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## Comparison of delayed hydride cracking behavior of two zirconium alloys

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### ABSTRACT

Delayed hydride cracking (DHC) is an important failure mechanism that may occur in Zr alloys during service in water-cooled reactors. Two conditions must be attained to initiate DHC from a crack: the stress intensity factor must be higher than a threshold value called  $K_{IH}$  and, hydrogen concentration must exceed a critical value. Currently the pressure tubes for CANDU reactor are fabricated from Zr–2.5Nb. In this paper the critical hydrogen concentration for DHC and the crack velocity of a developmental pressure tube, Excel, was evaluated and compared with that of Zr–2.5Nb. The DHC velocity values measured in Excel were higher than usually reported in Zr–2.5Nb. Due to the higher hydrogen solubility limits in Excel, its critical hydrogen concentration for DHC initiation is 10–50 wppm over that of Zr–2.5Nb in the range of 150–300 °C.

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### 1. Introduction

Zirconium alloys are used as the core structural materials in water-cooled reactors because of their low neutron absorption cross-section and good corrosion resistance. Nevertheless, some of the hydrogen or deuterium produced by the corrosion reaction is absorbed by the materials which is added to the initial concentration from the manufacturing process, may lead to component failure. Delayed hydride cracking (DHC) is one of the possible and important cracking mechanisms that may occur during service. It involves stress-induced hydrogen diffusion, precipitation and growth of hydrides and subsequent fracture, promoting crack propagation in steps [1–3].

For DHC occurrence it requires the presence of: internal or external tensile stresses, a stress concentrator and sufficient hydrogen concentration to form brittle hydrides at the stress concentrator. Two conditions must be attained to initiate DHC from a crack: the stress intensity factor must be higher than a threshold value called  $K_{IH}$ , for cracking the hydrides [4,5] and the hydrogen concentration must exceed a critical value for hydride precipitation which depends on temperature [6–8]. The pressure tubes in current CANDU reactors are fabricated from the alloy Zr–2.5Nb and this material is still considered the most acceptable for future CANDU nuclear power plants. Among other Zr alloys, Excel (Zr–3.4 wt.% Sn, 0.8 wt.% Mo, 0.8 wt.% Nb) has been studied which indicated

that it has similar  $K_{IH}$  values but higher DHC velocity than Zr–2.5Nb [9]. The advantages of Excel compared to Zr–2.5Nb are [10,11]:

- it has higher tensile strength under similar thermo-mechanical conditions (equivalent metallurgical condition),
- measured in-reactor creep rates of 5% cold-worked Excel tubes are about half those of 30% cold worked Zr–2.5Nb pressure tubes. Creep rates of annealed Excel tubes are less than a third that of the Zr–2.5Nb,
- annealed Excel has similar strength than cold worked Zr–2.5Nb but the dislocation density is lower. Assuming that the irradiation growth depends on the dislocation density and texture, for the same design strength annealed Excel would have a better in reactor performance than cold worked Zr–2.5Nb,
- for advanced reactors which have pressure tubes operating at higher temperatures, the higher tensile strength and better creep resistance are beneficial,
- hydrides are less susceptible to stress re-orientation,
- in addition, the corrosion rate of Excel is about twice that of cold worked Zr–2.5Nb but the hydrogen pickup is about half. Consequently the hydrogen absorption of both alloys is similar [10,11].

In previous work [12] the critical concentration of hydrogen for DHC was measured at different temperatures in two Zr–2.5Nb pressure tubes, a Canadian tube (CANDU) and a Russian tube (RBMK). The experimental results agreed with the theoretical

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**Table 1**

Composition of Excel Tube. F, B is the front end and back end of the tube respectively which comes out of the extrusion press first.

Tube number	Sn (wt.%)	Mo (wt.%)	Nb (wt.%)	O (wppm)	H (wppm)
254 F	3.38	0.78	0.82	1118	54
254 B	3.47	0.81	0.80	1115	34

**Table 2**

Mechanical properties of Excel tube (provided by AECL).

Test temperature (°C)	Test direction	0.2 Yield strength (MPa)	UTS (MPa)	Total elongation (%)
25	Longitudinal	736	845	12
25	Transverse	930	965	9
300	Longitudinal	525	580	12
300	Transverse	620	645	13

**Table 3**

Yield stress values of Zr–2.5Nb. Test direction: transverse [1].

Material	Test temperature (°C)	0.2% Yield stress (MPa)
CANDU	20	803
	250	567
RBMK	20	659
	250	494

prediction by Shi et al. [7]. The theoretical prediction depends on the yield stress and hydrogen terminal solid solubility. Based on the solubility limits of Excel [13], which are higher than those of Zr–2.5Nb, the authors of this paper predicted that this material had higher critical concentration. The crack propagation velocity and critical hydrogen concentration for DHC were measured in an Excel tube and the values compared with those of Zr–2.5Nb.

## 2. Material

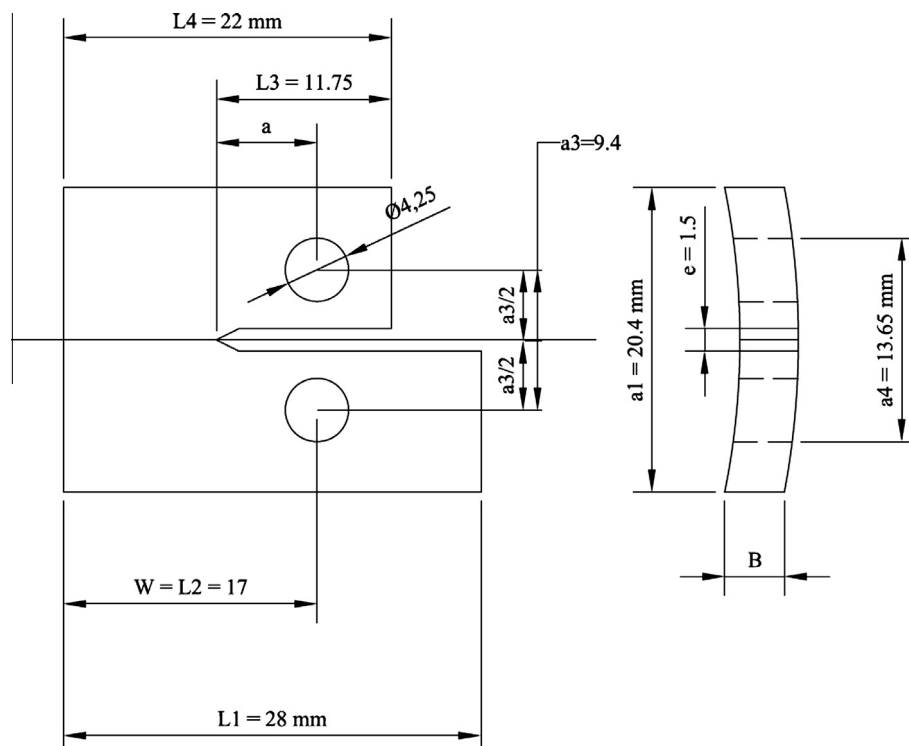
The composition and mechanical properties of the Excel tube employed in this work are detailed in Tables 1 and 2. The tube has been extruded at 860 °C (extrusion ratio 13.5:1) then cold-worked for about 3%. The results are compared with those of a Zr–2.5Nb pressure tube used in a previous paper [12]. Mechanical properties of Zr–2.5Nb are reported in Table 3.

## 3. Experimental

Samples, approximately  $30 \times 25 \times 4$  mm, were machined from the Excel tube and then hydrided using a gaseous charge method in a Sievert's device at temperatures around 350 °C in less than 30 min. The total hydrogen concentration (28–92 wppm) was calculated as the original concentration (28 wppm) plus those from the gaseous charged with an uncertainty of 10%. The samples were annealed at 383–385 °C for 22–27 h to attain a homogeneous hydrogen distribution. Curved Compact Toughness (CCT) specimens were machined from the annealed samples according to the guidelines of the IAEA Round Robin test program [1], see Fig. 1.

The specimens were tested in dead weight machines with a constant load applied perpendicular to the axial–radial plane of the original tube. Each specimen was subjected to a thermal cycle consisting of a heating ramp up to a peak temperature ( $T_{pk}$ ) higher than the dissolution temperature of hydrogen in Excel [13] and then cooling to different test temperatures ( $T_{st}$ ). Some  $T_{st}$  were chosen to be below the temperature ( $T_p$ ) corresponding to the terminal solid solubility for precipitation [13] to assure hydrides precipitated in the bulk during the test in order to obtain the maximum crack propagation rate at each temperature.

Once the test temperature was reached and stabilized, a constant load was applied. During testing the applied  $K_I$  was within the range 15–30  $\text{MPa}\sqrt{\text{m}}$ , where the DHC velocity is independent of  $K_I$  [1]. At the end of each DHC test, specimens were unloaded



**Fig. 1.** Schematic diagram of CCT specimen used for measuring DHC velocity along tube axial direction on axial–radial plane.

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