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Experimental investigation of the corrosion behavior of Eurofer97 steel in contact with Lithium ceramic breeder pebbles under specific Helium Cooled Pebble Bed breeding zone atmosphere

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ABSTRACT

For sub-sized fatigue specimens made of EUROFER97 in unconstrained contact to ceramic breeder pebbles, exposed to purge gas conditions for different durations in an oven, a chemical surface attack was observed which led to a significant reduction of fatigue lifetime [Aktaa et al., 2020]. To better reproduce the flow of the purge gas in the breeding zone of a Helium-Cooled Pebble Bed Breeding Blanket, the experiment was now repeated by placing the same kind of samples in the helium loop HELOKA HEMAT, where the gas is permanently circulated in a closed circuit, and its water content is controllable. The composition of the gas was monitored with a mass spectrometer and humidity sensors. The samples were exposed to a mixture of helium and 0.1vol% hydrogen at DEMO relevant operating conditions (550 °C, 1.2 bars abs.) for a duration of 8, 16, 32, and 64 days, respectively. New sample holders were designed which allow direct contact of the samples with the gas mixture. A special procedure for the handling of the test section was implemented to avoid contact of the hygroscopic pebbles with air humidity during the preparation phase and during the extraction of the samples from the test rig.

The present contribution discusses the various considerations on which the test rig design was based, followed by a description of the experimental setup and preliminary results of the testing campaign. These are similar to the results achieved in the oven experiment. In the future, the loop will be used for tests with up to 200 Pa partial pressure of steam in helium; a mixture that is considered relevant for the DEMO purge gas composition.

1. Introduction

In Europe, two driver breeding blanket (BB) concepts for fusion are developed for DEMO reactor specifications [1,2]: (i) the Helium-Cooled Pebble Bed (HCPB) concept, and (ii) the Water-Cooled Lithium Lead (WCLL) concept. For both concepts, a reduced activation ferritic martensitic steel is chosen as structural material, EUROFER97 (X10CrWVTa9–1). The structures are actively cooled with pressurized helium (300 – 520 °C, 8 MPa), or pressurized water (295–328 °C, 15.5 MPa), respectively. Both concepts are foreseen to be tested in ITER in the form of test blanket modules (TBM). The HCPB BB uses the advanced ceramic pebbles (Li_4SiO_2 + 35 mol Li_2TiO_3), which are enriched in ^6Li, as tritium breeder, and beryllide blocks as neutron multiplier. For the TBM and early DEMO HCPB BB concepts, helium mixed with 0.1vol% hydrogen at 2 bars pressure is used as a purge gas to remove the tritium generated.

In the breeding zone of the blanket, structures made of EUROFER97 (E97) are in direct contact with the breeder and neutron multiplier materials. The investigations reported in [3] indicate that a chemical surface attack is observed for E97 specimens in unconstrained contact to ceramic breeder pebbles. The oxide layer formed during the exposure time led to a significant reduction of the fatigue lifetime as indicated by the results of strain-controlled low cycle fatigue (LCF) tests at 550 °C (see Figure 5 and 6 in [3]). Gaisina et al. [8] found that the corrosion behavior of the steel is predominantly affected by purge gas impurities, namely water and oxygen. However, the used exposure device in their experiment did not allow keeping the water content on a specified level, aspect that is essential when replacing the share of hydrogen in the

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purge gas partly/completely by water steam as proposed in alternative purge gas concepts [4].

In the experiment reported in [3], the samples were placed in a crucible and exposed to a once-through gas stream of helium with 0.1vol % hydrogen in a heated oven. Measurements at the inlet of the oven showed that the water content of the gas was higher than certified by the gas supplier, namely about 0.1 %. At the outlet the water content was found to be around 1–2 %. It was concluded that water was generated or extracted from the pebbles. Another possible explanation would be that the surface of the pebbles was reduced. Still, using a once-through stream of gas does not allow for looking into cumulative effects that characterize a purge gas loop. To address this issue, a new experiment was proposed with the objective to improve the relevance of the test conditions, and to better quantify the effect of the pebbles and in a purge gas mixture. Thus, the samples are now tested in a helium loop where the gas is circulated and its composition monitored. In a first phase, the content of water is kept at a low level by a getter bed, so the influence of H_2O on the corrosion process of the samples can be minimized, thus allowing a direct comparison with the results of the tests done in [3]. In a second phase, it is foreseen to have a campaign with a gas mixture of helium and 200 Pa partial pressure of steam, which corresponds to a purge gas mixture proposed in [4].

The present paper discusses the experimental set-up and the relevant aspects of the first testing campaign (helium flow with water content as low as possible). First, the test rig is introduced followed by the design and manufacturing of the sample holders. A dedicated experimental procedure was developed to avoid contact of the hygroscopic pebbles with air humidity during the preparation process. Further, observations during the experiments are depicted. Preliminary results on the development of the oxide layer with time and its characterization are presented. The results are compared to earlier experimental results [3].

2. Description of the experiment

Similar to the experiments from [3], the E97 samples are to be aged for 8, 16, 32, and 64 days to observe the growth of the oxide layer. For each period of exposure time, nine Low Cycle Fatigue (LCF) samples are necessary for the fatigue tests. In addition, a cylinder is used for the investigation of the oxide layer growth. To minimize the air and humidity ingress when extracting samples from the test rig, it was decided to design and manufacture four distinct canisters, one for each exposure time.

2.1. Test rig and samples holder design

For the tests, sub-sized LCF specimens, 27 mm long, made from E97 batch 2, are used. The canisters that are designed as sample holders have to fit into the existing test section of the HELOKA facility (HEMAT loop), which is 68.8 mm in diameter and 660 mm long. Consequently, one canister has an outer diameter of 63 mm and is 76.3 mm long. More information about the loop is given in Section 2.4 – 2.6.

To allow easy installation and extraction after each testing period, the canisters are placed on a holding pin that ensures also fixation inside the test section. It consists of a spoked wheel and a tube, which allows the helium to flow through, so that the pressure loss of the insert is reduced to about 10 mbar for a flow of 2.4 g/s. Most of the helium stream flows through the central tube thus creating a low velocity region in the area where the canisters are, so that the flow conditions correspond to those of a breeding blanket.

2.2. Design of the canisters

Each of the four canisters contains the nine LCF samples and the reference cylinder surrounded by the pebbles. To ensure that the samples have a good contact with the pebbles, they are mounted on a positioning holder in form of a flower, so that they remain apart one from another during filling procedure and during the installation into the test rig.

To ensure that helium atmosphere around the samples continuously flows similar to the purge gas conditions in a breeder unit, the walls of the canister are made either out of a perforated plate or a metallic mesh. For the outer cylinder walls of the canisters, the perforated walls are manufactured using the same geometry and manufacturing technique as the one foreseen for a TBM breeding blanket box [5]. Slabs are cut from EUROFER sheets (E97, batch 3) and perforated by laser drilling (pro beam GmbH & Co. KGaA, Gilching, Germany), then formed to cylindrical walls and welded together. These side walls are intended for later investigations focusing on tritium permeation.

The bottom and top and the inner cylinder are made from X10CrWMoVNb9–2 (P92). To allow the helium to flow through the canisters, both the top and the bottom caps have openings covered with filters made from a commercially available wire mesh from Haver & Boecker OHG, Oelde, Germany, made out of X6Cr17.

The bottom part and the outer cylinder are welded together, as well as the flower and the inner cylinder. All parts were thoroughly cleaned in an ultrasonic tank, using the detergent Primasol (Primatec GmbH, Dörth, Germany). Fig. 1 shows all parts of the assembly except the samples.

2.3. Assembly procedure

The ceramic pebbles used in the experiment are from a fresh batch manufactured for this experiment in the KALOS facility [6] according to the established manufacturing procedure for EU HCPB Breeding Blanket concept. They were baked at 300 °C and stored in a chamber in nitrogen atmosphere prior to their use and they are delivered in vacuum packs.

As a first step, the samples are screwed into the “flower” plate, then the inner cylinder is screwed into the bottom part. To fill the canisters with the hygroscopic pebbles in absence of humidity, they are locked into a glove box filled with argon atmosphere. Oxygen and water content are kept below 5 ppm. The packages of the pebbles are opened only after they are put inside the glove box and the pebbles are poured into the canisters, as it can be seen in Fig. 2. The canister is placed on a shaker plate to allow a compact pebble bed. Shrink holes are avoided by gently vibrating the canisters. The pebble bed is not preloaded, and the pebbles

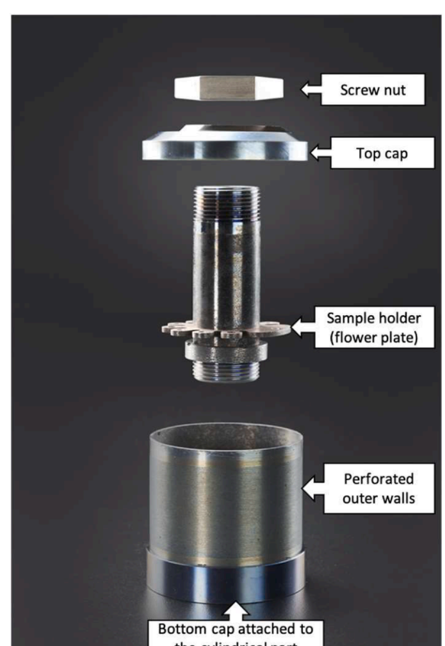

Fig. 1. Constituent parts of a canister (without samples).

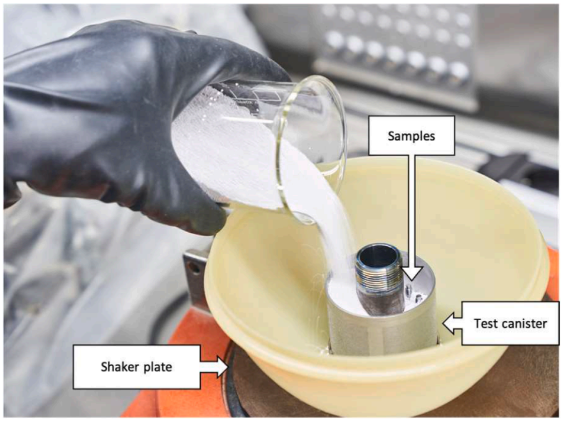

Fig. 2. Filling of the canisters with pebbles in a glove box.

are allowed to move during the installation process. After closing the canisters and locking them out, they are brought in a tight box under argon atmosphere to the HELOKA facility to be immediately installed in the test section.

2.4. Description of the experimental facility

The experiments are carried out in the HELOKA facility, using the HEMAT loop at the Karlsruhe Institute of Technology. The loop (Fig. 3) was built to investigate the corrosion behavior of new and improved materials in helium atmosphere at low pressure (max. 4 bars), but high temperatures (max. 650 °C). Depending on the temperature, mass flow values between 1 and 20 g/s are possible to achieve. Side channel flow compressors are used which produce a uniform flow without pulsations. Impurity gases like oxygen or hydrogen can be added in small quantities, in a controlled manner. The loop consists of an “eight configuration” with a helium – helium heat exchanger in the middle as energy recuperator. The control system is designed for long-term experiments running without surveillance.

2.5. Instrumentation

The loop is equipped with a number of type K thermocouples and pressure meters (from Siemens AG, Munich, Germany) to monitor its state. Most important are the thermocouples at the inlet and the outlet of the test section, which are used to control the heater in order to keep the temperature constant. A pressure differential meter is installed at the test section. Further, two gas sample openings leading to two humidity sensors from Thomsen Messtechnik GmbH, Greifenstein-Nenderoth (Germany), M-Probe, are connected at the inlet and at the outlet of the test section. The sensors are limited to a temperature of 70 °C, so the humidity can only be measured at some distance from the test section to allow the gas to cool down. To ensure a representative measurement of the coolant humidity, a small helium stream continuously flows through the sensors, from the test section area and back into the main stream.

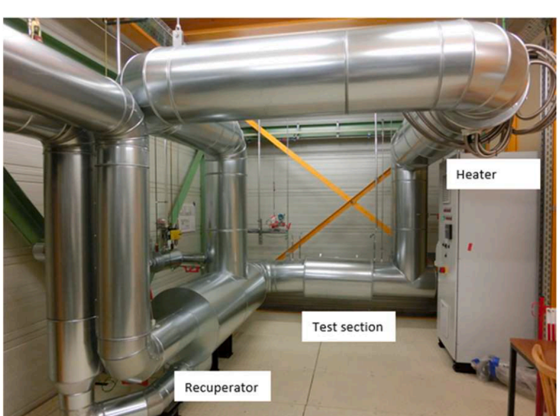

Fig. 3. High temperature part of HEMAT loop.

A mass spectrometer (GAM400, IPI GmbH, Bremen, Germany) is used twice a day to analyze the composition of the atmosphere in the loop. It is situated about 15 m away from the loop. The same as in the case of the humidity sensors, the measurements of the gas composition are done at room temperature.

2.6. Helium desiccation system HEPUR

The helium desiccation system HEPUR is integrated in the HEMAT loop in a bypass. It consists of a column filled with zeolite pebbles. When the helium atmosphere in the loop is getting too humid, i.e., at the humidity sensors a value of more than 200 ppmV is read, the valves that separate HEPUR from the main loop are opened for a period of 10 to 15 min and a part of the helium flow is directed through the bypass. The control is hand-driven. Before installation, the zeolite pebbles were heated out in a conventional oven for 120 h at 230 °C to reduce the water content.

2.7. Experimental procedure

A dedicated procedure was developed to reduce the contact time between the canisters filled with samples and pebbles and air humidity. Before opening the loop to install the test section, this is filled with argon. Argon as a heavy gas avoids the ingress of environmental air in the loop during the time it has to be opened. The test section is installed between two flanges. After closing, the loop is evacuated over night until end values of 7 to $6{\cdot}10^{-2}$ mbar are reached. Then, it is filled immediately with the test gas mixture (bought ready from Air Liquide, Deutschland GmbH, Düsseldorf, Germany) and a leak test is performed. If the helium leakage is below $1{\bullet}10^{-5}$ mbar.l/s, the loop is considered to be tight.

It takes about 1.5 days to heat up the gas atmosphere and the loop up to the required level, namely 550 °C. Once this value is reached, the temperature is kept constant for the given period, before the loop is cooled down for about 2 days to room temperature. Every 30 min, a measurement is taken on the status of the loop.

After each period, the test section is dismantled and transported to a small glove box on site, filled with argon, which was specially constructed and manufactured for this purpose. During this time, the loop is closed with plastic flanges and filled with argon, too. One canister is withdrawn and put into a container. The test section is installed back in the loop, while the container with the withdrawn canister is sent to the Institute of Applied Materials, where the thermomechanical investigations are made. Evacuation and filling of the loop is repeated, before the next experimental period is started. Removal of one canister takes less than 1 h.

3. Experimental results

3.1. Observations on the canisters

After the exposure time, the canisters show different colors, depending on the time they are aged in the loop (Fig. 4).

Also, the contact points between the canister walls made from EUROFER and the ceramic breeder pebbles are clearly visible already after 8 days exposure time, see Fig. 5.

After the experiment, the color of the pebbles turned from white to a light grey. No dust formation was observed for 8, 16 and 32 days. The situation changes for the 64 days canister, in which the presence of dust was observed. Some pebbles were baked to the material. Also, some of the pores of the filters and the side walls are then blocked. A black oxide layer was formed on the surface of the samples and the inner cylinder.

Fig. 4. View of all canisters after different exposure times.

Fig. 5. Contact points of the pebbles with the canister side wall made from EUROFER, after 8 days exposure time.

The canister walls are still permeable, as it can be seen in Fig. 6, in which one can see that the beam of a laser pointer still passes through the perforated wall.

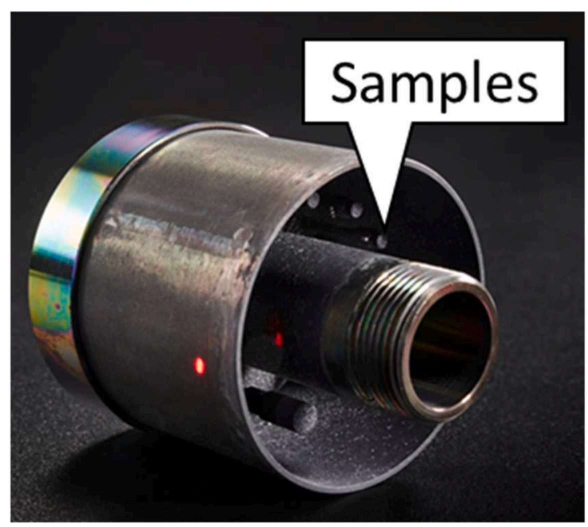

Fig. 6. The walls of canister 4 (64 days exposure time) are still permeable. Some pebble dust can be seen inside the canister.

3.2. Results of the humidity measurements

The present experiment intended to have an exposure of the samples in a low water-content atmosphere, therefore, the humidity level in the helium stream was carefully monitored.

Fig. 7 shows the measurements taken with the humidity sensors over time for run 4, 32 days. The duration of a run might not correspond to the exposure time of a canister. A canister exposed for 64 days has experienced runs of 8, 8, 16, and 32 days that are summed up to its total exposure time. At the beginning of the run, the water content is high, due to the opening of the loop.

The rising temperature has the effect to heat out the materials. The water content can be quickly reduced by using HEPUR. In the following, the content of water varies in parallel with the operation of HEPUR (about once per week, the humidity is reduced by applying the column). At the end of the run, the temperature is decreasing and the water content decreases, too. With HEPUR, it is possible to efficiently control the water content in the loop in the desired range of about 200 ppmV maximum.

3.3. Effect of the ceramic breeder environment on the corrosion layer of the fatigue samples

When comparing the samples exposed to a ceramic breeder environment in HELOKA HEMAT with samples treated under comparable conditions in a standard exposure device [3,7,8] it is found that the samples generally exhibit a difference in surface color as visible in Fig. 8.

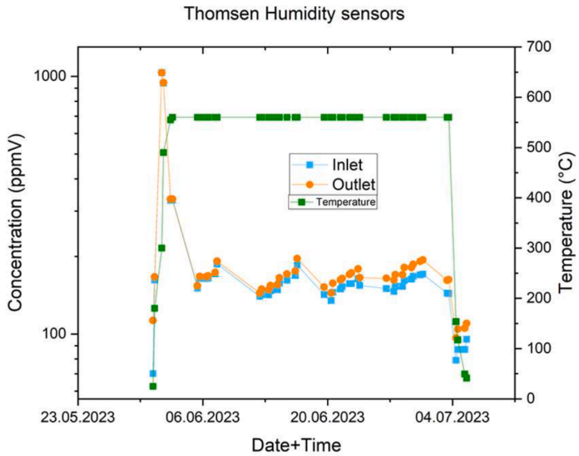

Fig. 7. Humidity measured with the Thomsen sensors.

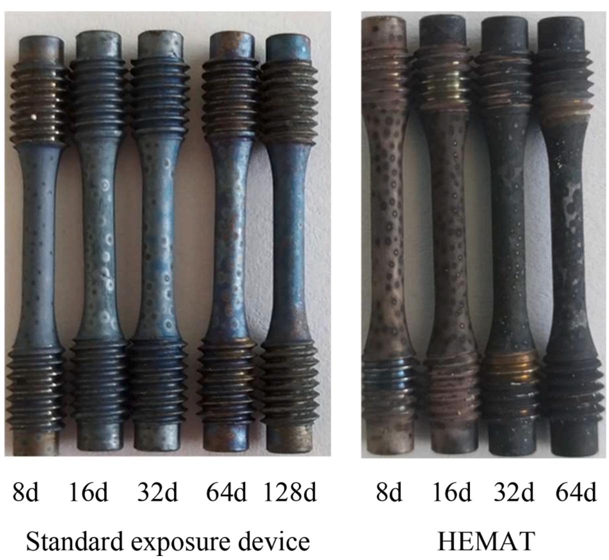

Fig. 8. Comparison of sub-sized LCF samples subjected to a ceramic breeder environment for different durations. Left: samples aged in a standard exposure device, right: samples aged in HELOKA HEMAT.

While the number of contact areas (EUROFER97 – ceramic pebble) is comparable, micro-cracks inside this so-called imprints, as observed in former exposure experiments [7,8], are not available. Fig. 9 shows a typical surface area including a contact zone.

Fig. 10 depicts details of the upper surface layer consisting of small hematite (Fe_2O_3) crystallites, enclosed by pure ferrite (α-Fe). In addition, Fig. 11 shows the cross-section of the entire corrosion layer. Compared to former experiments, performed in a standard exposure device (see e.g. [3,8]), the intermediate chrome rich layer reveals a varying thickness and the upper layer is predominantly consisting of individual crystallites and not of a more or less homogeneous ferrite film containing non-transformed hematite particles.

Furthermore, it is found that the initial growth rate of the corrosion layer in dependence of exposure times in HELOKA HEMAT is lower, than the one determined from exposure tests performed in the standard exposure device (see Fig. 12).

The observed differences show already a certain change in the corrosion behavior of the steel. When additionally comparing the results of XRD (X-ray diffraction) surface investigations performed on samples aged in a standard device (already shown in [7]) with results obtained from measurements performed on samples aged in HELOKA HEMAT,

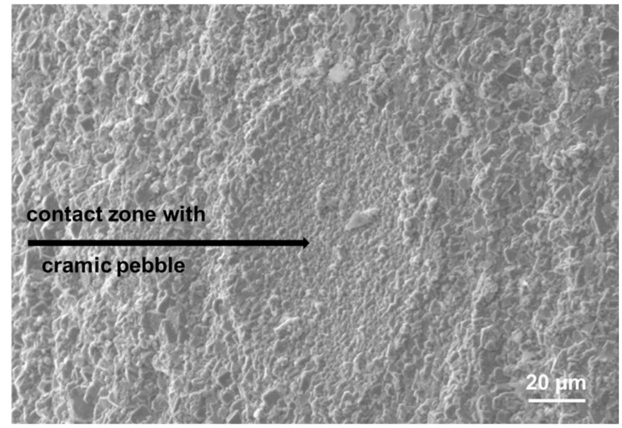

Fig. 9. SEM-SE micrograph showing the typical surface of a sub-sized LCF sample subjected for 64 days to a ceramic breeder environment in HELOKA HEMAT.

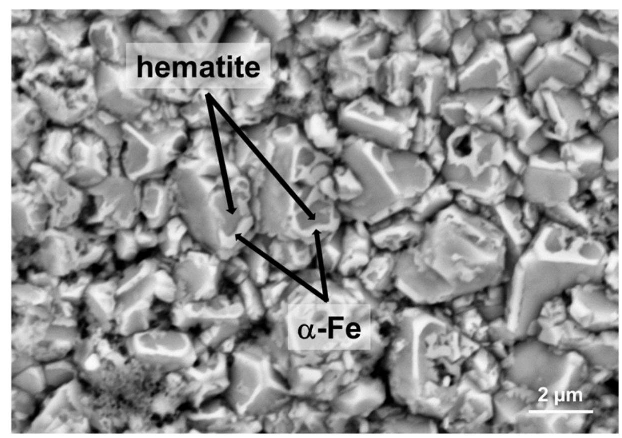

Fig. 10. SEM-BSE micrograph showing details of the typical surface of a sub-sized LCF sample subjected for 64 days to a ceramic breeder environment in HELOKA HEMAT.

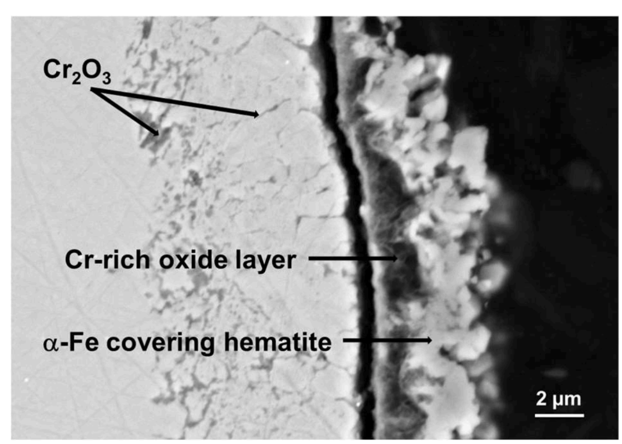

Fig. 11. SEM-BSE micrograph showing relevant details of the cross-section of a sub-sized LCF sample subjected for 64 days to a ceramic breeder environment in HELOKA HEMAT.

particularly differences in the share of iron oxide(s) and α-Fe in dependence of the exposure time are found. In Fig. 13, the results are depicted for both facilities exemplarily for aging durations of 8 days and 64 days, respectively. The differences in shape and absolute intensities

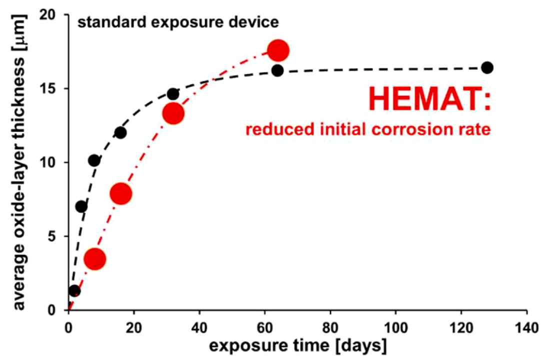

Fig. 12. Growth rate of the corrosion layer on the surface of EUROFER97 in dependence of both the exposure duration and the used test facility.

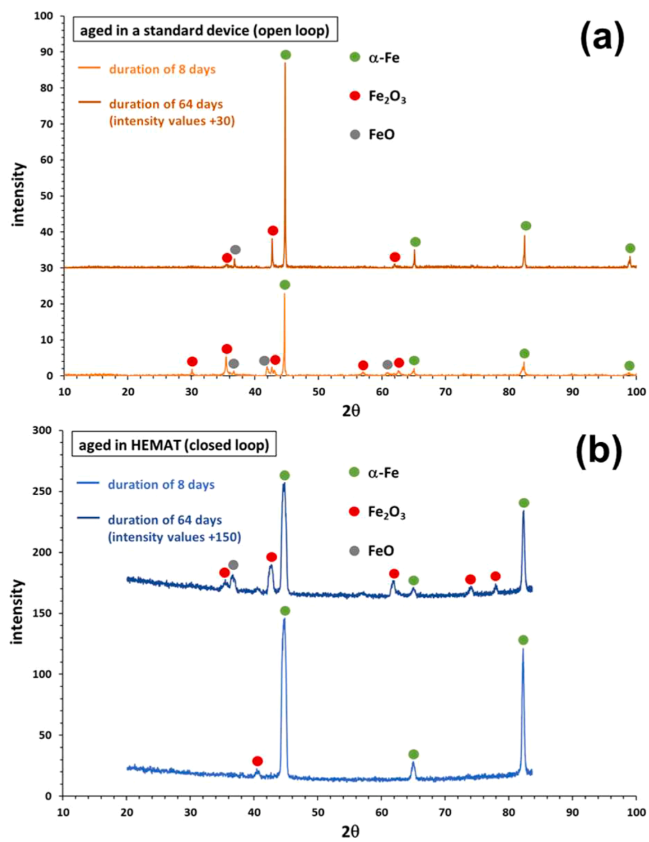

Fig. 13. Results of XRD surface investigations performed on samples aged in: (a) the standard exposure device [7] and (b) aged in HELOKA HEMAT for both 8 days and 64 days.

base upon the fact that the measurements on the samples aged in the standard exposure device were performed using a Bragg-Brentano set-up, whereas the samples aged in HELOKA HEMAT were investigated using a polycap X-ray lens with a diameter of 350 µm. It is observed that specimens aged in the standard furnace after an exposure duration of 8 days, besides the presence of ferrite reflexes, already show a lot of iron oxide reflexes. With increasing exposure duration, however, the number of oxide reflexes decreases and the intensity of ferrite reflexes increases. In other words, with increasing aging duration the share of α-Fe is increasing (Fig. 13(a)). In contrast, samples aged for 8 days in HELOKA HEMAT only show a single oxide reflex with low intensity. For longer exposure durations, both the number of oxide reflexes and the corresponding intensities significantly increase, while the intensities of ferrite reflexes decrease. So here, the share of α-Fe is decreasing with increasing aging duration (Fig. 13(b)).

The changes concerning the corrosion behavior can obviously be traced back on varying testing conditions. The standard exposure device consists of an open loop, exhibiting a continuous flow of fresh purge gas. As described in [3], the relative humidity was found to vary between < 0.1% (inlet gas) and up to 1.2% (outlet gas). Furthermore, the core components inside the heat affected zone (tubes of the furnace and canisters containing the simulated pebble bed – EUROFER97 samples embedded in ceramic pebbles) are made of alumina. HELOKA HEMAT, however, consists of a closed loop where the water content is continuously monitored and kept on a low level (see Fig. 7). Consequently, compared to samples aged in the standard exposure device, the growth rate of the corrosion layers is slowed down. Indeed, the layer thickness of the HELOKA HEMAT samples seems to increase stronger with increasing testing duration and is found to be even bigger for an exposure time of 64 days, however, one needs to consider that the outer layer predominantly still consists of single crystallites. As a result, compared to the upper layers formed in the standard furnace, the density is significantly lower (see Fig. 12).

Furthermore, the entire HELOKA HEMAT loop as well as the canisters containing the pebble beds are made of steel. One may suppose that particularly within the heat affected zone, metallic components like the canisters will show a certain hydrogen uptake. As a result, the reduced amount of hydrogen in the closer neighborhood of the specimens will inevitably lead to a decelerated reduction of the upper hematite layer to ferrite (see Fig. 13). A lower amount of α-Fe, enclosing hematite in the upper layer, may also explain the absence of micro-cracks inside the imprints even after longer exposure durations (particularly at elevated temperatures, the thermal expansion coefficient of hematite, $\alpha_{th} \sim 15 \times 10^{-6}$ 1/K, is significantly higher as the one of ferrite, $\alpha_{th} \sim 12 \times 10^{-6}$ 1/K and the one of the chrome rich intermediate layer, $a_{th} \sim 8.5 \times 10^{-6}$ 1/K). Cooling down after exposure will lead to residual tensile stresses inside hematite which will cause cracking at critical local stress conditions.

4. Conclusions and outlook

An experiment has been conducted to investigate the impact of the contact between Eurofer97 and ceramic breeder pebbles in purge gas atmosphere (helium + 0.1vol% hydrogen) at DEMO relevant operating conditions (550 °C, 1.2 bars abs.). Samples were exposed for 8, 16, 32, and 64 days, respectively. Special attention was paid to the design and the conduction of the experiment, so that the influence of the environment, in particular of air humidity, is excluded. The tests were run in the HELOKA HEMAT facility in a closed loop. It was found that an oxide layer is formed which increases with exposure time, but it is thinner than in preceding tests where the test conditions were less relevant to breeding blanket operating conditions.

The main objective of the experiment is to prepare further experimental investigations with different purge gas compositions, like He + H_2O as proposed by [4], or He + H_2O + H_2. A helium humidifier for this purpose is under construction at the moment. These mixtures are most relevant to the operation conditions of a future blanket system. Since the results of the mechanical LCF tests of the HELOKA HEMAT samples are comparable with those of the oven experiment, the results of the experiments in the loop are meaningful for DEMO operating conditions and present a step towards an experiment with steam.

CRediT authorship contribution statement

Regina Krüssmann: Investigation, Methodology, Software, Visualization, Writing – original draft, Writing – review & editing. **Bradut-Eugen Ghidersa:** Conceptualization, Methodology, Supervision, Writing – original draft, Writing – review & editing. **Mario Walter:** Conceptualization, Methodology, Resources, Supervision, Visualization, Writing – original draft, Writing – review & editing. **Thi Tra My**

Nguyen: Investigation, Visualization. **Viktoria Weber:** Investigation. **Philipp Heger:** Investigation. **Frederik Arbeiter:** Conceptualization, Investigation, Methodology. **Georg Schlindwein:** Investigation. **Guangming Zhou:** Conceptualization, Funding acquisition, Project administration. **Francisco A. Hernández Gonzalez:** Funding acquisition, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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