STRESS-STRAIN MEASUREMENT IN HEAVY LIQUID METALS

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The mechanical testing in heavy liquid metals is performed in order to obtain information about the influence of the liquid metal on Generation IV candidate construction materials. For testing in heavy liquid metal environment, it is required to test comparably with standards of mechanical testing in air at high temperatures. The paper summarizes the approach to the measurement of mechanical properties in such environment. Potential issues of force and deformation measurement are analyzed on examples of specific use in the project aimed on static fracture toughness testing in liquid lead. Technical solutions (e.g. extensometer and steel bellows) are discussed and suggested based on previous experimental experience.

KEYWORDS

Heavy liquid metal, strain, extensometer, mechanical properties, testing cell.

1 INTRODUCTION

Heavy Liquid Metal (HLM) environment is perspective cooling medium in primary circuit of Generation IV nuclear power plants (NPP). The European effort in this field is focused on Pb and lead PbBi-eutectic (LBE), particularly in construction of Multi-purpose hYbrid Research Reactor for High-tech Applications (MYRRHA, Belgium) using LBE-cooling system and Advanced Lead Fast Reactor European Demonstrator (ALFRED, Romania) using Pb-cooling system [Alemberti 2020]. The project Generation IV Materials Maturity (GEMMA) is finishing the general objective to qualify and codify the selected structural materials (for fuel cladding and, in some cases, for the main vessel and the internals) for the construction of Generation IV reactors, as envisaged within the European Sustainable Nuclear Industrial Initiative (ESNII). Centrum vyzkumu Rez (CVR) performed fracture toughness testing in liquid lead on preselected materials in frame of GEMMA project.

HLM environment is not a standard environment for test machine manufacturers, thus an appropriate testing equipment is individually developed [Chocholousek 2016], specifically the cell for the specimen’s exposition. The testing cell has to fulfill necessary requirements for temperature, which is preferably aimed on interval from 550 to 825 K, depending on liquid metal and its designed operating temperature.

The projects focused on material innovation, energy industry included, work in many cases with insufficient amount of experimental material [Spirit 2016], whether it is due to the material preparation difficulty (e.g. surveillance program for NPP) or costs minimalization. That leads to specimen miniaturization [Dzugan 2017], its influence on testing [Rund 2015] and issues connected to measurement of small deformations.

The small specimens testing requires additional adjustments on HLM testing device used in CVR, because certain technical solutions are not applicable on this case, as will be discussed below.

2 HLM CELL

HLM cell, see Fig. 1, is a key part of testing in HLM. It is situated in the suitable testing machine according to planned type of tests. In the frame of GEMMA project, an electromechanical creep testing machine was used due to its capability to perform slow strain rate tests (SSRT) or the cycling at low frequencies (up to 1 s^{-1}). The cycling capability was used for fracture toughness evaluation with regard to ASTM standards. HLM cell is mechanically connected to the testing machine, however, its software for temperature and oxygen amount regulation is independent. Maximal operational temperature in HLM (Pb, LBE) cell is up to 825 K.

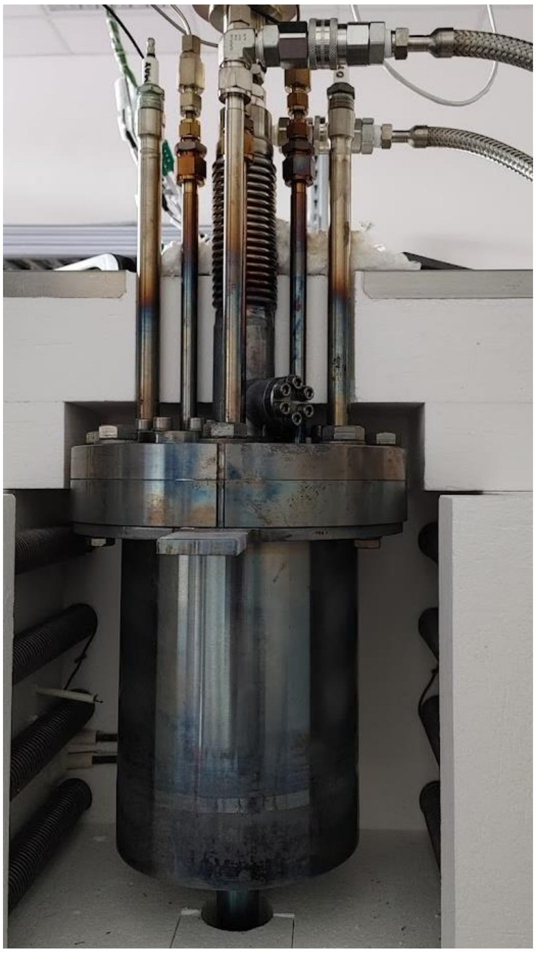

Figure 1. HLM cell for testing in PbLi in CVR. The basic concept for Pb and LBE is very similar (fixed vessel, attached cover with gas inlet/outlet and HLM level detection, steel bellows for gas tightness of upper tensile adapter), however the thermal insulation is not detachable.

Gas tightness is an important issue due to an oxygen amount in HLM (Pb, LBE), its monitoring and regulation. The oxygen is presented in HLM in form of dissolved PbO [NEA OECD 2015]. High level of oxygen leads to creation of protective oxygen layer (iron oxides) [Duchon 218][Kosek 2021], however high amount of oxygen products is undesirable for primary circuit operation. Low level of oxygen causes dissolution of protective oxygen layer and subsequently dissolution of particular alloying elements, e.g. Ni in nickel based alloys. That can lead to liquid metal embrittlement [Halodova 2018]. Thus the oxygen amount have to be maintained in optimal range [NEA OECD 2015], [Martin 2004]. Remark, there is a request for zero oxygen amount due to high oxidation of Li for PbLi fusion systems and therefore the gas tightness is required too. Protective oxygen layer does not exist due to low oxygen activity with iron, chromium and nickel [Courouau 2017].

Therefore, emphasis should be put on the sealing in places of inputs, i.e. gas input, thermocouples, level detectors and oxygen sensors. The oxygen sensors used in CVR are based on Bi-BiO according to [NEA OECD 2015], the main body is ceramic tube. Swagelok is a possible sealing system, however it takes not negligible risk of probe destruction in case of using inappropriate force during the mounting. Therefore cement with combination of gastight sealer between the ceramic tube and steel housing is a safer solution, however, it withstands lower temperatures. Temperature lowering can be achieved by sufficient length of the probe, extended out of heating area, where additional passive or active cooling system can be placed.

The testing HLM cell is a system working under about 2 bar (200 kN/m) argon gas overpressure. That also protects HLM against unwanted oxygen leak into the cell. The gas mixture of Ar + H_2 is used for decreasing oxygen amount in Pb and LBE. Pure Ar is used for maintaining required oxygen level or for slow oxygen increase, depending on actual gas purity.

In terms of specimen fitting, the cell contains two parts – a vessel with removable cover and a pull rod. The cover includes steel frame for specimen fitting on one side. The pull rod is the second specimen's fitting. These two parts are connected with steel bellows, see Fig. 1 and 5, to allow the mechanical loading of the tested specimen and to sustain the gas tightness.

3 STRESS MESUREMENT

Several technical issues need to be taken into account in case of stress measurement. First of all the load cell location is preferably on the bottom side of testing machine workspace for standard testing in air. That decreases the risk of heat transfer from furnaces to load cell. In case of testing in HLM cell, it depends on the concept of HLM filling and emptying. In CVR, there is a variant with two separated vessel systems, see Fig. 2, where one vessel is for HLM melting and preparation and the other vessel is for the mechanical testing. Both cells are separated during the testing and connected only at the time of HLM transfer. The alternative is permanent connection between the two cells.

First alternative with separated cells enables the load cell location on the bottom and the HLM cell weight (liquid metal included) is then as load offset at the beginning of mechanical test. The bottom location minimizes the heat risk on load cell without additional active cooling system. Another advantage is the possibility to use the HLM cell on a testing machines with moving crosshead at the bottom. First alternative also requires flexible connection (especially gas transfer pipes) and excludes firm fixation on testing machine, which can cause inaccuracy in load measurement due to the moment occurrence.

Alternative with permanently connected cells requires the load cell location in upper part of the testing workspace. Additional cooling system is recommended. Firm fixation of the whole system to testing machine is possible and probably inevitable. The system can work only with upper crosshead movement.

As mentioned above in HLM cell description, the pull rod and the cell is connected with steel bellows to ensure the gas tightness. However, the bellows put an inaccuracy into load measurement due to the stiffness. The stiffness of the bellows can be calibrated for particular test parameters, i.e. positions (data record of the crosshead absolute position required), testing speed and temperature. Load from the bellows can be evaluated and load record correction can be done to obtain precise results from mechanical testing, e.g. the load measurement with the bellows used in CVR requires correction max. ± 150 N (particular value depends on parameters above and appropriate calibration curve).

Using bellows to sustain the gas tightness appears to be inappropriate in case of small specimen tensile testing. Considering material of certain tensile stress properties, small specimen with small cross-section area requires proportionally lower applied load. That can lead to situation, where bellows' stiffness influence is in the order of measured load values of the small specimen. This approach is undesirable according to measurement accuracy, even with the appropriate calibration curves. Therefore additional development continues in CVR, focused on movable sealing around the moving pull rod. A sealing with higher compliance is being considered, however, its placement would be moved away from the heating zone to minimize the heat transfer and thus low the operating temperature of the sealing.

Figure 2. Separated HLM cells: Upper one for melting and lower one for testing in LBE and Pb, currently connected due to liquid metal filling.

4 STRAIN MEASUREMENT AND HLM EXTENSOMETER

Strain measurement is in HLM is more demanding issue comparing to stress. There is a wide range of sensors for deformation measuring in air, including laser or video extensometers able to measure small deformations on small specimens. HLM excludes using optical measurement and its conductive properties make unlikely the measurement via electric currents. Hence the contact measurement appears to be most suitable approach. On the other hand, HLM is not a standard environment for material testing and there is an absence of commercially usable solution.

4.1 Measuring without extensometer

Without an extensometer for HLM in CVR, the deformation measurement had to be done from crosshead position of the electromechanical testing machine. The measured deformation includes the deformation of the tested specimen, but also the elastic deformation of the whole alignment, i.e. pull rods, HLM cell and all adapters for specimen fixing. The measured results can be corrected thru stiffness evaluation for the whole tensile alignment at particular temperature. This approached was applied in GEMMA project, where calibration curves were used for applied load ranges.

In the frame of GEMMA project in CVR, fracture toughness measurement was performed on 3 mm thick 15-15Ti (EN 1515CrNiMoTiB / DIN 1.4970) TIG weld. Compact tension (13.5 mm diam. DCT) test specimens were manufactured in CVR. The specimens were fabricated by means of EDM cutting and grinding. In order to ensure the flatness of the DCT specimens, the plate was ground to approx. 2.5 mm thickness. The specimen was fixed into the holders inside testing vessel of the HLM cell. Liquid lead which has been pre-treated in the melting vessel (oxygen content was decreased by bubbling of Ar+6%H_2 gas) was transferred into the testing vessel at 773 K. Then, the testing vessel was cooled to the test temperature of 673 K in about 1.5 hour. The temperature was maintained at 673 K for several hours in order to reach the oxygen content of the required value (10^{-9} – 10^{-8} wt.%, measured by Bi-BiO oxygen sensors). The cyclic load pre-cracking of the specimen was performed on an electromechanical testing machine in load-control at testing temperature 673 K with frequency 1 s^{-1} and oxygen amount approx. 10^{-9} wt.%. The load at final pre-crack stage did not exceed maximal permitted load according to [ASTM 2018]. Fracture toughness testing of the pre-cracked specimens was performed in the same testing cell without specimen removing or changing the test conditions. Unloading compliance single specimen technique was applied [ASTM 2018]. The test was performed at constant displacement rate 2.6 $m \cdot s^{-1}$, the specimen displacement in load line was measured using the crosshead displacement. The step and the total displacement were chosen to obtain 30 to 40 unload/reload points before the reaching required final crack length. After test, the cell was heated to 773 K for HLM transfer out of cell and then naturally cooled to 300 K in Ar+H2 environment. Removed specimen was mechanically broken to open at room temperature in air. Initial crack, pre-crack from fatigue cycling and crack length growth due to fracture toughness test were measured from areas by means of optical stereo microscope, see Fig. 3.

The unload/reload step was approximately estimated by comparing with the crosshead position record obtained by the testing in air at 673 K with extensometer attached to the specimen. The corrections of stress due to bellows and correction of strain due to system compliance were done on crosshead position record obtained by the testing in Pb at 673 K. J- integral and crack growth were evaluated from corrected data according to real measured initial and measured crack lengths, Fig. 3. Evaluated results from testing in liquid lead are compared with results in air in Fig. 4.

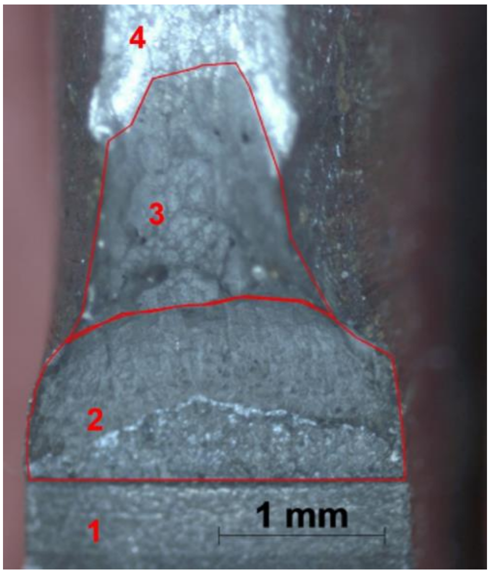

Figure 3. Fracture area of DCT specimen evaluated after fracture toughness test. 1. Notch area, 2. fatigue pre-cracking, 3. fracture toughness plastic crack growth, 4. final rupture area.

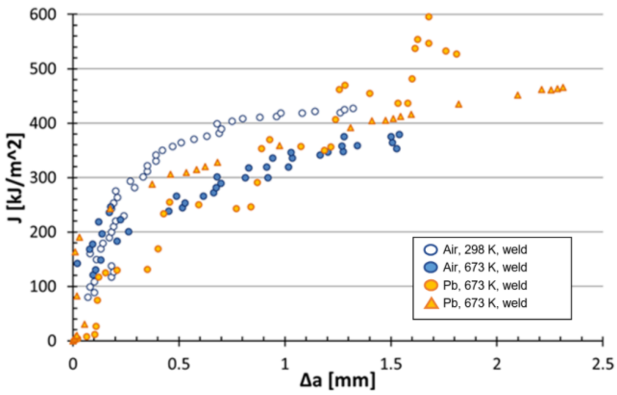

Figure 4. J-integral evaluation from fracture toughness stress-strain measurement on 15-15Ti TIG weld in air and in liquid lead at 673 K.

Comparing 15-15Ti TIG weld results in air and in liquid lead at 673 K results in approximate accordance of material behavior in both environments. Evaluated J-integral values on 13.5DCT specimen cannot be taken as a valid result in accordance with [ASTM 2018] standard, due to limits required by the standard. However supported by additional microstructure analysis, it gives sufficient comparing information of material (in particular form of processing and applied weld technology) behavior in both environments.

Evaluated results, Fig. 4, show differences, especially in elastic area at low measured deformations. Occasional scatter of results is evaluated at the end of fracture toughness tests in Pb. To make precise measuring and to decide, whether the reason of data scattering is in specimen or more probably in specimen fittings, extensometer is recommended.

4.2 HLM extensometer development

Using extensometer with HLM cell requires to withstand applied temperature up to 825 K, gas tightness to sustain protective atmosphere in the testing vessel and material without susceptibility to degradation due to HLM environment.

Development of contact extensometer for HLM applications is being performed in CVR. To avoid HLM degradation, Ni-alloys were avoided and commonly available austenitic stainless steels for high temperature applications in NPP were used. Ceramic materials were considered. However due to risk of liquid metal solidification in moving parts of extensometer and potential heating shocks applied during the cleaning process, ceramic parts were rejected in the first stage of development.

Due to the limited space in the cover of the testing vessel and amount of probes passing through the cover, concept of rod-in-tube extensometer was chosen as an appropriate solution with one required vessel input, see Fig. 5. Rod and tube are moving independently. Commercially available sensor can be used to measure mutual movement between rod and tube, however it has to provide gas tightness and be able to withstand the temperature at the upper ends of the rod and tube.

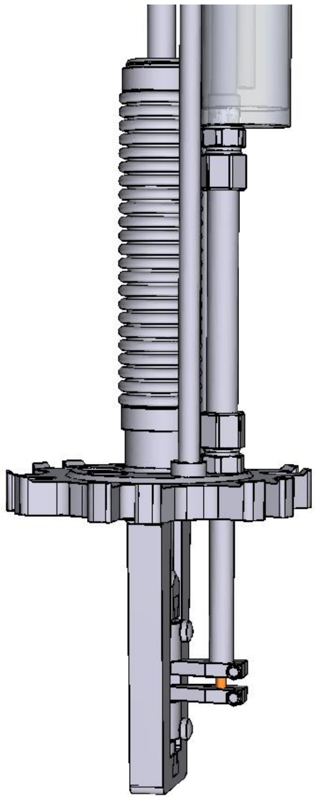

Figure 5. HLM extensometer for elongation and/or COD measurement. The extensometer is mounted to the cover of the HLM cell and it measures between bottom and top fitting located in load axis.

Temperature of commercially available sensor which is usually limited from 320 to 370 K.

The temperature can be lowered by extension of the rod and tube to area away from the heating zone with additional cooling system as optional solution for insufficient workspace.

On the side of the specimen location, the developed extensometer has fitting endings. Default concept was developed for rod shape tensile specimen, hence the measuring was placed on the adapters closest to the specimen. The rod-in-tube extensometers are suitable for crack open displacement (COD) on compact tensile specimens.

Modification of endings allows strain measurement directly on the specimen, but that requires modification of tested specimen too. Suitable shape of tensile specimen is supplemented with collars. Similar shape is used for creep testing.

HLM extensometer prototype is currently prepared for connection to electronics of the electromechanical testing machine. It will be tested in LBE environment and conclusions on operability will be drawn. In case of fully operational extensometer, additional changes for applications on small specimens will be considered.

5 CONCLUSIONS

General approach to HLM stress-strain measurement in CVR is summarized based on previous experiences with testing in liquid LBE and Pb.

Stress measurement with steel bellows concept requires additional load calibration and correction for particular test parameters. This concept is not recommended for small specimen testing, thus it requires additional modifications.

HLM extensometer is developed and is prepared for operational testing. The general concept and issues are introduced.

General approach to prolong the casing outside the heating zone is being considered. It appears to be appropriate solution enabling usage of materials and components providing gas tightness and working at lower temperatures up to approx. 370 K.

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REFERENCES

[Alemberti 2020] Alemberti, A. LFR GIF 2020 Annual Report. GEN IV International Forum, 2020, [online]. [Oct. 8th 2021]. <https://www.gen-4.org/gif/jcms/c_177507/lfr-gif-2020-annual-report>.

[ASTM 2018] ASTM E1820 - 18. Standard Test Method for Measurement of Fracture Toughness.

[Chocholousek 2016] Chocholousek, M. et al. Testing of T91 steel in heavy liquid metals, In 4th International Conference Recent Trends in Structural Materials Nov 9-11, 2016, 2016, pp 1-4, ISBN 978-80-87294-69-7

[Courouau 2017] Courouau, J.-L. Feron, D. Corrosion of structural materials by liquid metals used fusion, fission and spallation. [online] 1st IAEA Workshop on Challenges for coolants in fast spectrum system, Jul 2017, Vienne, Austria. [Oct. 8th 2021], <https://hal.archives-ouvertes.fr/hal-02417812>. hal-02417812

[Duchon 2018] Duchon, J. et al. Characterisation of oxides by advanced techniques. Acta Metallurgica Slovaca, Volume 24, Issue 1, 2018, pp 13 – 19. Doi: 10.12776/ams.v24i1.1031

[Dzugan 2017] Dzugan, J. et al. Mini-tensile specimen application for sheets characterization. IOP Conf. Series: Materials Science and Engineering 179 (2017) 012020. Doi:10.1088/1757-899X/179/1/012020

[Halodova 2018] Halodova, P. et al. Microstructural investigation of LME crack initiated in ferritic/martensitic steel T91 loaded in liquid lead-bismuth eutectic at 300 °C. Materials, Volume 12, Issue 1, 22 December 2018, Article number 38. Doi: 10.3390/ma12010038

[Kosek 2021] Kosek, L. et al. Performance of 9Cr Ferritic-Martensitic Steels in Flowing Lead for 8000 h. Journal of Nuclear Materials, Volume 554, October 2021, Article number 153092, Doi: 10.1016/j.jnucmat.2021.153092

[Martin 2004] Martín, F.J. et al. Oxide layer stability in lead-bismuth at high temperature. Journal of Nuclear Materials, Volume 335, Issue 2 SPEC. ISS., pp 194 – 198, 1 November 2004, Doi: 10.1016/j.jnucmat.2004.07.017

[NEA OECD 2015] Nuclear Energy Agency Organization for Economic Co-operation and Development, Handbook on Lead-bismuth Eutectic Alloy and Lead Properties, Materials Compatibility, Thermal-hydraulics and Technologies, 2015 NEA No. 7268 ed. [online]; Issy-les-Moulineaux, France: NEA OECD, 2015. [Oct. 8th 2021]. https://www.oecd-nea.org/jcms/pl_14972

[Rund 2015] Rund, M. et al. Investigation of sample-size influence on tensile test results at different strain rates. Elsevier Ltd. 1st International Conference on Structural Integrity, Funchal, Portugal, 2015. ScienceDirect [online] Procedia Engineering 114 (2015) pp 410 – 415. Doi: 10.1016/j.proeng.2015.08.086

[Spirit 2016] Spirit, Z. et al. Mechanical testing of anisotropy in ODS steel tubes [online], In 4th International Conference Recent Trends in Structural Materials, Nov 9-11, 2016, 2016, pp 1-4, ISBN 978-80-87294-69-7. Doi: 10.1088/1757-899X/179/1/012065

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