POLYTECHNIC UNIVERSITY OF BUCHAREST

FACULTY OF ENERGY

DOCTORAL SCHOOL OF ENERGY

PH.D. THESIS SUMMARY

CONTRIBUTIONS TO THE STUDY OF STRUCTURAL MATERIALS CANDIDATES FOR GENERATION IV NUCLEAR REACTORS

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THE CONTENT OF THE THESIS

Keywords:

Generation IV nuclear reactors, SCWR, structural materials, austenitic stainless steels, nickel-based alloys, oxidation, gravimetric corrosion tests, SEM-EDS, XRD.

LIST OF ABBREVIATIONS

ACR - Advanced CANDU Reactor
ASTM - American Society for Testing Materials
BSE - Backscattered Electron Detector
BWR - Boiling Water Reactor
CANDU – CANadian Deuterium Uranium
CNE –Nuclear Power Plant
EDS - Energy Dispersive Spectrometer
FESEM - Field Emission Scanning Electron Microscopy
GIF - Generation IV International Forum
IC - Intergranular Corrosion
JCPDS = Joint Committee on Powder Diffraction Standards
ODS - Oxide Dispersion Strengthened Alloys
PDF - Powder Diffraction Files,
PHWR - Pressurized Heavy Water Reactor
PWR - Pressurized Water Reactor
RIR - Reference Intensity Ratio
SCC - Stress corrosion cracking
SCW - SuperCritical Water
SCWR - SuperCritical Water-cooled Reactor
SE - Secondary Electrons
SEM - Scanning Electron Microscopy
XRD - X-Ray Diffraction

1. INTRODUCTION

1.1 Motivation

The selection of suitable candidate materials is a key issue for the development of the supercritical water cooled nuclear reactor (SCWR). Designing or choosing the most suitable materials means better durability, economy and safety. Because supercritical water (SCW) is a very aggressive corrosive environment, corrosion becomes a difficult problem for materials used in SCWR.

The corrosion performance of SCW candidate materials can be affected by several factors, including material composition and structure, SCW temperature and pressure, water chemistry, and exposure time. Several investigations were conducted to study the corrosion behavior of different candidate materials in different SCW environments, aiming to find the most suitable materials, as well as an effective method of corrosion control in SCW.

Austenitic stainless steels and nickel-based alloys have been considered as candidate materials for use in SCWR [1, 2, 3, 4] Efforts have been made to evaluate their corrosion performance in SCW media [5 , 6, 7]. However, further complex studies on the corrosion behavior of austenitic stainless steels and nickel-based alloys are still needed.

1.2 Scope and objectives

This research focuses on investigating the oxidation behavior of austenitic stainless steels and nickel-based alloys in the SCWR environment. Also, to investigate the morphological and structural changes of oxide films, analyzes were performed by advanced characterization techniques: corrosion tests by gravimetric analysis, Optical Microscopy, Scanning Electron Microscopy coupled with X-ray Spectrometry with Energy Dispersion (SEM-EDS) and X-ray Diffraction (XRD). The main objectives of this study are the following:

- Carrying out oxidation tests of austenitic stainless steels and nickel-based alloys in supercritical water conditions;
- Investigation of the oxidation behavior of austenitic stainless steels and nickel-based alloys by advanced characterization techniques;

- Evaluation of the influence of the exposure time, on the oxidation behavior of some austenitic stainless steels and of some nickel-based alloys in supercritical water conditions;
- Identification of materials with the best characteristics that could be used as internal components in future SCWR type reactors, from Generation IV.

2. ORGANIZATION OF THE THESIS

The doctoral thesis entitled ***"Contributions to the study of candidate structural materials for generation IV nuclear reactors"*** was structured in 6 chapters whose content is summarized in the following paragraphs as follows:

Chapter 1 presents the motivation for completing the doctoral thesis along with its purpose and objectives.

Chapter 2 is a documentation of the current state of the art in the study of the concepts of Gen IV supercritical water cooled reactors and the study of candidate materials for them. Following the documentation, austenitic stainless steels and nickel-based alloys were considered to be among the most interesting for the proposed purpose.

There are basically three directions for research and development of materials for SCWR:

1. Selection of existing materials that are most expected to meet SCWR requirements;
2. Development or design of new materials that are expected to have better characteristics than existing ones;
3. Modify existing materials to improve their characteristics.

The mechanical properties and corrosion resistance of stainless steels and nickel-based alloys make them viable options for reactor components exposed to high temperatures and pressures. Austenitic stainless steels and Ni-based alloys are already widely used in the design of nuclear power plants and are considered materials of interest for fuel sheaths and basic components in the proposed SCWR [26].

To date, several research papers have been published on the corrosion behavior of various materials, including stainless steels and Ni-based alloys, in supercritical water conditions [27, 28, 29, 30, 31, 32, 33, 34]. *Table 1* below shows the alloy classes studied under SCWR conditions.

The choice of an alloy that will maintain the integrity of the passivation layer under SCW flow conditions is an essential requirement for selecting materials for fuel sheath, pressure tubes and other radiation-exposed components [35].

Therefore, it is crucial to properly identify the mechanisms of processes that affect the lifetime of candidate materials under relevant conditions through extensive experimental tests [36, 37, 38, 39, 40].

Table 1 Alloy classes studied under SCWR conditions

Alloy Class	Temperature (°C)	Water chemistry	Exposure time (h)
Austenitic SS	290-650	DO from deaerated to 8 ppm	24-3000
Ni-base	290-600	DO from deaerated to 8 ppm Conductivity <0.1 mS/cm	24-3000
Ferritic-Martensitic	290-650	DO from deaerated to 8 ppm Conductivity <0.1 mS/cm	100-3000
Oxide Dispersion Strengthened steels	360-600	25 ppb DO	200–3000
Zr-base	400-500	Deaerated DO Conductivity <0.1 mS/cm	<2880
Ti-base	290– 550	8 ppm DO Conductivity 0.1 mS/cm	500

Chapter 3 described aspects and peculiarities regarding oxidation and corrosion in supercritical water, types of corrosion and parameters that influence corrosion and methods of physico-chemical analysis for the evaluation of corrosion behavior in SCW.

The methods of physico-chemical analysis by which the selected materials were evaluated, regarding the corrosion behavior in supercritical water, are the following:

- Gravimetric method
- Metallographic examination
- Characterization by scanning electron microscopy (SEM)
- Characterization by energy dispersive X-ray spectrometry (EDS)
- X-ray diffraction characterization (XRD)

Chapter 4 presents the materials selected for this study, the preparation of the samples, their test conditions, the equipment used and the analyzes performed.

As potential materials for Gen IV SCWR, I chose for this research four materials ***(OL 304L, OL 310, Incoloy 800HT and Inconel 718),*** because they have an optimal balance of structural strength and corrosion resistance required for applications in thermal power plants at high temperatures . They can be used for fuel sheaths and basic SCWR components. The chemical composition of the tested materials is given in the table below (*Table 2*).

Tabele 2 The chemical compositions of the materials tested according to the bulletins issued by the supplier

Alloy/chemical elements	**C**	**Al**	**Si**	**Mn**	**P**	**S**	**Cr**	**Ni**	**N**	**Cu**	**Mo**	**Co**	**Ti**	**Nb**	**Fe**
304L	0.03		0.46	1.37	0.028	0.0006	**18.07**	**8.11**	-	-	-		-		71.9
310S	0.063		0.71	1.61	0.016	0.001	**24.13**	**19.03**	-	-	-		-		54.34
Incoloy800HT	0.077	0.51	0.34	0.71	-	<0.001	**20.06**	**31.22**	-	0.41	-	0.61	0.52		45.19
Inconel 718	0.028	0.48	0.07	0.06	0.005	<0.001	**19.03**	**53.61**	-	0.02	3.05	0.16	1.04	5.5	17.2

Samples were taken in parallelepiped form from each material being delivered from Outokumpu Stainless AB Company [98]. Two and three samples of each material were prepared for each test. This represents a total of 10 samples for each test condition. The actual tests were performed in aqueous environment, under the specific conditions of the existing primary circuit in the SCWR reactor by autoclaving in demineralized water, in two static autoclaves, which operated at 550°C and 25 MPa pressure, parameters characteristic of SCWR systems.

Each sample, before exposure in aqueous environment at 550°C (25 MPa), was cut from the selected material in the delivery state, mechanically sanded on abrasive paper of different granules (# 600, # 800, # 1200) and then with diamond paste until metallic luster. After this process, the samples were coded for easy identification throughout the tests and exposed to the acetone ultrasound for 30 minutes. After ultrasound they were dried and weighed to an analytical balance with an accuracy of ± 1x10^{-4}g.

In order to obtain the oxide films, the samples were exposed in demineralized water, in two static autoclaves of 1l at a temperature of 550°C for different periods of up to 70 days. The scheme of the autoclave used can be seen in the figure below *(Fig. 1)*.

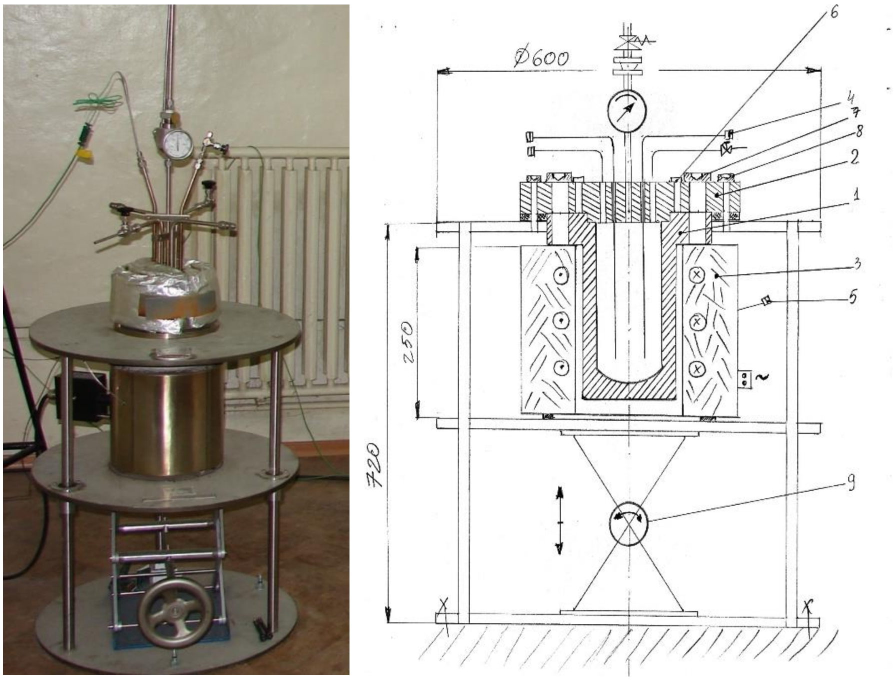

Fig. 1: 1l autoclave for supercritical temperatures and its scheme: 1-Autoclave body; 2-Autoclave lid; 3-Furnace; 4-Enclosure thermocouple; 5-oven thermocouple; 6-Detachment screw, 7-Autoclave cover-body tightening screw, 8-Autoclave cover-body fixing screw; 9-Jack down - oven lift.

The pH of the demineralized water in which the samples were exposed, measured at room temperature was about 6.7 (at high temperatures the pH decreases, reaching values around 4.5). The pressure worked was 25 MPa. To work with an oxygen content of approximately 2 ppm, at each autoclave exposure, oxygen was removed from the water by thermal degassing when the water reached a temperature of 100°C.

Analyzes / characterization and equipment used

During the autoclave testing, uniform corrosion of the samples was monitored by the gravimetric method; this consisted in determining the mass of the samples by weighing using a balance having an accuracy equal to (± $1x10^{-4}$) g, in order to establish the mass variations over time, every 10 days, after removal from the autoclave, washing and drying. The results obtained represent the mass gain of the materials.

Based on the initial weighings and those obtained after autoclaving, the variation of the mass ΔW in [mg], (final W-initial W) was determined, which was related to the area calculated in dm2, of the samples. In this way, the variation of the mass per unit area of the ΔW / S sample (mg /dm^2) was determined, which in relation to the exposure time, expressed in days, led to the corrosion rate in mg / dm^{-2}zi^{-1}.

The metallographic analysis was performed using the OLYMPUS BX51M optical microscope, which highlighted the structural constituents of the samples obtained from the tests performed in autoclaves. For this purpose, three samples from each material were selected. In the case of 304L and 310S austenitic stainless alloys, a comparative study was performed on bulk cross-sectional images, starting from the standard sample and continuing with the tested samples for 10 and 63 days, respectively. The metallographic study was performed in reflected light, at magnification of 500 ×, following electrolytic etching with oxalic acid.

Surface morphology of the samples after exposure were characterized by Scanning Electron Microscopy coupled with Energy Scattered X-ray Spectrometry (SEM-EDS) and X-Ray Diffraction (XRD).

Scanning Electron Microscopy coupled with Energy Scattered X-ray Spectrometry (SEM-EDS) were performed with Hitachi SU5000 Field Emission Scanning Electron Microscopy *(Fig. 2)*, at the Regional Research-Development Center for Innovative Materials, Processes and Products for the Automotive Industry (CRC&D-Auto) in the Advanced Materials Electron Microscopy laboratory within the University of Pitești.

Fig. 2 Hitachi SU5000 scanning electron microscope

XRD measurements were performed on a Rigaku Ultima IV diffractometer *(Fig.3)*, at the Regional Research-Development Center for Innovative Materials, Processes and Products for the Automotive Industry (CRC & D-Auto) in the Laboratory of Advanced Technical Materials with X-Radiation within University of Pitesti using Cu radiation and Bragg-Brentano geometry. For qualitative phase analyzes, X-ray diffraction spectra were acquired in Bragg-Brentano geometry, in the angular range 2θ (18^0-120^0), with a measuring step width of 0.05^0/min, scanning speed 1^0/min, acceleration voltage 40 kV, filament current 30 mA. Also, on the selected samples, quantitative phase analyzes were performed by the Reference Intensity Ratio (RIR).

Fig. 3 Rigaku Ultima IV Diffractometer

The net advantage of the equipment is the reduced time of purchase and use a small amount of sample.

Chapter 5 focused on the results of analyzes performed on the oxidation behavior of austenitic stainless steels and nickel-based alloys tested in supercritical water. The analyzes were performed by gravimetric method, Optical Microscopy, Scanning Electron Microscopy coupled with Energy Scattered X-ray Spectrometry (SEM-EDS) and X-ray Diffraction (XRD). The results of the analyzes were discussed in detail.

The last chapter, **chapter 6** presents the conclusions.

3. CONCLUSIONS

In the thesis we investigated the oxidation behavior of four materials, two austenitic stainless steels (304 L and 310 S) and two nickel-based alloys (Incoloy 800 HT and Inconel 718), in the simulated environment of the supercritical water-cooled nuclear reactor. generation IV. The testing of the materials was performed by autoclaving in supercritical water at a temperature of 550^0C and a pressure of 25 MPa at different periods of up to 70 days in order to contribute to the selection of the best structural materials in order to achieve generation IV reactors cooled with water. supercritical.

To investigate the morphological and structural changes of oxide films we performed analyzes by advanced characterization techniques: corrosion tests by gravimetric analysis, Optical Microscopy, Scanning Electron Microscopy coupled with Energy Scattered X-ray Spectrometry (SEM-EDS) and X-ray diffraction (XRD).

Corrosion rates for selected materials exposed to supercritical water were determined by weight gain over a period of up to 70 days. Low corrosion rates from 12 to 0.5 mg $dm^{-2}zi^{-1}$ for 304L, identified as the highest of the four materials tested and from 5 to 0.1 mg dm^{-2} day^{-1} for 310S identified as the smallest, demonstrated that 310S has a better corrosion behavior in the SCWR nuclear reactor environment. It was also found that the 310S, Incoloy 800 HT and Inconel 718 materials keep their values close, which represents a better corrosion behavior in the SCWR environment.

SEM images on 304L after being tested for 63 days in SCW show first separate islands until the formation of the oxide film covering the entire steel surface, which is in line with the weight gain results. The process of increasing the oxide layers during exposure can be described as an initial nucleation of the oxide in the selected sites, followed by a uniform growth of the oxide crystals until they connect with each other, resulting in a compact layer. EDS analysis indicates increased concentrations of Fe and O suggesting that the oxide formed on the surface of 304L steel could be iron oxide (Fe2O3). High values of weight gain and excessive formation of iron oxide on the surface may represent a low corrosion performance of 304L steel compared to other materials.

From the qualitative and quantitative phase analysis by X-ray diffraction, for 304L exposed in supercritical water for 63 days, two phases were identified: (CrFeMnFe) Cr0.193 Mn0.016 Fe0.697 Ni0.094 and (magnetite) Cr0.03 Fe2.96 Ni0.01 O4. The reference intensity ratio (RIR) was determined by the concentrations in complex mixtures. Observe the highest concentration (approximately 91%), represented by the phase Cr0.03 Fe2.96 Ni0.01 O4 (magnetite), followed by the concentration of the phase Cr0.193 Mn0.016 Fe0.697 Ni0.094 (approximately 9%) .

From the SEM analyzes of the 310S it is observed that after 63 days of testing under simulated SCW conditions, the oxide layer (observable at a magnification of x30k) remained thin and stable. EDS analyzes showed high concentrations of Fe and O (ie Fe2O3 formation) along with small amounts of Cr and Mn, which could suggest the formation on the surface of the chromium oxide (Cr2O3) sample or even a mixed type of oxide spinel. (Fe2CrO4).

By X-ray diffraction on 310S, exposed in supercritical water for 63 days, three phases were identified: Cr0.193 Mn0.016 Fe0.697 Ni0.094 (CrFeMnNi), Cr0.03 Fe2.96 Ni0.01 O4 (Magnetite) and Cr2 O3. The reference intensity ratio (RIR) determined the highest concentration (approximately 81%), represented by the phase Cr0.03 Fe2.96 Ni0.01 O4 (magnetite), followed by the concentration of the phase Cr0.193 Mn0.016 Fe0 .697 Ni0.094 (about 10%) and Cr2O3 about 8%.

It can be said that 310S steel has better corrosion resistance in supercritical water due to its high chromium content which leads to the development of a protective layer rich in chromium oxide (Cr2O3) formed during oxidation at high temperatures in SCW on the surface of the material.

SEM results on Incoloy 800HT after oxidation for 70 days in SCW showed two types of areas with different colors developed on the surface. The morphology of the particles found in both areas is very similar, there is a uniform growth of oxide grains until they connect, resulting in a compact layer. The results of the EDX analysis indicated high percentages of iron and oxygen suggesting the presence of magnetite (Fe3O4). The other oxides that appear on the surface of Incoloy 800HT tested for 70 days are considered to be chromium oxide (Cr2O3) and / or spinel oxide of NiCr2O4 type, because a higher concentration of chromium was detected.

From the qualitative and quantitative phase analysis for Incoloy 800HT exposed in supercritical water for 70 days of this sample, three phases were identified: Cr0,10Fe0.65Ni0.25 (CrFeNi), Ni0.4 Fe2.6 O4 and Cr2 O3 . The reference intensity ratio (RIR) determined the highest concentration (approximately 50%), represented by the Cr2O3 phase followed by the phase concentration Cr0,10Fe0.65Ni0.25 (CrFeNi), (approximately 39%) and Ni0. 4Fe2.6O4 (approximately 10%).

On the surface of the Incoloy 800HT alloy exposed to supercritical water, in the oxide films formed, even higher concentrations of Cr2O3 have been identified, indicating a more stable protective layer that effectively prevents the external diffusion of the alloying elements and the internal diffusion of oxygen ions. thus the corrosion rate of the substrate.

From the SEM analyzes of the Inconel 718 surface, it is observed that the polyhedral crystallites are distributed in the form of islands on a thin oxide scale, as in the case of the 800HT alloy, unless these crystallites are smaller. The surface EDX analysis indicates high percentages of O, Fe, Ni, Cr in the oxides developed as islands on the surface of the Inconel 718 samples. From the mapping it appears that Fe, Nb and O are concentrated inside the island area, while Ni and Cr are find nearby.

From the qualitative phase analysis for Inconel 718 exposed in supercritical water for 70 days of this sample, three phases were identified: Fe2 Ni O4, Cr2 O3 and Nb2 O5. From the analysis of the overlapping diffraction peaks, a systematic displacement is observed after the angle 2θ, to the left (the angle θ decreases), it results that following the oxidation tests appear in the oxide layer, on the identified peaks, tensile stresses which can be associated with a decrease in surface hardness (decrease in the density of atoms in the surface).

The study shows that 310S austenitic stainless steel and Incoloy 800HT nickel-based alloy have good corrosion resistance in supercritical water due to the high chromium content which leads to a more stable layer formed on the surface which makes them favorable in order to proposed.

In the field of the study, some recommendations based on the current contribution in this study are:

- From the literature, oxide layers deposited by oxidation tests usually form a multilayer. In order to highlight such multilayer structures, the research activities carried out so far can be continued with SEM-EDX analyzes in section;

- Due to limited experimental data, as autoclaving tests require long test periods, it is recommended to perform supercritical exposure tests in installations such as corrosion loops;

- It is desirable that the test method be able to simulate as many of the actual parameters as possible in the circuits in which the components made of the selected alloys in the SCWR nuclear reactors operate.

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