Set up Parameters of Hydride Orientation Test for Zirconium Alloy Tubes : A Review

SUN-DOO KIM, NAM-CHAN CHO, CHUL-HO PARK, YOUNG-MAN LEE
KOREA NUCLEAR FUEL CO.,LTD. / TECHNO SPECIAL ALLOY, chopark@knfc.co.kr

1. Introduction

Hydride orientation test for the zirconium alloy tube is frequently performed as one of the tests defined on the specification to confirm its texture preferences. In this study, the effect of set-up parameters of hydride test such as temperature, duration time and hydrogen gas pressure on hydride formation were investigated to see its effect on the formation of hydride and to find its optimal conditions for hydride test of zirconium alloy tubes.

A tube sample is plash pickled and then hydrided in a controlled furnace for a designated time period. (according to ASTM B353, Annex A2 or ASTM B811 Annex A2) It is cooled, mounted and prepared for a microscopic evaluation. A photo-micrograph of the area examined is read by the method outlined below to determine the Fn number of that area (outer, middle, inner)
The following is the Fn formula

$$Fn = \frac{\text{Total number of Fn}(>45°)}{\text{Totla number of Fn}}$$

This test's main parameters are time, temperature, pressure and mixed gas amount.
The purpose of this paper is to determine the suitable parameters and their optimal combination.

2. Test Results

2.1. Time and Temperature

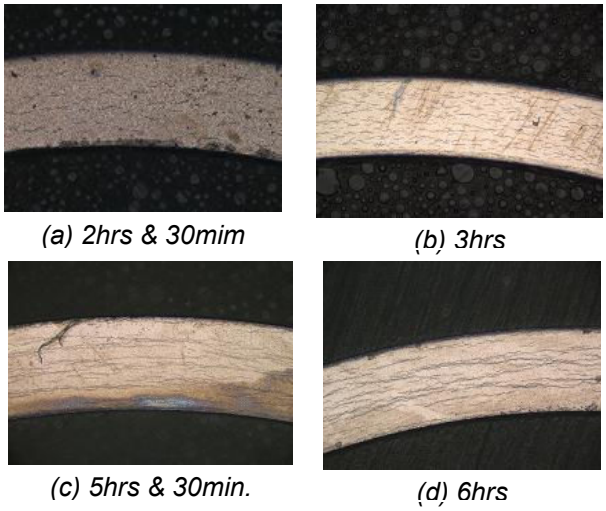
- Temperature Rising Time
  Many tests were performed for 2 ~ 3 hrs. After many tests, it is concluded that 2 hrs and 15 minutes was the most appropriate for Temperature Rising Time. Therefore, the temperature rising time was set at 2 hrs and 15 minutes. As the temperature tolerance in the furnace was +/- 1 ℃, it was chosen to hold the temperature in the furnace over 400℃ to meet the temperature holding condition.

- Temperature Holding Time
  Various holding time were tested in accordance with an increasing temperature holding time from 2 hrs up to 6 hrs. As a result of check and review of the test results(H_2 gas permeation trend), the optimal temperature holding time was fixed between 3hrs and 3.5hrs.(refer to table 1)

TABLE 1 - *Temperature holding time condition*

Holding time	H_2 gas permeation trend
2hrs ~ 2hrs 40min.	- Total 50 ~ 60 % level of permeation (≤ 100 lines)
3 hrs	- H_2 gas permeation amount : sufficent (> 100 lines)
3hrs ~ 6hrs	- H_2 gas permeation amount was sufficent. But It took so much time. Time can be decreased by Increasing pressure while pressure can be decreased by Increasing time.

The following is the H_2 gas permeation trend vs. temperature holding time (Refer to fig. 1 & 2)



(a) 2hrs & 30mim *(b) 3hrs*

(c) 5hrs & 30min. *(d) 6hrs*

Fig.1- *H_2 gas permeation trend depending on temperature holding time(100x)*

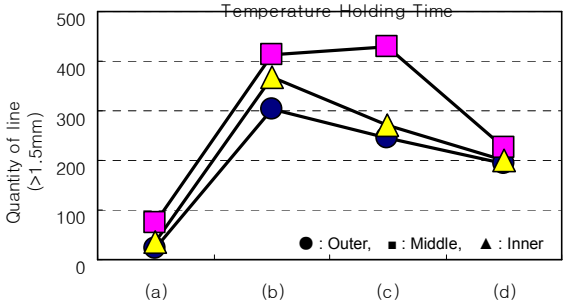


Fig 2. – *Quantity of the lines in fig. 1*

2.2. Pressure

- The mixed gas pressure
  The test was performed with the mixed gas pressure between 0.06 and 0.2Mpa. It was found that the pressure between 0.16 and 0.2Mpa was the most appropriate range. The 0.18Mpa

(1.8Bar) was the most optimal condition.[Refer to Fig.1(b)]

- Burette and Mass-cylinder pressure

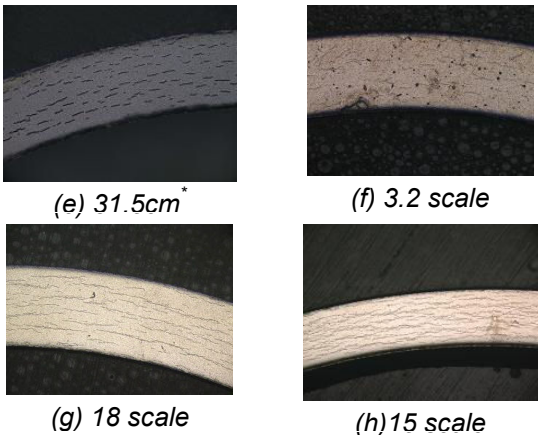
As the furnace temperature is increased, the movement of mixed gas becomes very active. Therefore, the pressure in both the burette and the mass-cylinder becomes high. The following table is the result of many tests.(Refer fig.3 & 4)

- As the gap between the incoming gas amount and the discharging gas amount becomes large and/or as the pressure in the quartz tube is increased,
- H_2 gas permeates into the samples very well.

Finally, when the mass-cylinder scale is maintained just above 15 scale, H_2 gas permeation was made very well. If the pressure of mass-cylinder is over or under 15 scale, the pressure can be controlled with the following methods :

TABLE2- *Pressure controlling method*

Pressure condition	Controlling method
Under 15 scale	Slightly open the incoming gas valve until the pressure goes up to 15 scale of mass-cylinder
Over 15 scale	Slightly open the discharging gas valve until the pressure comes down to 15 scale of mass-cylinder



*(e) 31.5cm** *(f) 3.2 scale*

(g) 18 scale *(h)15 scale*

Fig.3- *Burette and mass-cylinder pressure (100x)*

** In case of (e), scale reading is not possible*

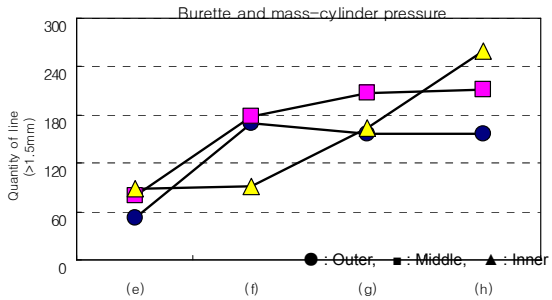


Fig.4- *Quantity of the lines in fig. 3*

2.3. Mixed gas amount

Many tests were performed under the conditions having various input/discharge gas flow ratio with the input gas flow increased and the discharge gas flow decreased. As a result of these tests and considering pressure condition, the incoming gas flow rate was set appropriately at 150ml/min. and the discharging gas flow rate was set appropriately at 80ml/min.. [Refer to Fig.3-(h)]

3. Conclusion

The optimal parameters for Hydrogen orientation test can be obtained through the integrated tests with main parameters such as time, temperature, pressure and mixed gas amount.

Here are more findings on this study as follow ;

- Time was the most critical factor having a direct effect on H_2 gas permeation formation.
- Pressure should be consistent during temperature holding time.
- Temperature was not an influential factor any longer as long as the temperature is over 400℃.
- The mixed gas consisted of H2 (2%) and Ar (Balance). Pressure is dependent upon both the gas incoming and the gas discharging flow rate in the quartz tube.

First of all, it was found that the most critical parameter was temperature holding time.
Most of H_2 gas permeation was made during 3hrs and 6hrs. The degree of permeation is almost same during them.
Considering all test parameters and those optimal combinations, 3hrs ~ 3.5hrs was the most appropriate for temperature holding time.

The optimal parameters are in the table below.

TABLE 3 – *Optimum parameters for H_2 gas permeation*

No.	Parameter	Range	Optimum value
1	Pressure of mixed gas container	0.65 ~ 2 bar	1.8Bar
2	Incoming gas amount (flow rate)	150ml/min.	150ml/min.
3	Discharging gas amount (flow rate)	80ml/min. ~ 100ml/min.	80ml/min.
4	Temperature Holding Time	3 hrs ~ 6hrs	3hrs ~ 3hrs 30min.
5	Pressure of burette and mass-cylinder	15 ~ 16 scale	15 scale

REFERENCES

[1] ASTM B353-02, Annex A2
[2] ASTM B811-02, Annex A2