REACTIVE INNOVATIONS

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An Electrochemical Probe for Characterizing Fatigue Damaged Metal Components

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The Importance of Characterizing Fatigue Damaged Metal Components

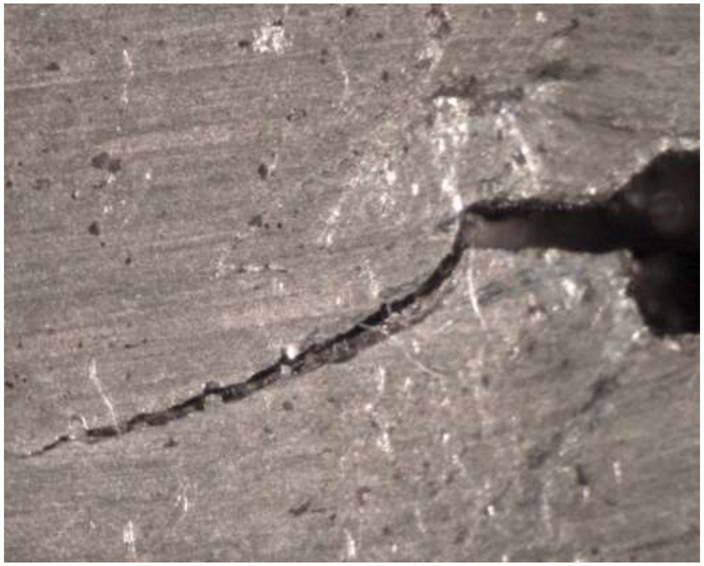
- The military needs an improved inspection system to detect, monitor, and characterize cracks and corrosion on titanium, steel, and aluminum components used in military equipment, vehicles, aircraft, and ships
- Nondestructive evaluation (NDE) methods for detecting and monitoring cracks are needed to not only detect cracks, but to characterize them for how they formed
    - whether from a one-time static load, or
    - as a result of repeated cyclic loads, or
    - from stress-corrosion cracking
- It is desired to have a hand-held device that can operate in-service preventing the component from being removed or excised from the vehicle or aircraft

Basis for Technical Approach

- Since heterogeneous chemical reactions occur at a nanoscale level, a size much lower than the grain size in a metallic component, one methodology to assess the surface morphology is to treat it like a catalyst and force a chemical reaction on it:
  - The surface morphology is dependent on the level of damage as a result of stress, fatigue, and/or corrosion that changes the grain structures locally,
  - A heterogeneous chemical reaction is dependent on the surface morphology like a catalyst,
  - Thus, the type and degree of damage in a component will affect how that surface behaves in a chemical reaction
- So how do you induce a chemical reaction?
  - Use electrochemistry to split water on the surface

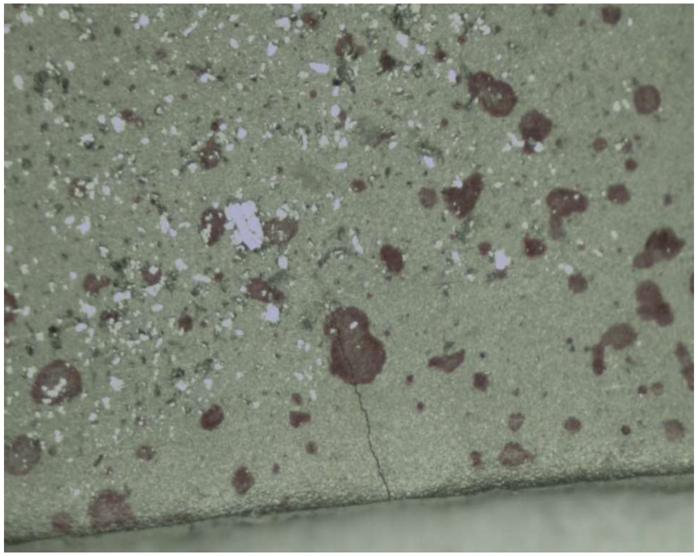
Representative Crack-Induced Images

Al 2024 static load induced crack at 8000 lb_f



- Static Load-Induced Cracks
  - Notch Coupon
  - Secure in Jig with L-piece
  - Apply Force to L-piece
    - 4000 lb_f
    - 6000 lb_f
    - 8000 lb_f

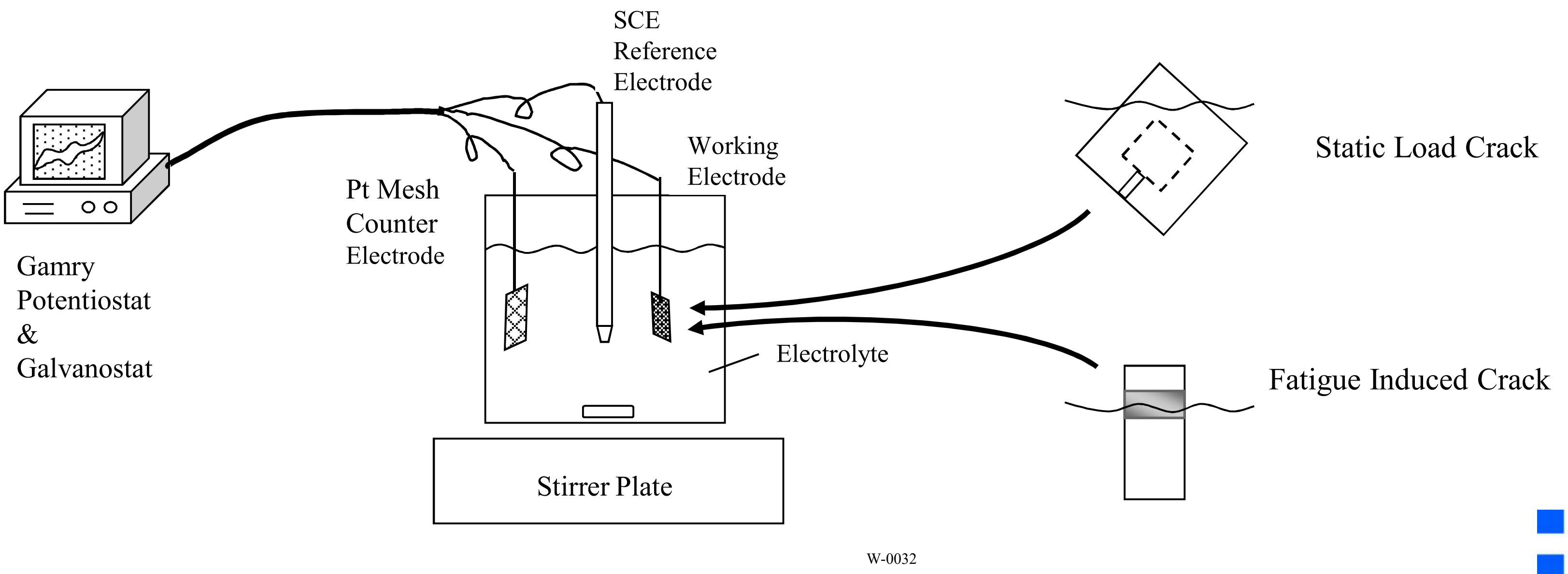
4130 Steel fatigued after 108,000 cycles



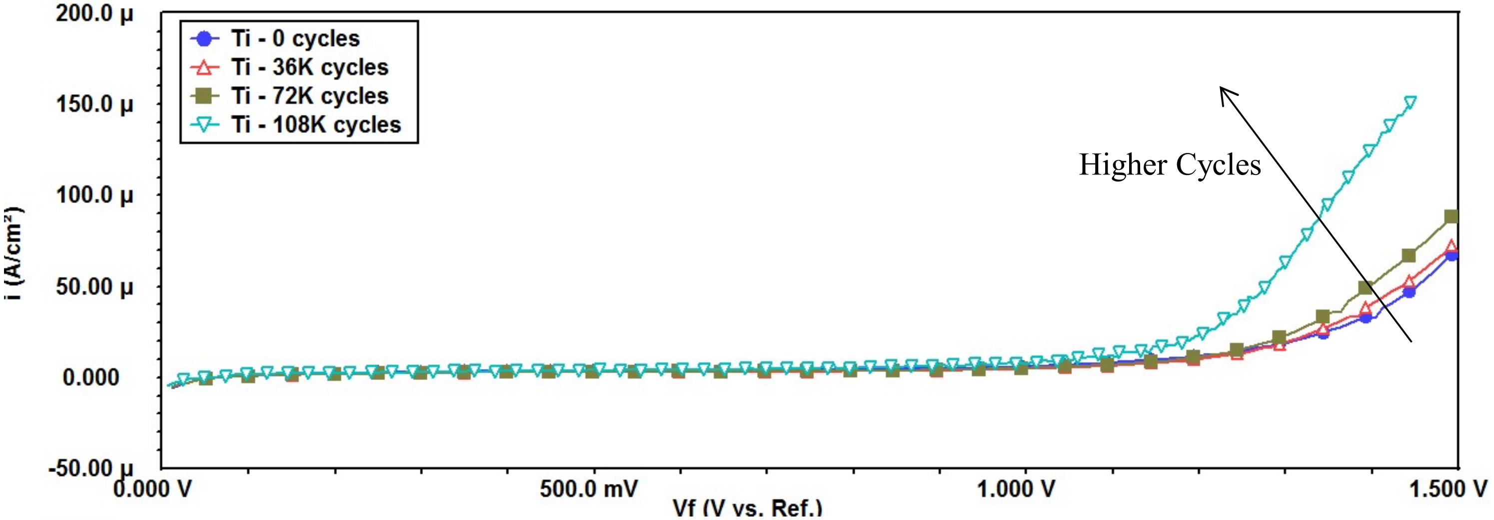
- Fatigue Induced Cracks
  - Secure in Fatigue Device
    - Parallel to Strike Piece
  - Run Fatigue Device
    - 1 hour – 18,000 cycles
    - 2 hours – 36,000 cycles
    - 4 hours – 72,000 cycles
    - 6 hours – 108,000 cycles

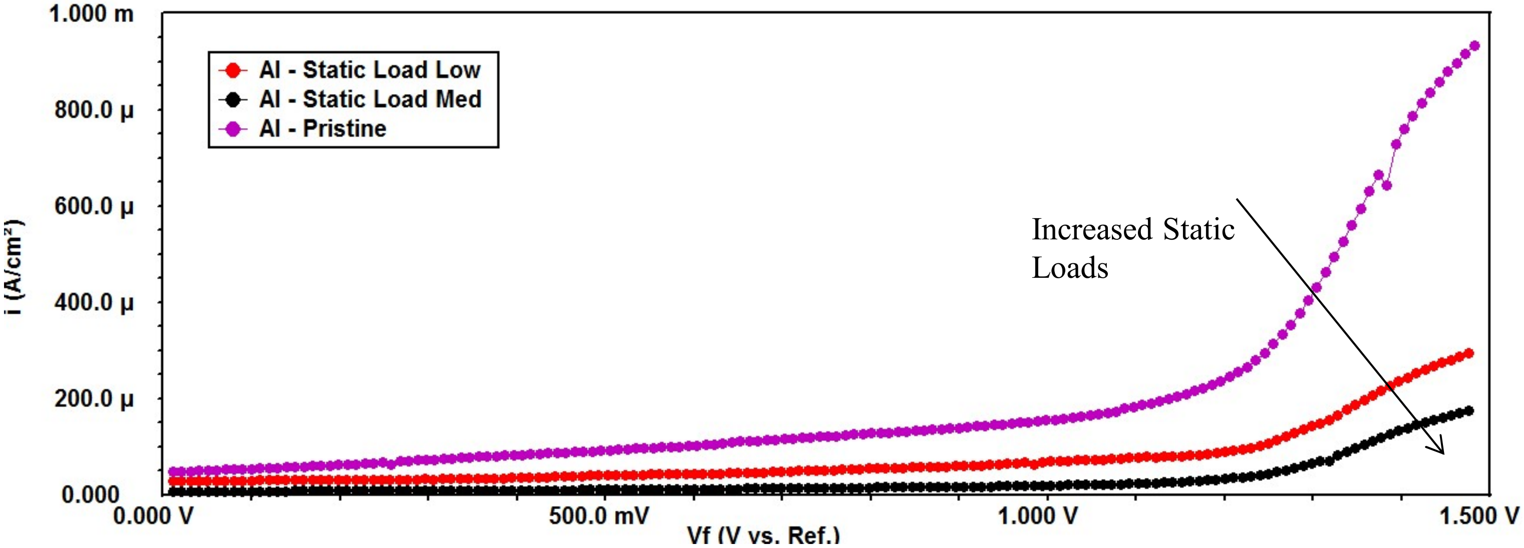
Fundamental Electrochemistry Investigation

- Fundamental question: can electrochemistry be used to interrogate cracked-damaged coupons of aluminum, titanium, and steel?
- Use the classic “wet-electrochemistry” method where the coupon is immersed in a beaker with other electrodes

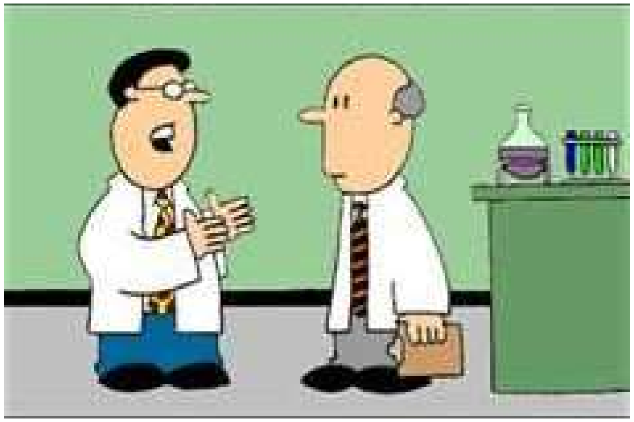


Cyclic Voltammetric Responses With O_2 Evolution for Ti Fatigue and Al Static Load


Higher Cycles


Increased Static Loads

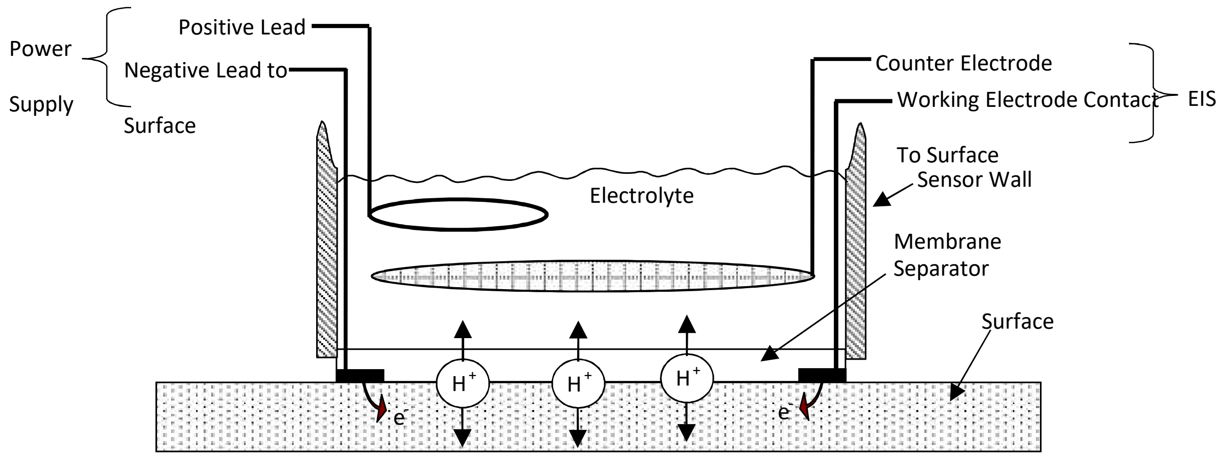
Now That We Know That Electrochemistry Can Detect Cracks, How Do We Apply it to Realistic Hardware?



It’s really simple, all we need now is a bigger beaker

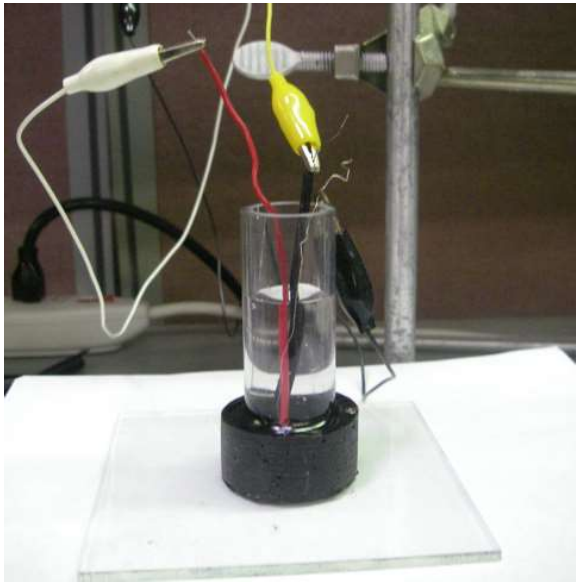
In-Situ Crack Probe Design

- An *in-situ* crack sensor was developed that packages the three-electrode system used in “wet-electrochemistry” into a probe
  - Consists of a membrane transducer that has a platinized current collector ring around its outer perimeter where the membrane transducer is mounted to a cylindrical tube
- The ion-exchange membrane allows protons to travel through it while electrons travel via a perimeter contact junction to the substrate component.

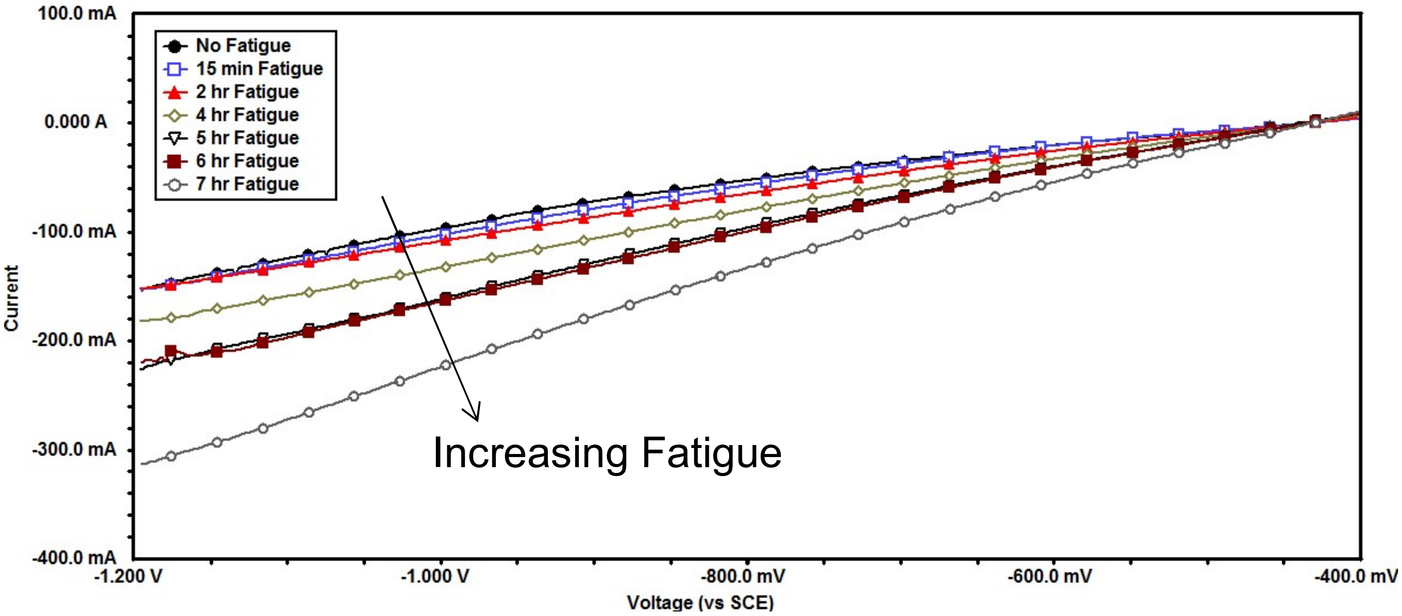


Crack Probe Components

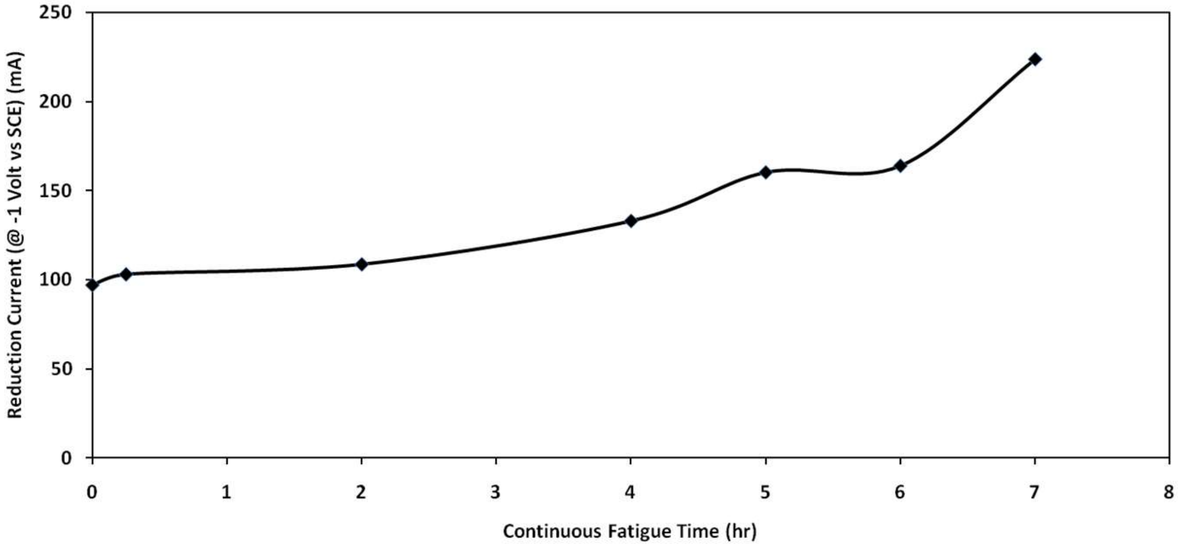
- Working electrode current collector
  - Pt catalyzed ring
- Reference electrode
  - In-house made Ag/AgCl electrode
- Counter electrode
  - In-house made Platinized mesh electrode
- Electrolyte
  - 0.5M H_2SO_4



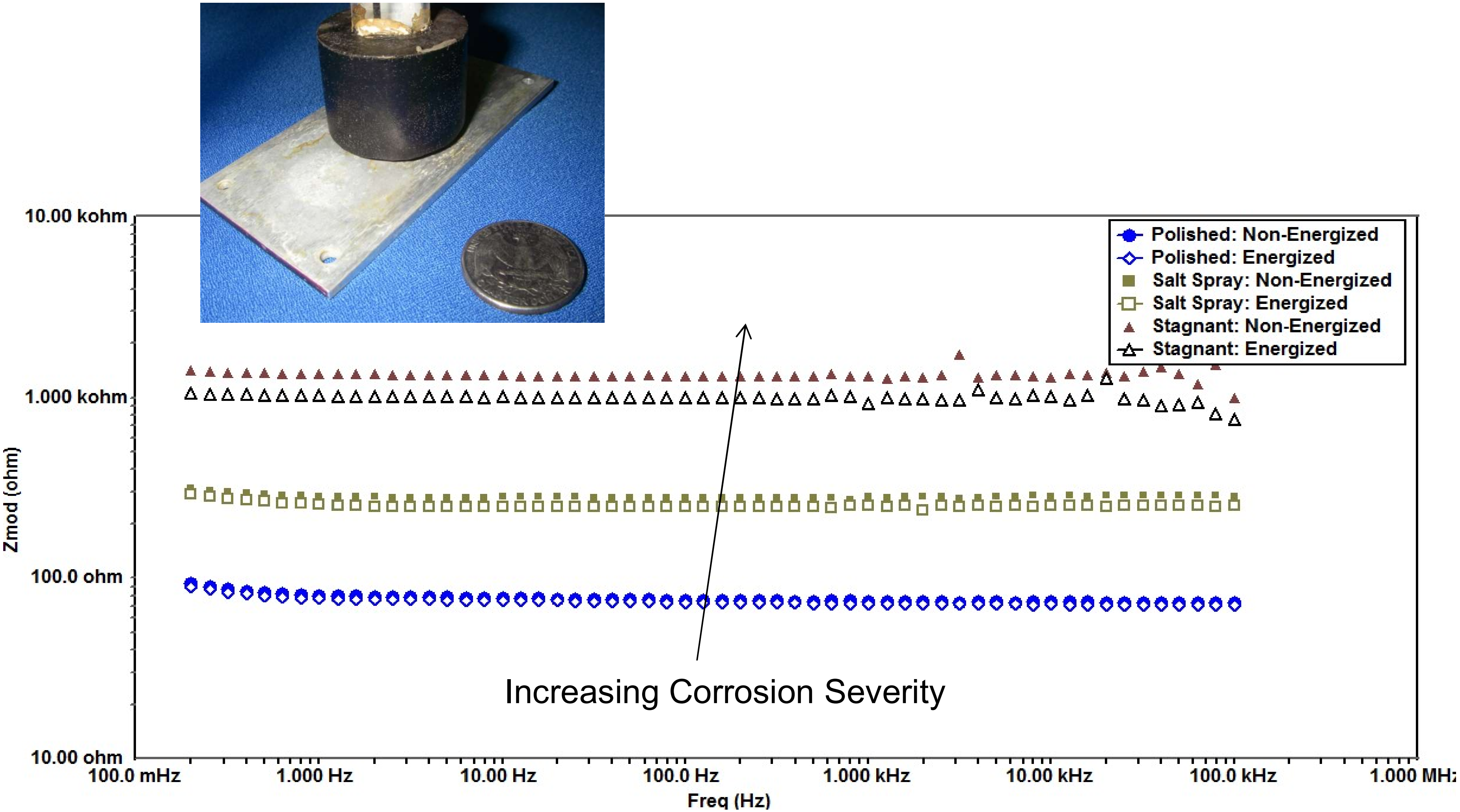
Electrochemical Response on Fatigued High Strength Steel



Increasing Fatigue

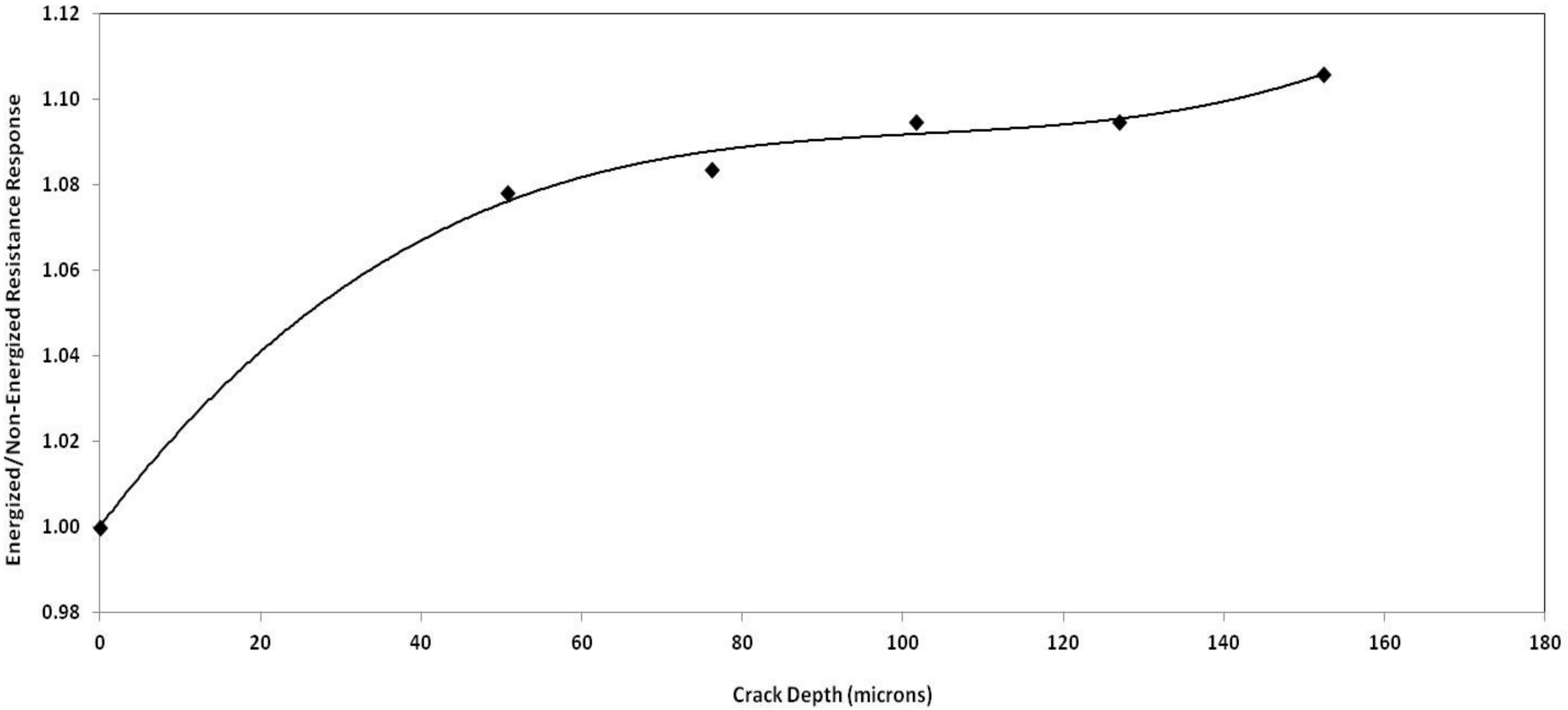


Impedance Response for Energized (Gas Evolution) and Non-Energized (No Gas Evolution) for Different Degrees of Corroded Aluminum (Al2024-T3)

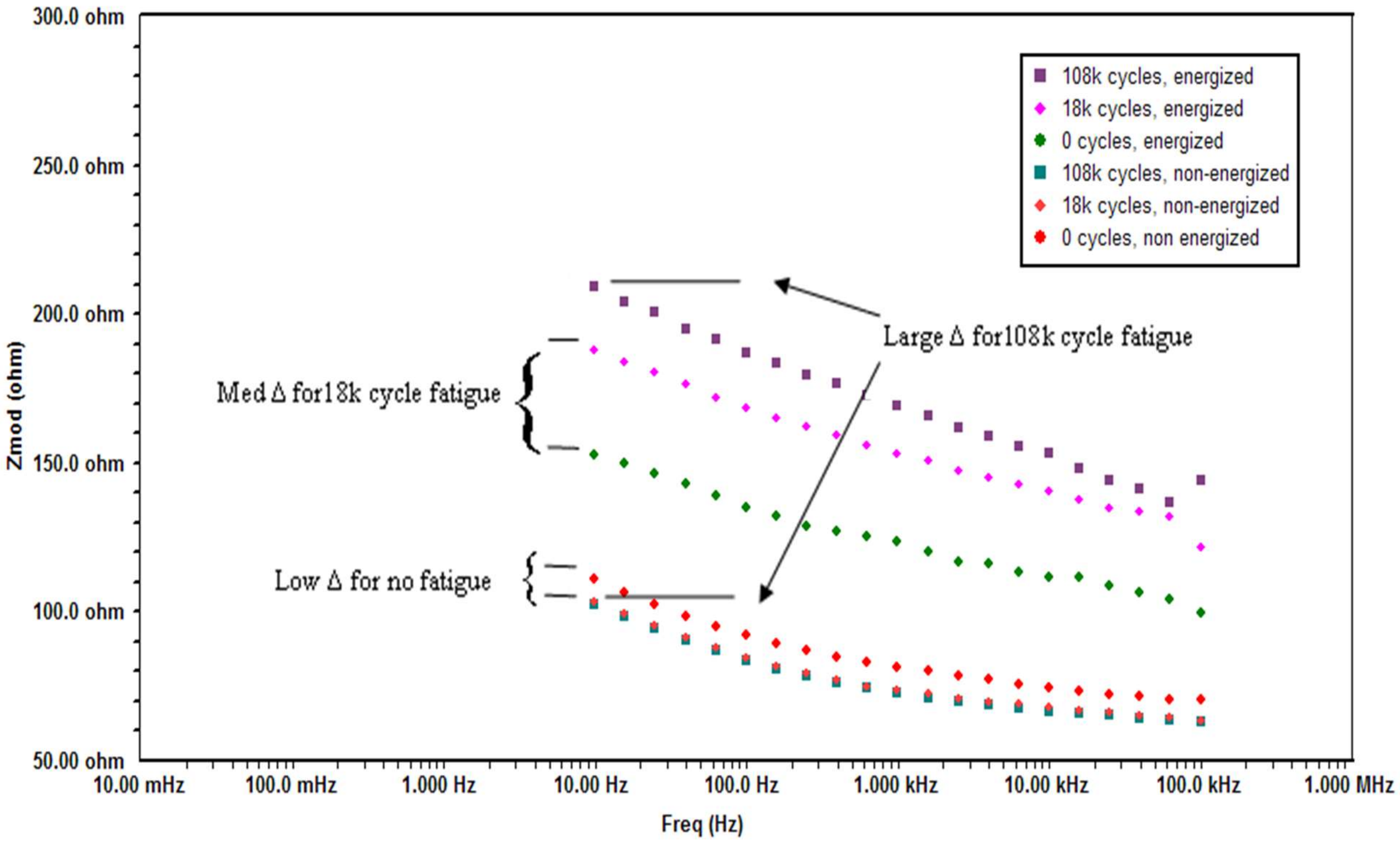


Increasing Corrosion Severity

Correlation of Reactive EIS as a Function of Crack Depth in Al2024-T3



Reactive EIS Response for Three Levels of Increasing Fatigue

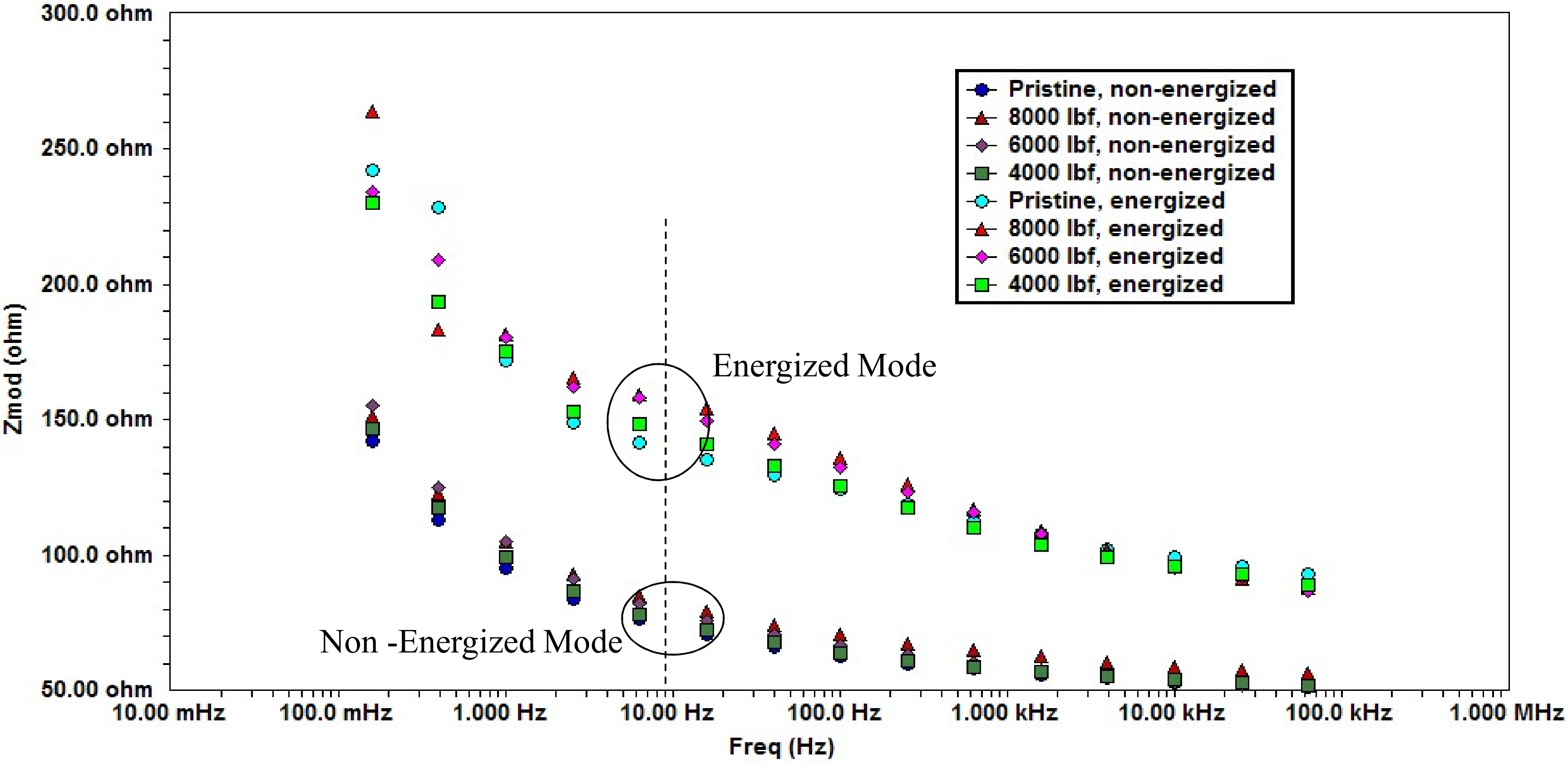


Large Δ for108k cycle fatigue

Med Δ for18k cycle fatigue

Low Δ for no fatigue

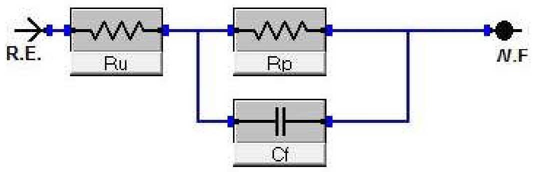
At 10 Hz, Differential Impedance (Energized – Non-energized) Increases for Al Coupons as Static Load Increases



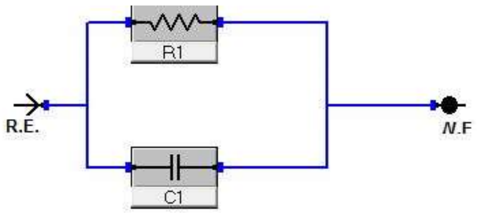
Static Load (lbf)	Delta Impedance (ohms)
Pristine	65.01
4000	69.42
6000	73.99
8000	75.08

EIS Spectrum Modeling Using Equivalent Circuits Helps Determine the Cause of Damage

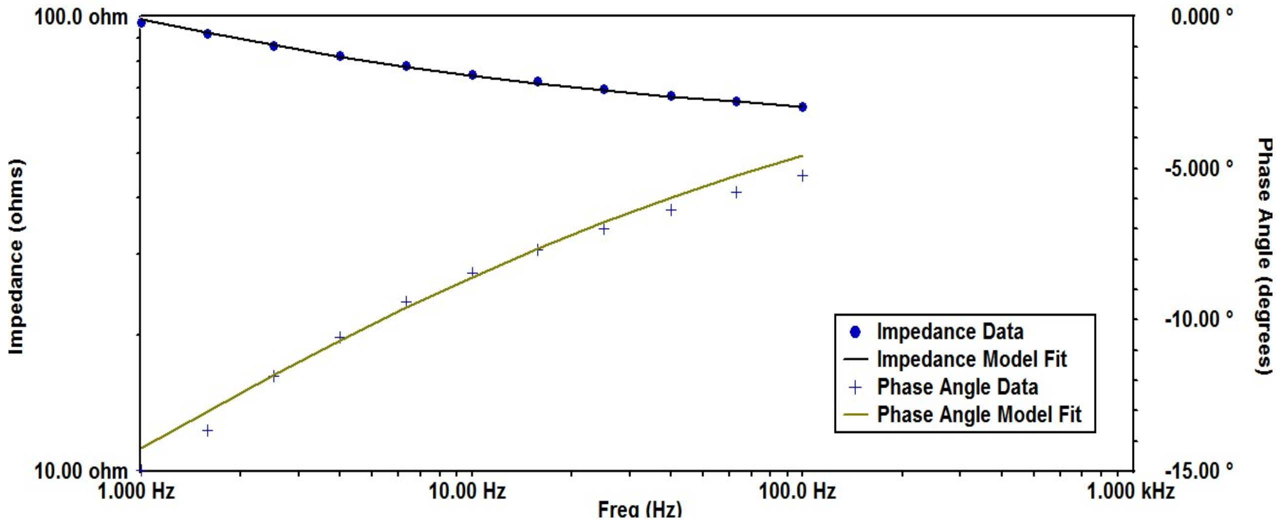
- Equivalent circuit models were made for Randle's and R circuits to numerically regress the circuit parameters (R_p C_f, R_u)
- Regressed the EIS spectrum over the 1 Hz to 100 Hz range
- Compared the parameter estimate values of the energized to non-energized tests for each crack type to create trend directions



Randle's Equivalent Circuit Model



R-C Equivalent Circuit Model



Reactive EIS Interrogation on Al 2024 Shows Distinct Parameter Trends for the Type of Damage

Static-Load Damage

Model	Parameter	Trend Direction of the Parameter (from Pristine to Damage)
Randles	Rp	Up
	Ru	Down
	Cf	Down

Fatigue-Induced Damage

Model	Parameter	Trend Direction of the Parameter (from Pristine to Damage)
Randles	Rp	Up
	Ru	Up
	Cf	Up

Stress-Corrosion Cracked

Model	Parameter	Trend Direction of the Parameter (from Pristine to Damage)
Randles	Rp	Down
	Ru	Down
	Cf	Down

Corroded

Model	Parameter	Trend Direction of the Parameter (from Pristine to Damage)
Randles	Rp	Down
	Ru	Down
	Cf	Up

The three parameter model gives distinct parameter trends for the type of damage present

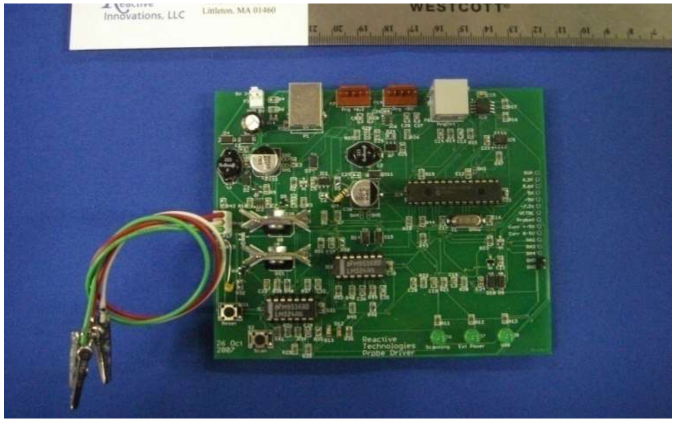
Summary

- Three representative aerospace metals were examined that include aluminum 2024, high strength titanium grade 5, and high strength 4130 steel alloy
  - Pristine and damaged coupons evaluated including those via static-load, fatigue, stress-corrosion cracking, and corrosion
- Fundamental investigations using traditional wet-electrochemistry show electrochemical trends due to type of damage and degree of damage
- Operational probe assemblies developed with 1” diameters
- Reactive Electrochemical Impedance Spectroscopy shown to differentiate the type of crack present in a metal system
- A methodology was developed to apply the Reactive EIS sensor using a systematic analysis framework for use by a non-electrochemical expert

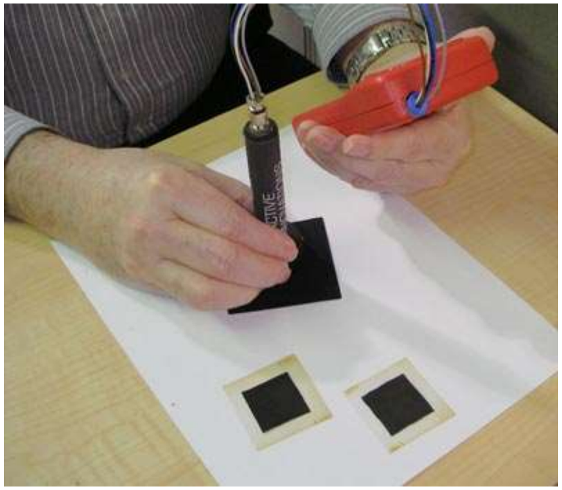
Product Vision

- A portable hand-held probe assembly that will enable an operator to characterize cracks, corrosion, and damage in metallic components, as well as characterize catalytic surfaces

Membrane Transducer Assembly, US Patent #8,221, 603 July 2012



Previously developed compact circuit board for CV interrogations



Acknowledgment

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