Development of Sensors for High-Temperature High-Pressure Liquid Pb/Pb-16Li Applications

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Abstract – Liquid Lead Lithium (Pb-16Li) is of primary interest as one of the candidate materials for coolant fluid and tritium breeder in liquid metal blanket concepts relevant to fusion power plants. For effective and reliable operation of such high temperature liquid metal coolant systems, monitoring and control of critical process parameters like pressure, level, temperature and flow is essential. However, high temperature operating conditions coupled with the corrosive nature of Pb-16Li severely limits the application of commercially available diagnostic tools. This paper illustrates indigenous test facility designs and experimental methods used to develop non-contact configuration radar level sensor and wetted configuration diaphragm seal pressure sensors for high temperature, high pressure liquid Pb and Pb-16Li. Calibration of these sensors at high temperature between 380ºC-400ºC and high pressure upto 10 bar was performed. Reliability and performance validation were achieved by continuous long duration testing of sensors in liquid Pb and liquid Pb-16Li environment for over 1000 hour. Estimated error for radar level sensor lies within ±10 mm and estimated error for pressure sensor lies within 1.1% of calibrated span over the entire test duration. Results obtained and critical observations from these tests are presented in this paper.

1. Introduction

One of the key missions of International Thermonuclear Experimental Reactor (ITER) is to validate the design concepts for tritium breeding blankets relevant to a power producing reactor like DEMO. ITER operation will demonstrate the feasibility of breeding blanket concepts that would lead to tritium self-sufficiency and high-grade heat extraction, which are necessary goals for DEMO [1-3]. India is working towards development of Lead Lithium Ceramic Breeder (LLCB) Test Blanket Module (TBM), which is planned to be tested in equatorial port no#2 in ITER [1]. LLCB blanket concept consists of lithium titanate ceramic breeder in the form of packed pebble bed with liquid Pb-16Li alloy eutectic (hereafter referred to as Pb-Li) acting as a tritium breeder, neutron multiplier and coolant. In order to achieve the intended operation of LLCB blanket, Pb-Li is circulated in a closed loop, called Lead Lithium Cooling System (LLCS), which extracts the volumetric heat generated within TBM internal structures along with its self-generated neutronic heat. LLCS operates at a gauge pressure of 1.2 MPa with temperatures between 300ºC–460ºC at TBM inlet and outlet respectively. LLCS is designed to extract a total heat load of 0.38 MW. LLCS consists of Pb-Li dump tank, centrifugal pump with sump tank, electrical heaters, Pb-Li/He heat exchanger, Pb-Li purification system and Pb-Li cover gas system. Process parameters like pressure, level, temperature and flow need to be monitored and controlled for effective functioning of LLCS as a primary cooling system. Instrumentation availability is limited for liquid metal applications and aggressive operating parameters of LLCS coupled with corrosive nature of Pb-Li presents additional major challenges for process sensors and diagnostic tools. As each operation cycle of ITER is planned as a 2-year campaign, precise validation of sensors requires reliable performance over long duration operation. As a first step to address some of these challenges, experimental studies were carried out towards calibration and rigorous performance validation as part of process sensors R&D activities for static liquid Pb/Pb-Li applications. To fulfil the test objectives, indigenous test facilities were designed and fabricated at Institute for Plasma Research (IPR). Calibration tests were performed using analytical methods. Long duration performance tests for over 1000 hour were carried out to assess the operational reliabilities for pressure sensors, level sensor and temperature sensor assembly at high temperature and high pressure conditions in liquid Pb/Pb-Li environments relevant to LLCS.
2. Sensor Selection

Application of liquid Pb-Li confined to fusion specific studies, corrosive nature of Pb-Li and high operational temperature requirements largely impeded the development and validation of process instrumentation for Pb-Li. Proper selection of measurement technique, sizing of sensor, engineering modifications/customization of commercial off-the-shelf sensors wherever applicable for specific requirements, installation considerations demanded by liquid metal applications and rigorous experimental validation are required for efficacious development of any measurement technology relevant to liquid Pb-Li.

2.1. Pressure Measurement

For a wetted pressure sensor in high temperature environment, sensing element along with signal-conditioning electronics must be isolated from the effect of extreme temperature. An indirect liquid metal pressure measurement approach was developed using gas pressure measurements [4]. However, as described, this technique does not allow instantaneous pressure measurements due to damping by large cover gas volume. In addition, such an approach essentially requires tight control over temperature to minimise errors from temperature dependent gas pressurization in a fixed volume. In this study, we have adopted piezo-resistive principle based remote diaphragm seal type pressure sensors with remotely mounted electronics for pressure measurement of liquid Pb/Pb-Li. Wetted diaphragm, acting as a process isolation seal, transmits the pressure upto remotely mounted sensing diaphragm of piezo-resistive sensor through a fine capillary filled with an incompressible, high temperature compatible fluid. Minimum volume displacement of capillary fill fluid ensures better dynamic response towards fast changes in pressure. For this study, two types of pressure sensors with 0-2 MPa measuring span were selected: a) remote diaphragm seal type pressure sensor with a 2-inch SS-316L wetted diaphragm with 1 mm bore flexible capillary containing high temperature compatible silicone oil and b) remote diaphragm seal type pressure sensor with a 1-inch SS-316 wetted diaphragm with 0.25 mm bore rigid stem configuration capillary containing sodium-potassium alloy (NaK), which is liquid at room temperature.

2.2. Level Measurement

A wetted configuration level diagnostics maybe rendered ineffective over long durations due to deposition of metallic oxides and impurities, corrosion of sensor probe and bending stresses exerted by high density liquid Pb/Pb-Li. To continuously measure the level in a non-contact manner, a pulse radar level sensor was selected. It is based on the principle of measuring transit time between the emitted microwave pulse and its reflected echo from the sensed material surface. The transit time is directly proportional to distance between the sensor and the medium. Unlike ultrasonic level sensors based on time of flight principle, radar level sensor provides a non-contact technique virtually unaffected by variations in process temperature, pressure or cover gas composition. To achieve a highly focused beam and smaller process connection size, basic sensor working on 26 GHz (K-band) frequency with a pulse repetition frequency of 3.6 MHz was selected. Isolation of electronics from high process temperature was achieved by a temperature isolator section. Antenna impedance cone of ceramic with graphite seal was selected for high temperature application.

2.3. Bulk Temperature Measurement and Temperature Profiling

To measure the bulk temperature of liquid Pb-Li and to study the feasibility of Pb-Li level estimation using vertical bulk temperature profiling, a compact Temperature Level Probe (TeLePro) assembly was conceptualised and customised using a K-type multilevel thermocouple. TeLePro concept provides a compact but rigid probe for vertical temperature profiling of bulk medium. It is adaptable for tanks with small diameter and tanks with internal installations. For this study, 21 temperature measuring junctions, separated by 20 mm each, were accommodated in a length of 400 mm within a single outer sheath of 6 mm diameter. Junction-1 corresponds to the tip of sensor probe. A properly sized Inconel-600 thermowell protects the sensor probe from corrosion, process pressure and bending stresses. Level estimation using TeLePro assembly is based on differential temperature measurement technique and can be further adapted to enhance the accuracy, response and resolution by selection of temperature sensor type, sheath diameter and location of sensing points respectively. The sensor customization might be limited by the manufacturing feasibility and detectable temperature gradients.
3. Experimental Details

Two calibration and test facilities were designed and fabricated specifically to provide the required test environments and to address the installation requirements of selected sensors.

3.1. Test Facility-1: Design and Experimental Procedure

Test facility-1 was designed for calibration and performance validation of non-contact configuration radar level sensor and silicone oil fill fluid based pressure sensor for liquid metal application. Major components include main tank, top nozzle, drain tank, side-section and connecting pipeline with an isolation valve. Material of construction for these components is SS-316L. **FIG.1(a)** shows the schematic diagram of test facility-1 and **FIG.1(b)** shows an image of process sensors installed on the main tank. Process test parameters are listed in Table I. Height of the main tank (600 mm) was made nearly equal to that of dump tank for LLCS. Other design constraints like internal diameter of main tank (492 mm) and height of connection nozzle were primarily governed by the installation requirements of radar level sensor. Due to large volume of main tank, lead was selected as an economical substitute for Pb-Li. Drain tank, with an internal diameter of 202.7 mm and height of 630 mm, allows for variation of liquid lead level in main tank using cover gas pressurization and venting.

**TABLE I: PROCESS PARAMETERS FOR TEST FACILITY-1**

<table>
<thead>
<tr>
<th>Process Medium</th>
<th>Liquid Lead</th>
</tr>
</thead>
<tbody>
<tr>
<td>Operating Temperature</td>
<td>380ºC – 400ºC</td>
</tr>
<tr>
<td>Operating Pressure</td>
<td>Upto 1 MPa (gauge)</td>
</tr>
<tr>
<td>Density of lead at 400ºC</td>
<td>10,584 kg/m³ [5]</td>
</tr>
<tr>
<td>Melting point of lead</td>
<td>327.4ºC [5]</td>
</tr>
</tbody>
</table>

Side-section is provided for the installation of diaphragm seal flange of pressure sensor. Side-section is provided with an adequate inclination from horizontal for gravity assisted draining of liquid metal when required. Grafoil gaskets were used between liquid metal wetted flanges for high temperature operation. Controlled shifting of liquid lead between the tanks was achieved through connecting pipeline with manual operation of isolation valve. Level switches based on conductivity principle (**FIG.1(c)**) were made using solid stainless steel rods of 2 mm diameter connected to an LED through a resistor and 9 volt DC supply. Porcelain insulator of spark plug provided the electrical isolation of electrode from tank structure. A glowing LED signified contact of rod end with liquid metal surface and provided a secondary reference for liquid metal level measurement. Resistive trace heaters, K type thermocouples and thyristor based control system were used to heat and control the temperature of test facility-1 components. Two layers of cerawool insulation blanket, each of 25 mm thickness, with fibreglass insulation tape were used for thermal insulation of test facility-1 components.

A known inventory (405 kg), in the form of solid lead ingots, was accommodated in the main tank. Radar level sensor was installed at the top nozzle. As per the height of main tank with top nozzle, radar level sensor was configured to measure liquid lead level \((H)\) in the range of 0-694 mm. Side-section was covered with a blind flange and all the ports of test facility were closed. Pneumatic pressure test and leak tests were performed to verify system integrity for required test conditions.
Subsequently, facility was evacuated upto 0.1 Pa (abs). Melt preparation was done in an inert environment by applying positive pressure of argon cover gas. A reference calibrated gauge pressure sensor was mounted on main tank to measure the cover gas pressure ($P_g$).

Using mass of lead taken, density of lead at operating temperature and dimensions of test facility-1, level of lead in the main tank was estimated analytically and compared with the output of radar level sensor. This completed first point calibration. For second point calibration, liquid lead was transferred to drain tank upto a level of 555 mm, estimated using a fixed conductivity level switch. The level of liquid lead remaining in main tank was calculated and compared with the output of radar level sensor. Afterwards, liquid lead inventory in the drain tank was transferred to main tank for long duration test. Test was performed for over 700 hour continuously with cover gas pressure upto 1 MPa (gauge). Readings were taken at regular intervals during this test duration and performance of radar level sensor was checked and validated for liquid lead application.

For pressure sensor testing, diaphragm seal flange of pressure sensor was installed on the side-section process connection flange. Zero adjustment required for the elevation of mounting position was carried out. In the set-up, sensor diaphragm seal was located at 145 mm above the bottom of main tank. Thus, only a part of liquid lead column ($H_{\text{effective}}$) generated pressure ($P_{\text{effective}}$) on the pressure sensor diaphragm.

\[ H_{\text{effective}} = H - 145 \text{ mm} \quad \text{.......................... (i)} \]
\[ P_{\text{effective}} = H_{\text{effective}} \cdot \rho_{\text{medium}} \cdot g \quad \text{.......................... (ii)} \]

where $\rho_{\text{medium}}$, in this case, is density of lead at operating temperature and $g$ is acceleration due to gravity. Total pressure ($P$) exerted on diaphragm seal is the summation of pressure applied through cover gas ($P_g$) and pressure head due to effective liquid lead column ($P_{\text{effective}}$).

\[ P = P_{\text{effective}} + P_g \quad \text{........................................... (iii)} \]

From equation (iii), it can be seen that by varying cover gas pressure alone, it is possible to vary total pressure applied to pressure sensor while simultaneously ensuring that diaphragm seal is in direct contact with liquid lead. Calculated total pressure $P$ applied to the pressure sensor was compared with pressure sensor output to estimate the error. Testing of pressure sensor was continuously done for over 310 hour and readings were taken at regular intervals during this test period.

### 3.2. Test Facility-2: Design and Experimental Procedure

Test facility-1 provided valuable operational experience of handling high-temperature, high-pressure liquid metal. In second phase, test facility-2 was designed for experimental validation of pressure sensors and TeLePro assembly in high-temperature, high-pressure liquid Pb-Li environment for extended duration. This study was intended to understand the deteriorating effects of corrosion, to test the compatibility of sensors with liquid Pb-Li and to check the feasibility of liquid Pb-Li level estimation using TeLePro. Tank-A with an internal diameter of 77.9 mm and height of 412 mm was used to test both the pressure sensors simultaneously. Tank-B with an internal diameter of 52.5 mm and height of 528 mm was used for testing of TeLePro assembly. Tubes were introduced between end of each side-section and tank-A top volume to help in the removal of trapped gas volume, if any, during the charging of Pb-Li in tank-A. In addition, these tubes help ensure proper drainage of liquid Pb-Li from side-sections due to active pressurization at ends. Tank-A and tank-B are connected through a connecting pipeline with an isolation valve. A total Pb-Li inventory of ~ 23 kg was estimated for this facility. Required inventory of Pb-Li was accommodated by melting in four batches. Testing of pressure sensors and TeLePro assembly were performed sequentially as shown in FIG.2(a) and FIG.2(b). Process test parameters are listed in Table II.

Zero adjustment required for the elevation of mounting position was carried out for silicone oil fill fluid based pressure sensor. For NaK fill fluid based pressure sensor, estimated pressure head due to inclination of stem capillary was estimated less than 1 kPa and was not considered in error estimation. For pressure sensors calibration and testing, temperatures of both the side-sections were maintained between 380°C - 400°C. Movable conductivity type level switches were used to estimate effective liquid Pb-Li heads for both the pressure sensors.
Level measurement was repeated for better estimation and an average was taken. For silicone oil fill fluid based pressure sensor, $H_{1\text{effective}}$ was 66 mm and for NaK fill fluid based pressure sensor, $H_{2\text{effective}}$ was 75 mm. Empirical relation [6] was used to calculate density of Pb-Li at 400°C. Total exerted pressures on the pressure sensors were estimated using equations (ii) and (iii). Calculated total exerted pressures were compared with actual readings obtained from respective pressure sensors to estimate the error. Two calibration cycles, each from 0 to 1 MPa (gauge) and vice-versa, were performed at the start and end of 1000 hour long duration test. Cover gas pressure was maintained in tank-A at regular intervals.

**FIG.2(c)** shows the customised TeLePro assembly before testing. TeLePro assembly was subjected to severe thermal aging in corrosive liquid Pb-Li at cover gas pressure upto 1 MPa (gauge). For first 500 hour, heater of tank-B was controlled using tank-B surface temperature measurement and for next 500 hour, junction-1 of TeLePro was used for heater control using bulk Pb-Li temperature measurement. Maximum bulk temperature upto 520°C was reached for extended durations. After completion of continuous 1000 hour reliability test, TeLePro development campaign for liquid Pb-Li level estimation was carried out. In principle, a reliable level sensing technique should be unaffected by process temperature and pressure conditions. To validate the assembly, steady state temperature profiles were obtained for two cases: a) different cover gas pressures at a constant temperature Control Set-Point (CSP); b) different temperature CSPs at a constant cover gas pressure. The sensor assembly was in liquid Pb-Li continuously for over 1240 hour. After completion of testing, TeLePro assembly was removed in hot condition from tank-B and facility heating was stopped.

**4. Results and Discussions**

For first point calibration of radar level sensor, analytically estimated liquid lead level in main tank was 198.42 mm while reading of sensor was 200.91 mm. Thus an error of +2.49 mm was observed. For second point calibration, analytically estimated level in main tank was 104.97 mm while reading of sensor was 112.60 mm. Hence, an error of +7.63 mm was observed. Observed error in level measurement of liquid lead was within [-7.42 mm, +9.58 mm] for over 1000 hour test duration. **FIG.3** presents long duration test data. The error band includes errors from manually performed dimensional measurements for configuration of radar level sensor, assumption of a constant bulk density of liquid lead for analytical estimation of true level, manual operation of isolation valve, error related to conductivity level switch and measurement accuracy of radar level sensor. The obtained level data suggests an absence of smooth surface which may be attributed to surface topography of floating
oxide layers on top of the melt. After prolonged exposure to test conditions, calibration check for radar level sensor was carried out, under ambient temperature conditions, using an aluminium reflector within the measurement range. In this study, error was estimated within [+1 mm, +5 mm]. As radar level sensor is a non-contact configuration sensor, its calibration and rigorous validation for liquid lead also corroborates its suitability for other liquid metals/metallic alloys including Pb-Li.

Estimated error in pressure measurement of liquid lead using silicone oil fill fluid based pressure sensor in test facility-1 was within 0.3% of span for over 310 hour test duration. FIG.4 presents long duration test data for pressure measurement of liquid lead.

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FIG.3. Long duration test data for non-contact radar level sensor

FIG.5(a) and FIG.5(b) show the test results obtained and condition of diaphragm seals after long term exposure to Pb-Li at high temperature and pressure. NaK fill fluid based pressure sensor showed good agreement with total applied pressure for complete test duration including calibration cycles. Error estimated for this sensor lies within 1.1% of span for over 1000 hour test duration. Silicone oil fill fluid based pressure sensor showed good agreement with total applied pressure during initial two calibration cycles and during long duration test. Estimated error lies within 0.9% of span for this test duration. But at the start of third calibration cycle, pressure sensor did not follow total applied pressure and displayed a gauge pressure reading between 0.4 MPa to 0.46 MPa for all applied pressures less than 0.4 MPa.

Deposition of Pb-Li with lustrous silvery characteristics was observed over the seal diaphragm of NaK fill fluid based pressure sensor while seal diaphragm of silicone oil fill fluid based pressure sensor was observed partially covered with lustrous Pb-Li and partially with a greyish-blackish layer. X-Ray Diffraction (XRD) analysis of samples taken from corresponding side-sections indicated presence of PbO and Li₂O. Oxide formation can occur due to various factors including presence of
free oxygen, Li₂O and PbO in the used Pb-Li ingots and surface oxidation of ingots during long term storage. In addition, high temperature long duration operation leads to severe corrosion and dissolution of structural material and impurities from tank walls. The abrupt behaviour observed for silicone oil fill fluid based pressure sensor can be due to damage/distortion of seal diaphragm, formation and deposition of oxides on the seal diaphragm, thermal expansion of silicone oil inside the capillary or can be a combination of one or more of the above factors. Further diagnosis was carried out by providing heat to the diaphragm seal at ambient pressure. The pressure sensor displayed a substantial increase (upto 0.38 MPa gauge) in measured pressure value with rise in temperature at ambient pressure. Diaphragm seals were chemically cleaned by immersing in an equal volume mixture of acetic acid, hydrogen peroxide and ethyl alcohol followed by cleaning with water. The diaphragm seal of silicone oil fill fluid based pressure sensor was observed to be distorted. In a similar trial with an identical pressure sensor, after 160 hour of continuous exposure to liquid lead followed by continuous 210 hour in liquid Pb-Li, a similar gauge pressure reading hold was observed between 0.42 MPa to 0.44 MPa for all applied pressures less than 0.4 MPa. This study suggested dominant thermal expansion of silicone oil fill fluid inside the capillary. Above made observations render silicone oil fill fluid ineffective for use in high temperature liquid Pb/Pb-Li applications over longer durations.

For TeLePro assembly development as a liquid Pb-Li level estimation technique, tank-B surface heater was controlled using a single point bulk Pb-Li temperature measurement with junction-1 of TeLePro. Due to thermal stratification in the thermally insulated tank-B, Pb-Li column was expected to arrange itself into layers of different densities, with lowest density layer on top of the melt. Due to high thermal conductivity of Pb-Li, more heat would be transferred from constantly heated tank walls to the TeLePro through bulk Pb-Li. Hence, an increasing temperature profile was expected in the bulk Pb-Li starting from the junction-1 up to the Pb-Li surface. Above the Pb-Li melt, due to presence of oxides layer and cover gas region with relatively lower thermal conductivity, transferred heat from tank walls to TeLePro would be less, leading to a reduction in temperature. Hence a decreasing temperature profile was expected in oxides and cover gas region. Steady state temperature profiles obtained are shown in FIG.6(a) and FIG.6(b).

For Case-I, temperature for the region near Pb-Li top surface decreased with an increase in cover gas pressure while the bulk Pb-Li temperature measured by lower junctions remained more or less the same. Observed profiles can be explained by the fact that the number of gas molecules increased with an increase in gas pressure, helping in more heat removal from region near top surface of liquid Pb-Li. In Case-II, overall temperature profile shifted upwards with an increase in temperature CSP due to an effective bulk temperature rise for Pb-Li and cover gas. Obtained temperature profiles from both the cases show that temperature increased continuously from TeLePro junction-1 to junction-15, remained nearly constant (within 3°C) from junction-15 to junction-18 and thereafter decreased continuously. For all observed cases, obtained temperature drops were Δ18-19 = 7°C to 12°C, Δ19-20 = 17°C to 25°C and Δ20-21 = 20°C to 29°C where Δab denotes temperature drop from junction a to junction b. Observed pattern and magnitude of this drop indicated liquid Pb-Li surface below junction-19 but very close to junction-19. Junction 19 lies at 366.4 mm as measured from the thermowell tip. TeLePro assembly was removed after over 1240 hour of continuous exposure to Pb-Li environment. A clear demarcation was observed on the thermowell with deposited greyish layers.
starting at a distance of nearly 370 mm from thermowell tip. This visual observation further corroborated that liquid Pb-Li level estimation can be done within reasonable accuracy limits using TeLePro concept. In the proposed design, precise level estimation is governed by the resolution, which is defined by the separation between successive sensing junctions.

FIG. 7(a) shows the observed deposited layers on thermowell. XRD analysis indicated PbO and Li₂O in the top deposited greyish layers. No evidence of nickel was present in the samples taken from different locations along the length of thermowell. Sensor probe was observed completely clean and protected from corrosion. FIG. 7(b) shows the thermowell after chemical cleaning. The obtained preliminary results from TeLePro tests are encouraging and further tests are planned for interface detection between liquid Pb-Li and oxide layers or between Pb-Li and cover gas, if active removal of oxides and impurities is performed.

5. Conclusion

Indigenous calibration and test facilities were designed and fabricated at IPR for development of level, pressure and bulk temperature measurement sensors for static liquid Pb/Pb-16Li applications. Since instrumentation availability is limited for liquid metal applications, rigorous test methods were applied to validate wetted configuration pressure sensor, non-contact configuration radar level sensor and bulk temperature measurement sensor. A differential temperature measurement based interface detection technique using bulk temperature profiling was proposed and extensively studied for liquid Pb-Li. High reliability and availability was observed for the sensors during these preliminary studies. Further design optimizations and compatibility with environmental factors (like magnetic field, radiation etc.) still need to be addressed for a complete development relevant to applications foreseen in heavy liquid metal coolant ancillary systems in fusion test blankets.

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References