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Influence of recoil-implanted and thermally released iodine on I-SCC of Zircaloy-4 in PCI-conditions: chemical aspects

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Abstract

The pellet cladding interaction (PCI) phenomenon can lead to cladding failure by iodine induced stress corrosion cracking (I-SCC) during power transients. In these situations, the aggressive species is present as both, recoil implanted iodine in the cladding, and gaseous iodine thermally released in the gap. The aim of this work is to determine the respective roles of implanted and gaseous iodine in the SCC phenomenon. Two types of SCC tests have thus been performed. In the first one, zirconium and iodine recoil implanted tensile test specimens were used, with implantation profiles typical of those existing in a cladding under PCI conditions either on the dose or on the induced damage standpoint. These tests have shown that recoil implanted iodine has no chemical effect on the development of the SCC cracks. The second type of tests was performed on reference tensile test specimens at 350°C with iodine released either, in oxygen containing atmospheres or, at increasing temperatures. The iodine efficiency for cracks initiation was found to be stronger when no oxygen is available for repassivation and when iodine is released at higher temperature. These two conditions being fulfilled during PCI loading, since no gaseous oxygen is available in the fuel-to-clad gap, and since iodine is released through the pellet radical cracks at high temperature, thermally released iodine can be considered as the chemical active species responsible for SCC.

1. Introduction

Pressurized water reactor (PWR) operation in ‘load following’ mode can occasionally lead to significant variations in the reactor fission power, and, as a consequence, in the thermal conditions prevailing in the fuel rods. Upon these variations, due to the expansion of uranium dioxide pellets constituting the nuclear fuel, the cladding surrounding these pellets can be subjected to mechanical stresses which may locally become very high. The combination of these mechanical stresses and the existence of a complex thermal and chemical environment within the fuel rod produces a physico-chemical interaction between the fuel pellets and the Zircaloy-4 cladding, which is known as the pellet cladding interaction (PCI). During a power transient, this specific interaction can lead to cladding failure through a mechanism identified in laboratory as iodine induced stress corrosion cracking (I-SCC). Iodine is effectively one of the fission products, and the failure surfaces obtained during laboratory I-SCC tests have been considered as relevant to PCI failures [1,2].

Iodine is present in the gap as a result of the constant limited release of volatile fission products and gases during irradiation, but mostly because of the sudden and large release occurring upon power transients. There are nonetheless diverging opinions in the literature as to whether there is a sufficient quantity of free iodine present in the gap to allow SCC. The iodine concentration threshold determined in laboratory for Zircaloy SCC ranges between 10^{-3} and 2.5 mg/cm^2 [3–5].

In a recent calculation, taking into account local release of iodine either in the inter-pellet zone or in the radical cracks of the pellet, as well as the CsI radiolysis

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efficiency depending on the local geometry, it has been shown that the concentration of free iodine in the gap can reach a value of 0.4 mg/cm^2 which is in good agreement with laboratory thresholds [6].

On the other hand, while many tests and calculations have been carried out to study the influence of gaseous iodine in the pellet-cladding gap on PCI-SCC failures, very little work has focussed on the role of recoil-implanted iodine in the cladding [7,8]. Owing to its kinetic energy (~73 MeV), the iodine created by fission in the peripheral zone of the pellet can be implanted by recoil in the cladding, thereby contributing to its mechanical surface deterioration, and leading to the presence of the chemical species responsible for SCC within the material. Since the stress corrosion cracks observed are, in fact, distributed in the surface layer damaged by the fission product recoils [9], it seems interesting to verify the exact role of implanted iodine, either from the chemical standpoint or with regard to implant-induced damage.

In this context, in addition to the calculations published in Ref. [6], the aim of this work is to determine the respective role of implanted and thermally released iodine on the SCC of Zircaloy-4 claddings after power transients. For this purpose, two types of tests have been performed.

Zirconium and iodine implanted tensile test specimens have been prepared with implantation profiles typical of those existing in the cladding under PCI conditions, either on the dose or on the induced damage standpoint. They have then undergone classical SCC tests in iodine environment.

On reference tensile test specimens, SCC tests have been carried out in iodine vapor, released at increasing temperatures, in order to simulate a release flowing through the pellet cracks, and combined with atmospheres containing increasing amounts of oxygen in order to estimate the influence of a passivation phenomenon on the SCC development.

The respective role of recoil implanted iodine and thermally released iodine is then discussed on a chemical standpoint.

2. Experimental procedure

Tensile test specimens with an active length of 12.5 mm were machined in two recrystallized sheets supplied by CEZUS, one of pure zirconium and the other of Zircaloy-4 with a standard composition (1.40% Sn, 0.20% Fe, 0.11% Cr, 0.13% O). Their thicknesses were 800 μm.

Implanted test specimens were first mechanically polished on one side, then iodine or zirconium implanted. The implantation characteristics are described in detail in the following section.

The test specimens were subjected to slow tensile tests at $\varepsilon = 4.7 \times 10^{-6}$ s^{-1} either until rupture or interrupted at 3% total plastic strain in order to study a strain range not too far from fuel rod local strain in PCI loading [10].

The implanted specimens, have been tested in iodine methanol at 25°C in order to overcome the passivation effects unavoidable in tests performed at high temperatures, and also to highlight a possible implantation induced damage effect. The iodine concentration was 5×10^{-6} gram of iodine per gram of methanol.

To study the influence of the iodine release temperature and the oxygen residual pressure on the SCC development, tests have been carried out at 350°C, iodine being released at 25°C before raising the temperature, or at 350°C during the temperature plateau. Iodine was combined with air, argon or vacuum.

All the tests have been duplicated with tests performed either on reference material (i.e. non-implanted) or in reference atmosphere (argon). All the tests characteristics are summarized in Table 1.

In order to characterize the development of stress–corrosion cracks either on the surface or in depth, a three-dimensional characterization technique classically used for steels or aluminum alloys has been adapted to zirconium alloys: cracks are first observed and characterized on the previously polished side of the test specimen which is then edge-coated. Successive polishing steps and optical microscopy observations are then performed in order to determine the in-depth crack size. This type of SCC crack characterization made on

Table 1
Characteristics of the tests

Temperatures (°C)	Atmospheres	Tested specimens
350	Argon	Zr: 2 (R)[a] Zry-4: 3 (R)
350	Air	Zr: 1 (R) Zry-4: 1 (R)
350	Iodine vapor released at 25°C in air	Zr: 2 (R)
350	Iodine vapor released at 25°C in argon	Zr: 1 (R)
350	Iodine vapor released at 350°C in air	Zr: 2 (R) Zry-4: 1 (R)
350	Iodine vapor released at 350°C in vacuum	Zr: 2 (R) Zry-4: 3 (R)
25	Iodine methanol	Non-implanted Zry-4: 4 (3%)[a] Zry-4: 3 (R)
25	Iodine methanol	Iodine implanted Zry-4: 2 (3%) Zry-4: 2 (R)
25	Iodine methanol	Zirconium implanted Zry-4: 2 (3%) Zry-4: 2 (R)

[a] Strained to rupture (R) or to 3% plastic strain (3%).

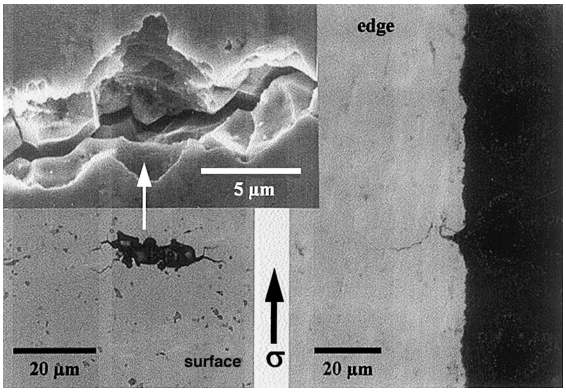


Fig. 1. Zircaloy-4 strained to 3% plastic strain in iodine methanol. Typical 3D observation of an SCC crack.

zirconium alloys is illustrated on Fig. 1. Fracture surfaces have been characterized by SEM.

3. Influence of recoil-implanted iodine on the susceptibility of Zircaloy-4 towards SCC

3.1. Implantation characteristics

To realize implanted specimens as close to fuel cladding as possible, the iodine recoil implantation profile obtained in the cladding has been calculated for the case of a two cycle PWR fuel rod submitted to a power transient. This calculation is detailed in Ref. [6].

For the chosen history, the number of iodine atoms created by fission is 6.4×10^{18} atoms/cm^3, most of them (90%) being stable or very long life isotopes (^{127}I and ^{129}I). The number of iodine atoms leaving the pellet per surface area corresponds to the iodine dose implanted in the cladding at the end of the power history. It reaches 10^{15} atoms/cm^2. The number of iodine atoms leaving the pellet with a given energy is calculated to establish the distribution profile of recoil-implanted iodine atoms in the cladding and the associated damage. Iodine atoms have a maximal range of 7.5 µm; they are preferentially implanted at the surface, where iodine concentration reaches 120 atomic ppm, and where the associated damage is equal to 1.4 dpa (Fig. 2).

The iodine implantations have been designed to produce an iodine profile in the test specimens similar to the one shown in Fig. 2. Zirconium implantations, on the other hand, were designed to separate the chemical role of the implanted iodine from its role as a source of damage.

To produce an implant profile over several microns, ion energies of the order of several MeV are required, thus involving the use of a coupled accelerator and implanter. In addition, iodine and zirconium ion sources, being difficult to obtain, are not frequently proposed. With the ARAMIS tandem implanter at the Orsay Nuclear Spectrometry and Mass Spectrometry Centre it was possible to achieve iodine implantations up to 14 MeV, corresponding to a recoil distance of 3.7 µm in Zircaloy-4, and zirconium implantations up to 10 MeV corresponding to a recoil distance of 2.5 µm. These implantation conditions have been considered as satisfactory since they allow to reproduce most of the iodine atoms profile present in a power ramp situation.

The implantations are made with a fixed incidence angle perpendicular to the surface of the material; several implanting runs had therefore to be made with variable energies and doses to reproduce accurately the real profile established in Fig. 2. The TRIM software was used to determine precisely these different iodine implantation energies and appropriate doses. A good balance was found with the conditions described in Table 2(a).

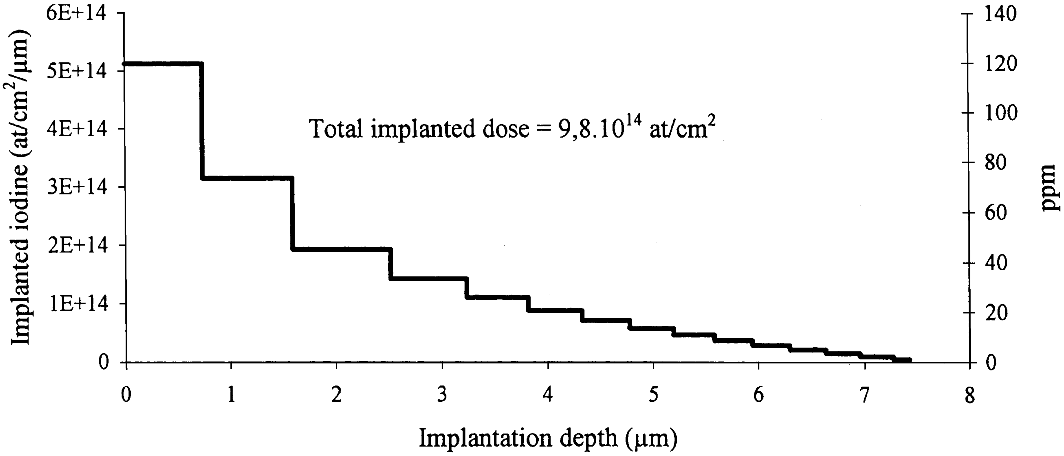


Fig. 2. Iodine recoil-implant profile established by calculation (from Ref. [6]).

Table 2
Energies and fraction of the dose for (a) iodine implantations and (b) zirconium implantations

Successive implantation energies and respective fraction of the dose				
(a) Iodine	14 MeV 20%	8 MeV 26,1%	4.5 MeV 27,2%	2.4 MeV 26,7%
(b) Zirconium	10 MeV 20%	5.5 MeV 26,1%	3 MeV 27,2%	1.4 MeV 26,7%

Zirconium implantation conditions were determined to produce a damage similar to that created by the iodine implantation. Consequently, zirconium implantations were also made in four runs, but with a 30% higher dose. The characteristics of these zirconium implants are presented in Table 2(b).

The implanted iodine profile formed with the above conditions has been characterized by SIMS analysis (primary source Cs^+, analysis of negative secondary ions), this method being classically used for determining ion implantation profiles [11]. The iodine profile experimentally produced and characterized is presented in Fig. 3. It appears to be in good agreement with the iodine recoil-implant profile established for a two cycle fuel cladding.

3.2. SCC results on implanted specimens

All the mechanical characteristics (strain at failure (A%), uniform strain (ε_u), yield stress ($\sigma_{0.2}$), and maximum stress (σ_R)) of the tests achieved to rupture, in iodine methanol, at room temperature, on iodine implanted, zirconium implanted and non-implanted specimens are gathered on Table 3.

For these three cases, results are rather similar, indicating that the surface recoil implantation does not have any influence on the mechanical behavior and on the SCC susceptibility of the material. It is worth noting that, since the tests were performed at room temperature, any possible implantation induced surface embrittlement would have been highlighted in these tests.

The absence of any effect of a surface recoil-implantation on the SCC susceptibility seems to be confirmed by some additional tests performed in iodine vapor at 350°C [10]. More work is however needed concerning this point.

Fig. 4 gives the cumulative distribution of the in-depth size of the cracks initiated on two implanted specimens for the two cases studied: iodine implantation and zirconium implantation. These distributions are compared to the distribution area defined by merging the results obtained on ten non-implanted specimens tested during two test series. This distribution area gives moreover a value of the dispersion range obtained on similar specimens tested in different test series: about 15 μm. The dispersion between the results obtained on different samples (iodine implanted, zirconium implanted or non-implanted) during one test series appears to be lower than that obtained on identical samples over two distinct test series. It can therefore be concluded that iodine or zirconium implants do not affect in depth crack growth.

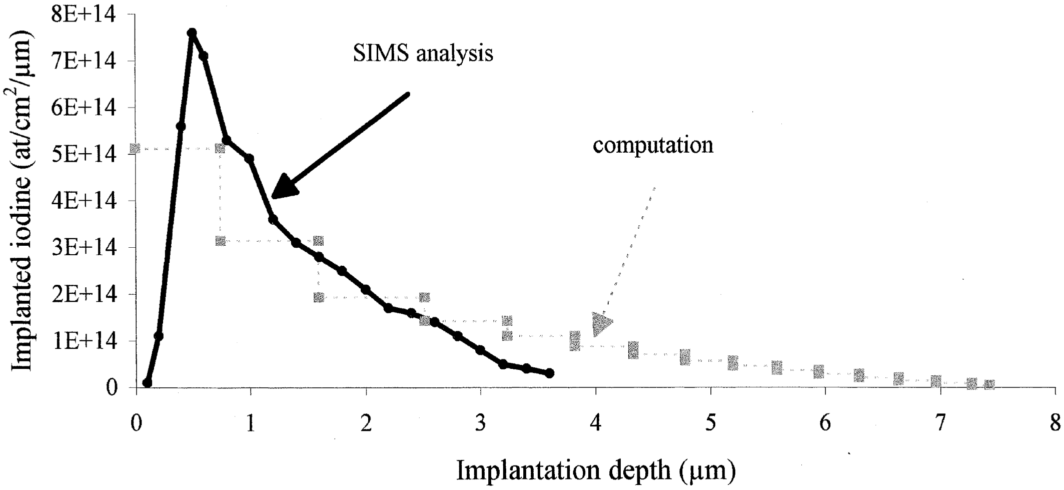


Fig. 3. SIMS iodine profile made on an iodine implanted specimen, compared to the iodine recoil-implant calculated profile.

Table 3
Mechanical characteristics measured in iodine methanol at room temperature

Nature of the specimen	A (%)	ε_u (%)	σ_R (MPa)	$\sigma_{0.2}$ (MPa)
Non implanted	18	7	440	360
Iodine implanted	18	8	425	365
Zirconium implanted	17	9	430	360

3.3. Comments and discussion

At this stage, some conclusions regarding the impact of mechanical damage and/or superficial chemical doping on the SCC susceptibility of Zircaloy-4 can already be drawn.

The superficial mechanical damage associated with recoil implantation, separated from any possible chemical effect, thanks to zirconium implantations, proves to be insufficient to cause any change in the mechanical behavior of the material. However, the damage induced by implanting, about 1.4 dpa over a maximum depth of 4 μm on the surface, is nonetheless of the same order of magnitude as that induced by neutron irradiation (1.6 dpa) through the entire thickness of identical tensile test specimens subjected to the same type of test and which have shown significant changes in their SCC susceptibility [10]. Since defects created by neutron irradiation or by ion irradiation (mainly dislocation loops) are of the same nature [12], it may be concluded that a surface damage is not sufficient to affect the SCC susceptibility of the material, whereas a bulk damage, by modifying drastically the whole mechanical behavior of the irradiated material, has a strong effect on stress-corrosion crack initiation and growth [10,13].

On the other hand, the iodine implanted dose, calculated to be typical of iodine recoil-implantation profile after two irradiation cycles and a power transient, appears to have no chemical effect on the SCC susceptibility of the material at room temperature and a similar tendency seems to be obtained at 350°C. At 350°C however, a diffusion phenomenon of implanted iodine, limited during laboratory tests and more pronounced during reactor life cannot be excluded. Yet, an iodine implanted specimen underwent a thermal treatment of 16 h at 350°C in a primary vacuum, typical of the time to failure in a slow strain rate test, after which the implantation profile was characterized by SIMS (Fig. 5). The comparison between the profiles determined before and after heat treatment shows that,

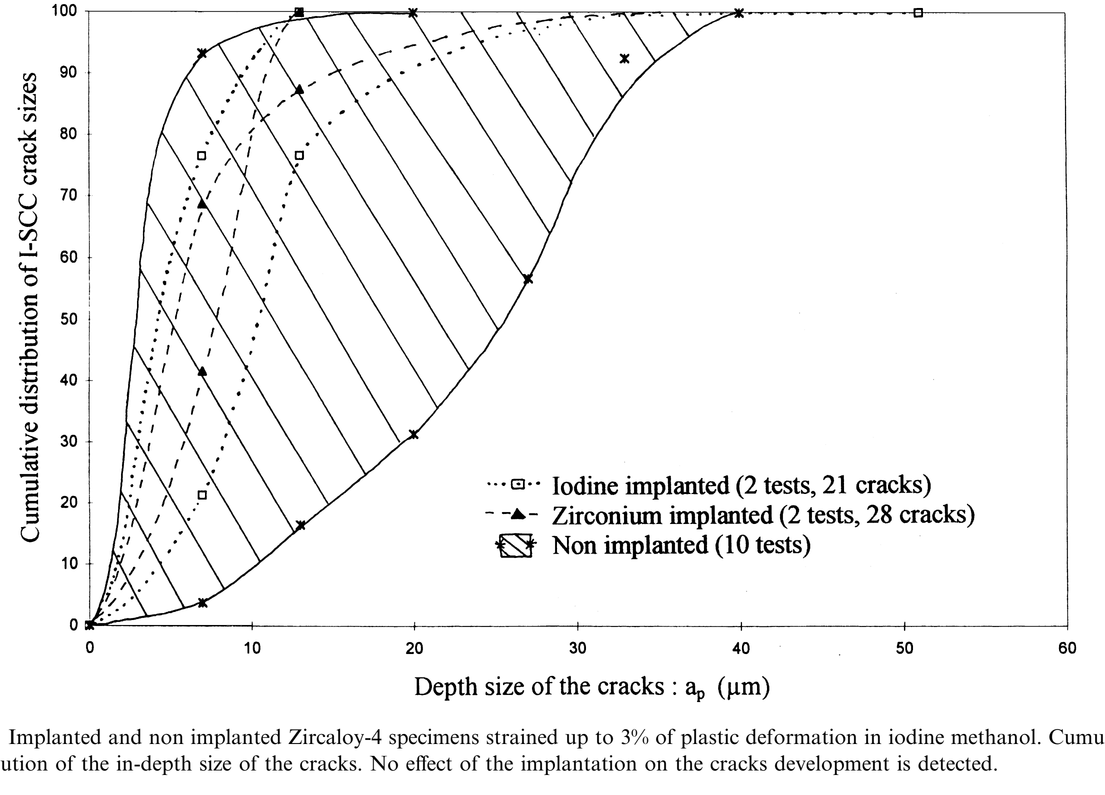


Fig. 4. Implanted and non implanted Zircaloy-4 specimens strained up to 3% of plastic deformation in iodine methanol. Cumulative distribution of the in-depth size of the cracks. No effect of the implantation on the cracks development is detected.

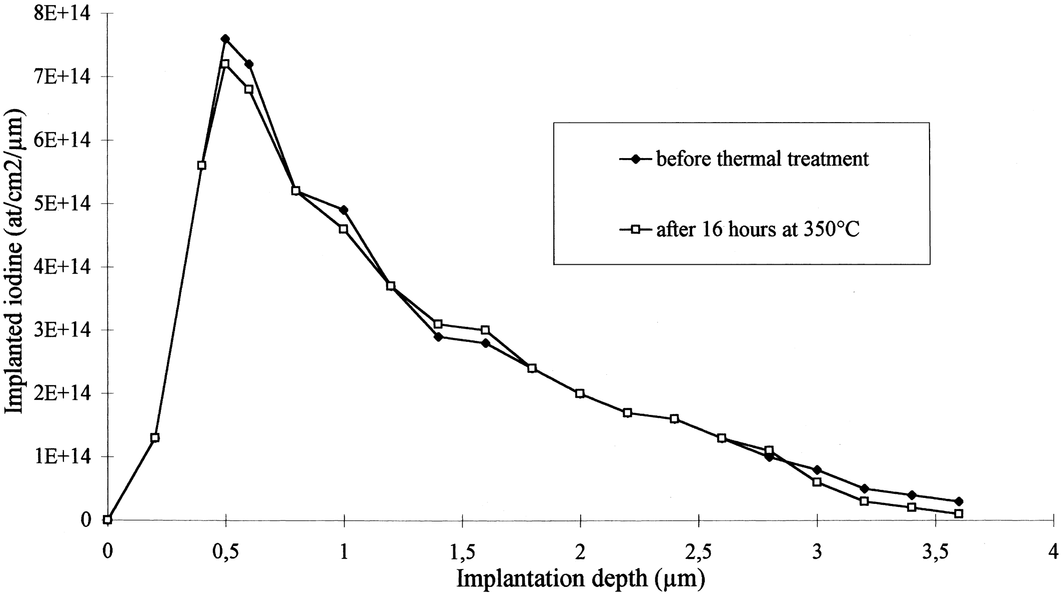


Fig. 5. SIMS iodine profile made before and after thermal treatment.

in these conditions, iodine has not significantly diffused within the material. An upper boundary of the diffusion coefficient $D^{\mathrm{I}}_{\mathrm{Zr}}$ of iodine in zirconium at 350°C can be deduced from these profiles. Indeed, a difference of 0.1 µm between the two profiles could have been detected. Thus, a diffusion distance lower than 0.1 µm, covered in 16 h leads to a $D^{\mathrm{I}}_{\mathrm{Zr}}$ value lower than 2×10^{-19} m^2 s^{-1}. This upper boundary is very closed to that proposed by [14]. Further more, due to its atomic size, iodine can only diffuse by a vacancy mechanism, which diffusion coefficients are ranging at 350°C between 10^{-26} to 10^{-32} m^2 s^{-1} [15]. These values are very low, indicating that iodine diffusion within the material, cannot have any effect during laboratory tests and that, in reactor, if it occurs, it should remain limited.

All these conclusions indicate that recoil-implanted iodine does not have any effect on SCC failures. This first part of the study gives an experimental confirmation of the result sounded out by a calculation of the amount of recoil-implanted iodine and thermally released iodine during PCI loading. Indeed, according to this calculation, an average concentration of 0.2×10^{-3} mg iodine/cm^2 is recoil-implanted in the cladding after a two PWR irradiation cycles, which is below the SCC concentration thresholds, while the local surface concentration of iodine in the gap can reach 0.4 mg/cm^2, when released through the radial cracks of the pellet [6]. As a consequence, according to the SCC concentration thresholds reported in the literature [3–5], the iodine thermally released from the pellet can be considered as the species responsible for SCC failures.

4. Influence of chemical conditions during thermal release of iodine on the susceptibility of zirconium and Zircaloy-4 towards SCC

Nevertheless, the comparison between the amount of free iodine present in the gap and the SCC concentration thresholds established in laboratory disregards the potential effects of thermochemical parameters such as the presence of residual oxygen in laboratory tests or the release temperature of iodine in the gap on the stress corrosion initiation. This point constitutes the aim of the second part of this study, on an experimental standpoint.

All the forthcoming experiments, were achieved with an amount of available iodine well above the SCC threshold, as it has been estimated in the range of 1–100 mg/cm^2.

4.1. Influence of residual oxygen on the cracks initiation

Some reference tests have first been performed without iodine, either in argon, the usual inert atmosphere, or in air, at 350°C. Zircaloy-4 specimens as well as zirconium ones exhibit exactly the same behavior when tested in air or in argon. In both cases, their fracture surface is completely ductile (Fig. 6), and their mechanical properties remain identical.

In the presence of iodine, Zircaloy-4 specimens hardly show any significant difference of their SCC susceptibility whether iodine is released in air or in secondary vacuum. In both cases, cracks are first intergranular and then propagate on a transgranular manner

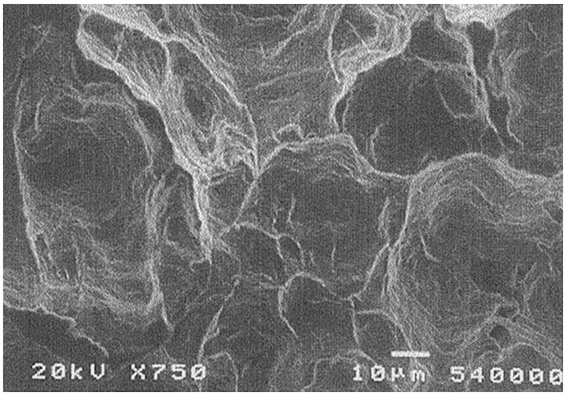


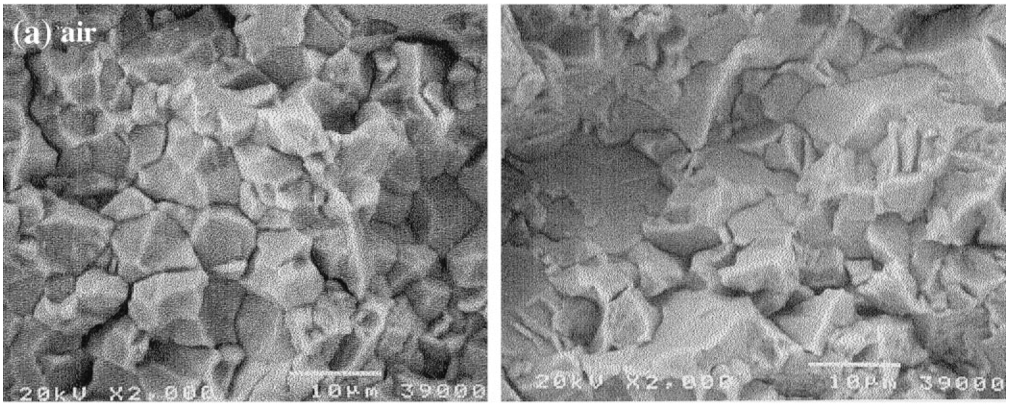
Fig. 6. Zirconium loaded at 350°C in air or in argon: ductile dimples all over the fracture surface.

by pseudo-cleavage (Fig. 7). However, the features of the fracture surface composed of large and numerous embrittled areas distributed all around the fracture surface makes the comparison difficult.

On the contrary, some differences are detected on the zirconium specimens. Indeed, since pure zirconium is less susceptible toward SCC, iodine induced embrittlement remains limited and can be accurately characterized by the few well-defined intergranular cracks on the fracture surface. If the morphology of these cracks remain very close whether iodine is released in air or in vacuum (Fig. 8), their number and length appear to be rather different. In Fig. 9, the number of intergranular cracks observed on the fracture surfaces is plotted versus their size for both cases, i.e. iodine released at 350°C in air or in vacuum. The number and the length of the SCC intergranular cracks are lower when iodine is released in air (4 cracks detected over 2 rupture surfaces) than when iodine is released in vacuum (15 cracks detected over 2 rupture surfaces). This result indicates that iodine is less aggressive for the initiation and the intergranular development of the SCC cracks when combined to air.

4.2. *Influence of iodine release temperature*

As the results obtained on Zircaloy-4 do not seem discriminating, due to its high susceptibility to iodine



IG development

mixed IG and TG propagation

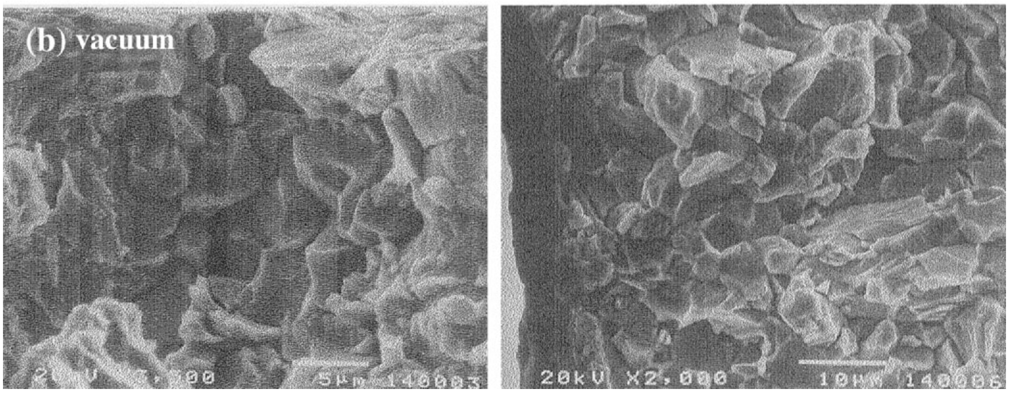


Fig. 7. Zircaloy-4 loaded in iodine vapor at 350°C, with iodine released in air (a) or in vacuum (b). In both cases, cracks are first intergranular, then mixed inter- and trans-granular.

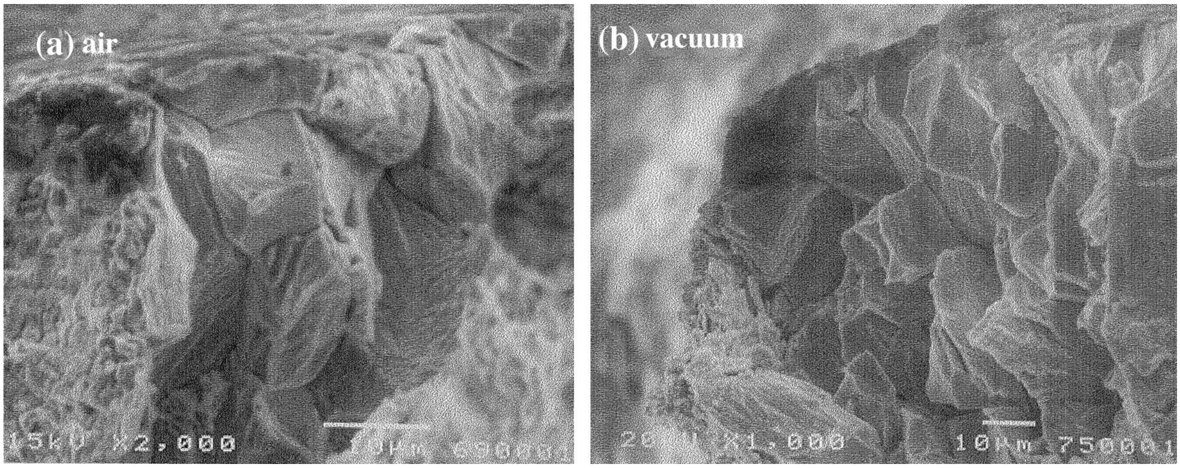


Fig. 8. Zirconium loaded in iodine vapor at 350°C, with iodine released in air (a) or in vacuum (b). In both cases cracks are intergranular.

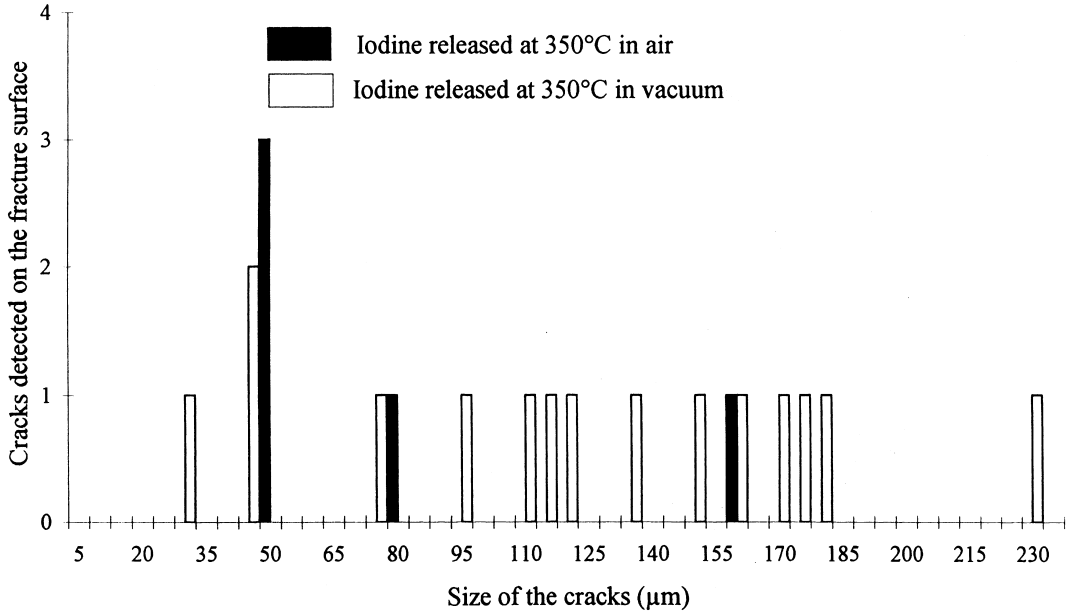


Fig. 9. Zirconium loaded at 350°C in iodine vapor up to rupture. The number of intergranular cracks detected on the fracture surfaces is plotted according to their size. 15 cracks are found over 2 rupture surfaces when iodine is released in vacuum while only 4 cracks are found over 2 rupture surfaces when iodine is released in air.

induced SCC in the laboratory conditions, the following tests, with variable iodine release temperatures, were conducted on zirconium specimens only. Two iodine release temperatures have been studied: 25°C (iodine is released in the chamber before starting the temperature increase) and 350°C (iodine, enclosed in a sealed container, is released when the plateau temperature is reached). It is worth noting that, due to this experimental procedure, since iodine sublimates at about 100°C, the time during which the specimen is exposed to iodine vapor decreases when increasing release temperature.

When iodine is released in air, the SCC behavior is very different depending on the release temperature: after a release at 25°C, no SCC cracks are detected on the zirconium fracture surfaces, whereas after an iodine release at 350°C, as seen in the previous section, several intergranular cracks are observed (Fig. 9).

When iodine is released in an inert atmosphere, either argon or vacuum, the release temperature again appears

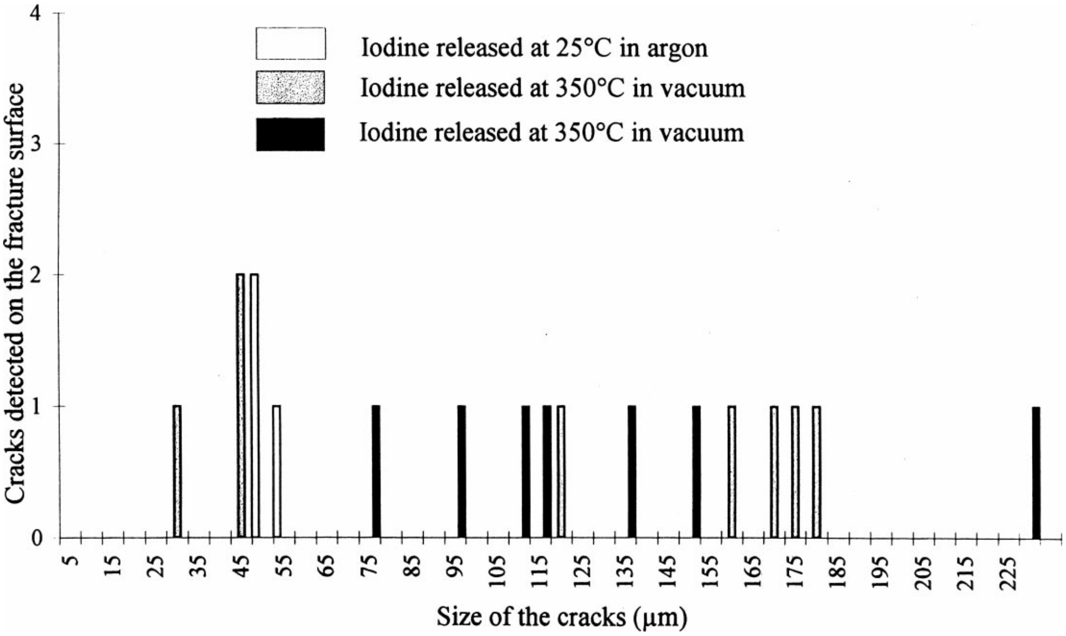


Fig. 10. Zirconium loaded up to rupture with iodine released in an inert atmosphere (argon or vacuum). The number of intergranular cracks detected on the fracture surfaces is plotted according to their size. The higher the iodine release temperature, the more numerous the number of detected cracks.

to have an influence: as shown on Fig. 10, intergranular cracks are more numerous and longer when iodine is released at 350°C. Indeed, after an iodine release at 25°C, 3 cracks are detected over 1 rupture surface, while 15 cracks are observed over 2 rupture surfaces after a release at 350°C.

Since the test duration varies simultaneously with the iodine release temperature, these two experimental parameters have to be separated. When iodine is released at 25°C, one can suppose that, during temperature increase, it gets adsorbed at the specimen surface as soon as sublimated. Before starting loading, when the temperature plateau is reached, it has always been checked that a purple vapor was present in the experimental chamber, indicating a saturation of the iodine partial pressure. When iodine is released at 350°C, this verification was also made before starting loading. So, in both cases, it can be assumed that a similar aggressive environment is available during the tensile test.

On the other hand, when iodine is released at 25°C, some iodine can effectively get adsorbed on the specimen surface during the temperature increase. Owing to the presented results, this adsorbed iodine appears to have no effect on SCC initiation, and confirms the importance of a direct reaction between the naked metal and iodine.

Thus, a clear effect of the release temperature of iodine is established: the higher the temperature, the more severe the SCC intergranular embrittlement.

4.3. Comments and discussion

These experimental results constitute meaningful complements to the calculations of, on the one hand, the available amount of free iodine in the gap, and, in the other hand, the iodine concentration thresholds for SCC.

Indeed, they show that the atmosphere to which iodine is combined, can be determining for the SCC susceptibility of the material. Specifically, even when the available iodine concentration exceeds largely the laboratory concentration thresholds, the presence of air appears to inhibit the cracks initiation and development. At this stage, it is reasonable to assume that such a result is due to a competition between, on the one hand, iodine adsorption and embrittlement of the depassivated metal and, on the other hand, its repassivation with oxygen. As a consequence, the lower the amount of residual oxygen, the more severe the zirconium embrittlement.

Keeping that result in mind, it is interesting to reconsider the signification of the concentration thresholds established in laboratory tests, in the presence of a significant oxygen residual pressure, for the PCI conditions, characterized by an atmosphere in the pellet-cladding gap having a very low oxidizing power. Owing to our results, these laboratory concentration thresholds are likely largely overestimated for the PCI conditions and, as a consequence, the amount of free iodine released in the gap during power transients should clearly be sufficient for SCC to occur. In this respect, Hofmann showed that the presence of an atmosphere enhancing passivation could lead to an increase in the iodine threshold (partial pressure or concentration) required for SCC failure [16].

The very low oxygen partial pressure prevailing in the gap of the fuel rod could then make it easier for the SCC iodine concentration threshold to be exceeded by lowering this PCI-SCC threshold value in relative terms.

On the other hand, the release temperature of iodine in the gap of the fuel rod can also contribute to a relative decrease of the PCI-SCC concentration threshold. Indeed, our results show that the higher the iodine release temperature (between 25°C and 350°C), the more severe the SCC intergranular embrittlement. Under a PCI loading, when iodine is released through the radial cracks of the pellets, it can flow directly from the center of the pellet which temperature reaches about 1000°C. In such condition, the iodine release temperature is undoubtedly higher than 350°C. This can also contribute to promote iodine induced embrittlement of the cladding.

5. Conclusions

The aim of this study was to establish experimentally the specific roles of recoil implanted and thermally released iodine on the cladding failures occurring during reactor power transients due to stress corrosion cracking.

Two types of SCC tests have thus been performed. In the first one, iodine and zirconium recoil implanted tensile test specimens were used, with implantation profiles typical of those existing in a cladding under PCI conditions either on the dose or on the induced damage standpoint. The second type of tests was performed on reference tensile test specimens at 350°C with iodine released either, in oxygen containing atmospheres or, at increasing temperatures. The main results are as follows.

The dose of recoil implanted iodine after a two PWR cycles irradiation, which is about 120 at. ppm at the internal surface of the cladding, corresponding to a damage of 1.4 dpa, appears to be not sufficient to induce any significant embrittlement of the cladding.

Recoil implanted iodine does not significantly diffuse in the material during the laboratory tests; and, as a consequence, an iodine diffusion process cannot account alone for the failures obtained in power transients.

Recoil implanted iodine has no chemical effect on the development of the SCC cracks in laboratory tests. Such a conclusion can be extended to PCI conditions.

Thermally released iodine appears to be the chemical species responsible for PCI-SCC failures moreover, its embrittling role is enhanced under PCI loading by the very low oxygen residual pressure in the fuel rod gap, which limits the repassivation phenomenon, and by the high release temperature of iodine, when emerging from the radial cracks of the pellets.

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