Short term creep of P91 in CO_2 environment utilizing confocal microscopy

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NATIONAL ENERGY TECHNOLOGY LABORATORY

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Solutions for Today | Options for Tomorrow

U.S. DEPARTMENT OF ENERGY

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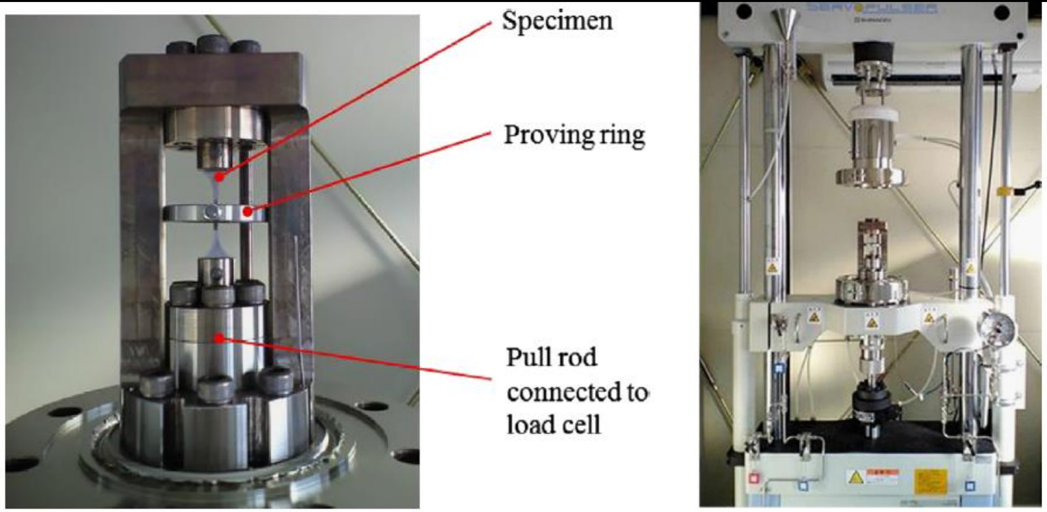
DISCLAIMER

Need for In-situ Strain Measurements

- **Problem: Difficult to measure strain in controlled environments at high temperature or pressure**
- **Existing environmental chambers have limited strain observation capabilities**
  - **Baffles prevent strain observation and spring rate must be accounted for when measuring load, I.E. Kubota et al.**
  - **Inferred from load cages, I.E. Saengas et al.**
- **Commercial systems not sealed, limited to 450-550^{o}C, (MTS)**
- **Typical In-situ strain observations are done in SEM under vacuum or low vacuum I.E. environmental SEM**

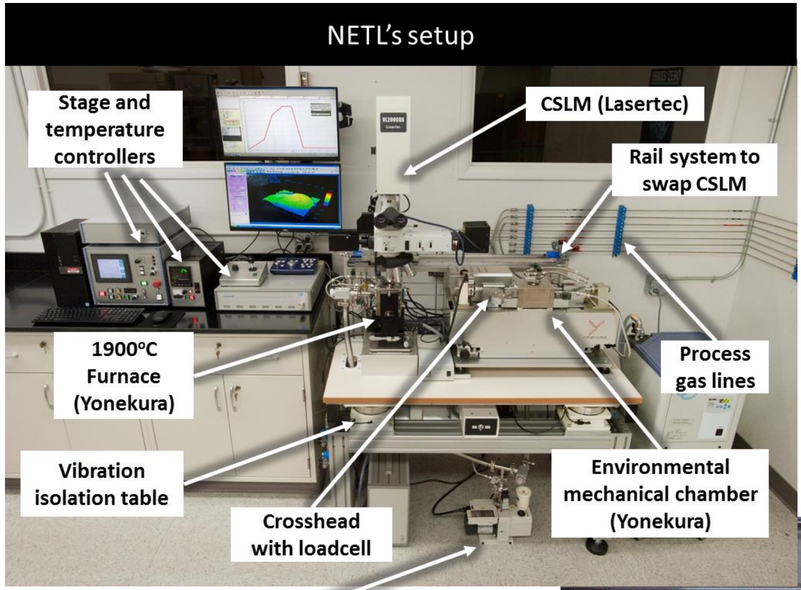
High pressure hydrogen chamber – Kubota et al.

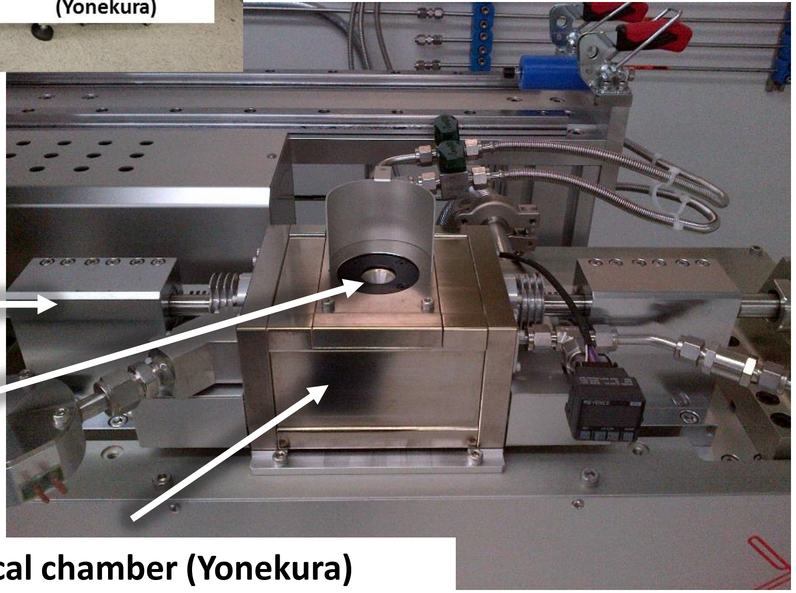
Elevated temperature / pressure autoclave – Saengas et al.



Confocal Scanning Laser Microscope (CSLM)

- CSLM mounted on rail system
  - Allows for easy access to both very high-temperature stage and mechanical stage
  - 405 nm laser
  - 2X to 10X objective lens with digital zoom capability
- Load stage inside sealed environmental mechanical chamber with quartz viewing window
  - Process gas or vacuum
  - Heating by focusing dual halogen lamps
  - 5000 N load stage
  - Max strain rate 20 mm/min
- CSLM allows for strain measurement at high temperatures in controlled environments



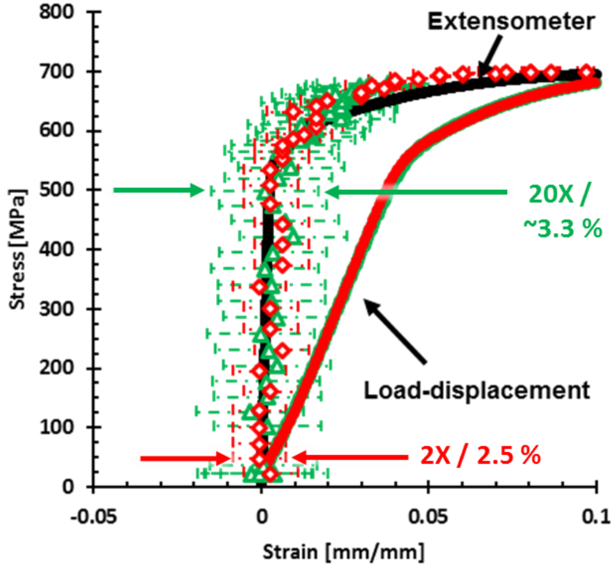


Environmental mechanical chamber (Yonekura)

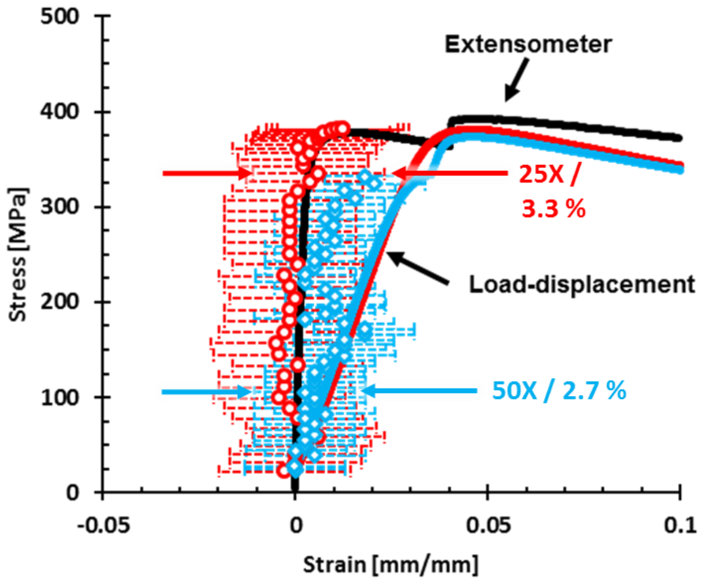
Previous Results

- Tested tensile properties of P91 steel in Air at 25°C, Vacuum at 550°C and CO_2 at 550°C
- Tensile curves overlapped with ASTM E8 generated data
- Greater experimental uncertainty at higher magnifications
- Noted image wobble was likely causing uncertainty
- Could be improved with automated strain measurements

25°C – P91 Steel



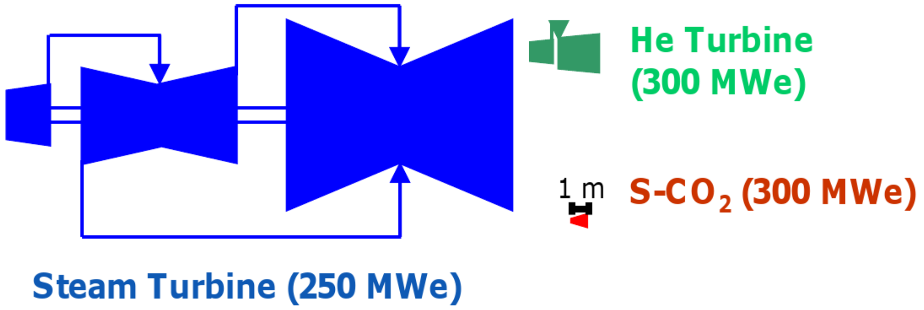
550°C – P91 Steel (Vacuum)

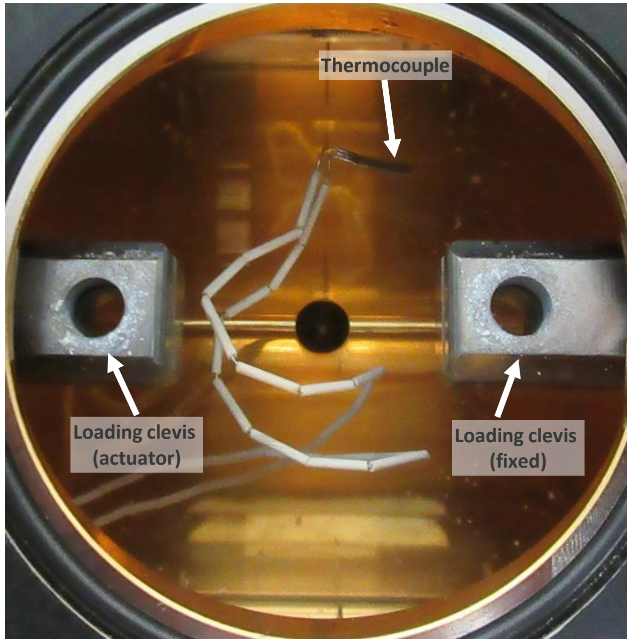


Rozman et al., Use of confocal microscope for environmental tensile testing, JOM, 70(10) (2018) 2270-2276

Creep Testing

- Building upon success of tensile tests
- Utilize system for *in-situ* controlled environment creep testing of materials
- Note corrosion properties pressure dependent for steels [Holcomb et al.]
- Test and analyze creep at 550°C under
  - CO_2 – Of importance to NETL advanced combustions projects
  - Ar – Simulates vacuum, -50 kPa-g was largest vacuum we could pull
- Target short term creep tests
  - 10 h
  - 20 h
  - 100 h
- Goal: Observe how CO_2 effects creep rates of P91 steel

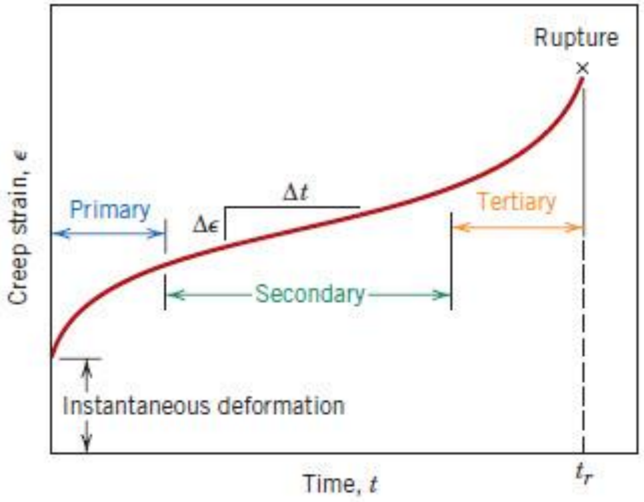




Holcomb et al., Oxidation of alloys for energy applications in supercritical CO_2 and H_2O, Corrosion Science, 109 (2016) 22-35

Creep Background

- When $T/T_{melt} > 0.4$, metals will elongate under load
- Strain rupture plots typically have three zones
  - Initial creep – rapid decrease in strain rate
  - Steady state creep – constant creep rate
  - Tertiary creep – rapid increase in creep rate
- Multiple models, Typically fit with exponential and Arrhenius fits
  - $\dot{\epsilon}_{ss} = K \cdot \sigma^n \cdot exp({-Q}/{RT})$
- Larson-Miller parameter (LMP)
  - Hardness was observed to follow a Time-Temperature relationship
  - Observed to be valid for creep as well
  - $LMP = T \cdot (\log t_r + C)$
- Monkman-Grant
  - Relationship between minimum creep rate and rupture time
  - $t_r \cdot \dot{\epsilon}_{ss} = Constant$

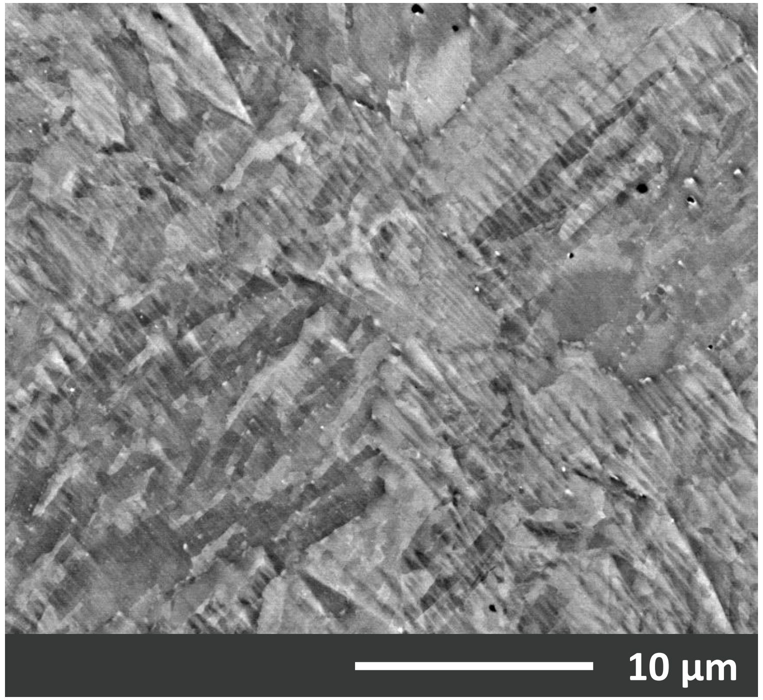


Material

- P91 steel from Arcelormittal Plate LLC
-

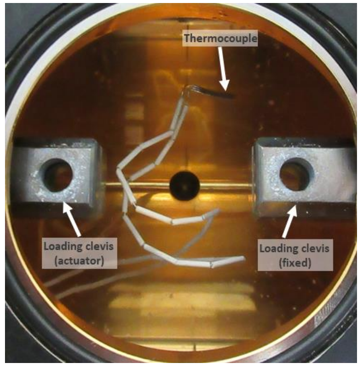
Element	Fe	C	Mn	P	S	Cu	Si	Ni	Cr	Mo	V	Ti	B	Al	Zr	Cb	N
Wt%	bal	0.09	0.45	0.01	0.004	0.09	0.33	0.09	8.37	0.9	0.224	0.002	0.001	0.007	0.001	0.072	0.0452

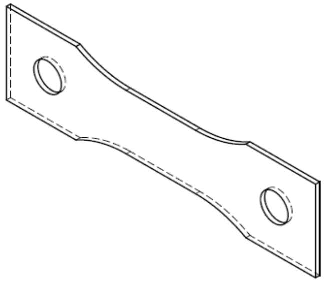
- Tempered martensite microstructure
- Small carbides line ferrite grain boundaries

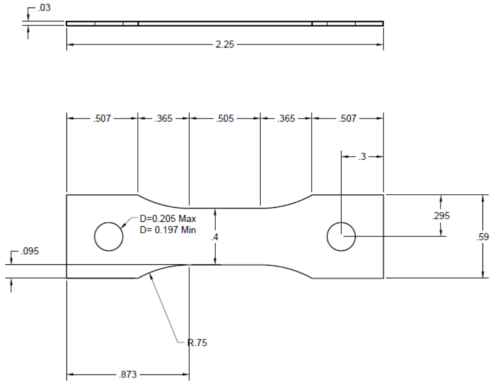


Utilizing Dog-bone Specimens

- CLSM equipped with pinned and threaded clevises
- Originally used thin dog-bone specimens
- Maximize surface area to enhance environmental interactions with surface
  - 0.76 x 10 mm gage cross sectional area
- Specimens bulged at loading pin interface
- Tested at 190 Mpa / 550C, Ar flow (50 SCCM)
  - Note 1800 N is known to buckle for this loading pin geometry
  - 190 MPa corresponds to 1100 N for this geometry



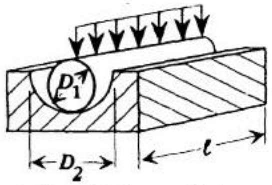




Slight bulge

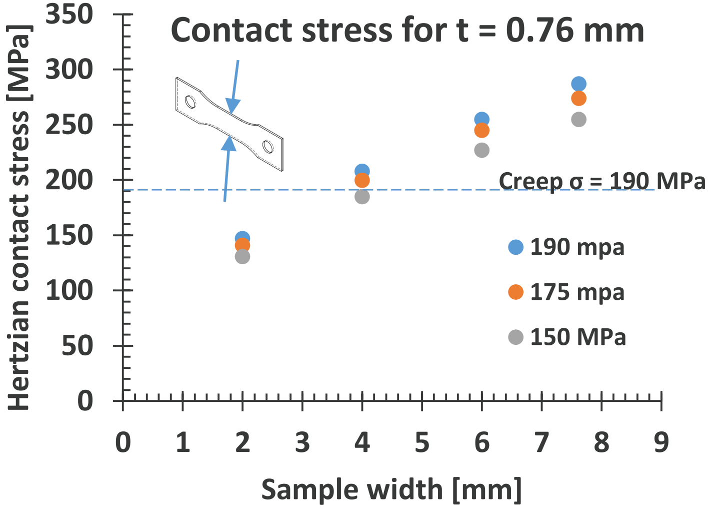
Hertzian Contact Stress

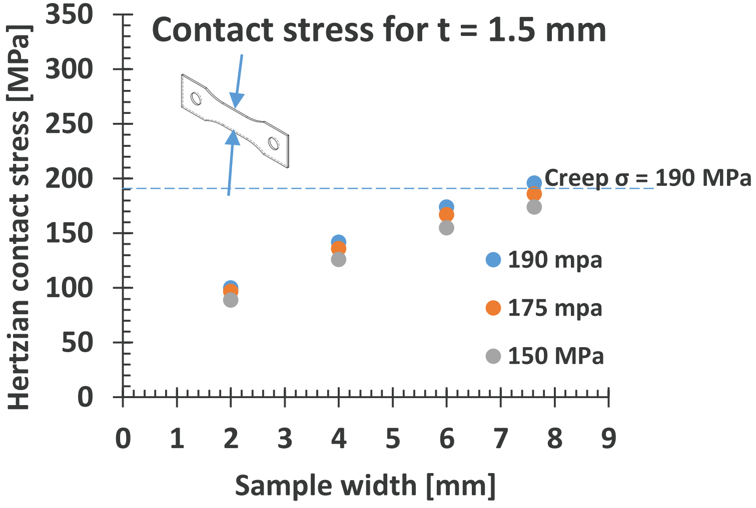
- Looked at Hertzian contact stress for cylinder in side cylinder
-

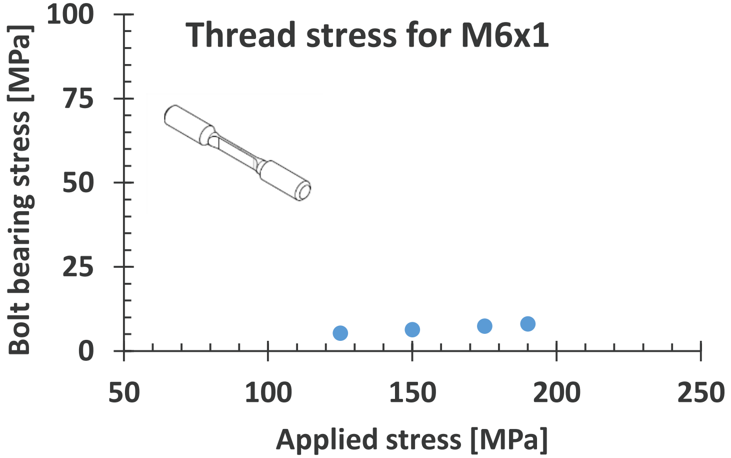


Cylinder in inner cylinder

- Contact stress was always 0.5x applied stress or greater for dog-bone specimens even when increasing thickness
- Redesign to utilize threaded ends
- Thread stress < 0.10x applied stress

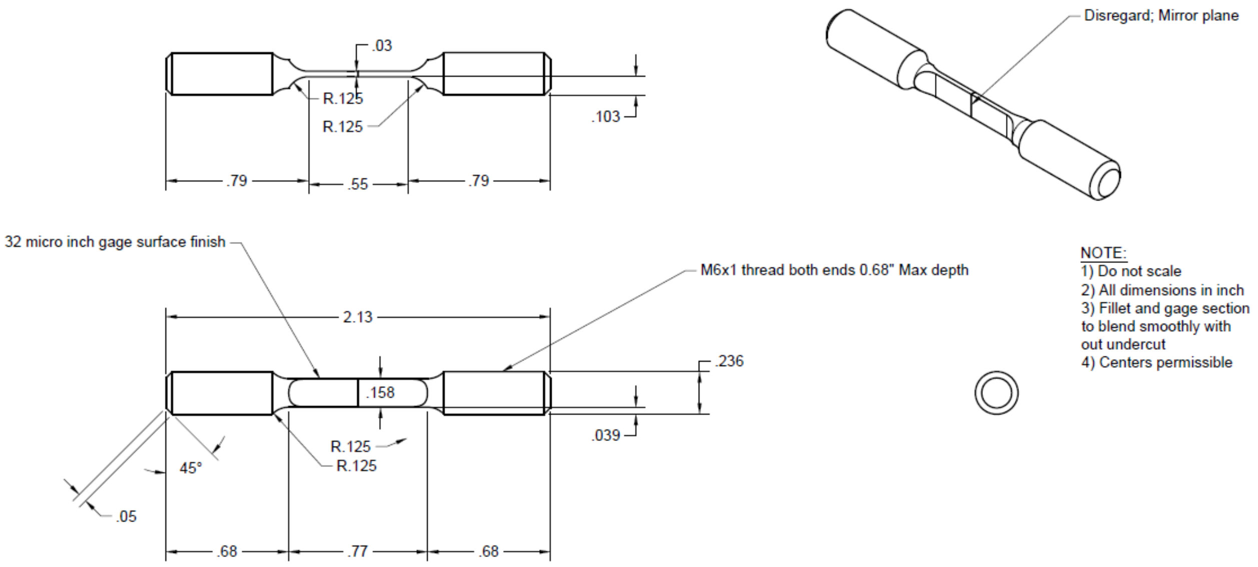






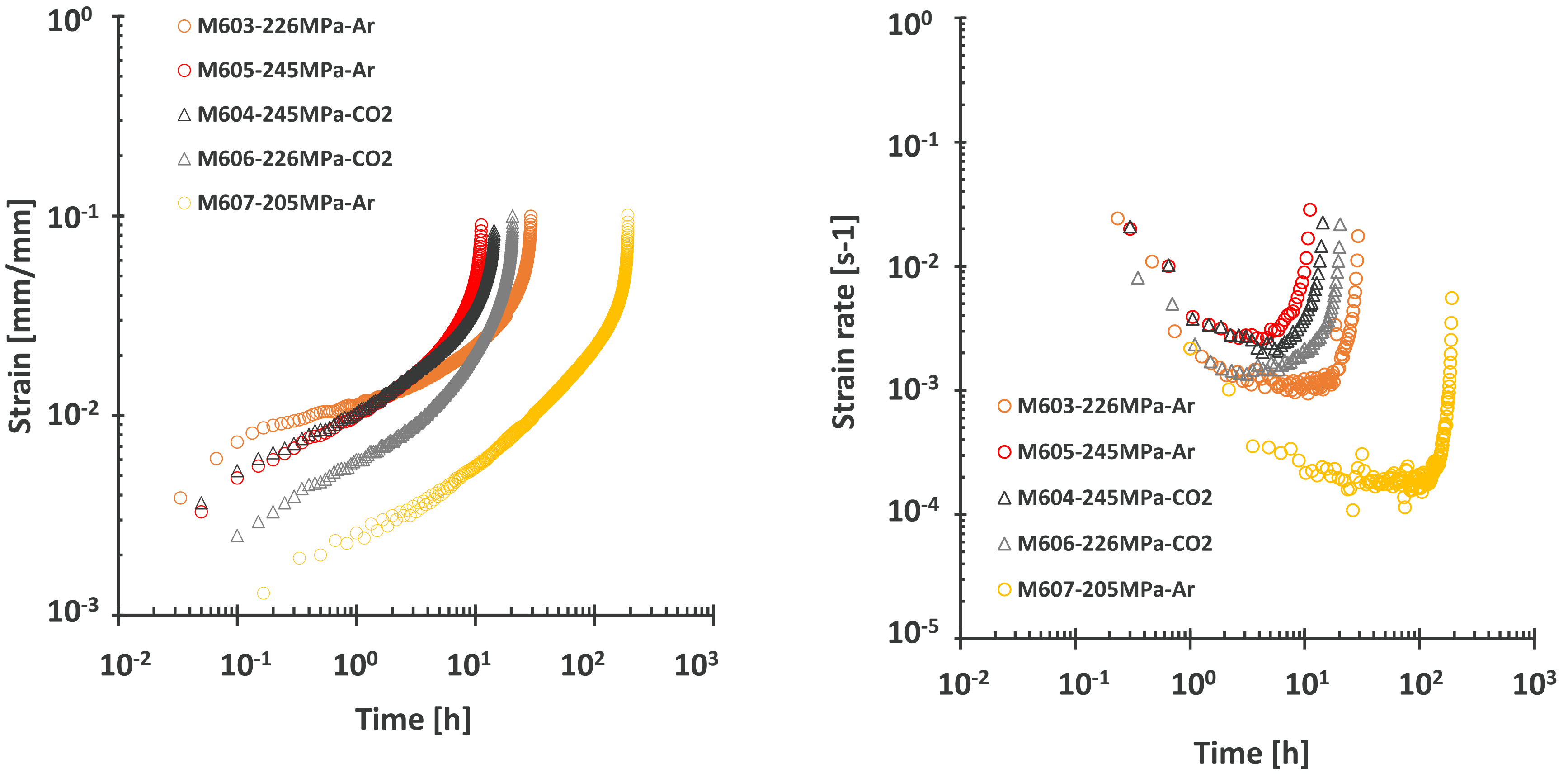
Experimental Parameters

- Utilized 'compression sample'
- Modified to reduce gage thickness
- Maximizes surface area
- Machined from ½" plate, L-T orientation, 1/8" away from edges
- Test at 550°C
  - Ar flow, 50 SCCM
  - CO_2 flow, 50 SCCM
- Creep at:
  - 245, 226, 205 MPa
  - Short term to evaluate system performance



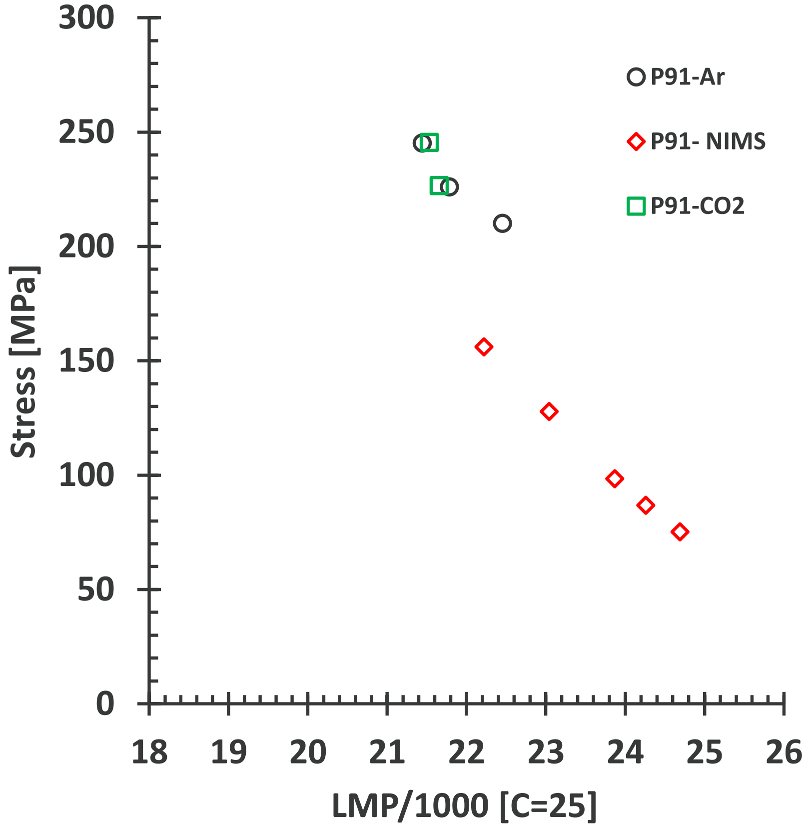
Creep Results

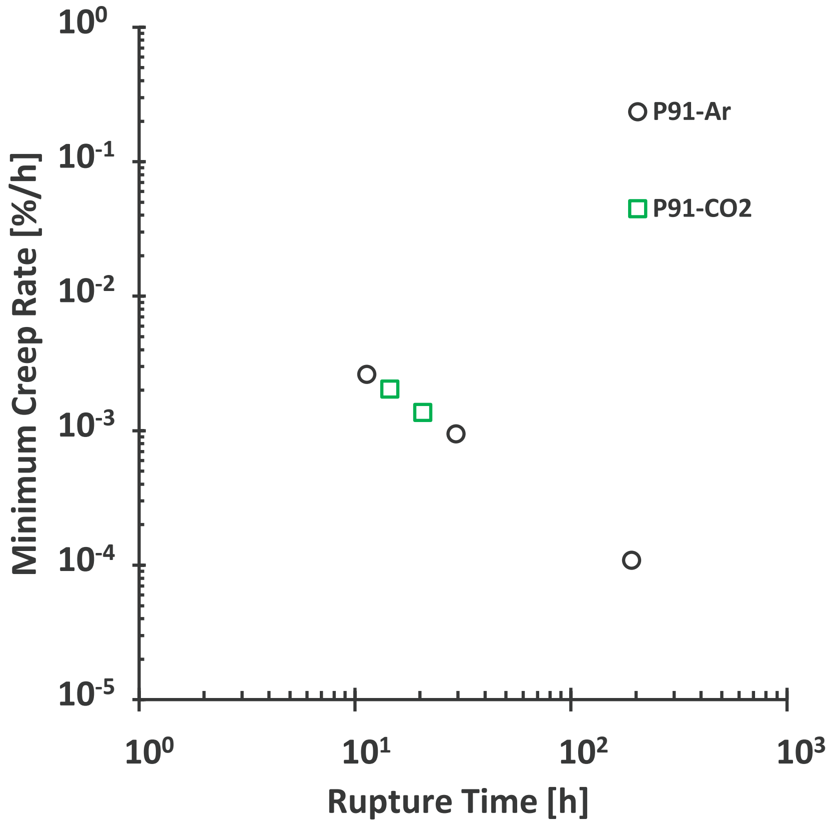
- Rates calculated from cross head displacement
- Stress intervals
  - 205, 226, 245 Mpa
  - Red-Yellow shades in 50 SCCM Ar flow
  - Black to Grey in 50 SCCM CO_2 flow
  - Darker shades higher stress
- While some scatter in time to failure is observed minimum creep rates and rupture time is consistent
  - Lower creep rates and longer rupture times for lower stresses
- No large effect of CO_2 relative to Ar



Creep Results

- $LMP = T \cdot (\log t_r + C)$
- Larson-Miller curve shows increase in performance relative to NIMS reported P91
- L-M shows no change in performance of P91 crept in CO_2 relative Ar
- Monkman-Grant shows linearized behavior, with CO_2 performance in line with Ar





Accuracy

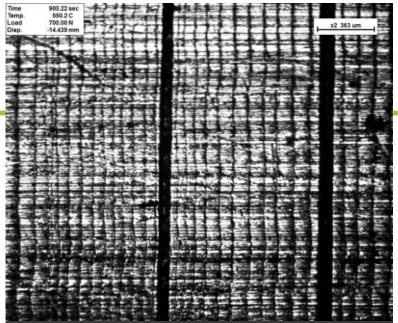
From tensile testing:

- **Manual measurements**
- **Best practices had repeatability to within 5 pixels**
- **Strain uncertainty estimated at 2.5%**
- **Reasonable rates were measured, with a bit of uncertainty**
- **Difficult to measure strain with corrosion layer in CO_2 experiments**

Note we encountered some software related issues.

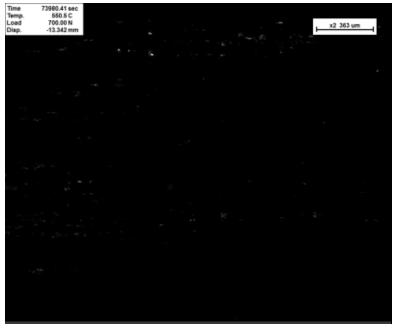
- **Images were not recoverable from a few tests**
- **One test had to be restarted**

Magnification	Length per pixel	Focus uncertainty	Approximate gage length	Total uncertainty
-	[µm]	[µm]	[µm]	[%]
2x	2.4	12	950	2.5
25x	0.19	3	120	3.3
50x	0.094	1.5	80	2.7

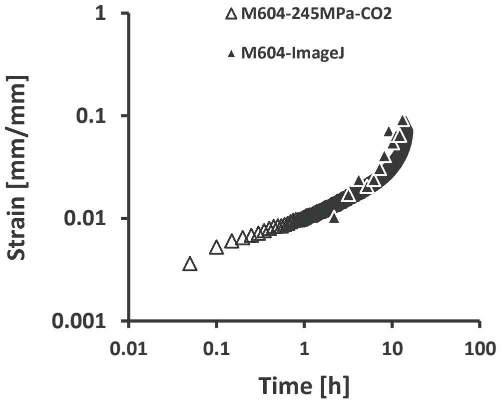


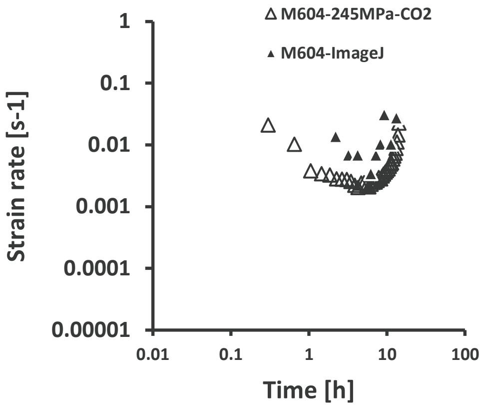
Start of test

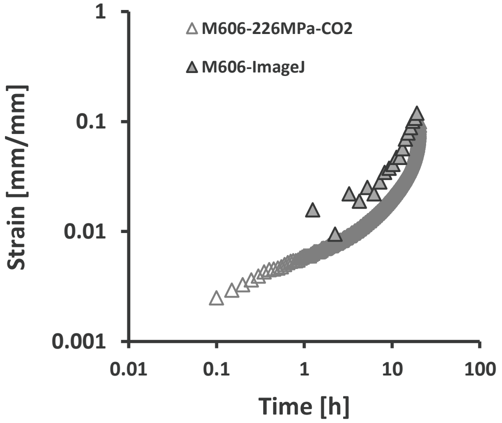
Mid test

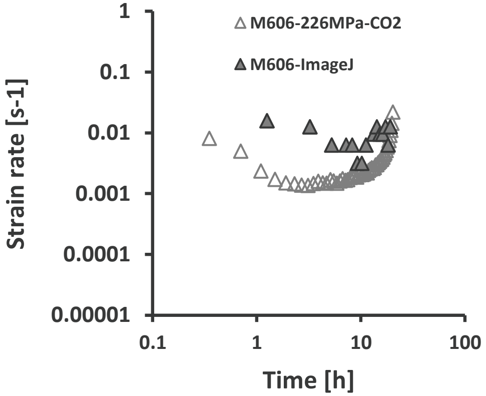


Near end test



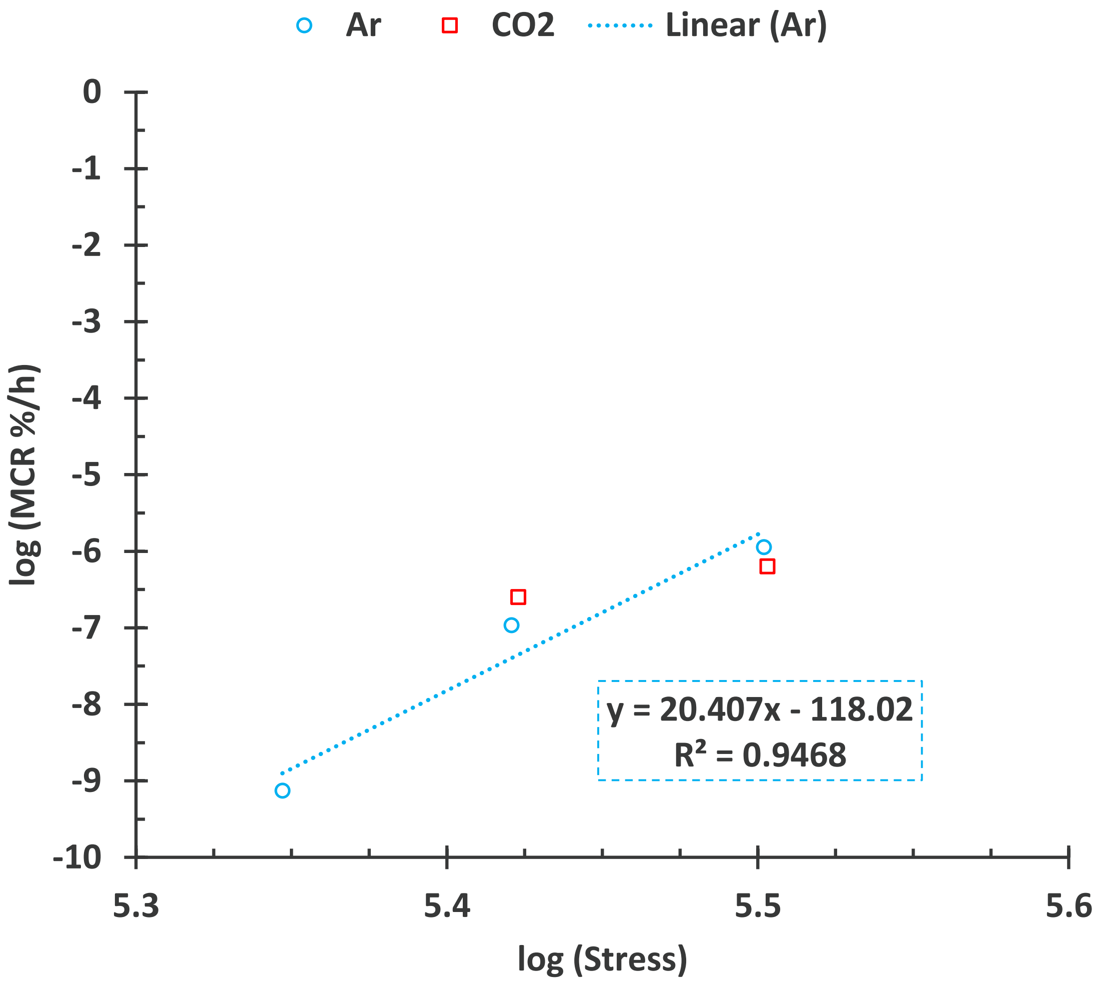






Creep Exponent

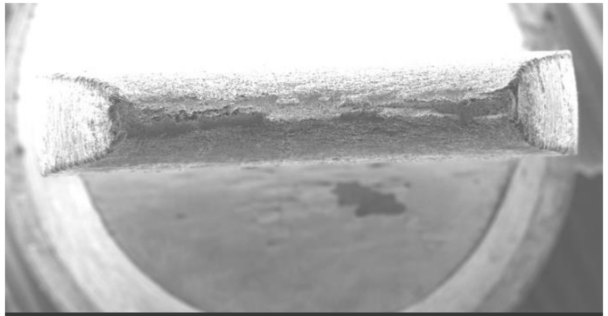
- Multiple models, Typically fit with exponential and Arrhenius fits
  - $\dot{\epsilon}_{ss} = K \cdot \sigma^n \cdot exp\left({}^{-Q}/_{RT}\right)$
  - 1 < n < 2 | creep mechanism is diffusion dominated
  - 3 < n < 7 | creep mechanism is dislocation dominated (climb)
  - n >> 7 | creep mechanism approaches ideal plastic behavior (glide)
- n ~ 20.4 for Ar exposures
  - Waiting on creep result at 205 MPa for n-value in CO_2
- High stress, short time experiments are more akin to slow strain rate tensile test than creep experiment
- Large 'n' values justified



Fractography

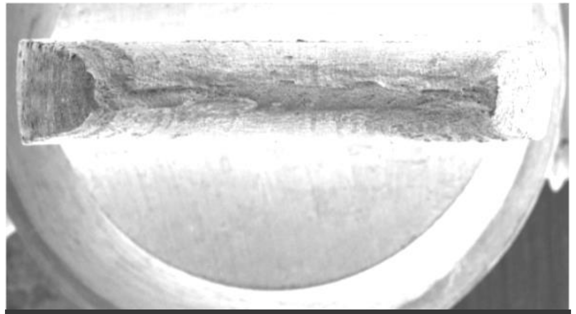
- **Ductile fracture surfaces observed independent of environment**
- **Less reduction of area in CO_2**
- **Highlighted in red**

Env.	Stress	TTF	Elong.	RA
-	[MPa]	[h]	[%]	[%]
Ar	226.1	29.4	11%	73%
Ar	245.2	11.4	13%	66%
Ar	210	191.1	11%	79%
CO2	245.5	14.6	10%	53%
CO2	226.6	20.7	10%	50%
CO2	210	-	-	-



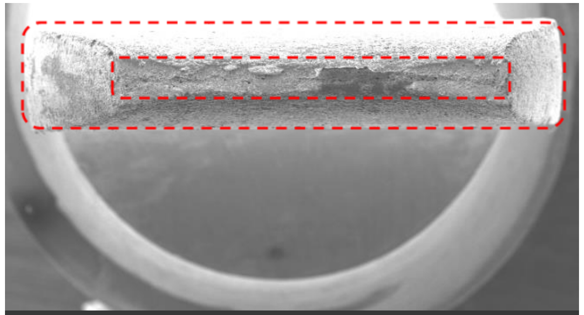
P91 / 205 MPa / Ar / 191 h

1 mm



P91 / 226 MPa / Ar / 29.4 h

1 mm



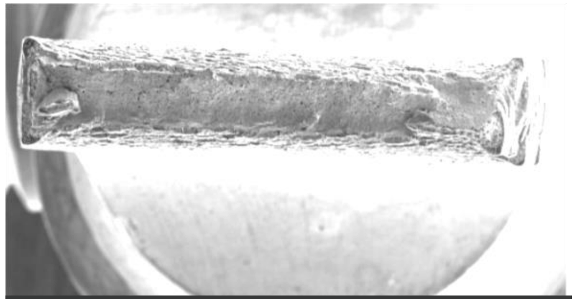
P91 / 245 MPa / Ar / 11.4 h

1 mm

In test

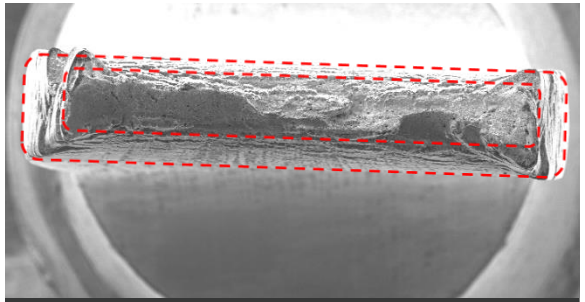
P91 / 205 MPa / CO_2 / xxx h

1 mm



P91 / 226 MPa / CO_2 / 20.6 h

1 mm



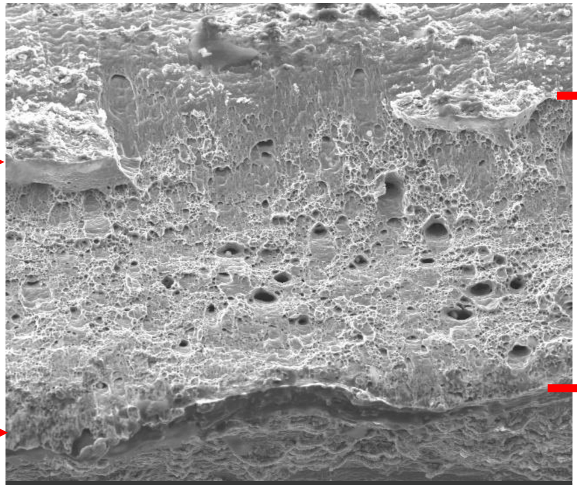
P91 / 245 MPa / CO_2 / 14.5 h

1 mm

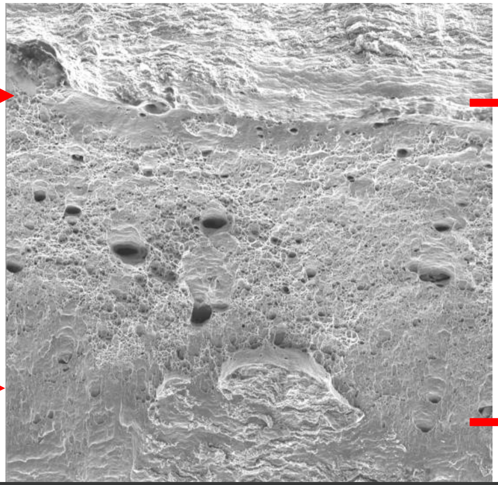
Fractography

- **Only see signs of ductile failure**
- **Arrows indicate specimen edge**

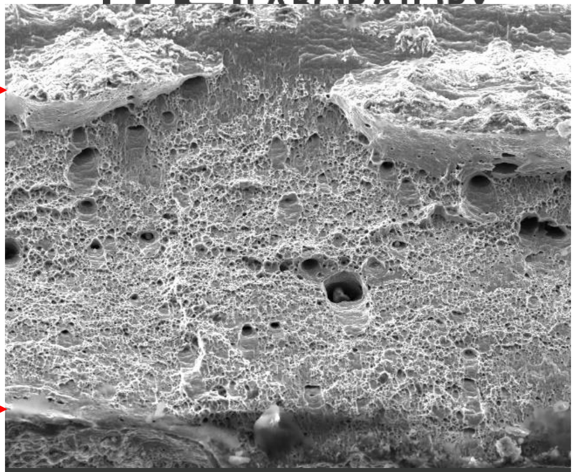
Specimen edge



P91 / 205 MPa / Ar / 191 h
50 µm



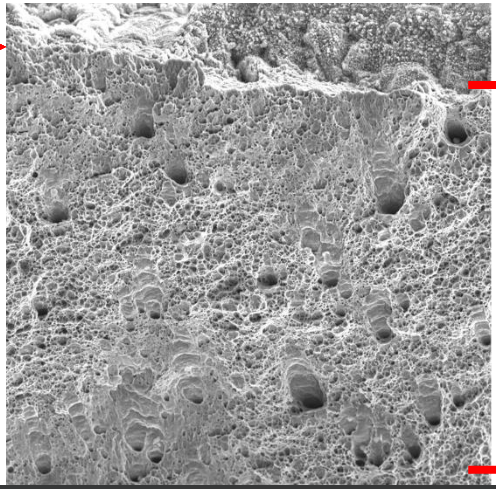
P91 / 226 MPa / Ar / 29.4 h
50 µm



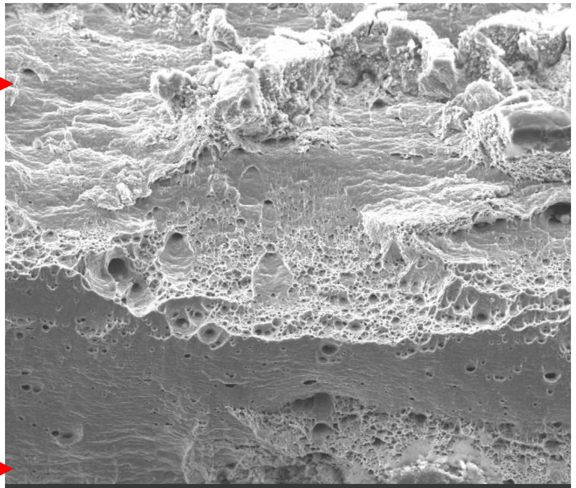
P91 / 245 MPa / Ar / 11.4 h
50 µm

In test

P91 / 205 MPa / CO_2 / xxx h
50 µm



P91 / 226 MPa / CO_2 / 20.6 h
50 µm

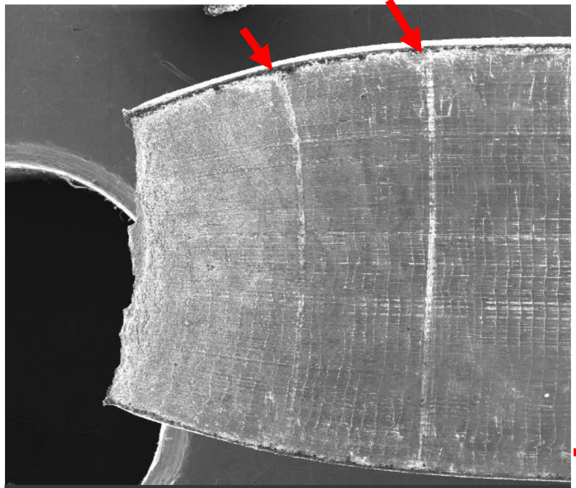


P91 / 245 MPa / CO_2 / 14.5 h
50 µm

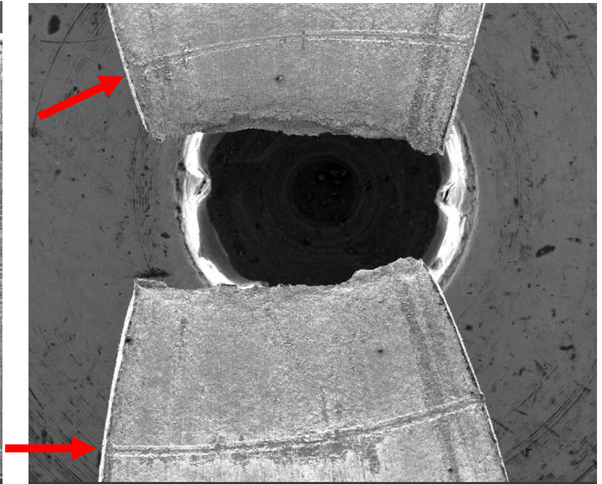
Fractography

- Signs of corrosion cracking on CO_2 specimen
- Arrows indicate gage scribe used to measure strain

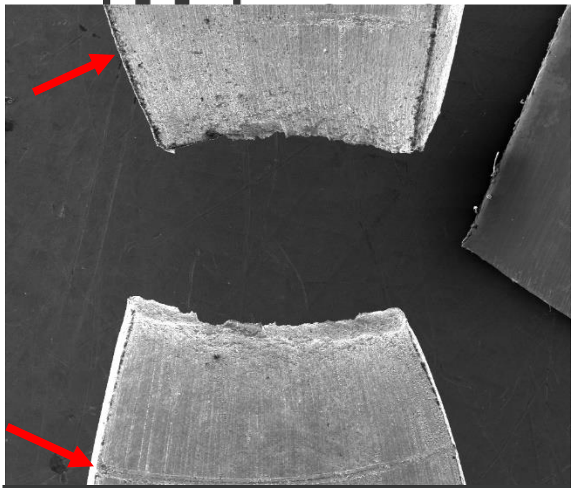
Gage scribe



P91 / 205 MPa / Ar / 191 h
1 mm



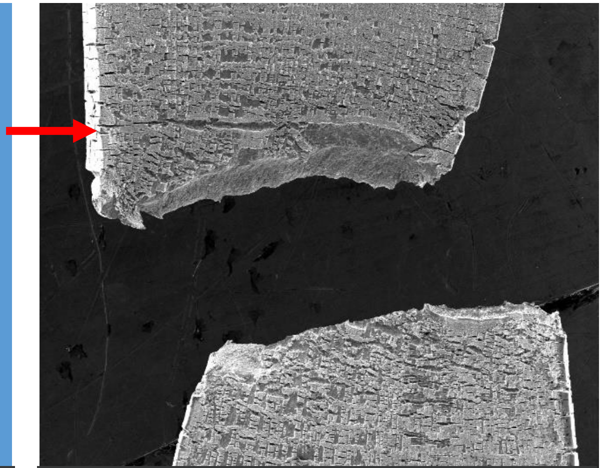
P91 / 226 MPa / Ar / 29.4 h
1 mm



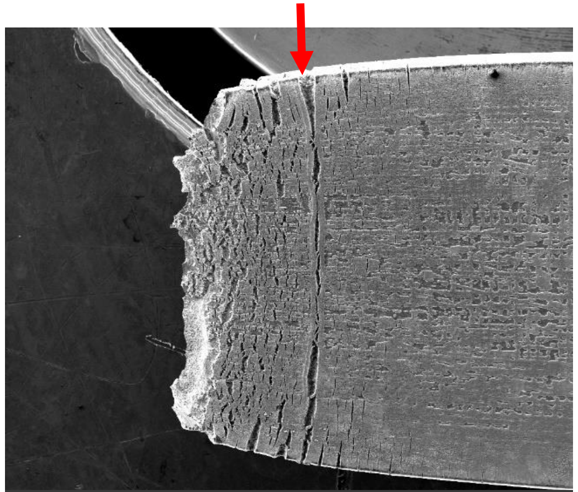
P91 / 245 MPa / Ar / 11.4 h
1 mm

In test

P91 / 205 MPa / CO_2 / xxx h
1 mm



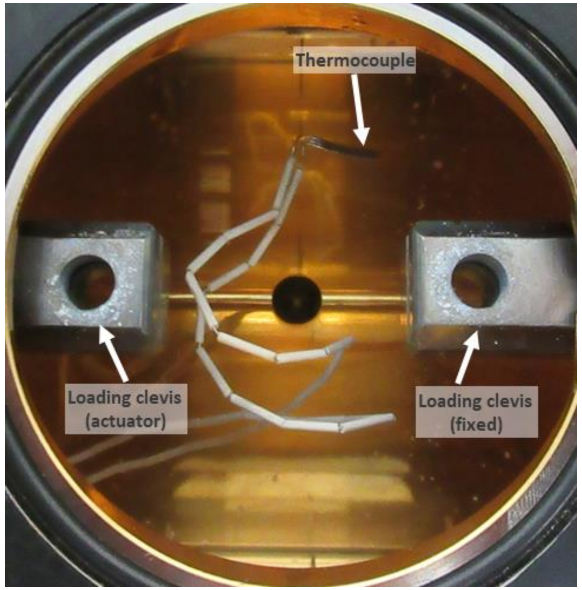
P91 / 226 MPa / CO_2 / 20.6 h
1 mm

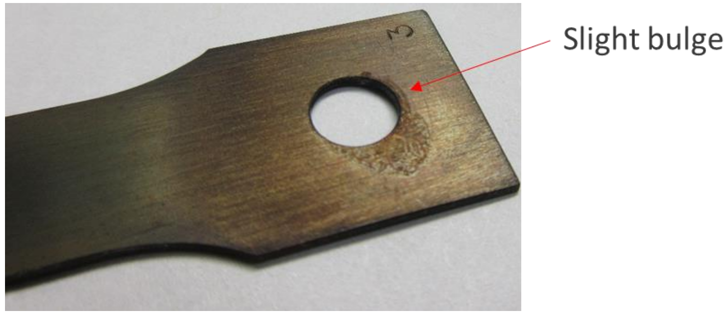


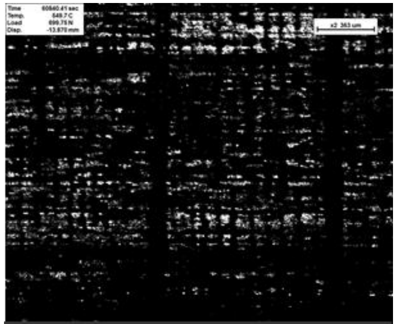
P91 / 245 MPa / CO_2 / 14.5 h
1 mm

Review

- Microscope system equipped with environmental chamber successfully tested creep performance of P91 steel
  - One must used ‘compression sample’ design, with threaded M6x1 ends
  - Note a few software issues (crashes/instabilities after long times)
  - More corrosive environments are unable to maintain contrast for imaging, need alternative way to place gage marks
- Automated measuring may increase accuracy of strain rates
- Balance between file size and data capture rate (GB per test)

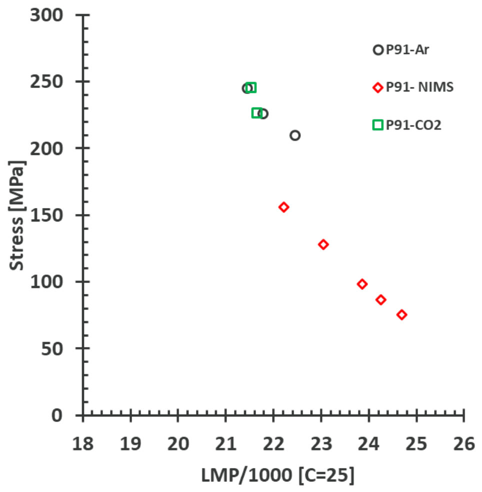


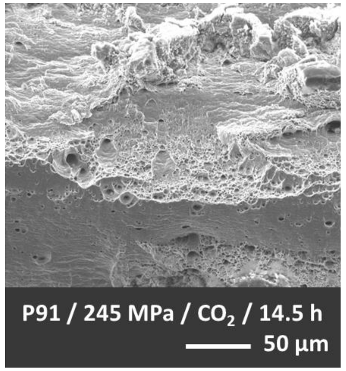



Mid test

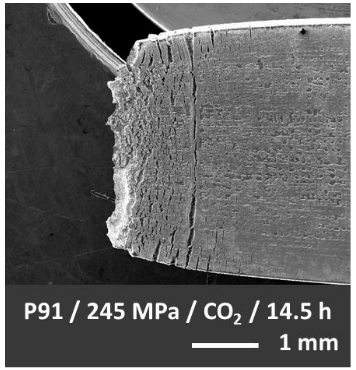
Review

- Tested P91 at 245, 226 and 210 MPa at 550°C in flowing Ar and CO_2 gas
  - No change in LMP, or minimum creep rates for CO_2 vs Ar flow
  - High stress exponent suggests mechanical performance analogues to a slow strain rate tensile test
  - No noted change in elongation, however, reduction of area was less for CO_2 tested specimens
  - Fracture surfaces show only ductile features
  - More surface cracking on specimens exposed to CO_2 flow
- As the LMP was higher for both CO_2 and Ar flow, we are looking to do creep testing in lab air to investigate the cause of better creep performance relative to NIMS data





P91 / 245 MPa / CO_2 / 14.5 h



P91 / 245 MPa / CO_2 / 14.5 h

Thank you

Questions?

<u>ACKNOWLEDGEMENTS</u>

Thank you to the organizers of this symposium

Kyle Rozman, Ph.D.
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