# Development of Electrolytic Hydriding System for Hotcell Testing

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## 1. Introduction

Various studies on degradation mechanisms caused by hydride have being performed. As a specimen preparation step, hydrogen charging methods have been developed for experiment of the hydride-induced degradation.

An electrolytic hydrogen charging method is widely used. Sufficient hydride layer is deposit on the surface of material and the hydrogen is diffusion-annealed from the hydride layer to the material. After the homogenization, the excess hydride layer is removed. In this method, the amount of hydrogen charged into the material cannot exceed the terminal solid solubility (TSS) at the homogenization temperature [1]. Because setup of the equipment is relatively simpler than other method, it is suitable for a hotcell testing.

The electrolytic hydriding procedures for an irradiated material has been developed to make specimens for delayed hydride cracking (DHC).

In this paper, the modified electrolytic hydriding equipment and handling tools for hotcell testing are introduced. Also, the commissioning test results of electrolytic hydriding on un-irradiated Zr-2.5Nb are described.

## 2. Methods

### *2.1 Electrolytic hydriding system*

In hotcell testing, equipment should be modified to easily handle using a manipulator because it is difficult for a researcher to access during the test. The hydriding equipment were modified as shown in Fig.1. The fume hood system was designed to minimize the damage of other equipment installed in the hotcell by vaporized electrolyte. The handling tools were developed to allow all process from setting up equipment to dismantling using manipulator. The performance of developed equipment and tools were verified in the operating area prior to installation in the hotcell.

### *2.2 Commissioning test of hydriding*

The commissioning test of hydriding on un-irradiated Zr-2.5Nb tube section was performed. As shown in Fig.2, the tube section has an internal diameter of 103 mm, a wall thickness of 4.2 mm and a height of 54mm.

The hydriding test conditions are shown in Table 1. The electrolytic hydriding was performed in a ~0.1 Mol/L sulfuric acid at 60 to 90 $^{o}$C with a current density of about 100 ~120 mA/cm$^2$.

Table 1. Hydriding test conditions

| Parameter | Test condition |
|---|---|
| Target Current | 45 ± 5 A |
| Target Temperature | 60 ~ 90 $^{o}$C |
| Target Hydriding Time | 48 hr |

## 3. Results

### *3.1 Evaluation of oxide and hydride layer thickness*

After the hydriding, two pellets were punched from the hydrided Zr-2.5Nb tube section to check the hydride layer thickness. Also, one pellet was punched from the as-received Zr-2.5Nb tube section to check the oxide layer thickness.

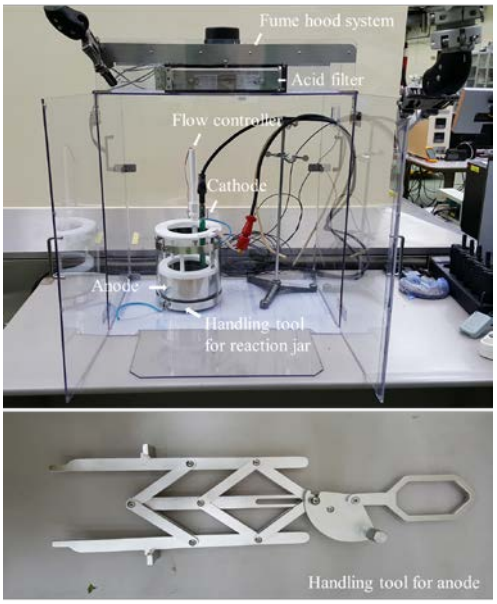


Fig. 1. Electrolytic hydriding equipment and handling tools

Fig. 2. Un-irradiated Zr-2.5Nb tube section

Metallography images are shown in Fig.3, Fig.4 and Fig.5. Also, results of oxide and hydride layer thickness measurement are shown in Table 2. As shown in the metallography images, the oxide thickness of as-received tube section is about 1.4 μm. The thick hydride layers were uniformly deposited on the surface and the thickness was about 29 μm. Minimum hydride layer thickness to achieve target hydrogen concentration is depending on testing conditions, however, more than 15 μm thick hydride layer is generally required. Therefore it was sufficient thickness to achieve target hydrogen concentration.

In hotcell testing, if sufficient hydride layer thickness is achieved, diffusion-annealing treatment is performed to reach the target hydrogen concentration. Diffusion-annealing temperature and time are determined in consideration of target hydrogen concentration and operating temperature [2]. Finally, pellets for Hydrogen/Deuterium (H/D) analysis are punched after removing remaining hydride layer.

Table 2. Results of oxide and hydride layer thickness measurement

| Thickness (μm) | Oxide | Pellet-1 | Pellet-2 |
|---|---|---|---|
| Min. | 0.606 | 25.506 | 24.291 |
| Max. | 4.545 | 32.793 | 32.186 |
| Mean | 1.413 | 29.009 | 29.278 |
| Std.Dev | 0.579 | 1.516 | 1.892 |

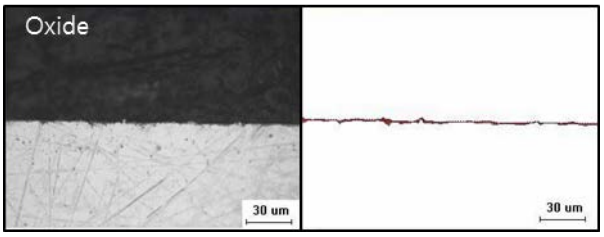

Fig. 3. Metallography images of oxide before hydriding

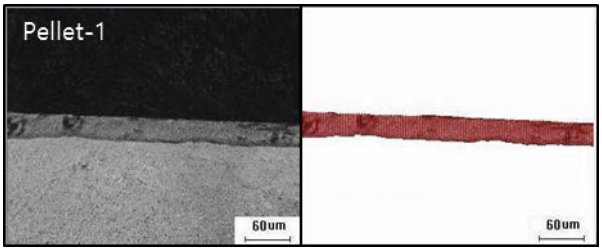

Fig. 4. Metallography images of pellet-1 after hydriding

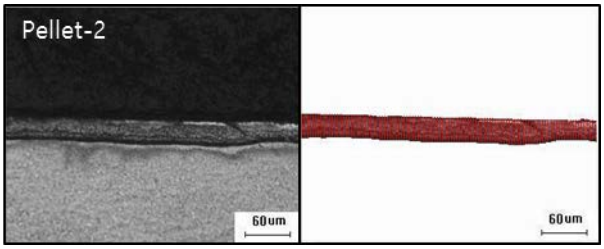

Fig. 5. Metallography images of pellet-2 after hydriding

## 4. Conclusions

The electrolytic hydriding equipment and procedure were successfully developed for hotcell testing. It was proven through the commissioning test on un-irradiated Zr-2.5Nb tube section.

The electrolytic hydriding on irradiated Zr-2.5Nb tube section are in progress and uniform and thick hydride layer have being achieved.

## REFERENCES

[1] R.N.Singh et al., “Hydrogen charging, hydrogen content analysis and metallographic examination of hydride in zirconium alloys”, BARC/2003/E/034.
[2] A.D.Lepage, W.A.Ferris, G.A.Ledoux, “Procedure for Adding Hydrogen to Small Sections of Zirconium Alloys”, FC-IAEA-03, November 1998.