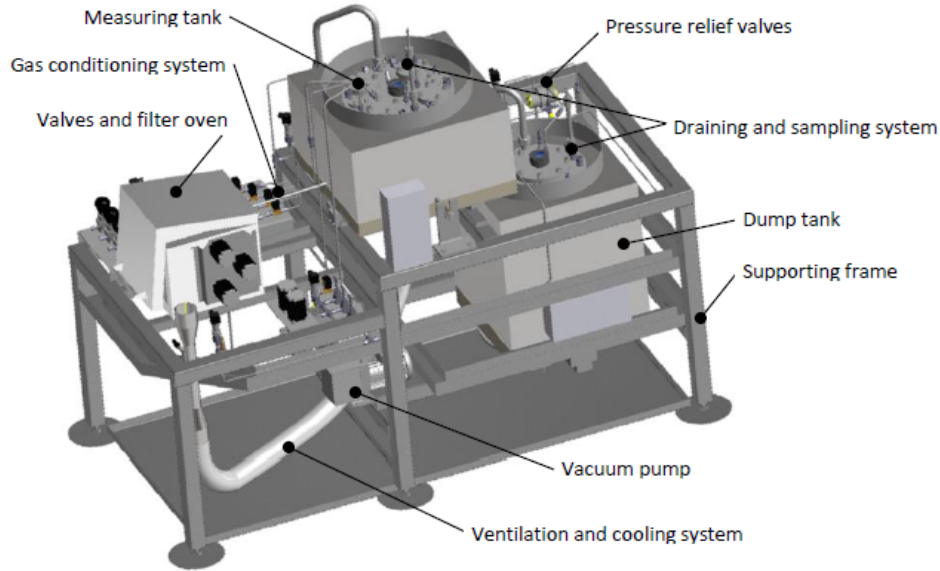
# Laboratory space requirements for LILLA

For the LILLA facility, a dedicated laboratory space was set up with specific requirements concerning, among others:

- Temperature in the facility room: 20-24°C
- Air supply in the control room: 500 m<sup>3</sup>/h
- Air supply in the facility room: 750 m<sup>3</sup>/h
- Room ventilation rate: 5-8x of the room volume per hour
- Under pressure in the facility room: 5-10 Pa
- Under pressure in the exhaust of the facility: 400-500 Pa
- Suction capacities:
  - Glove box: 10 m<sup>3</sup>/h
  - Facility: 30-50 m<sup>3</sup>/h
  - Facility room: 1 100 m<sup>3</sup>/h
- HEPA filters
- Washable painting, self-closing door, ventilation alarm, hydrogen and oxygen sensors



# New LILLA facility for material testing in liquid lead

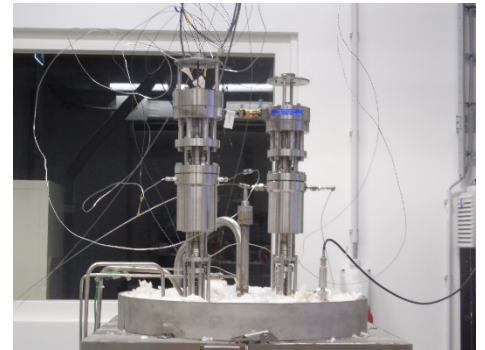
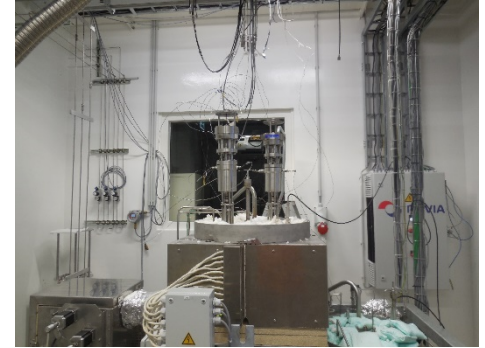


The facility consists of two cylindrical tanks, measuring tank and dump tank, and connecting piping to transport lead between the tanks as well as to deliver and extract gases to and from the facility, respectively.

The LILLA facility has been commissioned in 2018

# Main performance characteristics of the LILLA facility

- **Working temperatures in lead: up to 650°C**
- **4 test sections**
- Lead inventory: ca. 27 l / 280 kg
- Structural material: AISI 316Ti
- **Surfaces in contact with molten lead are protected by aluminium coating using pack cementation technology**
- **Possibility for active control of oxygen / gas composition, down to low oxygen concentrations ( $< 10^{-8}$  wt.%)**
- **Chemistry-controlling gas can be injected to cover gas space as well as directly to lead (below surface)**
- **Possibility for online sampling of lead composition during operation of the facility**
- **Possibility for filtering of gas and liquid lead**



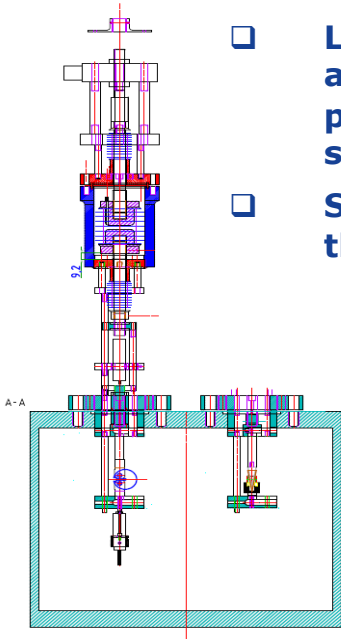
# Test sections for the LILLA facility

Test sections for the LILLA facility allow different types of tests:

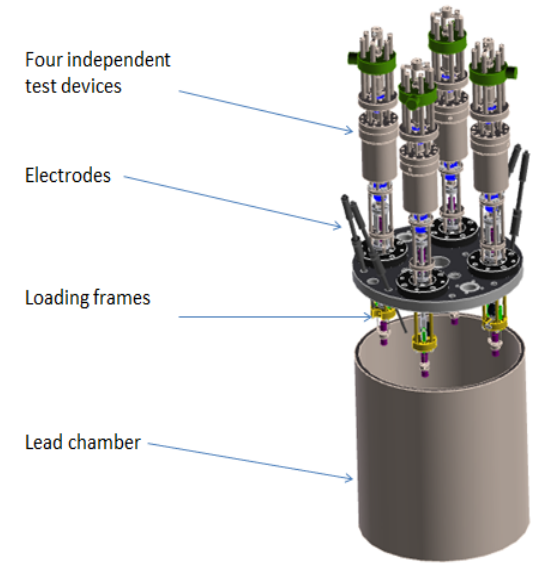
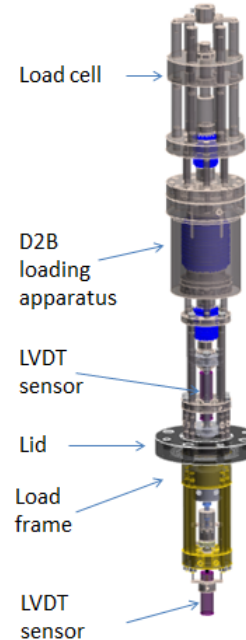
- Constant strain rate tensile (SSRT) tests
- Fracture Toughness Tests
- Crack Growth Rate Tests
- Small Punch and Segmented Cone Mandrel Tests

❑ **Load or displacement control is accomplished through unique pneumatic bellows-based system**

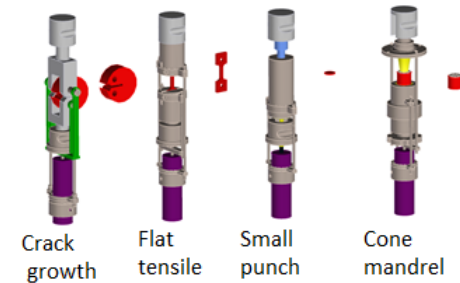
❑ **Strain feedback is accomplished through the LVDT sensor**



### Test section



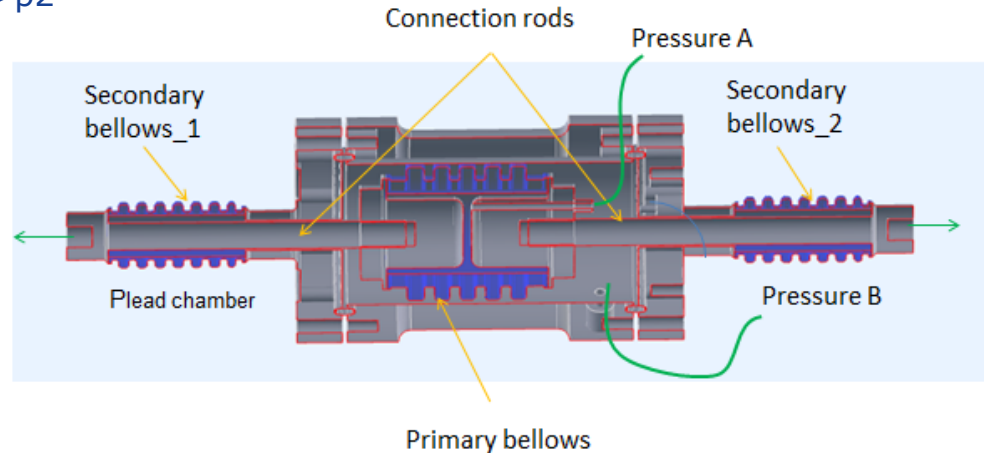
### Detachable Fixings





# The double2bellows loading apparatus

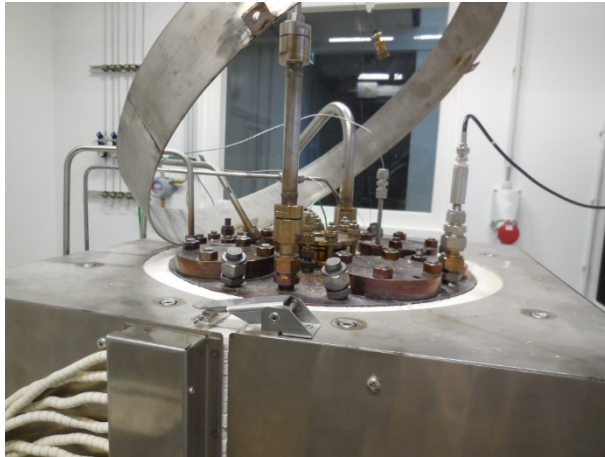
- Patented
- **The load in the test sections is generated by a new type of the pneumatically-powered double2bellows loading apparatus**
- **This device allows generating both tensile and compressive loads, which are measured by a commercial load sensor in a continuous way**
- Primary bellows pressure  $p_1$  (PLC controlled)
- Secondary bellows pressure  $p_2$  (PLC controlled)
- Push load ->  $p_1 < p_2$
- Pull load ->  $p_1 > p_2$



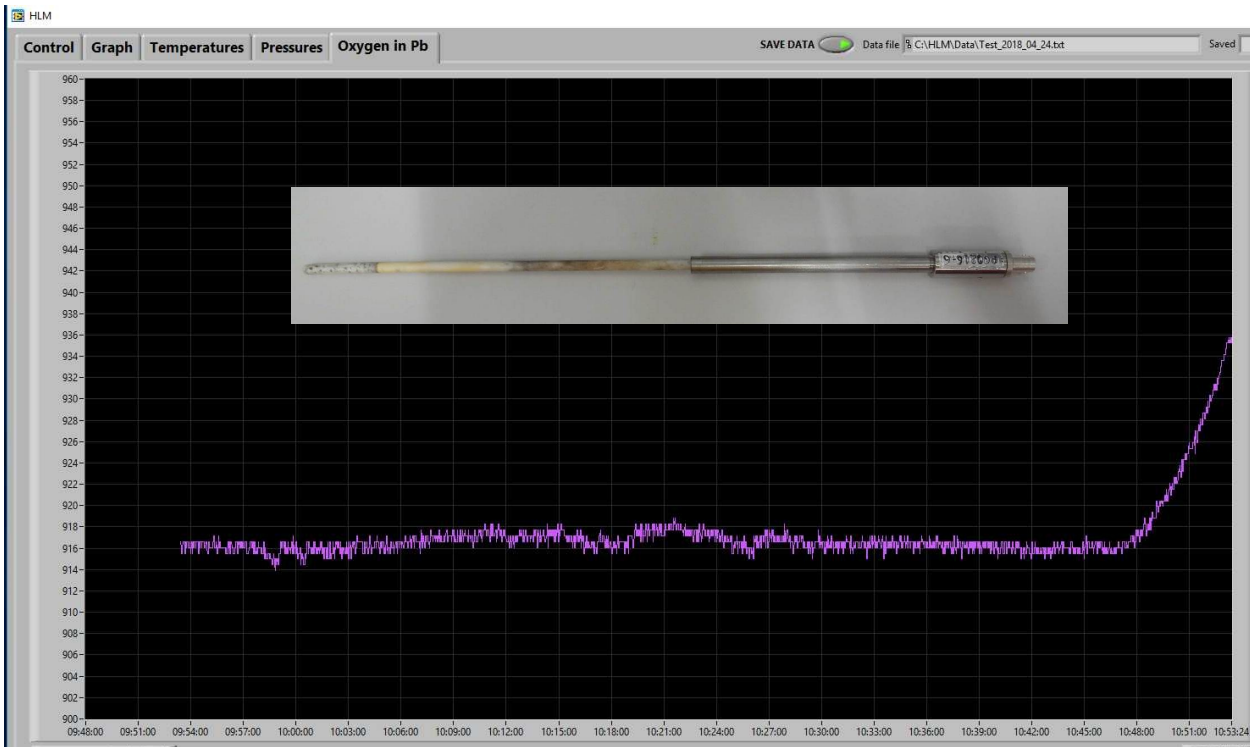
# Installation of Pt-air oxygen sensors



The oxygen control in molten lead is achieved by an appropriate admixture and delivery of Ar, Ar-H<sub>2</sub>, and air to the facility



- Oxygen sensors in lead (based on Pt-air reference system) control the amount of oxygen dissolved in molten lead
- Additionally, oxygen, hydrogen and humidity sensors control the gas chemistry



O<sub>2</sub> concentration  
in Pb at 450°C  
~10<sup>-5</sup> wt.%



This figure demonstrates, **the active control of the chemistry** in molten lead, where response of the Pt-air oxygen sensor measuring the amount of oxygen in lead is illustrated

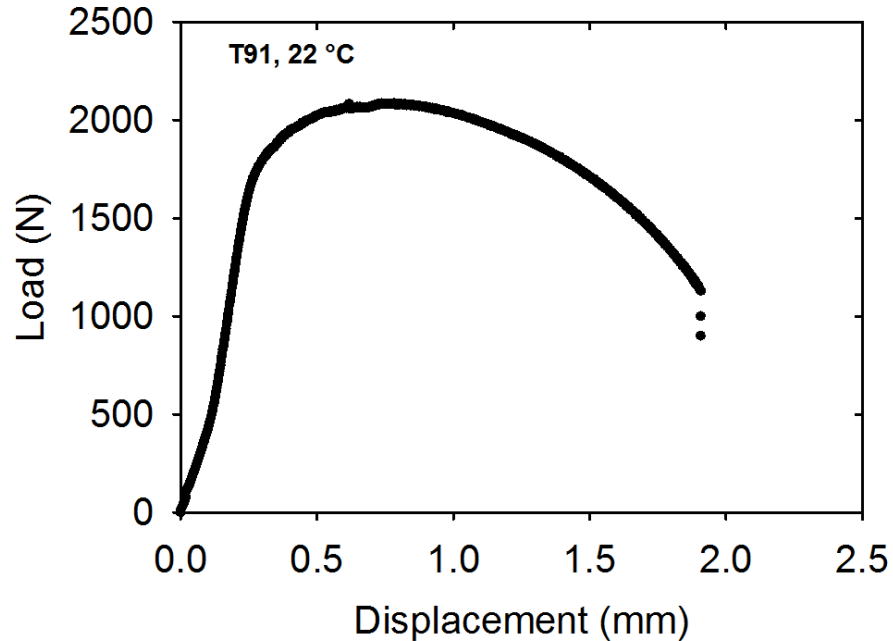
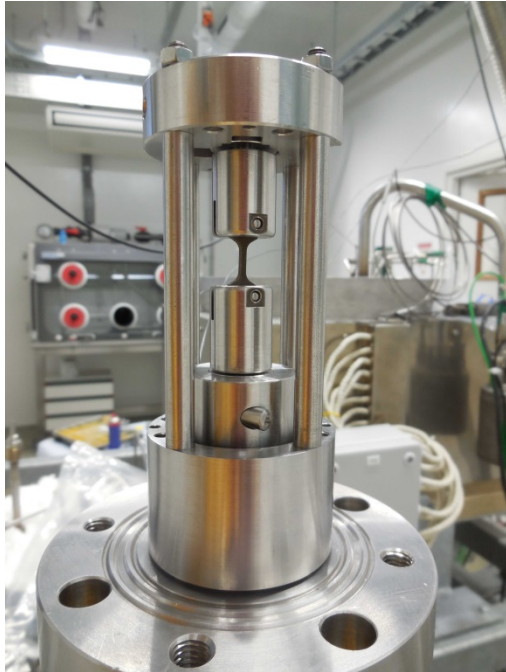
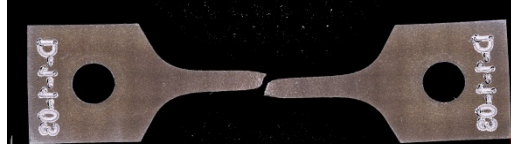
# LILLA commissioning tests

A series of commissioning and acceptance tests of the LILLA facility was conducted according to a **pre-defined programme to demonstrate that the facility operates in accordance with the technical requirements**

The commissioning tests included heat-up of the LILLA facility from cold to hot state (the target temperatures were 430°C for the dump tank and 550°C for the measuring tank, respectively). All **operating modes of the facility** were thoroughly tested, including:

- **Heating mode**, incl. vacuuming, facility flushing with Ar, lead melting in the dump tank, heating, gas / oxygen conditioning, and filling of the measuring tank with the conditioned molten lead
- **Nominal operating mode**, incl. maintenance of temperature, gas / oxygen conditions, as well as lead in the measuring tank
- **Cooling mode**, incl. cooling, draining and lead freezing in the dump tank;
- **Stand-by mode**, incl. Ar injection and control in the tanks
- **Purification mode**, incl. Ar/H<sub>2</sub> gas injection and H<sub>2</sub>, O<sub>2</sub>, and humidity control
- **Lead filtration**

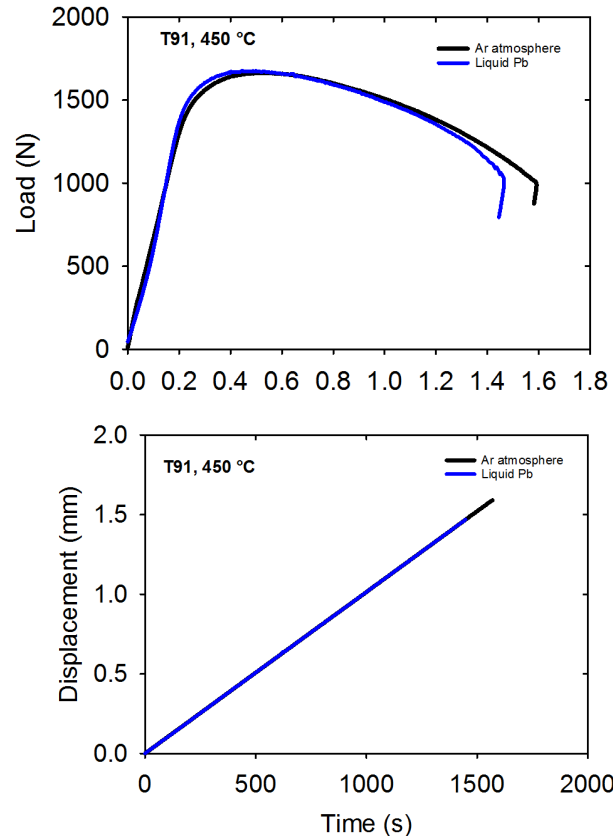
# First SSRT tests at room temperature



As a part of the commissioning tests, several tensile tests of T91 material were also performed in air at room temperature (illustrated on this figure) as well as in high temperature argon at 420°C and 450°C

# First SSRT tests at elevated temperature in Ar and Liquid lead

- Load as a function of the displacement was measured continuously during these tests and the full displacement curves for the tested T91 material were achieved
- The accuracy of load generated during the tests was better than  $\pm 2$  N, **achieving thus a very good linearity of the displacement response**



# Testing of T91 ferritic-martensitic steel at oxygen depleted conditions (1/5)

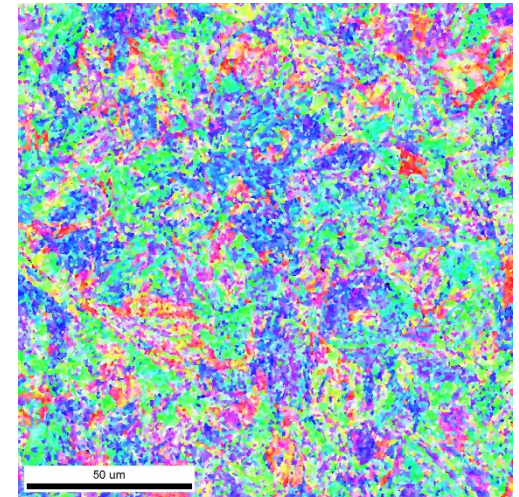
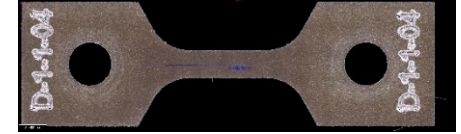
**Material: 9% chromium ferritic-martensitic steel T91 from EUROTRANS/DEMETRA and supplied by SCK•CEN;** the material was hot rolled into 15 mm thick sheet, heat treated 15 min at 1050°C and 45 min at 770°C

**Flat tensile specimens** with cross section of 1.5x2 mm<sup>2</sup> and **30 mm in length** were tested in **Ar and liquid lead**

**Specimens were wire cut and polished**

**Test conditions were:**

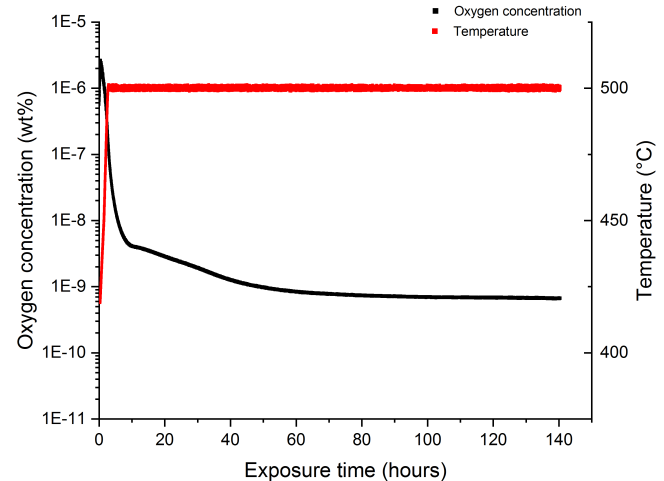
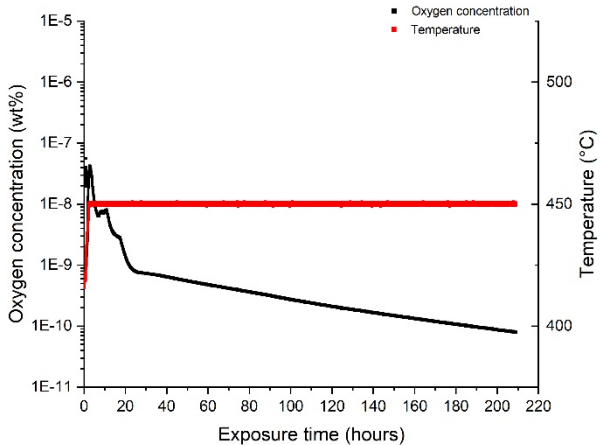
- Tensile testing up to rupture in the strain-controlled mode
- Temperature: 400°C
- Initial strain rate:  $1 \times 10^{-4} \text{ s}^{-1}$
- O<sub>2</sub> content:  $< 10^{-9} \text{ wt.}\%$  (oxygen depleted)
- Pre-exposure temperatures: 450°C or 500°C



# Testing of T91 ferritic-martensitic steel at oxygen depleted conditions (2/5)

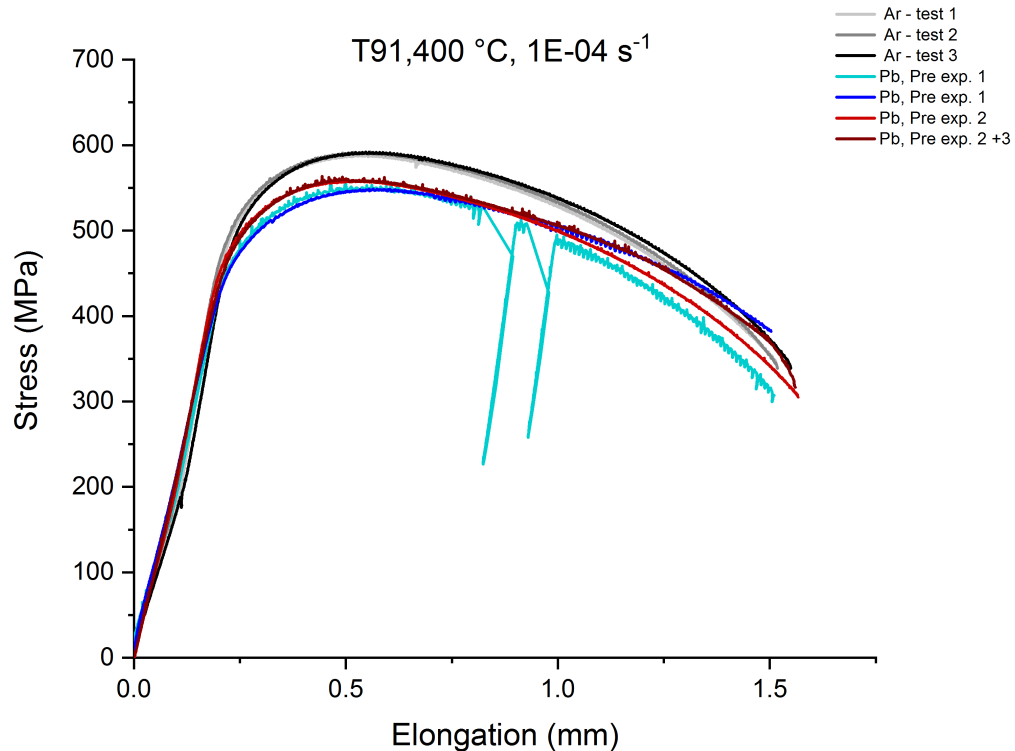
## Three pre-exposure conditions were used:

- **Pre exposure 1:** 180h in liquid Pb prior the test at 450°C, oxygen concentration  $\sim 10^{-9}$  wt.%
- **Pre-exposure 2:** 100h in liquid Pb prior the test at 500°C, oxygen concentration  $\sim 10^{-9}$  wt.%
- **Pre-exposure 3:** specimen kept 1000h in liquid Pb, 500°C, oxygen concentration  $< 10^{-11}$  wt.%, performed by SCK•CEN





# Testing of T91 ferritic-martensitic steel at oxygen depleted conditions (3/5)

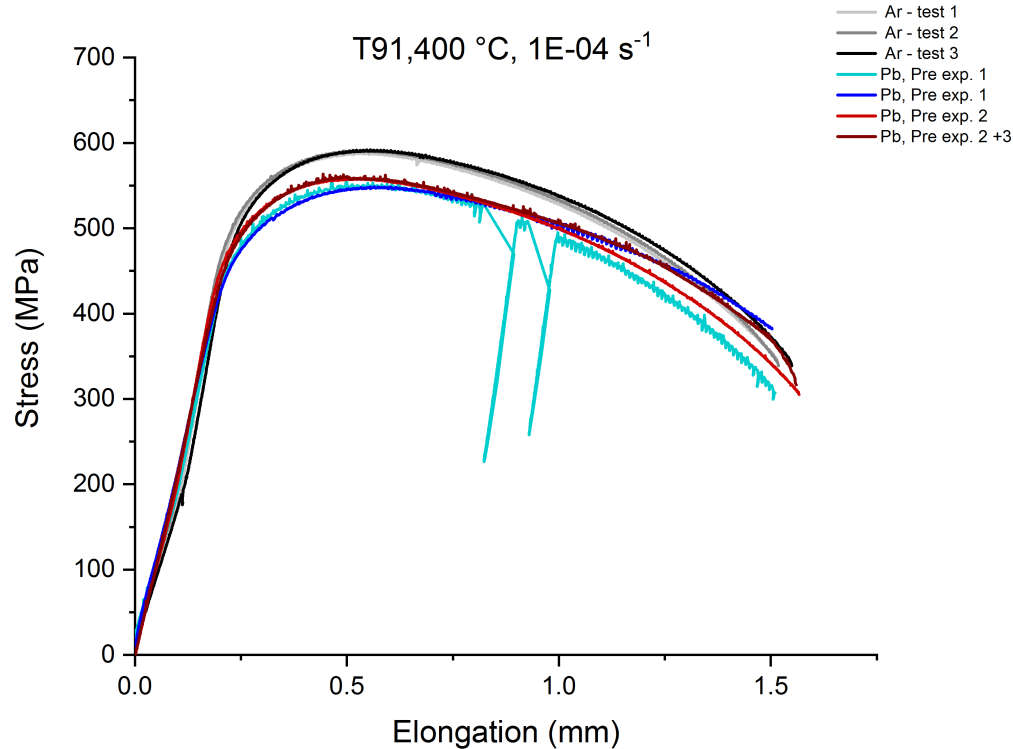


The **three tensile curves obtained in Ar** are displayed on top of each other which shows a **good repeatability**. The measured UTS (590 MPa) is in a good agreement with Van den Bosch et al. (2005).

**Four tests in oxygen-depleted liquid lead environment** (oxygen content <10<sup>-9</sup> wt.%) were successfully performed.

Two specimens were pre-exposed at **450°C** (pre-exposure 1) and one at **500°C** (pre-exposure 2). The fourth specimen was first pre-exposed 1000h at **500°C** in liquid Pb (pre-exposure 3) before introducing it into the LILLA facility, where it was additionally pre-exposed 100h at 500°C (pre-exposure 2).

# Testing of T91 ferritic-martensitic steel at oxygen depleted conditions (4/5)



One of the measured stress-strain curves exhibit two abrupt jumps in load. The cause of the jumps is not yet fully understood. The **observed behaviour could be material- and/or test control-related**, the former including **dynamic strain ageing**. The UTS for specimens tested in liquid lead was 30 MPa lower than in argon.

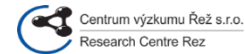
**There is no remarkable change in elongation at fracture; specimens tested in argon and in liquid lead environment** have the same ductility. This indicates that LME under the used test conditions did not occur.

# Testing of T91 ferritic-martensitic steel at oxygen depleted conditions (5/5)

- However, to **make a conclusion on susceptibility of T91 to LME in liquid lead further investigations need to be performed**
- It is necessary to verify that the used pre-exposures are effective to remove the protective oxide layer from the specimen surface
- The fracture surfaces of specimens tested in Pb are also currently under metallographic investigations to identify fracture modes and search for any sign of LME
- The previous studies in LBE showed that LME occurs in a certain range of temperatures combined with a range of strain rates. Therefore, **the temperature and strain rate may play an important role in lead, too.**
- After identifying suitable pre-exposure conditions, **SSRT tests will therefore be performed at different temperatures and with different strain rates in liquid lead**

# LILLA facility project stakeholders

- ❑ Support to **Member States** in safety assessment and licensing of the European (SNETP/ESNII) LFR demonstrator **ALFRED** and the European Pb/Bi-cooled Technology Pilot Plant **MYRRHA**
- ❑ Support to European Committee for Standardization **/CEN/**: extension of the **RCC-MRx Code** for the design of mechanical components of heavy liquid metal (Pb, Pb/Bi)-cooled nuclear reactors
- ❑ **EERA Joint Program on Nuclear Materials (EERA)**
- ❑ **Generation-IV International Forum (GIF)** LFR provisional System Steering Committee (SSC)
- ❑ **OECD/NEA** Expert Group on Liquid Metal Technology (EGLM)
- ❑ **GEMMA** (GEneration IV Materials MAaturity) **H2020 Project**

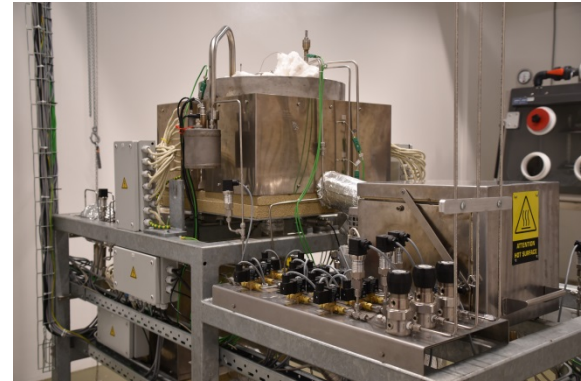
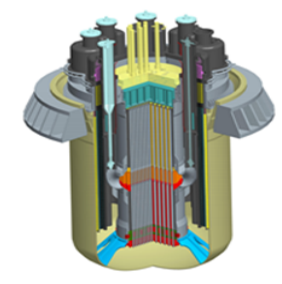
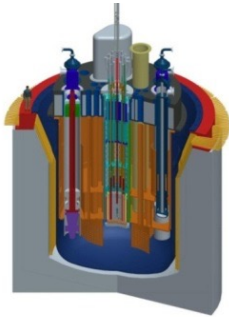


European  
Commission

# GEMMA: GEnIV Materials Maturity (2017-2021)

- **H2020 R&D collaborative project comprising 23 European and Korean partners, with a project budget of 6.6 M€**
- **Objective: qualification of structural materials and welded joints** for systems and components of ESNII Generation-IV demonstrators & prototypes foreseen in Europe: **ASTRID SFR, MYRRHA LFR, ALFRED LFR, ALLEGRO GFR**
- **The JRC contribution (51.4 person-months) to the project includes:**
  - coordination of WP2, incl. **testing of mechanical properties in air**, residual stress measurements in welds, and the **transformation of the generated experimental data to useful Design Rules in the RCC-MRx Design Code**
  - **in-situ mechanical tests of welded specimens in liquid lead (WP4)**
  - contribution to communication, dissemination, and **data management in MatDB**, in support to the **Horizon 2020 Open Data Pilot** allowing for verification and repeatability of project results (WP6)





**Thank you for your attention!**

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