

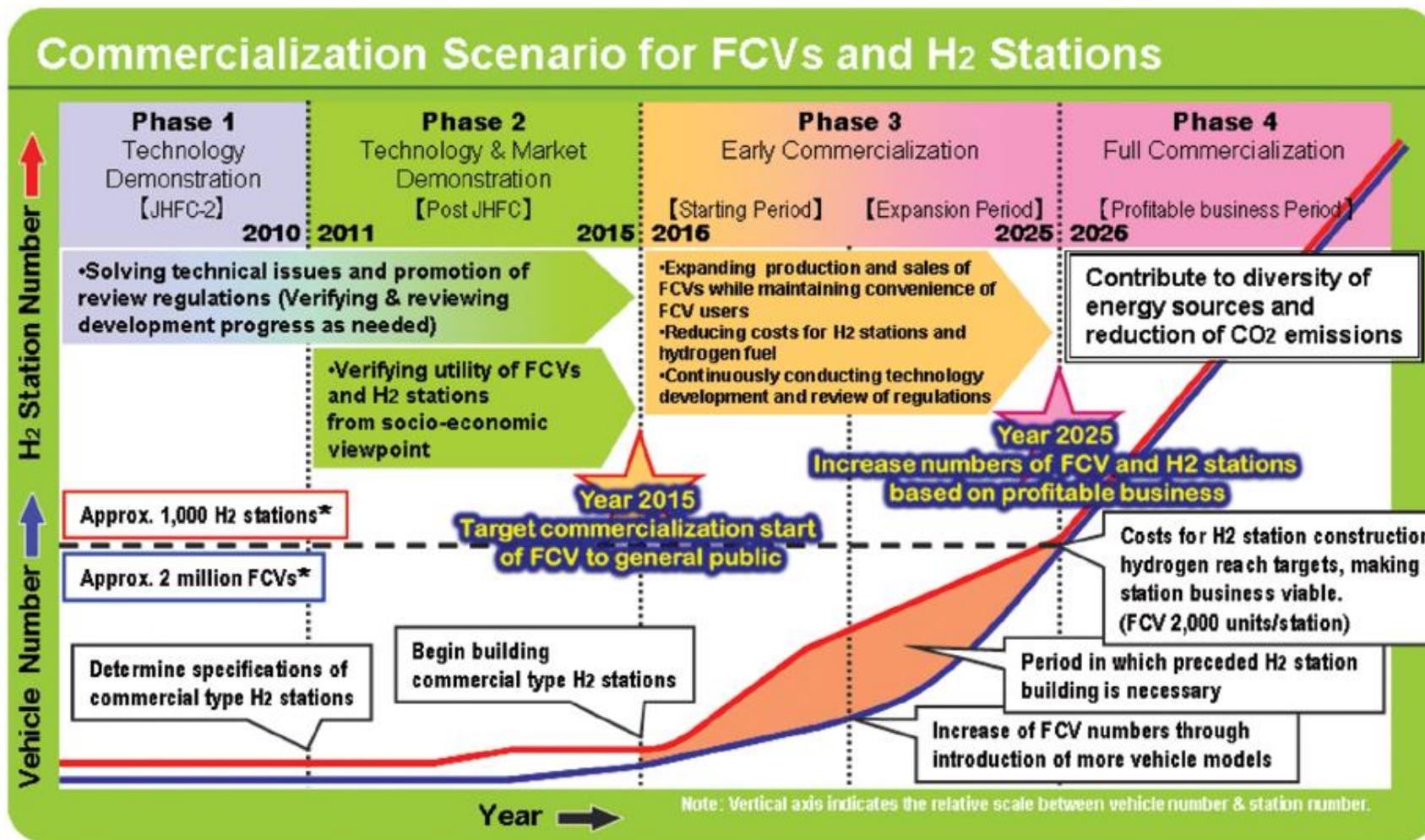
# Material Test Systems in High Pressure Hydrogen Gas at AIST Tsukuba

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\* Precondition: Benefit for FCV users (price/convenience etc.) are secured, and FCVs are widely and smoothly deployed

- 2015 · Commercialize FCV
- Build 100 hydrogen filling station

<http://fccj.jp/eng/index.html>

## HYDROGENIUS (Research Center for Hydrogen Industrial Use and Storage)

Collaborative Research Center between Kyusu University and AIST (National Institute of Advanced Industrial Science and Technology)



research collaboration agreements (1 May 2006)



**KYUSHU UNIVERSITY**  
 Fukuoka



Tsukuba



Yukitaka Murakami Dr.Eng., Ph.D.  
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 Vice President, <sup>2</sup>CNER, Kyushu University



**Fundamental Research Project on Advanced Hydrogen Science**

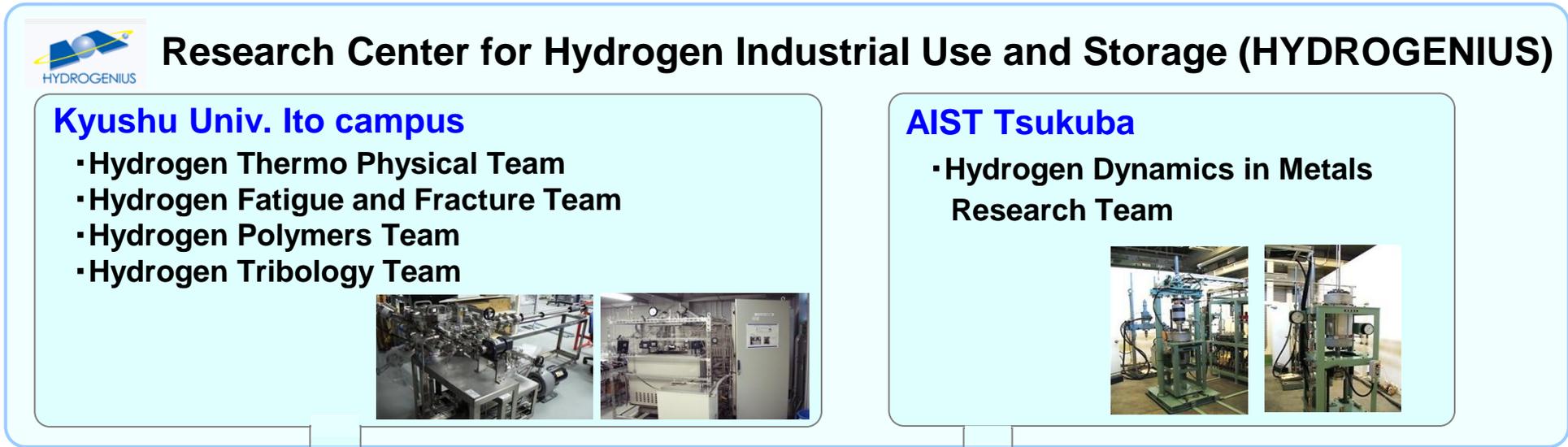
Funding

HYDROGENIUS (1 July. 2006)

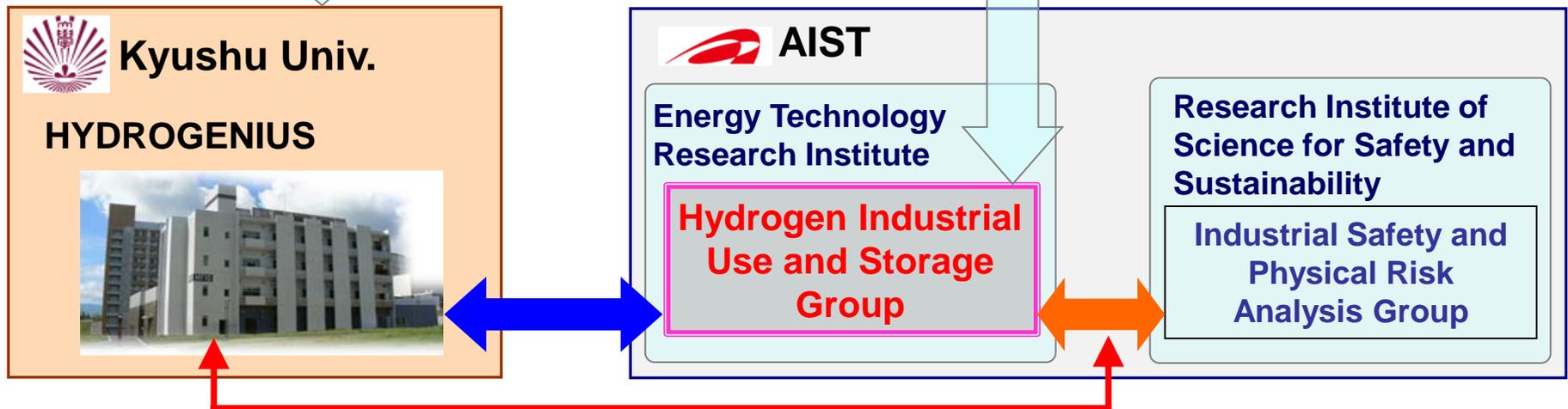


New Energy and Industrial Technology Development Organization

## 1<sup>st</sup> stage: 2006-2013



## 2<sup>nd</sup> stage: 2013-



## Collaborative Research Team for Hydrogen Industrial Use and Storage

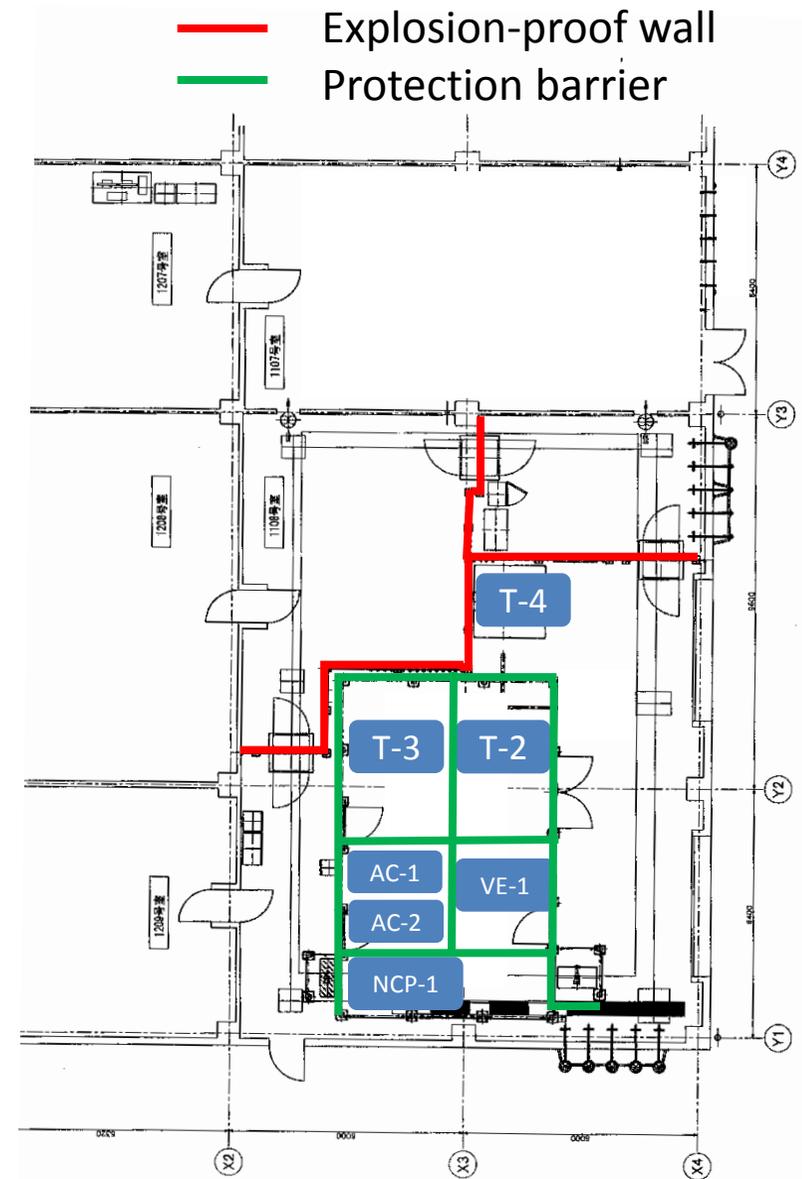
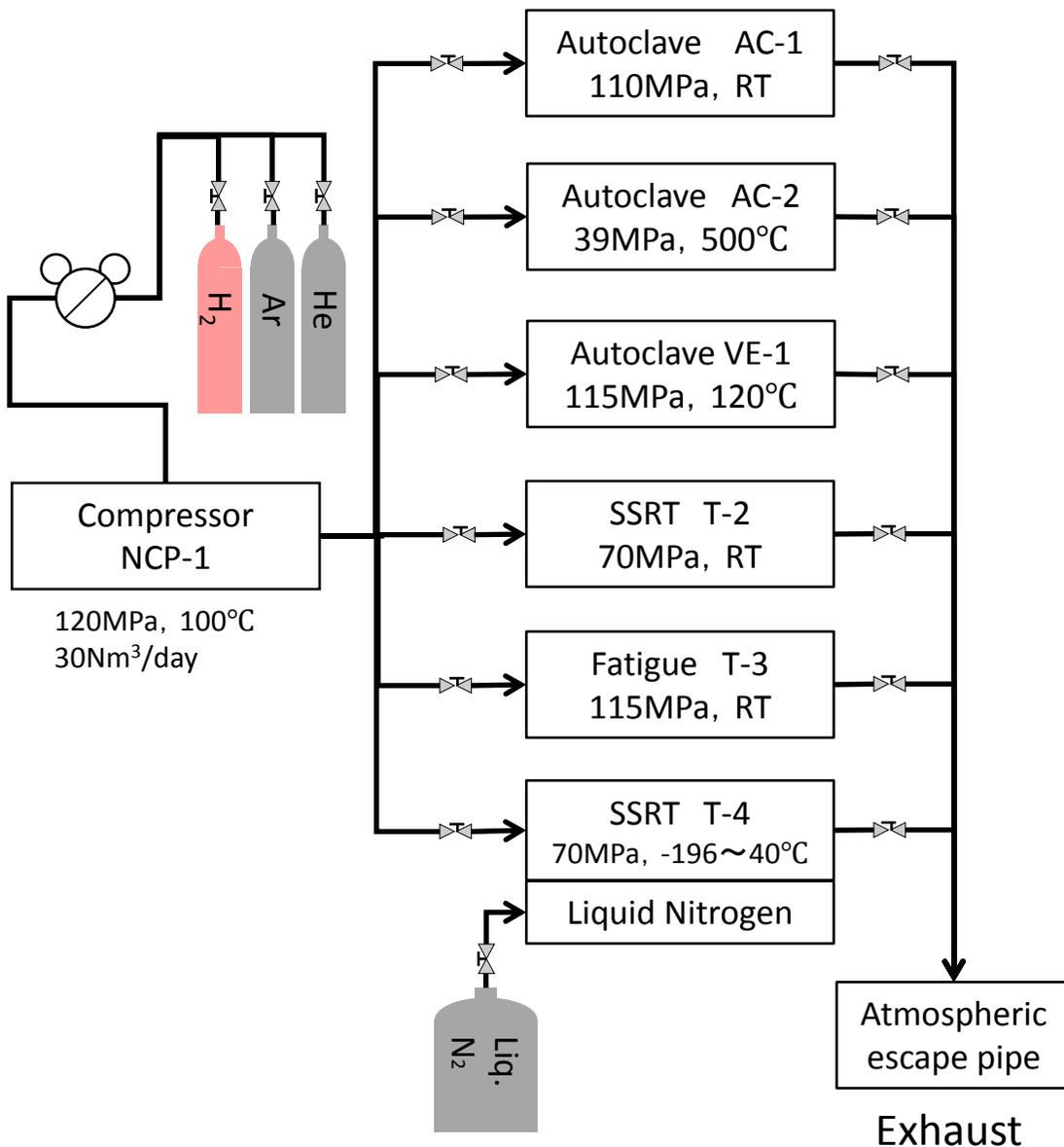
## 1. Material Test system

- Fatigue test : RT, 115MPa
- SSRT
  - RT, 70MPa
  - -190°C - RT, 70MPa

## 2. Autoclave: Static exposure system

- 500°C, 40MPa
- 120°C, 115MPa
- RT, 110MPa

# AIST Hydrogen facility in Tsukuba



## Materials test system at room temperature

**SSRT**



**70MPa**

**Fatigue test**



**115MPa**

## Static exposure system (Hydrogen charge test)



**500°C 40MPa**



**100°C 115MPa**



120MPa compressor



Protection barrier



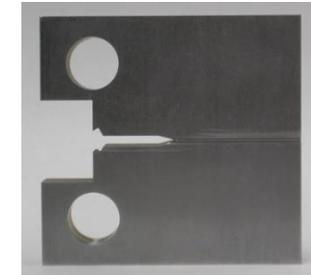
- Control facilities
- Explosion-proof wall
- PC control gas operating system
- PC control material test system
- Monitoring camera system



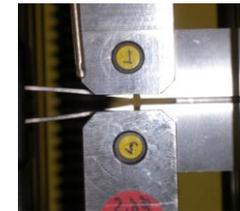
## Materials test system at room temperature

### 1. Rising-displacement fracture threshold test

Fracture test : 0.1 ~ 115 MPa in H<sub>2</sub>

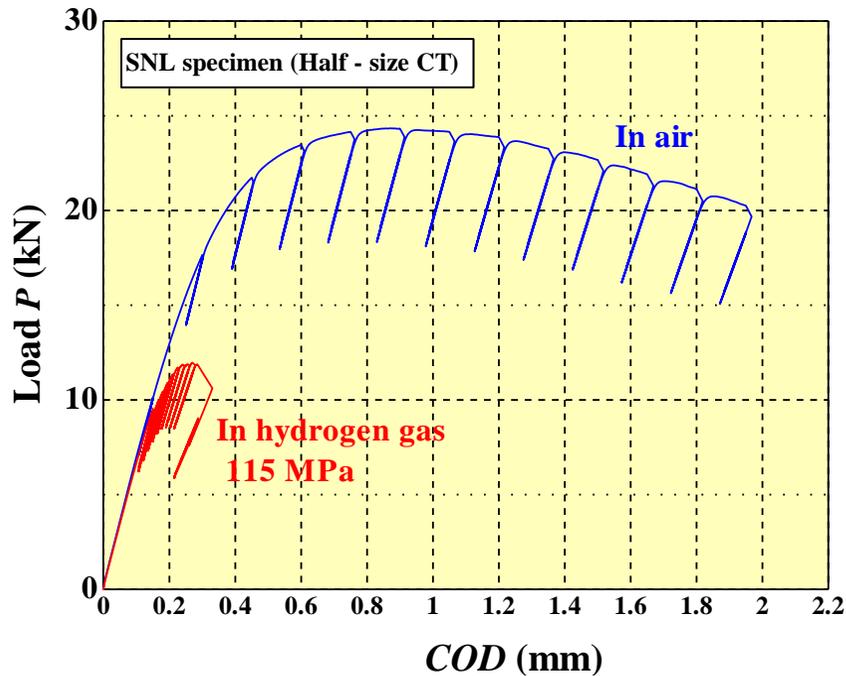


CT Specimens



Sample set up

## Rising-displacement fracture threshold test



## Unloading elastic compliance method

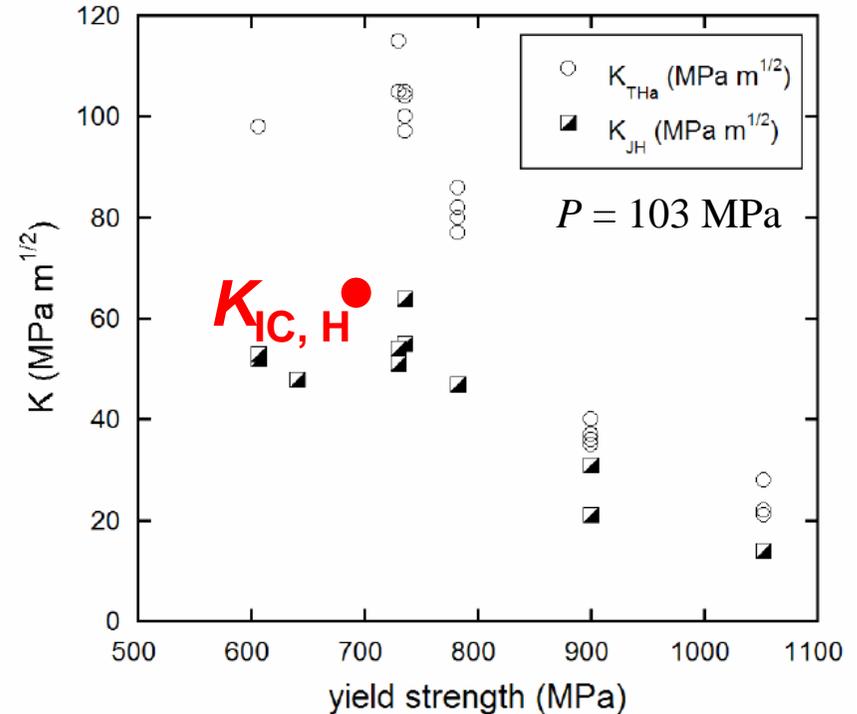


Fig. Crack arrest thresholds from constant displacement ( $K_{THa}$ ) and crack initiation thresholds from rising displacement tests ( $K_{JH}$ ) plotted as a function of yield strength in 103 MPa hydrogen gas\*<sup>1</sup>

\*<sup>1</sup> Kevin A. Nibur et al. SANDIA REPORT(2010)

● SCM435:  $\sigma_{ys}=700$ ,  $K=62.9$

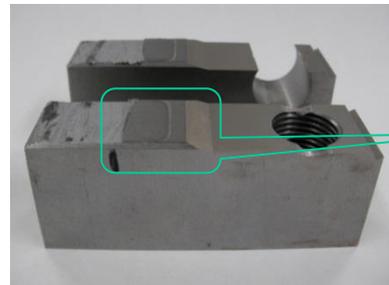
Materials test system at room temperature

2. Crack-arrest threshold under static loading test

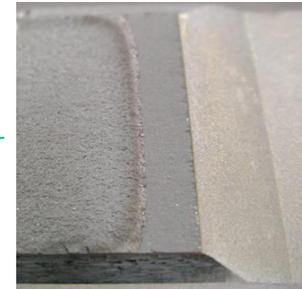
Fracture test : 0.1 ~ 115MPa in H<sub>2</sub>



Sample



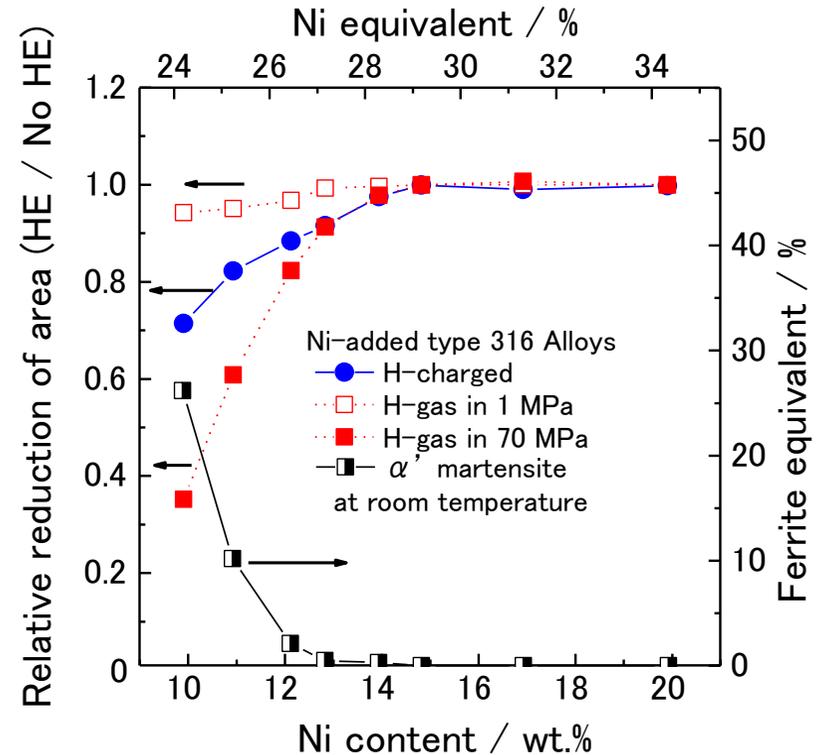
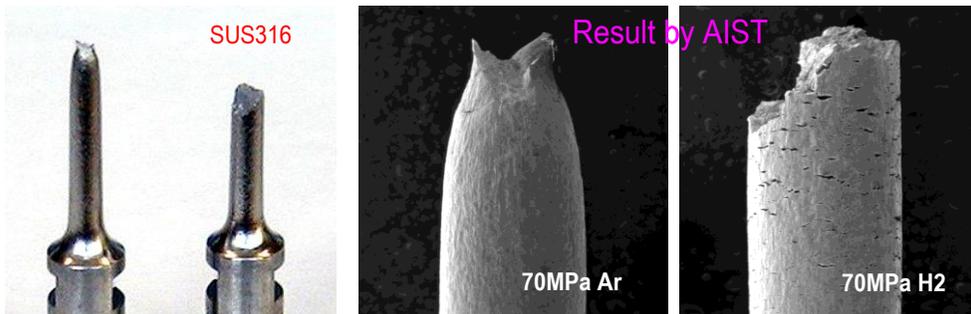
Fractured sample



Fractured surface

## Materials test system at room temperature

### 3. SSRT test : 70MPa in H<sub>2</sub>



Effect of Ni content on HE and IRHE of Ni-added type 316 stainless steel at room temperature.

## AIST Tsukuba facilities

### 1. Material Test system

- Fatigue test : Rising-displacement fracture threshold test
- SSRT
  - RT
  - Low temperature

### 2. Autoclave

- Static exposure system (Hydrogen charge)
- Crack-arrest threshold under static loading test

**“Collaborative work with SNL for technical evaluations and standards of hydrogen storage tank and vessel”**

## **Objective**

**To promote FCV, develop the evaluation method of the materials for the hydrogen storage tank and vessel, and clarify the mechanism of hydrogen embrittlement.**

## **Final goal**

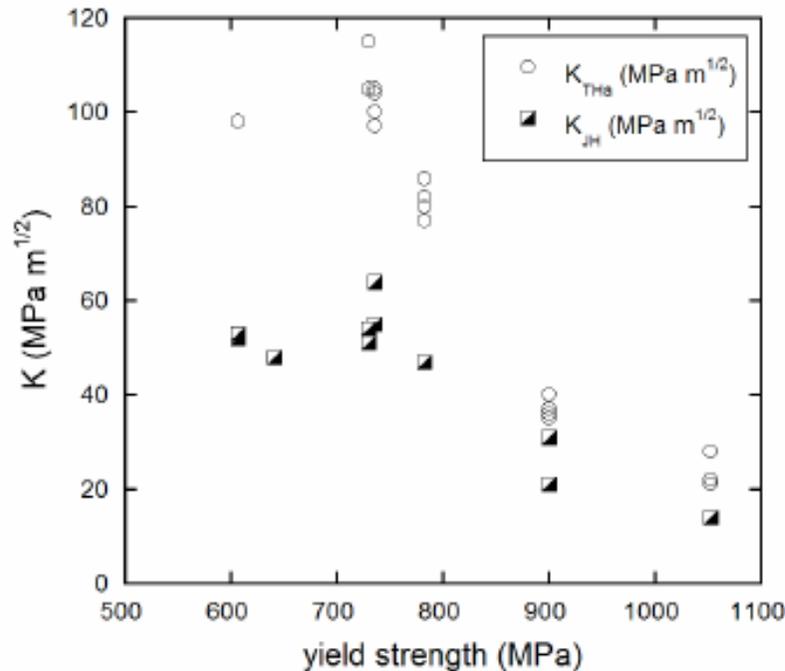
**Contribute the revision of international standards like ASTM or ISO in collaboration with Japan and US.**

## Background

The engineering analysis method for calculating the design life of steel hydrogen pressure vessels, [ASME KD-10](#) (Boiler and Pressure Vessel Code) requires measurement of **two fracture properties** for the steel containment materials in hydrogen gas.

1. The crack-arrest threshold under static loading ( $K_{THa}$ )
2. The fatigue crack growth relationship ( $da/dN$  vs.  $\Delta K$ ) under cyclic loading

Recent testing at Sandia to measure the fracture thresholds of ferritic steels in hydrogen gas demonstrated that the crack-arrest threshold under static loading ( $K_{THa}$ ) was higher than the crack-initiation threshold under rising-displacement loading ( $K_{THi}$ ) for lower-strength ferritic steels.



**SANDIA REPORT**  
 SAND2010-4633  
 Unlimited Release  
 Printed July 2010

Figure 19 Crack arrest thresholds from constant displacement tests ( $K_{THa}$ ) and crack initiation thresholds from rising displacement tests ( $K_{IH}$ ) plotted as a function of yield strength.

The rising-displacement fracture threshold may be a more conservative value to use in the life prediction analysis.

However, the rising-displacement fracture threshold is not commonly measured, so more testing directed at developing proper methods is needed

## Outline of the project

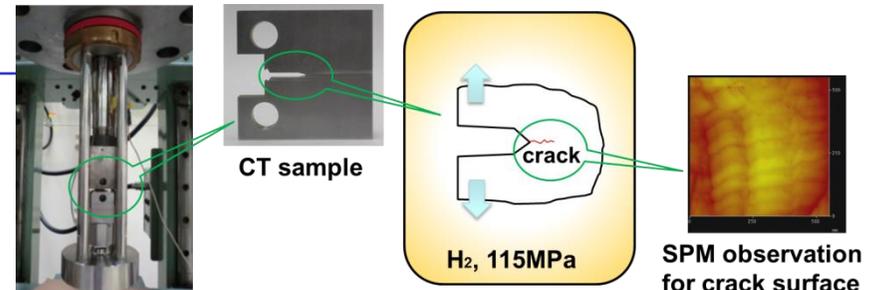
### 1. Measurement of Ferritic Steel Fracture Properties in Hydrogen Gas

Measure the rising-displacement fracture thresholds ( $K_{THi}$ ) for the same steels under identical mechanical loading and environmental conditions at AIST and Sandia.

- Sandia supply machined CT specimens from the SA372 Grade J steel to AIST.
- AIST supply machined CT specimens from the SCM435 steel to Sandia.
- The testing results from AIST and Sandia will be directly compared.

### 2. Identifying the Mechanisms for Hydrogen-Assisted Fracture in Stainless Steels

Carry out an experiment to clarify the nanoscale structures of the hydrogen-assisted fracture surfaces and crack profiles for the typical materials by using SPM techniques.



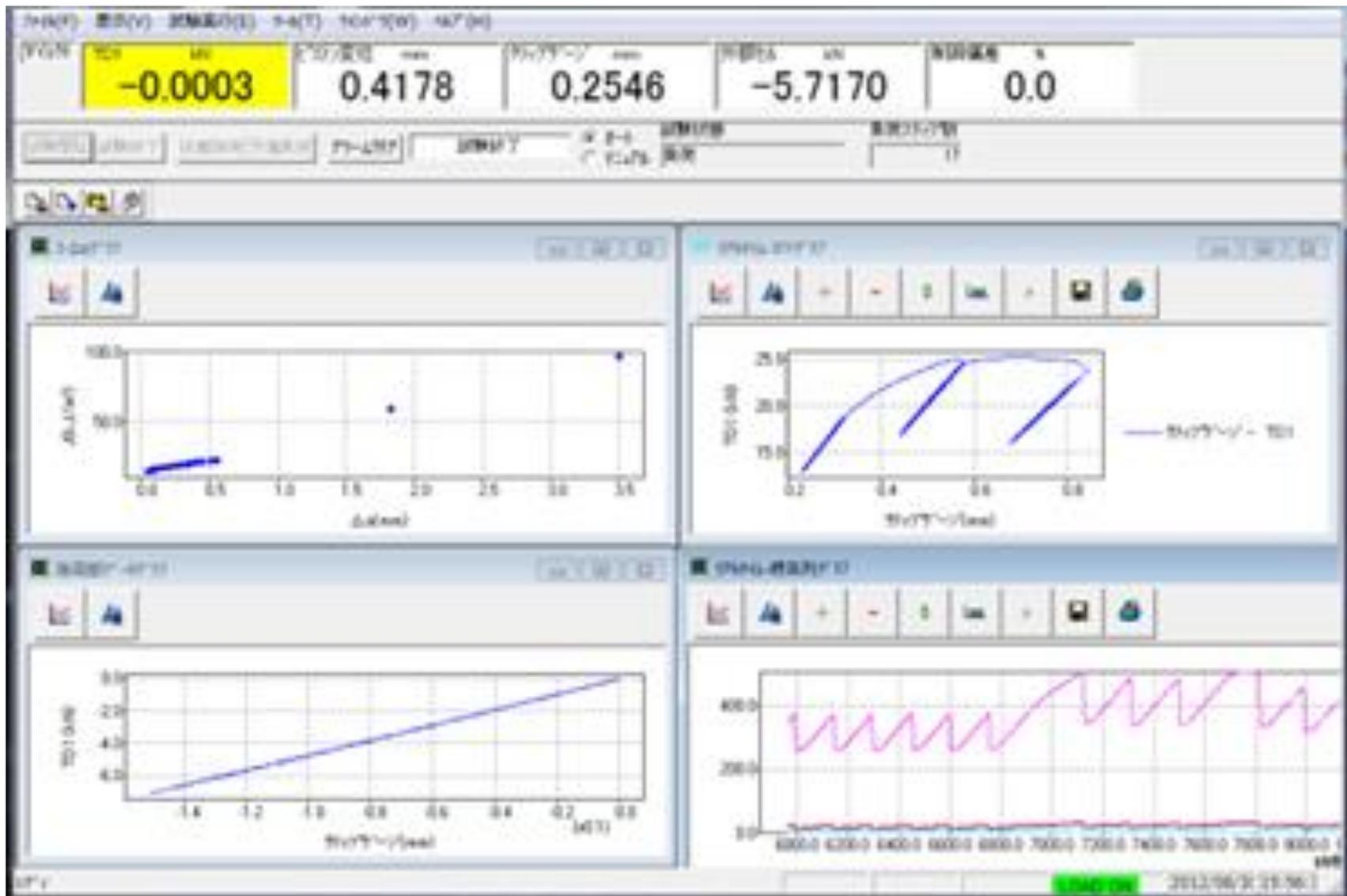


Table 1. Proposed test matrix for measuring rising-displacement fracture thresholds of Cr-Mo steels in high-pressure hydrogen gas.

Steel	H <sub>2</sub> pressure (MPa)	Load-line displacement rate (mm/s)
SCM435	10	$2 \times 10^{-3}$ (2-3 hours)
SCM435	40	$2 \times 10^{-3}$
SCM435	115	$2 \times 10^{-3}$
SCM435	10	$2 \times 10^{-4}$
SCM435	40	$2 \times 10^{-4}$
SCM435	115	$2 \times 10^{-4}$
SCM435	10	$2 \times 10^{-5}$ (300 hours)
SCM435	40	$2 \times 10^{-5}$
SCM435	115	$2 \times 10^{-5}$
SA372 Grade J	10	$2 \times 10^{-3}$
SA372 Grade J	40	$2 \times 10^{-3}$
SA372 Grade J	115	$2 \times 10^{-3}$
SA372 Grade J	10	$2 \times 10^{-4}$
SA372 Grade J	40	$2 \times 10^{-4}$
SA372 Grade J	115	$2 \times 10^{-4}$
SA372 Grade J	10	$2 \times 10^{-5}$
SA372 Grade J	40	$2 \times 10^{-5}$
SA372 Grade J	115	$2 \times 10^{-5}$