Methods of controlling concentration of oxygen dissolved in heavy liquid metal coolants (lead and lead-bismuth) of nuclear reactors and test facilities

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Abstract. In the paper presented are the various methods of maintaining specified HLMC oxygen potential and approaches to their implementation based on the experience gained in operating various experimental facilities and power plants located in the research institutes of the Russian Federation.

Key Words: heavy liquid metal coolants, methods of controlling, oxygen

1. Introduction

At the present time fast neutron reactors with lead and lead-bismuth coolants are being developed at the national and international levels based on high standards of safety, sustainability, economic efficiency, physical protection and stability in terms of proliferation. In Russia, the engineering designs of the “BREST-OD-300” and “SVBR” reactors have been completed. In China the lead-cooled reactor “CLEAR-I” is at the stage of preliminary engineering design. In Europe the lead-cooled demonstration reactor “ALFRED” and lead-bismuth research reactor “MYRRHA” (based on an accelerator-driven system) are at the stage of engineering design [1].

In Russia a considerable experience in safe operation of lead-bismuth cooled reactor systems for nuclear-powered submarines [2,3] and an experience of many years of efficient operation of research non-isothermal circulation facilities with lead-bismuth and lead coolants have been gained; an essential technological advance in the development of methods and techniques of heavy liquid metal coolant technology support is available [4].

By now the principal objectives of lead-bismuth and lead coolants have been determined, one of them being the arrangement of conditions for corrosion resistance (passivation) of structural steels during long service life (several decades with the reactor operating at power up to 100%) [5].

The features of heavy liquid metal coolants (HLMC) include their considerable corrosion-erosion impact on structural steels. Service life performance of structural steels in the primary circuit and equipment contacting HLMC is possible only under the condition that protective (passivation) coatings are available on the steel surfaces. Currently an “oxygen” technology of structural steel surface passivation has been selected and experimentally justified, which consists in the formation and integrity control of protective oxide films on the steel surfaces by way of maintaining the preset coolant oxygen potential.

For implementation of the steel corrosion resistance technology selected, it is required to ensure regular coolant makeup with dissolved oxygen. In the process of HLMC loops operation without a special coolant makeup with dissolved oxygen, spontaneous deoxidation of the coolant occurs to the level when reliable corrosion prevention of structural steels is not provided. For the stable and reliable protection of the steels contacting HLMC within a specified time of operation, a timely feed of the required quantity of dissolved oxygen into the coolant is needed.

The SSC RF-IPPE specialists have proposed and tested under the test-bed conditions the methods that make it possible to provide continuous maintenance of the preset oxygen regimes in HLMC of both nuclear reactors and research facilities. Today the work is under way on justification of technology implementation techniques for maintaining the preset oxygen regime level for the advanced designs of reactor facilities with lead-bismuth and lead coolants.

The key parameter to be measured, which specifies the oxidation capabilities of coolant is thermodynamic activity (TDA) of oxygen. Oxygen TDA control is carried out with the use of special oxygen TDA sensors that represent an important technique for HLMC oxygen concentration control.

The methods of HLMC oxygen concentration control that were applied in actual practice can be classified as follows:

- gas-phase methods;
- solid-phase method;
- “cold point” method.

Among the gas-phase control methods are: the use of “inert gas - oxygen” gas mixture and the use of “inert gas - water steam - hydrogen” gas mixture.

The solid-phase method consists in the use of prefabricated granules of lead oxide (or bismuth oxide) and their controlled dissolution in HLMC in special devices.

The cold point method means injection of dissolved oxygen in the event of lead oxide (slag) accumulation in the “cold points” of the circulation loop (areas with the minimum temperature).

The use of “inert gas - oxygen” gas mixture

Injection of oxygen into lead-bismuth or lead coolant in the course of its treatment with oxygen-containing gas mixtures includes a stage when solid-phase oxide phases of coolant components are formed; they are the intermediate products of the process and the direct sources of dissolved oxygen (Fig. 1).

Oxygen interacts with liquid lead to form solid-phase lead oxides:

$$2Pb + O_2 \leftrightarrow 2PbO.$$

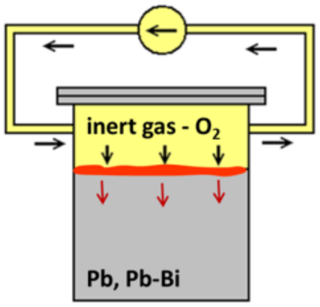


Figure 1 – The method of oxygen concentration control using the “inert gas - oxygen” gas mixture

Oxidation of liquid lead-bismuth takes place by way of oxygen interaction with the lead and bismuth components of this eutectic alloy by the reactions [6]:

$$2Pb + O_2 \leftrightarrow 2PbO,$$
$$4Bi + 3O_2 \leftrightarrow 2Bi_2O_3,$$
$$Bi_2O_3 + 3Pb \leftrightarrow 3PbO + 2Bi.$$

In the oxygen-containing gas medium or in the air, liquid lead or lead-bismuth surface oxidation takes place due to relatively fast formation of oxide film on the surface. It results from very low values of dissociation pressure typical of lead and bismuth oxides by the order of magnitude amounting to 10^{-20}–10^{-21} bar (at 500°C). After the period of initial Pb (Pb–Bi) oxide film formation on the surface, the further oxidation goes on in the diffusion stage and is limited by oxygen diffusion to the melt.

With such a method of injection, oxygen can be found in several forms:

a) in a disperse-suspended state, forming a solid phase of oxides;

b) in the form of oxide deposits on the “gas-metal” interface and on the surfaces of structural materials contacting the melt;

c) in the dissolved form.

The above method is implemented through the supply of the “inert gas-oxygen” gas mixture to the coolant surface (argon or helium are used as inert gas) or by way of gas mixture injection into HLMC flow. The attempts of gas mixture injection may result in fast injector nozzle plugging with solid-phase coolant oxides and impossibility of further operations.

Based on the experience of “inert gas-oxygen” gas mixture application for oxygen concentration control in HLMC circulation loops, it follows that one of the problems is low oxygen uptake by coolant. In other words, in order to maintain the oxygen TDA preset value, oxygen should be fed into the gas system in the quantity considerably exceeding the value required for HLMC oxidation to the preset level.

For example, at the test facility with the coolant volume of 90 liters through the use of regular feed of the “helium-oxygen” gas mixture into the gas volume of the buffer tank of the test facility, oxygen concentration was controlled within 10 000 hours. As a result of this control, the total amount of the oxygen fed was 275 g. After opening-up about 10 kg of slags were found in the loop, which were basically concentrated at the interface (in the pump tank) and in the areas with low coolant rates (gaps, dead ends etc.). It follows from the example that the amount of oxygen fed to the gas volume of the test facility essentially exceeded the amount of dissolved oxygen required for maintaining the preset TDA value, thus eventually causing the formation of solid-phase oxides in the form of slags.

Therefore, when the “inert gas-oxygen” gas mixture is used to control oxygen concentration in circulation loops, it should be taken into account that this method does not ensure complete dissolution of oxygen in coolant, and the efficiency of control can be extremely low. However, account should be taken of a possibility of undesirable accumulation of solid-phase lead oxides in the circulation loop with a potentiality of their subsequent uncontrolled (minimum) dissolution due to their passivation by dissolved iron that is found in the coolant at the initial stage of the facility’s non-passivated steel structures operation or found its way into the coolant from the structural steel through the protective film.

Among the peculiarities of the “inert gas-oxygen” gas mixture method implementation the following can be mentioned:

1) it is advisable to inject oxygen gas into the facility cover gas;
2) the process of dissolved oxygen injection into coolant includes a stage of formation of solid-phase lead oxides representing the intermediate products of the process and the direct sources of dissolved oxygen;
3) in the course of oxygen gas feed the structural steels and lead aerosols are oxidized in the gas phase;
4) performance of the method is limited to dissolution kinetics of solid-phase oxides, whose accumulation zones are extremely hard-to-predict;
5) the total amount of the oxygen fed using this method can essentially exceed the value needed for coolant oxygenation to the required level.

Among the key problems of the above-described method application, mention should be made of the following:

- low efficiency of oxygen uptake by coolant;
- risk of formation of considerable slag deposit amount both at the boundary of “coolant-gas” and the gas system, and in the coolant circulation loop;
- the process of solid-phase particles dissolution is virtually uncontrolled, their dissolution surface is unknown;
- performance of the method is limited and is specified by dissolution kinetics of the resulting lead oxides, whose surface can be “blocked” by iron impurity found in the coolant of steel circulation loop.

In spite of the described features, the above method of oxygen feed has found application and sometimes is used in the solution of research tasks. This method of control is efficient and convenient in small test facilities (up to 10 liters) with static sodium, which are designed for the study of structural steel oxidation processes, processes of impurities interaction in the coolant, testing oxygen control equipment and other R&D tasks.

2 The use of “hydrogen - water steam - inert gas” gas mixture

Another gas-phase method is the use of “hydrogen - water steam - inert gas” gas mixture, which found application for effective purification of circulation loops from solid-phase coolant oxides (hydrogen purification) [7]. Helium or argon can be used as inert gas.

When the “hydrogen - water steam - inert gas” gas mixture is fed into the loop (Fig.2) a process of water molecules dissociation accompanied by released oxygen passing into solution and the reverse process of hydrogen reduction of oxygen from the solution take place,

$$H_2O \leftrightarrow H_2 + O_{dis}, \qquad (1)$$

where O_{dis} is oxygen, dissolved in coolant.

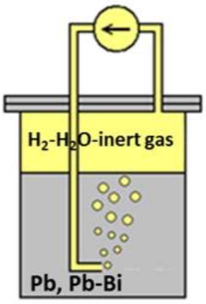


Figure 2 – The method of oxygen TDA control using the “hydrogen - water steam - inert gas” gas mixture

As the above gas mixture is fed into the loop, a direct interaction of the triple mixture components with metallic and oxide phases of the “coolant – structural materials - impurities” gas mixture system may take place under the coolant level:

$$x\ Me + y\ H_2O \leftrightarrow Me_xO_y + y\ H_2, \qquad (2)$$

where Me – components of structural materials and lead melt (Fe, Cr, Ni, Pb etc.); x, y – stoichiometric factors.

The oxidative regime required for oxygen concentration control is implemented in the event that reactions (1) and (2) are directed, respectively, towards water molecules dissociation and formation of oxides of structural material components. In this case structural material surfaces are oxidized both by oxygen dissolved in the coolant (1), and in the direct contact of steam-containing gas phase with the surfaces of structural materials (2). For the oxidative regime implementation, the gas mixture composition is selected, as well as the method of its injection into the circulation loop. Application of the “hydrogen - water steam - inert gas” gas mixture with the aim of maintaining the preset oxygen TDA levels in HLMC is not accompanied by solid-phase oxide formation.

In the course of this method implementation, the gas mixture feed to the coolant surface has an extremely low efficiency; therefore, it is advisable to perform gas mixture injection into the coolant flow.

When the control method under consideration is used, it should be kept in mind that the oxygen TDA level, which is equilibrium with the gas mixture and highlights its oxidative capabilities, is determined by the gas mixture composition; this composition can be specified by the parameter of the ratio of the water steam partial pressure and hydrogen (P_{H2O}/P_{H2}). However, when the method is selected for the circulation non-isothermal loop, it should be taken into account that the lines P_{H2O}/P_{H2} disagree with isoconcentration distribution of oxygen over temperature ranges.

The presence of gas phase in the coolant flow under specific conditions may affect the erosion processes. At the SSC RF - IPPE test facilities (Russia) the faults of inner surfaces of the injector devices were detected after 1000 hours of operation [8]. An influence of the two-phase flow depends heavily on the coolant rate, temperature and specific flow-round conditions of a certain loop element. In each case of gas mixture injection into coolant, attention should be given to this issue.

Another topical, issue is performance of the method of oxygen injection into coolant, i.e. the amount of dissolved oxygen fed into coolant with the aim of maintaining the required oxygen TDA value. In the event of the “hydrogen - water steam - inert gas” gas mixture use, the performance will be specified by the gas mixture flow rate and circulation loop temperatures.

Among the peculiarities of the HLMC oxygen concentration control method described above, the following can be mentioned:

1) Injection of gas mixture into the coolant flow is needed, and its transport over the entire circuit with the delivery to the major components in the course of circulation loop operation;

2) Oxygen TDA level, which is equilibrium with the triple mixture and highlights its oxidative capabilities, is determined by the gas mixture composition, i.e. the value of $P_{H2O}/P_{H2,}$ and is limited;

3) The lines P_{H2O}/P_{H2} disagree with isoconcentrational distribution of oxygen over temperature ranges of the circulation loop;

4) Performance of the method is limited. Analysis of the method capabilities under the conditions of each specific facility is required with regard to engineering capabilities of its implementation;

5) Presence of gas mixture in the coolant may have an effect on the structural steels;

6) Application of Ar-H_2O-H_2 gas mixtures is not accompanied by the formation of solid-phase oxides.

Among the problems of the “hydrogen - water steam - inert gas” gas mixture application, mention should be made of the following:

- selection and justification of the gas mixture composition in order to maintain oxygen concentration in the normalized range of values in different temperature zones of the circulation loop.
- selection and justification of gas mixture flow rate to ensure the flow capacity in oxygen that is required to maintain the predetermined oxygen regime;
- development of a method and device for effective injection of gas mixture into the circulating coolant flow;
- justification of the absence of zones on the facility heat exchange surfaces, where gas bubbles can be formed that will cause heat removal disturbance.

As it was mentioned earlier, the “hydrogen - water steam - inert gas” gas mixture found its application for purification of circulation loops from solid-phase slags based on coolant oxides. The mixture under consideration can be effectively used both for coolant oxidation, e.g. at research facilities in the isothermal operating conditions, in experimental installations without forced circulation of coolant in order to achieve the required oxygen TDA values, test setups for oxygen TDA monitoring equipment and other tasks.

3 Solid-phase method of oxygen concentration control

The most advanced method of oxygen concentration control in circulation non-isothermal loops with HLMC is a solid-phase method developed in the SSC RF - IPPE [9]. This control method consists in controlled dissolution of lead oxide granules fabricated by special technology and located in a specific reaction tank in the form of filling, through which the HLMC flow is arranged. Lead oxide granules contact heavy liquid metal and dissolve, thus enriching the melt with oxygen, which is transported further on throughout the whole circuit with the main coolant flow. (Figure 3). The engineering implementation of the solid-phase method of oxygen concentration control in HLMC is performed with the use of custom designed devices – mass transfer apparatus (MTA) representing an essential component of the HLMC technology system [10].

It should be noted that the use of prefabricated lead oxide granules in the mass transfer apparatus (MTA) ensures lead coolant makeup with the preliminary dissolved oxygen that rules out the formation and accumulation of solid-phase lead oxide susceptible to ferritization in the loop.

A possibility, in principle, of using a solid-phase lead oxide with the aim to control the oxygen concentration, and a possibility of creating effective systems of solid-phase control method implementation in circulation loops are basically specified by oxide dissolution kinetics (Figure 3). To date the dissolution kinetics of lead oxides of different forms has been studied in HLMC. The empirical dependences have been obtained for the calculation of kinetic characteristics of oxide dissolution processes under certain temperature and hydrodynamic conditions. The availability of the above data allows the calculation methods [11] to be implemented for determination of mass transfer apparatus characteristics with the aim of selecting the optimal design for a specific controlled unit.

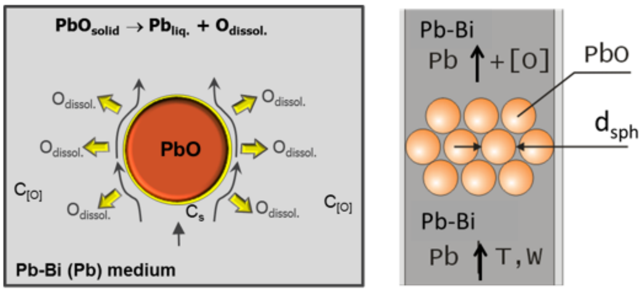
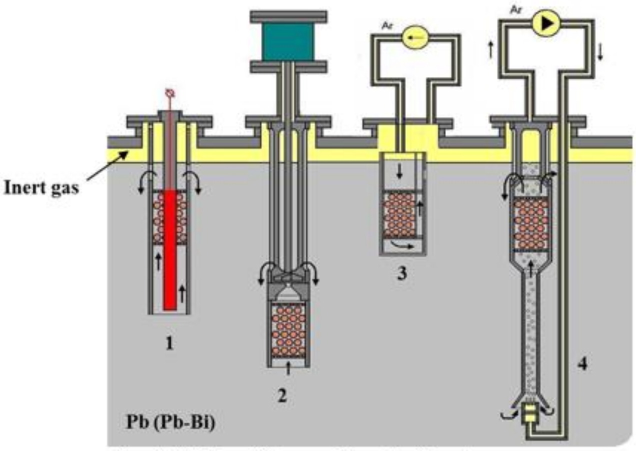


Figure 3. The scheme of lead oxide granules dissolution in lead or lead bismuth melts



1 – MTA with an electric heater;
2 – MTA with a built-in pump;
3 – MTA with discrete feed of gas medium;
4 – MTA with a gas-lift pump

Figure 4 –Mass transfer apparatus types

As a result of research and developments, the solid phase method of oxygen concentration control in lead-bismuth (lead) coolant has been justified comprehensively and recommended for industrial application in HLMC loops. Currently the SSC RF-IPPE have developed more than 50 MTAs representing the devices of various designs and principles of solid-phase lead oxide dissolution process structuring (Figure 4); considerable experience of their operation has been gained at the HLMC research facilities [10, 12].

Among the peculiarities of the above method, the following can be mentioned:

1) the use of solid-phase oxidizer (lead oxide) ensures lead coolant makeup with the previously dissolved oxygen that rules out formation and accumulation of solid-phase lead oxides in the loop;

2) a sufficient amount of lead oxide granules should be placed under the level of coolant in order to maintain the preset oxygen regime over the required period of MTA operation until its reloading;

3) a controlled coolant flow rate should be provided through the layer of solid-phase oxidizer inside the mass transfer apparatus;

4) technical solutions should be foreseen in the mass transfer apparatus to provide: the required surface of lead bismuth that makes it possible to inject the dissolved oxygen with sufficient flow capacity, to rule out lead oxide entrainment with the coolant flow from the

MTA and lead oxide reduction in the course of hydrogen purification of the circulation loop; besides, technical solutions preventing “poisoning” of lead oxide by metallic impurities found in the coolant (iron, chromium etc.) should be envisaged.

The solid-phase method of control is appropriate for oxygen concentration control in circulation loops virtually with any coolant volume: from research facilities with a few liters of coolant to nuclear power plants with coolant volume up to several hundred cubic meters.

4 “Cold point” method

Coolant makeup with dissolved oxygen can be accomplished when lead oxides are accumulated in “cold points” of the circulation loop, using a so-called “cold point” method. The capabilities of HLMC makeup with oxygen by the “cold point” method are given in Figure 5 for the options of lead and lead-bismuth coolants.

It follows from the data of Table 1 and Figure 4 that in lead-bismuth it is theoretically feasible to maintain in a natural way the required concentration of dissolved oxygen ($C_{[O]}$ ~ 10^{-6} % mas.) due to accumulation and dissolution of lead oxide deposits in the “cold point” of the loop.

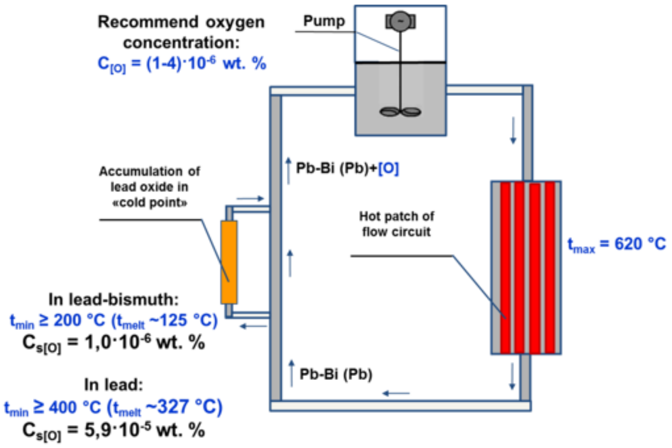


Figure 5 – The scheme of HLMC makeup with oxygen using the “cold point” method

The data on solubility of oxygen in lead and lead-bismuth eutectic melts are given in Table 1.

Table 1 – Solubility of oxygen in lead and lead-bismuth eutectic melts.

Lead-bismuth eutectic		Lead	
Temperature, °C	Oxygen solubility ($C_{s[O]}$), % mas.	Temperature,°C	Oxygen solubility ($C_{s[O]}$), % mas.
200	$1.0\cdot10^{-6}$	400	$5.9\cdot10^{-5}$
620	$2.5\cdot10^{-3}$	620	$4.0\cdot10^{-3}$

However, an in-depth analysis of all concurrent processes taking place in the circulation loop in the event of lead oxide accumulation and dissolution, demonstrates that this method is not only inefficient, but it can also cause negative consequences related to coolant technology. The major reasons for this conclusion are as follows:

1) A sufficiently large surface of lead oxide is needed to provide the required flow of dissolved oxygen (for arrangement of oxygen flow of 1 $g_{[O]}$/h using a “cold point” of 200 °C, the surface of about 50 m^2 is needed in lead-bismuth cooled facilities);

2) Mechanical corrosion of lead oxide, oxide suspension entrainment with the coolant flow to high-heat areas of the loop, and flow area blockage, core area included, should be eliminated, if this method is applied at the reactor facility;

3) In the course of hydrogen purification of the loop, with the use of the above option of oxygen regime maintenance, lead oxides will be reduced. At the same time, hydrogen recovery is a technological procedure needed after repairs, core components reloading etc., because of the slags formation when in contact with the air under various conditions (proved in practice). Therefore, an insolvable technical contradiction emerges;

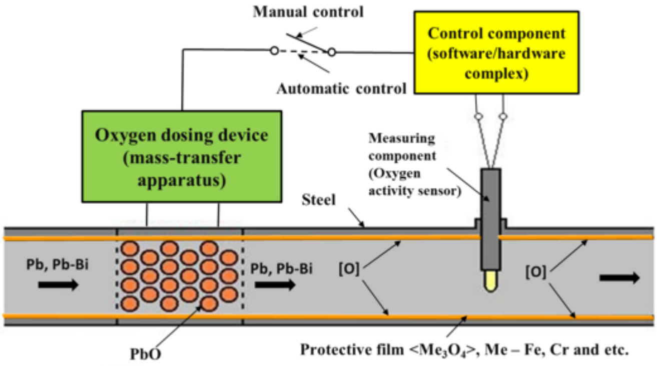


Figure 6 – The scheme of automated oxygen TDA

4) The formation of blocking film out of the impurities found in the coolant (iron, chromium, etc.) on the lead oxide surface is inevitable. It will result in considerable oxide dissolution retardation. In this case insoluble slag deposits are accumulated, and there arises a need to form new “fresh” lead oxide surfaces;

5) The lead oxide dissolution process under consideration will be uncontrolled in the course of facility operation (augmentation or decrease of dissolved oxygen flow is unfeasible).

In view of the above, the authors would not recommend the use of the “cold point” method with the aim of controlling oxygen TDA in HLMC. The above-mentioned negative processes are completely eliminated with the use of the solid-phase control method, which employs the same physical principle of lead oxide dissolution as in the “cold point” method; however, engineering and technological solutions are envisaged in order to eliminate negative factors.

5 Automation of the HLMC oxygen concentration control process

As applied to HLMC oxygen concentration control, automation is of paramount importance, the preset oxygen regime maintenance should be ensured in the course of nuclear power plant or research facility operation in the entire range of operating conditions.

With the use of mass transfer apparatus and oxygen control sensors, there is a possibility of creating a fully automatic system of oxygen concentration monitoring and control in HLMC.

Automation of the oxygen concentration control process in case of oxygen gas injection is a more complicated task than automation of the PbO solid-phase oxide dissolution process in the mass transfer apparatus. Based on the experience in the development of systems for automated maintenance of the preset HLMC oxygen regime, implementation of the automated system based on the solid–phase method presents no significant technical problems, moreover, it may employ standard controllers, using the signals of oxygen control sensors as a feedback channel.

In general, the automated system can be represented as composed of several basic components (Figure 6):

- A measuring component specifying all dependable current values of concentration or oxygen TDA in the coolant (oxygen activity sensors and thermocouples);
- An actuating component injecting the required quantity of dissolved oxygen into the coolant (mass transfer apparatus);
- A control component (hardware and software system) that receives information from the measuring component, and with the use of special algorithms generates commands to the actuating component for the accurate maintenance of the preset oxygen concentration in the coolant.

This automated system will make it possible to perform the required coolant makeup with dissolved oxygen by way of MTA oxygen input rate control with the use of the hardware and software system for the accurate oxygen TDA maintenance within the specified range virtually without the intervention of an operator.

As a typical example, Figure 7 illustrates the results of oxygen concentration control in the preset range at different setpoints. The values are given in computed concentrations for convenience of comparison with the recommended values of control range boundaries: $C_{[O]}$ = (1-4) $\cdot 10^{-6}$ % mas. Control was performed using the automated system based on the solid-phase method at the non-isothermal circulation test facility “TT-2M” (SSC RF – IPPE, Russia) with the coolant volume 200 liters. The test facility operated in the non-isothermal temperature regime: 520 °C – hot section, 420 °C – cold section. It should be noted that the automated system allowed oxygen concentration control to be ensured at different setpoints within the dissolved oxygen concentration range boundaries recommended for the setpoints with HLMC.

Several automated prototype systems for HLMC oxygen concentration control were developed and constructed in the SSC RF – IPPE [13]. Their testing under the test-bed conditions confirms the stable control of oxygen concentration in the circulation loop in the oxygen TDA value range from 10^{-7} to 10^{-1} to a high precision (at least 3 %), which confirms the promising outlook for this development policy.

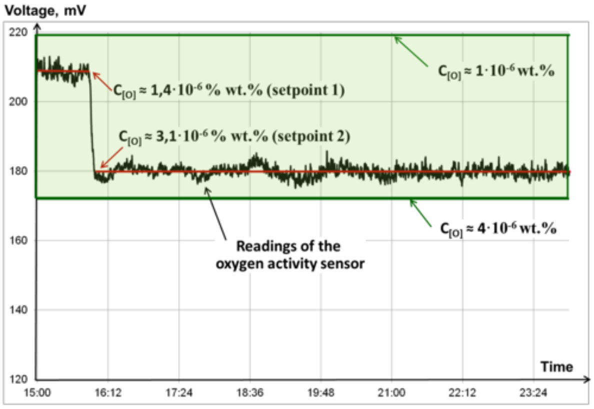


Figure 7 – Typical example of HLMC oxygen concentration control under the temperature conditions 400°C /520°C (“TT-2M” test facility, SSC RF – IPPE, Russia)

Conclusion

1. In the paper presented are the following methods of maintaining specified HLMC oxygen potential: gas-phase methods (the use of “inert gas - oxygen” gas mixture, the use of “hydrogen - water steam - inert gas” gas mixture); solid-phase method and «cold point» method.
2. The use of “inert gas - oxygen” gas mixture is efficient and convenient in small test facilities (up to 10 liters) with static sodium, which are designed for the study of structural steel oxidation processes, processes of impurities interaction in the coolant, testing oxygen control equipment and other R&D tasks.
3. The “hydrogen - water steam - inert gas” gas mixture found its application for purification of circulation loops from solid-phase slags based on coolant oxides. The mixture under consideration can be effectively used both for coolant oxidation, e.g. at research facilities in the isothermal operating conditions, in experimental installations without forced circulation of coolant in order to achieve the required oxygen TDA values, test setups for oxygen TDA monitoring equipment and other tasks.
4. The most advanced method of oxygen concentration control in circulation non-isothermal loops with HLMC is a solid-phase method. The solid-phase method of control is appropriate for oxygen concentration control in circulation loops virtually with any coolant volume: from research facilities with a few liters of coolant to nuclear power plants with coolant volume up to several hundred cubic meters.
5. The authors would not recommend the use of the “cold point” method with the aim of controlling oxygen TDA in HLMC.

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