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In-reactor performance of pressure tubes in CANDU reactors

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A B S T R A C T

The pressure tubes in CANDU reactors have been operating for times up to about 25 years. The in-reactor performance of Zr–2.5Nb pressure tubes has been evaluated by sampling and periodic inspection. This paper describes the behaviour and discusses the factors controlling the behaviour of these components in currently operating CANDU reactors. The mechanical properties (such as ultimate tensile strength, UTS, and fracture toughness), and delayed-hydride-cracking properties (crack growth rate V_c, and threshold stress intensity factor, K_{IH}) change with irradiation; the former reach a limiting value at a fluence of $<1 \times 10^{25}$ n m^{-2}, while V_c and K_{IH} reach a steady-state condition after a fluence of about 3×10^{25} n m^{-2} and 3×10^{24} n m^{-2}, respectively. At saturation the UTS is raised by about 200 MPa, toughness is reduced to about 40% of its initial value, V_c increases by about a factor of ten while K_{IH} is only slightly reduced. The role of microstructure and trace elements in these behaviours is described. Pressure tubes exhibit elongation and diametral expansion. The deformation behaviour is a function of operating conditions and material properties that vary from tube-to-tube and as a function of axial location. Semi-empirical predictive models have been developed to describe the deformation response of average tubes as a function of operating conditions. For corrosion and, more importantly deuterium pickup, semi-empirical predictive models have also been developed to represent the behaviour of an average tube. The effect of material variability on corrosion behaviour is less well defined compared with other properties. Improvements in manufacturing have increased fracture resistance by minimising trace elements, especially H and Cl, and reduced variability by tightening controls on forming parameters, especially hot-working temperatures.

1. Introduction

The pressure tubes used in a CANDU[1] reactor are made from Zr–2.5Nb. The tubes are fabricated by hot-working ingots in the (α + β)-phase to logs. These logs are machined to produce hollow billets that are approximately 560 mm long × 195 mm diameter suitable for extrusion. After water-quenching from the β-phase the hollow billets are extruded at 815 °C and cold-worked 27% to produce tubes that are approximately 112 mm outside diameter and 104 mm inside diameter. The tubes are stress-relieved at 400 °C for 24 h prior to installation in a CANDU reactor. The microstructure consists of elongated grains of hexagonal-close-packed α-Zr, partially surrounded by a thin network of filaments of body-centred-cubic β-Zr. These β-Zr filaments are metastable and contain about 20% Nb. The stress-relief treatment results in partial decomposition of the β-Zr filaments with the formation of hexagonal-close-packed ω-phase particles that are low in Nb, surrounded by a Nb-enriched α-Zr matrix. During service the pressure tubes operate at temperatures between about 250 and 310 °C, and with variable coolant pressures up to 11 MPa corresponding to hoop stresses of up to 130 MPa. The maximum flux of fast neutrons ($E > 1$ MeV)[2] from the fuel is about 4×10^{17} n m^{-2} s^{-1}. The performance of the pressure tubes during operation is a function of the fabrication history of the tubes and the operating conditions, both of which vary.

As part of an ongoing program to ensure safe and efficient reactor operation the properties of the Zr–2.5Nb pressure tubes have been monitored by a combination of in-reactor measurements, tests on tubes removed for surveillance purposes and irradiations in materials research reactors. This paper is directed at reviewing the most recent results of this monitoring program for periods of operation up to the design life of the reactor and also describes how manufacturing has been modified to improve some aspects of pressure tube performance. Performance targets described previously [1], are shown in Table 1. The pressure tube properties important for the performance of the reactor are: (i) fracture toughness; (ii) delayed-hydride-cracking (DHC); (iii) deformation; (iv) corrosion and hydrogen ingress. These properties are

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[1] CANada Deuterium Uranium, trademark of Atomic Energy of Canada Limited.

[2] All neutron fluxes and fluences are for $E > 1$ MeV for the remainder of this paper.

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dependent on the microstructure of the pressure tubes and thus are a function of the manufacturing process and operating conditions (radiation damage).

2. Fracture toughness

To ensure fitness for service, Zr–2.5Nb surveillance pressure tubes are periodically removed from the reactor to assess their mechanical properties. In-service, the properties change due to irradiation damage and due to deuterium pickup from the pressurized heavy water coolant. Most experimental data pertaining to the fracture toughness of irradiated pressure tube material have been obtained from tests of ex-service pressure tubes. The results have highlighted the important influence of the material's pre-existing toughness and the influence of the operating conditions on the fracture toughness of irradiated pressure tubes. The important variables that have been identified include the type of particles that are responsible for primary void nucleation, the influence of the deformation properties of the irradiated matrix and the influence of the stress state at the crack-tip.

Previously, it has been shown that pressure tubes manufactured from ingots of 100% recycled material (RM) have high toughness due to the reduced concentration of micro-segregated species such as Cl, P and C [2–4]. The parameter dJ/da is a measurement of the crack growth resistance at small crack extensions from curved compact specimens of irradiated surveillance pressure tubes [5]. As the Cl concentration increases there is a corresponding reduction in toughness but little further change above ≈3 wt ppm. For pressure tubes made from 100% recycled material the Cl concentration is <1 wt ppm; such material has high toughness. The strong correlation between toughness and Cl, Fig. 1, has led to changes in the manufacturing process and present specifications stipulate a Cl concentration <0.5 wt ppm [4].

In addition to the influence of the type and distribution of void nucleating particles, the fracture toughness of irradiated Zr–2.5Nb is also affected by the deformation characteristics of the matrix. The ability of the matrix to undergo slip, dislocation channelling and twinning, which are affected by the fluence and irradiation temperature, influences void nucleation, growth and coalescence. At low fluences ($<3 \times 10^{24}$ n m^{-2}) there is an initial sharp increase in the transverse ultimate tensile strength (UTS) of about 200 MPa [6–9]. Beyond this transient, the UTS increases gradually at a rate of approximately 3.2 MPa per increase in fluence of 1×10^{25} n m^{-2} [8]. These changes in the UTS are a result of the development of the irradiation-induced microstructure. The rapid evolution of the (a-type) dislocation structure at low fluences corresponds with the increase in strength [9]. In comparison, the (c-type) dislocation structure evolves at a slower rate over longer periods of time and may be responsible for the gradual increase in UTS observed at high fluences ($>1 \times 10^{25}$ n m^{-2}) [8,9].

The rate of change of fracture toughness by irradiation is also largest at low fluence, as shown in Fig. 2 [8]. The initial reduction is due to the increased susceptibility to void nucleation resulting from irradiation hardening [5,6]. However, unlike the transverse tensile properties there is little further change following this transient [7,8]. The initial reduction is evident for both low and high toughness pressure tube material. After irradiation, materials with high concentrations of Cl may maintain <20% of their initial low toughness whereas materials with low Cl maintain >40% of their initial high toughness. The consistency in the ranking of un-irradiated and irradiated material suggests that the fracture toughness is strongly influenced by the presence of pre-existing particles.

The toughness of irradiated Zr–2.5Nb pressure tube material is also influenced by the geometry of the test specimen [5,10]. Rising pressure burst tests should be used to establish the fitness for service of an irradiated pressure tube due to the potential influence of specimen geometry. The critical stress intensity factor (K_c) from rising pressure burst tests conducted from room temperature to 250 °C is shown in Fig. 3. The minimum K_c increases from room temperature to 150 °C and then appears to remain constant, which implies that the transition to upper shelf fracture behaviour is complete by ≈150 °C. At this temperature and above a lower bound curve (LBC) has been defined from the data of low toughness material. None of the burst test results fall below this LBC and, for the lowest value measured at 250 °C to date, K_c is 74 MPa $\sqrt{m}$. The scatter in the data reflects the influence of primary variables, including particles and the deformation characteristics of the matrix, and differences in the operating conditions, irradiation temperature and fluence. Despite the scatter, the high toughness of the 100% recycled material is clearly evident in the upper shelf regime ($T \geqslant 150$ °C), these values corresponding with critical-crack-lengths (CCLs) that are >60 mm.

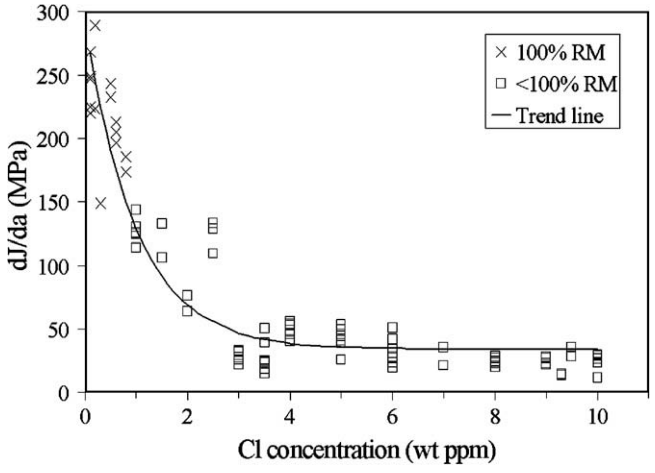

Fig. 1. Correlation of chlorine with the crack growth resistance (dJ/da) of curved compact specimens of irradiated pressure tube material tested at 250 °C. Results are for material with $P < 20$ wt ppm, fluence of $4–18 \times 10^{25}$ n m^2 and irradiation temperatures of 254–295 °C [5].

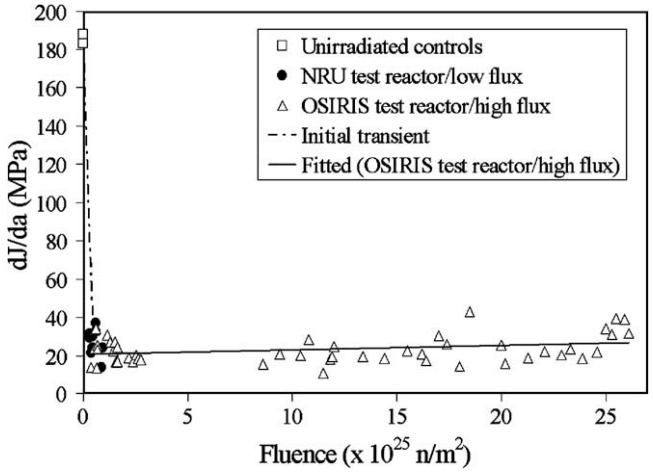

Fig. 2. Initial crack-growth-resistance (dJ/da) for axial cracking using curved compact specimens at 240 °C vs. fast neutron fluence. Data from pressure tube H737 (Cl = 4 wt ppm).

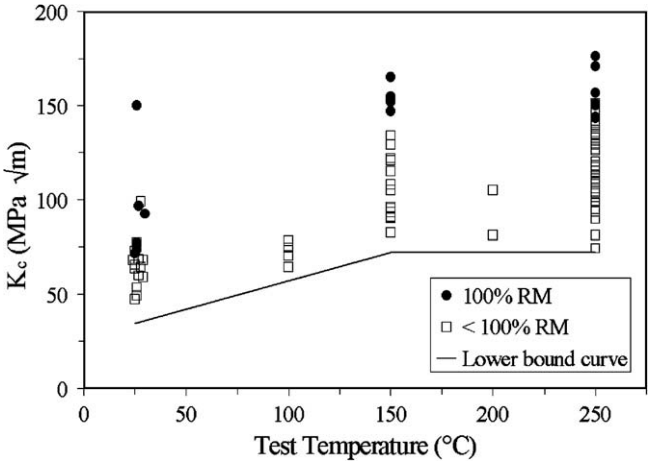

Fig. 3. Critical stress intensity factor, K_c, vs. test temperature for rising pressure burst test specimens from irradiated pressure tubing with an initial crack length between 45 and 65 mm.

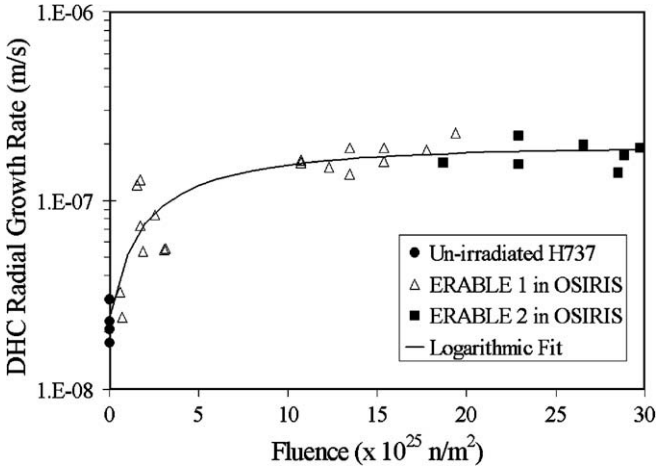

Fig. 5. Effect of fluence on DHC radial growth rates at 240 °C.

3. Delayed hydride cracking

Zirconium alloys are susceptible to a stable cracking process called delayed-hydride-cracking (DHC). DHC has two stages: (a) crack initiation that requires a minimum crack driving force (the threshold stress intensity factor, K_{IH}); and (b) stable crack growth that is weakly dependent on the applied K_I. The DHC crack growth rate is dependent on the test temperature and the state of the material [1,11]. The material state is a function of the fabrication process (controls the state of the β-phase, the α-grain size, network dislocation density and the texture) and the in-service operating conditions (controls the irradiation damage density and the state of the β-phase). For any given pressure tube the factors affecting DHC behaviour are irradiation conditions and test temperature.

Fig. 4 shows the effect of neutron fluence on K_{IH} for radial cracking using cantilever beam specimens from the CANDU reactor outlet rolled-joint region irradiated at about 290 °C and tested at temperatures between 140 and 270 °C. Previously it was shown that K_{IH} is weakly dependent on test temperature [11]. The results show that irradiation lowers the K_{IH} value by about 30% and the decrease appears to saturate at a fluence of less than 3×10^{24} n m^{-2}.

Fig. 5 shows the effects of irradiation and fluence on the radial crack growth rate at 240 °C [7,8]. The tests were performed on specimens containing nominally 100 ppm hydrogen at an applied K_I of 17 MPa √m. The data show that irradiation increases the DHC crack growth rate by a factor of about 10 and the effect saturates after a fluence between 5 and 10×10^{25} n m^{-2}.

The increase in DHC crack growth rate from irradiation is influenced by the state of the β-phase and the change in yield strength [7,8]. Irradiation reverses the decomposition of the β-phase and therefore enhances hydrogen diffusion relative to the as-fabricated state. Higher yield strengths lead to higher crack-tip stresses that increase the driving force for diffusion and affect the critical condition for hydride fracture.

The axial crack growth rate relationship with temperature on irradiated pressure tube material is shown in Fig. 6 [11] as an Arrhenius relationship because the main temperature dependence

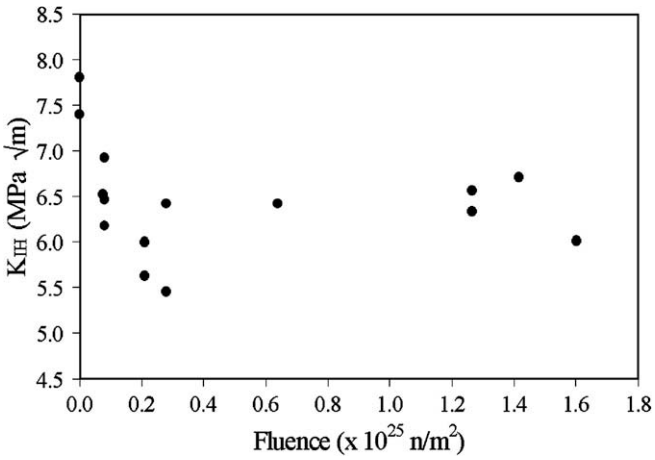

Fig. 4. Effect of fluence on K_{IH}. Material from CANDU reactor outlet rolled-joint region irradiated at about 290 °C and tested at temperatures between 140 and 270 °C.

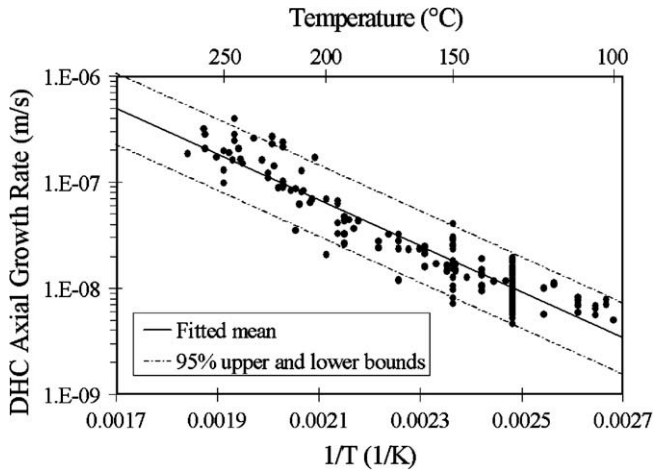

Fig. 6. The upper bound, mean and lower bound axial DHC crack growth rates for irradiated Zr–2.5Nb pressure tubes.

Table 1
In-service performance targets for current CANDU pressure tubes [1,15]

Property	Target
Deformation	<230 mm elongation, <5% diametral strain
Fracture toughness	CCL (250 °C, 10 MPa) > 60 mm
Delayed hydride cracking	K_{IH} > 4.5 MPa √m, V_C (250 °C) <2×10^{-7} m s^{-1}
Strength	Maintain specified value of >480 MPa
Corrosion	D-uptake per year: <1 ppm (inlet), <3 ppm (outlet)

of DHC growth rates resides in the temperature dependence of hydrogen diffusion and the solubility limit. The mean DHC velocity at 250 °C is about 2×10^{-7} m s^{-1}, the target rate specified in Table 1.

4. Deformation

Diametral expansion and elongation due to irradiation deformation may limit the useful life of pressure tubes in CANDU reactors and the maximum power for reactor operation. The deformation rates are a direct function of the microstructure (for example, crystallographic texture and grain size) and operating conditions (stress, temperature and neutron flux), but are also indirectly dependent on the operating conditions because of the modifying effects of the irradiation on the microstructure [12]. Therefore, the in-reactor deformation behaviour of pressure tubes is controlled both by the as-fabricated microstructure and the microstructure that evolves during irradiation.

For a given set of operating conditions there is considerable variability in deformation behaviour of pressure tubes that can be related to variations in the as-fabricated microstructure. Texture and grain thickness can affect both the anisotropy and magnitude of deformation strain. In general, pressure tubes that have a higher radial basal texture parameter, or Kearns factor (f_R), and have grains that are thinner in the radial direction tend to exhibit higher diametral strain and lower elongation rates compared with the average. These same microstructural variables affect the deformation behaviour along a given tube because of a gradual change in grain structure and crystallographic texture from one end of the tube to the other. The net effect is that pressure tubes tend to deform at a faster rate when the back-end of the tube (i.e., the end leaving the extrusion press last) is installed at the fuel-channel outlet. In current reactors the pressure tubes are installed with their back ends at the inlet end [12] to minimize the peak diametral strain. The primary cause of the difference in microstructure along a given tube is the temperature change during the extrusion process. This end-to-end variation itself varies from tube-to-tube, due to small variations in extrusion conditions from one extrusion run to the next, and also due to small variations in ingot chemistry and billet processing.

In some instances the deformation behaviour of pressure tubes cannot be explained simply from an examination of the forming variables. In these instances the deformation behaviour of the tubes is associated with the ingot from which they were manufactured. This 'ingot effect' is readily apparent from an examination of elongation data. Fig. 7 shows the elongation of groups of pressure tubes from different ingots in a CANDU reactor. Although the variation in the deformation behaviour can be related to differences in the final microstructure for these different groups of tubes, the relationship between ingot source and the microstructure is not always apparent. The source of the factors responsible for the ingot effect is the subject of current research. A key conclusion is that both ingot properties and extrusion properties affect deformation of pressure tubes. Gaining control of the ingot effect and maintaining control of the extrusion process should ensure a significant reduction in tube-to-tube variability in deformation.

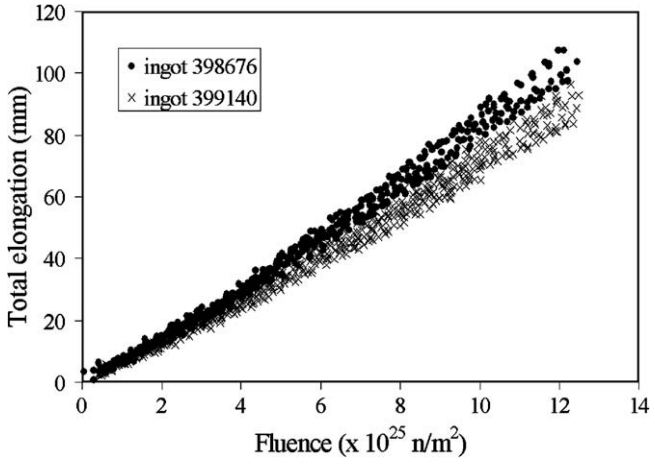

Fig. 7. Axial elongation of pressure tubes in a CANDU reactor showing the variability associated with tubes from different ingots.

5. Corrosion and hydrogen ingress

During operation, some deuterium generated by aqueous corrosion of the tube surface enters the metal. Additional deuterium also enters through the rolled-joint between the tube and the end fitting. The increase in deuterium during operation determines the susceptibility to hydride formation and this can limit the pressure tube lifetime [1]. For this reason it is important to have predictive models of deuterium ingress for fitness-for-service assessments for operating pressure tubes and for the development of new reactor designs. A predictive model for assessing the long-term oxidation of, and deuterium ingress into, the body of the pressure tubes has been developed from in-reactor tests of samples which had been pre-oxidized to obtain oxide thickness values representative of long-term behavior [13]. Deuterium ingress is modeled based on a fraction (2–10%) of the corrosion-freed deuterium entering the metal. The current version of the model contains relationships describing the oxidation rate as a function of oxide thickness, temperature, and fast neutron flux and fluence.

The model predicts a gradual increase in the deuterium pickup rate that is consistent with reactor data. In addition, the measured data are reasonably bounded by model predictions and fall below the target limits described in Table 1 [13].

6. Manufacturing of pressure tubes

In an effort to improve the properties of pressure tubes, several major changes in their fabrication have been explored and implemented. Zircaloy-2 was replaced by Zr–2.5Nb because of its greater strength and smaller pickup of hydrogen. The fabrication route of Zr–2.5Nb has had several versions. Pressure tubes made by routes based on quenching from the $(\alpha + \beta)$-phase close to their final dimensions were installed in Ignalina, KANUPP and Fugen whereas routes based on cold-working are used for pressure tubes in CANDU reactors of Canadian and Indian design. With cold-worked tubes, the focus of current improvements is to guard against fracture and to reduce microstructural variability to increase the predictability of behaviour.

Pressure tubes with consistent high fracture toughness are desirable. Early pressure tubes, while having acceptable values of fracture toughness, had high variability [14]. Laboratory and manufacturing studies [1,2,4] showed that the fracture toughness of as-fabricated tubes at 250 °C was increased by a factor of between 50% and 100% by:

- modifying the ingot charge make-up to lower the ingot carbon and phosphorus concentrations to $\leqslant$125 ppm (wt) and $\leqslant$12 ppm (wt), respectively, and
- using a four melt vacuum arc cycle or by using electrolytic powder to lower the chlorine concentration to <0.5 ppm (wt).

The efficacy of these changes is demonstrated in the distribution of the values of crack growth resistance in tubes made by double and quadruple melting of ingots made from Kroll sponge and

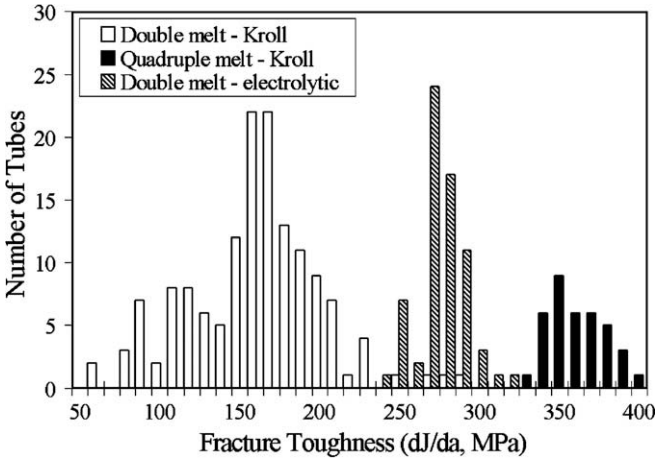

Fig. 8. Variation of crack growth resistance at 250 °C for three versions of Zr–2.5Nb.

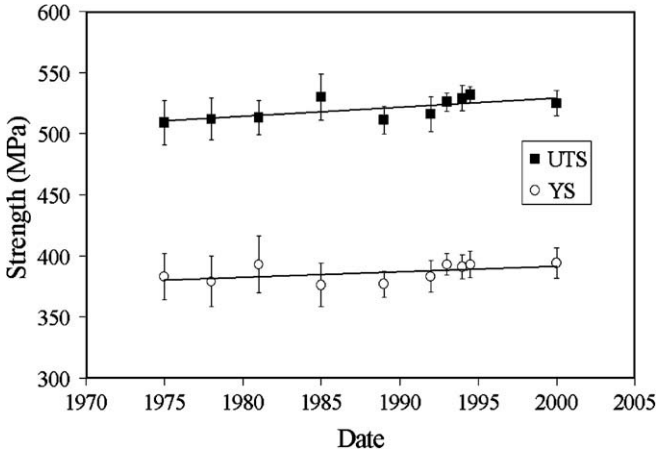

Fig. 9. Variation of yield strength (YS) and ultimate tensile strength (UTS) with year of production.

double melting ingots made from electrolytic powder, Fig. 8. This improved fracture resistance was gained without sacrificing strength [15]. Although the data presented in Fig. 8 are from unirradiated material, such tubes retain their high toughness after neutron irradiation. Modern tubes attain a minimum value of crack growth resistance of at least 250 MPa at 250 °C.

During reactor operation, pressure tubes may absorb about 3 ppm (wt) deuterium per year in some locations. If the initial hydrogen concentration is reduced, then the reduction leads to an increase in the time before hydrides are present at reactor operating temperatures. Several thousand samples were collected at all manufacturing stages. Analysis of these samples identified areas where both large and small reductions in hydrogen pickup could be achieved. Consequently, process equipment, such as gas heating furnaces, was replaced with electric furnaces, hold times at temperature were minimised and many other smaller, but no less significant, changes were made to the manufacturing process. As a result, new pressure tubes have an initial hydrogen concentration of $\leqslant$5 ppm (wt).

The extrusion process has a significant effect on the pressure tube properties, in particular, the mechanical strength and microstructural features important for deformation, such as basal pole texture and grain shape and size. Reducing variability for the extrusion feedstock, results in reduced variability for the extruded hollow and finished pressure tube. Manufacturing experience showed that a β-phase solution anneal followed by a water quench (known as β-quenching) reduced microstructural variability before extrusion. In the 1970's the manufacturing cycle was changed to include a β-quench for the solid logs after forging. In the early 1990's, the practice was modified to β-quench the pre-extrusion hollow billet. In the late 1990's, to reduce variability, the supplier installed an automated state-of-the-art solution annealing furnace and quench system that resulted in consistent:

- annealing temperatures,
- annealing times,
- transfer time from furnace to quench medium, and
- quench effectiveness.

Extrusion processing parameters have been established through plant trials so the resultant pressure tubes have low variation in their microstructure after cold-working [16]. Using the information from these trials, the supplier initiated process changes to reduce variability in the extrusion pre-heat conditions by installing:

- a state-of-the-art billet heating furnace,
- a billet transfer system with repeatable transfer times to ensure a consistent extrusion starting temperature, and
- an extrusion press that ensured that the extrusion conditions have small variability from tube-to-tube.

These changes in pressure tube fabrication have led to a more consistent product with smaller variation in microstructure and subsequent properties than attained previously. For example, although ultimate tensile strength has increased slightly in more recent production tubes, the variability has decreased Fig. 9. This knowledge provides a platform for further improvements based on feedback from reactor experience.

7. Conclusions

The primary pressure boundaries in CANDU reactors are the Zr–2.5Nb pressure tubes. Research projects combined with material surveillance tests have provided for safe and efficient reactor operation and improved power production capabilities. This knowledge has been further applied to evolutionary improvements in manufacturing and construction of new reactors.

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