2013 International Nuclear Atlantic Conference - INAC 2013
Recife, PE, Brazil, November 24-29, 2013
ASSOCIAÇÃO BRASILEIRA DE ENERGIA NUCLEAR - ABEN
ISBN: **978-85-99141-05-2**

INFLUENCE OF NEUTRON IRRADIATION ON THE STABILITY OF PRECIPITATES IN ZIRCALOY – A CRITICAL REVIEW

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ABSTRACT

The realization of RMB enterprise (Brazilian Multipurpose Reactor) will give the country a powerful tool to investigate the behavior materials subjected to irradiation. Among them, zirconium alloys, used as cladding of nuclear fuel in reactors type LWR. It is know that neutron irradiation can affect the stability of precipitates in zircaloys, generating as a result changes in theirs mechanical properties, important application of this alloys. This paper present a critical review of neutron irradiation effects on microstructural stability of zircaloys (2 and 4).

1. INTRODUCTION

The Laboratory for Irradiated Materials (LAMI), as part of the RMB (Brazilian Multipurpose Reactor), will permit examination and testing of the mechanical behavior of materials used as structural materials in nuclear reactors.

Among the most widely used nuclear material, the zirconium-based alloys, particularly zircaloy-2 and zircaloy-4 are generally chosen for their excellent mechanical properties and corrosion resistance, as fuel coating materials in nuclear reactors type LWR. The precipitates present in their microstructure play an important role in the properties of these alloys, being responsible for their optimizations. Many articles dealing with this subject in the literature under different aspects highlighting the influence and importance of correctly characterize such precipitates.

Analysis of zircaloy component undergoes typical neutron fluence with the passage of time, in typical operating conditions, may affect the stability of the precipitates, producing changes in the mechanical properties of these alloys, of paramount importance for the intended applications.

This paper present a critical review of the effects of neutron irradiation on microstructural stability of zircaloys (2 and 4), while present some devices for irradiations (“rigs”) in materials test reactors and also the facilities needed to their post irradiation evolution.

2. Precipitates

Different compounds of zirconium and alloying elements are precipitated in the matrix during thermal treatments. The general formula for these components are intermetallic $Zr_2(Ni, Fe)$ and $Zr(Cr, Fe)_2$. For zircaloy-4, Fe /Cr ratio is the same as the nominal composition of the alloy, while for zircaloy-2, due to the iron partition between two different intermetallic phases, the ratio covers a broad range of the ratio Fe/Cr in $Zr(Cr, Fe)_2$ and Fe / Ni in particles $Zr_2(Ni, Fe)$.

The influence of the precipitates size in the properties of the alloys occurs mainly in corrosion rate according Lemaignan et al. [1]. Better corrosion resistance for PWR, is obtained even in the presence of larger precipitates, while in BWR type reactors better resistance to localized forms of corrosion is observed when the material exhibits very small precipitates distributed.

An attempt to classify these particles in shape and size is given by Chemelle et al. [2] in his proposed scheme for the second phase particles of zircaloy-2 (Fig. 1). They are separated in accordance with the composition: Fe-Ni-Zr type particles numbered 1 to 3, Fe-Zr-Cr type, 4 to 6, and agglomerated particles classified as type 7. As zircaloy-4 is an alloy containing no Ni particles only numbered 4 through 7 can be correlated with its precipitation. In the scheme can be found small spherical precipitates (particle 1, with size between 0.2-0.4 µm and particles 4 with size between 0.2-0.3 µm), some of which shows contoured curved shapes and appear as single crystals . Type 5 particles occurring in grain boundaries have a rectangular shape and exhibit a twinned structure with stacking faults and with size between 0.3 and 0.6 micrometers. The larger particles (particles 3 and 6) have oval shapes and often are slightly angled. The agglomerates can be found with up to five particles. Examples of second phase particles are shown in Fig.2.

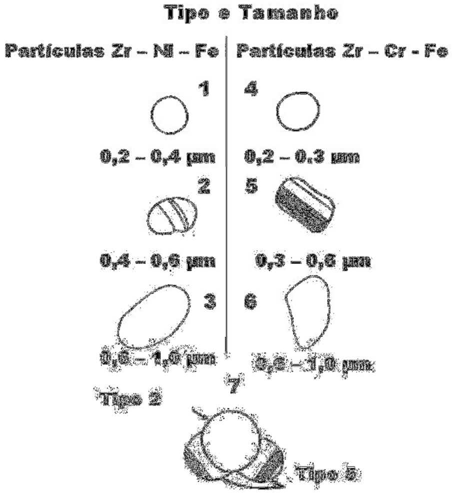

Figure 1: Chemelle's scheme for zircaloy-2 particles [2].

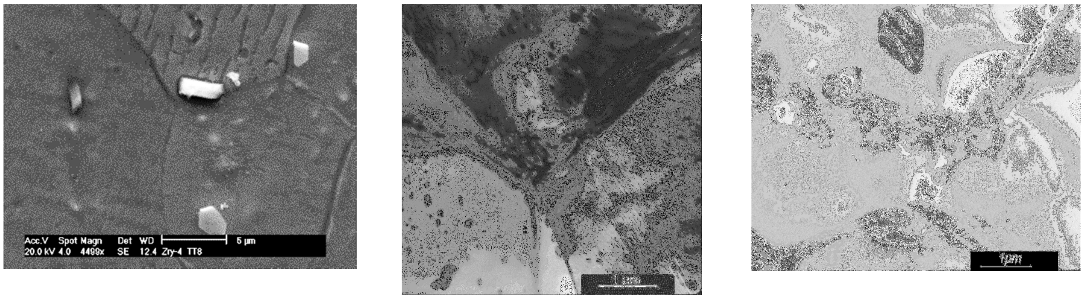

Figure 2: Second phase particles of zircaloy 4 (R. M. Lobo).

The crystal structures of the precipitates is identified as a hexagonal $Zr(Cr, Fe)_2$ and C14 type Laves phase in zircaloy-2 and zircaloy-4, tetragonal Zr_2 (Fe, Ni) in cubic zircaloy-2 and C15 type $Zr(Cr , Fe)_2$ Laves phase. The intermetallic $ZrCr_2$ can present two types of structure: a C14 type laves phase (hexagonal) and C15 type Laves phase (cubic). A transition is observed between C14 structure to C15 structure in the temperature range from 900°C to 994°C. By the Zr-Cr binary equilibrium diagram, the prototype of the C15 type $ZrCr_2$ (fcc) is a low temperature modification and the C14 type $ZrCr_2$ is the phase at high temperature. Although the C14 structure is not the most stable phase at low temperatures, it is retained because the transformation into the C15 structure is very slow. For Shaltiel et al.[3] the balance between these two structures depends on the ratio Fe / Cr according to an empirical rule proposed by the authors, which determines the crystal structure changes according to the composition from cubic to hexagonal structure and turning again to be cubic. Thus both hexagonal and cubic $ZrCr_2$ structures can be found at room temperature in zircaloy-4.

The difference in the temperature range where the cubic and hexagonal phases are stable is due to the difficulty of identifying precisely the crystal structure of the precipitates using only electronic diffraction pattern, according to Meng et al. [4]. Taking into account the diffraction electronic pattern, and the displacement of zirconium atoms necessary for the formation of hc in an arrangement for α-Zr type diamond cubic C15 Laves phase, it is verified that the most stable phase is the cubic in zircaloy-4. Based on the relative orientation of defective structures can be deduced that the formation of the C15 Laves phase is a process of displacement of atoms.

To identify and measure the composition of the second phase particles D. Arias et al. [(5] analyzed samples of zircaloy-2 and zircaloy-4 under different prolonged heat treatments. The average composition of the precipitates in zircaloy-2 was found equal to $Zr(Cr_{0,\,55}, Fe_{0,\,45})_2$ and $Zr(Fe_{0,\,60}, Ni_{0,\,40})$ and the boundary composition as $Zr\ (Cr_x, Fe_{1-x})_2$ with x = 0.45 and 0.63 and $Zr_2(Fe_y, Ni_{1-y})$ with y = 0.53 and 0.67, respectively, and the average value coincides with the estimated by Chemelle. The average composition of the precipitate for zircaloy-4 is $Zr(Cr_{0,\,58}, Fe_{0,\,42})$ and the second threshold Zr composition $(Cr_x, Fe_{1-x})_2$ with x = 0.09 and 0.63 respectively. The range of composition of precipitates is attributed to the different thermal treatments suffered by metallurgical alloy during processing and can be understood if one considers the ternary phase diagram.

3. Effects of Irradiation

Analysis of zircaloy component undergoing reactors typical neutron fluence and operating temperature show that many of the particles become totally or partially amorphous. According to Gilbert et al. [6], the amorphous-crystalline transformation is accompanied by a volume change, resulting in local plastic deformation and there is a decrease in iron concentration in these particles. The nature of these particles after irradiation can be understood in terms of the crystalline-amorphous transition temperature, there is an intermediate temperature range where the diffusion of the individual components is the main control factor for transformation. The diffusion may occur by a mechanism of vacancy or interstitial where iron appears to have greater mobility than chromium.

Many explanations of this phenomenon are given as related to the free energy, in any alloy system where spontaneous transformation to the amorphous state occurs when the free energy of the crystalline state becomes higher than in the amorphous state, or that involves the concept of critical density of defect when the density accumulated during irradiation exceeds the critical density, the transformation occurs. Another criterion is the degree of ionic and interatomic bonds in an intermetallic compound (ionicity) and the recrystallization temperature. For a system with less ionicity than 4 and a temperature of recrystallization to amorphous phase greater than or equal to 0.3 $T_{f,}$ material is ready to become amorphous under irradiation. Alloy systems with limited solubility or near composition range, show a greater tendency to become amorphous during irradiation. Alloy system displaying limited solubility or no, will have a low value of the critical density of defects since the free energy increase gradually, out of the stoichiometric composition.

Yang et al.[7] verifies that the amorphization of $Zr(Fe,Cr)_2$ precipitates with neutron irradiation at 290°C with a flow rate of $1x10^{25}$ n/m^2. The transformation begins on the borders of the precipitate and the thickness of the amorphous ring increases as the flow increases. Depletion of iron as these become amorphous precipitates is attributed to the dissolution of the solute induced by irradiation.

Commenting on these results, Griffiths[8] states that the dissolution of the intermetallic precipitates can be interpreted in terms of interaction between the precipitates and the flow of the auto-interstitials zirconium atoms, spreading parallel to the basal plane. This interpretation is consistent with findings of previous studies and the theory of radiation damage in zirconium alloys, based on interstitial diffusion anisotropy.

Samples of zircaloy-2 and zircaloy-4 analyzed by Griffiths et al.[9] at temperatures between 80 and 400°C with a flow of (<0.5 to 8.5) x 10^{25} n/m^2 verified that the intermetallic particles $Zr_2(Ni, Fe)$ and $Zr(Cr, Fe)_2$ undergo phase and morphology changes during irradiation. Below 80°C both become amorphous. In the interval between 250°C and 330°C, $Zr_2(Ni, Fe)$ particles will remain crystalline as the $Zr(Cr, Fe)_2$ become amorphous. This transformation depends on the local composition within the particle and occurs when the relative content of chromium is higher or there is no depletion of solute below the stoichiometric value. At temperatures of 370°C to 440°C both particles undergo radiation-induced dissolution. The redistribution of alloying elements results in the formation of precipitates in the matrix and at grain boundaries and the morphology of each type is determined by its composition.

The size and distribution of intermetallic precipitates influence the rate of corrosion of a reactor internal components, so the dissolution and reprecipitation of the particles is of great importance in the corrosion resistance of zircaloy-4 under irradiation. Motta et al.[10] presents a model to explain some characteristics of amorphous crystalline transformation, where the amorphization starts by a deviation of stoichiometry due to the interaction of atoms of Fe and Zr through the precipitate-matrix interface, forming a front that moves gradually toward the inside of the precipitate. This is also consistent with the kinetics of amorphization observed that predicts a amorphous layer thickness proportional to fluence and the absence of incubation period for the initiation of the transformation.

The amorphization alters the composition of precipitates in zircaloy-2 and zircaloy-4, according Etoh et al.[11] must be designated as the Zr-Cr-Ni and Zr-Cr-Fe type precipitates. Depletion of Fe precipitates in zircaloy-2 occurs faster than in zircaloy-4 and the effect of radiation on Zr-Cr-Ni zircaloy-2 precipitate is the dissolution of the precipitate. The precipitates of the type Fe-Zr-Ni dissolve faster than those of the type Fe-Zr-Cr and the dissolution ratio of each element is in the following order Fe> Ni and Ni ≥ Cr. The cause of the different behavior between the two types of intermetallic compounds may be due to different mobilities of point defects in both compounds , in other word, reordering of the crystal may occur in $Zr_2(Fe, Ni)$ but not in $Zr(Fe, Cr)_2$ under BWR operating conditions.

By analyzing precipitates in zircaloy-2, zircaloy-4 and in an alloy Zr-V-Fe irradiated by neutrons and ions, D. Pecheur et al.[12] found that the transformation is homogeneously distributed in the amorphous precipitates. The dose required to amorphize each precipitate type increases exponentially with temperature and irradiation when compared to ion irradiation with neutron cascades that induce collision, it is observed that the greater the damage the higher the critical temperature. The central region of the precipitates in zircaloy-2 (Fe / Cr = 0.7) become faster than the amorphous precipitates of $Zr (Fe, Cr)_2$ in zircaloy-4 (Fe / Cr = 1.7), which is according to the results of annealing after irradiation, showing that the amorphous phase is more stable in precipitates rich in Cr.

By studying neutrons pre-irradiated Zr-α, Griffiths et al.[13] found vacancies moving at surprisingly low temperatures (230-320 K). The climb planes of c-component network deslocations depend on the irradiation temperature: nonbasal climb at low temperatures (320K) and basal climb at higher temperatures (573 – 673 K).

The Zr and its alloys exhibit remarkable resistance to void swelling during in-reactor neutron irradiation. In contrast to other metals and alloys used for nuclear fuel cladding, such as stainless steel, Zr and Zr alloys exhibit little void formation and, as a consequence, little swelling when exposed to irradiation. In a study on formation of bubbles in Zr alloys under Kr ion irradiaton, Pergano et al.[14] showed that they form in all alloys at temperatures between 300-800°C and are larger in bulk specimen than in thin samples, which should be attributed to the greater loss of gas atoms and vacancies to the surface in the thin foil case. In bullk samples, bubble size increases with irradiation temperature, reaching a maximum diameter within the grains, from 10 nm at 300°C to 40 nm at 800°C. Large faceted bubbles and bubble coalescence were found, above 700 ° C.

Lefebvre et al.[15] attempt to explain irradiation effects on corrosion of zirconium alloy claddings, why a better corrosion resistence is obtained for BWR with small precipitates, while the reverse is true for PWR conditions. An important consequence of delay oxidation of the precipitates with respect to the matrix is that, since the oxidation of the metallic precipitates is observed to occur simultaneously to a Fe depletion, these precipitates, embedded in the zirconia deeper layer, act as sources of Fe. On non irradiated material, the oxide is growing on the matrix with strong preferential orientation relationships, leading to a strongly textured zirconia layer. For in reactor grow corrosion layers a tendency is observed for coarser oxide crystallites and less frequent areas showing a marked common orientation. In addition, large grains are found in the middle of the oxide layer showing that a recrystallization process has occurred during in reactor corrosion. An enhanced diffusion accelerates the precipitates depletion, reducing the time during which precipitates may act as a Fe reservoirs and by same process as long as precipitates are no sufficiently depleted, it spread Fe around the precipitates, increasing locally the stability of the tetragonal phase. This hypothesis appears to be consistent with the observation, several microns away from the metal-oxide interface, of areas of oxide having an extremely porous structure and a high Fe content. The irradiation enhance mobility of Fe in the oxide layer seems thus to contribute to decrease the protective character of the oxide layer, leading to a lower corrosion resistance under irradiation.

One of the limits to the time that a zirconium alloy component can stay in the reactor core is given by rate of absorption of hydrogen isotopes produced during the corrosion reaction between the alloy and the coolant water. Therefore, the irradiated zirconium alloys could in principle absorb relatively large amounts of hydrogen before precipitation of zirconium hydride occur. Higher effective hydrogen solubility would delay the nucleation of unwanted hydride blisters in zirconium base components, as pressure tubes, thus increasing their lifetime in service. Moreover, a smaller amount of hydrides would precipitate when the reactor cools after its shut down. Vizcaíno et al.[16] studied the solid solubility of hydrogen isotopes in neutron irradiated zircaloy-4 core components removed after 10,3 years in service conclude that all the inicial $TSSd_{irrad}$ (temperature of solubility on dissolution) values are around the reactor operating temperature range, it is predicted that under reactor operation conditions the fraction of hydrogen atoms in excess with respect to the zirconium alpha phase solid solubility are trapped by complex irradiation induced defects. The increase in $TSSd_{irrad}$ values with the thermal treatments could then be attributed to the gradual release of hydrogen/deuterium atoms from these defects. However, the final step of the recovery kinetics is difficult to understand, and the possibility of solutes and second phase particles in zircaloy-4 interacting with the hydrogen isotope atoms cannot be excluded. To measure the dissolution enthalpy of zirconium hydride precipitated Vizcaíno et al.[17] used the differential calorimetric technique found that the post irradiation thermal treatment releases the H atoms from their traps increasing the number of H atoms available to precipitate at end of each calorimetric run and or isothermal treatment. This behavior is compatible whit that observed in previous works in the terminal solubility temperature and supports the idea of the existence of a hydrogen trapping mechanism in defects created by irradiation. The defects are annihilated with the annealing, setting the hydrogen trapped in them free. In this way, the hydrogen fraction previously trapped is available to precipitate as hydrides when the material cooled down, increasing the $TSSd_{irrad}$ and the dissolution enthalpy in a subsequent run.

Onimus et al.[18] analyzes the changes in mechanical behavior of recrystallized zirconium alloy used as fuel cladding tube during neutron irradiation and verifies that major changes in mechanical behavior occurs in recrystallized zirconium alloys. Irradiation leads to a strong increase of the yield stress as well as the isotropic stress due to hardening by high loop density induced by irradiation. Strain rate sensitivity, as well as the associated viscous stress, is increased by irradiation which is explained by the activation of basal slip instead of prismatic slip for the non-irradiated material. The kinematic strain hardening is increased by irradiation due to the high strain incompatibilities between grains induced by the activation of basal channeling. The increase of kinematic strain hardening with irradiation can explain the increase with irradiation of the strain hardening rate at the onset of plastic flow. It is also proposed that the fast decrease of the strain hardening rate of the irradiated material is attributed to the propagation of basal channels from grain to grain. Nevertheless, the kinematic stress has not been measured for the irradiated material. The mechanical analysis has also proved that the strain rate sensitivity in stress relaxation, as well as the associated relaxed stress, is increased by irradiation. This is attributed to the activation of the basal slip as well as the important recovery of the kinematic stress during relaxation test due to the increase of the kinematic stress with irradiation.

X-ray diffraction line profile analysis (XRDLPA) technique was used by Sarkar et al.[19] to asses the microstructure of an heavy irradiated Zr-1.0%Nb-1.0%Sn-1.0%Fe alloy. The analysis reveals that there is a significant decrease of surface weighted average domain size with dose. The damage associated with neon beams (a heavy ion) is quite extensive and produces highly localized concentrations on defects, particularly vacancies and interstitials. These vacancy clusters collapse into dislocation loops and the dislocation density increases accordingly.

The irradiation hardening and change in microstructure following irradiation were characterized by Cokeram et al.[20]. Temperatures higher than 358°C results in a larger <a> loop size and lower <a> loop number density; this results in less irradiation hardening than observed for irradiations at lower temperatures. The calculated irradiation hardening results also match trends in measured irradiation hardening as a function of fluence for each material. Oono et al.[21] observed the irradiation effects on the hardness and the microstructural evolution of ε-Zr-hydride. The authors shows that the dominant mechanism of the deformation is the formation of new fine twin bands and tangled dislocations. Larger indentation induced cracks were also found by the surface observation on the indents print in the irradiated area. The strain of the irradiation induced defects made difficult to deform the ε-Zr-hydride.

Recrystallized zirconium alloys used as structural materials for PWR fuel assembly, undergo under neutron irradiation induced stress free growth which accelerates for high irradiations doses were studied by Tournadre et al.[22]. Results shows that after proton irradiation the irradiated layer exhibits a stress free strain which is consistent with the irradiation induced growth of recrystallized zirconium alloy and proves for the first time, that c-component dislocation loops can be created in zircaloy-4 RXA (recrystallization annealed) using 600 KeV Zr ion and 2 MeV proton irradiations at temperature range 573-623 K. The <c> loops created after charged particle irradiations are smaller than after neutron irradiations, and differences are larger for Zr ion irradiations than after proton irradiation. Proton irradiation appears to be more representative of neutron irradiation than Zr ion irradiation, at least concerning <c> loops. Moreover, thanks to the high penetration depth of protons, the thin foils are taken from the bulk material. Therefore proton irradiation allows avoiding the influence of the free surface which can be a sink of point defects.

The microstructure of pure Zr-base alloy (zircaloy-2) irradiated with Zr ions (self-ions) were studied by Yamada et al.[23]. It was found that the c-component dislocation structures formed in neutron-irradiated Zircaloy-2 can also be obtained by self-ion irradiation. The presence of alloying elements promotes the formation of the c-component dislocation structures. The self-ion irradiation technique is expected to be useful for the study of irradiation demage in various materials. In contrast to neutron irradiation, test conditions such as dpa values and temperatures can be precisely controlled, moreover, ion irradiation can induce a high level of damage in a comparatively short time.

Mechanical properties degradation of Zircaloy-4 e Zirlo with increasing hydrogen concentration was investigated by Lee et al.[24]. As hydrogen concentration increases, Young's modulus and total elongation decrease. This degradation is due to micro-crack development and void growth induced by hydrogen, because in the hydrided zirconium matrix, microcracks develop easily in the boundary between the base metal and precipitated hydrides when load is applied. Also, relatively small size hydride gives rise to voids at hydride-zirconium interface due to the loss of adherence between the hydride and zirconium metal.

4. CONCLUSIONS

Currently the analyses of irradiated materials are conducted mainly by mechanical testing laboratories [25] to evaluate the behavior of structural materials under the operational conditions of nuclear reactors. Samples from for such tests are taken from "rigs" radiation in periodic campaigns according to the prediction analysis.

The second phase particles are analyzed by more precise methods with high radiation flux and high resolution. Synchrotron radiation in the bulk microstructure of Zr alloys can give details down to the sub-micron scale [26].

The transmission electron microscope (TEM) coupled to an ion accelerator is used to perform "in situ" experiments ion irradiation in well-controlled experimental conditions in order to study the evolution of the damage, which is not provided by neutron irradiation experiments and can be continuously observed with the microscope. The main advantage of "in situ" TEM [27] ion irradiation is that the damage evolution can be continuously observed for a better understanding and spatial correlation of defect structures, and pre-irradiated microstructures at different dose levels.

Computer simulation [28] is used to predict the microstructure of irradiated materials through the evolution of various populations of loops defects, based on the experimental observations.

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