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EVALUATION OF THE EMBRITTLEMENT OF NUCLEAR CLADDINGS FOLLOWING MULTIPLE TRANSPORTATIONS

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ABSTRACT

The integrity of spent-nuclear-fuel (SNF) rod claddings must be ensured throughout all phases of the reprocessing or final disposal processes: handling, storage and transportation. In this regard, the potential degradation mechanisms specific to each step must be investigated, taking into account the cladding properties change due to in-reactor operation and subsequent thermo-mechanical transients. EDF is considering the building of a centralized interim SNF storage pool as a buffer step between SNF cooling-down in fuel pools and fuel reprocessing at La Hague facility. Under this scenario, some SNF assemblies might be subjected up to three dry transportations. During drying and transportation, residual heat of the SNF causes cladding heat-up and generates a high hoop stress because of the internal rod pressure elevation. All or part of the zirconium hydrides, stemming from in-core corrosion and initially oriented along the circumferential direction, are dissolved and could then precipitate as radial hydrides upon cooling. This phenomenon is known to cause additional cladding embrittlement and can be an issue for fuel retrieval.

The objective of this study was to evaluate the impact of several transportation–storage cycles on the cladding embrittlement by hydrides reorientation (HR). To address this issue, an experimental program was undertaken on unirradiated pre-hydrided (350 and 700 ppm) Zircaloy-4 cladding. One to three HR treatments were applied on cladding tubes (400°C, 80-120 MPa under internal pressure) and subsequent ductility was assessed by performing ring tensile tests at room temperature. A decrease of ductility was observed after the first treatment but no additional embrittlement was noticed after two or three treatments. Metallographic examinations were carried out to highlight the hydrides distribution after HR treatments and to explain the mechanical tests results. The findings support the idea that the multiple transportations are not more detrimental than a single one regarding cladding embrittlement.

INTRODUCTION

As a global leader in low-carbon energy, the EDF Group covers every sector of expertise, from generation to trading and transmission grids. In France, EDF operates 58 Pressurized Water Reactors (PWR) spread across 19 power stations all around the country. After their withdrawing from the reactor, spent nuclear fuel assemblies are stored for several years in on-site cooling ponds. Once sufficiently cooled, SNF assemblies can be transported to the ORANO reprocessing plant in La Hague. There are two key aspects in the French nuclear framework that make it original. The first one is the legal obligation to reprocess spent fuel in order to separate re-usable materials (plutonium and reprocessed uranium for MOX and RepU fuel fabrication) from final waste. The second one is the use of interim wet storage (i.e. at low temperature) rather than dry storage.

EDF is considering the building of a centralized interim storage as a buffer step between on-site cooling ponds and the reprocessing plant for MOx and RepU spent fuels. Under this scenario, some SNF assemblies might be subjected up to three dry transportations interspersed by two wet storage phases. During drying and transportation, residual heat of the SNF causes a heat-up inside the shipping casks. These casks are designed to withstand high internal temperatures (up to 450°C). The consequences on the SNF fuel rods must be assessed to ensure their integrity up to the reprocessing stage.

Due to their initial gas filling and the release of fission gas under irradiation, SNF fuels rods have a substantial internal pressure at room temperature, up to 5 MPa for a burn-up lower than 65 GWd/t [1] that generates a high hoop stress during drying and transportation. At the same time, all or part of the zirconium hydrides, stemming from in-core corrosion and initially oriented along the circumferential direction, can be dissolved. As the hydrogen solubility is very low at room temperature [2], hydrides will re-precipitate under a tensile stress during subsequent cooling down of the SNF. These conditions may induce a reorientation of the hydrides from the circumferential to the radial direction of the cladding [3], which is known to be detrimental to the cladding ductility [4–6].

Numerous studies have been carried out to determine the conditions of hydrides reorientation (HR) in various zirconium alloys in the hydrided or irradiated state [7–14]. Many have focused on the determination of the stress threshold under which no HR is observed or no ductility loss is observed. From this kind of study, operational limits were derived, such as the ones prescribed in NRC ISG-11 [15]: 400°C peak cladding temperature for all burn-ups, 90 MPa hoop stress for low and intermediate burn-ups, limitations of thermal cycling.

Several studies have been carried out to check the influence of thermal cycling on the HR phenomenon [8,10,13,16,17]. While some of them are linked to the drying phase before dry storage (limited temperature drop, high number of cycles), others just use cycling, and other experimental conditions, as a mean to obtain more and more radial hydrides without real connection to the actual SNF thermo-mechanical history. These studies were performed on a wide range of materials, using different techniques for hydrides reorientation. Most of them seem to indicate that thermal cycling induce a higher degree of reorientation, but results are scattered. Moreover, few studies focus on the effect of radial hydrides on the cladding material ductility [5,7–9,14], which is ultimately the most important feature from a safety point of view.

The aim of this study is to enhance the understanding of the potential effect of multiple transportations on the embrittlement of SNF rods according to EDF scenarios, using representative reorientation stress and focusing on the impact on ductility.

EXPERIMENTAL

Material and hydriding

Cold-worked stress-relieved Zircaloy-4 cladding was used for this study. Unlike Zr-1%Nb cladding [18], unirradiated Zircaloy-4 ductility can be deeply decreased by hydrides reorientation. Moreover, it has been shown that irradiation barely affects the stress threshold for reorientation in Zircaloy-4 [19]. Tube nominal dimensions were 9.5 mm outer diameter and 0.57 mm wall thickness. They were hydrided using a cathodic process at room temperature before undergoing a homogenization treatment at 430°C for 24 h in inert atmosphere. Two hydrogen contents were investigated, intermediate and high. Hot vacuum extraction measurements were performed on every specimen. For the specimens with intermediate hydrogen content, an average content of 375 ppm was found with a standard deviation of 35 ppm. For the high content batch, it was 760 ppm with a standard deviation of 40 ppm. Due to the hydriding process and the incomplete dissolution of hydrides at 430°C, specimens exhibit a hydride rim on the outer surface (Figure 2). Below the rim, hydrogen distribution was found to be homogeneous. Hydrides are quite long and mostly oriented along the circumferential direction. Spent fuel rods with rather high hydrogen content have necessarily a heterogeneous hydride distribution with a hydride rim (albeit thicker than in our samples). More information about the hydriding process and the hydride distribution can be found in [20].

Hydrides reorientation treatments (HRT)

100 mm long specimens were subjected to HRT in an internal pressure creep apparatus using argon gas. Tubes were sealed on one side and connected to a pressure circuit on the other side. Only constant internal pressure

(actively controlled) could be applied on this setup. The HRT temperature was 400°C, which is commonly used in this kind of study while being representative of peak cladding temperature during SNF transportation according to EDF R&D thermal finite-element analyses. According to [21], around 200 ppm of hydrogen is dissolved at this temperature and precipitation during cooling occurs at 320°C. Heat-up rate was around 8°C.min^{-1} and cooling rate was close to 1.5°C.min^{-1}, which is quite low for the SNF unloading after transportation. Simple calculations were computed to assess the cooling rate after unloading, results are illustrated in Figure 1. Typical HRT is as follow:

- The unstressed specimen is heat up to 400°C then maintained for several hours,
- Internal pressure is then applied,
- After two hours, the furnace is turned off and cooling takes place,
- When the temperature is lower than 100°C, the specimen is depressurized.

To investigate the effect of cycling, the process was applied one, two or three times for selected stresses. The list of all HRT specimens of this study is given in Table 1. The choice of experimental conditions is discussed in the results section.

Post-HRT mechanical tests

After HRT, plugs and tube fittings are cut off the specimen and the active length is sliced into 2 mm wide rings (up to 12 rings per specimen). Ring Tensile Tests (RTT) are then performed at room temperature with an opening rate of 1.8 mm.min^{-1}. Mandrels consist of two cylinder halves with a diameter of 8 mm. This type of tensile test is not analytical (there are bending effects to account for) but it was proven to be efficient as an overall ductility indicator [7] using the Displacement Energy Density (DED), which is the area under the displacement-stress curve. The DED of a given HR tube was defined as the average of the DEDs of all the RTT performed on its rings.

Determination of hydrides orientation

After tensile tests, one or two rings per HRT specimen were mechanically polished using a mixture of colloidal silica suspension and hydrogen peroxide during the final step. This provided the specimens with enough chemical contrast to expose the hydrides distribution. Pictures with a magnification of x400 were taken and stitched together to obtain a 300 µm wide view of the thickness, at a location not deformed by RTT. Image analysis was then undertaken to quantify the reorientation phenomenon. There is no general agreement about the best metrics for hydride reorientation. Some use the fraction (linear or surface) of hydrides with an orientation with respect to the radial direction lower than a fixed value (30, 40 or 45°), others use more complex parameters like the radial hydride continuity factor [9]. Because of a lack of a dedicated image analysis software, it was chosen in this study to take into account the orientation of the hydrides contour to define the Radial Hydride Fraction (Figure 3). Hydrides were considered radial if their orientation was in the range [0-30°] and the radial hydride fraction is named f_{0-30} in the following. From multiple analysis performed on one ring, the absolute error on f_{0-30} determination is ±2%.

RESULTS

Ductile to brittle transition following one HRT

Previous studies at EDF have shown that the ductility (through the DED metrics) of unirradiated Zircaloy-4 exhibits a ductile-brittle transition in relation with the stress applied during a hydride reorientation treatment at 400°C [22]. This transition can be fitted by a sigmoid function.

Based on these results, one-cycle HRTs were performed to obtain different states of embrittlement from the as-hydrided specimens (Figure 4 and Figure 5). Smooth ductile-brittle transitions were found for both hydrogen contents with small differences between them. The reference DED (as-hydrided) is lower for the high content despite reaching high strain (more than 30% in thickness reduction). This effect is most likely linked to the hydride rim, a brittle phase, which is thicker in high content specimens than in intermediate content ones. The transition curve for the high H-content is slightly shifted towards higher HRT stresses (around 10 MPa).

With the method used for the determination of the radial hydride fraction, f_{0-30} for reference specimens (as-hydrided) was found to be 3.5%. Except for one specimen (H_120_1), f_{0-30} increased with the stress applied during one HRT, up to 13.3% for specimen I_120_1. The correlation between DED and f_{0-30} is exposed in the next section.

From the study of RTT curves and width reduction at failure post-RTT, a DED of 0.15 $J.mm^{-2}$ was considered as the threshold under which the material could be considered brittle, i.e. without clear evidence of macroscopic plasticity. This ductility limit could be translated into maximal allowable stress during HRT with value of 100 and 110 MPa for intermediate and high H-content respectively.

Effect of cycling on ductility

In order to study the effect of consecutive HRTs on the cladding ductility, stresses of 79, 92 and 120 MPa were selected for the intermediate H-content while, due to a shortage of specimens, only 92 and 110 MPa were used for high H-content. These stresses correspond to rod internal pressures at room temperature ranging from 4.8 to 7.3 MPa. Each specimen was subjected to a complete HRT sequence before further investigation, meaning that, for example, specimens subjected to 3 HRTs were not investigated after 1 or 2 HRTs. Figure 6 and Table 1 show the effects of multiple HRTs on the room temperature ductility. No detrimental impact was found after 2 or 3 HTs when compared to the state after one HRT. Even more surprising, it seems that specimens subjected to repeated HRTs recover some ductility.

Except for one sample (H_92_3), tube cross-sections observations and radial hydride fraction determinations were performed. Figure 7 shows the evolution of f_{0-30} with the number of HRTs applied. The results are quite scattered but it seems that more radial hydrides are formed with repeated treatments. The results did not highlighted major differences between the two H-contents.

Figure 8 displays the relationship between ductility and radial hydride fraction. Despite the scatter, a clear trend is highlighted, for both H-contents. The logarithmic fits for each content has the same shape but a shift towards lower DED is found for the high H-content due to the difference of reference state between the two H-contents.

DISCUSSION

The literature about stress-induced hydride reorientation is already quite consistent due to the impact of this phenomenon on the management of fuel assemblies during the back-end of the nuclear cycle. But its variety in experimental approaches and studied materials makes it difficult to draw general conclusions for practical applications. The aim of this study is to be consistent with EDF needs even if that means that experimental conditions deviate from what is generally done. HRT stresses were chosen to be representative of internal pressure in spent fuel rods, not to limit the HR phenomenon. Moreover, the focus was not only put on HR but also on its impact on the cladding ductility which is ultimately the property to ensure.

One cycle HRT

The works performed by Chu [8,23] and Desquines [14] on HR constitute the two main reference points of this study even though their experimental approaches are different: Chu used pressurized cladding tubes at a constant stress of 160 MPa and a HRT temperature of 400°C while Desquines used C-shaped specimen under compression (heterogeneous stress distribution with a maximum of 230 MPa) and temperatures of 350 and 450°C. Both these references have derived models from their results. The predictions of the models differ and their applicability to this study is discussed hereafter. For a H-content of 375 ppm and a stress ranging from 79 to 120 MPa, Desquines's model predicts a radial hydride fraction between 15 and 33% while Chu's model predicts a fraction between 6 and 13%, closer to the experimental range [8-13%] found here. The high H-content is beyond the scope of the two references. The discrepancy with Desquines's model is probably due to the heterogeneous distribution of hydrides in this study (outer rim) showing that hydriding method, and thus the hydride distribution, has a major influence. Others studies with hydride reorientation measurements have also shown the limited amount of radial hydride (<15%) for similar stresses, albeit for different materials (irradiated duplex Zircaloy-4 [10], recrystallized Zircaloy-2 [13], CWSR Zr-Nb [24]). Above 100 MPa, an exponential effect of stress on the radial hydride fraction is highlighted.

From the mechanical point of view, Chu has studied the effect of radial hydrides fraction on the mechanical properties of rings tested in tension along the circumferential direction [8]. Using 1% residual strain as a brittle indicator, Chu found threshold radial hydride fractions of 33% and 6%, for H-content of 320 and 600 ppm respectively. Based on our definition of embrittlement, threshold fractions of 15 and 12% were found in this study for H-contents of 375 and 760 ppm. These discrepancies illustrate the scatter surrounding studies on hydride reorientation due to different laboratory techniques (hydriding, reorientation treatment, hydride reorientation fraction measurements, and post-reorientation ductility determination). Data from Daum provides another point of comparison linking HRT stress to post-HRT ductility in both fresh and irradiated Zircaloy-4 [19], without using the radial hydride fraction as an intermediate variable. Using a ring tensile test during HRT, a ring compression test for post-HRT ductility measurement and finite elements calculations for local stress determination, Daum found that the plastic strain at crack initiation follows a sigmoidal ductile-brittle transition curve with the peak HRT stress for specimens (fresh and irradiated) with a H-content of 300 ppm. For stresses lower than 90 MPa, 8% plastic strain is required to propagate an unstable crack. Above, a smooth transition takes place until the sample can fail in the elastic regime (around 130 MPa). This transition has a similar shape than the ones exhibited in Figure 4 and Figure 5, proving that radial hydride embrittlement is a progressive phenomenon without a cliff effect. Moreover, Desquines found that the stress threshold for hydrides reorientation in Zircaloy-4 is much lower than previously thought (50 MPa) [14]. Thus, hydride reorientation cannot be straightforwardly associated with cladding embrittlement; it is all about the degree of reorientation.

The shift between Daum's transition and the one found in this study is probably due to the difference in metrics used to assess cladding ductility, showing again that a standardization of techniques would be beneficial to improve knowledge.

The absence of an important difference between the two H-contents in this study is probably due to the limited amount of hydrogen dissolved on the cladding during the homogenization step after hydriding. At a temperature of 430°C, the H-content in the specimen's bulk is expected to be around 270 ppm. This raises the question of the relevancy of comparisons with uniformly hydrided samples with high H-contents. No uniform hydride distribution is expected in spent-fuel cladding having more than 100-150 ppm of hydrogen. The effect of hydride distribution, and not the only overall content, on the reorientation phenomenon should be looked into more precisely when studies are performed on unirradiated material.

Effect of multiple HRT

In this study, the results summarized in Figure 6 and Figure 7 show the limited effect of cycling on hydride reorientation and the absence of impact on cladding ductility for hydrogen contents of 375 and 760 ppm. Several studies were carried out to check the influence of cycling on the radial hydride fraction [10,13,16,17,23] with different techniques and materials. The overall conclusions is that thermal cycling (multiple HRTs) enhances the hydride reorientation phenomenon. But a closer look at the data shows some scatter between studies, but also the intertwined effects of several parameters such as H-content, stress applied and peak temperature during HRT.

For example, no sensible effects were highlighted by Chu before the 4th cycle (160 MPa HRT) at high H-content (600 ppm), while an important impact of cycling was exhibited for a H-content of 130 ppm (or 320 ppm) up to the 4th cycle [23]. In another study performed by Chernyaneva on recrystallized Zr-1%Nb with low HRT stresses (<70 MPa), it was shown that the effect of cycling continuously increases with increasing H-content (from 60 to 400 ppm) [17]. These results disagree with the ones obtained by Sakamoto on recrystallized Zircaloy-2 with low H-content (<100 ppm): the effects of cycling are only visible at high stresses (> 150 MPa) [13]. On irradiated duplex Zircaloy-4 cladding (400 ppm), Valance's results tend to prove that HRT cycles (1-3) have additive effects at all stresses and that the reorientation fraction follows Ell's model when the reference fraction (before HRT) is adjusted from previous HRT [10]. These examples prove the complexity of the subject and the need for experimental conditions as relevant as possible to real-life scenarios. In that respect, the study of Billone is probably the most valuable due to several characteristics: use of irradiated material (high burn-up Zirlo with hydride rim), of sealed pre-pressurized specimens for HRT and of ring-compression tests as a ductility indicator [16]. Focusing on the drying phases of spent fuel (ΔT = 100°C), Billone found that three cycles with a peak temperature of 400°C had no effect on radial hydride fraction or post-HRT ductility [16]. However cycling with a peak temperature of 350°C was found to increase the radial hydride fraction and the brittle-to-ductile temperature. While additional tests are required to confirm this trend, this effect is thought to be linked to incomplete hydride dissolution in the cladding bulk (below the rim) at 350°C and preferential dissolution of circumferential hydrides upon re-heating. These results could explain the absence of cycling effects found in this study, provided that they could be extrapolated to complete cooldown.

Another route for explaining the results of this study could be the memory effect as exposed by Valance [10]: when a thermal loading, without stress, follows a HRT, a partial recovery of the circumferential hydride distribution is observed. Because it was not possible to adjust the internal pressure during heating with our test setup, it was decided to apply stress only when the peak temperature is reached. The memory effect supposes that radial hydrides are more prone to dissolution than circumferential hydrides when no stress is applied. With the loading sequence used in this study, hydride reorientation after multiple HRT might be lower than what could be expected with sealed and pre-pressurized specimens.

CONCLUSIONS AND PERSPECTIVES

Within the framework of performing multiple dry transportations of spent nuclear fuel assemblies between wet storage phases, multiple hydride reorientation treatments were performed on unirradiated hydrided (375 and 760 ppm) Zircaloy-4 at 400°C and subsequent mechanical test at room temperature were carried out to determine the residual ductility. The effect of stress on hydride reorientation and material ductility was found to be progressive, with a smooth transition from ductile to brittle (around 100 MPa), without strong differences between the two hydrogen contents. Several stresses ranging from 79 to 120 MPa were selected to carry out the study of the impact of cycling on material ductility. No detrimental effect was found, suggesting that multiple dry transportations would not exacerbate the impact of the hydride reorientation phenomenon.

Further works could be performed to complete this study and extend its conclusions: tests on other cladding materials, investigations with lower H-contents (100-250 ppm), but also sensitivity studies for the estimation of safety margins.

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Table 1. List of tests with main results.

375 ppm						760 ppm					
Specimen	HRT		DED (J/mm²)		$f_{0\text{-}30}$	Specimen	HRT		DED (J/mm²)		$f_{0\text{-}30}$
	Stress (MPa)	cycles	avg.	SD			Stress (Mpa)	cycles	avg.	SD	
I_0_0	0	0	0.535	0.027	3.6%	H_0_0	0	0	0.474	0.009	3.5%
I_79_1	79	1	0.325	0.072	7.6%	H_79_1	79	1	0.364	0.059	4.3%
I_79_2	79	2	0.280	0.088	8.6%	H_92_1	92	1	0.262	0.074	6.8%
I_79_3	79	3	0.384	0.079	6.4%	H_92_2	92	2	0.294	0.084	8.0%
I_92_1	92	1	0.197	0.038	8.3%	H_92_3	92	3	0.314	0.109	N.D.
I_92_2	92	2	0.234	0.050	14.4%	H_110_1	110	1	0.156	0.050	10.8%
I_92_3	92	3	0.335	0.066	11.8%	H_110_2	110	2	0.209	0.070	10.4%
I_120_1	120	1	0.072	0.022	13.3%	H_110_3	110	3	0.206	0.042	13.7%
I_120_2	120	2	0.086	0.024	12.9%	H_120_1	120	1	0.123	0.027	8.4%
I_120_3	120	3	0.117	0.018	18.5%						

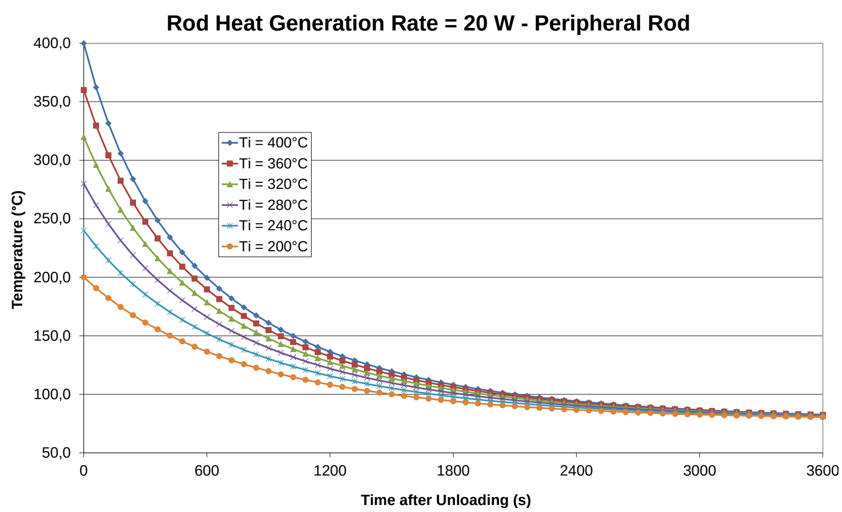

Figure 1: SNF fuel rod cooling evolution for different initial temperatures.

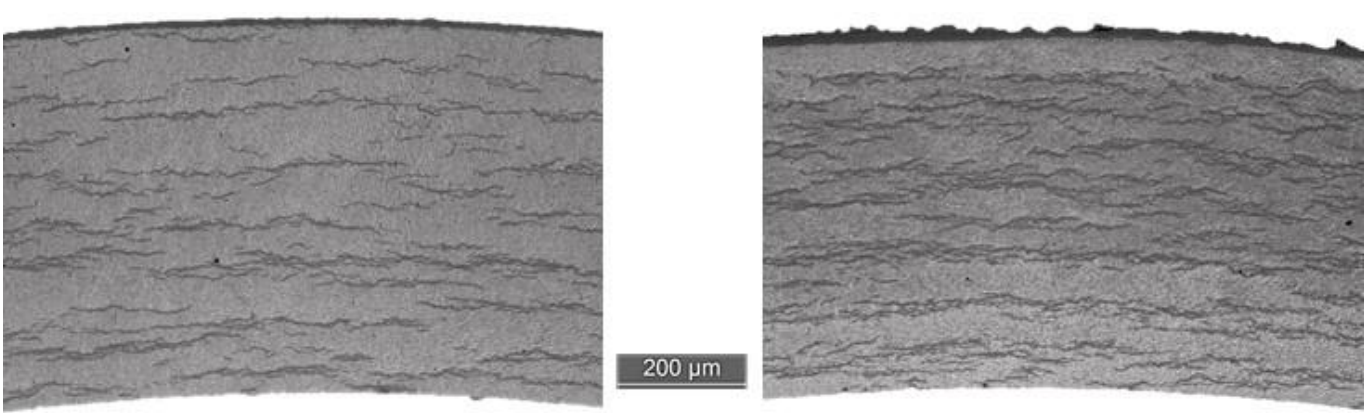

Figure 2. As-hydrided specimens of intermediate (left) and high (right) H-contents. The dark phase on the outer part of the cladding is the hydride rim.

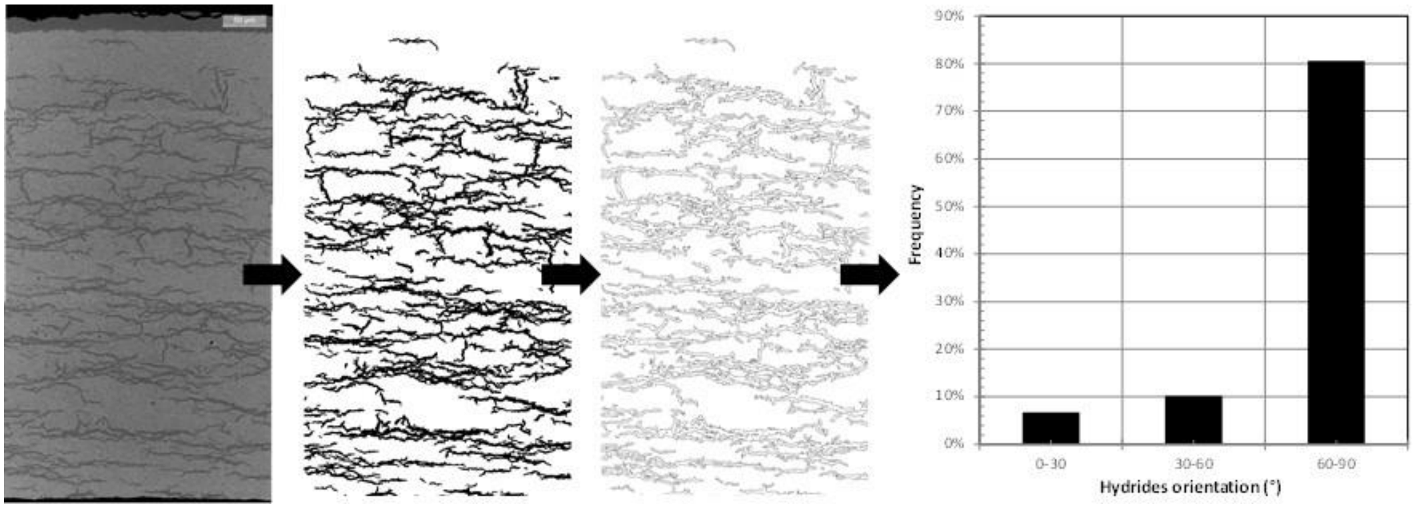

Figure 3. Assessment of hydrides orientation based on image analysis.

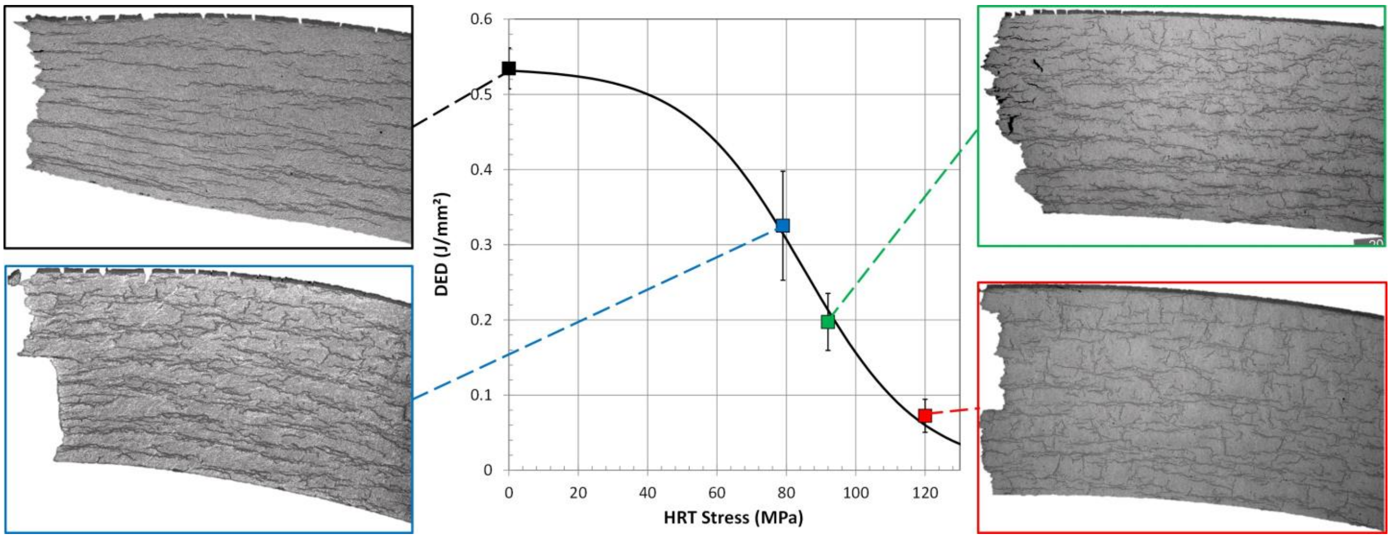

Figure 4. Impact of HRT stress on the DED and the fracture morphology after one-cycle HRT on specimens of intermediate H-content.

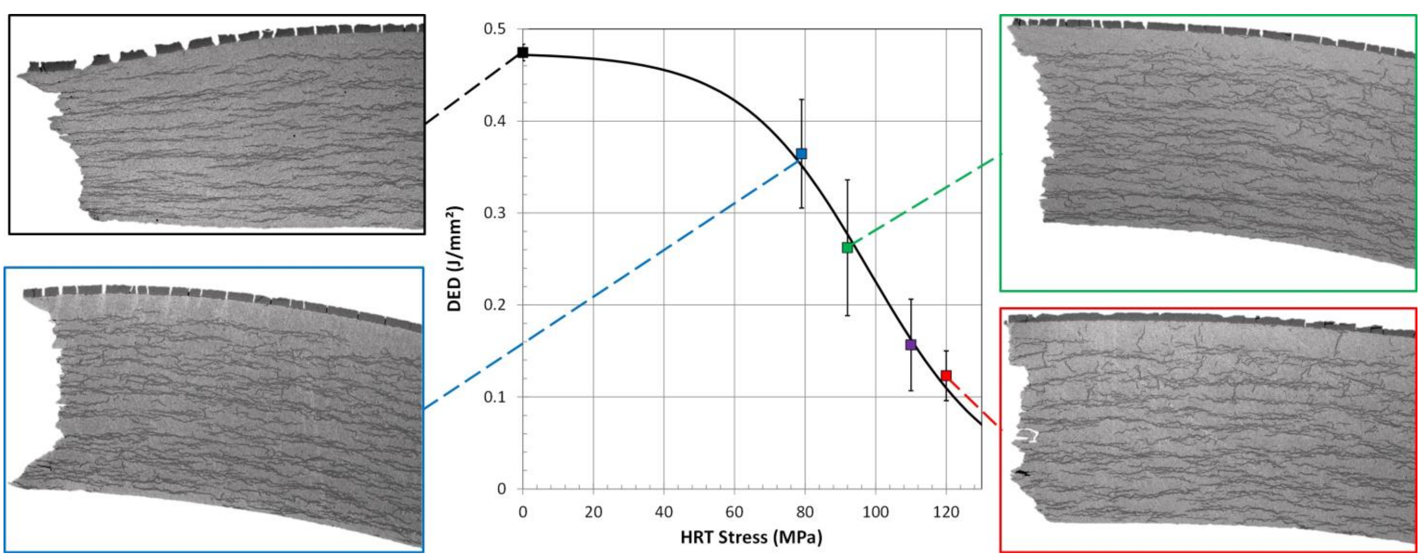

Figure 5. Impact of HRT stress on the DED and the fracture morphology after one-cycle HRT on specimens of high H-content.

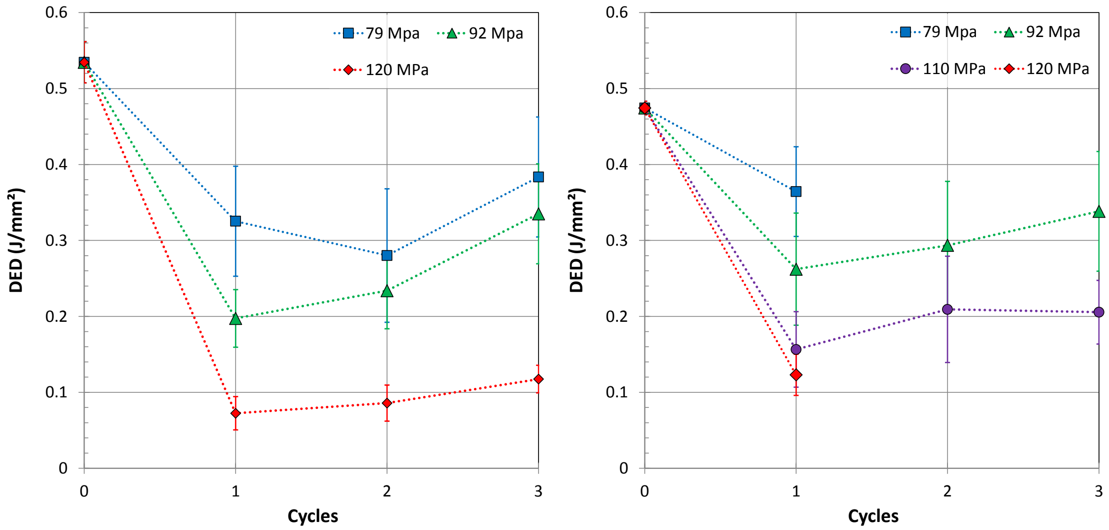

Figure 6. Effect of HRT number on the ductility of Zircaloy-4 cladding with intermediate (left) and high (right) H-content.

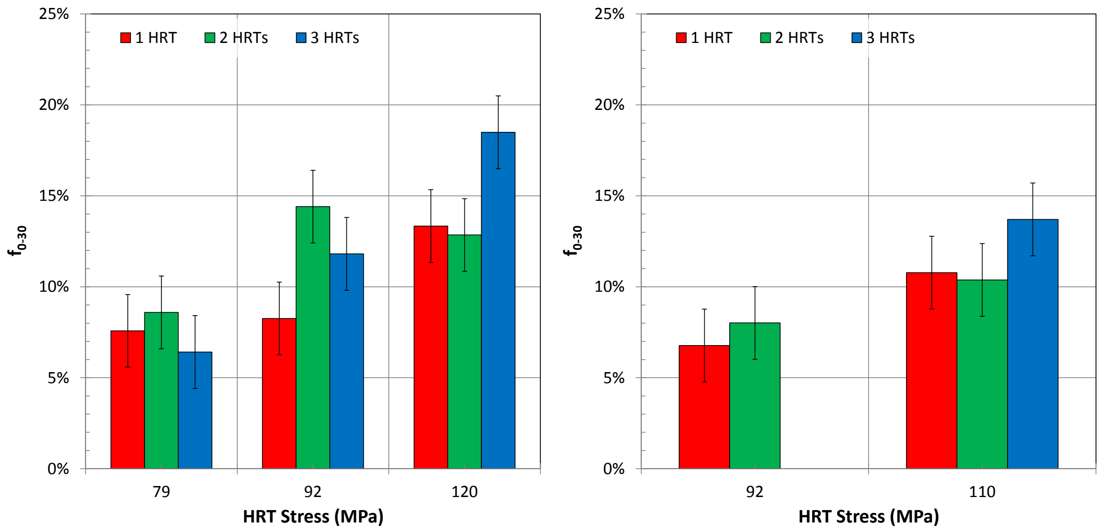

Figure 7. Effect of HRT on radial hydrides fraction in specimens of intermediate (left) and high (right) H-content.

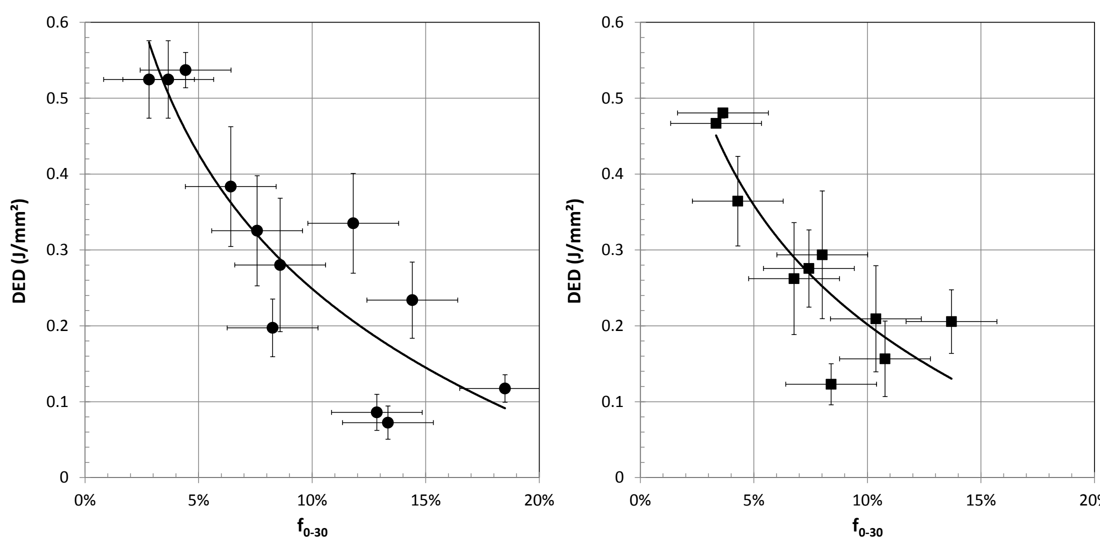

Figure 8. Correlation between DED and radial hydride fraction for intermediate (left) and high (right) H-content specimens.