Profile LFR-47

RACHELE

ITALY

GENERAL INFORMATION

NAME OF THE FACILITY	Lead-Alloy Chemistry Laboratory
ACRONYM	RACHELE (Reactions and Advanced CHEmistry for LEad)
COOLANT(S) OF THE FACILITY	Lead/Lead-Bismuth Eutectic
LOCATION (address):	Italian National Agency for New Technologies, Energy and Sustainable Economic Development, C.R. ENEA Brasimone, Italy
OPERATOR	ENEA
CONTACT PERSON (name, address, institute, function, telephone, email):	Dr. Alessandro Gessi, ENEA UTIS-CPM C.R. Brasimone 40032 Camugnano (BO), Head of Characterization and Material Tests Laboratory, Tel. +39 0534 801 273, alessandro.gessi@enea.it

STATUS OF THE FACILITY	In operation
Start of operation (date):	January 2014

MAIN RESEARCH FIELD(S)

- ☐ Zero power facility for V&V and licensing purposes
- ☐ Design Basis Accidents (DBA) and Design Extended Conditions (DEC)
- ☐ Thermal-hydraulics
- ☒ Coolant chemistry
- ☒ Materials
- ☒ Systems and components
- ☒ Instrumentation & ISI&R

TECHNICAL DESCRIPTION

Description of the facility

The lead-alloy chemistry laboratory in ENEA was born with the aim of studying the chemical behavior of liquid alloys and their interaction with the most promising structural materials in the working conditions of interest for the Generation IV Nuclear Fast and ADS Reactors cooled with liquid lead alloys.

To perform chemical compatibility tests of structural materials in liquid lead alloys, the laboratory has 10 experimental capsules, designed and manufactured by the KIT Institute (*Karlsruhe Institute of Technology,* Germany), and a muffle furnace.

The experimental capsules are used to perform corrosion tests of structural steels in liquid lead environment both in conditions of oxygen saturation (high oxygen content) and in conditions below the oxygen saturation value (low oxygen content). The conditions of high and low oxygen content in the liquid lead are obtained changing the type of cover gas (Argon for tests at high oxygen content and Ar/H_2 mixture for tests at low oxygen content).
Fig.1 a shows a capsule with its components. It consists of a cylindrical stainless steel can that is heated on the outer surface by means of a band heater (heating rate 2-3°C/min). An alumina crucible placed within the cylinder acts as a liquid lead container and avoids contact between the liquid metal and the cylinder steel wall. The capsule lid has holes for the insertion in the lead bath of a sample, a thermocouple, an alumina tube to introduce gas (oxygen-rich or -depleted gas), an electrochemical sensor for the oxygen determination in liquid lead and an molybdenum electrode. The thermal insulation is ensured by means of mineral wool and external aluminum half-shells around the cylinder. For more details on the capsules and their operation see reference [1].
The oxygen sensor (Fig.1b) is based on the Pt/air system as reference electrode and YSZ (Yttria Stabilized Zirconia) as selective electrolyte. The Mo electrode closes the electric connection when a metal sample is not present within the capsule.
The cover gas enters the capsules by means of a gas line connected to a cylinder. In addition, there is a bypass inserted in the gas line which allows to change cylinder and so the type of incoming gas (Ar or Ar/H_2). On the line and before each capsule there is a flowmeter for the control of flow rate of the incoming gas.

The muffle furnace is used to perform corrosion tests in the environment of oxygen-saturated liquid lead (high oxygen content). The muffle (L15/13 P330, Nabertherm) was equipped with an input for the cover gas (Argon) and for a thermocouple to monitor the temperature of the liquid lead during the tests. In the cover gas line and before the muffle there is a flowmeter for the control of the flow rate.
Corrosion tests are performed inside alumina crucibles and the samples are suspended in the liquid lead through a support. The test cycle is selected by setting the heating ramp (heating rate 2-3 °C/min, temperature and holding time).

The chemical control and monitoring of the cover gas in and out from lead baths are carried out using an oxygen gas analyzer and a hygrometer. The gas analyzer (E705-LOW, FER-Strumenti) (Fig.2a) allows the determination of the oxygen content as an impurity in the argon gas out coming from the cylinder. The hygrometer (Cermet II, Michell Instruments)(Fig.2b) is placed on the gas line coming out from the capsules and allows to monitor the water production during the tests using Ar/H_2 mixture.

Acceptance of radioactive material

No

Scheme/diagram

No

3D drawing/photo

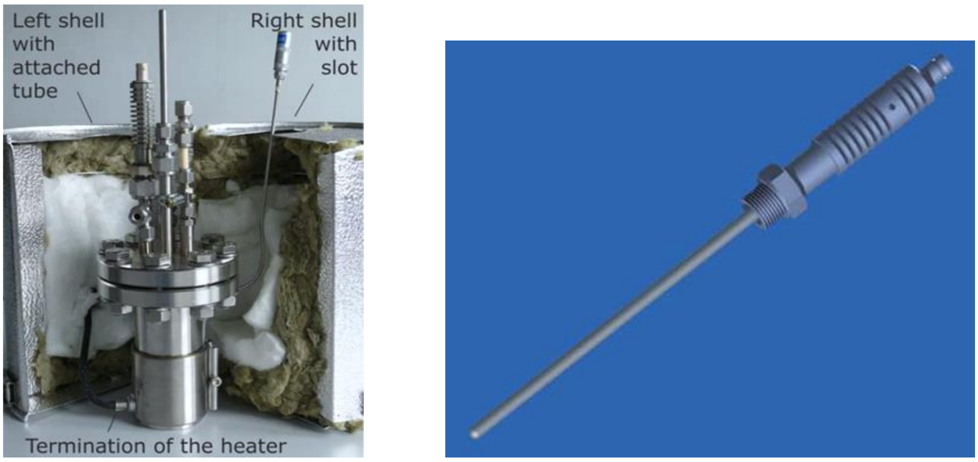

FIG. 1. Images from Ref. [1] showing a) KIT experimental capsule without insulation components and b) KIT oxygen sensor for corrosion tests in liquid lead at high and low oxygen content.

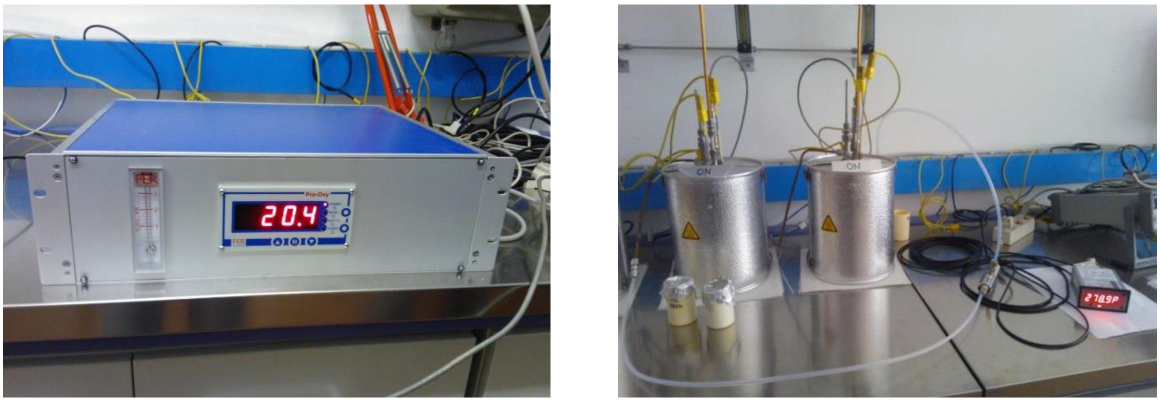

FIG. 2. Instruments for chemical control in gas, a) oxygen gas analyzer during calibration in air and b) hygrometer for the monitoring of water production in the cover gas out coming from KIT devices.

Parameters table

Coolant inventory	Up to 10 small alumina crucibles filled with LM in each furnace (furnace in total with 2 different oxygen partial pressures each)
Power	1 kW for each capsule
Test sections: 10	
TS #1-10	*Characteristic dimensions* cylindrical specimen of dimensions Ø6×50 mm, with an internal M3 screw thread
	Static/dynamic experiment static
	Temperature range in the test section (Delta T) Uniform, up to 550°C
	Operating pressure and design pressure ambient
	Flow range (mass, velocity, etc.)

Coolant chemistry measurement and control (active or not, measured parameters)	Oxygen control via gas phase Gas phase oxygen and humidity is controlled Oxygen sensor for the liquid metal
Instrumentation	Temperature Gas phase oxygen LM phase oxygen humidity

COMPLETED EXPERIMENTAL CAMPAIGNS: MAIN RESULTS AND ACHIEVEMENTS

Up to now the development of the laboratory has been completed, in particular for what concerns the electric connections structure, the gas lines and the data acquisition system. Finally, preliminary tests were carried out in order to optimize the samples exposure procedure. The experimental capsule were started and the operation of the oxygen sensors was verified, comparing the values of potential output measured with the theoretical values at different temperatures. For this purpose, oxygen-saturated liquid lead (obtained by air bubbling) within the capsules was brought to 550°C and then subjected to a cooling/heating cycle between 550°C and 450°C, during which the sensors potential outputs were acquired at different temperatures and compared with the theoretical ones. In addition, during the preliminary corrosion tests it has been found that the use of cover gas with not suitable purity (high oxygen content as impurity) leads to excessive oxidation of the molten lead (also using a hydrogen-containing cover gas), affecting the tests validity.

In the frame of the European program MATTER (Materials Testing and Rules) three types of materials were tested in the capsules KIT: T91 ferritic/martensitic steel and 316L and 15-15Ti(Si) austenitic steels. The three steels were exposed in oxygen-saturated LBE at 550°C. The T91 and 316L were exposed for 1500 hours, while the 15-15Ti(Si) for 4000 hours. In addition, the exposure test of T91 was used as a Round Robin Test to check with other European laboratories the reproducibility of the exposure method with KIT experimental capsules.

PLANNED EXPERIMENTS (including time schedule)

Since the beginning of 2014 started the activity concerning the study of the corrosion behavior in molten lead of steels for structural applications in Generation IV Nuclear Fast Reactors. This activity is mainly focused on the study of 15-15Ti(Si) and 316L austenitic stainless steels and T91 ferritic/martensitic steel in liquid pure lead. The steels will be tested in the muffle and in the experimental capsules up to 8000 hours of exposure in conditions of high and low oxygen content in liquid lead. Under the same conditions will also be tested the same types of steels but protected with different coatings (e.g. TiN, Ta, and Al-based coatings). These tests will be performed with the aim to evaluate both the effectiveness of the coating as a corrosion barrier and the goodness of the manufacturing process of the coating itself.

Presently corrosion tests of 15-15-Ti(Si) samples bare and coated with TiN are ongoing at high oxygen content. In addition, in order to start as soon as possible tests at low oxygen content, conditioning tests of the liquid lead baths are ongoing within KIT capsules. In support of these activities, the design of a "Multipurpose Facility" is currently in progress in order to perform further tests related to the topic of the lead chemistry control. Some of these tests will focus on the study of the effectiveness of the oxygen getters (eg, Ti or Ta) in the liquid metal and the study of the accuracy and reliability of sensors in the oxygen monitoring in the cover gas and liquid lead. Other tests will be focused on assessing the effect of corrosion inhibitors (Ti or Zr alloyed in the samples) and the ability to inhibit corrosion by means of the "solute/solvent" concept (adding in liquid lead one of the alloy elements of the structural steel, e.g. Ni, in order to limit its dissolution).

TRAINING ACTIVITIES

Training activities can be agreed with ENEA C.R. Brasimone in the frame of these experimental campaigns under the supervision of ENEA qualified staff.

REFERENCES (*specification of availability and language*)

1. Experimental Capsule for exposure of Steels to Oxygen-containing Lead Alloys: Technical documentation and user instructions, Corrosion Department of Institute for Applied Materials Material Process Technology (KIT), January 2013.