Pipeline and Pressure Vessel R&D under the Hydrogen Regional Infrastructure Program In Pennsylvania

Kevin L. Klug, Ph.D.

25 September 2007

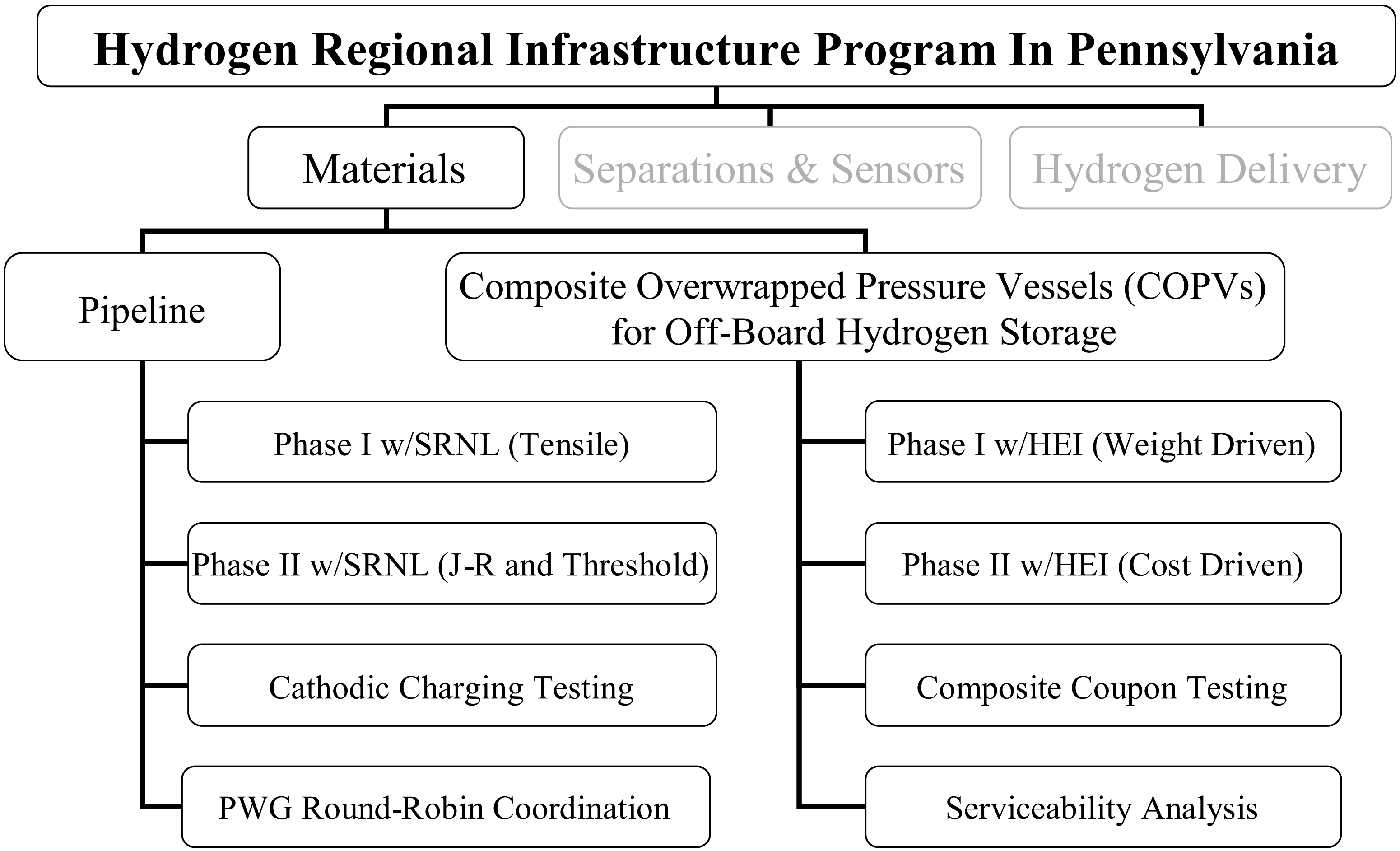
DOE Hydrogen Pipeline Working Group Meeting, Aiken, SC

Acknowledgments

- This material is based upon work supported by the Department of Energy under Award Number DE-FC36-04GO14229
- Partners
  - Savannah River National Laboratory (SRNL)
  - HyPerComp Engineering Inc. (HEI)
  - American Society Of Mechanical Engineers (ASME)
  - Pipeline Working Group (PWG)

Concurrent Technologies Corporation

Program Structure

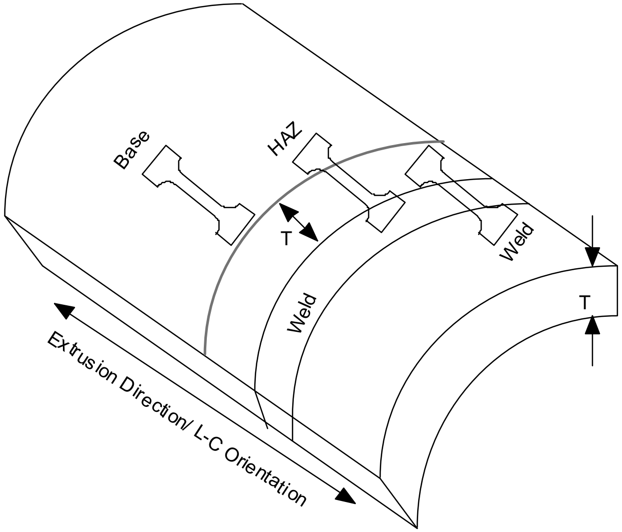


Pipeline and COPV data to be shared with PWG and ASME subject matter experts

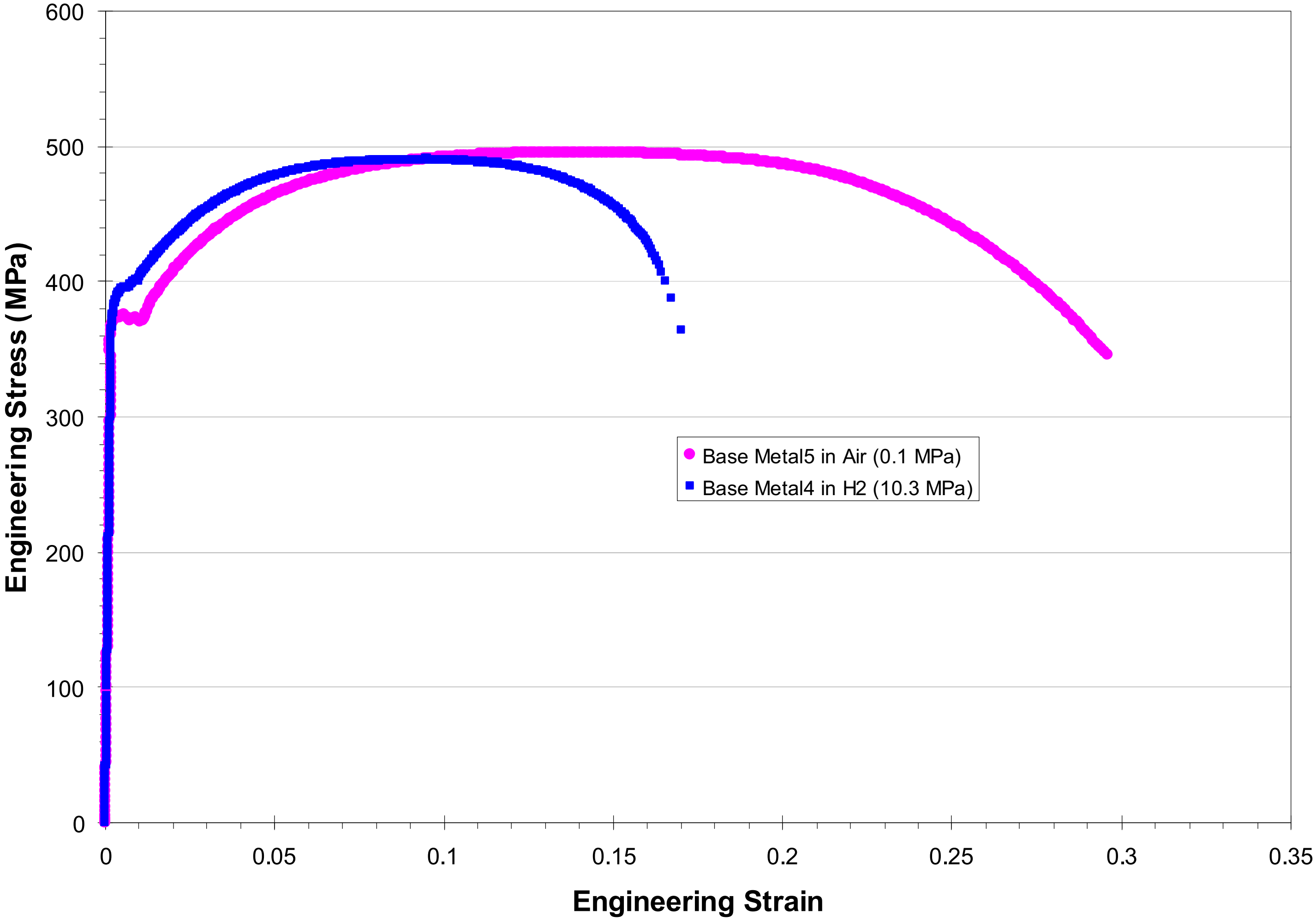
Concurrent Technologies Corporation

Pipeline Subtask – Phase I

- Alloy: 106 Grade B
- Multi-pass SMAW w/out stress relief
- Condition: base metal, weld and HAZ
- Orientation: L-C
- Atmosphere: 1500 psi H_2, ambient pressure Air
- Strain Rate: 10^{-4} /sec
- # of samples per matrix point: 6

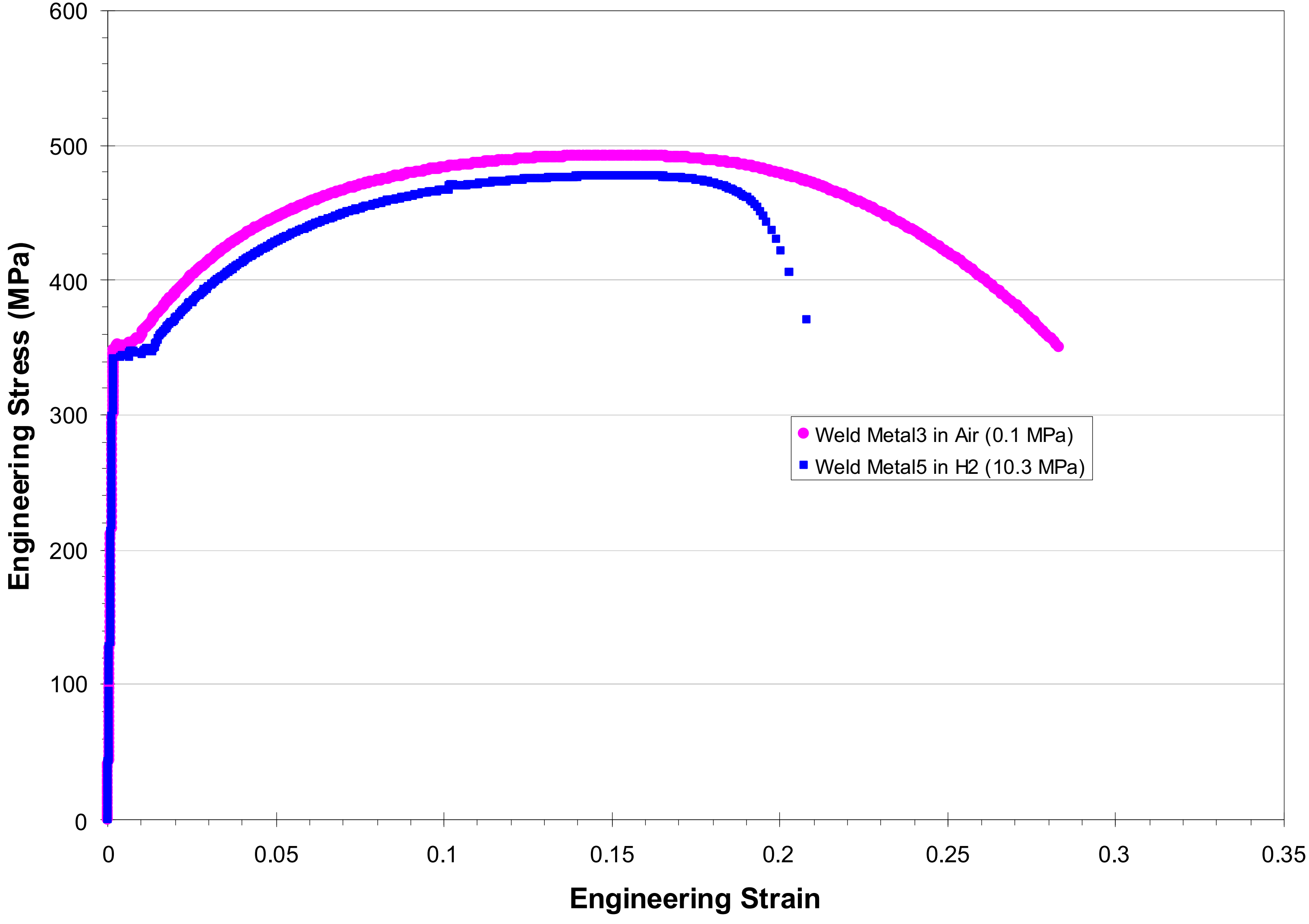


Phase I – Representative Result (Base Metal)



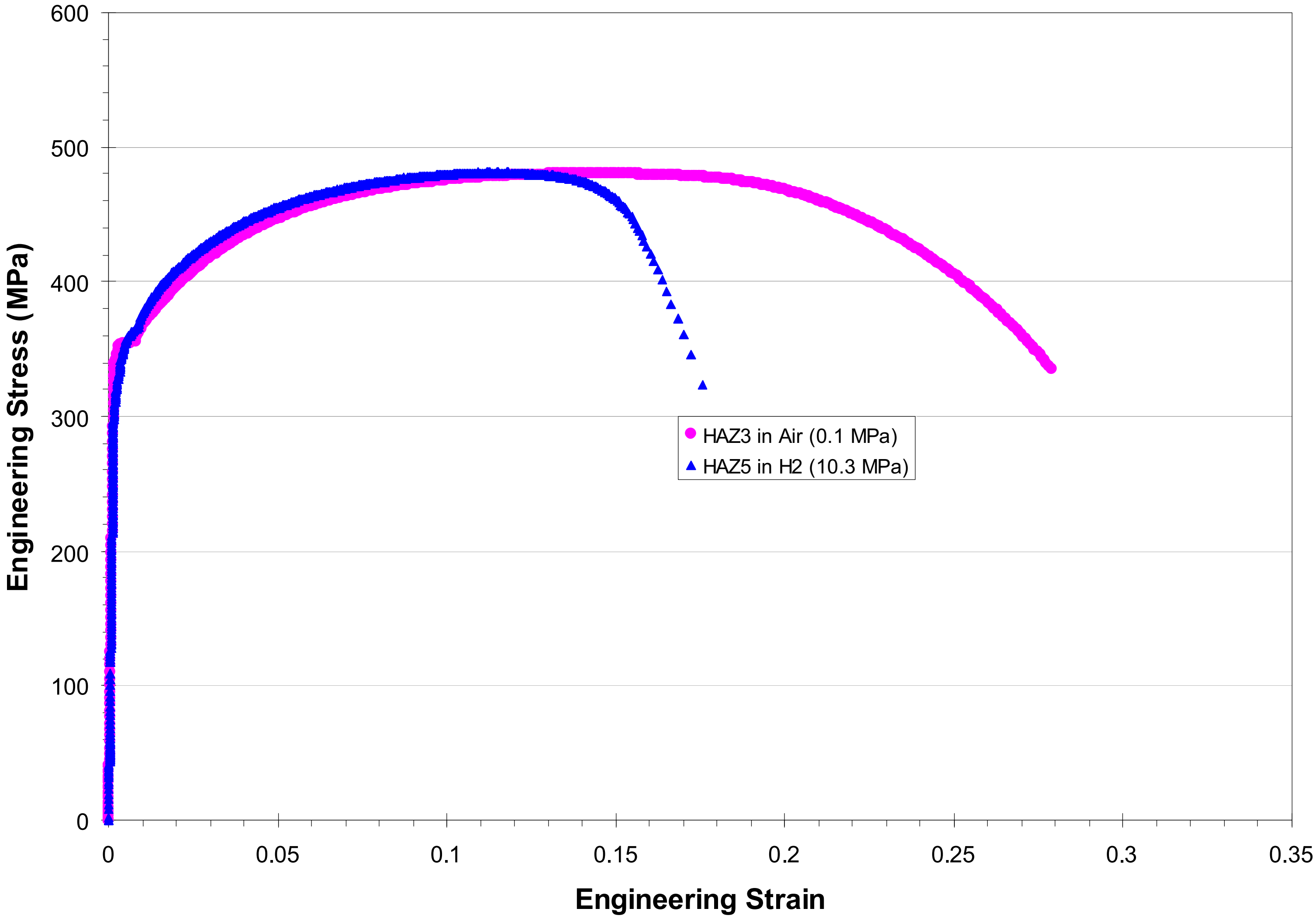
CTC
Concurrent Technologies Corporation

Phase I – Representative Result (Weld Metal)



Concurrent Technologies Corporation

Phase I – Representative Result (Heat Affected Zone)



Concurrent Technologies Corporation

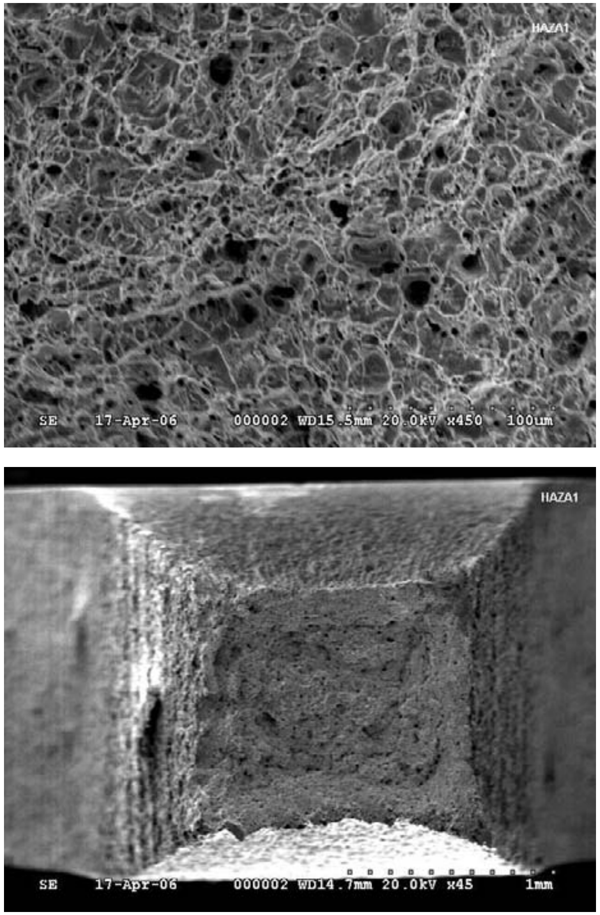
Phase I – Summary of Measurements

		Average Properties							
		0.2%Yield (MPa)	Dev.	UTS (MPa)	Dev.	Elong. at failure	Dev.	Reduction of Area (%)	Dev.
Base	Air	355.3	15.8	484.6	8.6	0.29	0.04	68.6	1.5
	H_2	357.1	32.9	486.4	17.8	0.19	0.04	30.8	5.0
Weld	Air	343.0	20.0	490.4	9.0	0.28	0.01	74.9	2.2
	H_2	350.0	16.1	480.9	12.0	0.21	0.02	30.5	6.4
HAZ	Air	349.3	20.8	482.3	7.6	0.27	0.04	71.0	1.7
	H_2	338.2	18.5	475.5	9.6	0.19	0.04	30.4	6.8

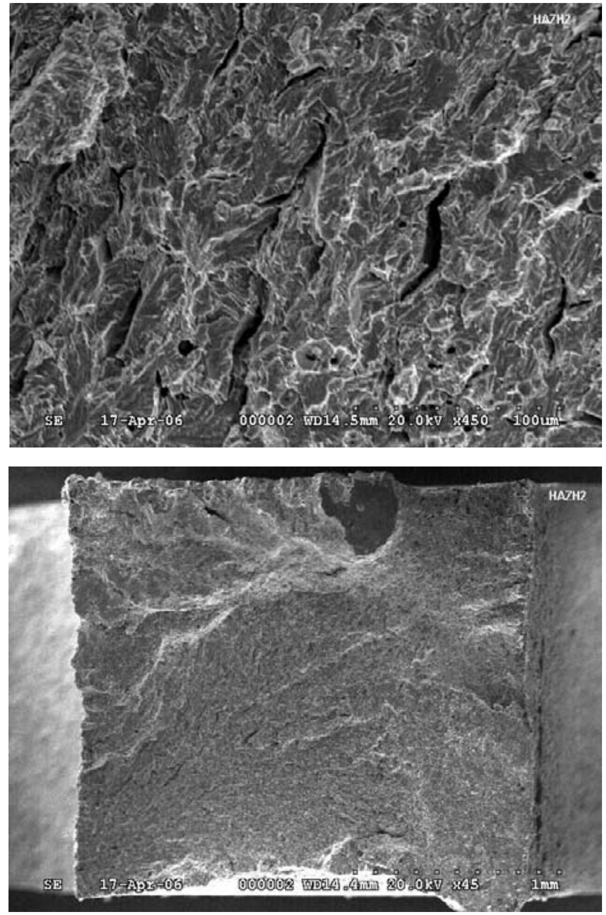
- Ductility reduced in 10.3 MPa hydrogen
  - The elongation to failure reduced ~30%
  - Reduction in area were reduced ~60%

CTC Concurrent Technologies Corporation

Phase I Fractography – HAZ Specimens



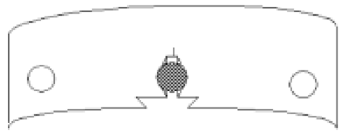
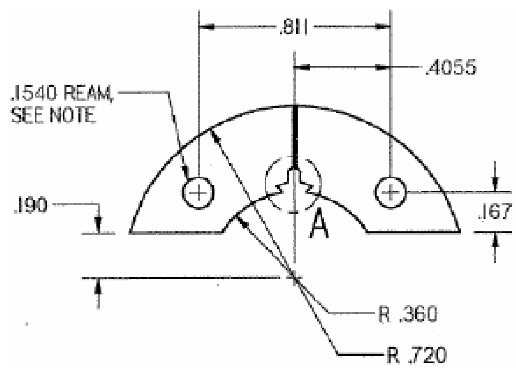
Air



Hydrogen

- Change in primary mode of fracture
  - Ductile rupture to quasi-cleavage.

Pipeline Subtask – Phase II

Specimen Geometry	Specimen Location	Number of Tests	Environment
Threshold Stress Intensity Testing			
 Note: Distance from load pins to crack ~ 0.5"	Base	2	Air
	Base	2	H_2
	HAZ	2	H_2
	Weld	2	H_2
Fracture Toughness Testing			
	Base	3	Air
	HAZ	3	Air
	Weld	3	Air
	Base	3	H_2
	HAZ	3	H_2
	Weld	3	H_2
All specimens C-R orientation, extracted from ASTM A106 (Grade B) 4.5″ O.D., 0.5″-thick pipe			

Concurrent Technologies Corporation

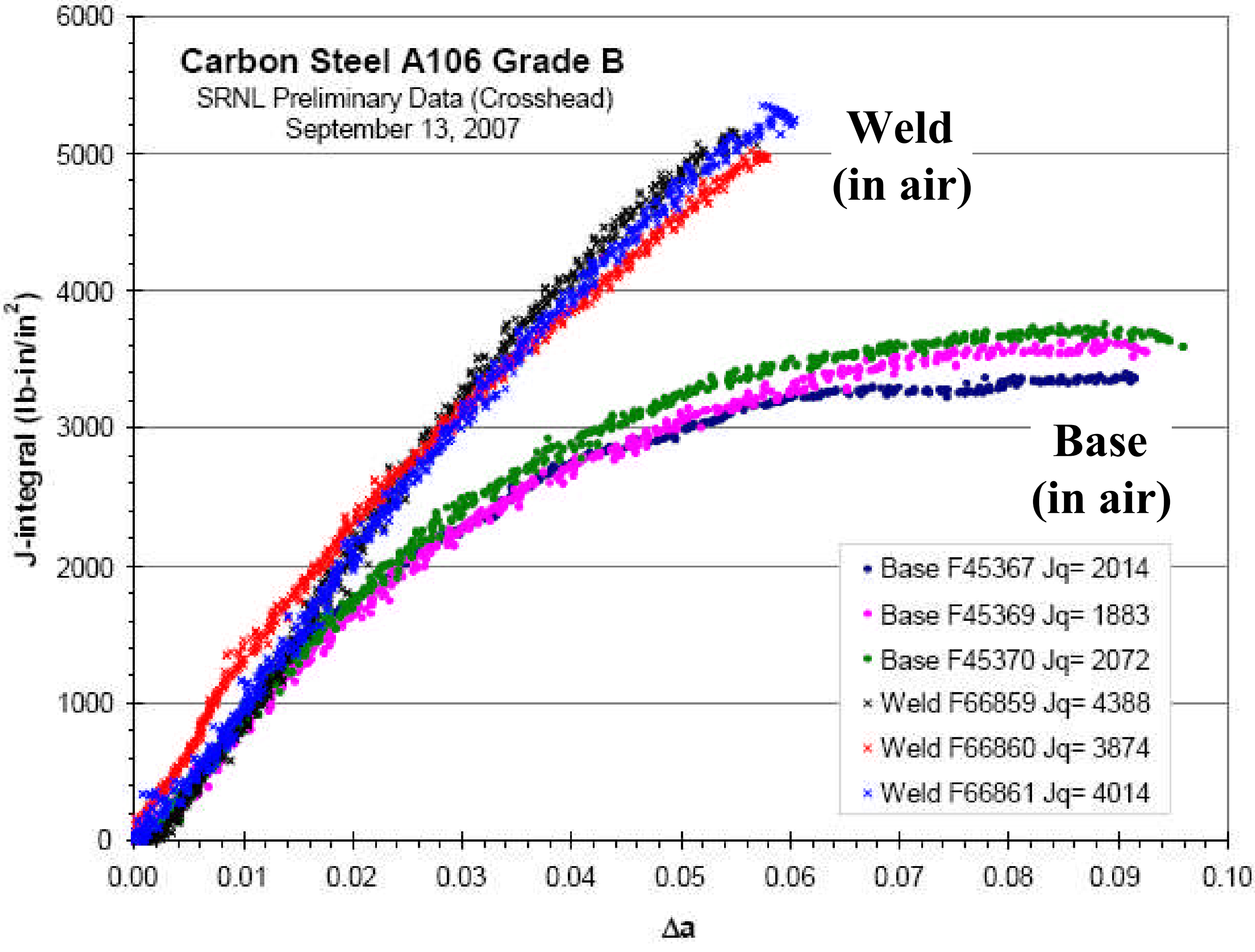
Pipeline Subtask – Phase II (Status)

Specimen Type	Location	Number of Specimens	Specimen Preparation: Machined?	Pre-cracked?	Side-grooved?	Testing at SRNL: Air/Hydrogen	Planned	Tested
J-R	Base	3	√	√	√	Air		√
	HAZ	3	√	√	√		√	
	Weld	3	√	√	√			√
	Base	3	√	√	√	Hydrogen	√	
	HAZ	3	√	√	√		√	
	Weld	3	√	√	√		√	
Threshold	Base	2	√	√	N/A	Air	√	
	Base	2	√	√	N/A	Hydrogen	√	
	HAZ	2	√	√	N/A		√	
	Weld	2	√	√	N/A		√	

- Status as of 19 September 2007
- All J-R tests are to be conducted at nominal room temperature and using ASTM E1820 as a guide
- All threshold tests are to be conducted at nominal room temperature and using ASTM E1681 as a guide
- All hydrogen testing to be conducted at nominal 1,500 psi pressure

Concurrent Technologies Corporation

Pipeline Subtask – Phase II (Initial J-R Test Results)



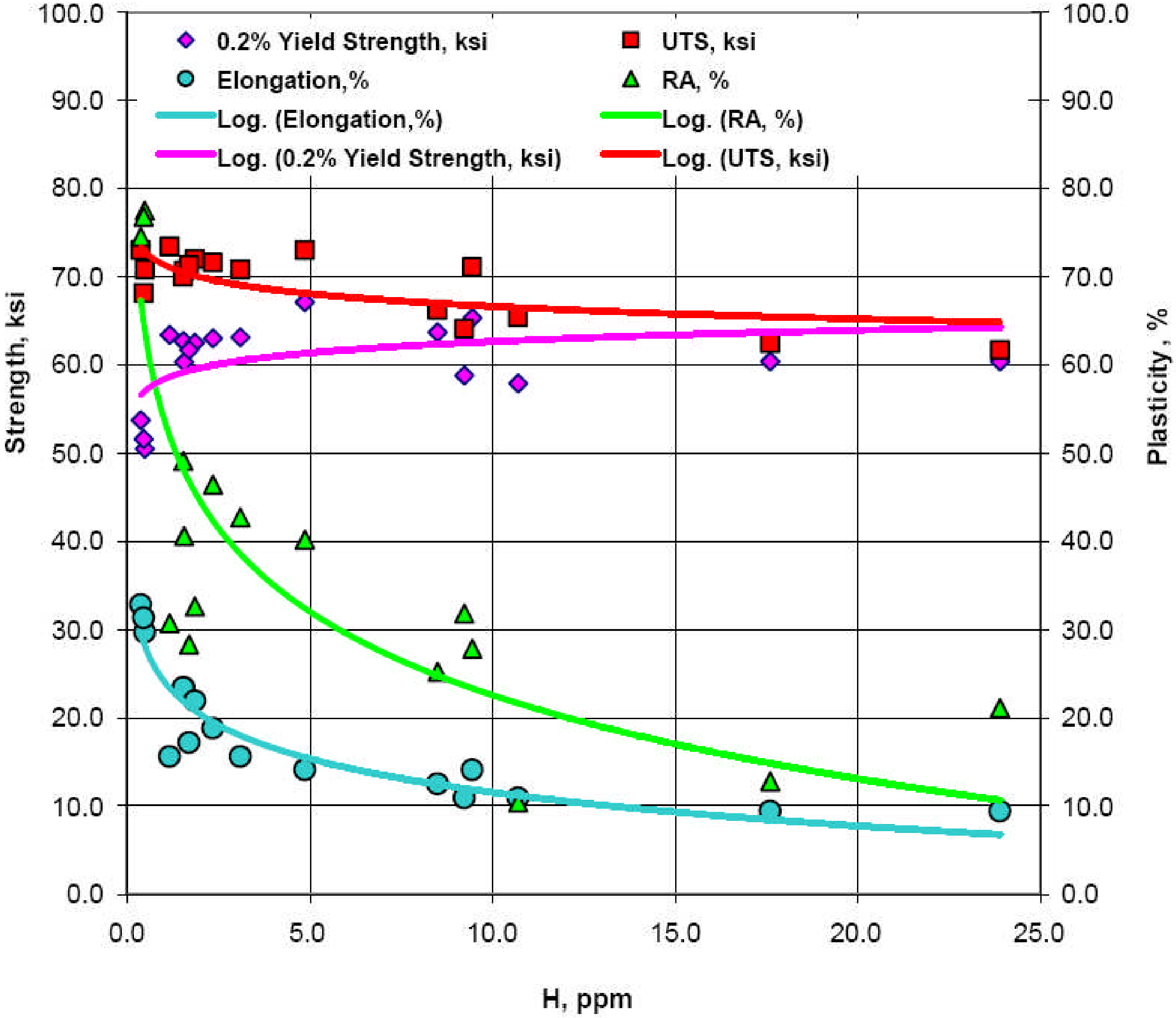
CTC
Concurrent
Technologies
Corporation

Pipeline Subtask – Cathodic Charging Effort

- Overview
  - Began as rapid data generator for numerical modeling efforts
  - Cathodic charging applied to base, HAZ and weld metal
  - Work being conducted at *CTC*'s Johnstown, PA facility
- Activities
  - Hydrogen concentration vs. charging time
  - Tensile properties vs. hydrogen concentration
  - Metallography and fractography
- Material
  - Description: 36″-long segment of longitudinally welded, 30″-diameter, 0.5″-thick X42 pipe
  - Specification: API 5L 42nd Ed. 07/01/00 PSL 2; API 5L 43rd Ed. 10/4/04 PSL 2; NACE MR0175
  - Procured from Petroleum Pipe & Supply Company (Pittsburgh, PA, USA)
  - Produced by Dura-Bond Pipe, LLC (Steelton, PA, USA)
  - Heat number 6104042; Heat code 801; Pipe number A1060

CTC
Concurrent Technologies Corporation

Pipeline Subtask –
Cathodic Charging Effort (Base Metal Results)



CTC Concurrent Technologies Corporation

PWG Round-Robin Testing (RRT)

- RRT framework determined at 22 August Pipeline Working Group (PWG) meeting at NIST-Boulder
- PWG prioritized test data needs, associated test standards, test conditions and tests for RRT
- Initial tests selected for RRT
  - Smooth Tensile
  - Permeation
- *CTC* volunteered to coordinate the RRT with participating laboratories and organizations; plan endorsed by PWG
- *CTC* will purchase pipeline material, machine and distribute specimens, collaborate with participants, summarize and publish RRT results

CTC Concurrent Technologies Corporation

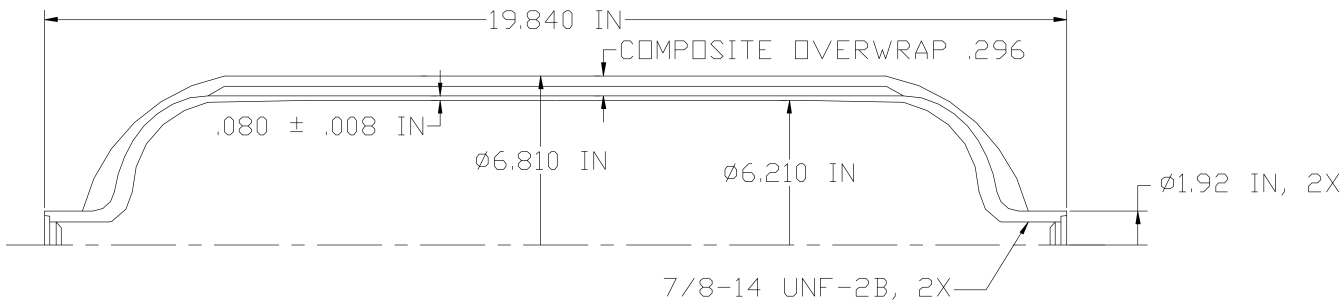
RRT - Organizations and POCs

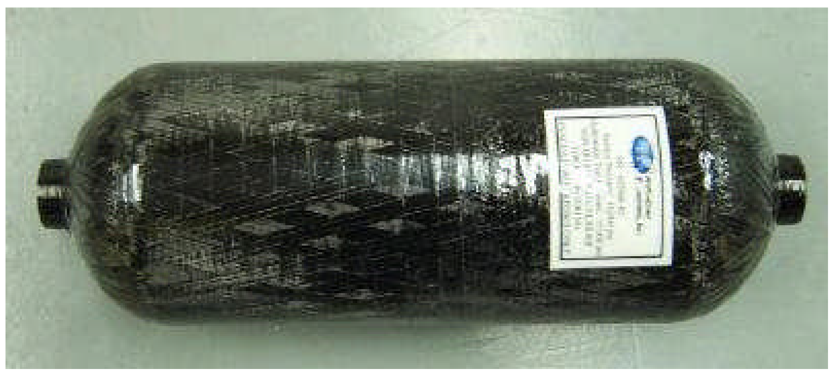
- Department of Energy (DOE)
  - Mark Paster and Tim Armstrong
- Concurrent Technologies Corporation (*CTC*)
  - Kevin Klug and Dave Moyer
- National Institute of Standards and Technology (NIST)
  - Dave McColskey and Rick Ricker
- Oak Ridge National Laboratory (ORNL)
  - Tim Armstrong, Zhili Feng, and G. Muralidharan (Murali)
- Sandia National Laboratory (SNL)
  - Brian Somerday
- Savannah River National Laboratory (SRNL)
  - Andrew Duncan and Thad Adams
- University of Illinois at Urbana-Champaign (UIUC)
  - Petros Sofronis

Concurrent Technologies Corporation

COPV Subtask – Phase I

- In Phase I, *CTC &* HEI developed a 10,000 psi service pressure, 7.5 liter Type III* COPV capable of nearly 26,000 psi with a hydrogen weight efficiency ratio of 5.2% (tank only)
  - Achieved with a non-optimized design
  - Weight (not cost) was primary goal at the time





* Type III COPVs utilized an aluminum liner and hoop and helical structural composite overwraps

CTC
Concurrent Technologies Corporation

COPV Subtask – Phase I (Results)

- Fabricated, (hydro) burst tested and fatigue tested (up to service pressure) twelve Type III COPVs
  - 7.75 liter water volume aluminum liner; 10,000 psi design pressure
  - Hoop and helical wrapped with carbon fiber
  - Designed to fail in sidewall
- Weight efficiency primary target – based on DOE goals at start of project*
  - 5.2% weight efficiency (tank only) achieved with non-optimized design
  - 0.035 kg of hydrogen per liter of storage volume
  - Tank cost $4,700/kg of stored hydrogen (note that cost reduction is primary focus of ongoing work)

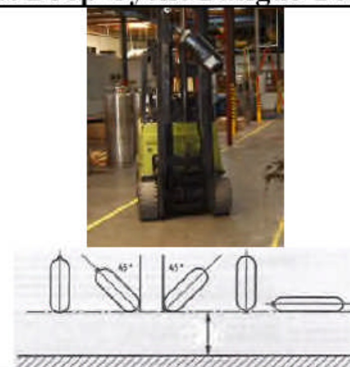
Burst Testing

COPV #	Burst Pressure (psi)
03280604	25,880
03080601	25,770
03220602	25,001
03270601	25,020
03280603	25,496
Avg. Burst Pressure	25,433
Standard Deviation	410.65
Coefficient of Variation	1.61%

Cyclic Fatigue Testing

COPV #	Cycles Achieved
03230601	3,161
03240601	3,466
03270602	3,047
Avg. Cycles Achieved	3,225
Standard Deviation	216.63
Coefficient of Variation	6.72%

Post Drop Cyclic Fatigue Testing



COPV #	Cycles Achieved after Drop
03270603	2,760
03280601	2,436
03280605	2,921
03290602	2,184
Avg. Cycles Achieved	2,575
Standard Deviation	329.72
Coefficient of Variation	12.80%

* DOE Multi-Year Research, Development and Demonstration Plan—Hydrogen Delivery (Revision 1, 2005; Table 3.2.2)

COPV Subtask – Phase II

- Primary focus = cost reduction
- Pursue Type II COPV instead of Type III used in Phase I
- 15 liter/34CrMo4/COTS SCUBA vessel (rated to 3,400 psi) used as proof-of-design liner
- T700 hoop wrap only (i.e., no helical wrap)
- Reduced COPV service pressure (6,700 psi vs. 10,000 for Phase I)

Off-Board Gaseous Hydrogen Storage Tanks (for forecourts, terminals, or other off-board storage needs)			
Category	2005 Status	FY2010	FY2015
Storage Tank Purchased Capital Cost ($/kg of H2 stored)*	$820	$500	$300
Volumetric Capacity (kg H2 /liter of storage volume)**	0.023	0.030	>0.035

* Storage Tank Capital Cost: These costs are based on the H2A Components Model V1.1. The model uses a current cost of $820 per kg of hydrogen stored for a 1,500 kg/day Forecourt station. This is based on quotes from vendors for steel tanks capable of 6,250 psi working pressure. The 2015 target cost is set to achieve the overall delivery cost objectives.

** Forecourt Storage Volumetric Capacity: The 2005 value is based on the specific volume of hydrogen at room temperature and 6,250 psi. The 2015 target is based on the specific volume of hydrogen at room temperature and approximately 12,000 psi. Off-board storage tank technology could use carriers as opposed to or in addition to compressed hydrogen as a means to store hydrogen. The most important target is system capital cost. However, the footprint for the storage must also be taken into consideration where space is limited such as at forecourts. For this reason, it is assumed that the hydrogen volumetric content of the storage volume should be at least as high as for 10,000 psi hydrogen gas.

From DOE Multi-Year Research, Development and Demonstration Plan—Hydrogen Delivery (Revision February 6, 2007; Table 3.2.2)

CTC® Concurrent Technologies Corporation

COPV Subtask – Phase II (Results)

Type II COPV Test Results	
Burst Testing	
Serial Number	**Burst pressure (psi)**
061907-01	15,955
062507-03	15,193
062107-03	15,944
062507-02	15,468
mean	**15,640**
standard deviation	375
Cycle Testing	
Max pressure = 8,375 psi (1.25 X service pressure); ~ 4 cycles/minute	
Serial Number	**Number of cycles to leakage**
062107-05	11,799
062507-01	5,174
062607-05	9,600
062607-06	9,642
mean	**9,054**
standard deviation	2,783
Drop Cycle Testing	
Max pressure = 8,375 psi (1.25 X service pressure); ~ 4 cycles/minute	
Serial Number	**Number of cycles to leakage**
062107-01	8,223
062507-04	6,532
062607-04	8,831
062607-07	7,335
mean	**7,730**
standard deviation	1,008

- Estimated price = $641/kg H2; volume efficiency = 0.029 kg of H2/L of storage volume; weight efficiency (tank only) = 2.47%

COPV Subtask – Composite Coupon Testing

- HEI supplied composite test plates composed of same material system used in Phase II COPVs
- Tensile and fatigue testing of specimens from plates
  - Feed data into serviceability analysis to improve COPV design
- *CTC* leads: Dr. V. Shkolnikov and G. Hostetter
- Test vendor: Axel Products, Inc. (Ann Arbor, MI)

Test mode		Fiber orientation	Load direction	Sought properties	Specimens	
					Configuration*	#
Mechanical	Tensile	Unidirectional	11, 22	σ_{U11}, E_{11}, υ_{12}, ε_{11} σ_{U22}, E_{22}, υ_{21}, ε_{22}	A 0.1 × 1 × 10"	5 + 5
	Shear	Unidirectional	12, 13, 23	τ_{12}, G_{12}, τ_{13}, G_{13}, τ_{23}, G_{23}	B 0.18 × 0.75 × 3"	5+5+5
	Tensile	Quasi-isotropic 87, -87, 17, -17	11	σ_{U22}, E_{22}, υ_{21}, ε_{22}	C	5
Thermal	Expansion	Unidirectional	11, 22, 33	CTE	E	5

*) Specimen configurations shall correspond to ASTM standard requirements, specifically: "A" - dog bone specimens corresponding to ASTM D3039, "B" – bars as per ASTM D5379; "C" beyond the existing standards, D and E to be determined with regard to ASTM C177, E228, WK3997.

Test #	Test categories	Rate s^{-1}	Frequency Hz	Stress range $\sigma_{min}/\sigma_{max}$	Stress level $\sigma_{max}/\sigma_{ult}$	Temperature*	Specimens**
1		100				T_0	3
2						T_1	3
3		10				T_0	3
4	High strain rate***					T_1	3
5		1				T_0	3
6						T_1	3
7		0.1				T_0	3
8						T_1	3
High strain rate sub-total							24
9						T_{-1}	3
10	Short-term	0.01				T_0	3
11						T_1	3
12						T_2	3
Short-term sub-total							12
13						T_{-1}	3
14			0.1	0.1	0.9	T_0	3
15						T_1	3
16						T_2	3
17			0.1	0.1	0.8	T_0	3
18	Cyclic					T_1	3
19						T_{-1}	3
20			0.1	0.1	0.7	T_0	3
21						T_1	3
22						T_2	3
23			0.05	0.1	0.8	T_0	3
24	Sustained				0.8	T_0	3
25						T_1	3
Fatigue sub-total							39
Total							75

*) Temperature levels being selected shall correspond to anticipated operational conditions and test lab capability; those presumed to be: T_{-1} = 0°C (32°F), T_0 = 20°C (68°F), T_1 = 40°C (104°F), T_2 = 60°C (140°F), until otherwise is specified

**) 3 is the minimal acceptable number of specimens; more than 3 specimens per a test mode is preferred

***) Secondary priority

Concurrent Technologies Corporation

COPV Subtask – Serviceability Analysis

- MATLAB-based analysis developed by Dr. Vladimir Shkolnikov
- Predicts COPV mechanical response based on:
  - Exposure to variable force-temperature conditions over a tank's service life
  - Fatigue properties of the composite used in the tank structure
- Technique is capable of providing sound design knock-downs and safety factors to improve COPV design
- Technique described in "Serviceability Characterization of Composite Storage Tanks" presented at 2007 ASME Pressure Vessels and Piping Division Conference, July 22-26, 2007, San Antonio, TX

CTC® Concurrent Technologies Corporation

Summary

- **Hydrogen Regional Infrastructure Program In Pennsylvania**
  - Materials
    - Pipeline
      - Phase I w/SRNL (Tensile)
      - Phase II w/SRNL (J-R and Threshold)
      - Cathodic Charging Testing
      - PWG Round-Robin Coordination
    - Composite Overwrapped Pressure Vessels (COPVs) for Off-Board Hydrogen Storage
      - Phase I w/HEI (Weight Driven)
      - Phase II w/HEI (Cost Driven)
      - Composite Coupon Testing
      - Serviceability Analysis
  - Separations & Sensors
  - Hydrogen Delivery

Pipeline and COPV data to be shared with PWG and ASME subject matter experts