CORROSION AND HYDRIDING OF ZIRCALOY FUEL CLADDING
-THE INSIDE (AND OUTSIDE) STORY

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Abstract

Zirconium alloy fuel cladding corrodes, and absorbs hydrogen, both from the inside and the outside. The detailed mechanisms for oxide growth and hydrogen absorption are quite different, however, because of the different conditions ("protectiveness") of the oxide films formed in the two environments. Historical changes in the understanding of the processes in the two very different chemical environments on the two sides of the cladding are summarised, and current views of the hydrogen uptake process are summarised. The lack of protectiveness of the inside oxide also contributes to easy PCI corrosion.

1. Introduction

Since this is an historical session, it may be interesting to start with an anecdote illustrating the aphorism "that what goes around, comes around". One of the earliest studies of zirconium alloy corrosion that I was involved in was an attempt at AERE Harwell to develop alloys that could be used in the SCHWR (Steam Cooled Heavy Water Reactor), an aborted predecessor of the SGHWR design (Steam Generating Heavy Water Reactor) which, like its close relation Gentilly - 1, got to the prototype stage. SCHWR was to produce a steam-cycle as nearly identical as possible to the standard steam-cycle of U.K. coal fired generating stations (500 - 550°C, 1000 - 1500 psi). After much study of many zirconium alloys in 500 - 600°C steam it became evident that there was an irreducible minimum degradation of all zirconium alloys at these temperatures determined by the diffusion of (and embrittlement by) oxygen in the metal. These rates of embrittlement (even if oxide film growth could be limited) were too high for a reasonable lifetime for any Zr components of acceptable thickness [1]. The hydrogen absorption rates were also very high for many alloys at these temperatures, leading to severe hydrogen embrittlement when cold [2,3], and a severely accelerated oxide growth ("nodular corrosion") was found as steam pressure was increased [3,4,5]. This dilemma has recently been resurrected by the CANDU-X programme, which will have to achieve success by a different route (insulated pressure tubes and ceramic fuel cladding perhaps). However, it is not the purpose of this paper to report on what might have been, but to show how our current views of the corrosion and hydrogen uptake mechanisms have developed over time.

The first zirconium alloy to see extensive in reactor service was Zircaloy-2, whose composition was fixed in August 1952 [6], by Admiral Rickover and the U.S. Naval Reactors Branch. For some time after this, however, details of in-reactor corrosion performance were not available [7]. There had been a number of laboratory studies of the kinetics of oxidation of zirconium (of various purity levels) in pure oxygen [8]. The tendency, therefore, was to compare data on the corrosion kinetics of Zircaloy-2 (and the later modification giving lower hydrogen uptake- Zircaloy-4) with those of pure zirconium in oxygen. It soon became evident that while zirconium corroded worse in steam and water than in oxygen, the behaviour of the best alloys in water could approximate to a lower limit for behaviour given by the oxidation kinetics of high purity zirconium in oxygen at the same temperature (figure 1) [9]. This led to the inference that solid state diffusion processes were controlling both oxidation and hydrogen uptake, and that there was always at least a thin barrier layer oxide at the oxide/metal interface.

When results for oxide growth in water under irradiation became available the tendency was to extend this comparison to these results and look for an "irradiation enhancement factor" to explain any differences between the supposedly diffusion controlled out - and in - reactor corrosion rates [10]. There were clearly two common sets of conditions for these tests. One which gave little or no increase when out - and in - reactor data were compared [8, 11] and the other where visibly accelerated corrosion of a non-uniform, nodular, type (similar to that observed in ≥500°C, 1000 psi steam) was observed (figure 2) [12, 13]. it was soon established that these two conditions corresponded to PWR (hydrogen overpressure) and BWR (excess radiolytic oxygen) water chemistries respectively, and that the behaviour in reactor could be fitted to a pair of curves for corrosion rate and hydrogen uptake rate versus the H : O ratio in the reactor water (figure 3) [14]. The data used to construct this figure included CANDU data showing that, at least initially, some CANDU reactors had apparently operated with water chemistries close to the cross-over between the curves for the two conditions.

There have subsequently been many attempts to explain the causes of nodular corrosion, and to determine whether there really was an irradiation enhancement of corrosion in PWRs. There is not space to summarise all the proposed hypotheses, or experimental results here. Readers are referred to the two IAEA - TECDOCs [15, 16] on this subject, although there are many other possible sources. The second TECDOC (1998) was nearly twice as long as the first one (1993), demonstrating the big change in the availability of in-reactor data between their two respective cut-off dates for inclusion, 1990 and 1995 respectively.

2. The Outside Story

2.1 Understanding the Corrosion Mechanism in High Temperature Water

Understanding what was happening in-reactor proved to be quite difficult so long as attempts were following the hypothesis that it was necessary to modify some solid-state diffusion processes in order to see an effect of irradiation. This was particularly difficult where fuel cladding had been operating under high heat flux conditions. The clue to what was happening came with the observation that the protective ZrO_2 film could be dissolved locally in concentrated LiOH solutions [17-19]. The other key observation was the realization that, if the local temperature within the oxide film (under high heat flux conditions) exceeded the saturation temperature, the water in pores in this film would not just boil and create a steam atmosphere, but would concentrate until the LiOH concentration reached that which had a boiling point equal to the local temperature. Other species (e.g. boric acid in PWRs) might also concentrate by the same mechanism, but since B_2O_3 is volatile in steam, severe boiling tends to deplete it. Thus, there was a mechanism for achieving high LiOH concentrations in small pores where interdiffusion was restricted without maintaining protective high concentrations of boric acid simultaneously . This led to a series of papers [16,20,21] trying to explain the observed phenomena on this basis.

There has been some reluctance to accept that the problems with high burnup, highly rated PWR fuel during long cycles in high outlet temperature reactors could be a simple water chemistry related problem. Thus, much time and expense has been devoted to developing new zirconium alloys to meet the most severe thermal hydraulic conditions. Alloys such as ZIRLO and M5 (a modified Zr-1%Nb alloy) have shown better behaviour than optimised Zircaloy-4 [16,22,23] behaviour, but have not been immune to through wall corrosion failures in the most severe conditions.

2.2 Applicability to CANDU Fuel Cladding

CANDU water chemistry contains LiOH, although in lower concentrations than in PWRs, but contains no boric acid to protect the Zircaloy-4 under boiling conditions. Thermal hydraulic conditions tend to be less severe than for PWRs and channel outlet temperatures are lower. When combined with the comparatively low burnups (7,000 MWd/te in CANDU compared with 70,000-80,000 Mwd/te aimed at for PWRs) this results in oxide thicknesses being usually well below the $10\mu m$ at which the onset of these LiOH concentration effects appears to become severe (figure 4). If fuel burnups were to be significantly increased then the absence of corrosion problems on the outside that has been the norm for CANDU fuel cladding may not persist.

Pressure tubes, of course, have much longer exposures to reactor water than fuel cladding, and oxides routinely exceed $10\mu m$. However, the high heat flux that drives the concentration process is absent. The effectiveness of the concentration mechanism represented by the corrosion process (which removes water molecules from the solution at the bottoms of fine pores) has yet to be clearly demonstrated.

2.3 Nodular Corrosion in Oxygenated and Irradiated Water

Nodular corrosion of fuel cladding has been a significant problem only in BWRs, where the absence of an hydrogen overpressure allows radiolysis, and the boiling partitions the radiolytic products so that an excess of oxygen remains in the water. The only other set of conditions under which a similar appearing nodular corrosion of the Zircaloys occurs is in high pressure (≥ 10 MPa) high temperature (≥ 773 K) steam in laboratory autoclave tests. While such tests have been used to diagnose inferior quality cladding that is particularly susceptible to nodular corrosion, the precise mechanism for nodular formation in BWRs still eludes us, although many hypotheses have been proposed [15,16]. The original Burns and Moore hypothesis [24] invokes a direct relation between oxidising radicals (produced by water radiolysis) and the onset of nodular corrosion. How this could operate when the ZrO_2 surface is a good catalyst for recombination of radical species has never been clear.

Recently Russian authors [16,25,26] have proposed a mechanism in which hydrogen peroxide (present at elevated concentrations during radiolysis) degrades the oxide by enhanced dissolution of ZrO_2. It is not clear whether the Russians intend this dissolution to be highly localised, but since nodular corrosion is a localised form of oxide growth, such localised dissolution must automatically be invoked. Interestingly enough, some early reports of severe corrosion in highly oxygenated water in the laboratory where the oxygen had been added at the start of the experiment (as hydrogen peroxide), but not when it was added as dissolved oxygen (from a high pressure gas cylinder), showed oxide films (figure 5) with appearances very similar to those observed after enhanced corrosion in concentrated LiOH solutions [17-19]. Attempts to find evidence to support an hypothesis that hydrogen peroxide can cause localised dissolution of ZrO_2 (or other minor phases in the oxide film) have so far been unsuccessful.

2.4 Hydrogen Absorption

Very early in the investigations of the corrosion of zirconium alloys [7] it was discovered that the hydrogen content of the alloy increased during corrosion in water or steam. Experiments [30] tended to show that the source of this hydrogen was the water molecules decomposed during the formation of the oxide film, and that molecular hydrogen dissolved in the water was not absorbed unless high hydrogen overpressures (many times greater than those used in reactor) were present [15,16]. Thus, hydrogen uptake was seen in terms of the fraction (%) of corrosion produced hydrogen that was absorbed, and percentage values >100 were thought to indicate the involvement of other processes (e.g. reaction with dry H_2). The temptation was to see the hydrogen uptake mechanism, like the oxidation mechanism, as a solid state diffusion process through a barrier oxide film. This idea seemed to be supported by SIMS profiles (figure 6) for deuterium in the oxide film [31]. However, these profiles did not propagate with time in the expected fashion, and could be exchanged (H for D, and vice-versa) very much more rapidly than the time taken to develop them in the first instance (figure 7) [32]. When it was found that both Li and B isotopes in the oxide could be exchanged almost as rapidly as hydrogen isotopes it was finally concluded that all these species were merely absorbed on pore walls in the oxide and that the profiles were actually the profiles of free surface area that decreased monotonically through the oxide thickness [33,34].

Understanding the hydrogen absorption mechanism was then reduced to answering the question "how far do these pores penetrate through the oxide?" If they penetrate right through to the oxide/metal interface then no diffusion through a barrier oxide is involved. Experiments suggest [33,34] that the small pores and cracks do penetrate to the oxide/metal interface, at least for a brief period of time, and that at any instant there will always be some pores or cracks penetrating to the interface. These active sites have been decorated by depositing Cu on them cathodically (figure 8) [34], and the sites identified when the Cu deposits were subsequently dissolved have usually been small cracks in the oxide, rather than the even smaller pores. This technique tends to decorate the most active sites in preference to less active (smaller) ones. So there may still be small sites that are active for hydrogen absorption, but do not get identified.

Since these pores and cracks that penetrate the oxide will be the cathodic sites at which protons are discharged during the corrosion reaction, the percentage hydrogen uptake will be determined by the ease with which the hydrogen can escape out of these small pores and cracks into the water. Hydrogen which cannot escape will form small hydrogen bubbles at the bottom of these pores, and could then be absorbed directly by the metal, since there will be a period when no barrier oxide is present.

The cathodic current will flow at whatever sites have the lowest electrical resistance, and it is possible therefore for the cathodic current from oxidation of a large area of surface to flow at a small region where the oxide is very conductive. Evidence for this has recently been confirmed [35]. In this instance the cathodic current, and the hydrogen uptake, for some short lengths of fuel cladding occurred at the welded end plugs. Oxide thicknesses at the welds were not significantly greater than on the cladding surface, but the oxides were more conducting. Such an effect can explain the very irregular deuterium distributions seen in some CANDU fuel cladding (figure 9) [36].

3. The Inside Story

Oxide films also form on the inside of fuel cladding, although the conditions under which they form are very different from those under which the oxides on the outsides form. Thermodynamically ZrO_2 is more stable than UO_2 [15,16], so that zirconium will reduce UO_2 if it gets the chance. If the two are not in contact this process is very slow as, in a tightly closed system such as a fuel element, the oxygen partial pressure inside is very low and the kinetics are correspondingly slow. Thus, in LWR fuel, in which the cladding is initially free-standing and there is a gap between the cladding and the UO_2, there is little or no growth of oxide until the cladding creeps down and makes contact with the UO_2. When this occurs oxide growth on the Zircaloy cladding can occur by solid state diffusion from the UO_2 to the Zr. However, although the kinetics of oxide growth are enhanced by contact with the UO_2, the thermodynamics (low pO_2) remain the same so that the oxide properties are typical of those formed in low pressure oxygen environments [9,16]. The growth of such oxides entails a large fraction of the oxygen dissolving in the metal, rather than forming a coherent ZrO_2 film. This results in extensive porosity in the oxide similar to that which develops when "normal" oxide films are heated in vacuum or low pressure hydrogen environments [37]. Metallographic sections of relatively thick oxides formed on the fuel cladding of high burnup LWR fuel have sometimes been so porous that large pores visible in low power optical micrographs have been seen both in the ZrO_2 film and the adjacent UO_2. The development of this "rim effect" in high burnup UO_2 is another story that is not relevant to this discussion.

Thus, the oxides formed on the inside of fuel cladding tend to be very porous and non-protective, and this will be equally true of those formed on CANDU fuel, which, because of the good contact between cladding and UO_2 (even at the start of irradiation) will be formed by oxygen diffusion from the UO_2. Because of the porosity of these films they offer no protection against hydrogen absorption from the inside of the cladding, or against iodine released during power ramps. The CANLUB graphite coating may provide a partial barrier to oxygen diffusion to form the oxide on the Zircaloy, but does not seem to prevent oxide growth completely.

Interdiffusion of carbon into the UO_2 may be a complementary process to oxygen diffusion in the other direction. Hydrogen from the fabrication process can remain entrained in the UO_2 for hundreds of days (provided the centre temperature is low) and the cladding on fuel pins exposed in D_2O in NPD continued to increase in hydrogen content (as well as the deuterium content) throughout its life (figure 10) [12]. This is not hydrogen from residual moisture in the UO_2, which causes "sunburst" type hydriding failures within a few days of reactor startup, but H entrained in the UO_2 during sintering in an H_2 atmosphere.

Conclusion

Our understanding of the mechanisms of corrosion and hydrogen absorption by Zircaloy fuel cladding have changed radically over the last forty years, and are now (possibly) much nearer to reality. In general, problems with CANDU fuel from either oxide growth or hydrogen isotope absorption have not been of any consequence. However, this seems to be largely a result of the short exposure time, and will not necessarily persist if burnups are increased by a large factor.

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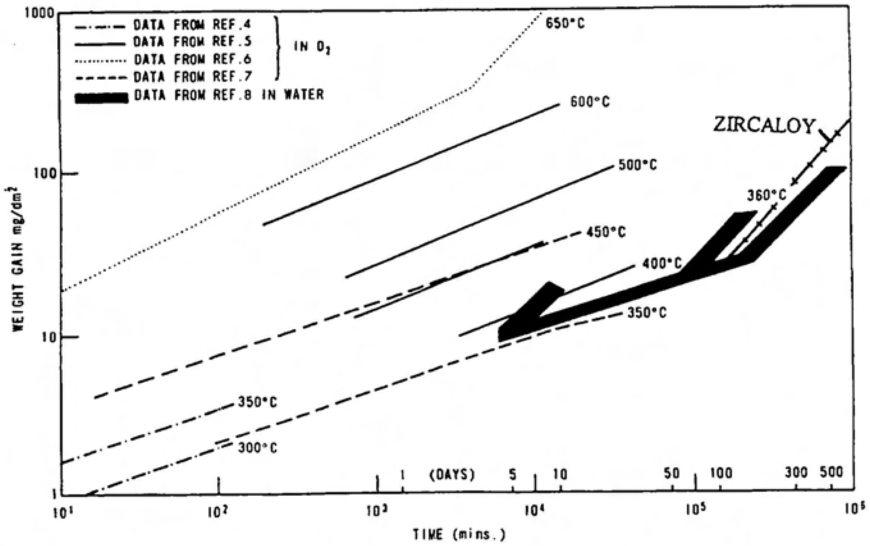


Fig.1. LONG-TERM OXIDATION OF UNALLOYED ZIRCONIUM IN OXYGEN AND WATER AT 300-650°C. THE THREE BRANCHES OF THE WATER CURVE CORRESPOND TO THE BEHAVIOUR OF DIFFERENT GRADES OF ZIRCONIUM [9].

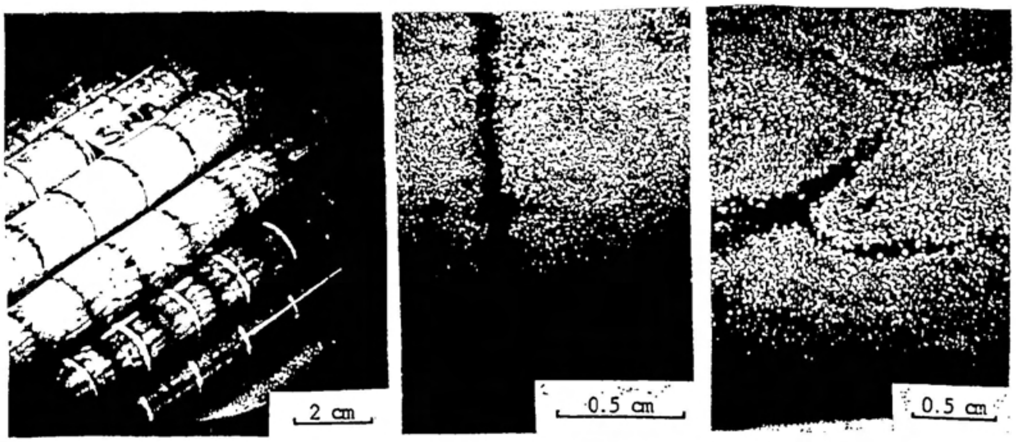


Fig. 2. NODULAR CORROSION IN A BOILING LOOP TEST (U-106) WITH INSUFFICIENT AMMONIA ADDED TO SUPPRESS RADIOLYSIS. CORROSION WAS POSITION DEPENDENT (a) AND PELLET INTERFACES (b) AND CRACKS (c) WERE SITES OF VARIABILITY [12].

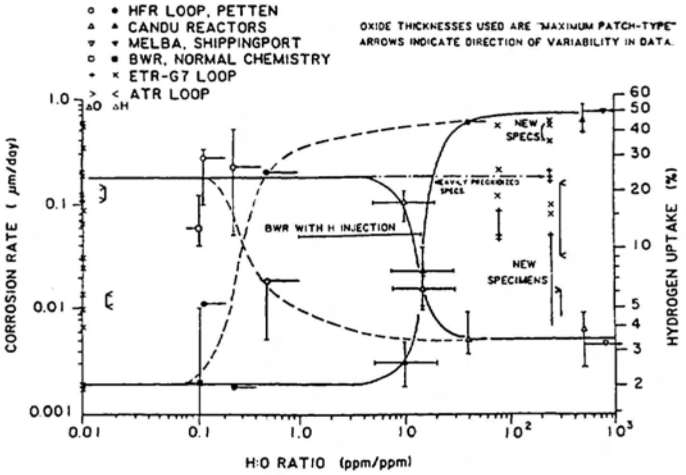


Fig. 3. EFFECT OF WATER CHEMISTRY ON CORROSION AND HYDRIDING OF ZIRCALOY-2 IN-REACTOR [14].

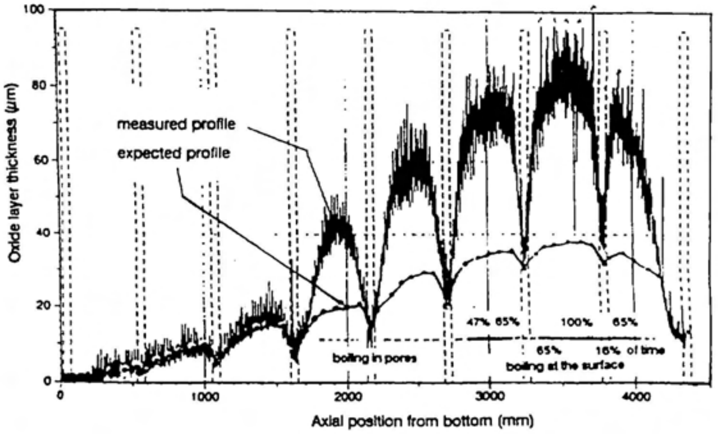
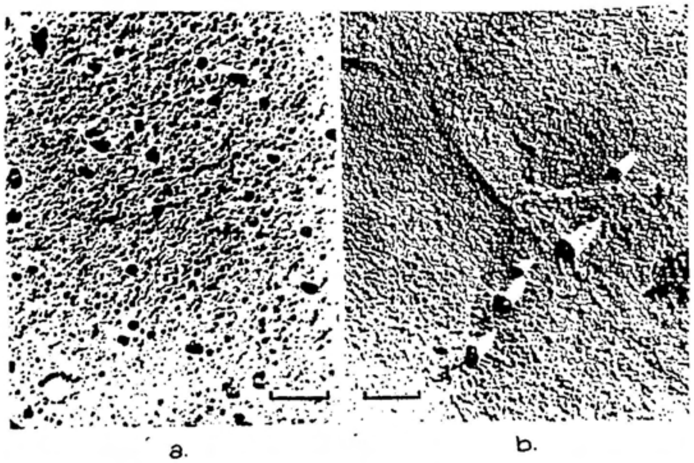


Fig. 4. ACCELERATED CORROSION OF A FUEL ROD EXPOSED FOR 2 CYCLES IN PWR-H.



ig. 5. SIMILARITIES BETWEEN OXIDE SURFACE FEATURES OF IRCALOY SPECIMENS CORRODED IN H_2O_2 (a) AND pH 12 LiOH b) [27,17].

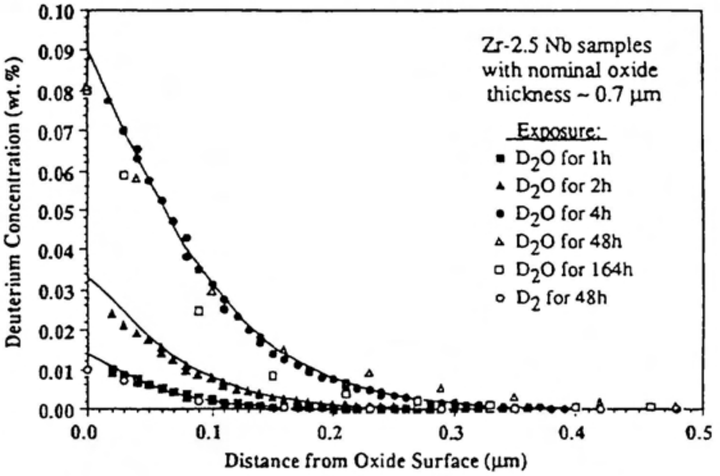


Fig. 6. DEUTERIUM PROFILES IN A THIN OXIDE FILM THAT DID NOT DEVELOP ACCORDING TO FICK'S LAW [31].

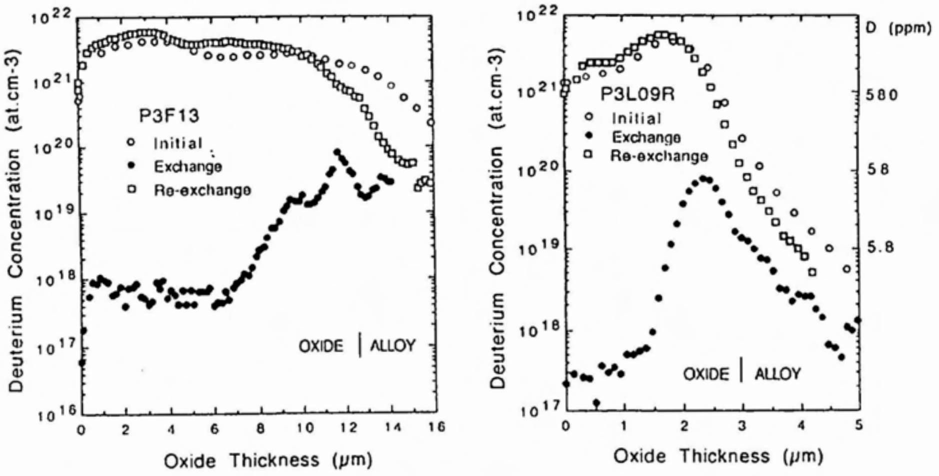


Fig. 7. RAPID EXCHANGE OF H / D (300°C, 7d.) IN OXIDE FILMS ON PRESSURE TUBES THAT TOOK 1.5 YEAR (P3L09R) AND 10.4 y. (P3F13) TO GROW [32].

Fig. 8. CATHODIC SITES IN A 1.2μm OXIDE FORMED ON ZIRCALOY (a) AND DETAILS OF ARROWED SITE (b) BEFORE AND (c) AFTER Cu DISSOLUTION [34].

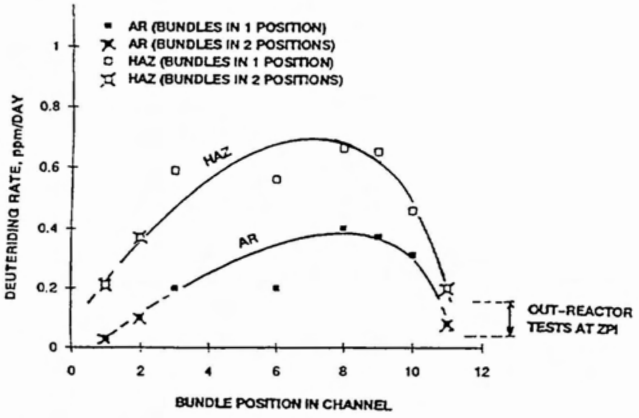


Fig. 9. PREFERENTIAL HYDROGEN UPTAKE AT BRAZE HEAT-AFFECTED ZONES IN CANDU FUEL CLADDING [36].

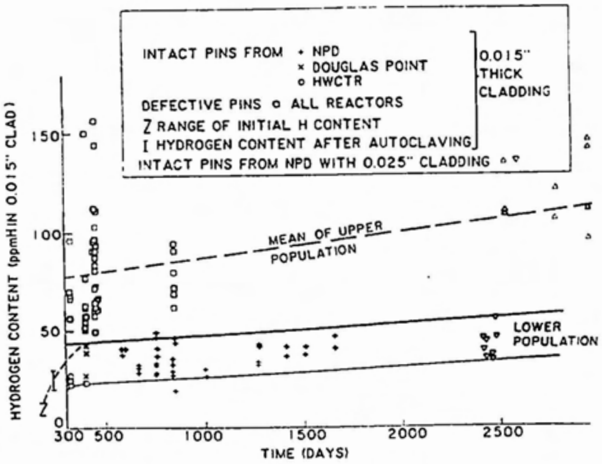


Fig. 10. INCREASING HYDROGEN CONTENTS WITH TIME IN IRRADIATED D_2O [14].