

DEVELOPMENT OF A HOT CELL TEST SET-UP FOR LIQUID METAL EMBRITTLEMENT (LME) STUDIES IN LEAD-LITHIUM AND LEAD-BISMUTH

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ABSTRACT

Liquid Metal Embrittlement (LME) is defined as the brittle fracture (loss of ductility) of usually ductile materials in the presence of a liquid metal. The sensitivity to LME is likely to increase with irradiation hardening as localised stresses can promote the aggressive action of a liquid metal. To investigate the mechanical response of irradiated materials in contact with a liquid metal, an instrumented hot cell is being developed. The testing machine installed inside will allow mechanical testing of active materials in liquid lead lithium and liquid lead bismuth under well controlled chemistry conditions. Typical mechanical tests that can be carried out are SSRT, constant load and rising load tests at temperatures from 150°C to 500°C. A special requirement of the hot cell set-up is that the irradiated samples can be retrieved from an irradiation rig without any supplementary damage. Therefore a dedicated dismantling set-up has been designed as well.

The LME test set-up will consist of three vessels; besides the autoclave and loading unit two more vessels are used to prepare a test. The first vessel (melting tank) is only used to melt the lead mixture for the first time. Impurities and the always present oxide layer on top of the liquid metal can be removed. The second vessel (dump tank) is used to condition the liquid metal with a gas, for example hydrogen. After the conditioning, the liquid metal mixture is ready to be used for testing. Part of this set-up consists of a vacuum pump and an argon gas supply, which are used to prevent the contact of liquid metal with air and humidity. Tensile tests can be performed with a loading unit allowing strain rates in the range of 10^{-3} to 10^{-7} s⁻¹ with a maximum load of 20 kN

In this presentation we will focus on the technical design of the test set-up and the special features that have been developed to allow testing in a hot cell environment.

KEYWORDS **LME, SSRT, HOT CELL, LEAD-LITHIUM, LEAD-BISMUTH.**

1. INTRODUCTION

Liquid Metal Embrittlement (LME) is defined as the brittle fracture (loss of ductility) of usually ductile materials in the presence of liquid metal. The sensitivity to LME is likely to increase with irradiation hardening [1,2]. Therefore a hot cell Slow Strain Rate Test set-up will be developed, which allows testing of active materials in liquid metal like lead-lithium and lead-bismuth. A special requirement is the capability to test α -contaminated tensile specimens, which results from the irradiation of tensile specimens in lead-bismuth.

The test set-up will be designed in such way that tests can be performed in both lead-lithium (related to the fusion research) as in lead-bismuth (related to the MYRRHA program). As lead-lithium is a reducing system and so easier to be removed from the interior of the test set-up, tests will first be carried out in lead-lithium and then in lead-bismuth. The conditioning systems are slightly different, but will both be integrated in the design.

The main characterization of LME is the very high crack growth rate, being in the range of one centimetre to several meters per second [2]. Therefore the main emphasis of LME investigation has been on initiation assuming that when LME starts, the construction has reached end of life. Due to these very high crack growth rates, little or no attention has been given to fracture mechanics type of testing. In case of smaller crack growth rates a fracture mechanics approach would certainly increase our understanding of the phenomena [3,5,6]. It has also been stated that local stress concentrations or stress raisers might be a requirement for LME to start/run [2-4]. So when defining the type of mechanical testing to be carried out, we didn't only look at initiation tests but also at fracture mechanics testing. The following tests are considered to be important.

- Slow Strain Rate Tests (SSRT)
- Constant load
- Rising load
- Crack growth rate (fracture mechanics)

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2. TEST SET-UP FOR LME STUDIES

2.1. MECHANICAL TESTING

All tests will be carried out under static conditions. The test set-up will consist of three vessels to melt and condition the appropriate lead melt, which will be exposed to the metal samples. Part of this set-up consists of a vacuum pump and an argon gas supply, which are used to prevent the contact of the lead with air and humidity. Mechanical tests can be carried out at temperatures from 150°C to 500°C. Slow strain rate tensile tests will be performed with a loading unit allowing strain rates in the range of 10^{-3} to 10^{-7} s^{-1} with a maximum load of 20 kN. Figure 1 shows the Slow Strain Rate Test set-up. The autoclave vessel is transparent to enable to view the inside construction.

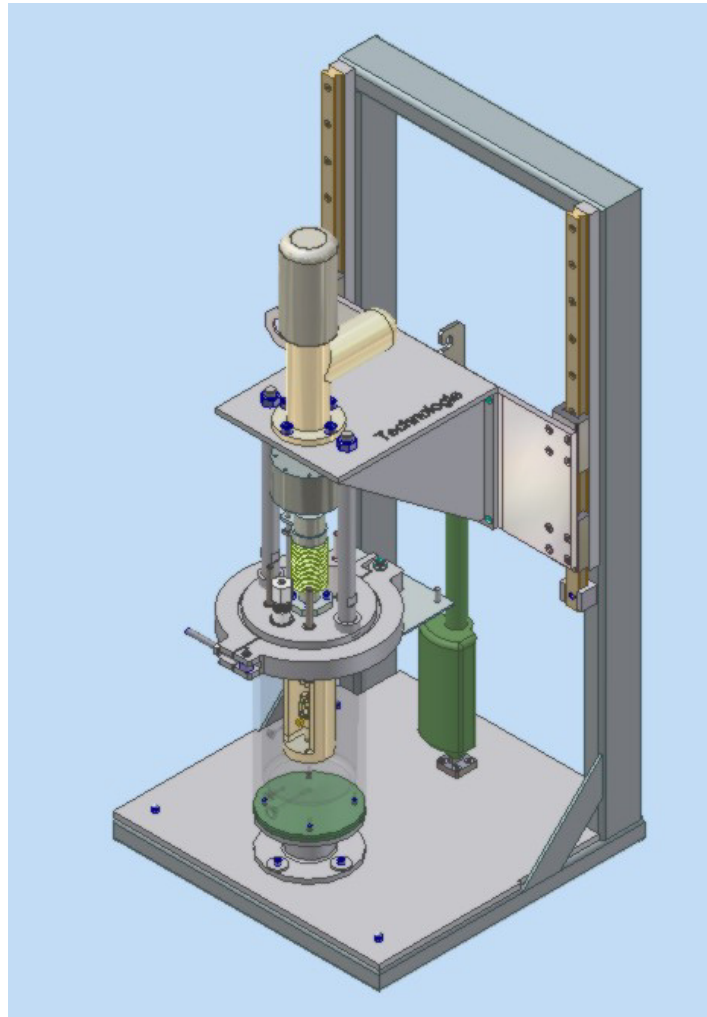


Figure 1. Overview of the mechanical test set-up

The mechanical set-up consists of a loading actuator, which can provide the required displacements to perform a slow strain rate tests and a load cell to measure the applied load. The tensile specimen is positioned in a test rig fixed to the autoclave lid, with a feed through for the pull rod. The whole test assembly can be immersed in an autoclave filled with the appropriate liquid metal. A remote controlled lifting system is used to move the test rig up and down. The autoclave is connected to a conditioning system (see paragraph 2.4 "Liquid metal conditioning system"), which is used to prepare the liquid metal melt. The specifications of the test set-up are summarized in table 1.

Table 1. Specifications LME test set-up

Temperature	Max 500°C
Pressure	Max 4 Bar
Maximum load	20 kN
Displacement rates	$9 \cdot 10^{-2}$ to $3 \cdot 10^{-6}$ mm.s ⁻¹
Strain rates (gage length 10 mm)	$9 \cdot 10^{-3}$ to $3 \cdot 10^{-7}$ s ⁻¹
Maximum displacement	30 mm
Specimens to be tested	Tensile specimen
Number of autoclaves and loading units	1
Autoclave volume	3.6 L
Autoclave materials	316L
Material conditioning system	316L
Conditioning gasses	Hydrogen, Argon

2.2. SPECIAL FEATURES FOR TESTING IN A LIQUID METAL ENVIRONMENT

The mechanical test set-up contains some special features which are necessary when tests are performed in a liquid metal environment. A metal bellow feed through is constructed for the transfer of the pull rod through the autoclave cover. When the pull rod moves up and down, the bellow is expanded or compressed.

Temperature control is necessary for the autoclave, the connecting tubes and the valves. Before a valve is opened or closed, it must be sure that the temperature is high enough so that the lead lithium mixture is completely liquid. Otherwise the valve will be damaged. For the autoclave a double heating system is used, to avoid solidification in case of heater break down.

Special valves will be used for the handling of the liquid metal. These valves are of the diaphragm type, as they are less sensitive to failure.

Vapor traps are placed at all gas inlets and outlets to avoid damage of the pressure meters and gas valves. Lead-lithium or lead-bismuth vapor will condensate in the vapor traps and so not in the valves or pressure meters. A staple of small plates creates a large surface area where the metal vapors can condensate.

2.3. SPECIAL FEATURES FOR A HOT-CELL ENVIRONMENT

A special closing clamp to fix the autoclave cover to the autoclave has been designed that can be used by a manipulator. This closing clamp is visible on figure 1. This closing clamp is fixed to the autoclave by a horizontal plate at the back side of the autoclave. With the manipulators both parts of the clamp can be put around the autoclave flange and autoclave cover. The bolt is moved to the clamp end and the nut is turned to close the clamp. For the small operating pressure (maximum 3 bars) this closing system is leak tight. A C-ring shaped sealing is used of Inconel 600. This sealing is not in direct contact with the liquid metal. (Ni-alloy does dissolve in liquid PbLi).

The autoclave cover can be removed from the autoclave with a lifting system composed of a linear actuator. This actuator is fully remote controlled and therefore particular suitable for application in a hot cell. In case of malfunctioning of this lifting system, the actuator can be disconnected from the autoclave cover by removing the connector pin. A tackle can then be used to open the autoclave.

The autoclave cover can be opened and closed by remote control. Also the closing clamp can be closed from a distance by using a manipulator. To put a tensile specimen in the tensile test rig, the manipulators are also necessary. Two nuts are screwed to the thread of the tensile specimen to be tested. Two conic shaped rings are put under the nuts to centrally fix the tensile specimen in the test rig. The tensile specimen with nuts and rings is placed in the special holders, which have an opening in front.

2.4. LIQUID METAL CONDITIONING SYSTEM

Figure 2 shows a simplified flow sheet of the test set-up for LME testing.

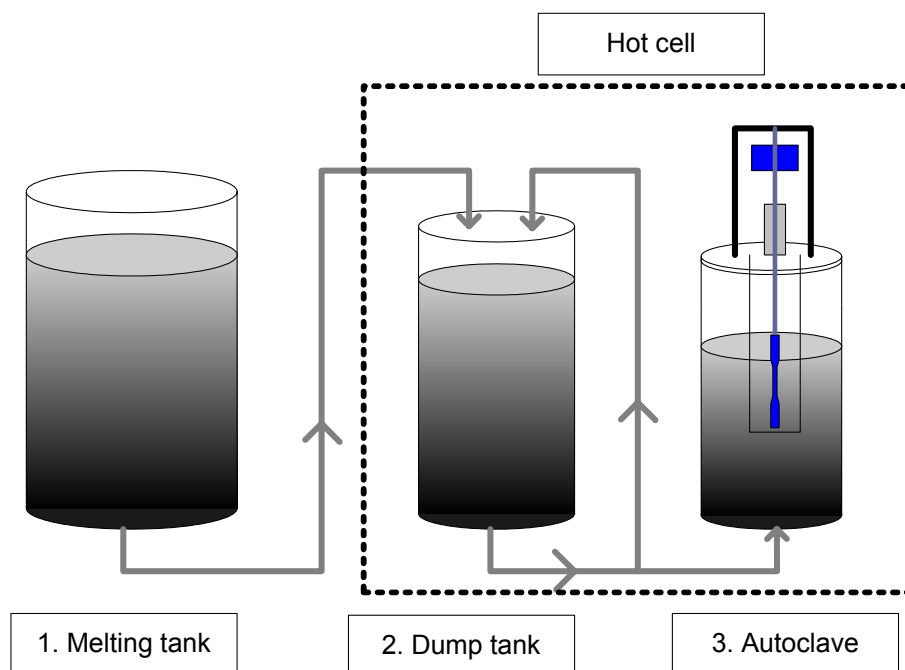


Figure 2. Simplified flow sheet of the LME Slow Strain Rate Test set-up; (1) melting tank, (2) dump tank and (3) autoclave

Besides the autoclave and loading unit (3) two more vessels are used to prepare a test. These two vessels are used to condition the liquid metal. The first vessel (melting tank) is only used to melt the lead for the first time. Impurities and the always present oxide layer on top of the melted liquid can be removed. The second vessel (dump tank) is used to condition the lead melt with a gas (for example hydrogen). After the conditioning, the liquid metal mixture is ready to be used for testing.

To prevent damage of the individual valves vapour traps are present all over the set-up. These vapour traps should prevent the condensation of Pb vapour in the valves, thereby ruining their proper working. Also each valve will be connected to a thermocouple to prevent opening or closing of a valve, when the temperature is too low. The whole set-up will be designed for a test temperature up to 550°C for tests in lead-lithium and 450°C for tests in lead-bismuth. This will allow testing at higher temperatures than 300°C, but also prevents problems with the local solidification of lead. In case the melt composition deviates locally from the eutectic composition the melting temperature will be (much) higher.

A typical test is then performed as follows. The first vessel (melting tank) is filled with solid particles of lead. Then the melting tank is heated to melt the lead. Melting can be performed under argon atmosphere. Then the melted lead is transported to the second vessel (dump tank) leaving the oxides formed during melting in the first vessel. The vacuum pump is used to move the liquid metal from the vessel (1) to vessel (2). From the second vessel, the lead melt can be transported to the third vessel in the same way. A tensile specimen will be positioned in the tensile rig. This rig is present in the third vessel. Then the third vessel is filled with the melted lead and a slow strain rate tensile test can be carried out. When this tensile test is finished, the lead melt is brought back to the second vessel. A new test campaign can be started using the lead melt in vessel two. Only when the lead melt is too much contaminated it will be replaced. The lead melt will be removed from vessel 2 in small portions (max 5 kilo = maximum of the manipulators) into small containers suitable for (temporarily) storage of the lead as waste. From the melting tank through a tube penetrating the hot cell wall, the dump tank will be refilled.

3. DISMANTLING OF IRRADIATION CAPSULES AND STORAGE OF THE WASTE

Tensile specimens will be irradiated in the BR2 reactor in capsules filled with lead-bismuth. This will cause contamination of the samples with polonium. These capsules have to be dismantled in the hot cell to retrieve the tensile specimens for testing and to separate the contaminated lead-bismuth as waste. In addition the lead-lithium and lead-bismuth melt will be contaminated after a certain period of time due to general corrosion of the specimens. Then this lead melt should be removed from the dump tank in small portions and be treated/conditioned in such way that it can be stored as waste.

A special dismantling set-up has been designed and developed that can dismantle an irradiation capsule filled with tensile specimens and lead-bismuth (Figure 3). A typical size of such an irradiation capsule is 10 mm in diameter and 500 mm in length. This dismantling set-up can carry out the following actions:

Cutting, the irradiation capsule is cut at the top and the bottom (figure 4).

Melting, the lead-bismuth is melted from the capsules without removing the samples (figure 5).

Removing tensile specimens, the tensile specimens are removed after the lead-bismuth has been removed, without interchanging them. From each specimen should be unequivocally known its original position in the irradiation rig. (figure 6).

Cleaning, the specimens should be cleaned afterwards in hot oil with an ultrasonic probe to remove the last lead-bismuth that is stuck to the samples.

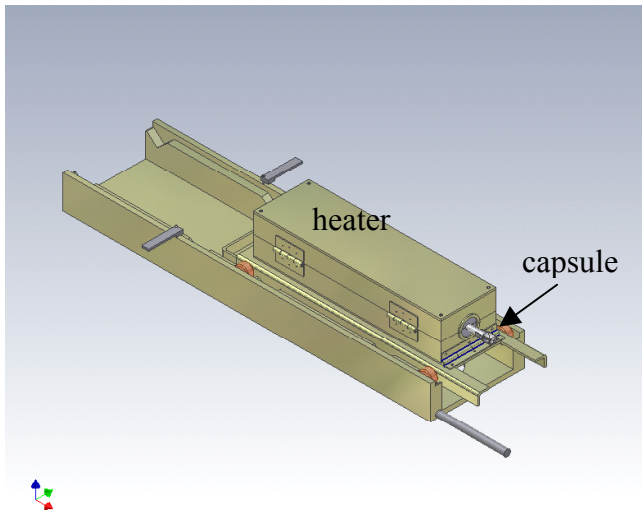


Figure 3. Dismantling set-up

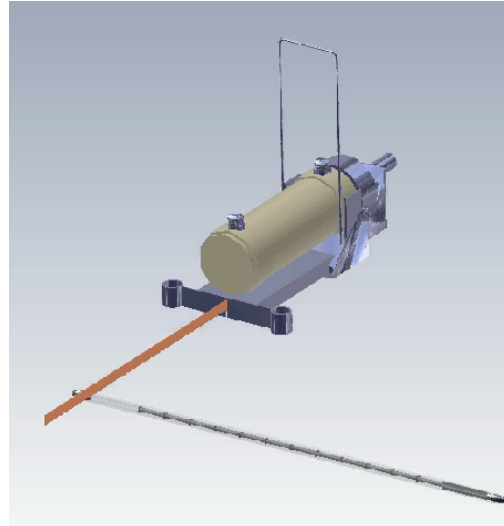


Figure 4. Cutting the capsule

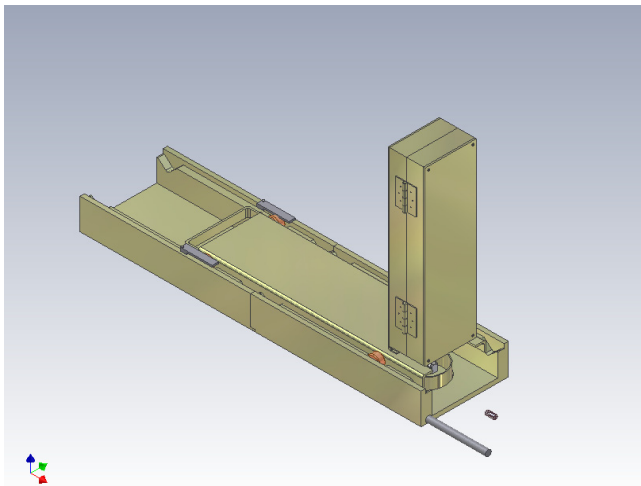


Figure 5. Melting the lead-bismuth in the capsule

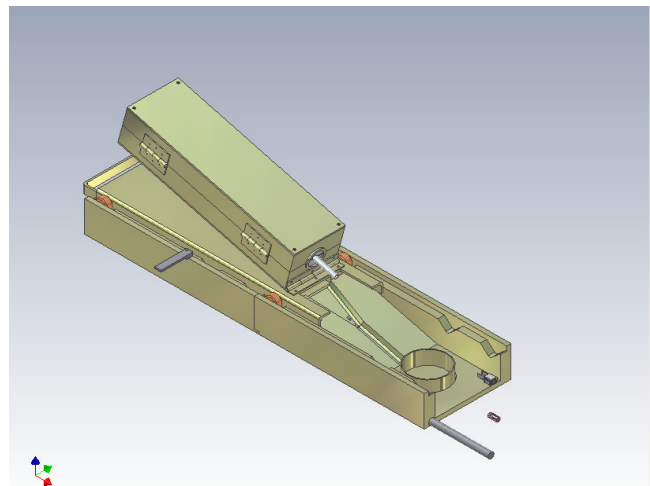


Figure 6 Removing the samples

The contaminated lead-lithium and lead-bismuth has to be stored in the hot cell. A simple but adequate storage system has been designed. It consists of small containers (maximum 5 kilo) that can be placed in cylinder shaped spaces that are submerged in the hot cell floor. These spaces are surrounded by lead blocks to shield for radiation. On top are lead plugs with covers of magnetic materials (ferritic stainless steel) that can be removed with a magnet. Figure 7 shows a picture of the storage facility in the hot cell and a schematic of the storage principle.

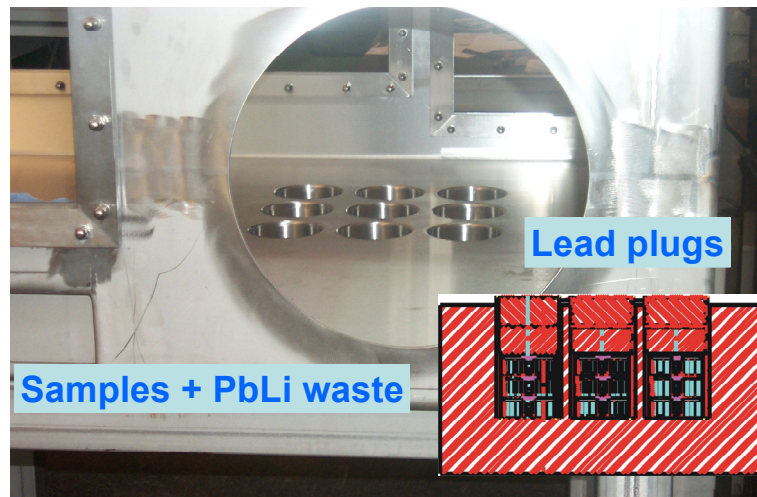


Figure 7. Storage of lead-bismuth radioactive waste

4. CONCLUSIONS

A hot cell set-up for Liquid Metal Embrittlement (LME) studies in lead lithium and lead bismuth is being developed with the following characteristics:

Irradiated samples can be retrieved from the irradiation rig without any supplementary damage.

Tests can be performed under well controlled liquid metal chemistry.

SSRT, constant load and rising load tests can be carried out at temperatures from 150°C to 500°C.

5. REFERENCES

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