



# Design of the LIMELIGHT Test Rig for Component Testing for High-Temperature Thermal Energy Storage with Liquid Metals

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**Abstract:** Thermal energy storage systems for high temperatures >600 °C are currently mainly based on solid storage materials that are thermally charged and discharged by a gaseous heat transfer fluid. Usually, these systems benefit from low storage material costs but suffer from moderate heat transfer rates from the gas to the storage medium. Therefore, at the Karlsruhe Liquid Metal Laboratory, liquid metals are investigated as alternative heat transfer fluids for such heat storage systems, making use of the broad temperature range, in which the metals are in a liquid state, and their efficient heat transport capabilities. In this work, the design and construction of a high-temperature test rig using liquid lead is presented. The goal of the experiments is to demonstrate the operability of a pump, valves and measurement equipment at 700 °C in a challenging corrosive environment. Based on material pre-tests in stagnant lead at 700 °C, which are also shown in this study, aluminizing and pre-oxidation of the pipes and components are applied for enhanced corrosion protection.

Keywords: thermal energy storage; high temperature; component tests; corrosion; liquid metals

## 1. Introduction

The European Green Deal of 2019 declared the goal of a climate-neutral EU in 2050. As a quarter of the EU's energy is consumed by industry, there is a particular focus on this sector, especially because it currently uses mainly fossil fuels, which result in greenhouse gas emissions of 513 million tons of  $CO_2$  equivalents per year [1].

High-temperature processes, e.g., in the iron and steel industry, require heat above 700 °C [2]. For integrating fluctuating, renewable energy sources, high-temperature heat storage systems are proposed. Compared with other energy storage technologies, heat storage systems are relatively simple, efficient and cost-effective. This is particularly true, if the energy in the industrial processes is needed in the form of thermal energy. At such high temperatures, mainly heat storage systems based on a solid storage material and a heat transfer gas are being developed [3]. However, those systems suffer from poor heat transfer, which is why liquid metals are suggested as alternative heat transfer fluids [4,5]. They offer a broad liquid range and can transport heat more efficiently than conventional fluids such as gases or salts [6].

Liquid metal candidates suitable for high-temperature applications are alkali metals (e.g., sodium or sodium–potassium), heavy metals (e.g., lead or lead–bismuth–eutectic) and fusible metals (e.g., gallium or tin) [7]. Among those, liquid lead is a promising candidate because its large operational experience in the nuclear sector, its lower safety requirements



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). compared with liquid sodium as there is only a slow oxidation with air and water and its relatively low material costs compared with fusible alloys [7]. This is why lead (Pb) was chosen as the liquid metal for the test rig presented in this study.

The thermal conductivity of liquid metals is an order of magnitude higher than that of conventional fluids, leading to large convective heat transfer coefficients [8]. This is why liquid metals are proposed to be used in concentrating solar power plants [9]. At the Karlsruhe Liquid Metal Laboratory (KALLA) at the Karlsruhe Institute of Technology (KIT), their application as heat transfer fluids for thermal energy storage systems is being investigated. In the recent past, a packed-bed storage system with liquid metals being used as heat transfer fluid has successfully been tested at KALLA on a lab scale [10]. In the experiments, a 1-kWh heat storage system with zirconium silicate as filler material has been tested at temperatures of up to 380 °C. As heat transfer fluid, lead-bismuth eutectic was used. In 36 cycles in total, the charging (380 °C), discharging (180 °C) and standby behavior of the storage system was investigated. Besides the demonstration of a packed-bed system with liquid metal as the heat transfer fluid, testing of the measurement procedure and the derivation of important design parameters for a larger scale system were the reached goals of the experimental campaign. Currently, a demo-scale packed-bed system with a thermal capacity of 100 kWh is under construction. The heat storage will be integrated into an existing lead-bismuth loop at KALLA. As packed bed material, zirconium silicate will again be used. The envisaged charging temperature will be ca. 450 °C, the discharging temperature ca. 180 °C, limited by the melting temperature of lead-bismuth eutectic at the lower end (ca. 125 °C [11]) and the temperature restrictions of the materials and instrumentations of the test rig.

For temperatures up to 550 °C, various corrosion tests have been performed, and compatible material is well-known [11]. However, for high temperatures of up to 700 °C, compatible key components such as pumps and valves are not yet state of the art due to the corrosive nature of the liquid metals at elevated temperatures [12,13]. This is why at KIT, in a current joint project with the company KSB SE & Co. KGaA and the German Aerospace Center (DLR) funded by the German Federal Ministry for Economic Affairs and Climate Actions (BMWK), a test rig is being set up that will allow the testing of components at 700 °C. This paper will give an overview of the status regarding the materials and the design of the components of the high-temperature test rig.

In the literature, few works on the design and setting-up of a high-temperature (700 °C and higher) heavy liquid metal test loop are reported.

Anderoglu et al. [14] describe the design of a lead loop with a maximum operating temperature of 700 °C for material tests for lead-cooled fast reactors. The material chosen for the test loop was FeCrAl-based oxide-dispersion-strengthened (ODS) steel. The layout was relatively straightforward, with one loop and the liquid lead flow conveyed by an electromagnetic linear induction (EM) pump. As the material of the EM pump was SS316, 500 °C need to be maintained in the pump [15]. Therefore, the flow rate was limited for tests > 600 °C to achieve sufficient heating in the test section. The flow rate was indirectly determined with a heat exchanger. The main goal was to analyze the corrosion of inserted material specimens in the heated test section under different thermo-hydraulic conditions and not the hydraulic components themselves. Therefore, no valves or flow measurement devices were tested in the loop.

Piazza et al. [16] report on the design of a liquid lead storage tank with temperatures of 600–750 °C in the frame of the EU project NEXTOWER (concentrating solar tower application). The material used was Alloy 800 with an internal cladding of FeCrAl, which showed good resistance against the liquid metal at 800 °C [17]. As the heat was planned to be transferred to a gas via heat exchangers, liquid metal was not pumped in that concept. No experimental results of the storage tests are published yet, to the best of the authors' knowledge.

Kim et al. [18] present a liquid lead loop for demonstrating a two-tank heat storage solution for concentrating solar power at temperatures of 500–770 °C. No pumps or valves

are integrated in the design. The "hot" and "cold" liquid metal are each transported using gas pressure (argon cover gas) on the surface of the liquid metal. The mass flow is determined via the changing level of liquid lead–bismuth in the heat storage tanks.

All in all, while the above-mentioned literature shows the increasing interest and relevance of heavy-liquid-metal-based thermal storage, no test loop for temperatures of 700 °C, including the testing of a pump and valves at such temperatures and, at the same time, the monitoring of operating conditions via differential pressure, flow and temperature measurements, has been reported yet, to the best of the authors' knowledge.

We aim to close this gap by testing the operability of a pump, valves and measurement equipment in liquid lead at 700 °C. This paper presents the design and construction of an according loop and the underlying scientific and technical considerations.

#### 2. Materials Used for the Liquid Metal Loop

#### 2.1. Liquid Metal: Lead (Pb)

The test rig LIMELIGHT (liquid metal loop for high temperature) will use lead as liquid metal. It has a melting temperature of 327.5 °C [11]. The equations for the thermophysical properties (density  $\rho$ , thermal conductivity  $\lambda$ , dynamic viscosity  $\mu$ , specific heat capacity  $c_p$ ) are given in the following equations [11] and are listed in Table 1 for 700 °C and 1 bar:

$$\rho/\left(kg/m^{3}\right) = 11441 - 1.2795 \cdot T/K \tag{1}$$

$$\lambda/(W/(m K)) = 9.2 + 0.011 \cdot T/K$$
 (2)

$$\mu/(\text{Pa s}) = 4.55 \cdot 10^{-4} \cdot exp(1069/(T/K))$$
(3)

$$c_p / (J/(kg K)) = 175.1 - 4.961 \cdot 10^{-2} \cdot T/K + 1.985 \cdot 10^{-5} \cdot (T/K)^2 -2.099 \cdot 10^{-9} \cdot (T/K)^3 - 1.524 \cdot 10^6 \cdot (T/K)^{-2}$$
(4)

Table 1. Properties of liquid lead (Pb) at 700 °C [11].

Lead Properties	
Density $\rho$	10,196 kg/m <sup>3</sup>
Thermal conductivity $\lambda$	19.9 W/(m K)
Dynamic viscosity $\mu$	1.4 mPa s
Specific heat capacity $c_p$	142.1 J/(kg K)

#### 2.2. Structural Material

The material of the pipes of the liquid metal test loop, which are heated up to 700 °C, is a high-temperature chromium–nickel alloy (Alloy 800H, 1.4958). Alloy 800H offers good creep strength at temperatures above 600 °C, good resistance in oxidizing conditions as well as metallurgical stability in long-term use at high temperatures. To improve its corrosion resistance, a protective alumina layer is added to the inner wall of the pipes. This is performed in a pre-treatment by the generation of aluminum in a pack cementation process with subsequent oxidation. Previous works have shown that alumina-forming metals and coatings show a high resistance to liquid metals [19].

For the parts of the loop, which do not experience the maximum temperature of 700 °C, such as the sump tank (Section 3.3), stainless steel (1.4571) is used without any additional coating.

The loop will allow isothermal tests of the components at 700 °C to assess the material compatibility of the components over time. For comparison, the compatibility of the material is also being tested in stagnant liquid lead at the same temperature. Within the frame of the project, further materials are being tested in stagnant conditions. However,

this is beyond the scope of this paper, which focuses on the test rig and its design and components.

#### 2.3. Pre-Tests of Structural Material under Stagnant Conditions

As part of a corrosion screening test, alloys with a wide range of different Nickel (Ni) contents, including aluminized alloys, were tested in stagnant oxygen-containing liquid lead for up to 5000 h. The screening test was carried out in the COSTA (Corrosion test facility for Stagnant liquid lead Alloys) facility at the Institute for Pulsed Power and Microwave Technology (IHM) at KIT [11]. The COSTA facility allows corrosion tests at defined and controlled oxygen contents, as it determines the amount of dissolved oxygen in the liquid lead and thus, the corrosion mechanism of the materials. The target amount of dissolved oxygen partial pressure via the H<sub>2</sub>/H<sub>2</sub>O ratio that flows in the quartz glass tube. To measure the actual oxygen concentration, an oxygen sensor was installed in the gas outlet. The specimens being tested were immersed in alumina crucibles filled with lead and then the crucibles were inserted into the quartz glass tube inside the COSTA furnace. They were exposed to liquid lead with an oxygen concentration of  $2 \times 10^{-7}$  wt% at temperatures of 600 °C and 700 °C.

As shown in Figure 1a, 316Ti, an austenitic steel containing around 12 wt% Ni, exhibited Ni dissolution at 700 °C after 5000 h that resulted in sub-surface ferritization. In alloys with a higher Ni content, the attack by liquid lead on Alloy 800H (1.4958) and Alloy 718, with 32 and 52 wt% Ni, respectively, was observed at 600 °C (Figure 1b,c). It is obvious that the liquid lead attack becomes more intense with higher Ni content in the alloys and with elevated temperature.



**Figure 1.** Representative results of the corrosion screening tests in liquid lead, (**a**) 316Ti after 5000 h exposure at 700 °C, (**b**) Alloy 800H after 5000 h exposure at 600 °C, (**c**) Alloy 718 after 2000 h exposure at 600 °C, (**d**) aluminized and pre-oxidized 316Ti after 5000 h exposure at 700 °C, (**e**) aluminized and pre-oxidized Alloy 718 after 5000 h exposure at 600 °C.

A commercially available method for protecting such materials in liquid lead at high temperatures is the deposition of an aluminide diffusion coating on the surface by pack cementation [20]. The presence of an aluminide coating in an oxygen-containing atmosphere promotes the formation of a protective  $Al_2O_3$  (alumina) layer, as Al can easily diffuse outward from iron- or nickel-aluminides at high temperatures. If such an alumina layer fails locally, the remaining amount of Al is still sufficient to maintain the alumina layer. However, in liquid lead at elevated temperatures, the dissolution of Al and Ni from the aluminide layer is faster than the formation of the alumina layer. Therefore, for an optimal barrier in high-temperature applications, it is necessary to perform pre-oxidation, which allows the formation of a thin alumina layer on aluminide prior to the exposure.

After 5000 h of exposure at up to 700  $^{\circ}$ C, no corrosion problems were observed in aluminized and pre-oxidized 316Ti and Alloy 718 (Figure 1d,e). Hence, aluminization with subsequent pre-oxidation has been found to be promising in preventing corrosion problems by avoiding the dissolution of Ni, even in an alloy containing up to 52 wt% Ni. Based on these results, it can be assumed that pre-oxidation of an aluminized Alloy 800H (1.4958), which is selected for the construction of the test rig, significantly improves the corrosion resistance in liquid lead at 700  $^{\circ}$ C.

## 3. Design of the LIMELIGHT Facility

## 3.1. Overall Layout of the Test Rig

The high-temperature test rig can be divided into two sub-systems: the liquid metal loop (with main loop and sump tank) and the auxiliary systems (gas system and instrumentation). In Figure 2(left), the main loop including the sump tank is presented. The dimensions of the support structure are 2.6 m  $\times$  2.3 m  $\times$  4.0 m. A photo shows the state of construction (Figure 2(right)). On the lower right hand side in the photo, the sump tank is visible. Above that, the pipe system is installed.



**Figure 2.** Left: simplified 3D design of the liquid-lead-containing parts of the test rig LIMELIGHT (blue arrows: flow direction of main loop; green arrows: flow direction during draining/filling of the loop); right: progress of the construction work showing the sump tank and the main tubes, photo: M. Lux/KIT.

The blue arrows indicate the flow direction of the liquid lead in the high-temperature loop. At the top, the pump and valves of the project partner KSB SE & Co. KGaA, which are to be tested, are shown. The pump is a centrifugal pump and will be immersed into a so-called "pump tank". The pump is designed to convey max. 4 m<sup>3</sup>/h liquid lead up to a height of 4.5 m, which corresponds to ca. 4.5 bar. The pump and the valves are connected to the pipes with a DN25 flange. Upstream of the test valves, the liquid metal flow is determined by flow meters that are based on the Venturi effect and are designed according to ISO 5167-4 [21]. The sump tank is the lowest part of the loop. In case of an emergency, the liquid metal, which is circulating in the upper part of the loop, is drained into this

tank. It also holds the liquid before the start of operation and when the test rig is not in operation. At the start of operation, the drain valve is open and gas pressure is used to force the liquid metal into the upper part of the loop. In order to keep the lead in a liquid state at all times, trace heating is installed at the outer surface of all liquid-metal-containing parts of the test rig. The trace heating is also used to heat up the medium and keep it at 700 °C for the high-temperature component test. During operation, the drain valve is closed, and approximately 60 L of liquid lead are in the upper part of the test rig (blue arrows in Figure 2(left)). For thermal insulation to the environment, a combination of layered mineral wool (Insulfrax<sup>®</sup> mat and ProRox<sup>®</sup>) is used. An insulation thickness of 200 mm ensures a maximum surface temperature of the loop of less than 60 °C. This ensures safety aspects regarding protection against contact and burns.

Argon is used as a cover gas in the loop to prevent the formation of lead oxide. Argon is also used as the sealing gas for the rotating mechanical seal of the pump. The piping and instrumentation diagram (PID) of the test rig is shown in Figure 3. The test components (pump and valves) are highlighted in blue. The absolute gas pressure in the sump and the pump tank (PIR) will be measured with FESTO SPTW pressure transmitters. Furthermore, the differential pressure of both valves and both Venturi nozzles (F) is determined using Siemens pressure transducers and measuring instruments. The procedure for the pressure measurement is further explained in Section 3.6. The level (L) of the liquid metal in the tanks will be detected by BERU ZE 18-12 electrodes, which close an electric circuit once the electrically conductive liquid metal reaches the sensor. Bulk temperatures and surface temperatures (TIR) are measured with 91 thermocouples (type K) in total.



Figure 3. Simplified PID of the liquid metal loop of the LIMELIGHT test rig.

#### 3.2. Design of the Pipe System

The pipe system will obtain linear expansion due to the high operating temperature of 700 °C. In order to reduce stresses in the pipe system and loads on the flange connections and pipe nozzles, the pipe system is freely suspended, contains expansion bends in all spatial directions and long pipe sections are avoided. The pipes have an outer diameter of 33.7 mm and wall thickness of 3 mm. The maximum flow velocity is 2 m/s corresponding to a maximum mass flow of 11.3 kg/s. All pipes are inclined so that, in case of an emergency, all components and pipes can be drained solely by gravitational force in the sump tank via the drain valve (green arrows).

#### 3.3. Design of the Sump Tank

The sump tank (Figure 4) is made of stainless steel 1.4571 without an additional protective coating, as it is not experiencing the maximum temperature of the test rig (700 °C). It was designed in accordance with the AD2000 regulations [22] and manufactured in-house. The sump tank is keeping the liquid lead inside above melting temperature in case of standstill and before start of operation. In case of an emergency, the material only experiences a short-time temperature rise to 550 °C, as the incoming lead at 700 °C is mixed with the remaining part of the colder liquid lead, which is kept in the sump tank. The total volume of the sump tank is 170 L. The 3D drawing in Figure 4(left) shows the DN25 connections for the instrumentation (temperature, level, pressure). Additionally, a 20 mm pipe is installed to connect the safety valve (pressure relief valve), which opens in case of excessive overpressure. The DN25 pipe (filling pipe) is used for filling the upper part of the loop with liquid lead at the start of operation and draining in case of emergency and at the end of operation. Figure 4(right) presents a photo of the sump before connecting it to the pipe system of the loop. The figure also shows the protection trough, which is installed to collect the liquid metal in case of a leakage. It is further described in Section 3.5.



**Figure 4.** Sump tank for liquid lead loop (left: CAD model; right: after construction, photo: M. Lux/KIT).

#### 3.4. Design of the Pump Tank

Figure 5 shows 3D designs of the pump tank with (centre) and without the immersed pump (left) and a photo after construction (right). It was designed in accordance with the AD2000 regulations [22] and in critical areas, a finite element analysis was performed. The pump tank was manufactured in-house. At the DN400 flange of the pump tank, connections for temperature, level and pressure are provided, as well as connections for the inert gas stream and the safety valve, which opens in case of an overpressure. The volume of the pump tank is approximately 70 L. From the pump tank, the circular flow of liquid lead passes the valves and the Venturi nozzles and goes back into the pump tank through a DN25 pipe. Metallic gaskets are used as sealings for the flange connections of the components to the DN25 pipe.



**Figure 5.** Pump tank for the liquid lead loop (left: 3D drawing; center: 3D model including immersed pump; right: after construction, photo: M. Lux/KIT).

## 3.5. Leak Detection and Design of the Safety Trough

To detect leaks in the pipe system, leakage trays are installed in vertical pipe segments of the loop. Those have special electrodes (BERU ZE 18-12 electrodes) that can detect an accumulation of liquid lead and by that, a leak in the loop.

Furthermore, a protection trough, made of stainless steel, was constructed from three segments welded together at the overflow points. The trough is designed according to the example of water protection troughs and extends over the entire base area of the test rig. In the event of a leak, the liquid lead from the above installed components would safely be collected and solidify. It is shown in Figure 4(right) underneath the sump tank.

#### 3.6. Realization of Pressure Measurement

Flange-type diaphragm seals are used to measure the pressures or differential pressures of the molten lead at high temperature. The diaphragm seals consist of a blind flange, a membrane and a chamber filled with a high-temperature silicone oil in behind. This chamber is connected to the pressure transmitter via a flexible metallic hose. To close these diaphragm seals and to create pressure on the membrane, closing flanges were designed and constructed, which have connections to the main pipe system. Due to the necessary operating temperature above the melting point of lead, metallic seals are used.

## 3.7. Planned Start-Up of the Loop and High-Temperature Experiments

Commissioning will take place gradually. Before filling and heating up the loop with lead, a functional test of components is performed. Afterwards, the loop will slowly be heated up. At first, the lead bars will be melted in the sump tank at 400 °C using the electric heaters on the outer surface of the sump tank. The rest of the test rig will be heated up to 400 °C using the electric heaters (max. total power 50 kW) at the outer surface of the pipes and components. Then, the main loop will be filled by applying (argon) gas pressure onto the surface of the liquid lead in the sump tank. In the next step, the filling/draining valve is closed, and the loop will be heated up with a heating rate of maximum  $\approx 1$  K/min until 600 °C is reached. When the loop is filled, the speed of the pump will gradually be increased.

The pump and valves will then be tested in moving liquid lead under isothermal conditions, first at a constant temperature of 600 °C, then, after further gradual heating of the tubes and components, at a constant temperature of 700 °C (Figure 6).



**Figure 6.** Planned temperature ramps and plateaus for high-temperature component tests in the LIMELIGHT facility.

## 4. Conclusions

A new liquid lead test loop is designed and currently under construction in the Karlsruhe Liquid Metal Laboratory (KALLA) at the KIT. The goal is to test key components (pump and valves) for the operation of heat storage systems with flowing liquid lead at up to 700 °C to gain important insights in the material compatibility and measurement experience regarding temperature, pressure, flow and level in such challenging conditions. As material for the tubes, alumina-coated Alloy800H is selected and will be tested for its corrosion resistance under dynamic conditions in liquid lead. Combined with the parallel demonstration of a packed-bed heat storage solution with liquid metal as the heat transfer fluid, currently performed at the KALLA, this shall close one important gap in developing high-temperature heat storage with liquid metal.

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