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Advanced Reactor for Cogeneration of Heat & Electricity-R&D

Report on results from out-of-pile and in-pile experiments

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Summary

The high temperature gas-cooled reactors (HTGR) may provide electricity or heat for remote areas or industrial users in developed and/or developing countries. Despite the high thermal efficiency and enhanced safety, the HTGR systems impose some challenges, especially in the field of the key components, their materials, design and testing. Experience with previous high temperature gas cooled reactor designs indicates that the helium will contain significant amounts of impurities during operation. One of the critical technical issues is long term performance of alloys at high temperature in this environment. The corrosion issues in HTGR are probably less critical compared to such issues in other nuclear reactors of Generation IV. Nevertheless it is important to take them into account adequately. It is shown that corrosion phenomena in such systems derive from the presence of low concentrations of the impurities. These impurities H2O, CO, CH4, H2 and fine particulates in the helium can produce oxidation, decarburisation and carburisation of materials depending on the relative and absolute partial pressures of the impurities and the temperature. Major corrosion effects can be eliminated by control of the environment and by selection of appropriate structural materials. Efforts in this deliverable have focused on the material testing in an out-of-pile facility, the High Temperature Helium Loop (HTHL-1). The section 2 below summarises corrosion behaviour of selected structural materials studied in simulated HTGR-helium environment, specifically in the high temperature helium loop HTHL-1. Further, some results from the in-pile test in the reactor LVR-15 are presented in section 3 and a new in-pile facility the HTHL-2 covered in section 4.

Approval

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1 Introduction

The high temperature gas-cooled reactors (HTGR) may provide electricity or heat for remote areas or industrial users in developed and/or developing countries. Despite the high thermal efficiency and enhanced safety, the HTGR systems impose some challenges, especially in the field of the key components, their materials, design and testing.

Experience with previous high temperature gas cooled reactor designs indicates that the helium will contain significant amounts of impurities during operation. One of the critical technical issues is long term performance of alloys at high temperature in this environment. The corrosion issues in HTGR are probably less critical compared to such issues in other nuclear reactors of Generation IV. Nevertheless it is important to take them into account adequately. It is shown that corrosion phenomena in such systems derive from the presence of low concentrations of the impurities. These impurities H_2O, CO, CH_4, H_2 and fine particulates in the helium can produce oxidation, decarburisation and carburisation of materials depending on the relative and absolute partial pressures of the impurities and the temperature. Major corrosion effects can be eliminated by control of the environment and by selection of appropriate structural materials.

Efforts in this deliverable have focused on the material testing in an out-of-pile facility, the High Temperature Helium Loop (HTHL-1). The section 2 below summarises corrosion behaviour of selected structural materials studied in simulated HTGR-helium environment, specifically in the high temperature helium loop HTHL-1. Further, some results from the in-pile test in the reactor LVR-15 are presented in section 3 and a new in-pile facility the HTHL-2 covered in section 4.

2 Out-of-pile experiments in HTHL-1

The experimental High Temperature Helium Loop (HTHL-1) represents experimental equipment for testing processes in the high temperature helium cooled reactor. The loop operates with maximum helium flow of 10 m^3h^{-1}, maximum working pressure equal or less than 7 MPa and at a temperature up to 900°C. The connection to the analytical assembly is made over a reduction section modifying the temperature and pressure of the stream entering the gas chromatograph. The HTHL is also equipped with the helium purification system of a similar design as that of HTR-10. This system is designed as variable, enabling e.g. series connection of several purification units. Those units are a particle filter, CuO oxidizer, molecular sieve beds, low temperature adsorber. The photo of the HTHL-1, the active channel and the purification system is shown in Fig.1. Verification and testing of helium purification methods are one of the HTHL purposes. Furthermore the HTHL-1 is an out of pile loop for material testing under conditions of the helium coolant. Detailed description of the HTHL-1 loop has been described already elsewhere [1].

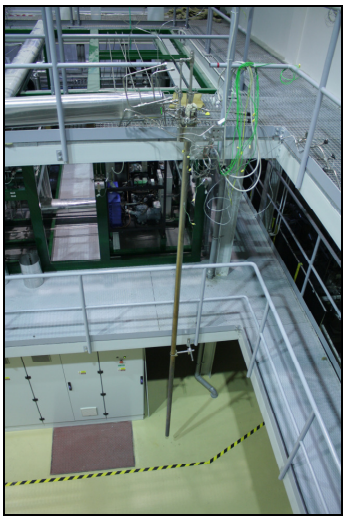


Fig. 1 Photo of the High Temperature Helium Loop (HTHL-1)

2.1 Test conditions and samples

Two types of materials Alloy 800H (welded plate) and steel P91 were used in the present work. Their compositions are listed in Table 1.
Alloy 800H consists of Fe-Cr-Ni with a minimum carbon content of 0.05 % by weigh. Alloy 800H is proposed for use at temperatures higher than 590°C and it provides good creep properties. This alloy has been approved under ASME Code for nuclear service up to 760°C [2]. A WIG-welded plate (dimension: 500mm*150mm*16mm, filler material: Nicrofer S7020 – FM 82 (Filler Metal 82)) has been provided by ThyssenKrupp VDM. Plate has been cut into various segments for microstructural study and the high temperature exposure tests in helium. The width of HAZ was rather large. Samples from the steel P91 were prepared from the supplied bar. The bar diameter was 91mm.
Test conditions, temperature and pressure in the loop were kept at 760-800°C and 3,5MPa. During the exposure also gas impurities were added. Impurity levels in the flowing helium are controlled through a feedback mechanism based on gas chromatography with HID detector (GC-HID) measurements of the gas chemistry at the inlet from a HTHL-1 containing the test materials. Humidity in the gas was controlled by an optical hygrometer BARTEC.
The duration of the test was 100 hours. During the test campaign the temperature was kept at 760-800°C and pressure 4MPa. The corrosion behaviour was determined on exposed coupons using optical and scanning electron microscopy to characterize the nature and extent of environmental interaction.

Table 1 Composition of Alloy 800, steel P91 and material of the loop internals steel 1.4541

(%)	**Alloy 800H**	**P91**	**1.4541**
C	0.02	0.08-0.12	Max. 0.08
Mn	1.00	0.30-0.60	Max. 2
P	0.020	Max. 0.020	Max. 0.045
S	0.010	Max. 0.010	Max. 0.015
Si	0.35	0.20-0.50	Max. 1
Cr	21.0	8.00-9.50	17-19
Ni	32.0	Max. 0.40	9-12
Ti	0.40	Max. 0.01	Max. 0.7
Al	0.40	Max. 0.02	—
Cu	0.30	—	—
Mo	—	0.85-1.05	—
V	—	0.18-0.25	—
N	—	0.03-0.07	—
Nb	—	0.06-0.10	—

2.2 Results and discussion

The interaction of P91 and Alloy 800H with the reactive impurities H_2O, CO, H_2 and CH_4 in simulated cooling gas of the primary circuit of the High Temperature Gas Cooled Reactor (HTGR) has been investigated. The concentration of impurities added to helium loop is given in Table 2. It is known from the previous studies that this interaction causes corrosion effects that can significantly influence the mechanical properties. From the view of high-temperature corrosion, chromium is the most important alloying element [3]. Usually the alloy forms a dense and compact layer of Cr_2O_3. The oxide layer grows slowly and protects the bulk material from penetration of other elements such as carbon. Another high temperature oxides that were expected to be formed due to bulk material composition are SiO_2 and Al_2O_3.

Table 2 Concentration of impurities in helium; HTHL-1 loop test

	H_2	CH_4	CO
ppm	10	10	50

2.2.1 Alloy 800H

Significant oxidation on the surface of specimens Alloy 800H (welded) was observed, see Fig. 3. The surface of each coupon and its chemical composition has been investigated by means of SEM coupled with the EDX analysis. The chemical composition of the surface of the weld material (Nicrofer S7020) is relatively homogeneous. The main oxides formed on the surface are chromium oxides and manganese oxide, (see Table 3).

Table 1 Chemical composition of the surface oxides formed on the welded material Alloy 800H (5S-10-21)/weld S7020 (5S-1-7) after exposure in impure He at 800℃ for 100 h

wt.%	O	Al	Si	Ti	Cr	Mn	Fe	Ni
5S-1-7	29,1	1,1	0,0	1,2	40,7	23,0	2,2	2,7
5S-10-21	31,5	0,8	0,3	1,5	50,0	8,8	5,9	1,1

Comparing the chemical composition of the surface oxide and the welding filler, the oxides formed on Alloy 800H contained more chromium and significantly less manganese. Manganese is an austenite stabiliser. An isothermal section of the Mn–Cr–O phase diagram at 818 ℃ was calculated [4]. Results from the phase diagram confirmed our findings after the exposure test in high temperature impure helium. Welding material Nicofer S7020, richer in manganese (3.2%), formed a scale layer (probably Mn_3O_4) that might have prevented the base material against carburization. The internal precipitation of the carbides in the weld material is somewhat less than in welded Alloy 800H, see **Error! Reference source not found.** a) and b).

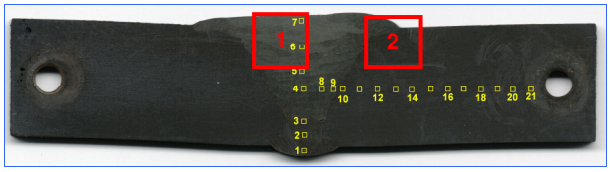
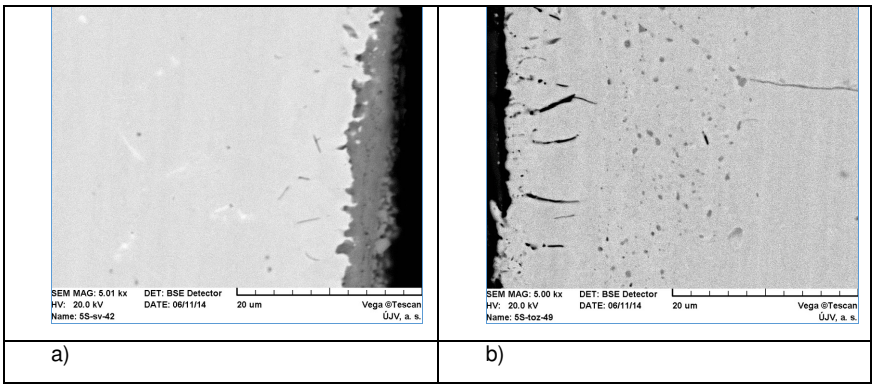


Fig. 3 Corrosion coupon after the exposure test in HTHL-1; 1-weld material; 2- Heat affected zone (HAZ)

The microstructure - cross-section of welded sample is shown in Fig. 4 a) and b). Alloy 800H contains 0.40% titanium and 0.02% carbon and hence tends to form titanium carbide and titanium nitride or carbonitride during high-temperatures exposure. Precipitates in the form of carbides were found in the austenitic matrix (40 microns from the gas-metal interface) and along the grain boundaries, see Fig. 4b).

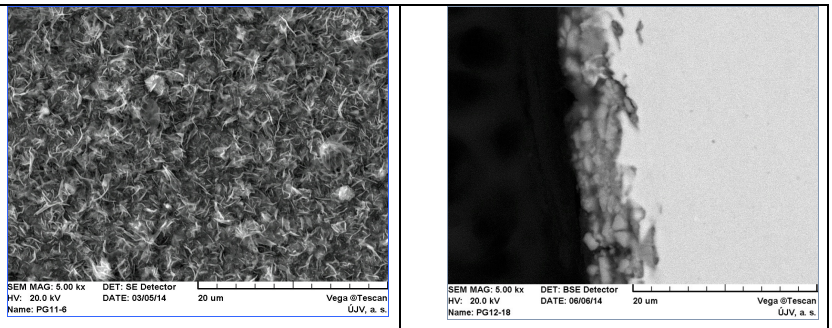


a) b)

Fig. 4 Cross-sectional SEM micrographs of the welded specimen exposed to impure helium at 800℃ for 100 h. a) weld material Nicrofer S7020; b) Heat Affected Zone Alloy 800H.

2.2.2 P91

The micro structure of P91 steel in the initial state is formed by carbide-ferritic mixture, where a carbide (most likely $M_{23}C_6$) are separated mainly at the borders of martensitic material.
Specimens of P91 were also exposed to the same condition as the previous material Alloy 800H in HTHL-1. It was found that the corrosive layer after exposure is non–uniform and consists mainly of Fe oxides, on a base of Fe_3O_4. Beneath the outer oxidation layer, a layer depleted in chrome was observed. For steel P91 the sub-corrosive layer contained no carbides.



a) b)

Fig. 5 SEM micrographs of specimen of P91 exposed to impure helium at 800℃ for 100 h. a) surface; b) cross-section

2.2.3 Oxidation of the internals in the HTHL-1

The internals of the HTHL loop were homogeneously oxidised. The thickness of the oxide slightly varied depending on the base material and process temperature and the scales consisted of multiple oxide layers. Several samples of the oxide layer were collected directly from the loop internals that are made of austenitic steel (1.4541). Fig. 6 shows some representative pieces of the oxide layer removed from the base material after more than 2000 hours of discontinuous operation. Region 1 – represents the outer oxide layer that was in direct contact with gaseous helium and it is mainly composed of iron oxide (FeO). The internal oxide zone – Region 2 is at the interface (between oxide layer and base material) is composed of magnetite (Fe_3O_4). This finding is consistent with the oxide growth at high temperatures (> 600℃), the inner oxide is formed by magnetite and the hematite is the outer oxide together with wustite. In some parts of the inner layer – Region 3 shows the oxide layer enriched in chromium, silica and manganese. Even though the base material (see Table 1) of the internals also contains nickel, no nickel was identified in any of the external oxide layers.

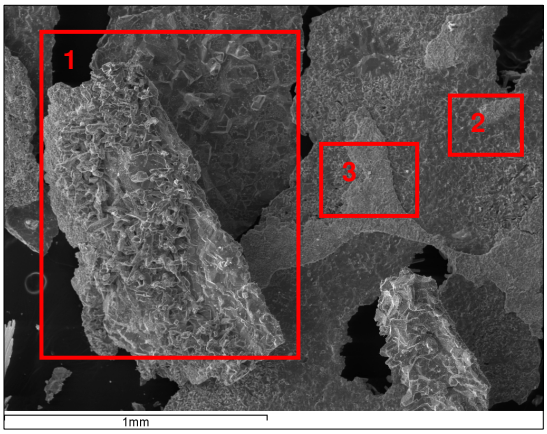


Fig. 6 Products of surface oxidation on the internals of the HTHL-1 loop: 1) oxide in contact with helium gas; 2) and 3) interface between the oxide layer and base material

2.3 Conclusions

Exposure of welded Alloy 800H and steel P91 in impure helium at 800℃ leads in all cases to external oxide scaling. The internal chromium-rich carbide precipitation occurred in welded Alloy 800H. The addition of impurities to the gas leads to more significant changes to the nature of the oxide scales compare with previous tests in pure helium.

The welded specimens form an outer scale layer composed of chromium and manganese oxide. Welding material Nicofer S7020 forms a scale oxide layer richer in manganese that might slightly prevent the base material being carburized. The amount of internal carbide was greater beneath the scale in Alloy 800H. Nevertheless the Alloy 800H retains a protective chromium oxide scale in impure helium for 100hours.

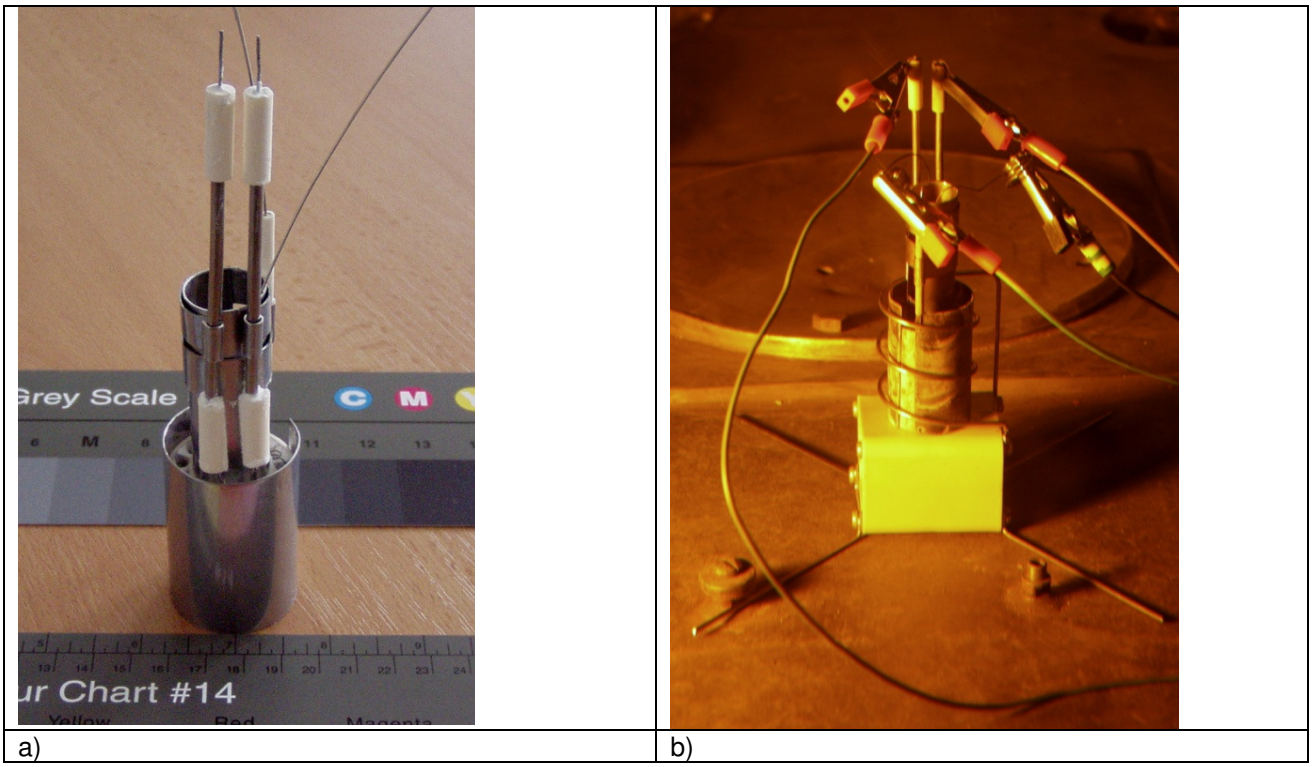
Degradation of the material due to the high temperature oxidation in impure helium was the highest for the case of the P91 material. The outer oxide layer was not uniform and already partly spalled off.

Also the oxidation of the austenitic material in the loop itself has been studied. The austenitic steel forms external iron oxide - wustite and internal magnetite or spinel in impure high temperature helium.

Nevertheless the nature of the oxide layer on the internals was rather thin, compact and without sign of scaling despite more than 2000 hours exposure including thermal cycling.

3 In-pile experiment of ceramic heating elements

During the operation of the loop HTHL 1, a decrease in the electric insulation resistance of the ceramic heating wires has been observed. Such a decrease led to a limited temperature increase of the heating elements in the test section of the loop. Therefore it was necessary to develop and validate new heating elements. The heating elements isolated by alumina C799 (Luxal 203 - see properties. [5]) were successfully tested during out-of-pile experiments in HTHL-1, therefore C799 heating elements were also selected for in-pile test. The main goal of the irradiation test was to verify whether C799 deteriorates and reduces its insulating properties.
To this aim, a specimen, was irradiated in the zone of LVR-15 (average flux of fast neutrons of about 2.5 x 1018n / m_2s), for about 6 months. After irradiation the dependence of the resistivity of the temperature ceramic insulation was verified. The experimental design did not allow for reaching a temperature higher than 770°C during the irradiation. However, it has been concluded that even after 6 months the insulating properties of the tested ceramics have not changed significantly. Heaters with the isolated alumina C799 will be used in helium loop HTHL2.



a) b)

Fig. 7 Test heating elements before exposure to irradiation; b) measurement of electric resistance after exposure to irradiation

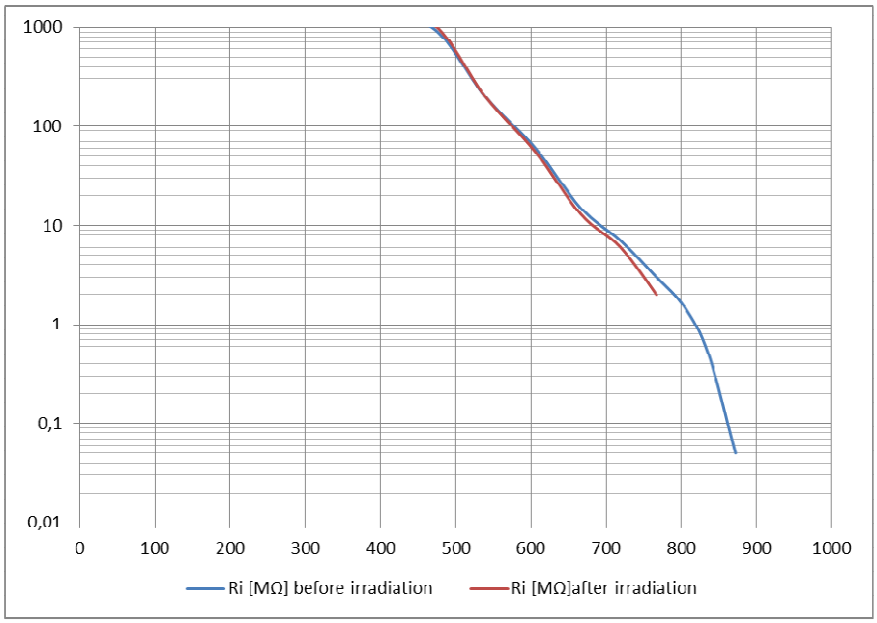


Fig. 8 Dependence of the resistance of C799 ceramic Ri on temperature, before and after the irradiation

4 In-pile high temperature loop HTHL-2

Experimental High Temperature Helium Loop 2 (HTHL-2) is an improved version of the currently operated loop HTHL-1. Detailed description of the HTHL-1 loop has been described already elsewhere [6].

Both loops represent experimental equipment for material testing in the simulated environment of the high temperature helium cooled reactor. Additionally, the influence of the neutron flux on tested structural materials in the high temperature helium environments will be studied in HTHL-2. During in-pile operation, the Light water moderated reactor (LVR-15) will be the source of neutrons. The power of the reactor LVR-15 is 10 MW. It is a cooled-tank-type reactor with a forced-circulation cooling system. It utilizes 19.7% enriched ITR-4M fuel (27 fuel assemblies). The average water inlet temperature is about 43℃ and the outlet temperature approximately 48℃.

The loop will operate with maximum helium flow of 36 $kg.h^{-1}$, with the maximum working pressure equal or less than 7 MPa and at a temperature in the specimen holder zone up to 900 ℃. The connection to the analytical assembly is adjusted in order to safely enter the gas monitoring system. The facility can provide 30kW electrical power. The helium is circulated by means of two stages circulation pump. The maximum helium temperature shall not exceed 200℃ in the pump.

Table 4 Characteristic parameters of HTHL-2 loop

Available thermal power	
Electricity power	30kW
Medium	Helium
Temperature of He	Up to 900℃
System pressure	Up to 7MPa
Flow rate of He	36 kg.h–1
Purity of He	99.9%

The HTHL-2 will consists of the active channel, a helium purification system, impurities-dosing system (e.g., CO_2, H_2, H_2O, O_2, and N_2), and a helium purity monitoring system. The schema of the HTHL-2, the active channel and the purification system is depicted in 10. The helium purification system is similar to the one in the HTR-10 facility. This system is designed as variable, enabling for example a series connection of several purification units. These units include a particle filter, CuO oxidizer, molecular sieve beds and low temperature adsorber. Verification and testing of the helium purification methods are one of the HTHL

purposes. Compared to the HTHL-1 assembly, there are some modifications to the helium purification system. Which are described further later in this document.

The helium storage tanks, helium purification system, central controlling system, and secondary-cooling system are installed outside of the reactor in the dedicated area, see **Error! Reference source not found.**.

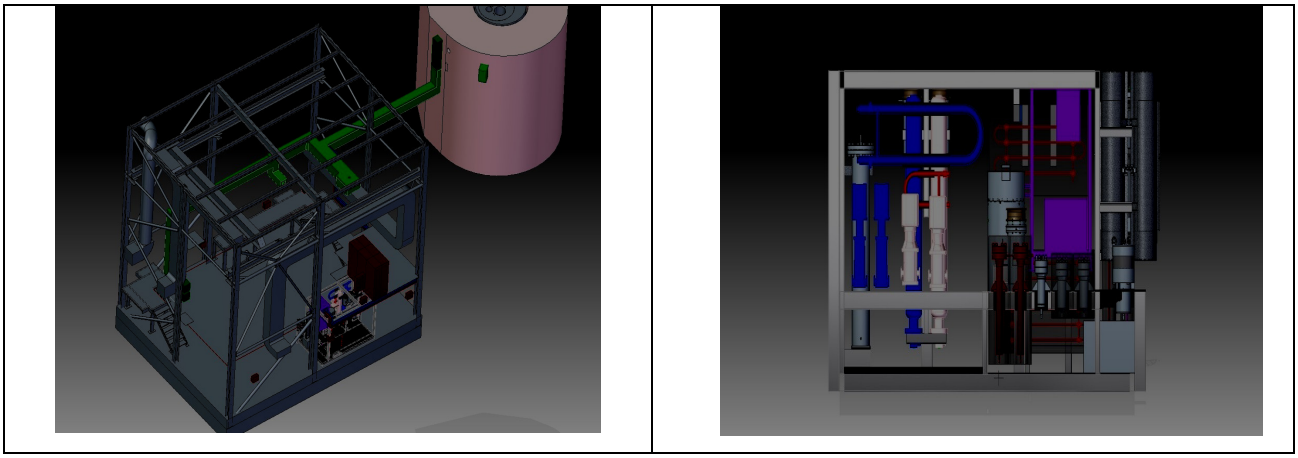


Fig. 9 3-D model of HTHL-2 loop placed in the vicinity of the LVR-15 reactor

4.1 Active channel

The specimen holder with tested structural materials (including metals and ceramics) is placed in the active channel. The active channel consists of a very complex system of coaxial pipes, a regenerative heat exchanger, electrical heaters and other supporting systems (a water-cooling unit, insulation) and specimen holder. The electrical heater heats up the helium up to 900℃ just before it enters the samples holder area. The holder is located in the lower part of cylinder-shape space with diameter of 50-mm. The total length of the active channel is 6 meters.

When inserted in the LVR-15 reactor, the irradiation channel will occupy only one cell of the reactor grid. Therefore,from very beginning, the design of the channel was limited not only by many geometrical and material parameters, but also by the maximum allowed active channel wall temperature in the core. The maximum temperature in the active channel wall shall not exceed 500℃. Most of the active channel internal parts are made of titanium-doped steel with 18% Cr and 10% Ni. The material used for the heated tube is Incolloy.

One of the very specific and technically challenging features of the loop is the two-stage circulation pump. The main problem is the unique design of the loop, the circulation pump has to be rather efficient, but small, because it is placed just above the active channel.

4.2 Helium purification and monitoring systems

One of the purposes of the HTHL-2 is to verify and test the gaseous-coolant purification procedures for a V/HTR system, or similarly for the GFR system. The impurities present in the primary system in helium can substantially accelerate corrosion of structural materials, material embrittlement and loss of strength.

The impurities contained in the helium, mentioned above, have to be reduced to specified values by a helium purification system. For this purpose a helium purification system is installed. The operational pressure of the purification should be equal to the pressure in the main loop system. The maximum mass flow required is 4 kg/h. The system is designed to be relatively flexible, so that the flow rate can be changed within a wide range. Also some components will need to be removed without disassembling the entire purification part of the loop. The flow scheme for the purification system is given in 10. The helium inlet temperature is controlled by the combined cooling and heating system of the active channel. There are several steps in order to assure the required purity of the helium exiting the active channel. After the helium is cooled down to 100℃ gas passes through two mechanical filters, where larger particles of possible corrosion products, graphite dust, fission products and aerosols are removed. During normal operation, the contamination of the primary circuit of the HTR is due to the fission products fraction released by diffusion from the intact fuel particles and directly from the defective particles [7]. An important part of metallic fission products and of the

iodine isotopes are deposited on to the inner surface of the primary circuit, particularly on the colder parts (plate-out) [8]. The expected fission products released by the fuel elements may enter the primary loop and combine with the dust, resulting in the dust having high loads of cesium, strontium, iodine, silver and tritium.[9].

The copper oxide bed, with a temperature of 250 °C, oxidize H_2 (including tritium) and CO to H_2O (vapour) and CO_2, respectively, while O_2 is also removed by adsorption on copper. Then helium is cooled down to 50°C and it passes through the molecular sieve to remove further impurities H_2O and CO_2. The absorption of CH_4, N_2, and O_2 is achieved in a low-temperature absorber, which is filled with activated carbon and cooled down in two stages by a compressor and liquid nitrogen to minus 195°C. Here, the remaining CH_4, N_2 and other impurities not retained in the preceding steps are removed.

The main impurities expected to be identified in the VHTR and GFR coolants are H_2, tritium, H_2O, CO, CH_4, CO_2, N_2 and O_2. These impurities penetrate to the coolant from various sources: leakage from surrounding environment, desorption from construction materials or as a result of subsequent chemical reactions.

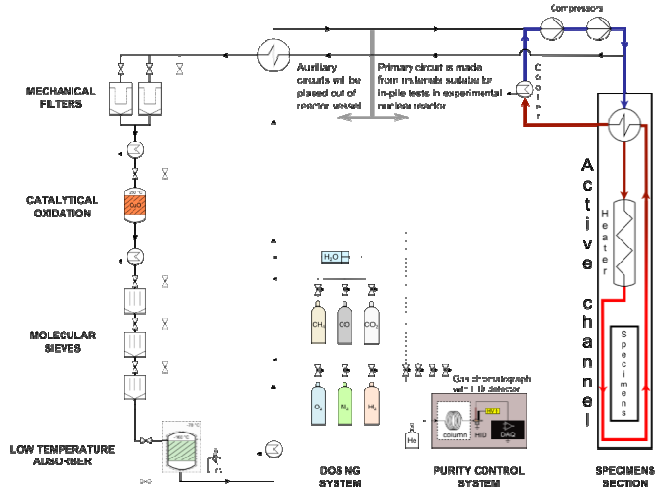


Fig. 10 Schematic diagram of HTHL loop

Even relatively low deviations from the recommended values of concentrations of the above mentioned impurities may lead to corrosion or damage of construction materials. Therefore, their investigation is crucial for new reactor designs. Impurities are planned to be dosed to the HTHL through precise electric valves from the dosing tank into the primary circuit. Once the impurities are dosed, the procedure for their removal consists, as already described of the purification steps above [10].

Exact determination of low concentrations of impurities requires precise analytical methods and eventually, also sampling of the gaseous coolant. For such cases the HTHL is equipped with a sampling panel. The sampling lines were designed with regard to minimal contamination of the sample by the surrounding air.

The adopted instrumentation to measure the impurities concentration levels consists of gas chromatographs, infrared optical analysers for CO, oximetry, dew point monitors for H_2O content and ionisation chambers for tritium.

4.3 Conclusions

The development of new technological experimental helium loops shall help to resolve technical issues imposed by the HTGR systems. The HTHL-2 loop will be used to investigate the effects of operation including neutron flux and chemical conditions of the VHTR and GFR .

5 Overall conclusions

Generation IV reactors are a set of mostly theoretical nuclear reactor designs currently being researched. While the gas-cooled fast reactor system requires still a substantial R&D support, the V/HTR system could displace fossil fuel power plants in the near future. The capability of the high temperature gas cooled

reactors (HTGR) to produce process heat above 600℃ makes it an efficient reactor type in a number of various applications such as cogeneration of electricity and other non-electric applications such as seawater desalination, hydrogen production, district heating or cooling, enhancement of oil shale recovery or any energy-demanding industrial application. [11]. Despite the high thermal efficiency and enhanced safety, the HTGR systems impose some challenges, especially in the field of the key components, their materials, design and testing.

The corrosion issues in HTGR are probably less critical compared with such issues in other Generation IV nuclear reactors. Nevertheless it is important to take them into account adequately. It is shown that corrosion phenomena in such systems derive from the presence of low concentrations of the impurities. These impurities H_2O, CO, CH_4, H_2 and fine particulates in the helium can produce oxidation, decarburisation and carburisation of materials depending on the relative and absolute partial pressures of the impurities and the temperature. Major corrosion effects can be eliminated by control of the environment and by selection of appropriate structural materials.

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