Materials Testing in Hydrogen

National Institute of Standard and Technology
Boulder, Colorado USA

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PHMSA
Pipeline Transportation of Emerging Fuels and R&D Forum
Interagency Panel
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NIST Material Measurement Laboratory (MML)

- NIST's mission is to **promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life**.

- MML Mission Statement: **“The MML strives to establish relationships that result in advances with broad scientific impact in chemical, biological and materials measurements, and maintain NIST's longstanding role and reputation as a neutral partner.”**

Project Outline

Measurements

- Tensile
- Fracture
- Fatigue
  - FCGR
  - Strain-life
- Microscopy

Modeling

- Pipeline/Pressure Vessel Lifetime
- Code Modification
- Physics-based Fracture and Fatigue Crack Growth Rate Model

Science

- Advanced Correlative Fractography and Metallography
- Neutron and Synchrotron X-ray Experiments
- Tomography
- Deuterium Embrittlement

Project Impact

Measurements

- DOT
  - HAZMAT
  - PHMSA
- DOE
- ASME
- Praxair
- Air Liquide
- Mannesmann
- Europipe
- Emerson/Micromotion
- EVRAZ

Modeling

- ASME B31.12 Code Modifications
  - Pipeline code
- ISO 11114-4
  - Pressure vessel code

Science

- Acta Materialia
- Materials Science and Engineering A
- Review of Scientific Instruments
- Corrosion Science
- International Journal of Fatigue
- Applied Physics Reviews

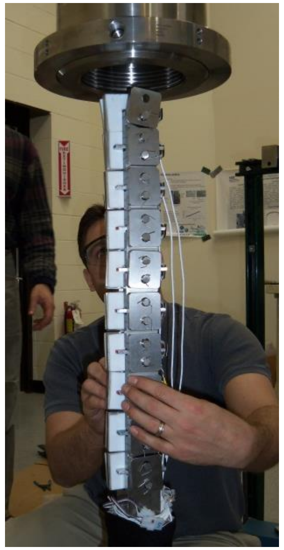
Investments: Laboratory

- **Location:**
  - Tucked under Boulder's Flatirons in the foothills of the Rocky Mountains
- **History:**
  - Materials testing in hydrogen began in 2012
  - **$2M investment for lab + equipment**
  - **$8.5M investment since start of project**
- **Safety:**
  - Control room electronics separate from testing facility
  - Testing facility can be monitored remotely

Investments: Testing Facility

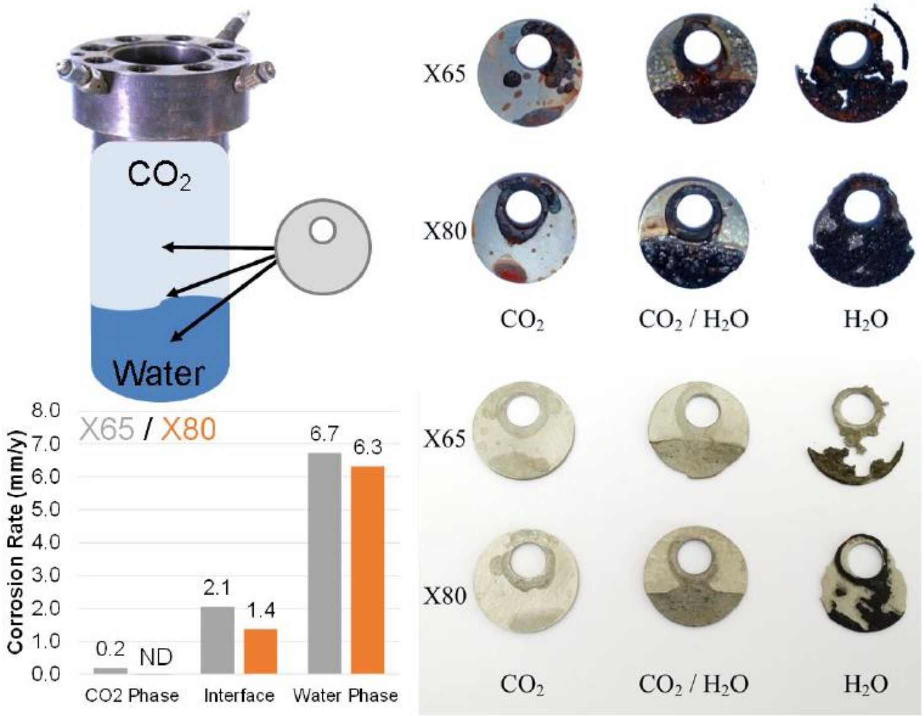
	Equipment			
	Load Frame 1	Load Frame 2	Pressure Vessel 1	Pressure Vessel 2
Capacity	100 kN (22 kip)	250 kN (55 kip)	138 MPa, 20 ksi	34 MPa, 5 ksi
Features	Strain-life	8 simultaneous FCGR tests	-	-

	Test Control	
	Strain rate	Frequency
Tensile	10^{-6} s^{-1}	-
Fatigue	10^{-6} s^{-1}	0.01 - 10 Hz, Fully-reversed



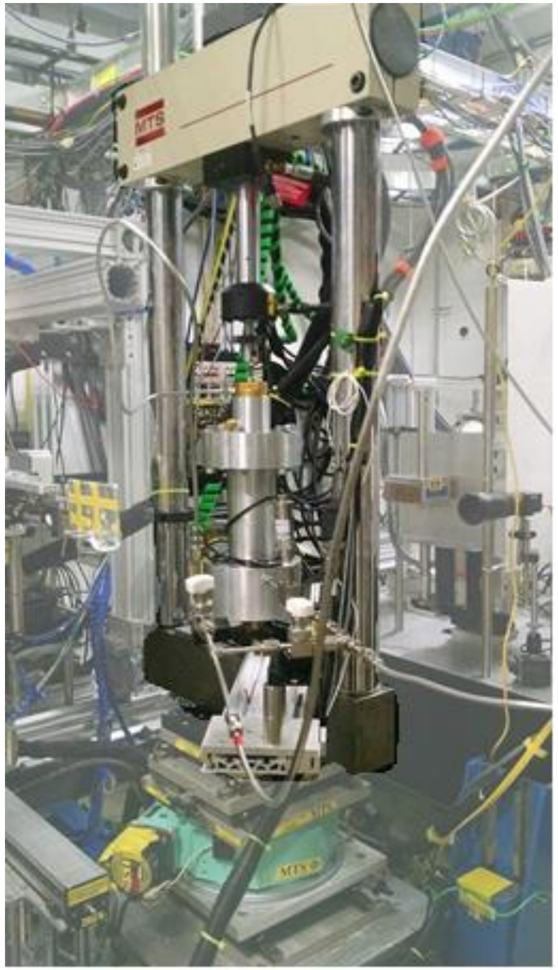
Supercritical CO_2

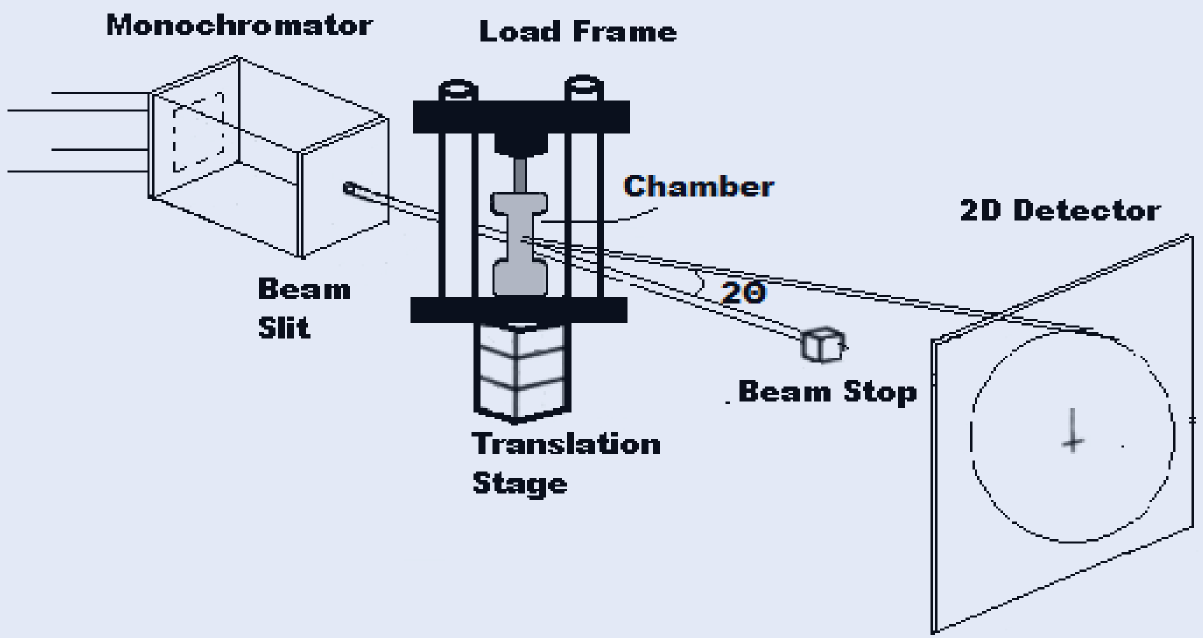
Figure 1: Photos of the Supercritical CO_2 Corrosion Test Facility showing the walk-in fume hood setup (left) and the interior lab layout (right).



Clark, Brandi N., et al. "Preliminary Results from the NIST Supercritical Carbon Dioxide Corrosion Test Facility∗." *CORROSION 2017*. OnePetro, 2017.

Investments: Chamber for Neutron and High-energy X-ray Diffraction





Experimental setup at the APS 1-ID beamline. The sample chamber was mounted in a hydraulically-driven load frame positioned in the beam on a translation stage.

Key on-going work

Measurements

- Tensile
- Fracture
- Fatigue
  - FCGR
  - Strain-life
- Microscopy

Modeling

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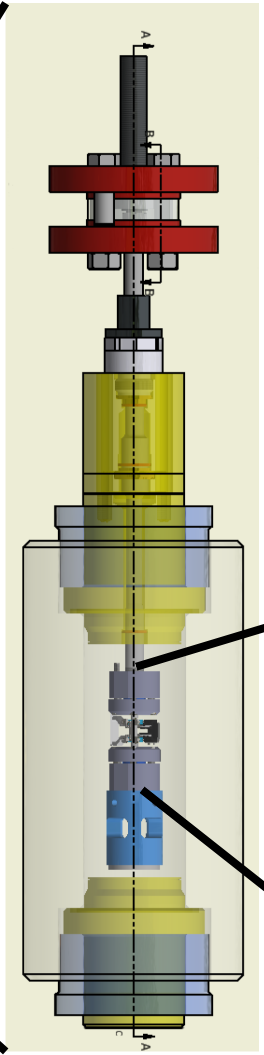
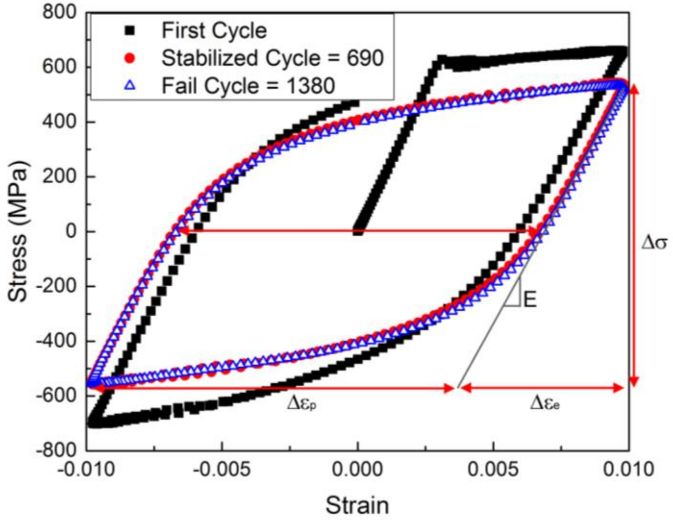
Fully-reversed Strain-life Testing in H_2 Gas

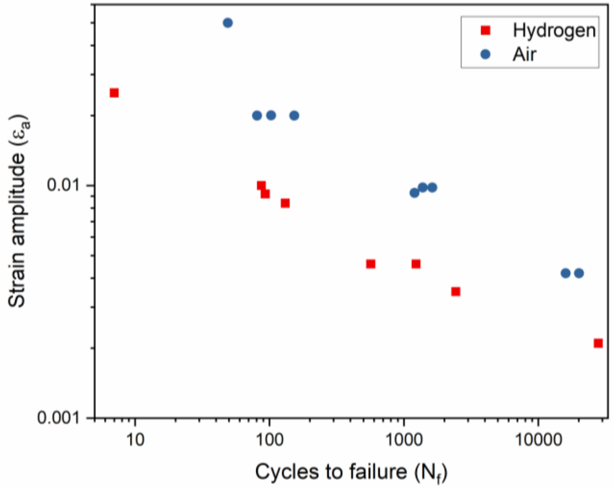


- *Only facility* capable of fully-reversed strain-life testing in H_2 gas on round-bar specimens due to testing difficulty

- Provides huge amount of information compared to stress-life, fatigue, etc.

- Results show drastic differences in plastic and elastic strain damage in H_2



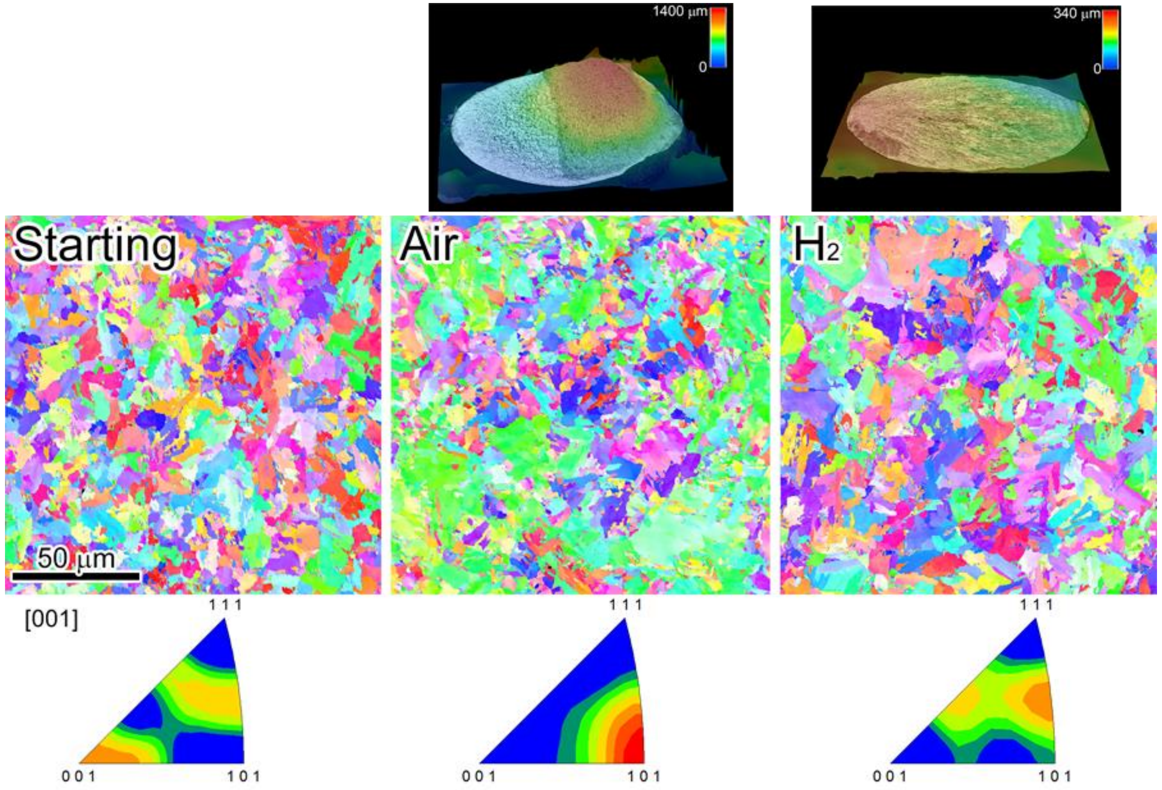


Correlative Fractography: Understanding hydrogen's influence on fracture and deformation

Fractography ⟷ **Metallography**

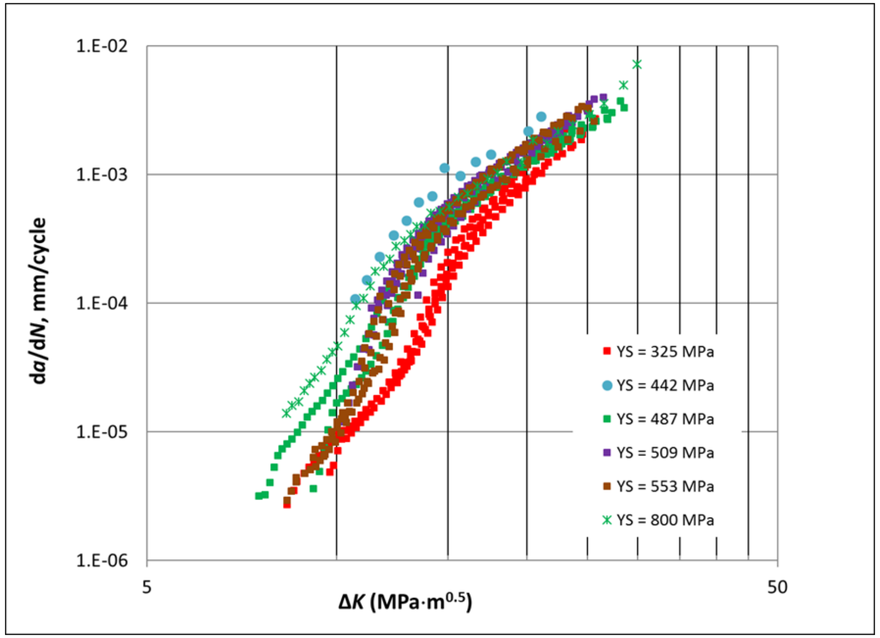
Damage Mechanism

- Example: Strain-life testing of pressure vessel steel
  - **Fracture features** are more brittle with H_2
    → different fracture process
  - **Microstructure** shows **less rearrangement** with H_2
    → H_2 impacts deformation processes



Success: Modifications to ASME B31.12 Pipeline code

- Fatigue crack growth rate (FCGR) is increased in H_2 environment compared to air
- H susceptibility in fatigue does not have same correlation with strength as under tensile loading



- Data collected for ASME B31.12 code modification
  - 10 air tests
  - 74 H_2 tests

ASME B31.12-1600 psi:
Current Code:
t=0.272” (Sch 30) for X52
t=0.255” (Sch 30) for X80
N/A @ X100

*H_f=1
t=0.272” (Sch 30) @ X52
t=0.177” (Sch 20) @ X80
t=0.133” (Sch 20)@ X100

29,000 pounds/mile of steel saved from Sch 20 to Sch 30

Success: Modifications to ASME B31.12 Pipeline code

- Fatigue crack growth rate (FCGR) enhanced in H_2 environment compared to air
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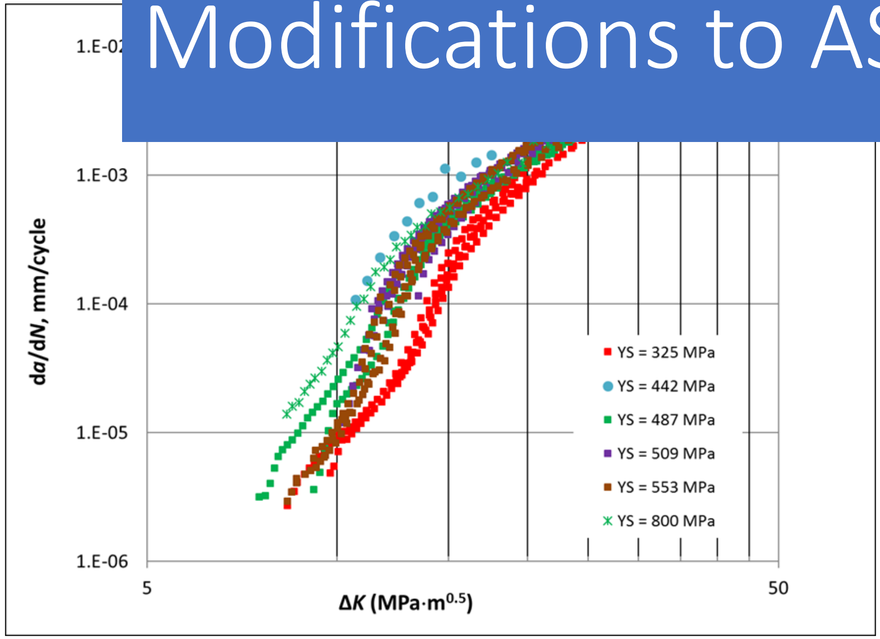
Modifications to ASME B31.12 save 29,000 lbs/mile



N/A @ X100

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29,000 pounds/mile of steel saved from Sch 20 to Sch 30

Success: Unification of ISO 11114-4 Tests for Hydrogen Pressure Vessels

Cumulative Damage Parameter (Air)											
		TO									
		Burst Disk	CT-LC	CT-DC	Tensile	IS 0.038%	IS 0.08%	IS 0.1%	IS 0.375%	IS 0.5%	
FROM	Burst Disk	1	0.11	0.02	0.11	2.90E-08	1.43E-04	4.01E-04	0.01	0.03	MULTIPLY BY
	CT-LC	9.47	1	0.19	1.00	2.75E-07	1.36E-03	3.80E-03	0.08	0.30	
	CT-DC	48.90	5.17	1	5.14	1.42E-06	7.01E-03	0.02	0.43	1.56	
	Tensile	9.51	1.00	0.19	1	2.76E-07	1.36E-03	3.82E-03	0.08	0.30	
	IS 0.038%	3.44E+07	3.64E+06	7.04E+05	3.62E+06	1	4.93E+03	1.38E+04	3.03E+05	1.10E+06	
	IS 0.08%	6.98E+03	737.26	142.72	734.07	2.03E-04	1	2.80	61.43	223.10	
	IS 0.1%	2.49E+03	263.21	50.96	262.07	7.24E-05	0.36	1	21.93	79.65	
	IS 0.375%	113.63	12.00	2.32	11.95	3.30E-06	0.02	0.05	1	3.63	
	IS 0.5%	31.29	3.30	0.64	3.29	9.09E-07	4.48E-03	0.01	0.28	1	

CT-LC: compact tension specimen, load-controlled test
CT-DC: compact tension specimen, displacement-controlled test
IS: in-service condition

Success: Unification of ISO 11114-4 Tests for Hydrogen Pressure Vessels

Cumulative Damage Parameter (Air)

		TO									
		Burst Disk	CT-LC	CT-DC	Tensile	IS 0.038%	IS 0.08%	IS 0.1%	IS 0.375%	IS 0.5%	
FROM	Burst Disk	1	0.11	0.02	0.11	2.90E-08	1.43E-04	4.01E-04	0.01	0.03	MULTIPLY BY
	CT-LC	9.47	1	0.19	1.00	2.75E-07	1.36E-03	3.80E-03	0.08	0.30	
	CT-DC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1.56	
	Tensile	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	0.30	
	IS 0.038%	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	.10E+06	
	IS 0.08%	6.98E+03	737.26	142.72	734.07	2.03E-04	1	2.80	61.43	223.10	
	IS 0.1%	2.49E+03	263.21	50.96	262.07	7.24E-05	0.36	1	21.93	79.65	
	IS 0.375%	113.63	12.00	2.32	11.95	3.30E-06	0.02	0.05	1	3.63	
	IS 0.5%	31.29	3.30	0.64	3.29	9.09E-07	4.48E-03	0.01	0.28	1	

Modeling to Unify Tests under ISO 11114-4

CT-LC: compact tension specimen, load-controlled test
CT-DC: compact tension specimen, displacement-controlled test
IS: in-service condition

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