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# Hydrogen ingress through EDM surfaces of Zr–2.5Nb pressure-tube material

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**Abstract**

Mechanical test specimens can be easily manufactured from Zr–2.5Nb pressure tubes using electrical discharge machining (EDM). We discovered that zirconium specimens manufactured by EDM using a brass electrode and paraffin oil as dielectric fluid picked up a large amount of hydrogen during irradiation in 250°C water. This paper describes the investigation of the hydrogen ingress route. Our results showed that EDM in paraffin oil using a brass electrode modified the surface such that it became permeable for hydrogen when the specimen was later exposed to water. Out-reactor experiments on unirradiated material showed that irradiation was not necessary for the hydrogen ingress but might influence the ingress rate. The hydrogen ingress rate for out-reactor tests was estimated to be in the range $2 \times 10^{17}$–$8 \times 10^{17}$ atoms/m$^2$ s at 300°C water. There was a large scatter in the results indicating that the surface conditions, water chemistry and radiolysis of the water may influence the hydrogen ingress rate. 

## 1. Introduction

Electrical discharge machining (EDM) is a simple and cost effective technique for cutting and shaping irradiated material. Our first application was for axial slits in irradiated pressure tubes to represent cracks in fracture tests [1]. Using shaped electrodes, tensile specimens were successfully machined from highly irradiated fuel sheathing using EDM [2] and subsequently a similar technique was used to make curved compact toughness (CC(T)) specimens [3] to measure fracture toughness of irradiated pressure tubes. No artifact in the results from these specimens could be attributed to EDM. However, specimens machined this way and irradiated in a light-water loop in NRU at 250°C were embrittled by a large amount of hydrogen. These specimens were used in a study on how to recover the good fracture toughness of irradiated pressure tube by heat treatment [4].

Most of the fracture toughness reduction of the Zr–2.5Nb pressure-tube material due to irradiation can be recovered by annealing at 500–550°C for 10–20 min [4]. To study how quickly the toughness would return to the value prior to annealing, some of the heat-treated materials were cut by the EDM process into CC(T) and transverse tensile specimens [5] and then re-irradiated in the NRU reactor at 250°C for 78, 143 and 221 days at a fast flux of about $1.4 \times 10^{17}$ n/(m$^2$ s), $E > 1$ MeV. The hydrogen embrittlement was discovered during mechanical testing of these specimens after re-irradiation. This paper describes the investigation of the hydrogen ingress route.

## 2. Experiments

### 2.1. The EDM process

The EDM process used for cutting the irradiated CC(T) and transverse tensile specimens is as follows. The brass electrode was made with 1.5-mm thick plates with the following composition (wt%): Cu 64, Zn 33, Pb

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2.2, Ni 0.04, Fe 0.02 and S 0.02. The brass plates were arranged into the shape of the specimen and the electrode was used like a cookie-cutter to cut through the thickness of the pressure tube. Fig. 1 shows the electrode used for CC(T) specimens. The dielectric was Texaco EDM fluid, a paraffinic oil, but the exact composition was not known. Negative polarity was used, i.e., the electrode was negative with respect to the grounded specimen. A current of about 7 A was used during machining. The voltage was about 100 V open circuit, and it dropped to between 30 and 60 V during cutting.

After EDM, the specimens were cleaned with xylene, then separated from the blanks using a slow-speed saw with water as coolant.

### 2.2. Irradiated materials

Curved compact fracture toughness CC(T) and transverse tensile specimens used to study the recovery of fracture toughness were cut from a CANDU (**CA**-**N**ada **D**euterium **U**ranium; registered trademark of AECL) fuel channel F13 from the Pickering Unit 3 reactor (P3F13). The section of tube had been in service at a fast neutron flux of $2.1 \times 10^{17}$ n/(m$^2$ s) ($E > 1$ MeV) to a fluence of $7 \times 10^{25}$ n/m$^2$. Rings were cut from this pressure tube and heat treated in vacuum either at 500°C for 20 min or 550°C for 10 min.

The rings were cut into $50 \times 50$-mm$^2$ blanks using a low-speed saw without coolant. The blanks for transverse tensile specimens were mechanically milled to a thickness of 2 mm before they were cut by EDM. For the CC(T) specimens, the oxide on the inside surface of the pressure tube was removed by abrasion so that there was good electrical contact for the EDM process. Fig. 1 shows the CC(T) specimen before separation from the blank. After the specimens were separated from the blanks using a low-speed saw with water as coolant, they were loaded into a light-water loop in NRU. After re-irradiation at 250°C, the CC(T) specimens were pickled using a solution of 45% nitric acid, 10% HF and 45% water for about 30–90 s and then washed in acetone. The transverse tensile specimens were not pickled.

Some irradiated materials from P3F13 were cut by EDM and exposed to heavy water in an autoclave at 300°C to determine if irradiation during exposure to water was required for the heavy hydrogen ingress.

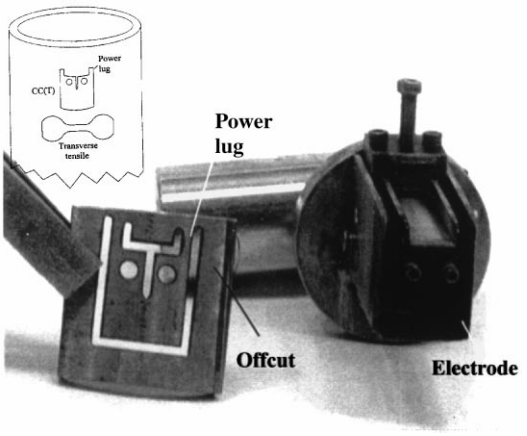


Fig. 1. The brass electrode used for the EDM process and the resulting CC(T) specimen before separation from the blank. Note that the power lugs were cut using EDM on two *T* planes and the offcut was EDM cut on only one surface.

### 2.3. Unirradiated materials

To test whether microstructural changes due to irradiation of the Zr–2.5Nb pressure-tube material was necessary for the hydrogen ingress, some strip specimens were made using unirradiated material. They were machined by EDM as described in Section 2.1 on the three principle planes as shown in Fig. 2. To maximize the hydrogen ingress, two parallel surfaces were cut by EDM. The specimens with the plane normal in the radial (R), transverse (T) or longitudinal (L) directions are called the R, T, L specimens, respectively. The T and L specimens were about 1.6 mm thick and the R specimens were 4 mm thick between the parallel EDM surfaces. These specimens were exposed in the out-reactor RD-5 loop at 300°C for 20 days. RD-5 is a heavy-water loop, and thus the sources of the hydrogen isotopes that entered the specimens could be distinguished. The heavy water in the loop was supersaturated with deuterium at about 3–4 cm$^3$/l.

### 2.4. Hydrogen analyses

Hydrogen analysis was performed using a high temperature vacuum extraction technique. The specimen

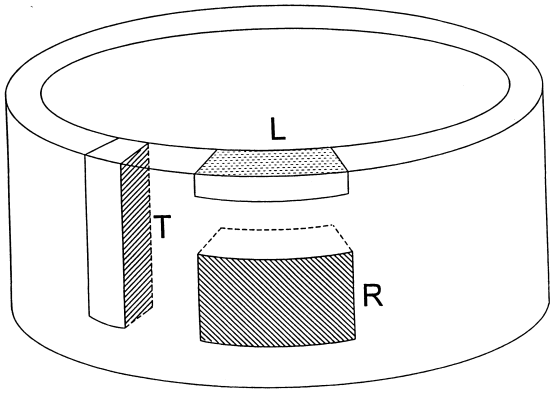


Fig. 2. Orientations of the EDM surfaces of unirradiated specimens.

was inductively heated to 1200°C to extract the gas. Hydrogen isotopes were collected through a hot palladium thimble. The H/D ratio was determined by mass spectrometer. The accuracy of the hydrogen concentration measurements was about ±10%.

## 3. Results

### 3.1. Metallurgical examinations

#### 3.1.1. Irradiated specimens

Three irradiated transverse tensile specimens that had different histories were examined metallographically:

(a) as machined by EDM,

(b) cut by EDM and heat treated (550°C, 10 min), and

(c) cut by EDM, heat treated (500°C, 20 min) and re-irradiated to $1.8 \times 10^{24}$ n/m$^2$ (143 days at 250°C in water).

The metallography of these specimens are shown in Figs. 3–5. There was a hydride concentration just under the heat-affected zone of the EDM surface of specimen (a). The heat treatment of 550°C for 10 min had dispersed the hydrides and no hydride concentration was observed under the EDM surface of specimen (b) (Fig. 4). After re-irradiation in water at 250°C, very heavy concentrations of hydrides were found in specimen (c) (Fig. 5).

A scanning electron microscopy examination of the EDM surface layer using WDX showed that there was a porous surface layer contaminated with Cu and Zn, elements found in the brass electrode (Fig. 6). It was inconclusive if C was present in this layer.

#### 3.1.2. Unirradiated specimens

Unirradiated specimens machined by EDM were exposed in the heavy-water RD-5 loop for 20 days at 300°C. Metallography of these specimens is shown in Fig. 7. The hydride concentration of these specimens had increased and the hydrides were distributed uniformly in these specimens, showing that neither radiation damage of the material nor irradiation during the water exposure was required for the high hydrogen ingress rate.

### 3.2. Hydrogen analysis

#### 3.2.1. Irradiated materials

The hydrogen concentration of the transverse tensile specimens increased linearly with time of exposure in NRU (Table 1). The hydrogen concentration of the CC(T) specimens were measured at the power lug (Fig. 1) and at the centre of the specimen (Table 2). In both cases, the hydrogen concentration increased linearly with exposure time in the reactor (Fig. 8), indicating that the hydrogen ingress rate is independent of the hydrogen concentration.

Specimens from irradiated materials were exposed in air and in an autoclave ($D_2O$), at 300°C for two months, to study the effect of the environment. The results, Table 3, show that only the specimens that were exposed to water picked up hydrogen isotopes. The small increase of hydrogen (H) concentration after exposure to $D_2O$ could be due to the light-water impurity in the heavy

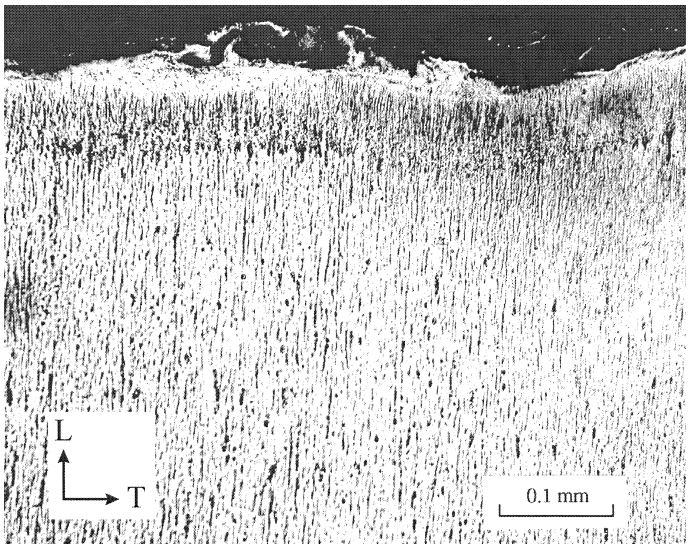


Fig. 3. Cross section of an irradiated specimen after EDM. Note the hydrides located about 0.1 mm from the EDM surface.

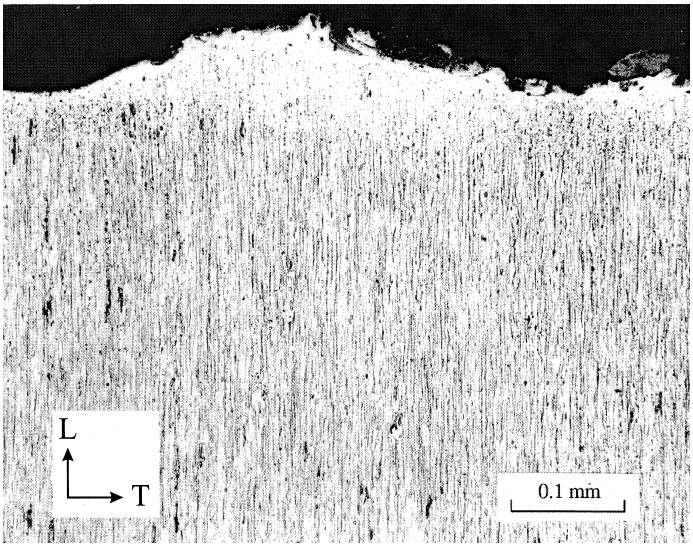


Fig. 4. Optical micrograph of a EDM specimen after heat treatment (550°C, 10 min). Note that there is no hydride concentration under the heat-affected zone.

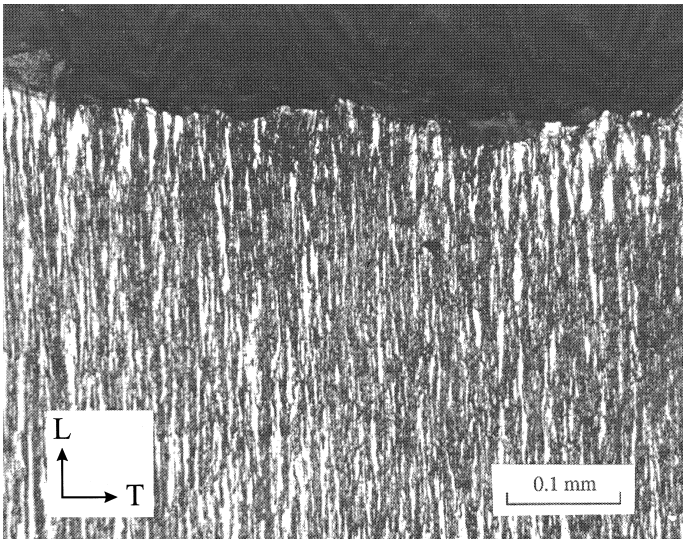


Fig. 5. Heavy concentration of hydrides in a specimen that was re-irradiated in NRU to $1.8 \times 10^{24}$ n/m$^2$ (143 days at 250°C).

water. Tables 2 and 3 also contain the results from an 'offcut'. Offcut was the leftover material after the CC(T) specimen was removed (Fig. 1). In this case, the offcut specimen was cut by EDM on only one side of the $T$ planes. The offcut received no heat treatment and no re-irradiation in NRU, but it also picked up deuterium when exposed to heavy water in the autoclave, indicating that the heat-treatment process was not responsible for the high hydrogen ingress rate.

*3.2.2. Unirradiated materials*

Small unirradiated strips after EDM were exposed in the RD-5 heavy-water loop. Hydrogen concentrations before the exposure were about 0.1–0.15 at.%. These were close to the normal hydrogen concentrations of as received material, which was $0.1 \pm 0.06$ ($2\sigma$) at.% [6], indicating that hydrogen introduced into the specimens during EDM was small, probably $<0.05$ at.%.

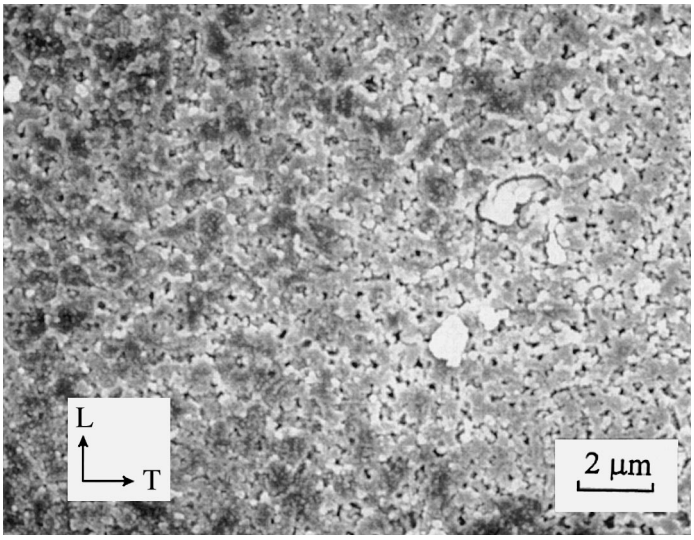


Fig. 6. Porous layer of the EDM surface.

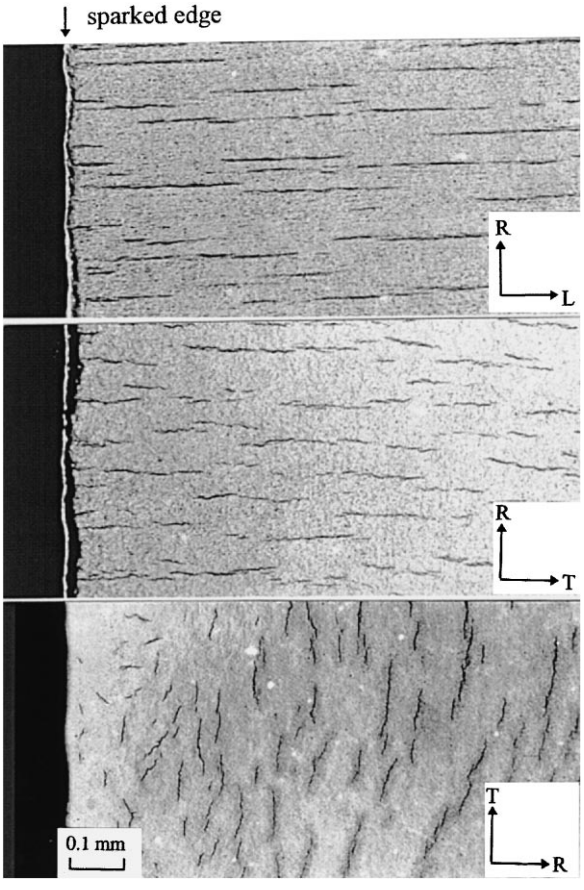


Fig. 7. Hydride morphology for specimens exposed in the RD-5 loop for 20 days.

Table 1
Hydrogen analysis of transverse tensile specimens after irradiation in a light-water loop in NRU at 250°C

| Specimen I.D. | Heat treatment (°C/ min) | Re-irradiation fluence ($n/m^2$) | Re-irradiation time (d) | [H] (at.%) | [D] (at.%) | Total hydrogen isotopes (at.%) |
|---|---|---|---|---|---|---|
| A2.TT3 | None | 0 | 0 | 0.18 | 0.023 | 0.2 |
| A2.TT4 | 550/10 | 0 | 0 | 0.14 | 0.014 | 0.15 |
| A4.TT2 | 550/10 | $9.0 \times 10^{23}$ | 78 | 8.4 | 0.054 | 8.5 |
| A4.TT5 | 500/20 | $1.8 \times 10^{24}$ | 143 | 20 | 0.086 | 20 |
| A4.TT1 | 500/20 | $2.8 \times 10^{24}$ | 221 | 22 | 0.095 | 22 |

Table 2
Hydrogen analysis for CC(T) specimens after irradiation in NRU at 250°C

| Specimen I.D. | Heat treatment (°C/min) | Re-irradiation fluence ($n/m^2$) | Re-irradiation time (d) | Power lug | | Specimen centre | |
|---|---|---|---|---|---|---|---|
| | | | | [H] (at.%) | [D] (at.%) | [H] (at.%) | [D] (at.%) |
| T10-1-OC [a] | 550/10 | 0 | 0 | 0.05 | 0.01 | 0.05 | 0.01 |
| A1.C8 | 550/10 | $9.0 \times 10^{23}$ | 78 | 6.4 | 0.05 | 1.9 | 0.02 |
| | 550/10 | $9.0 \times 10^{23}$ | 78 | 6.4 | 0.05 | 1.8 | 0.02 |
| A5.C1 | 500/20 | $1.8 \times 10^{24}$ | 143 | 12.0 | 0.05 | 3.5 | 0.02 |
| A7.C8 | 550/10 | $1.8 \times 10^{24}$ | 143 | 13.0 | 0.05 | 3.3 | 0.02 |
| A1.C6 | 550/10 | $2.8 \times 10^{24}$ | 221 | 20 | 0.06 | 5.9 | 0.02 |
| | 550/10 | $2.8 \times 10^{24}$ | 221 | 20 | 0.06 | 4.6 | 0.04 |

[a] Offcut of specimen T10-1. This material was heat treated and then cut by EDM but not re-irradiated in NRU.

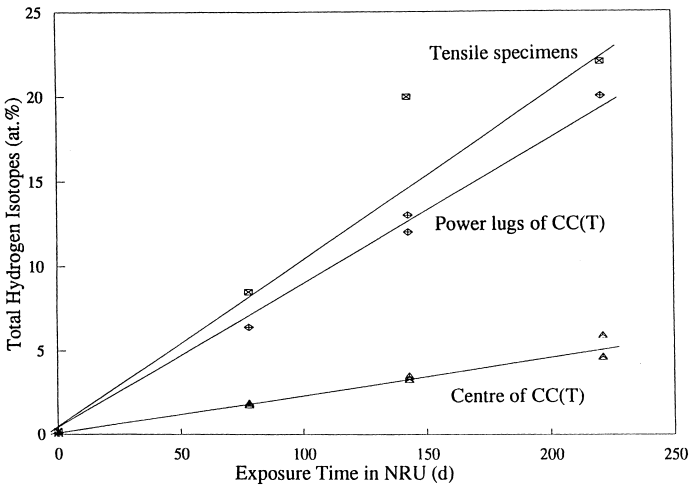


Fig. 8. Hydrogen concentration as a function of exposure time in NRU.

Table 3
Hydrogen isotope concentration of irradiated samples exposed in an air furnace and autoclave for two months at 300°C

| | Exposed in air | | | Exposed in $D_2O$ | | |
|---|---|---|---|---|---|---|
| | [H] (at.%) | [D] (at.%) | Total (at.%) | [H] (at.%) | [D] (at.%) | Total (at.%) |
| Power lug | 0.16 | 0.03 | 0.19 | 0.26 | 1.5 | 1.8 |
| Offcut | 0.09 | 0.02 | 0.11 | 0.28 | 2.4 | 2.7 |

Table 4
Hydrogen analysis for specimens exposed in the RD-5 loop at 300°C for 20 days the concentrations of total hydrogen isotopes (hydrogen plus deuterium) are given

| EDM plane | Thickness | Hydrogen isotope concentration (at.%) | | |
|---|---|---|---|---|
| normal | (mm) | Initial | After exposure | Ingress |
| *T* | 1.6 | 0.10 | 1.05 | 0.95 |
| *R* | 4.05 | 0.14 | 0.58 | 0.44 |
| *L* | 1.6 | 0.15 | 1.12 | 0.97 |

Following exposure for 20 days at 300°C in the RD-5 loop, the total hydrogen isotope concentrations had increased significantly, Table 4.

*3.2.3. Surfaces machined by other methods*

As part of the same study on recovery of properties, delayed hydride cracking (DHC) cantilever beam specimens were machined mechanically by Dr Sagat of AECL and were irradiated in NRU in 250°C water for 221 days at the same time as our transverse tensile and CC(T) specimens. The DHC specimens picked up less than 0.02 at.% hydrogen during the irradiation in 221 days [7]. Comparing with results in Table 2, this result indicates the mechanically machined surfaces were not as permeable to hydrogen as those cut by EDM. Mr Wallace of Ontario Hydro cut unirradiated CC(T) specimens using a traveling brass wire EDM process with water as coolant. These EDM surfaces were generally smoother than those cut by the 'cookie cutter' electrodes. These specimens did not have the high hydrogen ingress rate we experienced when they were irradiated in NRU [8].

*3.3. Estimate of hydrogen ingress rate*

Irradiated CC(T) and transverse tensile specimens had irregular shapes, unsuitable for hydrogen-ingress calculations. Therefore, the hydrogen ingress rate through the EDM surfaces was estimated using unirradiated specimens tested in the RD-5 loop. If we assume that hydrogen ingress is through the EDM surface only when it is exposed in water (the ingress through a mechanically machined surface is small compared with that through the EDM surface, see Section 3.2.3), and that the ingress is proportional to the surface area $A$ and independent of the hydrogen concentration in the specimen as indicated in Fig. 8, then for time $t$, the number of hydrogen atoms that enter the material is

$$n = \lambda A t, \tag{1}$$

where $\lambda$ is a rate constant in atoms/(m$^2$ s).

If the zirconium sample has volume $V$, the number of Zr atoms in that volume is given by

$$N = \rho V/(Za), \tag{2}$$

Table 5
Hydrogen ingress rate in RD-5 loop at 300°C for unirradiated material after EDM

| EDM plane normal | $\lambda$ (atoms/m$^2$ s) |
|---|---|
| *T* | $1.9 \pm 0.4 \times 10^{17}$ |
| *R* | $2.2 \pm 0.4 \times 10^{17}$ |
| *L* | $1.9 \pm 0.4 \times 10^{17}$ |

where $\rho$ is the density of Zr $= 6.49 \times 10^3$ kg/m$^3$, $Z$ is the atomic weight of Zr $= 91.22$, $a$ is the atomic mass unit $= 1.66 \times 10^{-27}$ kg.

The atomic percent of hydrogen, $C$, is thus

$$C = 100\lambda A t/(\lambda A t + \rho V/(Za)) \tag{3}$$

$$= 100/(1 + 4.28 \times 10^{28} T/(\lambda t)) \tag{4}$$

and

$$\lambda = 4.28 \times 10^{28} TC/((100 - C)t) \tag{5}$$

where $T$ is the thickness between the two parallel EDM surfaces.

The data in Table 4 were used to estimate $\lambda$ and the results are shown in Table 5. From these data, the hydrogen ingress rates for the three EDM surfaces are the same within experimental error (~20%).

## 4. Discussion

*4.1. Hydrogen ingress during the EDM process*

It has been established that water-rinsed, traveling-wire EDM process can induce hydrogen pick up in Fe-based alloys and CP Ti [9]. We also found that a higher hydride concentration under the EDM surface of our Zr–2.5Nb specimens (Fig. 3). It is not clear if this was the result of redistributing the hydrogen in the heat-affected zone or if the hydrogen has been pumped into the specimen. Our data showed that if the EDM process did induce hydrogen ingress, the amount would be less than 0.05 at.% (see Section 3.2.2) for the geometry of our specimens. However, unlike the alloys studied by Foecke et al. [9], the mechanical properties of zirconium alloys would not be affected by this relatively small increase in hydrogen concentration [10,11] and we did not investi-

gate this in detail. In the following discussions, we shall concentrate on the heavy hydrogen isotopes ingress when the EDM specimens were exposed to water. As shown in Table 1, the total hydrogen concentration increased from ~0.2 to 22 at.% in 221 days.

### 4.2. Requirements for a high hydrogen ingress rate

The following summarizes the observations reported above:

All our specimens that have been in paraffin oil using brass electrode and then exposed in water had high hydrogen ingress rate.

When the EDM materials were exposed to air, they did not pick up hydrogen.

Irradiation is not necessary for the hydrogen ingress, neither as a means to change the microstructure of the material nor during the exposure to high temperature water. Unirradiated specimens exposed in the RD-5 loop and irradiated specimens exposed in an autoclave both picked up hydrogen. The EDM process alone contributes very little hydrogen compared with subsequent hydrogen ingress when exposed to 300°C water.

Thus, based on the above observations, the hydrogen ingress into specimens occurred through the EDM surfaces when they were exposed to water. Irradiation was not necessary for the high ingress rate to occur.

### 4.3. Hydrogen ingress rate of irradiated CC(T) and transverse tensile specimens

In Section 3.3, the hydrogen ingress rate of unirradiated material exposed in the out-reactor RD-5 loop at 300°C was calculated. Using the ingress rate of $2.0 \times 10^{17}$ atoms/m$^2$ s to calculate the total hydrogen isotope ingress of the power lug of a CC(T) exposed to $D_2O$ in an autoclave for two months (Table 3), the result was 1.7 at.%, in excellent agreement with the experimental result of 1.8 at.% (Table 3). The total hydrogen concentration of the offcut specimen after being exposed in $D_2O$ (Table 3), however, was higher than expected. The offcut was EDM cut on only one surface, and the thickness was about half that of the power lug. If the hydrogen only entered the material through the EDM surface, we would expect similar hydrogen concentration to that of the power lug. However, the offcut had a higher concentration of deuterium, although the hydrogen concentration was about the same.

The hydrogen concentration in the power lugs of the CC(T) specimens exposed in NRU were also calculated for exposure time of 78, 143 and 221 days. Assuming a width of 3 mm, the predicted hydrogen concentrations were 2.1, 3.7 and 5.7 at.%, respectively. The measured values were about 3–4 times higher than this despite the lower exposure temperature in NRU. (Note that the ingress rate used in the calculation was derived from tests conducted at 300°C in the RD-5 loop, but the specimens were exposed at 250°C in NRU.) It is possible that the water chemistry of the NRU coolant and radiolysis of the water during irradiation may play an important role in the hydrogen ingress rate.

The EDM surface-to-volume ratio of the transverse tensile specimen is 25% lower than that of the power lugs of the CC(T) specimens, but the hydrogen concentration was about 15% higher in the transverse tensile specimens. The reason for the discrepancy is not known.

### 4.4. The EDM surface

Electrical discharge machining produces a recast surface layer. The electrode and the dielectric fluid will contaminate the surface. Paraffinic oils contain carbon, and carbon contamination during cutting does occur when these oils are used as dielectric fluids [12]. Because of the low solubility of carbon in zirconium, zirconium carbides may be present at the recast layer. Second-phase particles are considered to play a role in hydrogen ingress mechanism [13,14]. There is little information on the role of zirconium carbides on hydrogen ingress in zirconium alloys because carbide concentrations are generally low. In this situation, there might be more carbides on the recast layer acting as cathodic sites for hydrogen generation and entry. The fact that Mr Wallace's specimens, cut by traveling-wire EDM process using water as dielectric fluid, did not pick up hydrogen seems to support this argument, as these specimens would not have the carbides on the surface layer. However, the positive identification of the carbides in the EDM layer requires further work. Using SEM / EDX, we were unable to identify the carbides in this layer. The zirconium peak and the carbon peak are close to each other which makes the carbide detection difficult. It is also possible that the carbide particles were too small ($<0.7$ μm) to be imaged using SEM / EDX. At this point we can only put forth this idea as a possible mechanism.

The electrical conditions of machining may affect the roughness and porosity of the EDM surface. More importantly from the hydrogen ingress point of view, it would also influence the decomposition of paraffin oil and carbon deposit, which in turn affects the hydrogen ingress rate. This could be the reason for the difference in hydrogen ingress rates between the CC(T) specimens and the transverse tensile specimens.

### 4.5. Implications

In studies of the combined effects of hydrogen and irradiation on the mechanical properties of zirconium alloys, it is required to add hydrogen to the irradiated specimen at low temperatures to prevent loss of irradi-

ation damage. There are several techniques being developed at AECL and ingress through the EDM surface can potentially be one of them. Before method endorsement, the effects on the ingress rate due surface conditions, water chemistry, and radiolysis of water during irradiation need more studies. If additional hydrogen is not wanted, EDM using brass-electrode/paraffin oil should be avoided, unless the material would not be exposed to high-temperature water later. In such cases, water rinsed traveling-wire EDM process and mechanical machining can be used.

## 5. Conclusions

Mechanical testing specimens of Zr–2.5Nb had been machined by EDM using "cookie-cutter"-type, brass electrode in paraffin oil. A large amount of hydrogen was found in these specimens after irradiation in an NRU light-water loop at 250°C. An experimental program was initiated to determine the hydrogen ingress route and the following conclusions can be drawn from the experimental data reported here.

Hydrogen can enter through a EDM surface of Zr–2.5Nb material when the specimen is exposed in 250°C water.

Ingress rate seems to be independent of the plane of the EDM surface. The ingress rate is about $2 \times 10^{17}$ atoms/($m^2$ s), measured at 300°C in an out-reactor heavy water loop.

Neither modification of the material due to irradiation nor irradiation during exposure to water are necessary for high hydrogen ingress rate.

Hydrogen ingress rate seemed to be higher when irradiated in NRU. Possibly radiolysis of the water and/or the water chemistry has an influence on the ingress rate.

The effects of surface conditions, water chemistry, and radiolysis of water on the ingress rate need more studies.

To avoid the high hydrogen ingress into Zr–2.5Nb specimens when they have to be exposed to water, milling or traveling brass wire EDM with water as dielectric fluid can be used.

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