

## Effect of cooling rate on delayed hydride cracking in CANDU Zr-2.5Nb pressure tube

Eun Young Na, Sung Woo Park, Young Suk Kim, Kyung Soo Im, Yong Moo Cheong, Kang Soo Kim  
Zirconium Team, Korea Atomic Energy Research Institute, Daejeon 305-353, Korea  
e-mail : yskim1@kaeri.re.kr

### 1. Introduction

Dutton and Puls suggested a delayed hydride cracking (DHC) model where a driving force for DHC is the stress gradient between the notch and the bulk region far away from it, which has been acknowledged as a rational hypothesis for now [1,2]. However, their DHC model has some defects: firstly, it cannot explain why DHC velocity (DHCV) becomes constant regardless of an applied stress intensity factor even though the stress gradient is affected by the applied stress intensity factor at the notch tip, secondly, it cannot explain why the DHCV has a strong dependency on the way how to approach the test temperature by a cooling-down or a heating-up even under the same stress gradient and thirdly, it can not predict any hydride size effect on the DHC velocity. Recently, Kim suggested another DHC model where a driving force for DHC is a difference in hydrogen concentration between the crack tip and the bulk region or  $\Delta C$  [3,4].

Therefore, the objective of this study is to verify the plausibility of Kim's DHC model. To this end, this study has investigated the effects of the hydride size and phase on the incubation time and the DHCV in a CANDU Zr-2.5 tube using the water-quenched, liquid nitrogen-quenched and furnace-cooled specimens.

### 2. Experiments

All the test specimens used were of curved compact tension (CCT) shape with 17mm in width taken from a CANDU Zr-2.5Nb tube (used in the Wolsong NPS). The CCT specimens were subjected to an electrolytic charging to form a thick hydride layer on their surfaces followed by a homogenization treatment at 307 °C for 26 h to theoretically contain 70 ppm H. Thus, all the hydrogen must be in solution at this homogenization temperature of 307 °C. Some of the solution-treated specimens were then quenched, either in water or liquid nitrogen, to produce fine hydride dispersions. Others were furnace cooled from the solution temperature, to produce a much coarser hydride distribution. A pre-fatigue crack of 1.7 mm was introduced using an Instron 8501 to obtain the ratio of the fatigue length and the CT length ( $=a_0/W$ ) equal to 0.5. The applied stress intensity factor was 12 MPa $\sqrt{m}$  at the beginning stage of the pre-fatigue crack and decreased to 10 MPa $\sqrt{m}$  after the fatigue crack grew to 1.7 mm. DHC tests were performed at various temperatures, ranging from 144 °C to 200 °C using a 6 channel creep machine. The test temperatures were reached by either a cool-down or a heating-up, as shown in Fig. 1. The CCT specimens were loaded at an initial stress intensity factor of about

15 MPa $\sqrt{m}$ , 30 minutes after the desired test temperature was reached. An initiation and growth of the crack were monitored by a DC potential drop method. The crack length was determined on the fractured surfaces by dividing the area of the DHC crack calculated by an image analyzer by the width. After DHC testing, the X-ray analyses were conducted on the fracture surfaces and the bulk region away from the crack tip to identify the hydride phase distribution.

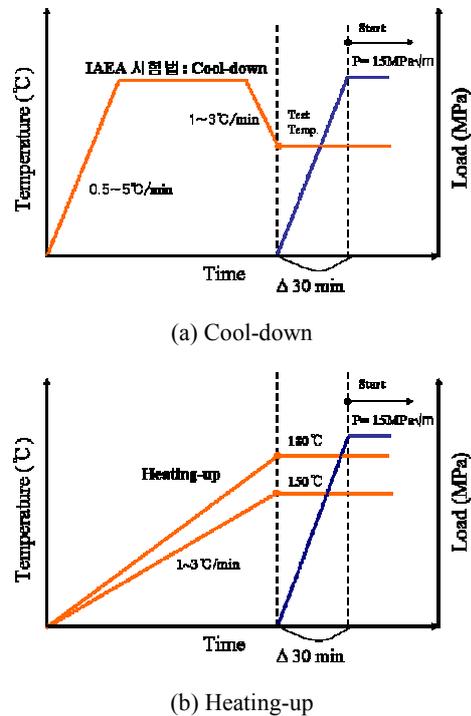
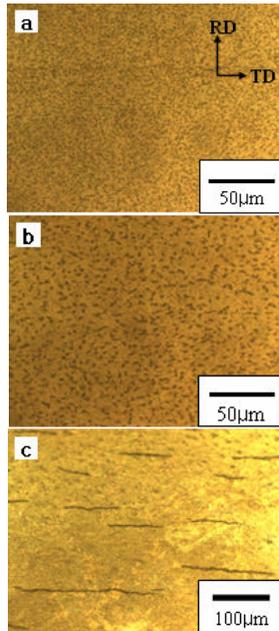


Fig. 1 Temperature cycles to which the CCT specimens were subjected for the DHC tests along with the loading schedule.

### 3. Results and discussion

#### 3.1 Hydride Morphology

The size and distribution of the hydrides in water-quenched or liquid nitrogen-quenched and furnace-cooled material are shown in Fig 2. As expected, a very fast cooling from the solution temperature produces a very finely dispersed hydride distribution (average hydride spacing  $\sim 1$  to 3  $\mu\text{m}$ ). Fine hydrides in the water-quenched specimens are mostly  $\gamma$ -hydrides. In contrast, the furnace cooling results in a coarse distribution of hydrides (average hydride spacing  $\sim 100\mu\text{m}$ ), which are expected to be predominantly the  $\delta$ -phase.



RD: radial direction, TD: tangential direction

Fig. 2 (a) The finest hydrides in the water-quenched, (b) Fine hydrides in the liquid nitrogen-quenched and (c) coarser hydrides in the furnace-cooled Zr-2.5Nb CCT specimens.

### 3.2 DHC Velocity with the cooling rate

Fig. 3 shows the DHCV of the Zr-2.5Nb tube with the cooling rate at four different temperatures upon an approach by a heating. The quenched specimens (water, or liquid nitrogen) had a much faster crack growth rate than the furnace-cooled specimen at various temperatures, ranging from 144 °C to 180 °C. Also, the incubation time for the furnace-cooled specimen was considerably long compared with the quenched specimen.

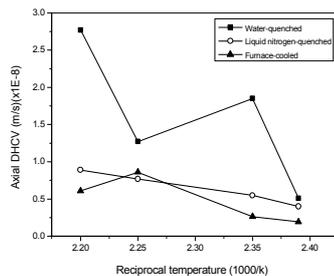


Fig. 3 DHC velocity of the CCT specimen at 144, 150, 170 °C and 180 °C by heating-up method as a function of cooling rate.

To confirm the dependency of DHC velocity on the cooling rate, DHC tests were again conducted to the CB specimens with the test temperatures reached by cool down from the peak temperature of 310 °C. As shown in Fig. 4, the water-quenched was confirmed to have a slightly higher DHC velocity both at temperatures of 180 and 200 °C even though the difference in DHC velocity is small. Kim proposed that DHC caused by a stress-induced transformation from the  $\gamma$ -hydrides and the  $\delta$ -hydrides. This can occur because most hydrides

formed in the water-quenched specimens and the furnace-cooled specimens are  $\gamma$ -hydrides and  $\delta$ -hydrides, respectively. The results as shown in Fig. 3 and Fig. 4 demonstrate that the water-quenched specimen with the  $\gamma$ -hydrides should have a much higher DHCV than the furnace-cooled specimen with the  $\delta$ -hydrides upon an approach to the test temperature. Another thing to note is that the water-quenched specimen would have a DHC crack growth even at temperatures higher than 180 °C where the furnace-cooled specimen fails to initiate a DHC crack growth. It is related to the formation of the  $\gamma$ -hydride in addition to the  $\delta$ -hydride that a DHC has occurred in both specimens at temperature, the stress-induced  $\gamma$ - $\delta$  transformation will occur only at the crack tip subjected to a tensile stress, causing a difference in the hydrogen concentration or  $\Delta C$  between the crack tip only with the  $\delta$ -hydrides and the bulk region with a mixture of the  $\delta$  and  $\gamma$ -hydrides.

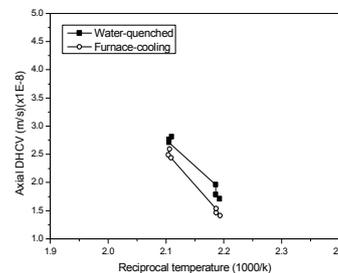


Fig. 4 DHC velocity of the CB specimen at 180 and 200 °C by cool-down method as a function of cooling rate.

## 4. Conclusion

The delayed hydride crack (DHC) tests were conducted on Zr-2.5Nb compact tension specimens with the test temperatures reached by a heating and a cooling. The maximum crack velocity is obtained of a water-quenched specimen. The size and distribution of the hydrides became larger for the furnace-cooled specimens with coarser hydrides than that for the water-quenched specimens. This different DHC behavior of the Zr-2.5Nb tube with the cooling rate after a homogenization treatment is due to a precipitation of the  $\gamma$ -hydrides only in the water-quenched Zr-2.5Nb tube. This experiment will provide supporting evidence that the terminal solid solubility of a dissolution (TSSD) of  $\gamma$ -hydrides is higher than that of  $\delta$ -hydrides and furthermore it will confirm the feasibility of Kim's DHC model.

## REFERENCES

- [1] R. Dutton, K. Nuttal, M.P. Puls and L.A. Simpson, *Metall. Trans.* 8A p. 1553, 1977.
- [2] M.P. Puls, L.A. Simpson and R. Dutton In: L.A. Simpson, Editors, *Fracture Problems and Solutions in the Energy Industry*, Pergamon Press, p. 13, 1982, Oxford.
- [3] Y.S. Kim, S.S. Park, S.S. Kim, Y.M. Cheong, K.S. Im, Proceedings of the Symposium on Zirconium, p. 58, 2002.
- [4] Y.S. Kim, in: TMS 2003 Annual Meeting & Exhibition Program and Guide: JOM 55, p. 383, 2003.