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Long-term corrosion behavior of ODS-Eurofer in flowing Pb-15.7Li at 550 $^{\circ}\mathrm{C}$



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ABSTRACT

Low activation ferritic-martensitic steels (RAFM-steels) are foreseen as structural materials in different blanket designs (HCLL, DCLL, WCLL) with Pb-15.7Li as breeding and partly also as cooling medium. In HCLL and WCLL designs the structural material will be in direct contact with the flowing liquid breeder at operating temperatures up to 550 °C. In the past, investigations concerning the corrosion behavior of RAFM-steels like F82H-mod. and Eurofer showed that these alloys are attacked by the flowing breeder. These corrosion tests pointed out, that the corrosion attack depends mainly on flow velocity and temperature. All these alloys were 'single' phase ferritic-martensitic steels without any additions. However, structural materials with better strength and creep resistance like ODS-Eurofer or ferritic steels are gathering interest in blanket development and will be required at least for DCLL application due to higher operation temperatures. Nevertheless, reliable data concerning compatibility with Pb-15.7Li are missing until now.

In this paper results from long-term corrosion testing of ODS-Eurofer will be reported for exposure times up to 1.5 years at a flow velocity of 0.1 m/s. The evaluated data for ODS-Eurofer corrosion will be compared with values of 'single' phase 'classical' Eurofer. The observed corrosion attack and mechanisms will be discussed in detail considering the testing conditions and the microstructure of the RAFM-steels. © 2016 The Authors. Published by Elsevier Ltd.

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1. Introduction

The performance of future fusion systems strongly depends on the operation temperature of the components. Conventional designs considered 'classical' Reduced Activation Ferritic-Martensitic (RAFM) steels (e.g., Manet, F82H-mod. or Eurofer) as structural material for fabricating of, e.g., blanket components with operation limits at about 550 °C [1,2]. In contrast, advanced designs are dealing with increased temperature limits and steels with improved strength properties, e.g., of type ODS-steels [3]. Several blanket designs, e.g., HCLL, WCLL and DCLL (Helium Cooled, Water Cooled and **D**ual **C**oolant Lead Lithium) [1] will use liquid lead lithium as breeding material. All these blanket designs have in common that the liquid breeder is in direct contact with the structural material. Thus, the applicability of the developed RAFM-steels as structural material will essentially depend on their compatibility with the liquid breeder to ensure the required reliable and safe system operation.

* Corresponding author. E-mail address: wolfgang.krauss@kit.edu (W. Krauss). 'Classical' RAFM steels were characterized concerning their corrosion stability in Pb-15.7Li environment at different testing conditions. In the beginning the early developed steels like Manet, F82H-mod., Optifer and Eurofer were examined at low temperatures (753 K or 480 °C) [3-6]. Later on, testing temperatures were increased to 823 K (550 °C) which is the envisaged operation temperature of HCCL blankets for ITER or DEMO. Corrosion attack at 480°C and a flow velocity of 0.22 m/s resulted in a moderate material loss of roughly 90 μ m per year. However, the slight increase in operation temperature by 70 K shifted the material loss to 400 μ m per year at the same flow velocity [7].

These tests showed that the dominating corrosion mechanism is dissolution of steel elements out of the matrix by the liquid breeder. All these tested 'classical' RAFM-steels exhibited a strong dependency of corrosion attack on temperature and the flow velocity. Reducing the flow velocity of Pb-15.7Li to 0.10 m/s resulted in a decline of material loss down to about 220 µm per year at 550 °C testing temperature, however, still representing an amount of roughly 2 kg of corrosion products dissolved per square meter of surfaces exposed to Pb-15.7Li. These testing conditions showed for the 'classical' RAFM-steels that corrosion is a serious issue and that corrosion products are moving with the flowing breeder and can

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form precipitates [8] which may cause line plugging. The observed corrosion attack, under the selected testing conditions in the turbulent flow regime, was found to be in agreement with physicochemically based corrosion modeling (MATLIM-code) [9]. However, experimental and modeling work analyzed only 'classical' RAFMsteels without any added oxide particles for improvement of the mechanical properties until now. Ab initio it cannot be ruled out that the compatibility of such steels with modified microstructure to Pb-15.7Li may be different compared to 'classical' RAFM-steels. Thus, a corrosion testing campaign of ODS-Eurofer was launched and performed in the corrosion testing loop PICOLO at KIT to provide data for this kind of alloy and to compare it with 'classical' RAFM-steels.

2. Experimental

2.1. Corrosion testing loop PICOLO

The corrosion testing loop PICOLO was designed for analyzing the compatibility of steels in a flowing Pb-15.7Li environment [10]. PICOLO is a loop designed for forced flow of molten Pb-15.7Li. The loop can be operated at flow velocities in the range of roughly 1 cm/s to 1 m/s in the test section. The Pb-15.7Li flow is powered by an electro-magnetic pump (EM) which is installed in the cold leg of the loop together with other essential components like a magnetic trap (MT) and a magnetic flow meter (MF). This cold leg of the loop is fabricated from austenitic steel (DIN 1.4571) and operated at a temperature of about T=623 K (350 °C) or slightly above. The hot section is manufactured from a ferritic-martensitic steel (DIN 1.4914) and consists of components like a counter flow heat exchanger (CFHE), electrical heater (EH) and the test section (TS). The test section is connected via an expansion vessel (EV) to a glove box system, which is operated under controlled and purified Ar atmosphere. Sample loading is done there by immersion of a screwed stack of samples into the test section which has a diameter of 16 mm. The cylindrical samples have a diameter of 8 mm and are positioned in concentrically arrangement in the test section. Thus, the Pb-15.7Li is flowing in a gap of 4 mm width around the samples in the test section in the direction from top to bottom. At the flow velocity of 0.10 m/s, which was chosen for the testing campaign of ODS-Eurofer, a turbulent flow regime will be present in the test section. This testing condition was selected to generate clearly visible attack of the samples also at shorter exposure times. The chosen flow velocity ensures turbulent conditions but is close to the regime of mixed flow regime (approx. 0.09 m/s). The testing campaign was set up for a whole duration of about 1.5 years to obtain reliable long term corrosion data. Fig. 1 shows the schematic view of PICOLO and the positioning of the samples in the test section. It should be mentioned that flow velocities in other components of the hot leg of the loop (e.g. EH) are much lower and that the test section is coated inside by an Al-based barrier. Thus, system corrosion should not affect the behavior of the test samples.

PICOLO loop was operated at the same flow and temperature parameters during this ODS-Eurofer campaign as at the preceding campaign testing behavior of 'classical' Eurofer. Testing was performed in Pb-15.7Li which was renewed 12,000 h earlier after a mayor revision of PICOLO loop. The fresh Pb-15.7Li was delivered by GMH Stachow-Metall GmbH, Germany and the main relevant impurities Fe, Cr, and Ni (the elements which will be enriched by corrosion products during compatibility testing) were found to be below resolution limits of about $1 \mu g/g$ detected by ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometry). Samples of Pb-15.7Li extracted from the test section after conditioning the loop for some 100 h showed impurity levels of Fe, Cr and Ni of about 6, < 3, and 30 wppm, respectively. They did not change significantly over the whole duration of two testing campaigns lasting



Fig. 1. Schematic view of PICOLO loop and arrangement of samples in the test section.

roughly 24,000 h. However, it has to be mentioned that the measured impurity values are the sum of the element concentration caused by dissolved atoms in Pb-15.7Li and the element amount in precipitates. Such particles are formed in the cooler loop sections due to oversaturation and will not be completely removed by the trapping device. They are transported with the liquid metal circulating in PICOLO. Analyzing inner sides of the loop walls showed that such precipitates may deposit anywhere in the loop there favorable conditions exist like magnetic fields or low flow velocity, e.g., in the electrical heater component. Particles found in this hot section [8] may be an indication that they will enter the test section some centimeters away. The Ni concentration observed in the extracted Pb-15.7Li indicates that corrosion is present at all loop sections also at cooler parts which are operated in the temperature range of 350 to 400°C.

2.2. Samples for corrosion testing

The samples for corrosion testing in PICOLO loop were fabricated from plate material with orientation along the rolling direction of the raw material. In the first processing step rods were cut out of the center of 15 mm thick plates by EDM (Electro Discharge Machining) and the final shape was given by turning in the second



Fig. 2. Samples used in corrosion testing fabricated from 'classical' Eurofer and ODS-Eurofer with cylindrical shape and assembled to the test stack.

step. The dimensions of the fabricated 'classical' Eurofer and ODS-Eurofer samples were 8.0 mm in diameter with an overall length of about 35 mm. The samples were equipped with 6 mm male and female threads for assembling them to a stack of samples. Fig. 2 shows virgin samples made of both RAFM-steels and the assembled stack together with the flow orientation of Pb-15.7Li in the test section. The surface of such samples machined by turning exhibited a surface roughness of about $R_a = 0.5 \,\mu m$. Atomic Emission Spectroscopy analyses (AES) indicated that a very thin surface scale is present after removing typical manufacturing impurities by ultrasonic cleaning. One kind of contamination is of absorbed CH_x molecules. The other component is oxygen. The thickness of this oxide scale is in the range of up to about 5 nm. It is a Fe-oxide with a small amount of Cr. The chemical composition of the used RAFM-steels Eurofer and ODS-Eurofer were analyzed by ICP-OES. Both steels had rather similar composition concerning the metallic components (e.g., Cr \sim 9%, W \sim 1%, Ta \sim 0.1%) and the ODS particle concentration was about 0.3% Y2O3. More detailed data on the composition of samples tested in PICOLO loop are given in [13] and the processing of ODS-Eurofer inclusively of the microstructural characterization can be found, e.g., in [3,11,12].

3. Results

3.1. Corrosion testing

The compatibility testing of ODS-Eurofer and 'classical' Eurofer was performed in PICOLO loop at the flow velocity of 0.1 m/s. The testing temperature was 550 °C (823 K) with a deviation of smaller than 2 K. The flow velocity was controlled and adjusted by variation of the pumping power to be in a $\pm 5\%$ range with short time deviations not exceeding \sim 10% to obtain mostly constant test conditions. The samples designed for long term exposure (e.g., 11,000 h) were mounted at the top of stack i.e. at the Pb-15.7Li entrance into the test section. The first sample at the bottom of the test section was an ODS-Eurofer sample followed by a specimen made from 'classical' Eurofer. However, the quantity of 'classical' Eurofer samples was kept at a minimum. This type of samples was used as reference material to allow a reliable comparison with corrosion data from the proceeding campaign. Samples for short time exposure were positioned at the bottom end of the stack and removed samples were replaced by fresh ones.



Fig. 3. Appearance of samples made from 'classical' Eurofer and ODS-Eurofer after removal from the test section with adherent Pb-15.7Li layer.

3.2. Sample preparation for corrosion analyses

Samples were removed from PICOLO loop for analyzing microstructural effects and for measuring the material loss by corrosion. Fig. 3 shows samples after extraction from the loop covered by an adherent Pb-15.7Li layer. The removed samples are cut by sawing for these different analytical objectives. The cutting positions were chosen to be constant for all samples. The smaller piece indicated by (a) is used for optical and SEM / EDX analyses (Scanning Electron Microscopy / Energy Dispersive X-ray analyses). The longer bottom part (b) is designed for diameter measurements after cleaning the surface by etching.

3.3. Corroded surfaces

The first ODS-sample was removed after 1000 h testing time from PICOLO loop. This sample showed clearly visible traces of corrosion attack. However, this attack may be interpreted as type of strong surface roughening at some areas but not at all surface parts. Fig. 4 shows the cross sections of both alloy types after exposure for 2000 h to Pb-15.7Li. The micrographs show that all areas of the sample are well wetted by the breeder Pb-15.7Li and that corrosion is affecting all areas. However, the surfaces exhibit clearly visible elevations which indicate that delayed attack took place due to incubation effects. This short term behavior is well known from tests performed earlier also at higher flow velocity [6,7]. Micrographs of longer exposed samples, e.g., 3000 h, showed that these elevations are smoothed out. SEM analyses showed that corrosion products (e.g., Fe, W) are present in the adherent Pb-15.7Li layer.

The longer term exposed samples showed that the plateau like elevations are disappearing and that smooth surfaces are formed as it can be seen in Fig. 5. The micrographs presented show for both alloys the ferritic-martensitic structure (FM) and due to magnification by optical microscopy no ODS particles. In general the surface structure of the analyzed samples did not show any significant differences in roughness and global appearance. No washing out of FM needles was seen and rather smooth surfaces are present which may indicate that ODS particles have no or less impact on corrosion attack. All these features indicate that the dominating corrosion mechanism is dissolution. Optical microscopy and SEM/EDX analyses indicate that under this testing conditions ODS-Eurofer and 'classical' Eurofer steels behave very similarly by inspecting the surface appearance of the contact zone to the flowing breeder.



Fig. 4. Cross sections of ODS-Eurofer and 'classical' Eurofer samples exposed for 2000 h to flowing Pb-15.7Li with adherent layer.

3.4. Corrosion attack

The corrosion attack was determined from diameter reduction. The diameter of the fresh test samples was measured by using a laser technology with a resolution of ca. 1 μ m. The samples were rotated to determine the diameter in dependence on radial positions and moved in axial position to measure diameter versus length of the sample. Two different methods were used to evaluate the diameter of the corroded samples as illustrated in Fig. 6.

Method one used the cross sections processed for microstructural analyses and the diameter was measured under a microscope by laterally moving the cut from one edge to the other by means of a micrometer step controller. The second method was the laser technology. Before measurements, the Pb-15.7Li crust was removed from the specimens by gently etching in a solution of acetic acid, ethanol and hydrogen peroxide to prevent attack of the matrix. Both methods determined the diameter in dependence on radial positions. The evaluated values for one special axial position are the arithmetic average of all measured radial values (step angle $\Delta
ho = 30^{\circ}$) at that position. This value will be used for evaluating the corrosion attack vs exposure time. Fig. 7 shows the corrosion behavior of ODS-Eurofer in dependence on axial and radial position for long term exposure to Pb-15.7Li. This sample was positioned slightly above the center of the test section and should thus represent most reliably corrosion behavior under undisturbed turbulent flow conditions. The first general information from this figure is that a significant reduction of the diameter took place. A reduction of roughly 0.5 mm was observed after an exposure of 10,000 h. The more detailed view shows that corrosion attack in the center part of the sample, which is unaffected by machining (thread cutting,



Fig. 5. Etched cross sections of ODS-Eurofer and 'classical' Eurofer samples exposed for about 10,000 h to Pb-15.7Li.



Fig. 6. Schematic view of measuring techniques for diameter determinations in dependence on radial and axial positions.

flats for wrenches), is rather constant (constant diameter vs axial position for one specific radial value). The second information is that the corrosion attack of this concentrically positioned sample in the Pb-15.7 flow shows very low fluctuation with radial position. The deviation in roundness do not exceed 50 μ m or 10% compared with the totally removed layer thickness. Thus, Fig. 7 points out that homogeneous corrosion attack is present also for ODS-Eurofer as stated earlier for 'classical' Eurofer [6,7,13].



Fig. 7. Corrosion attack in dependence on radial and axial position of ODS-Eurofer after 10,000 h.

The corrosion behavior in dependence on the radial position is shown in Fig. 8 for both steels analyzed under this testing campaign. The diagrams depict the virgin diameters (upper curves) and the diameters after corrosion attack (curve in center) of ODS-Eurofer and 'classical' Eurofer. Visible is that both steels undergo a reduction of diameter due to corrosion attack. The reduction in diameter is rather similar for both steels with about 70 µm. Both attack samples show a scattering of the diameter in dependence on radial position around an average value of similar extent. This 'type' of roughness is a contribution to incubation effects at this early stage of exposure to flowing Pb-15.7Li. The diagram shows as general information that both samples exhibit similar behavior under these corrosive conditions.

3.5. Corrosion rate

The corrosion behavior was analyzed for both steels in the PI-COLO loop at flow velocity 0.1 m/s. The longest exposed samples remained for approx. 11,000 h in the PICOLO loop. Under this testing campaign most of the exposed samples were of type ODS-Eurofer. Only a limited number of 4 other samples were analyzed under this campaign to provide reference data for comparison with earlier performed corrosion campaigns. The first ODS-Eurofer sample was removed after about 1000 h.

The longest exposed ODS-Eurofer sample stayed in the test section for roughly 11,000 h just as a 'classical' Eurofer sample. Beyond this pair of samples two other pairs were exposed for direct comparison one removed at 2000 and the other at 4000 h, respectively. The corrosion rates evaluated for all samples exposed to Pb-15.7Li under this campaign are shown in Fig. 9. The corrosion rate is given as reduction of the radius of the sample and represents thus directly the wall thinning due to dissolution / corrosion process. The values are normalized to an exposure time of 1 year. The three pairs of samples show rather similar behavior in direct comparison of corrosion rates. This indicates that ODS-Eurofer and 'classical' Eurofer exhibit similar compatibility behavior. The corrosion rates evaluated for ODS-Eurofer in the exposure range of 1000-11,000 h are found to be near the value of about $200\,\mu m/year$. The scattering within a band of up to 10 % of the evaluated value is caused by different factors ranging from, e.g., position effects in the test section to sample preparation effects. 'Classical' Eurofer samples analyzed in a previous campaign showed similar values and fit very well to the observed corrosion rates of ODS-Eurofer. The previous and actual data set for 'classical' Eurofer indicates that the same testing conditions were present over the whole test duration. Thus a sufficient high reliability is given in the face of the limited number of tested ODS-Eurofer samples that corrosion attack is near 200 µm/year and that both steel types behave similarly.

Fig. 10 shows the corrosion attack in dependence on the exposure time. Beyond the actual test data evaluated for ODS-Eurofer, this diagram gives also values for 'classical' Eurofer tested at different flow velocities. The values obtained for both shown testing velocities indicate most reliably a linear correlation between corrosion attack and exposure time. The corrosion rate at a flow velocity of 0.1 m/s is clearly lower than the rate evaluated at 0.22 m/s, which was about 400 µm/year. This corrosion rate of 'classical' Eurofer is very reliable due to similar behavior of other earlier tested RAFM-steels [6]. This correlation indicates that the corrosion process is mainly governed by a dissolution reaction as earlier pointed out [4-7,9]. The sequence of data points obtained for ODS-Eurofer fit very well to corrosion values of 'classical' Eurofer for the flow velocity 0.1 m/s. Most of the data points are near to the estimated slope line indicating material losses of 200 µm/year. An additional argument for the reliability of the obtained value of 200 µm/year is the fact that it is about half of the values observed at a flow velocity of 0.22 m/s. Only a very limited number of samples exposed rather long (~12,000 h) show a relatively high material loss



Fig. 8. Corrosion attack in dependence on radial position of ODS-Eurofer and 'classical' Eurofer after short exposure time of about 2000 h.



Fig. 9. Corrosion attack in dependence on exposure time for ODS-Eurofer and 'classical' Eurofer.



Fig. 10. Comparison of corrosion rates at flow velocities 0.22 and 0.1 m/s and of ODS-Eurofer with 'classical' Eurofer.

compared to all other samples. This may be caused by local effects, e.g., mounting near to the upper end of the test section or by replaced virgin samples in front causing higher turbulence. It has to be mentioned that flow velocity at 0.1 m/s is near to the transition zone from turbulent to mixed flow regime. Considering such local and equipment specific details an average corrosion rate of $200 \,\mu\text{m/year}$ seems the most reliable one.

4. Conclusions

The corrosion testing campaign at 0.1 m/s was successfully performed up to approx.. 11,000 h of exposure time. Beyond determining the compatibility behavior of ODS-Eurofer this campaign increased also the limited data base of test results obtained at 0.1 m/s flow velocity and testing temperature of 550 °C and led to an increase of reliability for these data The total number of tested ODS-Eurofer samples is small and no statistics exist at the moment. The correlation with the results obtained from earlier RAFMtests will replace this lack. Additionally, the rather constant reaction of the samples over the whole test campaign has to be valued together with the microstructural behavior. All these facts imply that the corrosion rate evaluated is well anchored with the value of about 200 µm/year. The comparison of ODS-Eurofer with 'classical' Eurofer showed that the ODS particles have no visible impact on corrosion rate under the applied testing conditions and that reactions in the contact zone due to Pb-15.7Li are not changed i.e. dissolution remains the dominating and corrosion controlling mechanism at 0.1 m/s flow velocity. The observed material loss can be classified as homogeneous attack in the flow direction. It was also found that the cylindrically shaped test samples did not lose their roundness. However, the corrosion tests also showed that small variations in the flow profile e.g., higher turbulence or modified flow velocity generate a response. This issue is visible in variations of the mass loss around the given average value of 200 µm/year. The new results of ODS-Eurofer corrosion testing in PICOLO loop are in line with earlier tests of RAFM-steels performed in PICOLO [6,7,13]. They indicate that material loss also causes a similar amount of corrosion products for this steel with improved mechanical properties with the same risks shown earlier [8]. Surely, in a future fusion system flow velocities will be smaller and thus also the production of corrosion products has to be scaled down but mass loss will not vanish and has to be controlled and managed.

This will imply – T-permeation is the same concern for ODS-Eurofer as for 'classical' Eurofer due to the same matrix – that corrosion barriers (with simultaneous positive impact on Tpermeation reduction) have to be considered also for ODS-Eurofer applications in future development to manage corrosion issues.

The large number of corrosion campaigns performed in PICOLO loop will lead to the general result that the type of the FM-steel (Manet, 'classical' Eurofer, CLAM-steel, ODS-Eurofer) has no or minimal impact on corrosion attack as long as they exhibit similar microstructure and composition. However, their behavior may change and diverge under varied testing / application conditions (e.g., laminar flow, back flow) or, e.g., changed impurity levels.

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