Proceedings of the Korean Nuclear Society Autumn Meeting
Taejon, Korea, October 2000

Experiment of Hydrogen Embrittlement of Tritium Storage Vessel Material

Hai Yong Jung and Kun Jai Lee

Korea Advanced Institute of Science and Technology (KAIST)
373-1 Kusong-dong, Yusong-gu, Taejon, Korea 305-701

H. Chung and S. Paek

Korea Atomic Energy Research Institute (KAERI)
150 Duckjin-dong, Yusong-gu, Taejon, Korea 305-353

Abstract

The tritium storage is one of the most important problems for the safety of tritium removal facility. In current, many researches for tritium immobilization have been carried out. The research for tritium storage could be divided into two parts, one is for the metal getter of tritium and another is for the integrity of tritium storage vessel. Especially, the integrity of tritium storage vessel is up to the tritium embrittlement of vessel material, for tritium vessel is mostly made of metal material. In this work, the evaluation of the tritium embrittlement for the tritium storage vessel material is performed with the equipment that is made for high temperature and high vacuum. However, tritium is the radioactivity material, so hydrogen is used for this work. In this work, three metals were chosen for the vessel candidate material, carbon steel, austenitic stainless steel (SUS) 304 and 316L. The experiment was carried out for the several conditions of temperature and pressure. The property change of metal was investigated through the tensile test. Austenitic stainless steel has a high resistance for the hydrogen embrittlement from the result. But the obvious gap between SUS 304 and SUS 316L is not revealed, because the experiment condition may be not sufficient to show the difference between SUS 304 and SUS 316L.

I. INTRODUCTION

The technology of tritium storage is necessary for the entire safety integrity of tritium removal system. Therefore, many studies for tritium storage have been accomplished by many researcher in Korea. In case of other foreign country, tritium storage vessel had been designed and has been used in many tritium removal facilities. Korea has a plan to start the TRF in 2006, so the technology of tritium storage should be developed as soon as possible.

Many countries and companies are undertaking the storage or disposal of tritium extracted from the heavy water of the power reactors. Fixation as a metal tritide is the prime candidate for form of the tritium, although pressurized gas also is being considered. In both methods a primary container will be required. Austenitic stainless steels among many metal materials have a low permeability to hydrogen and are comparatively resistant to any deleterious effects of hydrogen on mechanical properties. In this paper the effects of hydrogen on mechanical properties of candidate materials of storage vessel are considered.(1)(2)

The evaluation of the hydrogen embrittlement of the tritium storage vessel is carried out through the experimental system. An experimental system is designed and equipped to evaluate hydrogen effects between metal hydride and vessel material. Hydrogen absorbed in metal as metal hydride form might be desorbed due to the external high temperature. In this case an inner pressure increase, therefore, the inner wall of container has a possibility to be embrittled by hydrogen in a condition of high temperature and pressure. The hydrogen effects on vessel material were tested by tensile test and the tensile stress of each metal in several conditions showed the change of mechanical property due to hydrogen embrittlement. The evaluation of hydrogen embrittlement of three candidate materials is to select a proper material through comparing the hydrogen effects of each metal and to recommend the safety scenario of tritium storage.

II. EXPERIMENTAL

1. Specimen Preparation

Several candidate metal materials were evaluated for hydrogen embrittlment. They include carbon steel, austenitic stainless steel 304 and 316L. A round-bar specimen of each metal, the shape which is illustrated in figure 1, is prepared to be tested through tensile test after hydrogen exposure experiment. The specimen is based on ASTM Standard E8 -"Test Methods for Tension Testing of Metallic Material".(3) The composition of each material is summarized in table1.

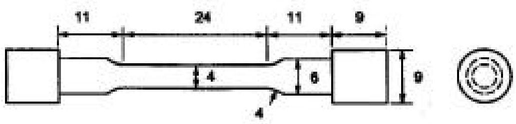

Figure 1. Shape of specimen for tensile test

Table 1. Composition of candidate material used in the experiment (w/o)

	C	Mn	Si	Cr	Ni	P	S	Others	Fe
AISI 1045	0.42-0.48	0.60-0.90	0.15-0.35	≤ 0.20	≤ 0.20	≤ 0.03	≤ 0.035	-	Bal.
SUS 304	0.08	2.00	1.00	18.0-20.0	8.0-10.5	0.045	0.03	-	Bal.
SUS 316L	0.03	2.00	1.00	16.0-18.0	10.0-14.0	0.045	0.03	2.0-3.0Mo	Bal.

2. Equipment

The entire experimental apparatus is schematically drawn in figure 2 and figure 3 shows the real apparatus picture. The apparatus consist of a part of high temperature and a part of vacuum.(4) For high temperature, electric furnace, which can heat up to max. 1400°C, is used. A part of heating in which the specimen and Ti-powder are placed, are made up of quartz glass. The apparatus could be evacuated to 10^{-6} torr by a vacuum pump that is made up of a rotary pump and a diffusion pump.

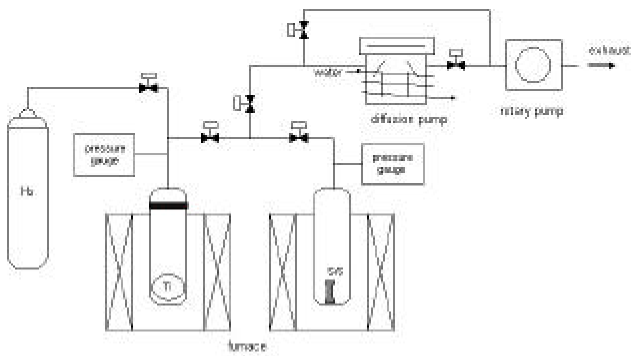

Figure 2. Schematic drawing of apparatus

Fuigure 3. Picture of a installed real apparatus

3. Procedure

Experiment of hydrogen embrittlment can be divided into several steps: activation, charging, discharging, and tensile test.(5)

Activation

The titanium must be activated before it can be absorb hydrogen readily. Activation can be accomplished by heating the titanium to about 800°C about 1-2 hours under 10^{-6} torr vacuum to remove volatile impurities and oxides from the titanium surfaces. To increase the activity, the evacuation may be followed by exposing the titanium to hydrogen. The absorbed hydrogen is then desorbed from the titanium by evacuation and heating at about 800°C. This procedure is repeated at 2-3 times.

Charging

After activation, hydrogen is absorbed into titanium powder at about 800°C. Titanium powder that absorbed hydrogen is cooled at room temperature.

Discharging

Hydrogen stored in titanium will be recovered (dischaged) by heating and evacuation. Titanium powder absorbed hydrogen at a charging step and a specimen of candidate material are placed in a quartz tube. Prior to heating, the apparatus is evacuated by a vacuum pump to 10^{-6} torr and then the hydrogen gas is released out of the titanium powder by heating it at a specific temperature. The temperature and pressure are continuing during the expected time. The experiments were carried out at temperatures of 600°C, 800°C, and 1000°C and during times of 8h. After heating, the specimen was cooled to room temperature in air.

Tensile test

Tensile test methods cover the tension testing of metallic materials in any form at room temperature, specially, the methods of determination of yield strength, yield point elongation, tensile strength, elongation, and reduction area. Tension tests provide information on the strength and ductility of materials under uniaxial tensile stresses. This information may be useful in comparisons of materials, alloy development, quality control, and design under certain circumstances.

Tensile test was accomplished to investigate the change of mechanical property during the discharging stage at room temperature. Figure 4 and 5 illustrate the view of tensile test.

Figure 4. View of tensile test.

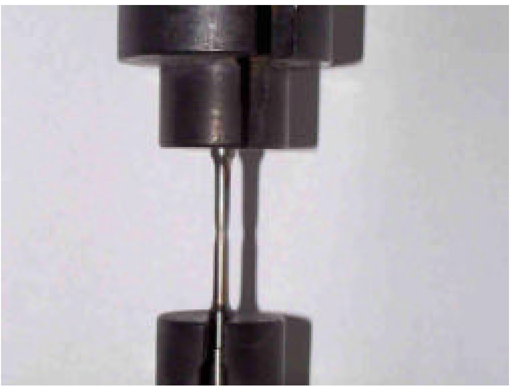

Figure 5. View of the specimen during test.

III. RESULTS AND DISCUSSION

The experiments were accomplished at several conditions of heating temperature. To compare the thermal effects and hydrogen embrittlement of metal material, the experiment are independently carried out at same temperature in a condition of vacuum in which have no titanium powder and the hydrogen is desorbed from titanium powder. The experiments were performed in duration time of 8h, because the resistance of SUS against hydrogen is so high that the change of mechanical property may not be discovered in short time. The experiment in duration time of 8h, therefore, was carried out for AISI 1045 (carbon steel), SUS 304, and SUS 316L.

The experiment results are summarized in table 2 and shown in figure 6 to figure 8. The tensile strength of material was compared each other as experimental conditions. Table 2 show the decrease of tensile strength of material by hydrogen attacking. The change of tensile strength in case of AISI 1045 is lager than any others. It comes obviously out in case of high temperature, 1000°C. Carbon steel might have a weakness for hydrogen embrittlement. As generally known, austenitic stainless steel is a good material has the resistance against hydrogen. In this experiment, the change of tensile strength for austenitic stainless steel was indistinctly discovered, especially, at temperature of 600°C, 800°C.

In case of SUS 316L, It was at 1000°C that the decreasing of tensile stress could be found. On the other hand, SUS 304 is relatively weaker than SUS 316L for hydrogen embrittlement at high temperature.

Table 2. Tensile strength of material in each experimental condition.

	AISI 1045			SUS 304			SUS 316L		
Temp.	600	800	1000	600	800	1000	600	800	1000
Vacuum	690	672	727	727	614	592	717	660	568
Hydrogen	694	642	603	733	609	571	713	665	556
Change (%)	+0.6	-4.5	-17.1	+0.8	-0.8	-3.5	-0.6	+0.8	-2.1

IV. CONCLUSIONS

Hydrogen embrittlment of candidate metal material has been measured by tensile test under the various conditions. The change of tensile strength has been obviously discovered in case of carbon steel and in case of high temperature condition. The hydrogen effect has not been found out for austenitic stainless steel and in case of low temperature condition.

Austenitic stainless steel 316L might be a proper material for tritium storage container in a view of hydrogen embrittlement. The more experiments, however, should be needed to strongly recommend the material for tritium storage container. If the experimental procedure and condition are modified and added, this evaluation method should be able to present more information, for example, safety scenario and so on.

Acknowledgment

This work has been carried out under the Nuclear R&D Program by MOST.

References

1. AECL, "TRITIUM IN AUSTENITIC STAINLESS STEEL VESSELS: THE INTERGRITY OF THE VESSEL." AECL-6972, 1980

2. AECL, "THE CONTAINMENT OF TRITIUM IN AUSTENITIC STAINLESS STEEL VESSELS " AECL-7159, 1981

3. ASTM, "Annual Book of ASTM Standard", 1997

4. M. SUGISAKI, H. FURUYA, " Tritium Solubility in SUS 316 Stainless Steel", Jour. Of Nuc. Material 120, pp36-40, 1984

5. L.K. Heung, "Titnium For Long Term Tritium Storage", WSRC-TR-94-0596, 1994

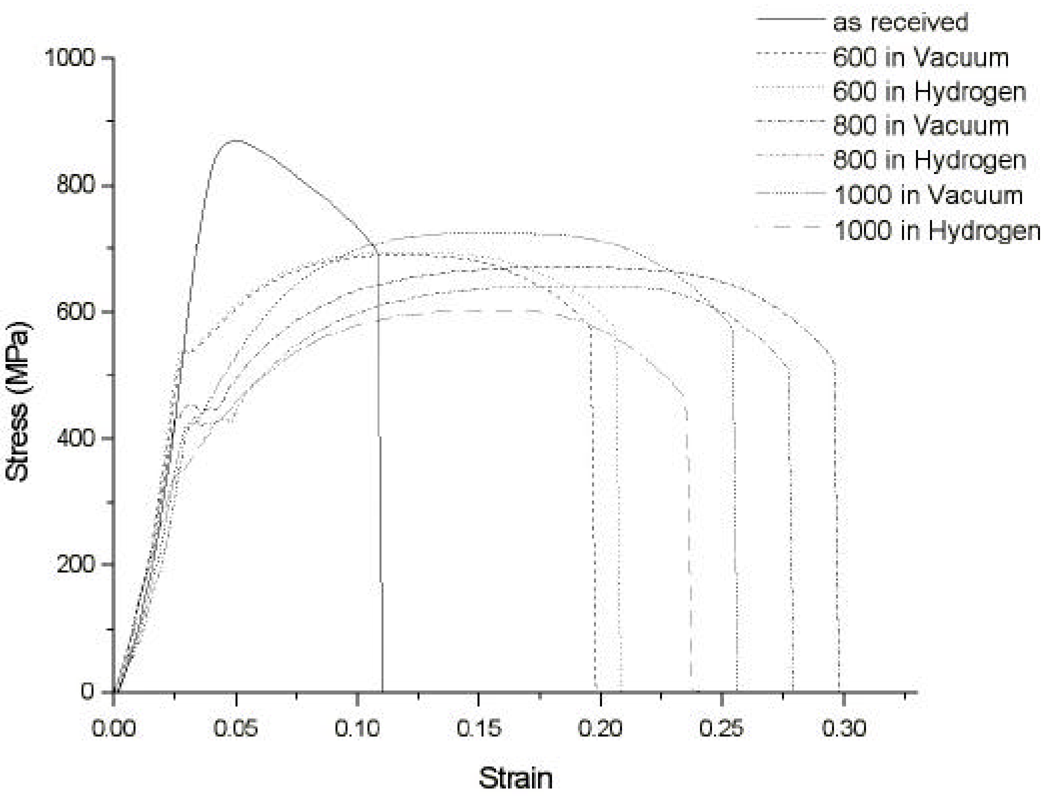

Figure 6. Tensile test of AISI 1045

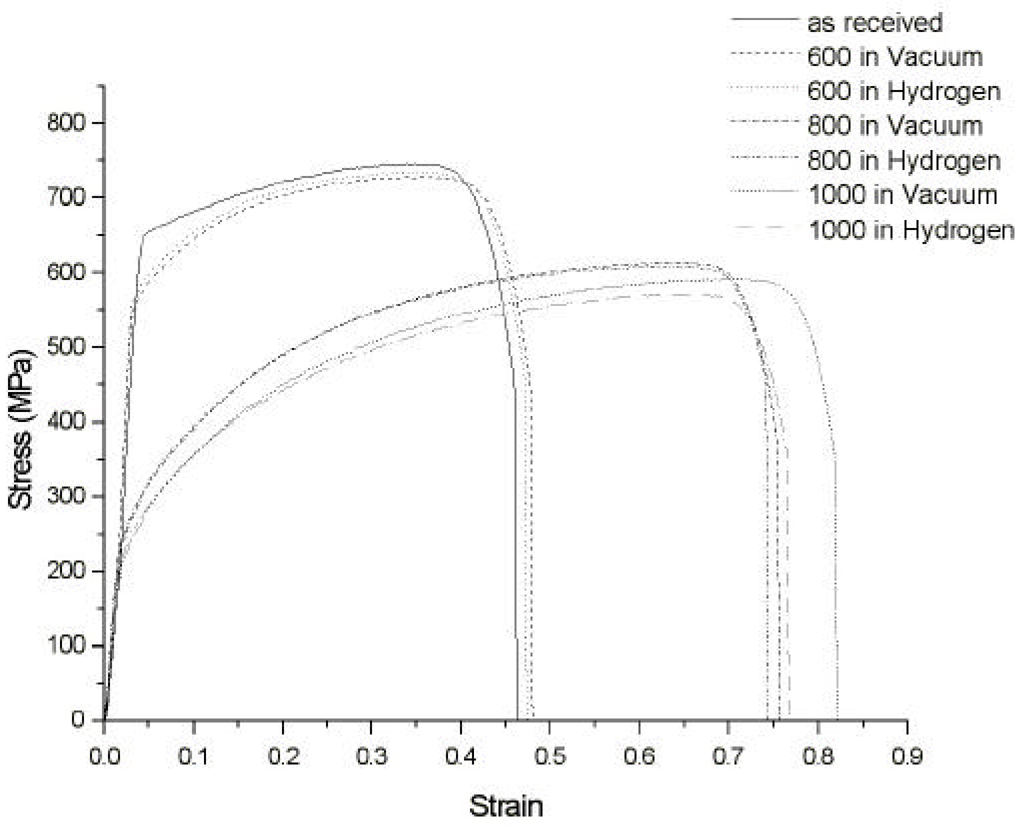

Figure 7. Tensile test of SUS 304

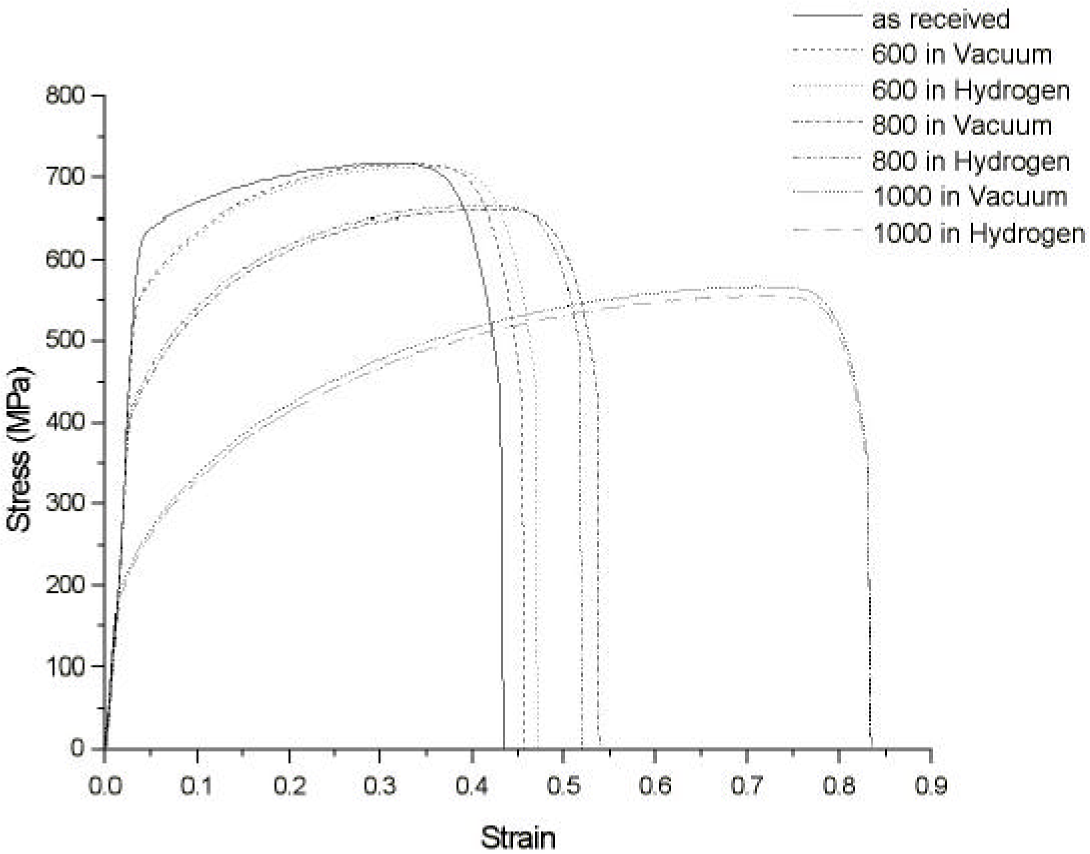

Figure 8. Tensile test of SUS 316L