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Romania's contribution to the development of the knowledge society in the field of nuclear energy research

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Abstract. Institute for Nuclear Research Pitesti (INR) is the main responsible R&D (Research and Development) organization for the development of national technical support of nuclear power in Romania. The INR activity is oriented with priority towards applied and engineering research within R&D programs, connected to present and future specific issues of NPP, especially those using CANDU 6 type reactors.
Over the last years, INR has been working on R&D programs to support a comprehensive and integrated Plant LIfe Management (PLIM) program for Cernavoda Unit 1 that will extend the operational life of the plant.
The objectives of the experimental programs consist in the identification of the main types of specific degradations of the fuel channel structural materials correlated with the primary water chemistry parameters as follows:
• evaluation of the fuel channel material (Zr – 2.5%Nb) corrosion behavior in normal and abnormal operation conditions. Characterization of structural materials before and after testing;
• assessment of restrictions on the service life of components caused by the progression of the degradation.
The paper presents the principal results of the research activities on INR Nuclear Materials and Corrosion Department in the field of fuel channel material corrosion behavior with the aim of PLIM and LTO.

Keywords: fuel channel, corrosion, Zr – 2.5%Nb, Plant Life Management (PLIM), Long Term Operation (LTO).

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1. Introduction

The objective of the Cernavoda NPP (Nuclear Power Plant) Plant Life Management (PLIM) program is to understand and deal with the various aging related degradation mechanisms and associated failure modes to determine the most effective inspection and maintenance strategies for all plant Structures, Systems, and Components (SSC). The main goals of the PLIM program in a PHWR (Pressurized Heavy Water Reactor) NPP are:

• to maintain the long-term reliability and safety of the plant during the design life;
• to maintain the long-term availability and capacity factors of the plant with controlled and reasonable generating costs during the nominal design life;
• to avoid surprises through identification of potential ageing issues ahead of their occurrence and provide means for monitoring and mitigation to ensure reliable component performance;
• to preserve the option of extending the life of the plant with good safety and availability at a reasonable cost, beyond the nominal design life of 30 years, up to 50 years;

The INR R&D programs in support of the PLIM program are focused on:

• understanding operating environment and degradation mechanisms and developing models;
• developing and applying inspection and monitoring technology;
• applying models to field data to predict component behavior and recommend maintenance and management activities, and/or develop and qualify improved components or systems.

Until now, considerable progress has been made in understanding the degradation mechanisms and developing ageing management programs for the major critical components, such as fuel channels, steam generators and feeder assemblies.

The corrosion of the structural materials in a nuclear installation is the principal degradation mechanism, especially taking into account the trends to increase the thermal rates and the need to decrease the exposure of operators. Therefore, the investigation of the structural materials corrosion in correlation with the water chemistry, as well as the corrosion products deposition and their removing from the primary and secondary circuit of a NPP is a very active field and both the experimental works and the understanding of the mechanisms involved are submitted to some rapid changes and permanently open to the researches.

To improve the service life and the structural components performances (such as: sheathe, pressure tubes, feeders, end-fittings, SG (Steam Generator) -tubes) it is necessary to study and monitor the corrosion of all structural materials from the primary circuit by controlling certain chemical parameters, which can reduce the aggressiveness of the coolant on specific materials used in system. During the last decade significant improvements were achieved in the reduction of the corrosion of structural materials of the primary circuit and dose rates by the improvement of water chemistry conditions.

One of the most important NPP structure is the Primary Heat Transport System (PHTS) having in view its role in active zone cooling and heat transfer to steam generators. In PHTS the chemical control is directed to keep chemical parameters within specified limits in order to mitigate the corrosion of the key equipment and related piping, to control the corrosion rate and impurities concentration, such as corrosion and fission products and to minimize activity transport and heat transfer surfaces fouling.

By operation in aqueous environment at high temperature and pressure, the structural materials from PHTS cover with protective oxide films, which maintain the corrosion rate in admissible limits. A lot of potential factors exist, which conduct to degradation of the protective films and consequently to intensification of the corrosion processes. The existing experience of different nuclear reactors shows that the water chemistry has an important role in maintaining the integrity of the protective oxide films. In order to minimize the adverse effects, an optimal water chemistry control and corrosion monitoring programme were established. The understanding of the corrosion degradation phenomena that conduct to failure of some components from PHTS of CANDU NPP implicates the investigation of the structural materials corrosion processes, in different conditions of water chemistry and temperature. To investigate the corrosion process of some structural materials from PHTS (Zr and Ni alloys) of CANDU 6 reactor were performed the following activities: out of pile corrosion experiments in different conditions of water chemistry, corrosion experiments in autoclaves assembled in by-pass of CANDU 6 reactor PHTS, corrosion analysis performed on some corroded components. The gravimetric method, optical metallographic microscopy and SEM analysis have been used to evaluate the corrosion behaviour of the pressure tube material. The obtained results allowed us to establish the contribution of the water chemistry in the initiation and evolution of some accelerated corrosion processes.

2. Experimental

In INR Nuclear Materials and Corrosion Department, the corrosion of CANDU PHTS structural materials was investigated extensively, allowing us to accumulate data and knowledge on corrosion mechanisms related to material characteristics, water chemistry and testing conditions.

These data were obtained by analysis of specific samples tested in static autoclaves and different electrochemical methods (cyclic polarization, potentiodynamic, potentiostatic, galvanostatic and electrochemical impedance spectroscopy – EIS).

Surveillance of some structural materials corrosion from primary and secondary heat transport system of CANDU-6 reactor is performing by out of pile experiments, corrosion experiments in autoclaves placed in bypass of CANDU reactor PHTS and corrosion analysis performed on some corroded component.

On the basis of the experiments and analysis performed, have been determined oxidation and hydriding kinetics of zirconium alloys (Zr-2.5% Nb) as a function of the initial surface state the water chemistry (pH) and testing temperature and the

corrosion of structural materials coupons exposed in PHTS autoclaves at U1 NPP Cernavoda, [1] ÷ [7].
In order to minimize the adverse effects, an optimal chemistry control has been proposed. Objectives of chemistry control are: minimize the degradation of the diverse system materials, minimize transport of radioactivity and prevent fouling of the core by magnetite deposition. These objectives are achieved by respecting PHTS chemistry specifications in normal operation, presented in Table 1.

Table 1. PHTS's chemistry specifications in normal operation

Parameter	Type	Specification range (Desired range)	Rationale
Dissolved Deuterium	Control	3-10ml/kg (3-7ml/kg)	Lower to prevent radiolytic oxygen formation Upper to minimize deuterium pick-up Zr-alloys
Dissolved Oxygen	Diagnostic	<10 mg/kg	Minimize Zr-Nb alloy corrosion Minimize pitting corrosion of carbon steel and cracking of stainless steel Minimize activity transport through minimizing crud formation Minimize corrosion of Incoloy-800
pH_a	Control	10.2 – 10.8 (10.2 – 10.4)	Lower limit is set by minimize deposition of magnetite in core Upper limit set to: minimize Zr-alloys and martensitic steel corrosion and minimize FAC of carbon steel
Conductivity	Diagnostic	8 - 25μS/cm (8 - 20μS/cm)	Used in conjunction with pH_a readings
Lithium Ion	Diagnostic	0.4 – 1.1mg/kg (0.5– 0.7mg/kg)	Used in conjunction with pH_a readings
Chloride Ion	Control	<200μg/kg (<50μg/kg)	Minimize pitting corrosion of Zr-alloys, carbon and martensitic steel Minimize cracking of stainless steel

In order to determine water qualities, e.g. pH, conductivity, lithium and dissolved D_2 concentration, cooling water to be measured is cooled down and depressurized, then is sampled and brought to a chemical laboratory in the plant for chemical and radioactive nuclide analysis.
The chemical species and the radioactive nuclides collected on membrane filters are analyzed by gamma-ray spectrometer and then, the measured data are transferred from analyzers to the computer system for evaluation. Accumulated data are stocked in a host computer allowing easy observation of the plant water chemistry.
Water chemistry standards for reactor systems and operating modes are presented in the PHTS Chemistry Operation and Control Manual. This manual lists acceptable operating conditions and corrective actions, including the response time that must be met if monitoring of water chemistry reveals inconsistencies with standards.

Chemistry control of the Primary Heat Transfer System and Auxiliaries is required to:

- mitigate the corrosion of system components;
- limit the production rate of radioactive corrosion products;
- minimize the fouling of the heat transfer surfaces by controlling the crud movement and removal;
- maintain heat transport storage tank cover gas deuterium and oxygen concentration below explosive limits.

The aggressiveness of PHT System water is reduced by controlling dissolved oxygen content, pH, impurities, radioactive crud and fission products.
The samples from Zr-2.5%Nb were tested at 310° C, 10.1 MPa in demineralized water with the pH value adjusted with LiOH = 10.1.

3. Results and discussions

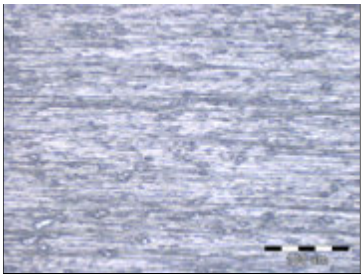
Fuel Channel Ageing Services under the Fuel Channel PLIM program include provision of expertise, supply of specialized tooling and equipment, work plans and procedures, analysis and assessment of inspection results and generate Fitness for Service (FFS) assessments. On the basis of the experiments and analysis performed, have been determined: oxidation kinetics of zirconium alloy Zr-2.5%Nb as a function of the initial surface state the water chemistry and testing temperature and the corrosion of structural materials samples tested in the laboratory facilities or exposed in PHTS autoclaves at U1 NPP Cernavoda, [8] ÷ [24].

3.1. Out of pile corrosion experiments (laboratory tests)

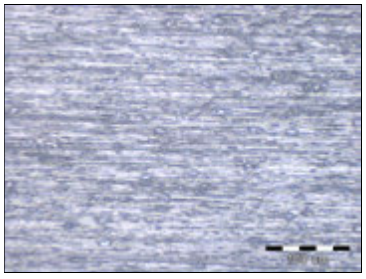
The results obtained by optical metallographic microscopy are presented in the Fig.1.

Zr-2.5%Nb sample tested 137 days: 2.4μm_x1000

Zr-2.5%Nb sample tested 149 days: 2.5μm_x1000



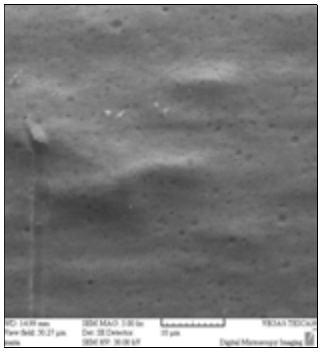
Zr-2.5%Nb sample tested 137 days, 2.4μm, x200



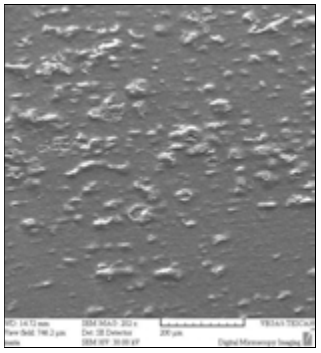
Zr-2.5%Nb sample tested 149 days, 2.5μm, x200

Fig.1. The aspect of oxide films and surface morphology formed on Zr-2.5%Nb samples after different testing periods at 310ºC.

The results obtained by Scanning Electron Microscopy (SEM) analysis are presented in Fig. 2.



Zr-2.5%Nb sample tested 137 days: 2.4μm_x1000



Zr-2.5%Nb sample tested 149 days: 2.5μm_x1000

Fig. 2. The aspect of Zr-2.5%Nb samples surface morphology after different testing periods at 310ºC.

Gravimetric analysis

When degrading corrosive protective films, and consequently accelerating degradation processes through localized corrosion, there are many factors, one of the most important factors being water chemistry. The chemical control in the main circuits of heat transport aims to keep the chemical parameters according to the specified limits, in order to reduce the corrosion of the structural components, to control the concentration of impurities and fission products and to reduce the transport of the activity, as well as the contamination of the thermal transfer surfaces.

The corrosion kinetics of the Zr-2.5% Nb material is shown in Fig.3.

From the plotted graphs it is observed that the corrosion rates decrease as the test time increases.

After a short initial period in which the oxidation is carried out according to different speed laws, the corrosion (weight gain ΔW due to the formation of the oxide layer) follows a cubic shape relation:
$\Delta W=k_c.t^{1/3}$ where:
ΔW- weight gain (mg/dm^2);
t- the exposure time (days) and
k_c- corrosion rate constant at a given temperature ($mg/dm^2.day$).

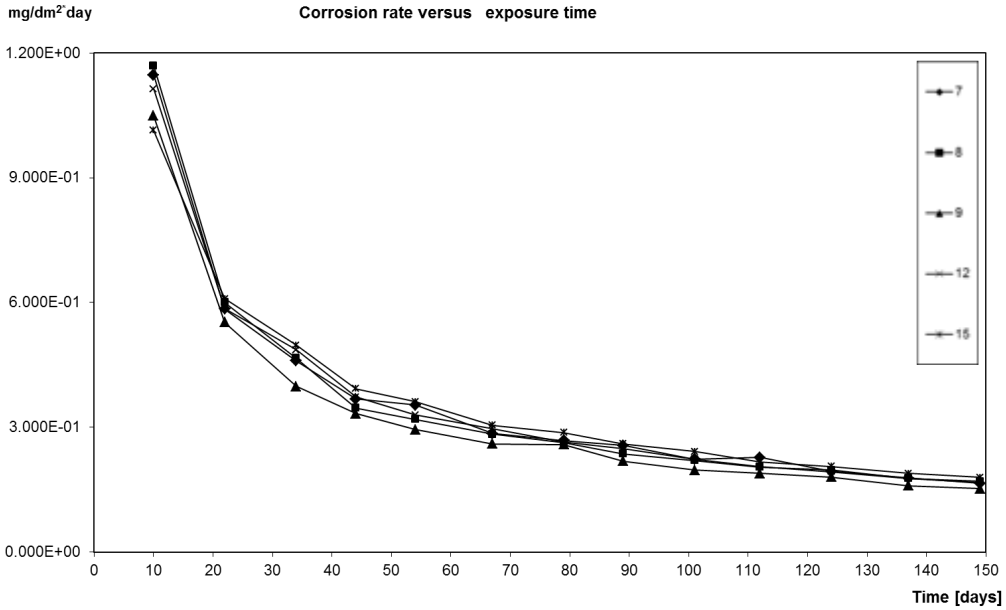
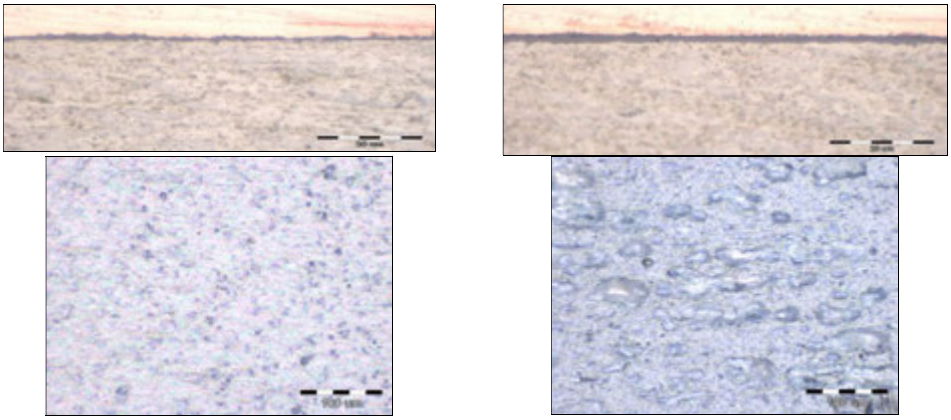


Fig. 3. Corrosion kinetic for Zr-2.5%Nb.

3.2. Corrosion experiments in autoclaves assembled in by-pass of CANDU 6 reactor PHTS

The zirconium alloy coupons (Zr-2.5% Nb), identified as Y4 - 47 and Y2 - 62 were extracted from the Y4 autoclave, at the exit of the reactor (at the steam generator); respectively Y2 autoclave at the reactor inlet (at recirculation pump) and thus the test conditions are different. The coupons subjected to the examination also had different exposure periods in the specific environment, the coupon Y4 - 47 being extracted after a period of less than 5 years, and the coupon Y2 - 62, after an exposure period of more than 5 years. At the visual examination of the coupons from Zr-2.5% Nb, there were differences between the two coupons regarding the surfaces of the formed oxides. The results obtained by optical metallographic microscopy are presented in the Fig. 4.



<5 years: 0.7-1.4μm >5 years: 1.2-2.2μm

Fig. 4. The aspect of oxide films and surface morphology formed on Zr-2.5%Nb after different periods of exposure in the PHTS autoclaves system.

Coupon Y2 - 62 has a glossy black oxide and numerous white lines along the length of the coupons. This suggests that, before mounting on the support, the coupon had traces of deep mechanical processing, which oxidized differently from the rest of the material. Generally, in the case of Zirconium alloys the white color of the oxide is specific to the stoichiometric oxide ZrO_2, and the black, gray color signifies the presence of a non-stoichiometric ZrO_{2-x} oxide.
It is expected that the oxide layer will not have a uniform thickness but will be thicker on the white oxide lines and thinner in the black oxide regions.
In the case of coupon Y4 - 47 it was found that the surface is completely covered with a black, glossy oxide, Fig.5 and Fig.6.

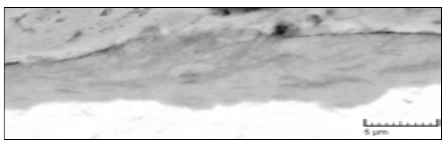


Fig. 5. Aspects of the oxide from sample Y2-62 from Zr-2.5% Nb.

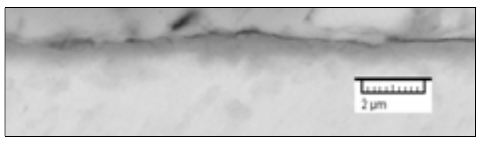


Fig. 6. Aspects of the oxide from sample Y4-47 from Zr-2.5% Nb.

Surface oxide analysis In the case of oxidized samples in the autoclaves of the primary circuit, the analyzes were performed on the topographic images, of secondary electrons obtained at a scanning electron microscope of the TESCAN VEGA II LMU type.
When examining SEM from sample Y4-47 from Zr-2.5% Nb (exposure under 5 years) it was found that the oxide forms a continuous film and no surface defects (exfoliation) of the oxide film were observed and no crystallites with defined geometric shapes were present. The oxidized surface (Fig. 7) is smooth and free of scratches, suggesting that a thin oxide was formed which blurred the traces of mechanical processing and grinding. Throughout the surface of the coupon you can

see pores with dimensions smaller than 1 micron that form in or under the thin layer of oxide. From place to place, small swelling in the oxide is observed but not accompanied by the degradation of the oxide surface. Examination of the surface of the Y2-62 coupon (exposure over 8 years) (Fig.8) showed that the oxide formed has numerous swellings accompanied by cracks and detachments of oxide, partial exfoliation. The swelling may be due to the partial detachment of the oxide at the metal-oxide interface or to an uneven thickness of the oxide.

In the case of coupon Y2-62 from Zr-2.5% Nb it was noted that the oxide does not have a uniform thickness, as the metal oxide interface is corrugated (Fig. 9 and Fig.10). Occasionally, oxide nodules of larger thickness are present. A relatively uniform oxide layer is observed throughout the surface, with a small thickness between 2 and 4 microns and oxide nodules with a thickness of around 8 microns.

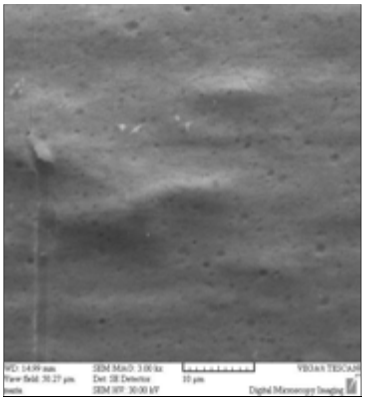


Fig. 7. Oxides surface on sample Y4-47 from Zr-2.5% Nb - SEM image (x3000)

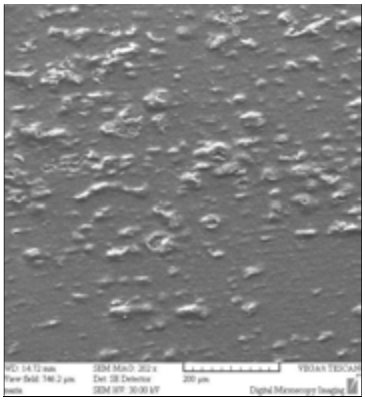


Fig. 8. Oxide surface on sample Y2-62 from Zr-2.5% Nb - SEM image (x200)

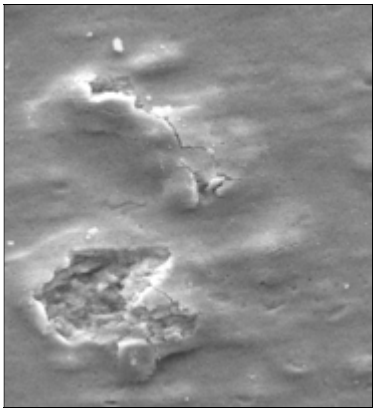


Fig. 9. Oxide on Zr-2.5% Nb sample surface Y2-62 SEM image (x1000)

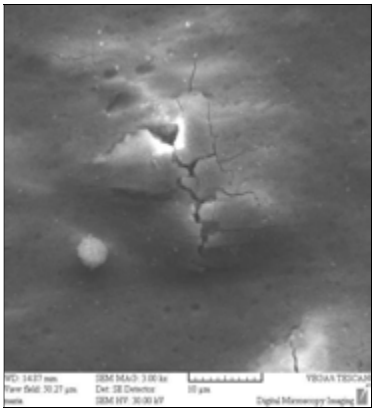


Fig. 10. Oxide on Zr-2.5% Nb sample surface Y2-62 SEM image (x3000)

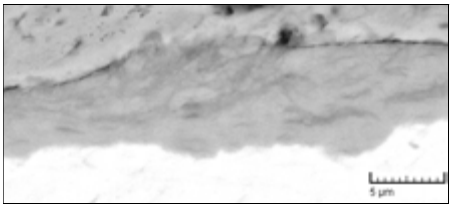


Fig. 11. Aspect of the oxide from sample Y2-62 from Zr-2.5% Nb

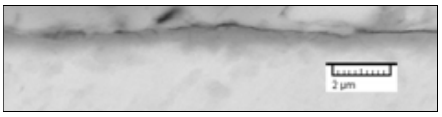


Fig. 12. Aspect of the oxide from sample Y4-47 from Zr-2.5% Nb

As for the Y4-47 coupon, the oxide layer on it is very thin, the thickness measurements made on back-scattered electron images at 10,000 magnification show values of: 0.39, 0.79, 0.45, 0.69 microns. The metal oxide interface is not clean and is corrugated as illustrated by Fig.11 and Fig. 12.

4. Conclusions

The paper presents the principal results of the research activities on INR Nuclear Materials and Corrosion Department in the field of Zr-2.5%Nb alloy corrosion behavior with the aim of Ageing Management and LTO.
The experimental program consists in: out of pile corrosion experiments (laboratory tests) and corrosion experiments in autoclaves assembled in by-pass of CANDU 6 reactor PHTS (in-situ corrosion monitoring).
The oxidation of Zr-2.5%Nb alloys was strong dependent of temperature. Generally, the formed oxides were continuous and uniform, but, at long period of exposure the porous character of oxides and appeared fissures at exterior surface oxides which increased of time, advancing to oxide/metal interface.
The scanning electron microscopy analysis pointed out the appearance of pores and small fissures on oxides surface with thickness beginning from approximately 2μm corresponding of transition point. The oxide surface at oxide/metal interface did not present fissures even in case of the thick oxides.
Reactor coolant aggressiveness is controlled by monitoring the dissolved oxygen content, pH and the level of impurities, crud and fission products.
Corrosion testing of the samples from Zr-2.5% Nb alloy was performed for a maximum of 149 days. The exposure medium was demineralized water with pH = 10.1 adjusted with LiOH and the temperature and pressure had values of 310^0C, respectively 10.1MPa.
The films formed on post-autoclaving samples and the morphology of the samples surfaces were evaluated by metallographic microscopy and scanning electron microscopy.
The coupons of Zr-2.5% Nb which were extracted from the autoclave system from U1 Cernavoda NPP after an exposure less than five years and over five years were examined visually, by metallographic (optical) microscopy and by SEM. It was possible to compare the corrosion behavior between the two types of samples, the

results being affected by the fact that the testing time in the autoclave was incomparably smaller than the testing in the autoclave system.
The oxides formed on Zr-2.5% Nb are adhesive and protective at an operating time of less than 5 years, their thickness being under one micron. At longer exposure times (over 8 years), coupons contain oxide swellings on the surface that are actually oxides that grow like nodules, crack and partially lose in the cooling agent. This was observed on the available coupons and it was observed that there are deep traces of grinding and the surface is not finely polished. Zirconium oxides from the cooling agent were transported to the autoclave and fixed to other steel coupons, EDS (Energy-dispersive X-ray spectroscopy) analyzes showing the presence of zirconia among the magnetite crystals present on the surface.
In normal conditions of the operation and water chemistry, the general corrosion did not limit life of structural components. But, in abnormal chemistry conditions, localized corrosion processes can conduct to failure of some components and leakages of the coolant.
The topic of corrosion of in-situ alloys in water has been the demonstration of a proper and systematic management of the ageing of the Unit 1 components of the Cernavoda NPP, thus creating the premises of the renewal of the license and the life extension.

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