MTS Cryogenic & High-Pressure Corrosion Material Testing Solutions

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Cryogenic Applications

High-Pressure Corrosion Applications

Agenda

1. Introduction
2. Hydrogen Background
3. Main Hydrogen Applications
4. Hydrogen Storage
5. Expected Testing Requirements
6. Are main Applications Cryo or High Pressure?
7. Cryogenic Testing
8. High-Pressure Corrosive Testing
9. Integrated Top Industrie High-Pressure Corrosive Testing Solution
10. Extensometry
11. Q&A

Hydrogen Background

- Smallest & lightest Element on Earth: 50 l Gasoline >> 36 kg vs. 50 l Hydrogen >> 0.0041 kg

- Hydrogen can be a multi-purpose energy carrier to power hydrogen-based mobility and a medium to store and distribute
  - » Hydrogen used in fuel cells has bettter energy to weight ratio than lithium-ion batteries
  - » Rechargeable in a few minutes, similarly to gasoline vehicles

- Calorific values compared…

» Liquid Hydrogen T=20 K, -253,15 C° p = 1 bar	2.360 kWh/m³
» Petrol	9.200 kWh/m³
» Diesel	9.700 kWh/m³

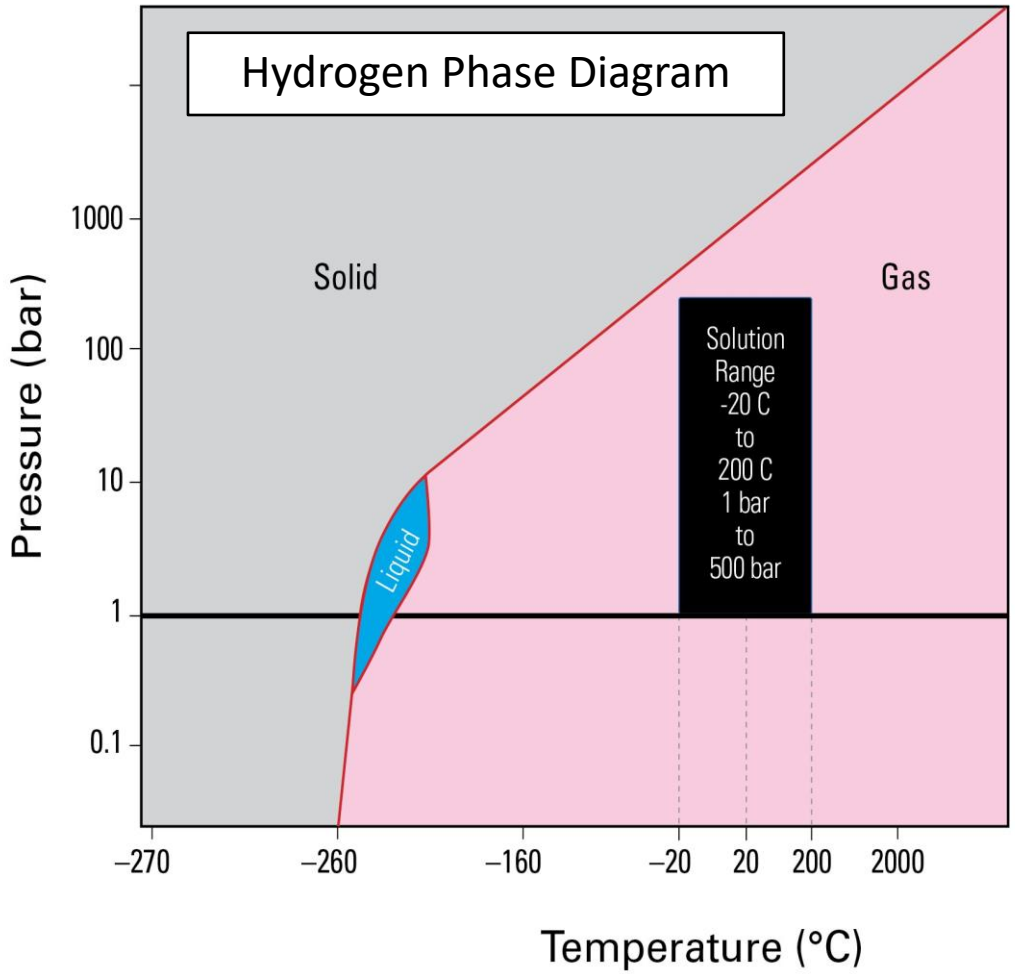
- Due to availability and technological challenges of H_2, battery electric has developed much faster and started to dominate the passenger car market

Main Hydrogen Applications

- Power to Gas:
  - Stationary storage of Hydrogen produced with renewable energy
  - Working pressure approx.. 50 bar to 200 bar at ambient temperatures
- Mobile Applications:
  - H2 powered cars, coaches and large trucks with fuel cells or combustion engines
  - Almost standard working pressure 700 bar at ambient temperatures
- Fueling Stations and H2 Shipping:
  - Possibly CHSS 1000 bar to 1200 bar but also LHSS expected
- H2 as aviation fuel:
  - Aviation will require LHSS at cryo temperatures with a high probaility
- Hydrogen powered cargo vessels and ferries
- Steel Industry
- Chemical Industry
- Gas Power Plants

Hydrogen Storage

- Storage:
  - » Compressed Hydrogen Storage Systems (CHSS)
    - Temperatures: -50 to 90°C
    - Pressure: up to 700 bar
  - » Liquified Hydrogen Storage Systems (LHSS)
    - Temperature: < -250°C
    - Pressure: 1 bar (near atmospheric pressure)
- Historic Material Test Standards do not cover the material behaviour under H_2 sufficiently.
- Extensions are discussed in international bodies…
- Currently this is still in transition from research to industrialization as has led to some H2 related inquiries



Expected Testing Requirements

- Requirements for material compatibility and hydrogen embrittlement of austenitic steel started with SAE H2 Compatibility Expert meeting in 2015
- Following material test related standards have been under discussion in the GTR-13* working group

  - **SSRT (slow strain rate tensile) and Fatigue tests**
    - Austenitic stainless steels — 228 ±5 K (-50°F/ -45°C)
    - Aluminum alloys — 293 ±5 K (70°F/20°C)
    - Other materials
      - Temperature where the material shows a minimum of tensile ductility in gaseous hydrogen
      - 228 to 363 K (193°F/90°C)
    - Our current research shows testing up to 1.5 x working pressure.

  - **HG-SCC (Humid Gas Stress Corrosion Cracking)** test method for aluminum (GTR13-3-02)
    - This may not be a high-pressure nor a cryo application

  - Aging tests for Composite materials (Not considered in this presentation)

Are main Applications Cryo or High Pressure?

- In most mobile Applications stored energy to weight/volume ratio is important
  - » Higher H2 densities are achievable through use of lower temperatures.
  - » Cryogenic H2 –Storage have been developed down to -253°C

But…

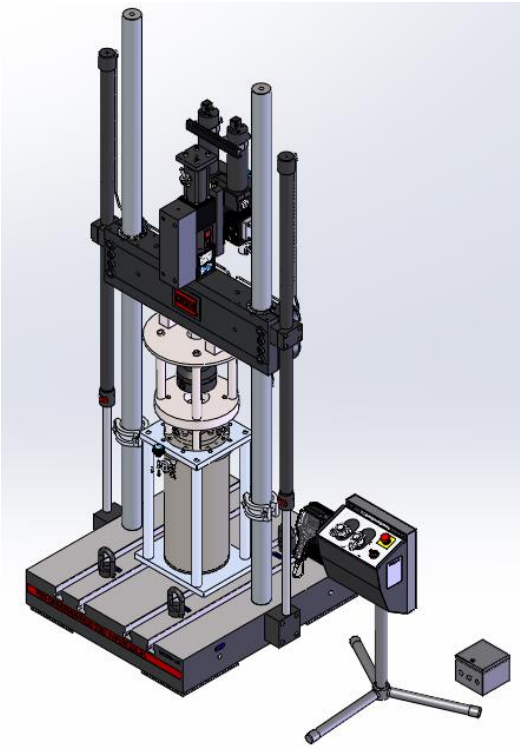
- Cooling uses as much as 45 percent of the stored energy content
- Need of significant insulation
- Complex and heavy tank technology reduces the effective gravimetric energy density

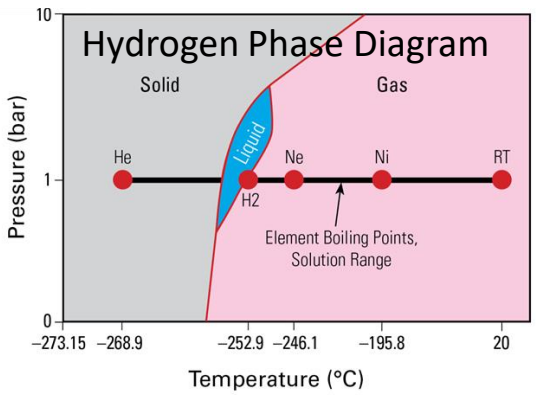
Assumption:

- Main applications will be “High Pressure”
  - » Around 700 bar
  - » Temperature Range from -50°C to 90°C
- GRV Industry has already set that as “almost” standard
- Aviation may be different…

Cryogenic Testing

- Industry Concerns
  - » Structural metals and composites
    - Strength, elastic modulus, ductility, fracture toughness, thermal conductivity, and thermal expansion
- Users
  - » Space Applications
  - » Energy Production and Distribution
  - » Mobility
- Qualifying and assessing materials under:
  - » Cryogenic Temperatures up to Room Temperature
    - Liquid Helium: -269°C (4K)
    - Liquid Nitrogen: -196°C (77°K)
    - (Liquid Hydrogen: -253°C (20°K))





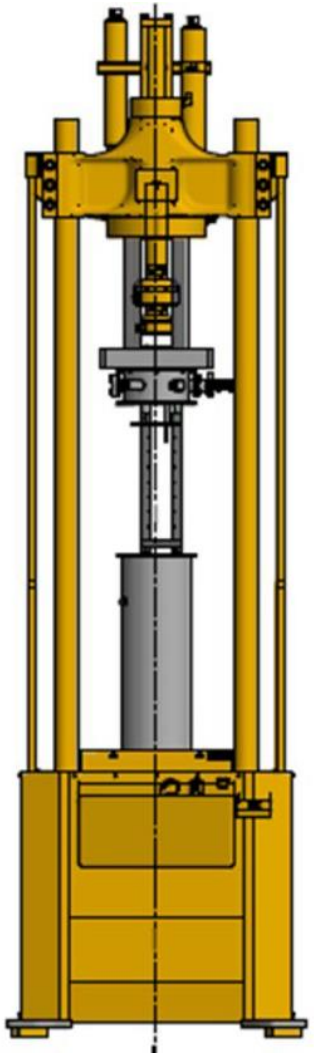
Cryogenic Testing: Material Characterization

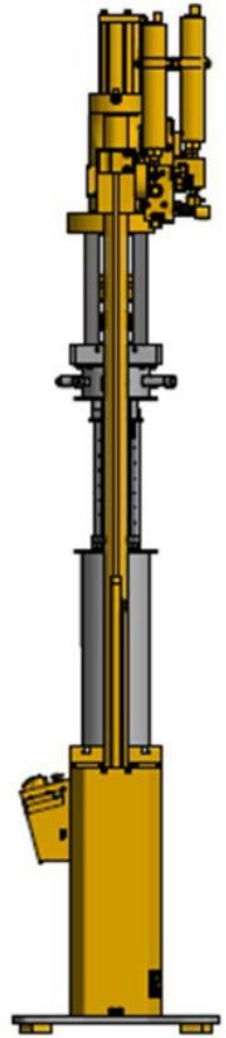
- Tensile testing
  - » Determine critical mechanical properties like: Yield strength, yield point elongation, tensile strength, elongation, and reduction of area
- Fatigue testing
  - » Determine fatigue properties of metals in the fatigue regime using test specimens subjected to uniaxial forces under low or cryogenic temperatures.
  - » Common test standards apply
- Fracture mechanics testing
  - » Determine mechanical properties like: Mode I fracture toughness & plane strain fracture toughness (KI_c), fatigue crack growth
  - » Common test standards apply
- Compression testing of composites

Cryogenic System: MTS Landmark

- MTS 370.100 or 370.25 frame sizes
- Crosshead mounted actuator and extended columns
- Integrated 100/250 kN Cryostats
- Hydrostatic Bearing optional
- Available as 100 kN or 250 kN Version

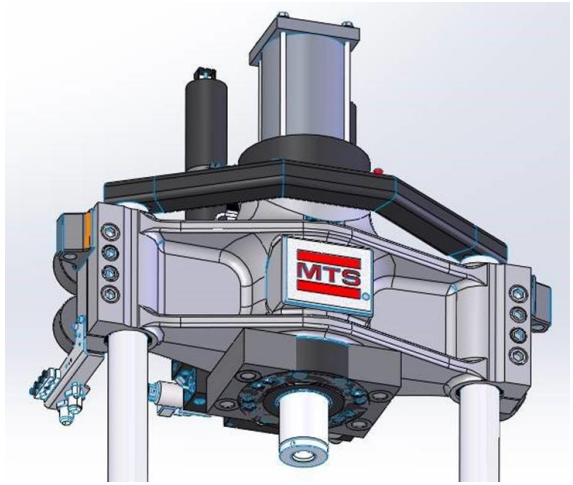
- 370.10 Base Frame
  - » Test height 1600 mm
  - » Test Width 533 mm
- 370.25 Base frame
  - » Test height 2174 mm
  - » Test Width 635 mm

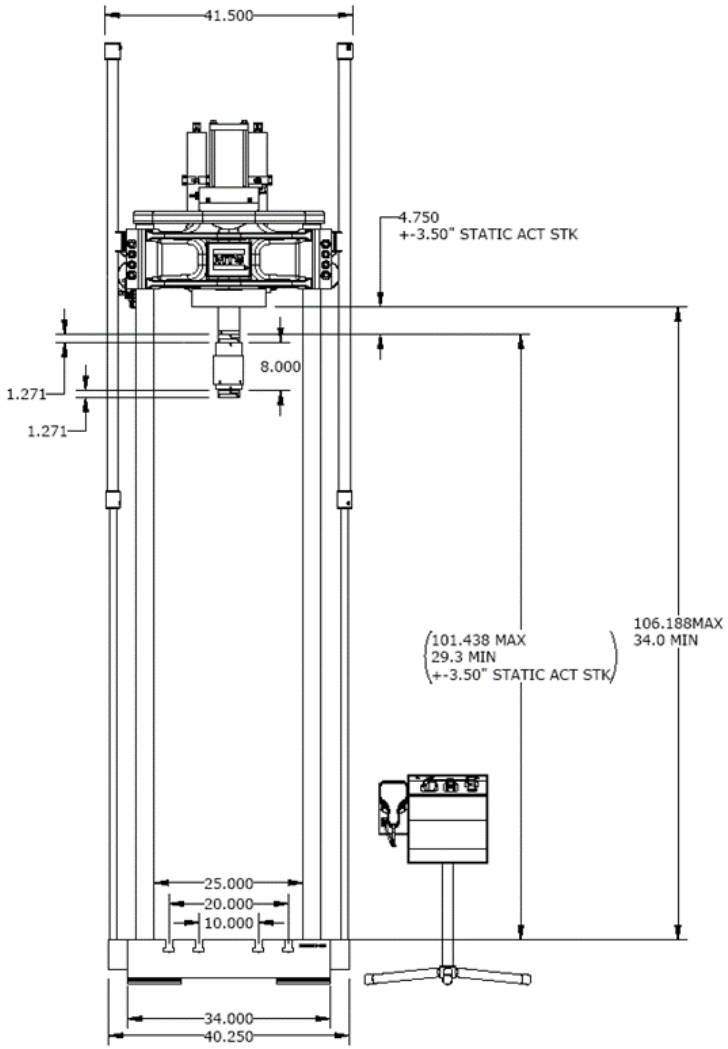




Cryogenic System: MTS 322 / Landmark Hybrid

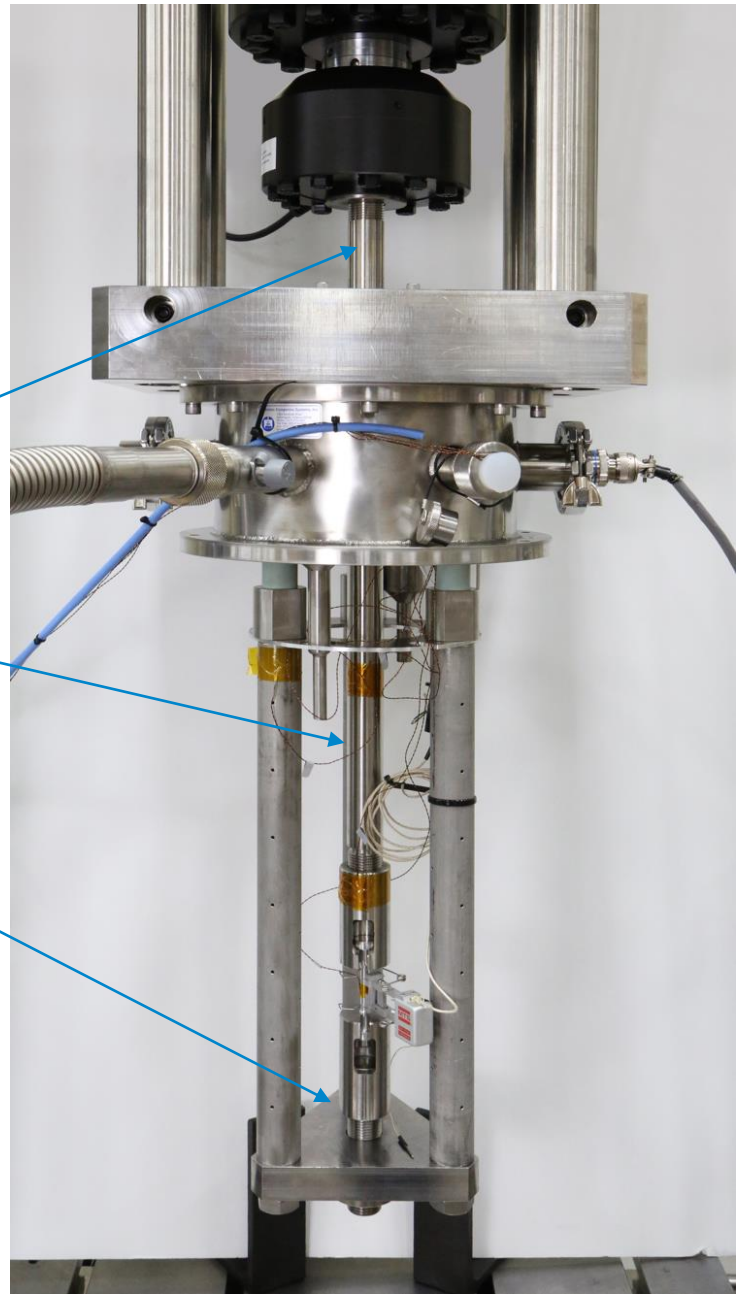
- Versatile MTS322 frame
- Using Landmark 100 kN or 250 kN Actuator
- Different T-Slot Options
- Different column position options
  - » Custom Test height
  - » Test Width 635 mm





Cryogenic System Detail

- Tensile Test Cryostat
  - 100 kN capacity self-reaction frame
    - 3 post structure
    - Sample space 115 mm dia x 430 mm length.
    - Threaded high strength stainless steel upper and lower pull rods
    - High strength lower reaction platen with concave spherical alignment seat
    - Lower pull-rod with spherical alignment nut
  - Specimen grips
  - X-Y alignment adjustment with laser alignment check



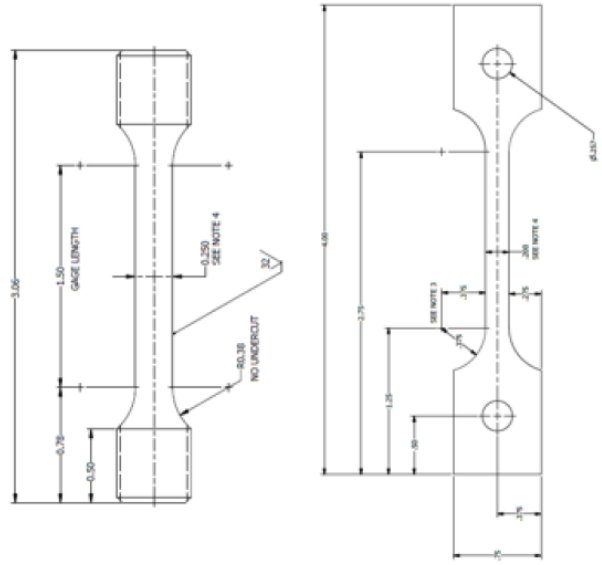
Cryogenic System Detail

- Tensile Test vs Reversed Cycle Fatigue Cryostat:
  - Tensile testing
    - Insulators are made of a composite material
  - Reversed Cycle Fatigue
    - Insulators are made of metal
    - Insulating less and requiring more cryogenic fluid to be fed
    - Increases the performance envelope to fatigue testing

Cryogenic Testing: Standards and Fixtures

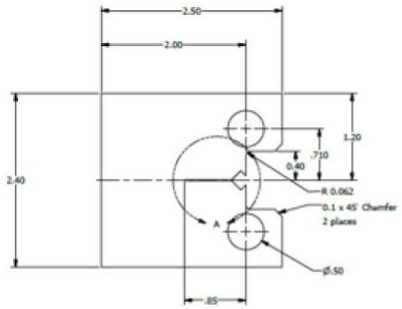
- Common Cryogenic Test Standards
  - » ISO (EN) 6892-3, ISO 6892-4 (Liquid Helium)
  - » ASTM E1450 (Liquid Helium)
  - » GB/T 228.3, GB/T 228.4 (Liquid Helium)

- Testing fixtures available for
  - » Tensile testing (round or flat specimen)
  - » Fully reversed fatigue testing (threaded or buttonhead)
  - » Fracture mechanics CT testing
  - » Compression testing (FRP composite specimen)

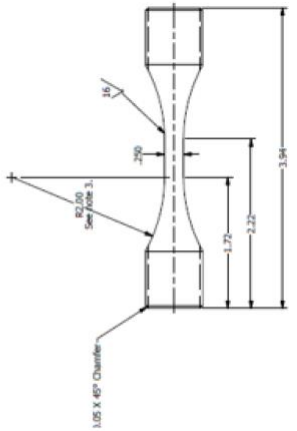


Round Tensile

Flat Tensile



Fracture Mechanics CT

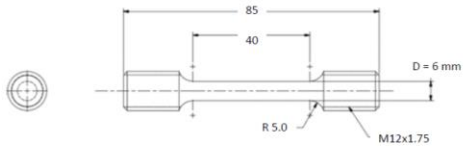


Round Fatigue

Gripping at Cryogenic Temperatures

Threaded Tensile Specimen Grips
Model MR-TTS Grips
Grips for low temperature tensile tests of cylindrical threaded tensile specimens

- High Strength Threaded grips, for specimens with M12 threaded ends and 6 mm dia and 40 mm Gage Length
- Custom designs available for other specimen, pull rod and cryostat specifications



Product	Part Number	Part Name	Description	Quantity
Threaded Tensile Specimen Grips, ASTM E8 and E1450	4013-5014-COM	THREADED TENSILE GRIP, FEMALE THD M27 to M12	Tensile grips for 6 mm dia gage dia tensile w M12 Thd ends	2

Model MR-BFG-50-13W
- Bolted-Friction Grips for low temperature tensile tests

- Versatile design for cryogenic tensile tests as a replacement solution for bulky wedge grips
- Typically used for low temperature tests at liquid nitrogen (77 K) or liquid helium (4 K)
- Can be used on flat metal (ASTM E8,E1450) FRP composites (ASTM D3039) and polymer tensile specimens (ASTM D638)
- Clevis Grip Gap = 40 mm, maximum allowable tabbed specimen thickness = 10 mm
- Custom design grips are available upon request

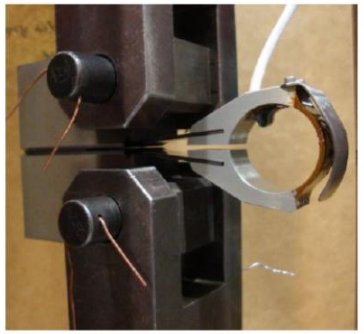
Used for FRP Composite tensile specimen geometries recommended in ASTM D3039

TABLE 2 Tensile Specimen Geometry Recommendations[A]

Fiber Orientation	Width, mm [in.]	Overall Length, mm [in.]	Thickness, mm [in.]	Tab Length, mm [in.]	Tab Thickness, mm [in.]	Tab Bevel Angle, °
0° unidirectional	15 [0.5]	250 [10.0]	1.0 [0.040]	50 [2.25]	1.5 [0.062]	7 or 90
90° unidirectional	25 [1.0]	175 [7.0]	2.0 [0.080]	25 [1.0]	1.6 [0.062]	90
balanced and symmetric	25 [1.0]	250 [10.0]	2.5 [0.100]	emery cloth	—	—
random/discontinuous	25 [1.0]	250 [10.0]	2.5 [0.100]	emery cloth	—	—

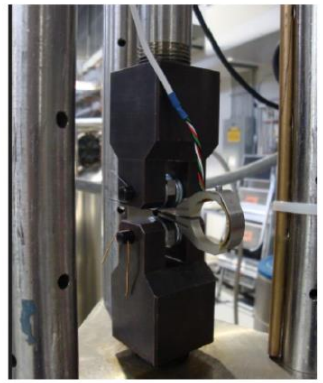
[A] Dimensions in this table and the tolerances of Fig. 2 or Fig. 3 are recommendations only and may be varied so long as the requirements of Table 1 are met.

Compact Tension Clevis Grips
- Fracture Mechanics Clevis Grips for Fatigue and Fracture



MR-CTCG-1.0 Grips with a MR-COD Gage

- **Model MR-CTCG-XX**
- Meet ASTM E399, E647 and E1820 specifications for testing compact tension specimens
- 3 types different types for 1.0, 0.75 and 0.75 scale CT specimens
- Precision machined from high strength Maraging Steel
- Custom design grips are available upon request

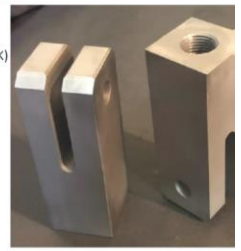


MR-CTCG-0.5 Grips with a MR-COD Gage

Gripping at Cryogenic Temperatures

Flat Tensile Specimen Clevis Grips

- Grip Model MR-FTSCG-6
- Typically used for low temperature tests at liquid nitrogen (77 K) helium (4 K)
- Grips and Pins are made of High Strength Maraging Steel
- Clevis Grip Gap = 6.5 mm, Pin Dia =
- Pull Rod female interface thread of M16x1.75
- Maximum load capacity = 75 kN
- Custom design grips are available upon request



COMPONENTS

Product	Part Number	Part Name	Description	Quantity
Flat Tensile Specimen Clevis Grips, ASTM E8 and E1450	4013-5581-COM	TENSILE CLEVIS GRIP, 6 mm gap	Clevis grip - flat tensile, 6x5 mm gage section	2
	4013-5574-COM	CLEVIS GRIP PIN, 6mm dia	Hardened steel clevis pins	2

RCF Grips –Specimen is bench mounted and then transferred to the machine

Reverse Cycle Fatigue Wedge Grips for flat metal specimens

Versatile grips can be used for;

- **Tensile Testing**
- **Tension-Tension Fatigue**
- **Fully Reverse Cycle Fatigue**

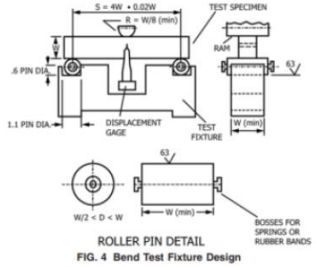
RCF Grip –shown here with curved wedge inserts for conduit tube fatigue testing for ITER

- Flat Specimen Axial RCF Grips
- Readily used as wedge grips for cryogenic tensile tests of flat specimens
- High strength steel grips accept flat specimens with grip sections up to 3 mm thick and 25 mm wide.
- Serrated face wedges are mechanically preloaded during set-up on bench
- The grips have axial alignment and zero backlash features to meet ASTM E466 and E606 specifications.

Three Point Bend Fixtures- designed to set on the self-aligning compression platen

- For Apparent Interlaminar Shear Strength (ILSS) of composites- A compact 3 pt bend fixture to enable measurements of Apparent Interlaminar Shear Strength (ILSS) for short beam shear specimens according to ASTM D2344
- For Fracture toughness and Fatigue Crack Growth Rate of Metals- Meet ASTM E399, E647 and E1820 specifications for testing 3 Pt bend specimens

ILSS Test Fixture (ASTM D2344



ROLLER PIN DETAIL

FIG. 4 Bend Test Fixture Design

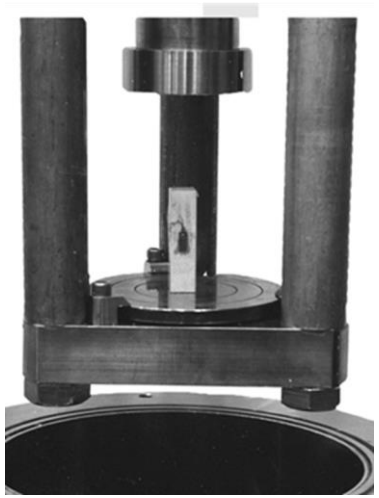
Fatigue and Fracture of Metals (ASTM E1820)

Gripping at Cryogenic Temperatures

Top view of lower platen and spherical bearing plate

Compression Test Platens designed for use with MR-100kn Reverse Cycle Fatigue Cryostat Model MR-CTP-100

- Compression platen can be used for cryogenic axial compression tests of metals, composites and polymers according to ASTM E9 and D695
- Clean surface platens provide an excellent stage for integral test fixtures such as;
  - 3 pt bend Interlaminar Shear of Composites
  - Modified IITRI and Celanese fixtures



Optional Testing at Intermediate Cryogenic Temperatures

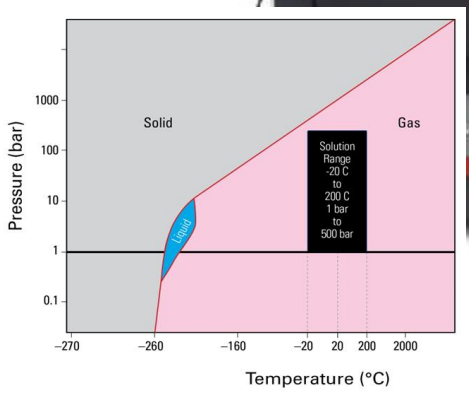
- The Cryogenic Temperature Control Option converts the systems into a variable temperature cryostat that enables testing at intermediate test temperatures in the range from 20 K to 300 K (± 1 K) on both the TTC or RCFC cryostats.

- Cooling media (typically liquid nitrogen or liquid helium) is used to reduce the cryostat chamber temperature

- The test temperature is obtained by heating the specimen with resistive heating elements
  - For low temperatures such as 20 K, a continuous supply of liquid helium provides the cooling media
  - For temperatures between 77 K and 200 K a static bath of liquid nitrogen in the bottom of the test dewar provides the cooling medium

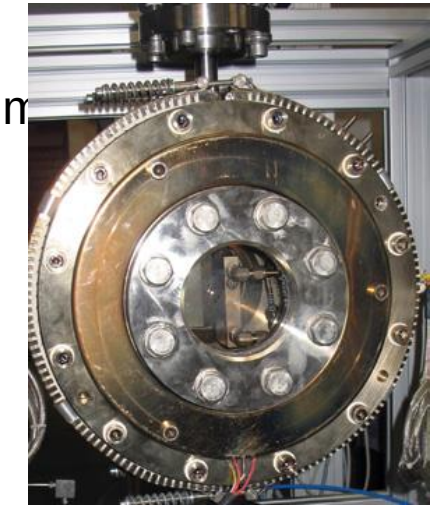
High-Pressure Corrosion Testing

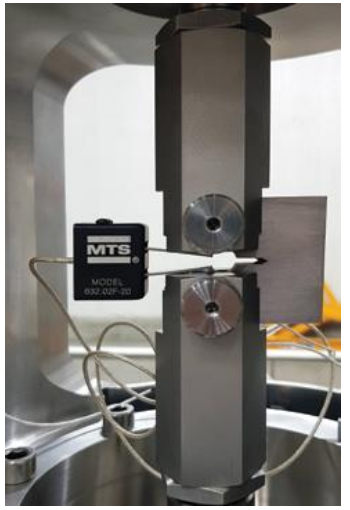
- Industry Concerns
  - » High pressure, high temperature environments increase possibility for gasses to be absorbed into materials to promote corrosion and environmental assisted cracking such as hydrogen embrittlement and stress corrosion
- Industry Users
  - » Geothermal or Nuclear Energy
  - » Gas and Oil
  - » Gas container manufacturers
- Qualifying and assessing metallic or composite materials under:
  - » Ambient & high-pressures
  - » Ambient & high-temperatures
  - » Corrosive gasses
    - Hydrogen, Methane, Carbon Dioxide, Mixed Gases



Integrated Top Industrie High-Pressure Corrosive Testing Solution

- Accommodates gases, fluids or liquids: gas H_2, CH_4, CO_2, m gases, syngas …
- Force Range
  - » 100 kN or 250 kN
- Pressure range:
  - » 1 to 500 bar for H_2 up to 1000 bar for other media
- Temperature range
  - » -20°C up to 180°C for H_2 up to 400°C for other media
- Internal volume capacities: 2L / 6L / 14L / 20L
- Orientation: Vertical or radial
- Specimen
  - » CT Specimen, Flat – pin loaded, Round - threaded





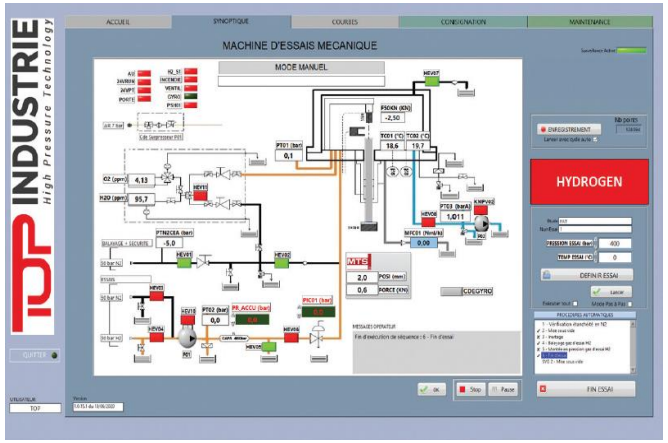
Integrated Top Industrie High-Pressure Corrosive Testing Solution

Integrated Top Industrie High-Pressure Corrosive Testing Solution

Landmark with Autoclave

Test Area Enclosure with ventilation

Grips / Fixtures & Extensometers



Supervisor & Data Acquisition System

- MTS Landmark test system with dual crossheads, hydraulic power supply, controller & application software
- Optional contacting & non-contacting (DIC) strain measurement solutions
- High pressure autoclave with gas (including Hydrogen) feeding and temperature control system
- Optional test area enclosure with hydrogen gas detection, ventilation, and emergency stop integration
- Grips / fixtures, crack monitoring options & safety systems integrated into MTS Landmark system

Extensometry

- A few recommendations and remarks:
- Our extensometers can be used in Hydrogen and we do make “Hydrogen hardened” models.
  - Choice of strain gages
  - Use of A286 parts
- As this environment is very unpredictable and damaging we sell them with no warranty
  - Commercially effective, extensometers used under these envirionments should be seen as consumables and spare examples should be available
  - We are able to repair extensomters
- Calibration
  - From our experience with high pressure and high temperature environments (including triax systems which go to 20000psi and above) is that the effects on our extensometers and load cells are negligible.
  - Gas in liquids (excitation down as bubbles will add noise to the signal, by isolation effects)
  - Consider that the units should be calibrated with less than 2 Volt excitation so the heating does not boil the liquid and create noise on the signal.

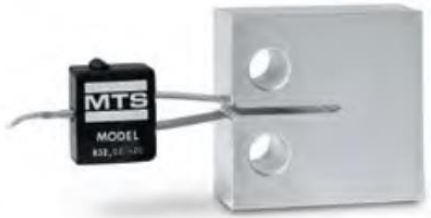
Extensometry

- Embrittlement
  - Use A286 parts
  - Avoid embrittlement
  - Maintain preload at cryo temperatures and yield at room temperature
- Hydrogen entrapment / Pressure drop
  - H2 permeates into glue and in any encapsulation under High Pressure
  - May blow the gage off when pressure relieved too fast (more or less anecdotical)
  - Our recommendation as lowest cost option for low temp liquid hydrogen testing are our low-temp-versions of COD gages and extensometers working from -269°C to +65°C
- ATEX
  - Until now there was no need for ATEX certification
  - Risk Assessment and Measures to be done by operator

MTS Measurement Equipment Offer

Our recommendation as lowest cost option for low temp liquid hydrogen testing are our low-temp-versions of COD gages and extensometers working from -269°C to +65°C

- Clip-On Displacement Gage 632.02F-21

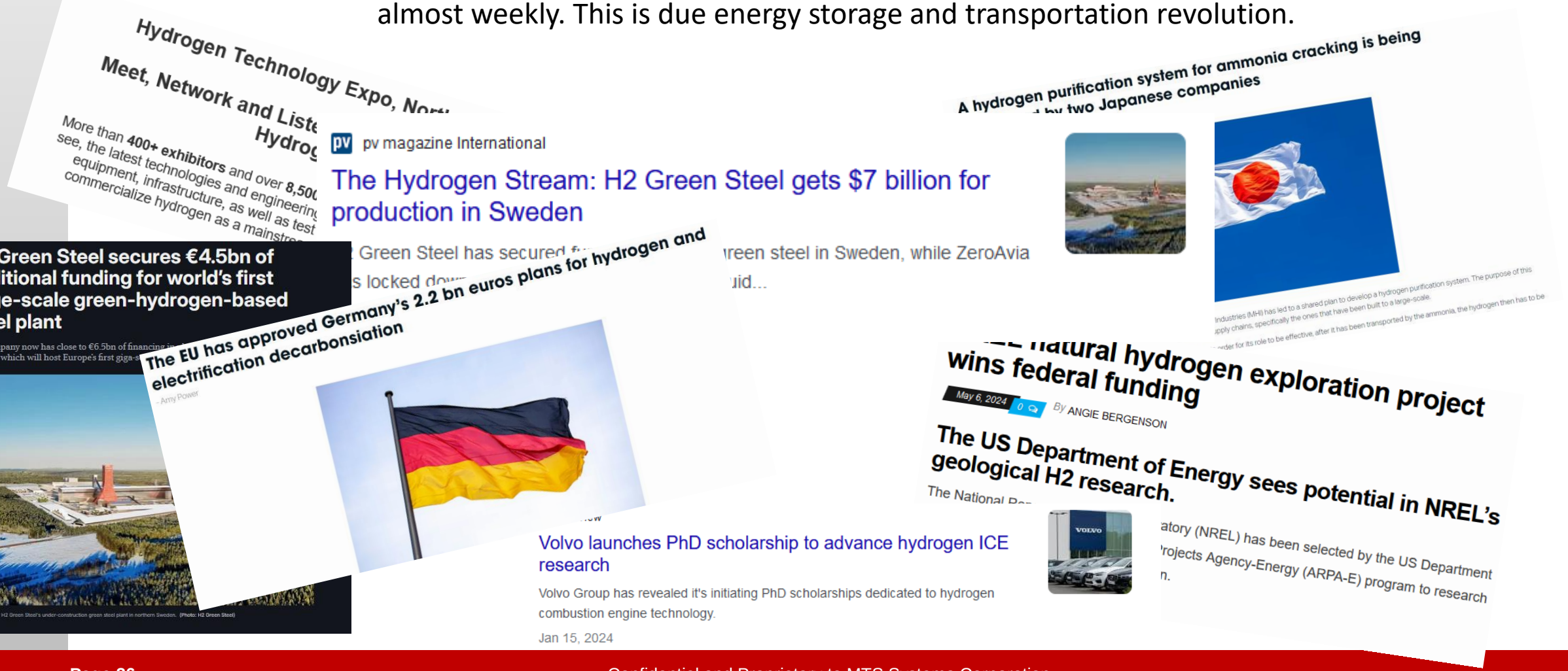


- MTS 634.11 Extensometer

Opportunities

Cryogenic Applications **High-Pressure Corrosion Applications**

Both markets are growing rapidly with billions of government and industry funding being announced worldwide almost weekly. This is due energy storage and transportation revolution.



Q & A

Questions?