FORT WAYNE METALS
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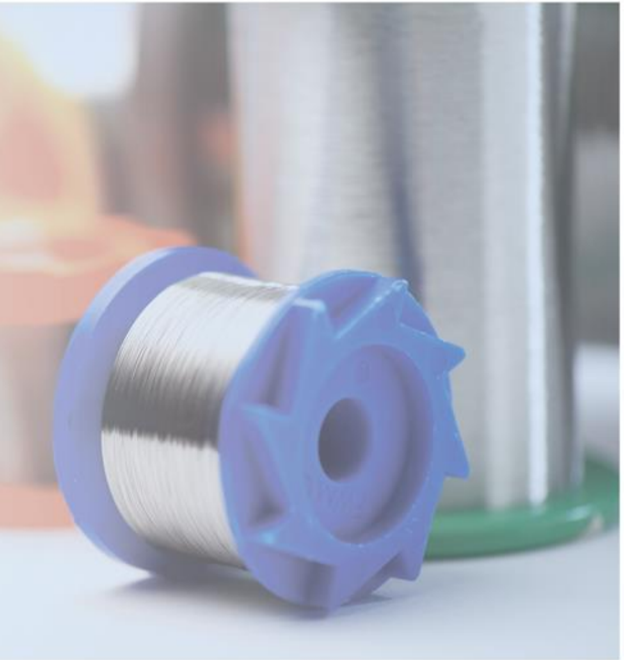
Stress Corrosion Testing of Mg Wire

Adam Griebel, Alexis Nicolette-Baker,
Anh Pham, Sam Friedman, Jeremy Schaffer



Updates in Bioabsorbable Metals 2020 | August 25, 2020 | Virtual

Precision melting and integrated supply chain

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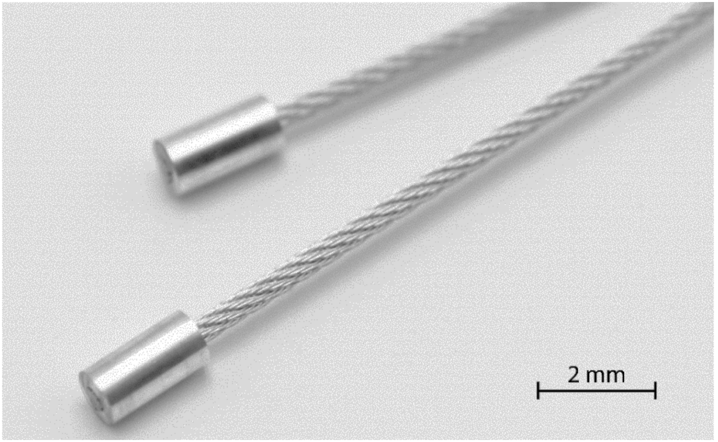
Absorbable Wire in Medicine

Orthopedic devices (cables, screws, pins, etc.)



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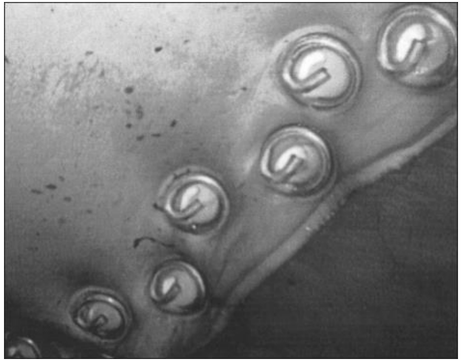
Soft Fixation (staples, ligation, sutures)

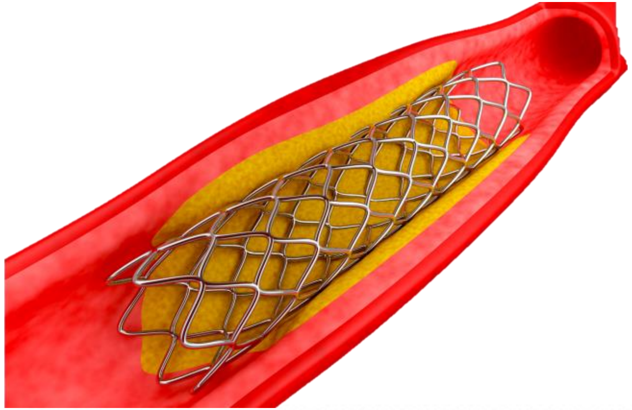


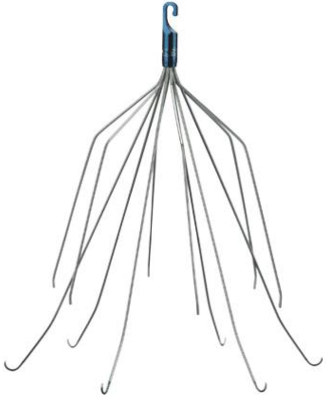
Absorbable Wire in Medicine

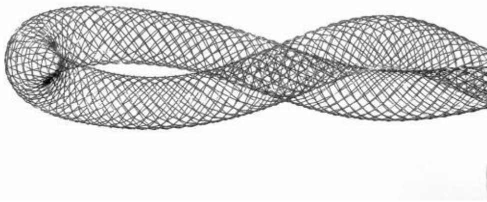
Orthopedic devices (cables, screws, pins, etc.)

Soft Fixation (staples, ligation, sutures)

Stents and intraluminal scaffolds







Absorbable Wire in Medicine

Orthopedic devices (cables, screws, pins, etc.)

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Stents and intraluminal scaffolds

Scaffolds

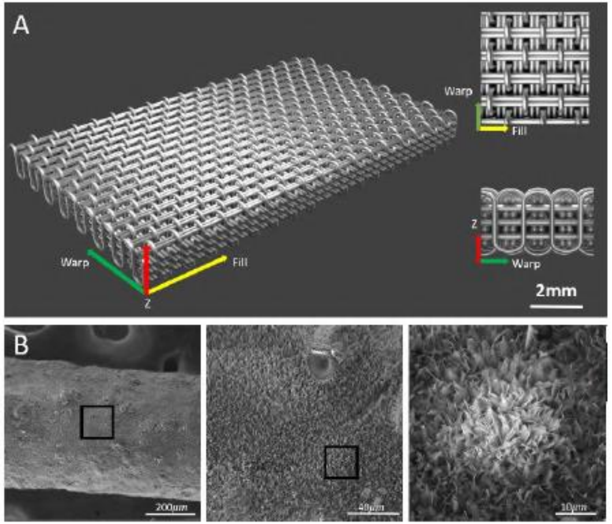


Fig. 1: (A) schematic of Mg 3D weave; (B) Mg alloy wire coated with HAp

Xue et al, Biometal 11, (2019)

Mg wire in medicine

1924

A STUDY OF MAGNESIUM WIRE AS AN ABSORBABLE SUTURE AND LIGATURE MATERIAL *

M. G. SEELIG, M.D.

ST. LOUIS

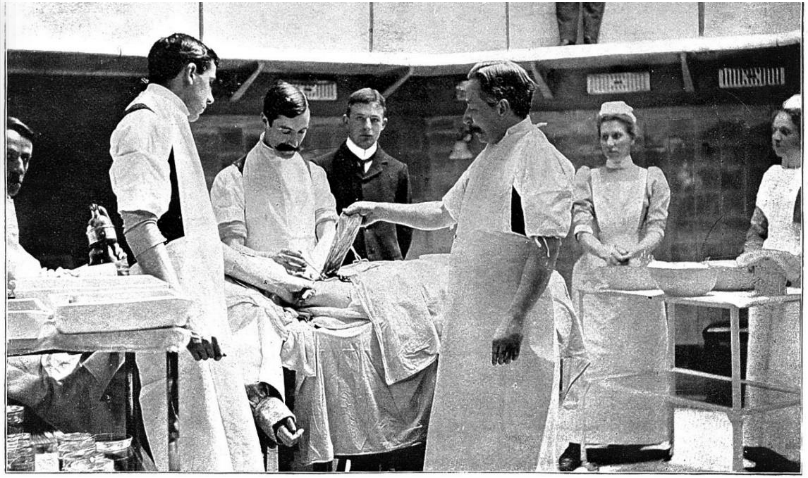
In adapting the metal for this purpose, there were several requirements to be met:

1. A wire of sufficient tensile strength to withstand the strain of sewing and tying.

2. A wire of sufficient pliability or ductility to withstand the flexing encountered in tying the surgeon's double knot.

3. A wire of high and uniform purity to insure corrosion taking place uniformly when in contact with blood serum.

4. A wire permitting some measure of control in the rate of absorption.



Why do we do corrosion testing?

Why do we do corrosion testing?

- Screen
  - Which alloys might work?
  - Which alloys will not work?
- Optimize
  - Which composition/process/surface works best?
- Inspect/Verify
  - How repeatable is it?
  - Did this batch meet our requirements?
- Answer fundamental questions
  - What factors influence corrosion, and how can we control them?
  - What is the corrosion mechanism?
- Predict *in vivo* performance?

Corrosion Assessment

- Electrochemical (PDP, EIS)
- Immersion
- Stress Corrosion
- Corrosion Fatigue

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New approaches in evaluating metallic candidates for bioabsorbable stents
Bowen, Drelich, Buxbaum, Rajachar and Goldman
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New approaches in evaluating metallic candidates for bioabsorbable stents

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4 **Rupak M. Rajachar** PhD
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5 **Jeremy Goldman** PhD
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Effect of stress on the corrosion behaviour of magnesium wire in m-SBF

S Zhang [1], X Li[2], H Wu[2], C Zhao[2], J Liu[1], Y Zhang[1], X Zhang[1,2]
[1]Suzhou Origin Medical Technology Co., Ltd. [2]School of Materials Science and Engineering, Shanghai Jiao Tong University

Stress corrosion of cold drawn and aged WE43 wires in different setups

P Maier[1], AJ Griebel[2], WD Mueller[3], JE Schaffer[2]
[1]University of Applied Sciences Stralsund, Stralsund, Germany. [2]Fort Wayne Metals Research Products Corp., Fort Wayne, IN, USA. [3]Charite Berlin, Universitätsmedizin, Germany

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Corrosion Bending Fatigue of RESOLOY® and WE43 Magnesium Alloy Wires

Authors Authors and affiliations

Petra Maier, Adam Griebel, Matthias Jahn, Maximilian Bechly, Roman Menze, Benjamin Bittner, Jeremy Schaffer

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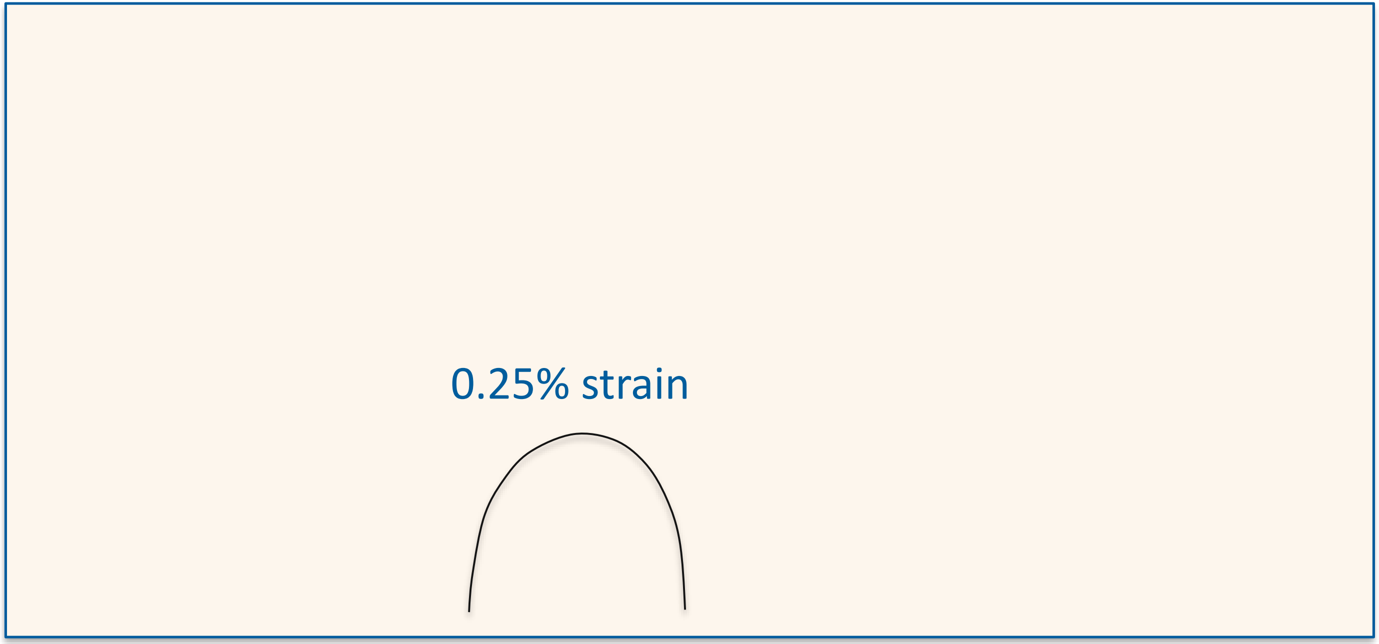
Volume 113, 1 September 2020, Pages 627-645

Full length article

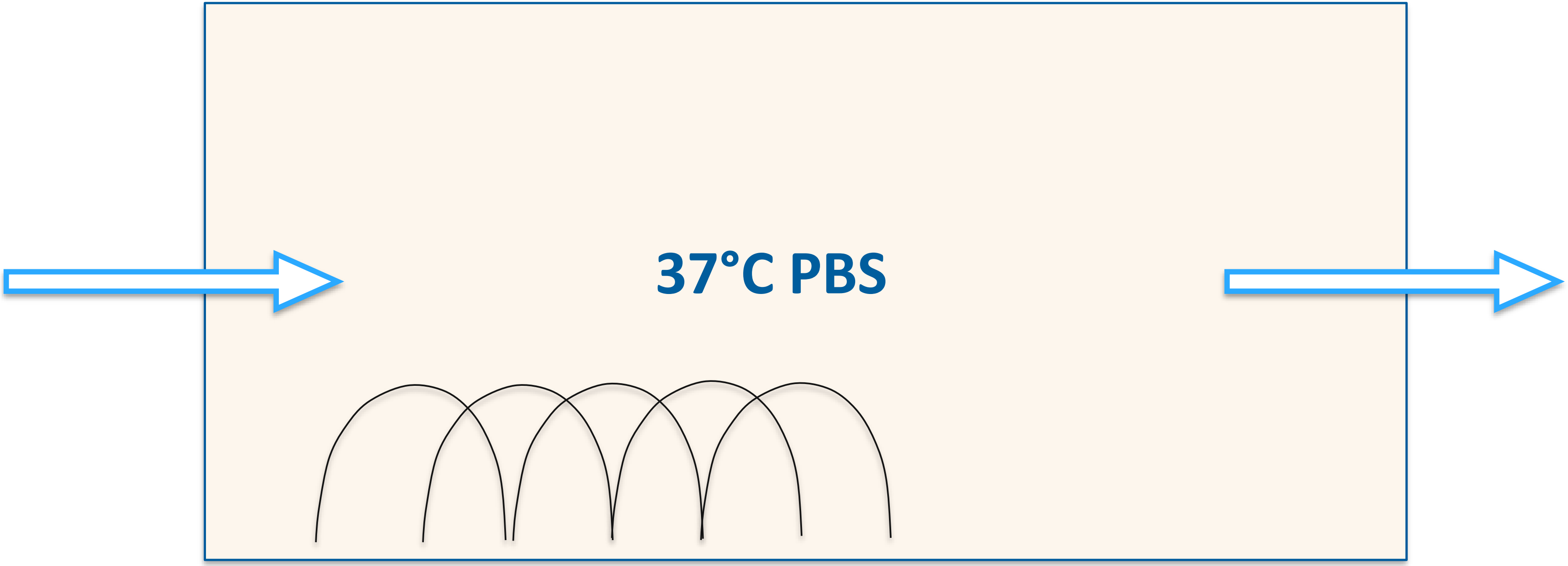
Effect of strain on degradation behaviors of WE43, Fe and Zn wires

Kai Chen [a, b, 1], Yun Lu [a, b, 1], Hongyan Tang [a, b], Yuanming Gao [a, b], Feng Zhao [a, b], Xuenan Gu [a, b], Yubo Fan [a, b, c]

Strain Stress Corrosion at FWM



Static strain-corrosion



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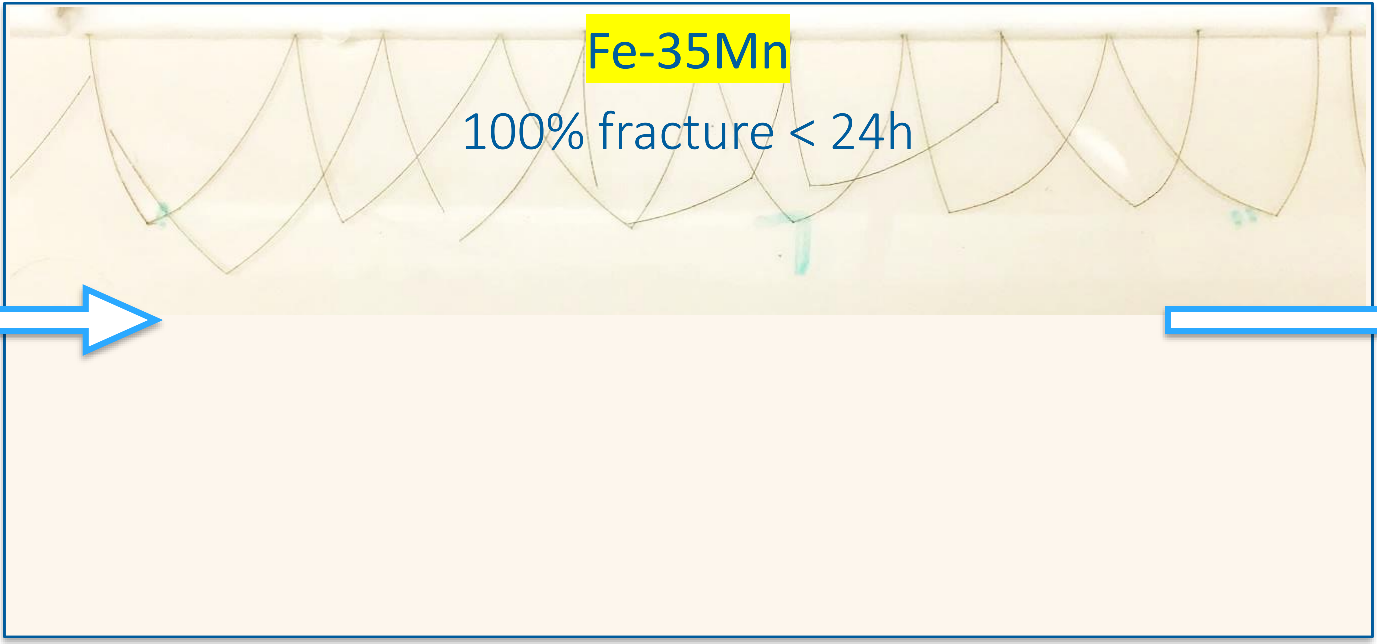

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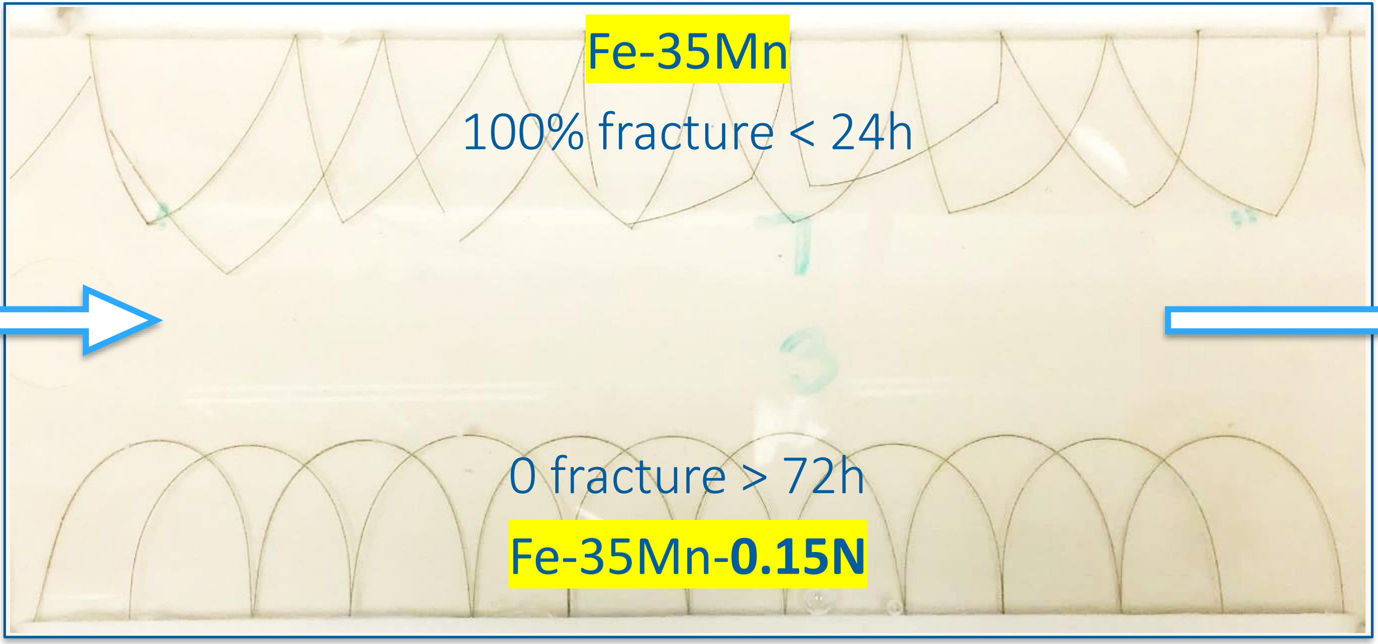
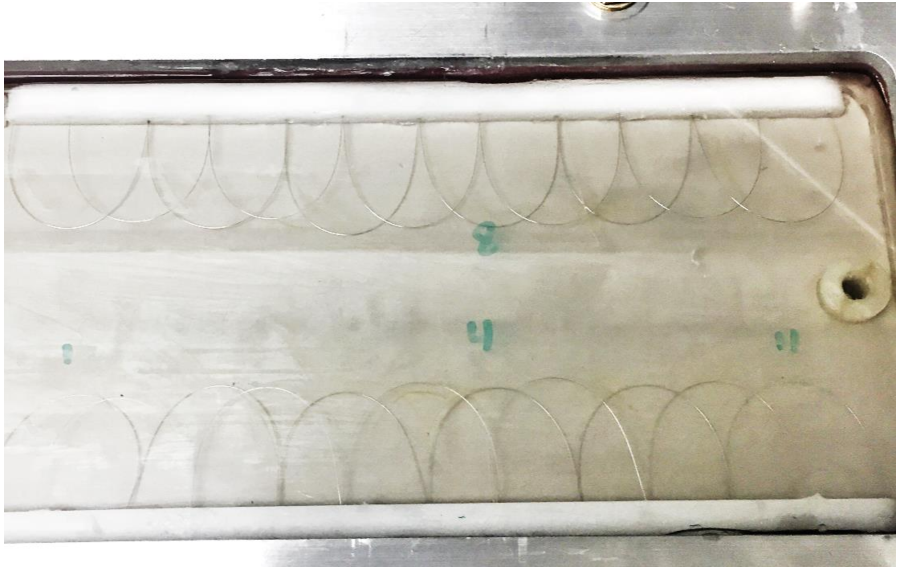


Strain-controlled corrosion: Mg (WE43)



0 hrs

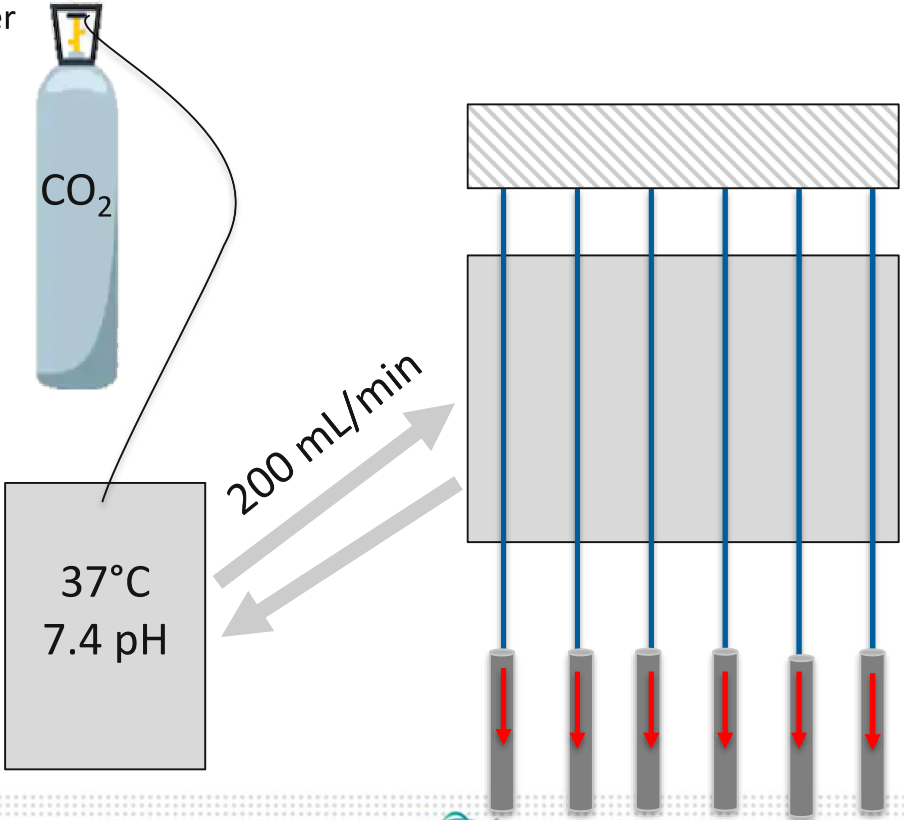
96 hrs

Aim

A corrosion testing method that can efficiently and repeatably assess corrosion and stress-corrosion behavior of fine Mg wires

Stress-Corrosion System Design

- 6 horizontal wires through 150 mm test chamber
- Stainless steel deadweights
- Electrical contact break detection system
- Media
  - 5L of Modified Hank's Solution
    - 1.6 g/L sodium bicarbonate
    - 0.265 g/L calcium chloride dihydrate
  - pH 7.4 +/- 0.1
    - Buffered with CO_2 bubbling
  - 37 +/- 1°C
  - 200 mL/min flow
- Data Outputs:
  - Time-to-failure
  - Post-corrosion cross-sectional analysis



Stress-Corrosion System Design

Test Plan

- Case 1: Compare alloys and process conditions at a given stress
- Case 2: Investigate influence of stress on corrosion rate at a given time
- Case 3: Generate stress-life curve for a given alloy and evaluate corrosion morphology

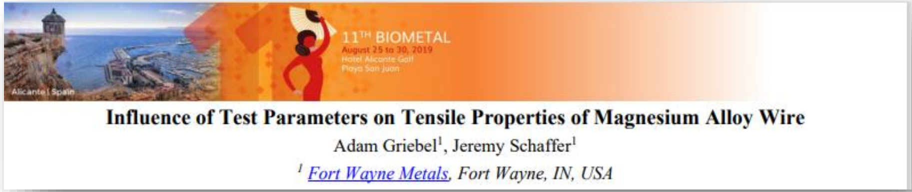
Alloys Investigated

Name	Mg	Li	Zn	Ca	Y	Nd	Zr	Mn	Fe	Cu	Ni
LZ21	96.0	2.0	1.21	0.35	< 0.01	< 0.01	< 0.01	0.4	0.004	< .001	< .001
WE43	92.8	< 0.01	< 0.01	< 0.01	3.9	3.0	0.3	< .01	0.004	0.008	0.001
L4	96.4	3.5	< 0.01	0.01	< 0.01	< 0.01	< 0.01	0.03	0.004	0.008	<.001
ZX10	98.6	< 0.01	1.03	0.26	< 0.01	< 0.01	< 0.01	0.12	< .001	0.001	< .001

Wire Production

- VIM, cast to ø50 mm
- Extruded to ø12.7 mm
- Wire drawing to ø0.24, 0.25 mm

Tensile Properties


11TH BIOMETAL
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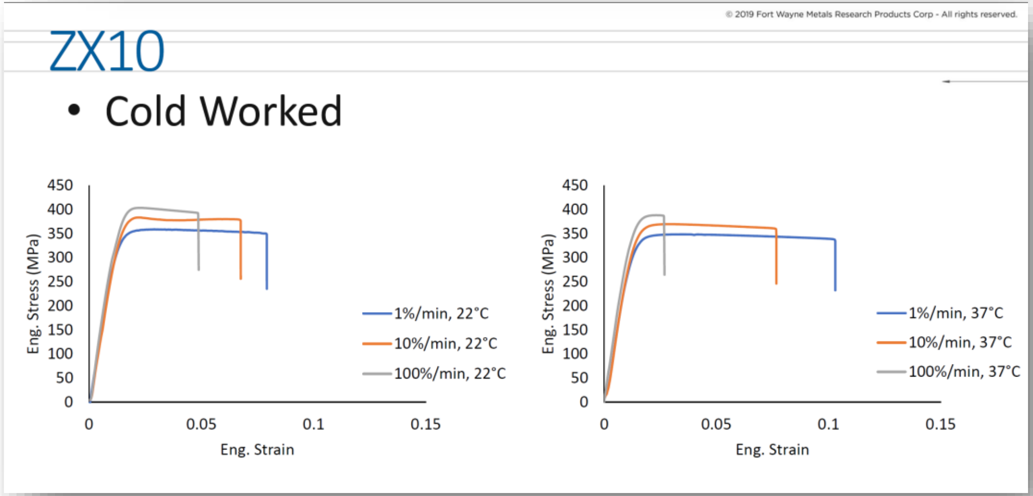
Influence of Test Parameters on Tensile Properties of Magnesium Alloy Wire

Adam Griebel[1], Jeremy Schaffer[1]

[1] *Fort Wayne Metals, Fort Wayne, IN, USA*



Tensile Properties

Influence of Test Parameters on Tensile Properties of Magnesium Alloy Wire

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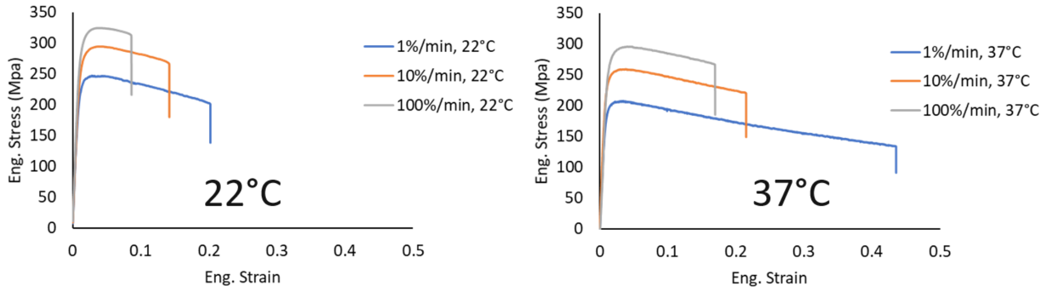
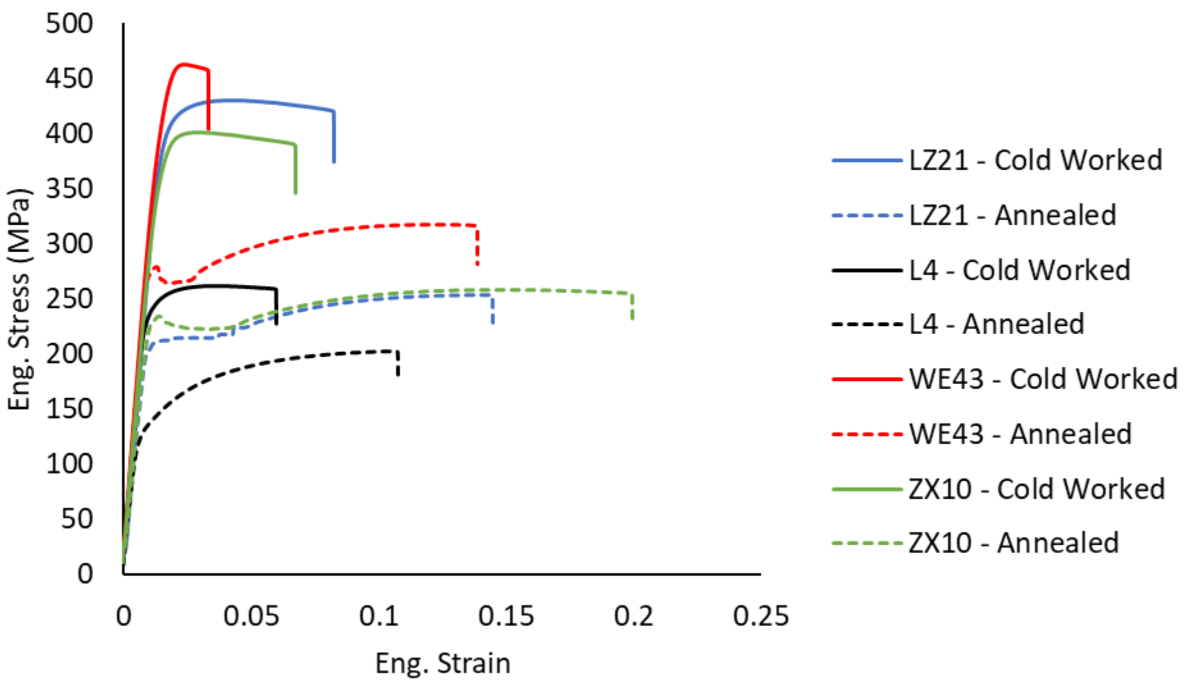
Mg4Li

- Cold Worked



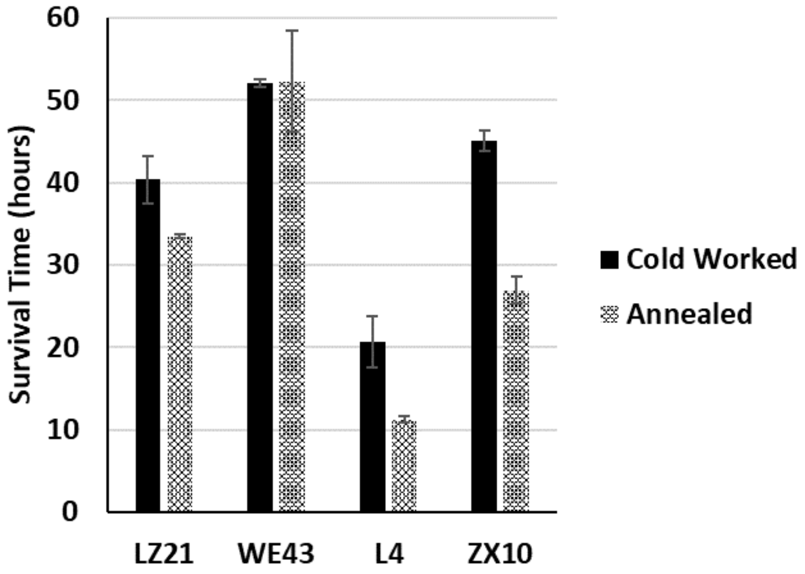
Mg wire tensile

- 127 mm GL, 20%/min, 22°C



Case 1: Comparing Alloys

- LZ21, WE43, L4, ZX10
  - 0.24 mm, 110 MPa initial stress, Both Cold Worked and Annealed conditions



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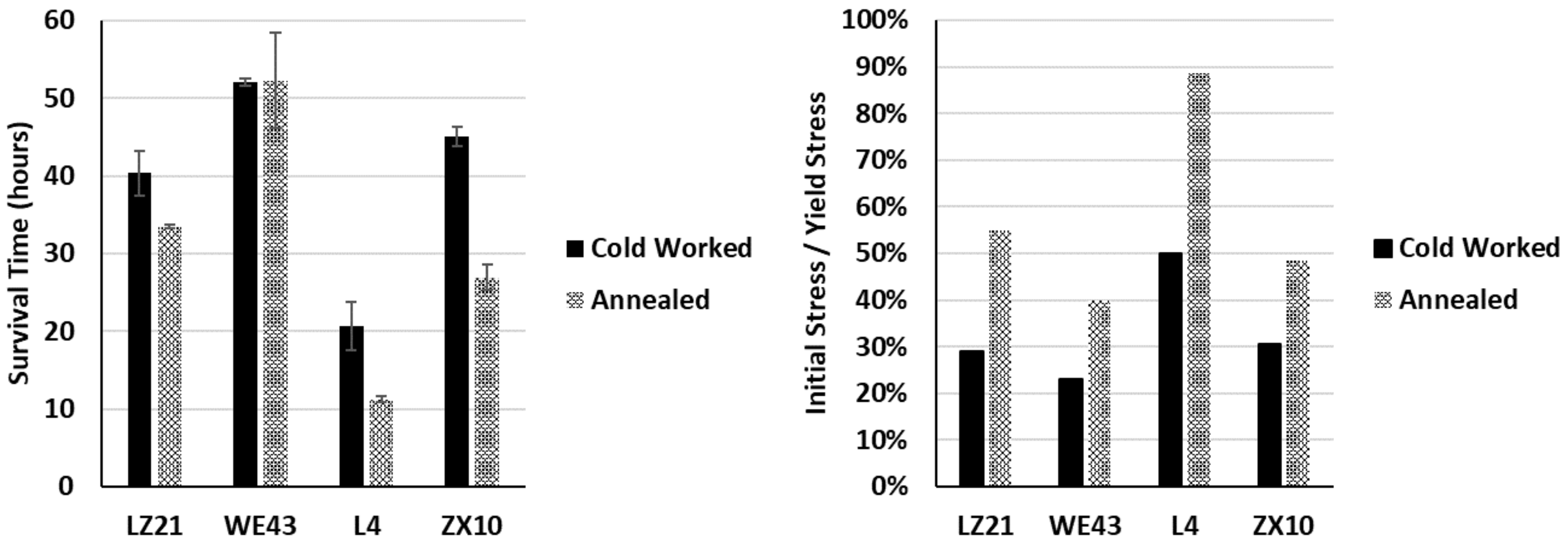

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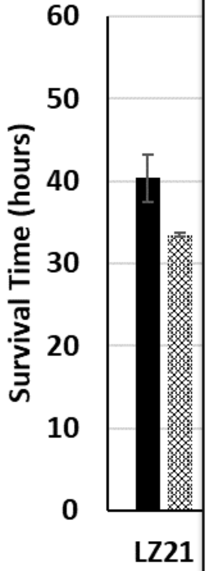
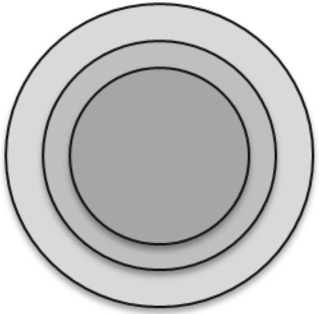
  - 0.24 mm ... d conditions

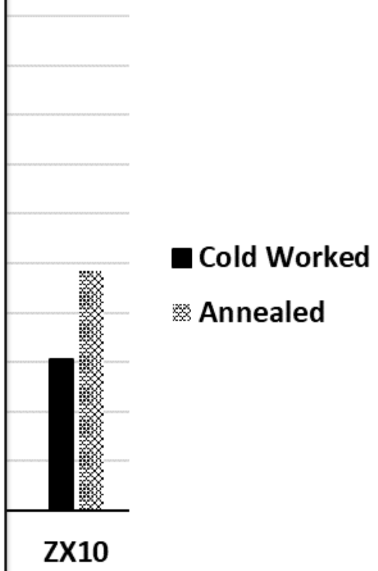


Constant Force ≠ Constant Stress

$$\text{Stress} = \frac{\text{Force}}{\text{Area}}$$



Wire corrodes → area decreases → stress increases



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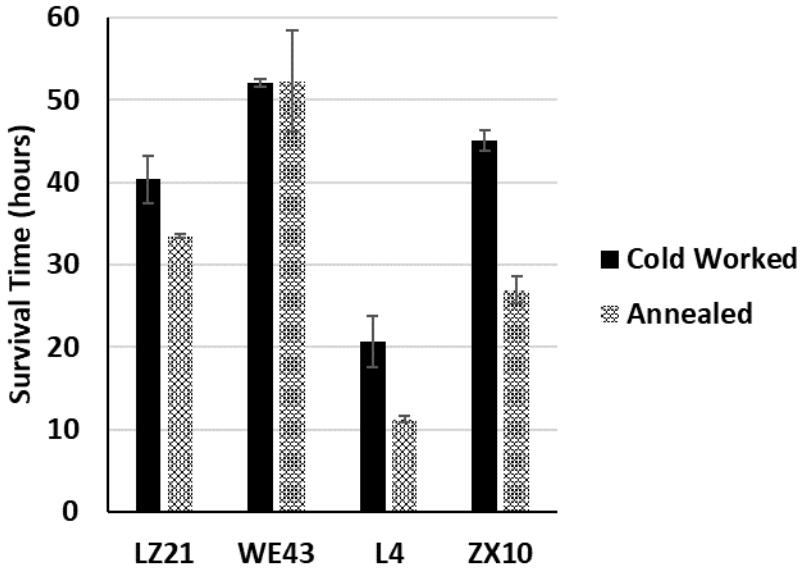
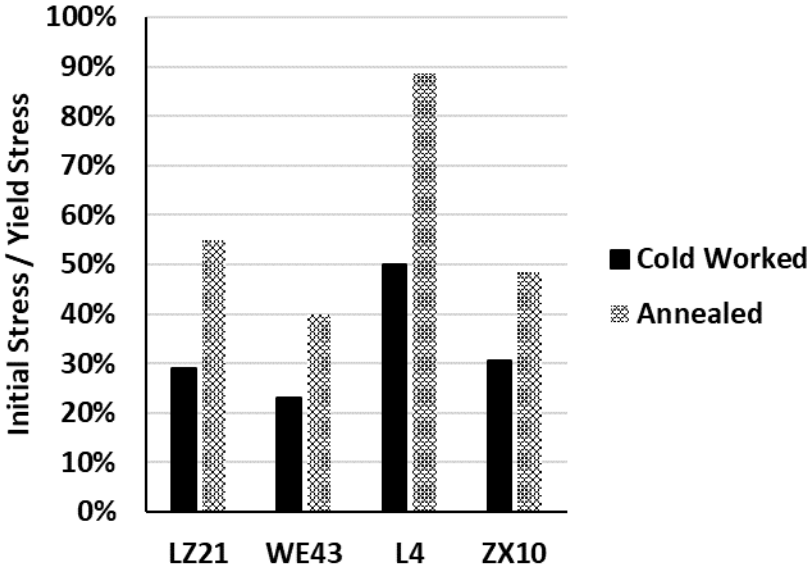
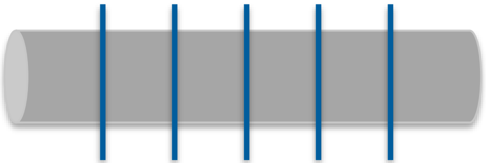
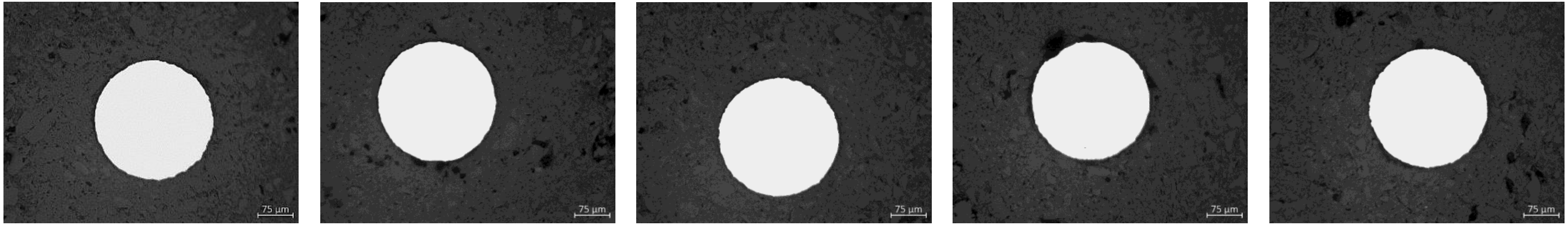




Case 2: Influence of Stress



- 0.25 mm ZX10, cold worked, 16 hours
- Stress = 0, 75, 150 MPa
- Analysis:
  - Collect 5-6 cross-sections (mount/polish)
  - Measure remaining metal area and maximum penetration in ImageJ
    - Calculate Average Penetration and Pitting Factor.



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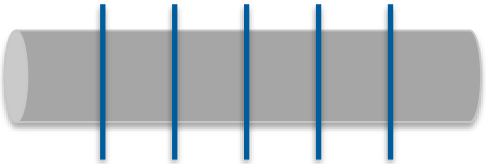

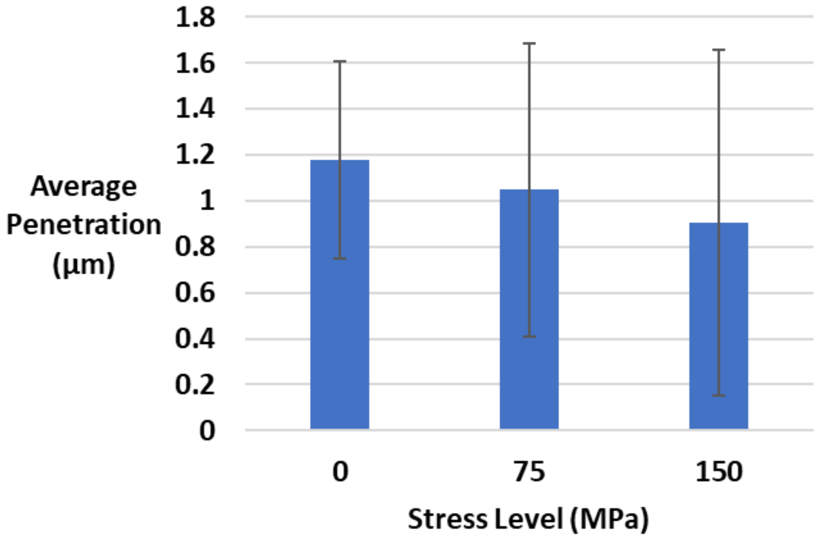

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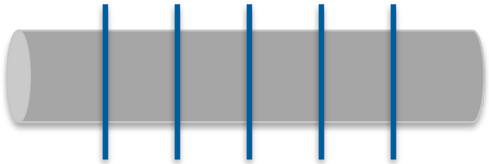
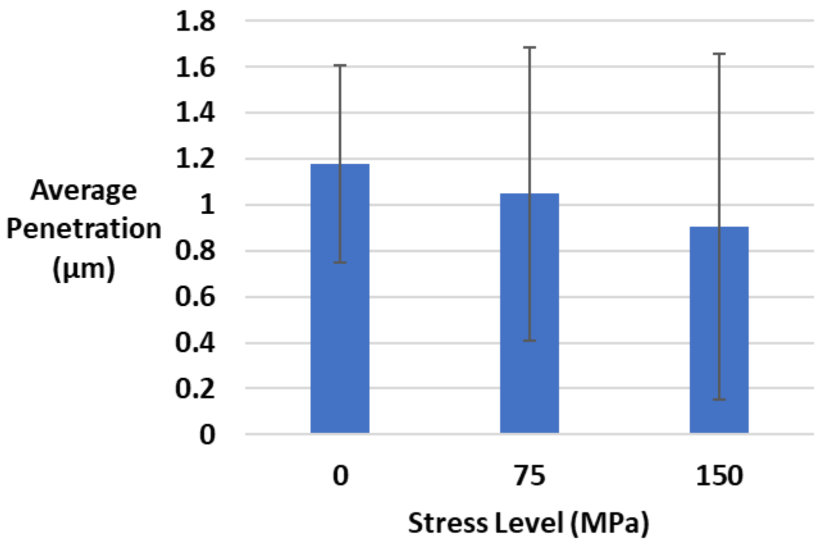




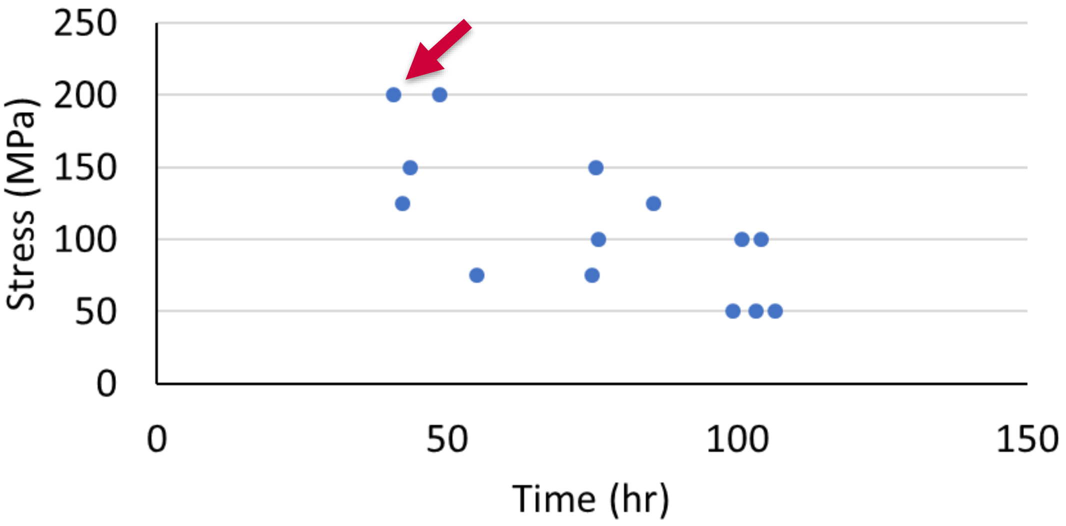
Stress does not increase corrosion rate(?)

Case 3: Stress-Life Curve

- 0.25 mm ZX10, cold worked
- Held at initial stresses of 50-200 MPa
- Outputs:
  - Survival time
  - Cross-sectional analysis

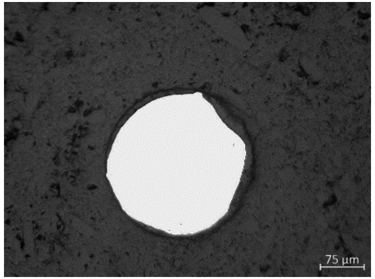
Case 3: Stress-Life Curve

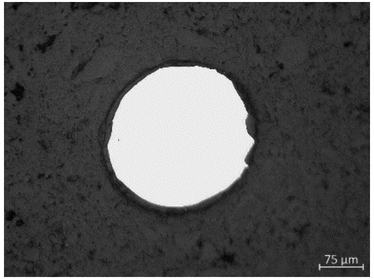
- 0.25 mm ZX10, cold worked

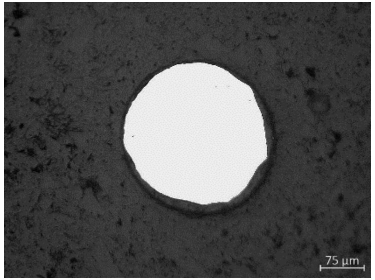


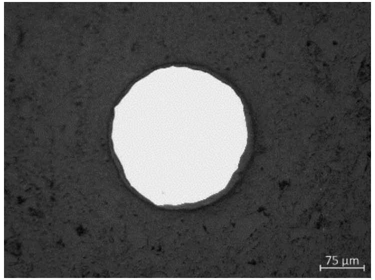
Case 3: Stress-Life Curve

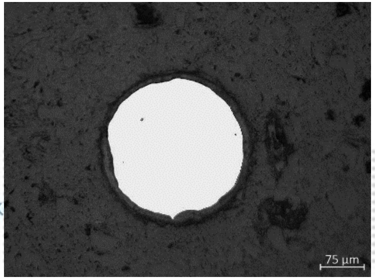
- 200 MPa, 40.8 hours

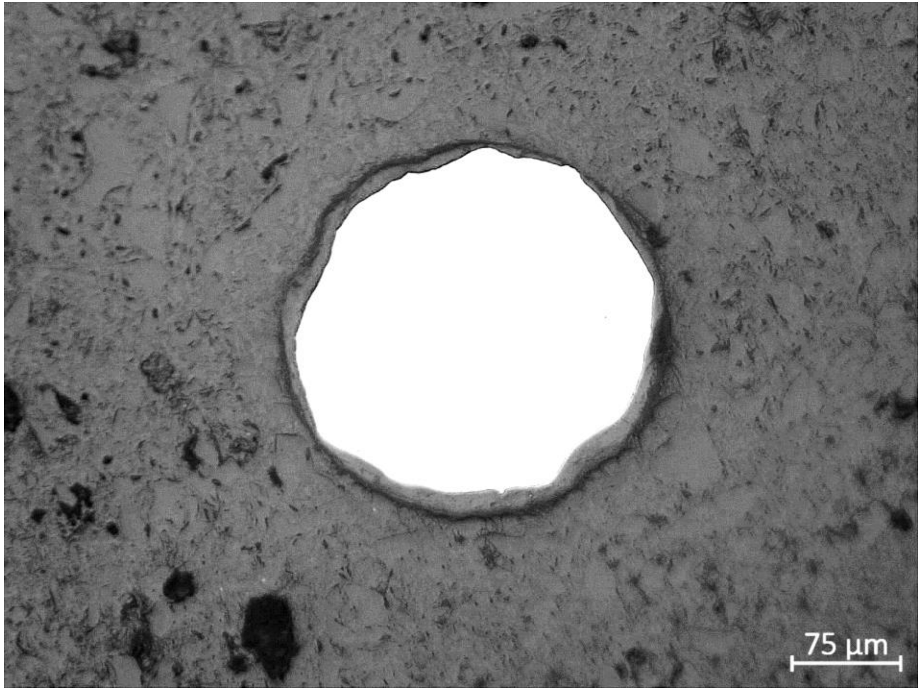






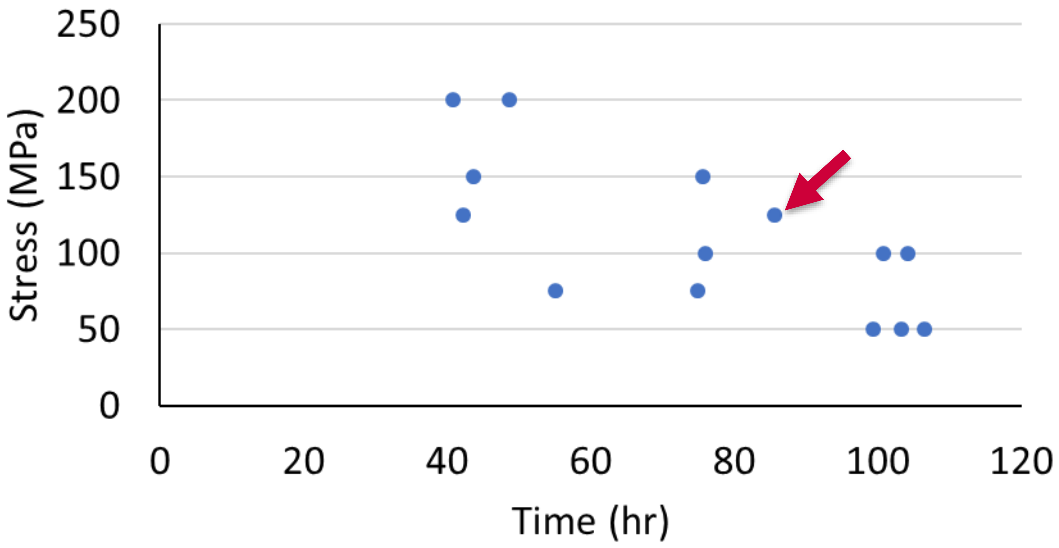






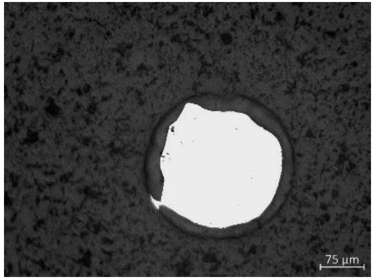
Case 3: Stress-Life Curve

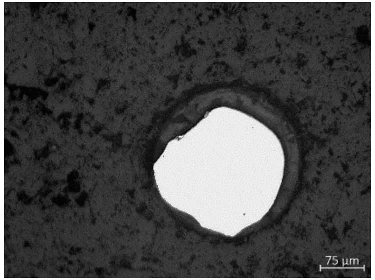
- 0.25 mm ZX10, cold worked

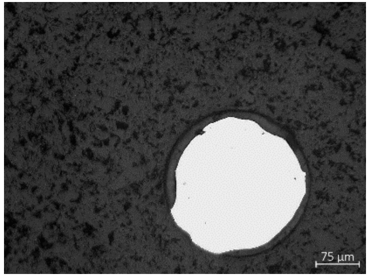


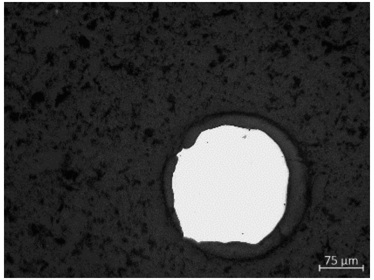
Case 3: Stress-Life Curve

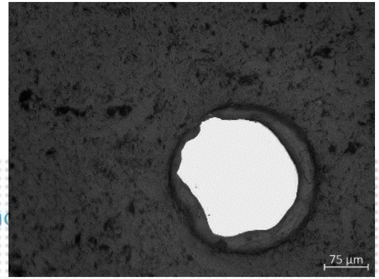
- 125 MPa, 85.6 hrs

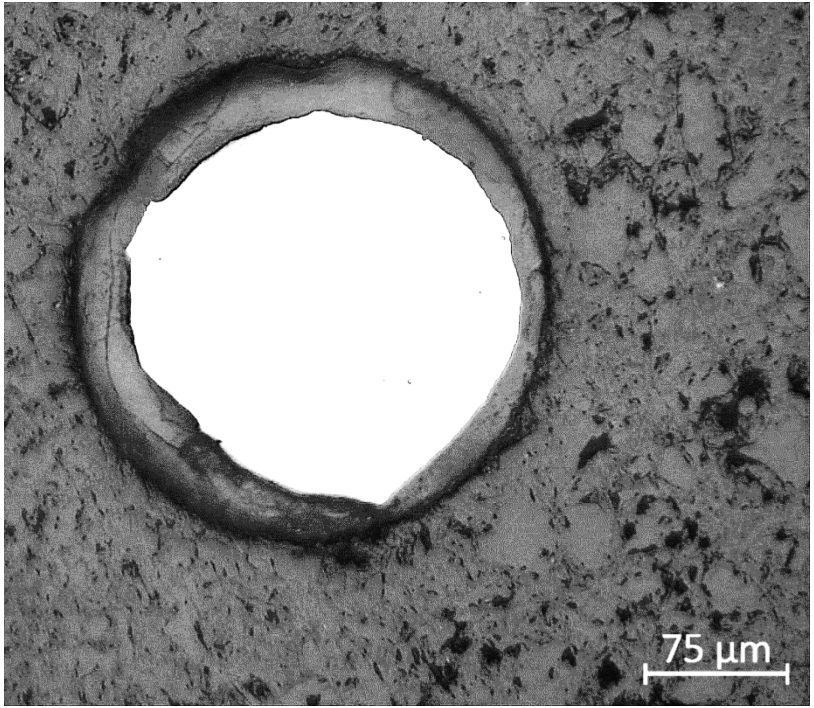






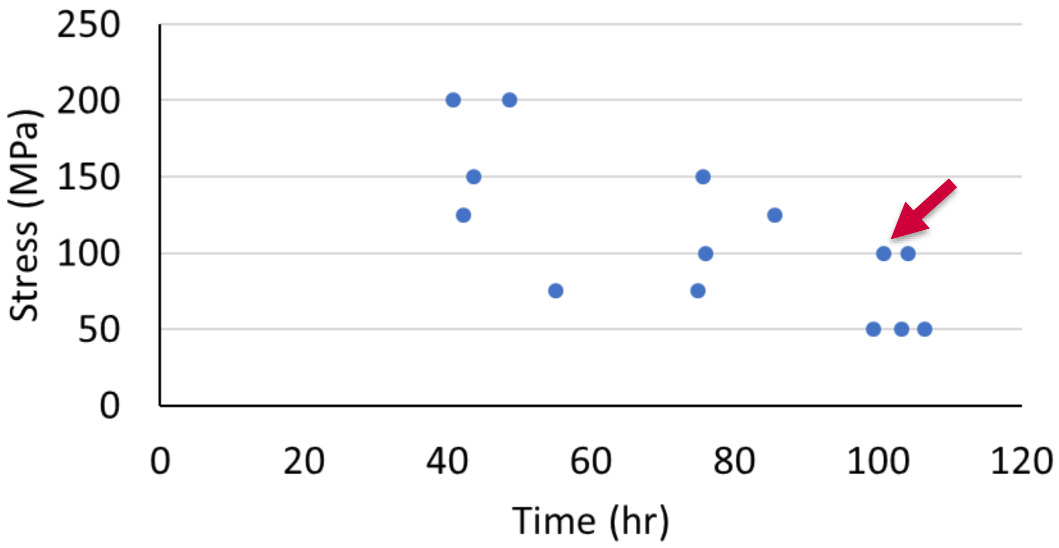






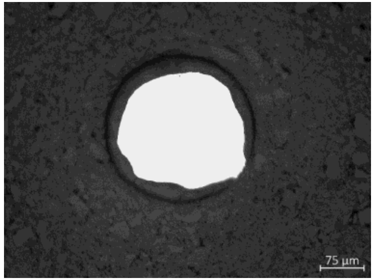
Case 3: Stress-Life Curve

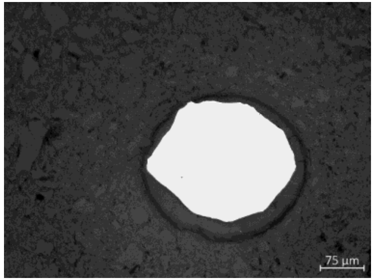
- 0.25 mm ZX10, cold worked

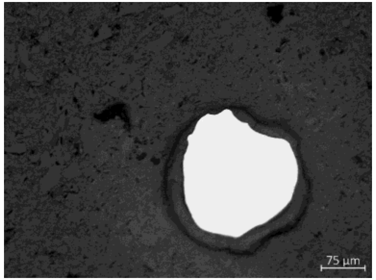


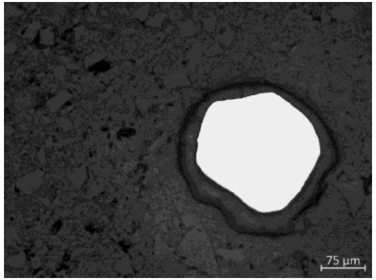
Case 3: Stress-Life Curve

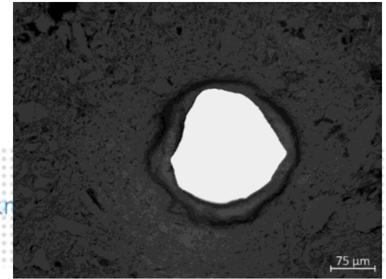
- 100 MPa, 100.8 hrs

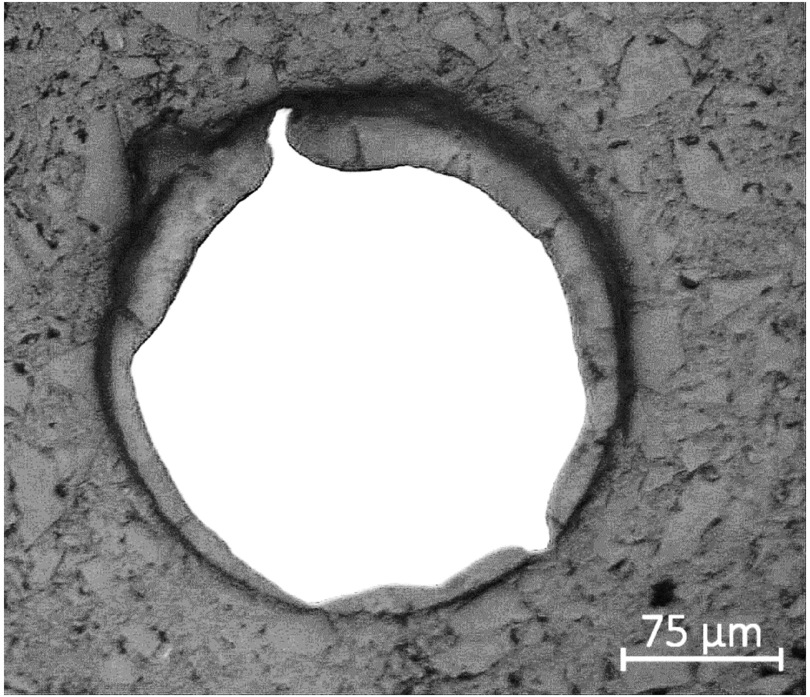






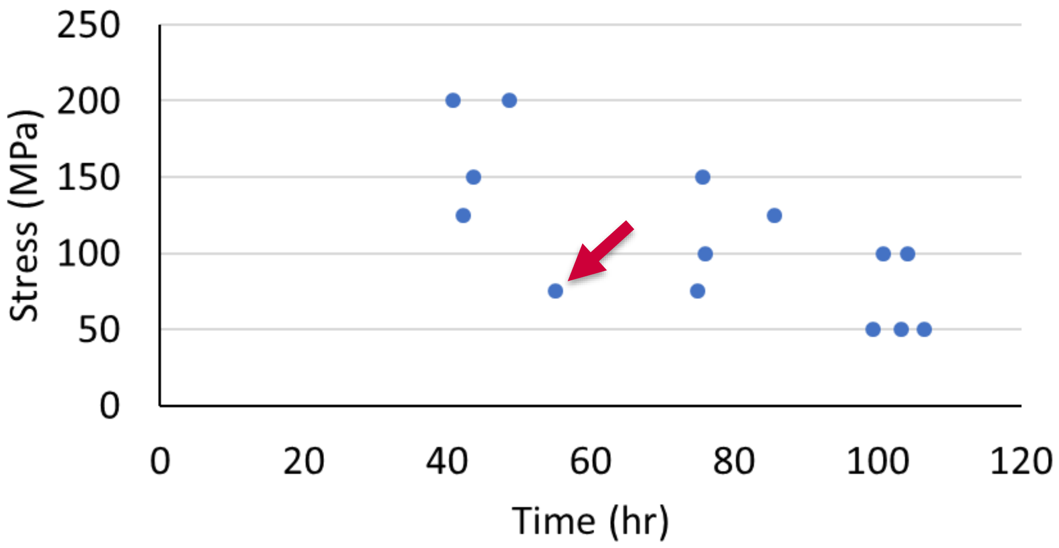






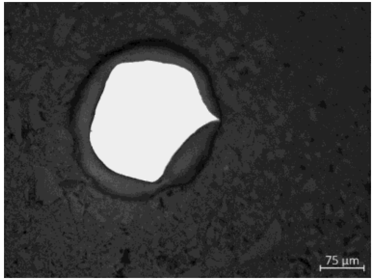
Case 3: Stress-Life Curve

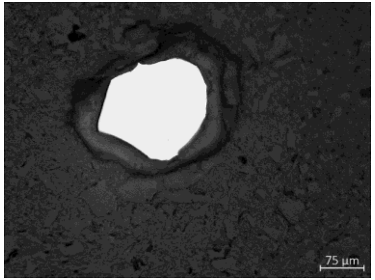
- 0.25 mm ZX10, cold worked

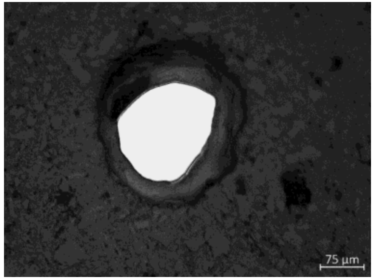


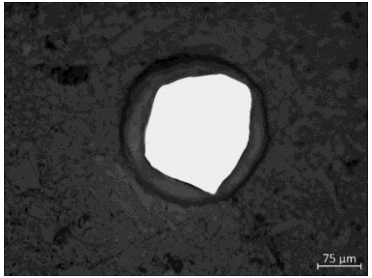
Case 3: Stress-Life Curve

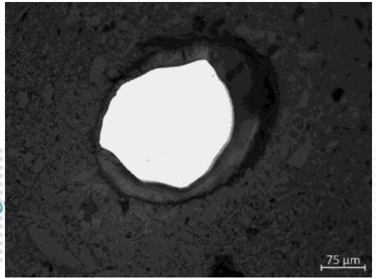
- 75 MPa, 55 hrs

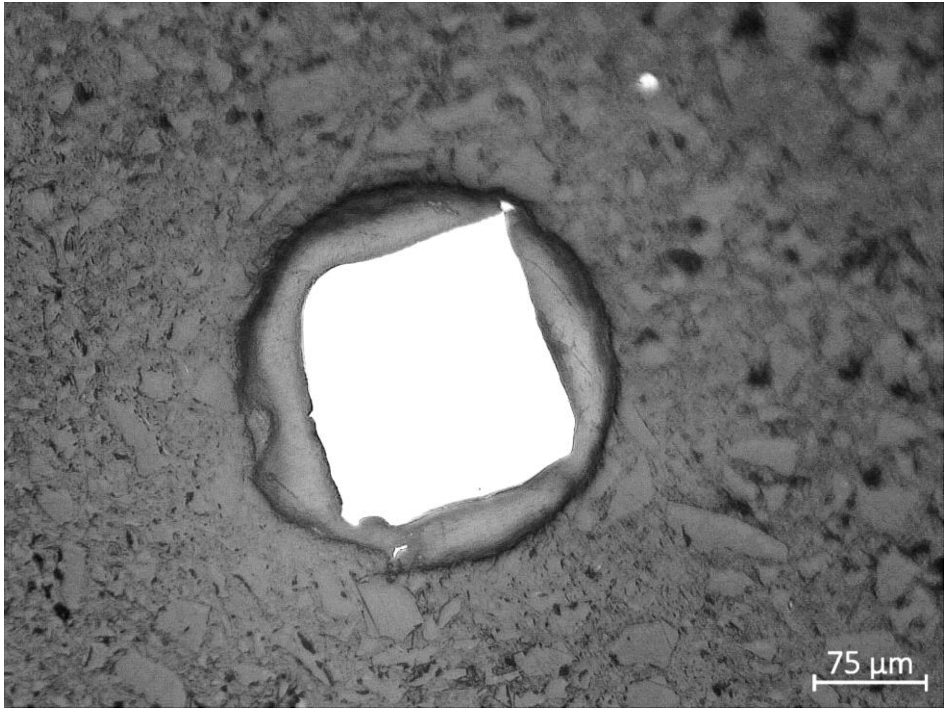




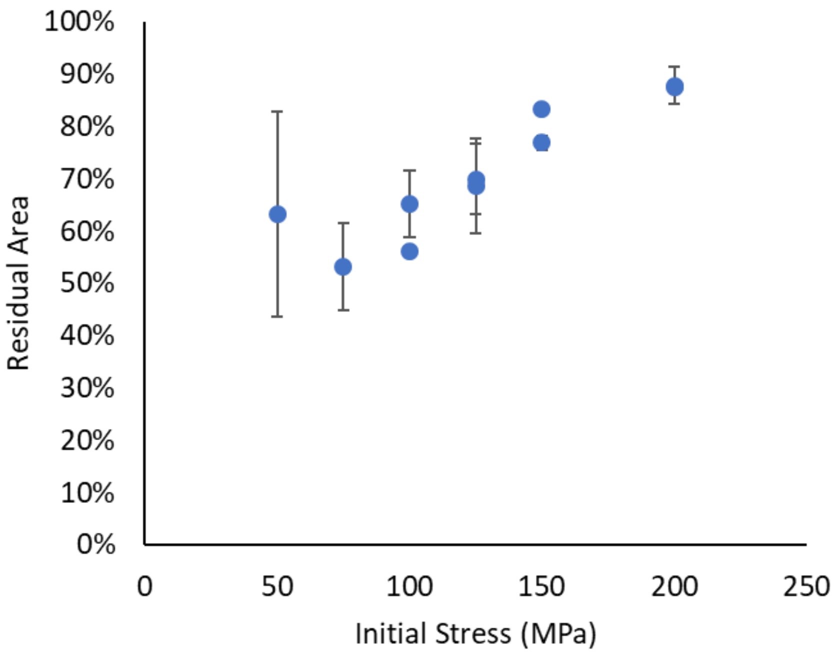


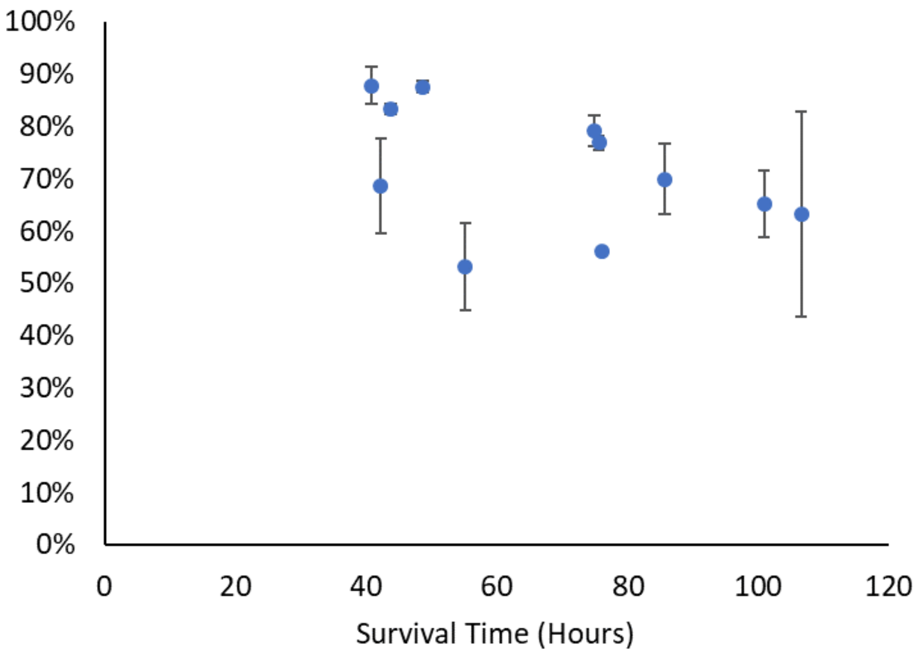




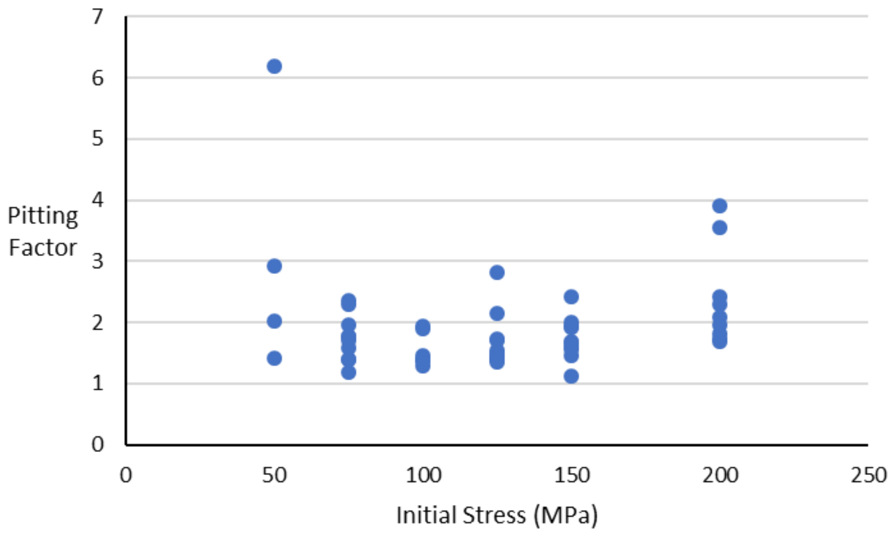


Case 3: Residual Area

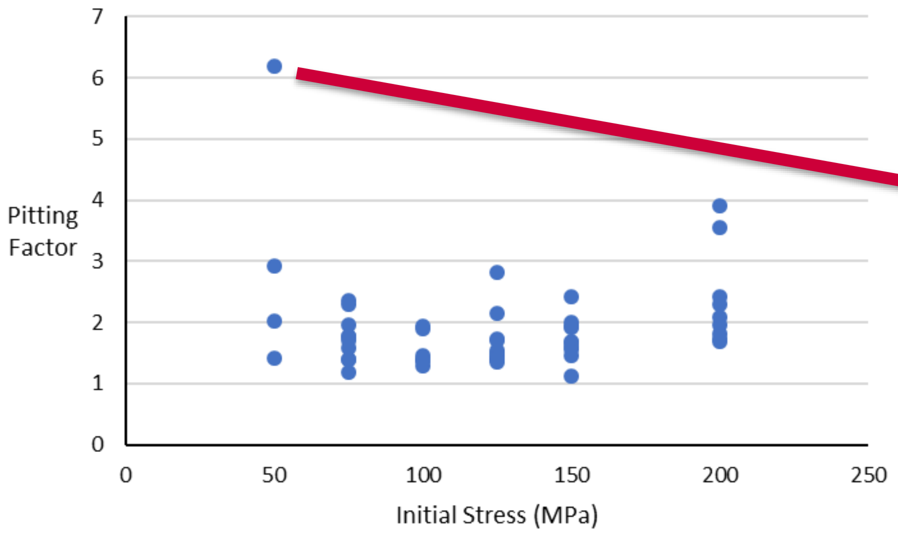


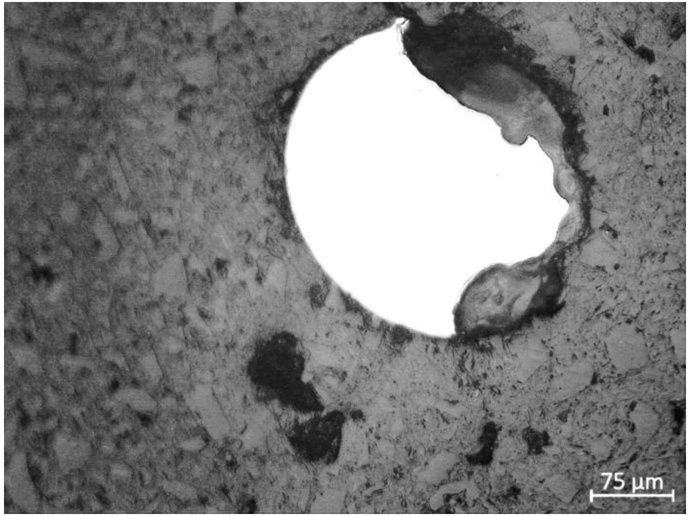


Case 3: Pitting Factor

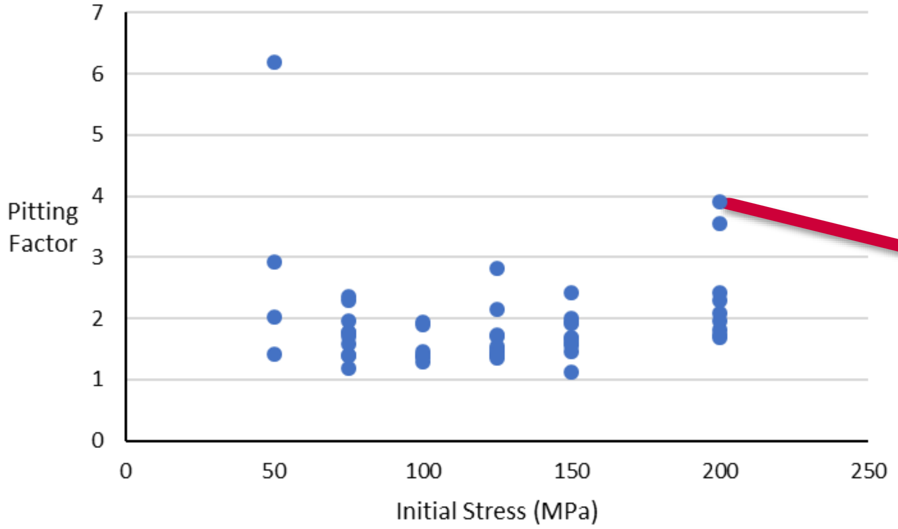


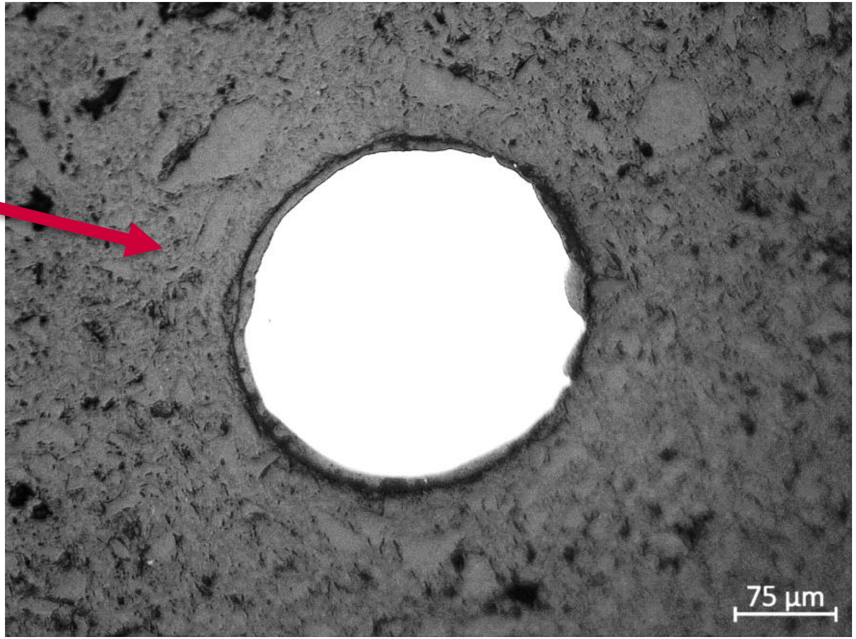
Case 3: Pitting Factor



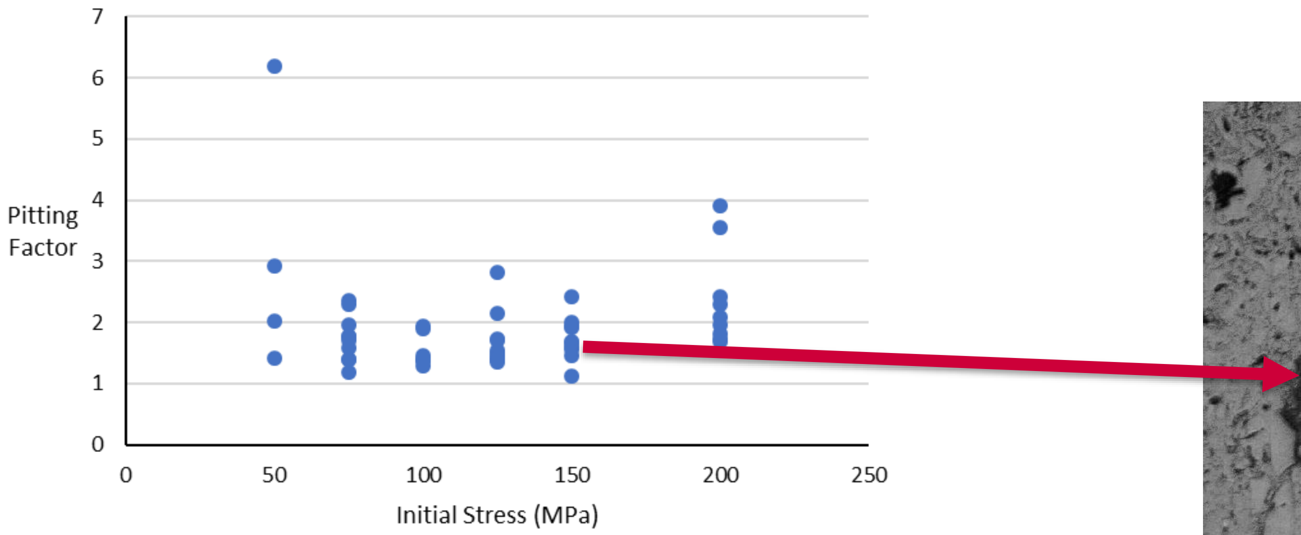


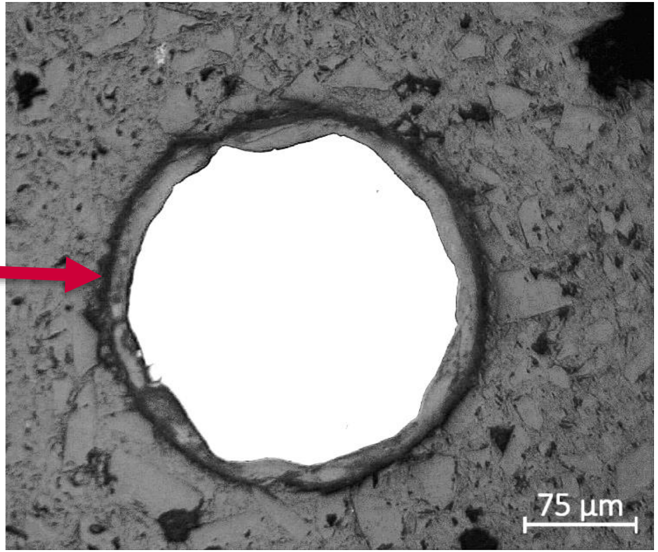
Case 3: Pitting Factor





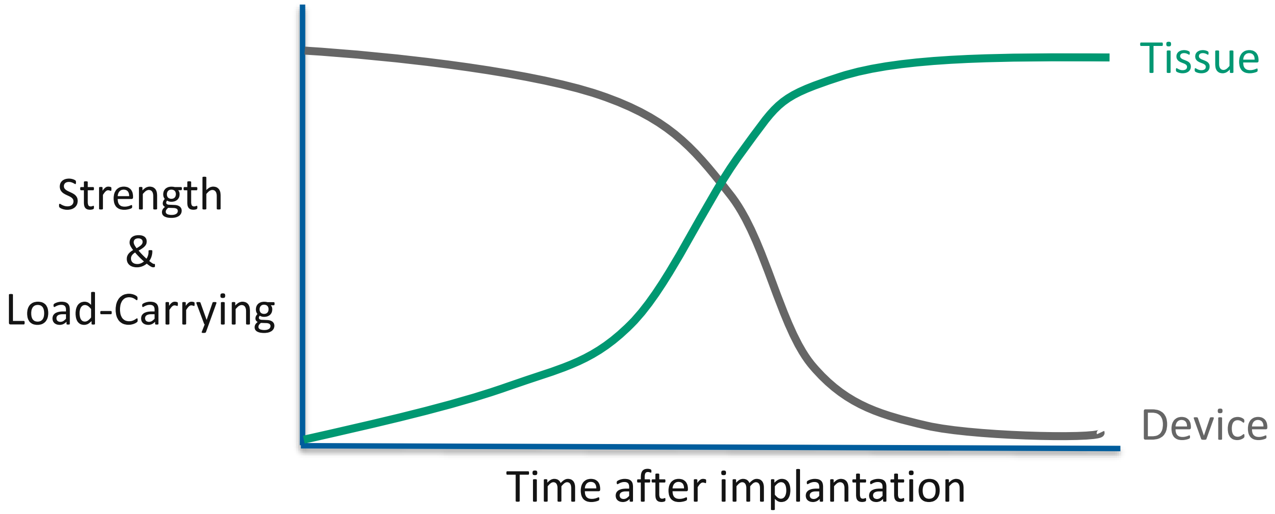
Case 3: Pitting Factor



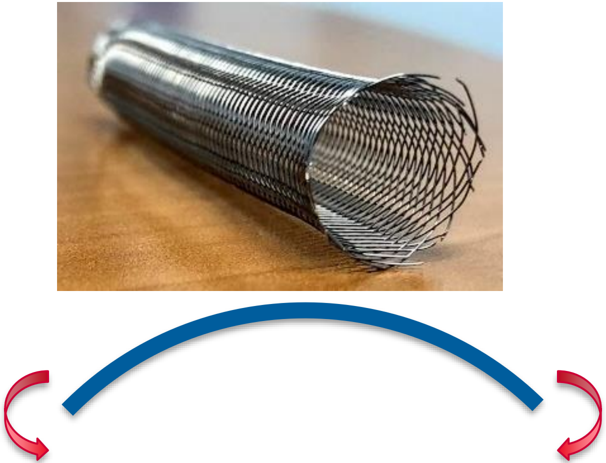


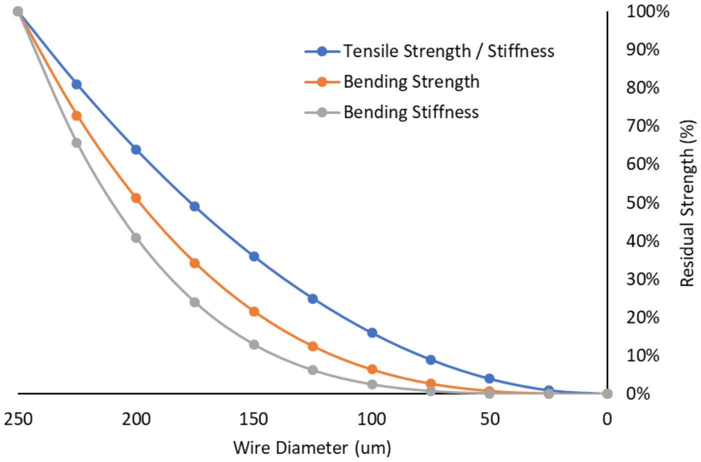
How does this aid in the development of absorbable devices?

Mechanical Transfer



Actual loading depends on device





Future work

- Case 1: Comparing Alloys
  - Confounding influence of strength
  - Hold constant time (pre-failure)
- Case 2: Does stress increase corrosion?
  - Longer duration (e.g. 36 hrs)
  - Additional alloys
- Case 3: Stress-Life curve
  - Additional alloys
  - Higher N
- Additional Studies
  - Microstructure vs corrosion (grain size, secondary phases)
  - Effect of impurities (Fe/Ni/Cu)
  - Surface/Coatings
  - Mechanical strength after set time

Conclusions

- A simple stress-corrosion system was designed for testing of Mg alloy wires.
- The method
  - Effectively distinguishes Mg alloys and process conditions
  - Enables corrosion uniformity assessment
  - Will allow for efficient corrosion checks in a manufacturing environment

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ISO/TS 20721

Implants for surgery — General guidelines and requirements for assessment of absorbable metallic implants

GENERAL INFORMATION

Status : Under development **Publication date :** 2020-09

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ICS : 11.040.40 Implants for surgery, prosthetics and orthotics

LIFE CYCLE

A standard is reviewed every 5 years

00 › 10 › 20 › 30 › 40 › 50 › 60.00 Publication › 90 › 95

Thank you!

See you in 2021!

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