

Acceptance Criterion for Hydrogen Embrittlement Testing of Coated Fasteners

by Louis Raymond

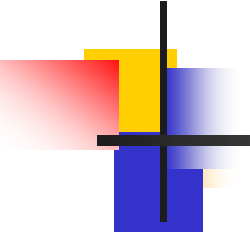
Industrial Fastener Institute Science Award'06, IAE Engineer of the Year'94

Ph.D. UC Berkeley'63, B.S., M.S. Carnegie Mellon University'56,'58

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LRA/FDI/RSL™ —

(LRA) L.Raymond & Associates
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- **Failure/Life/Risk Analysis** — integrates system from research, analysis, testing and quality assurance. Includes Laboratory SEM/EDX, Metallographic, mechanical testing facilities. Specialize in fastener materials, design, testing and analysis.

(FDI) Fracture Diagnostics International, LLC

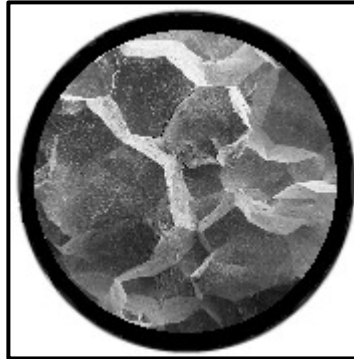
- **Manufactures RSL™** tension and bend testing equipment, including auxiliary environmental and mechanical testing chambers/adapters and ASTM F519/F1940 certified specimens

(RSL™) RSL™ Technology Center

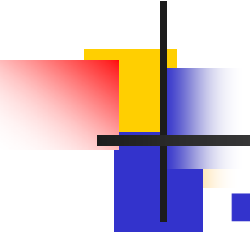
- **Laboratory Testing Services** and hands-on training courses for RSL Testing in air, environment and in both tension and bending. Offers on-site training courses

Hydrogen Embrittlement

- Time delayed fracture in steel due to time-delayed, hydrogen induced cracking under the influence of stress
- Photographic Fingerprint -"IG" Intergranular cracking

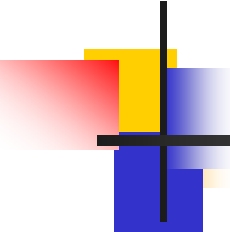


- Just because coated fastener is made "FREE" of hydrogen embrittlement after applying a coating — **NO IHE**; does not mean the at is will not fail by hydrogen embrittlement in service (**EHE**)



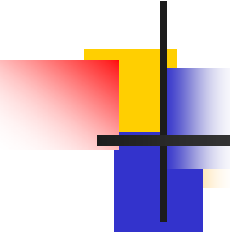
Source of Hydrogen

- **Coatings** on fasteners are a source of hydrogen either,
 - During application of the coating due to coating process
 - Internal Hydrogen Embrittlement (**IHE**)
 - or
 - During service exposure under stress in a moist service environment due to galvanic couple between coating and steel acting as an in situ hydrogen generation pump
 - Environmental Hydrogen Embrittlement (**EHE**)



Acceptance Criterion

- Acceptance criterion for **hydrogen embrittlement** is based on service performance.
- **PROBLEM:** Parts pass the hydrogen embrittlement tests, yet fail in service
- **Objective:** An acceptance criterion that eliminate hydrogen embrittlement failures in service by requiring a **threshold stress** for the onset of hydrogen induced stress cracking (Hydrogen Embrittlement) that has a margin of safety in bending.

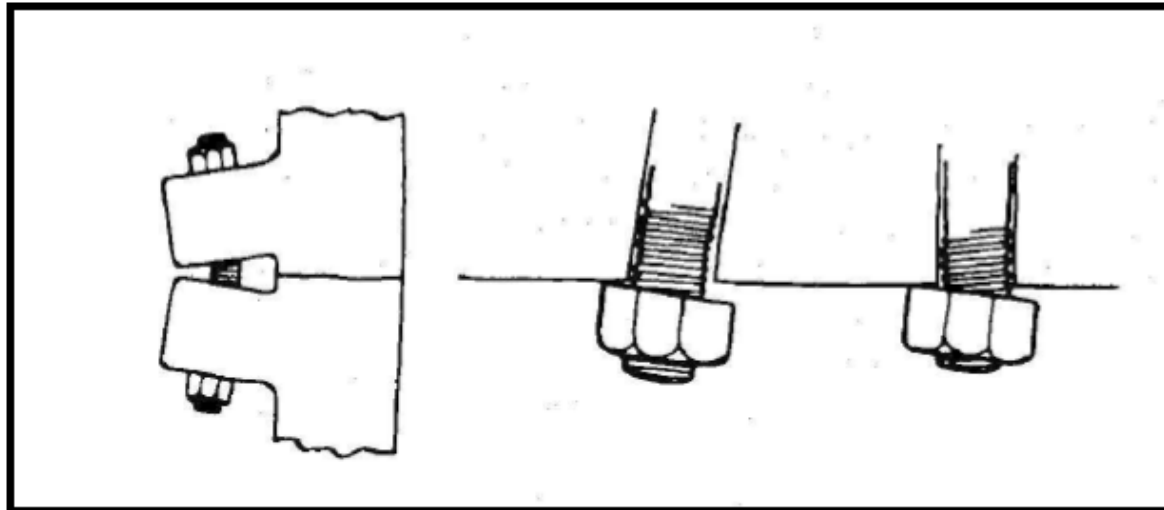


Why is it an issue?

- Many different hydrogen embrittlement test methods — not equivalent
- Pass one test — Fails another or fails in-service
- Main difference — loaded to different stress levels — does not take **bending** into account

Bending

Reason that Bending must be accounted for —



**Because joint and nut surfaces
are never exactly perpendicular to thread axes,
a bolt almost never stretches uniformly when it is tightened;
instead it bends to some degree.***

*Reference: Bickford, John H. "An Introduction to the Design and Behavior of Bolted Joints." Dekker 1990. 2nd Edition p 252.

Causes for Service Failures

- **Processing error – 20%**
 - (Insufficient hydrogen bake-out treatment)
 - **Fault:** Manufacturer
- **Over-torquing – 10%**
 - (Lubricant, wrench)
 - **Fault:** User
- **Bending in combination with Coating – 70%**
 - (Long thin screws, others)+ (Galvanic couple as a hydrogen pump)
 - **Fault:** Design & Coating Selection

Standard IHE Test Methods

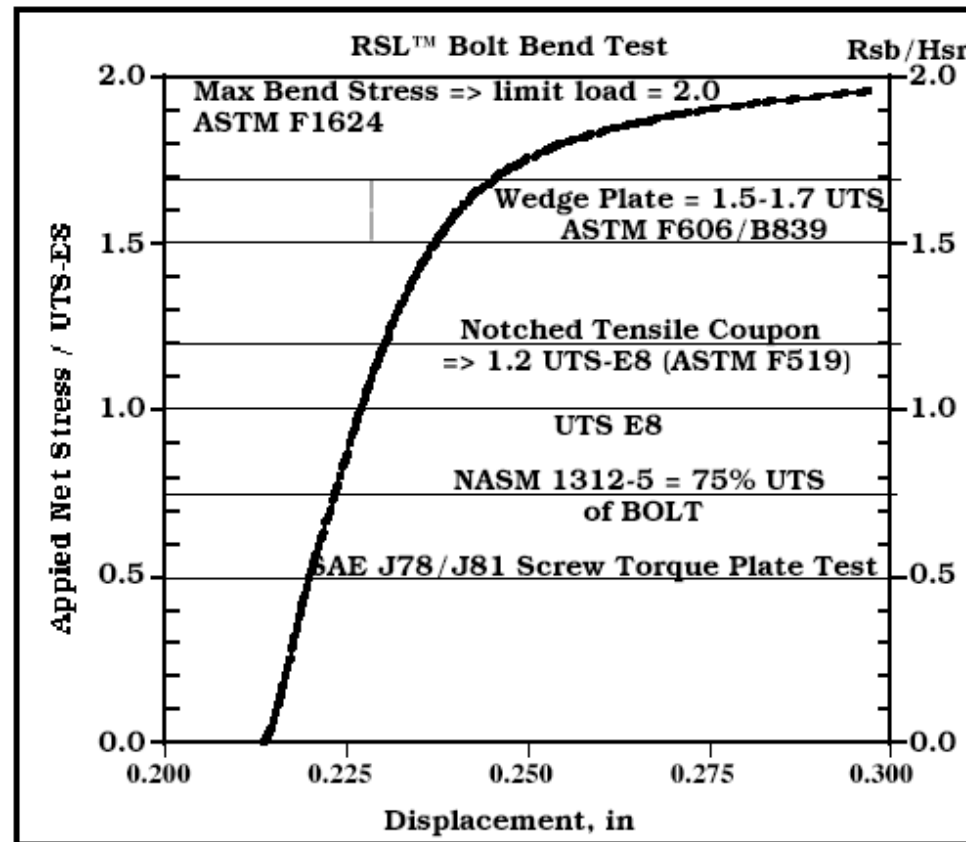
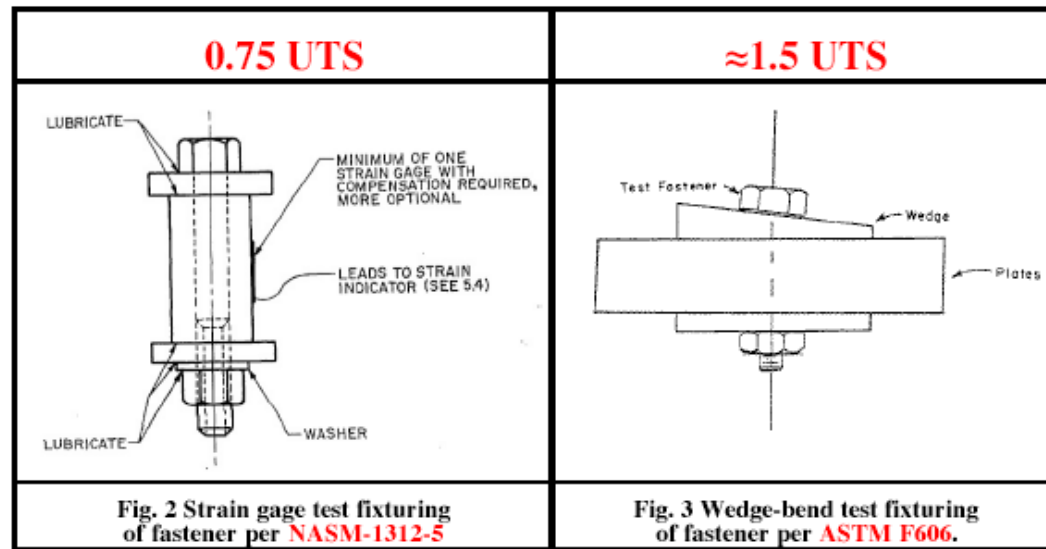


Fig.1 An actual pure bend test on a SAE Grade 5 fastener in an RSL™ Bend Testing Frame to illustrate the ability of a fastener to attain maximum possible local service stresses approaching 230% of the smooth bar yield strength or about 2.0 UTS per ASTM E8. Superimposed are the applied proof stresses corresponding to the various hydrogen embrittlement or stress durability tests currently being used for quality assurance.

IHE Test Fixtures

Fastener self-loading fixtures for fasteners in tension and bend
at **75 % NFS**



Local stress in bending per F606 is about **2X** that of NASM 1312-5.

RECOMMEND: That ≥ 1.2 UTS_{E8} be used as acceptance criterion for hydrogen embrittlement of fasteners or notched round bar tensile specimens.

NOTE: To attain ≥ 1.2 UTS_{E8} **fasteners must be tested in bending.**

Hydrogen Embrittlement Testing

In **tension**, **ASTM F519, Type 1a.1 Notched Bar** specimen with **$d/D = 0.7$** considered an acceptable criterion for Aerospace & Aircraft

- Therefore, it was selected as the minimum acceptable value, or the minimum threshold stress $\geq 75\%$ NTS, where $NTS \approx 1.6$ UTS, or

per **F2078**, the Hydrogen Susceptibility Ratio

$H_{sr} = \text{NetThresholdStress} / \text{UTS} \geq 1.2$, or

Acceptance Criterion: $H_{sr} \geq 1.2 \leq 2.0$

Note: Cannot be achieved with **bolt** because

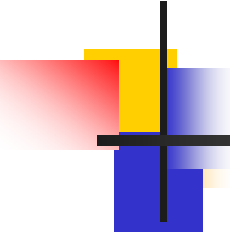
$d/D > 0.7$; i.e., $d/D \geq 0.85$

Therefore,

**Fasteners must be tested in bending,
and specimen is tested in bending**

Material Hydrogen Susceptibility Index

- Hydrogen susceptibility ratio (**Hsr**) per **ASTM F2078**
 - **Hsr = Threshold stress for HSC/UTS** of fastener
- For **Hsr ≤ 1.0**, threshold stress is below UTS and fastener has **no margin of safety in bending** during installation
- For **Hsr > 1.0**, threshold stress is above the UTS and the fastener **does have a margin of safety in bending** during installation
- Therefore, for margin of safety in **bending** of uncoated steels having a resistance to hydrogen embrittlement is for **Hsr > 1.0 and ≤ (2.0 => limiting or max stress)**
- To accommodate **bending** and to include a margin of safety on bending, and to be **consistent with ASTM F519**, Type 1a.1 specimen, it is recommended that **as a minimum Hsr = 1.2 or Hsr ≥ 1.2 ≤ 2.0**.

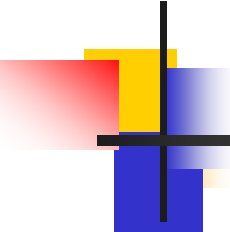


Testing Protocol

- **Baseline** is Fast Fracture Strength in bending, FFS(B)
- **Rsb**: Specimen strength ratio in bending per **ASTM E812**
 - $Rsb = FFS(B)/UTS \leq 2.0 = \text{Max limit load}$
- **Measurement:**
 - Hydrogen susceptibility ratio (**Hsr**) per **ASTM F2078**
 - **Hsr**= Threshold stress for **HSC/UTS** of fastener
- **Test Method:**
 - **(IHE) Processing – RSL™** Test in air
 - **Bare** — manufacturing of steel
 - **Coated** — application of coating
 - **(EHE) Environmental – RSL™** Test in service environment w/Holiday (defect in coating)

Test Specimens or Fasteners?

- A **rapid, inexpensive method** to evaluate a material and coating is to use an **ASTM E1280**, 0.4"W, Charpy-sized specimen with a modified notch in bending.
- It can be used for testing, either bare, or coated to obtain a long list of data starting w/HRC=>UTS; micro/SEM/EDX/fractography. In addition, K_{IC}, NFS(B), R_{sb}, H_{sr}; K_{ISCC}; OCP of coating on steel; FFS(B) of coated steel; w/Holiday; w/Holiday; or under an imposed cathodic potential to measure the permeability of the coating.
 - **Note:** The notched specimen give very similar result to bolts with similar root radius. fpc is conservative. Fastener also has **R_{sb} ≤ 2.0 as a limit load**



Influence of Coating

- In service, the main influence of a coating in a moist environment is the galvanic reaction generating hydrogen on the steel substrate.
- The coating is generally anodic to the steel fastener, "**cathodically**" protecting it.
- The fastener must also be cathodic to the structure, both of which are charging the fastener with nascent (atomic) hydrogen in a moist or marine environment.
- **Result** => the fastener is being charged with hydrogen in a moist or marine environment.

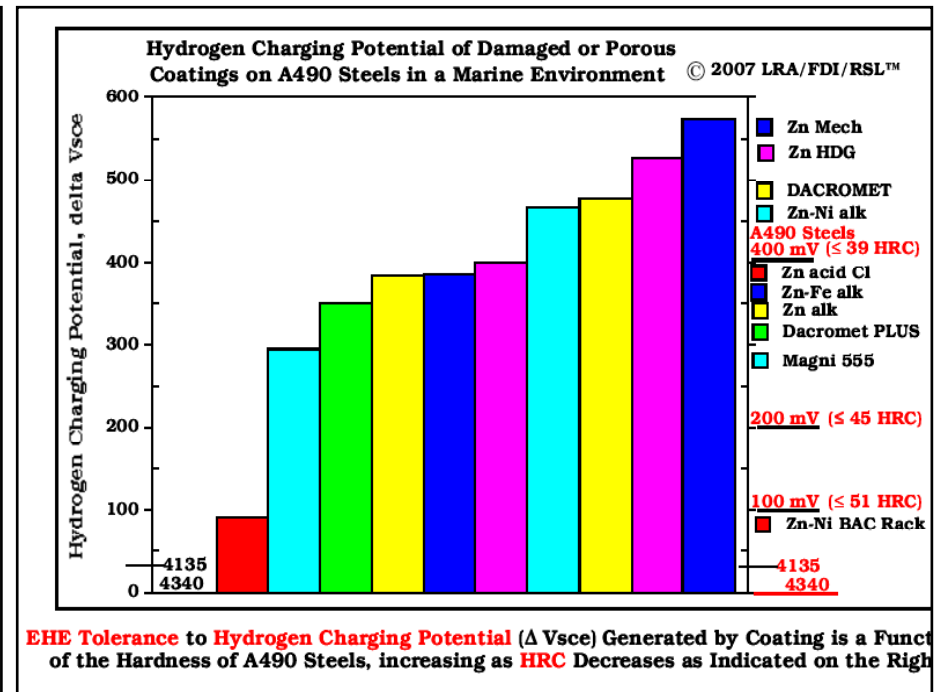
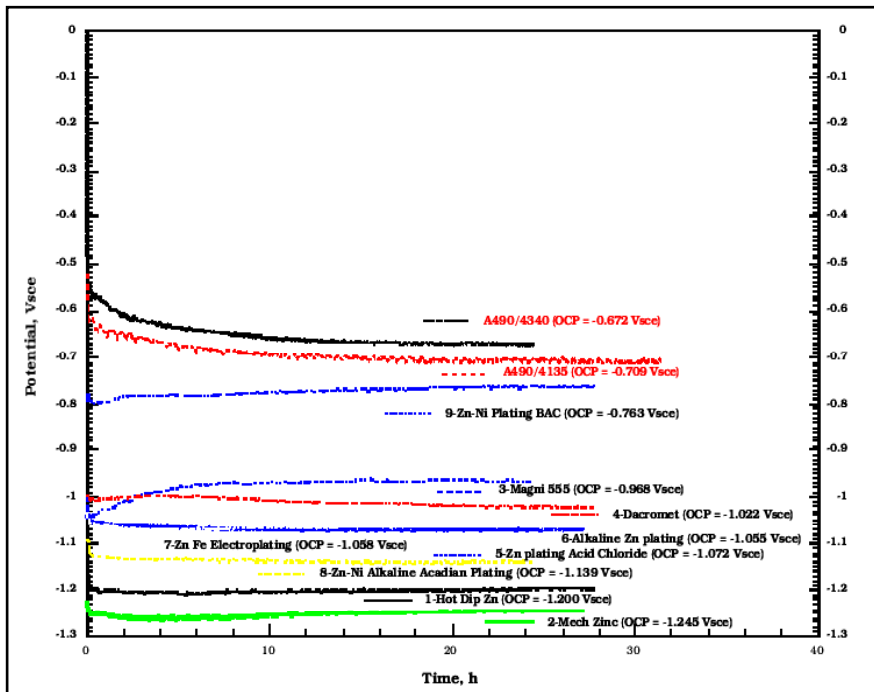
Cathodic Charging

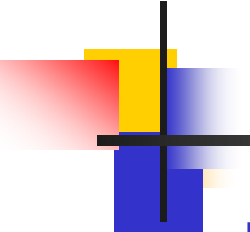
- **OCP difference** Between coating and steel bolt = **HCP**
 - **OCP** — Measure OCP per **ASTM G82**, **V_{sce}**
 - **HCP** — Hydrogen Charging Potential, **ΔV_{sce}**
- Coatings on bolt are anodic to steel substrate — sacrificially corrodes, results in bolt being cathodically protected; i.e. charged with hydrogen
- OCP of coating – OCP of bare bolt = **ΔV_{sce}**

As ΔV_{sce} ↑, Threshold stress ↓
- “**Worst case**” damaged coating exposing bare metal at root of thread (**Holiday** in coating) — “Full charge”

Approach w/Coating

- Measure the OCP in service environment, (a 3.5% NaCl solution is generally used)
- Quantify the hydrogen charging effect (ΔV_{sce})



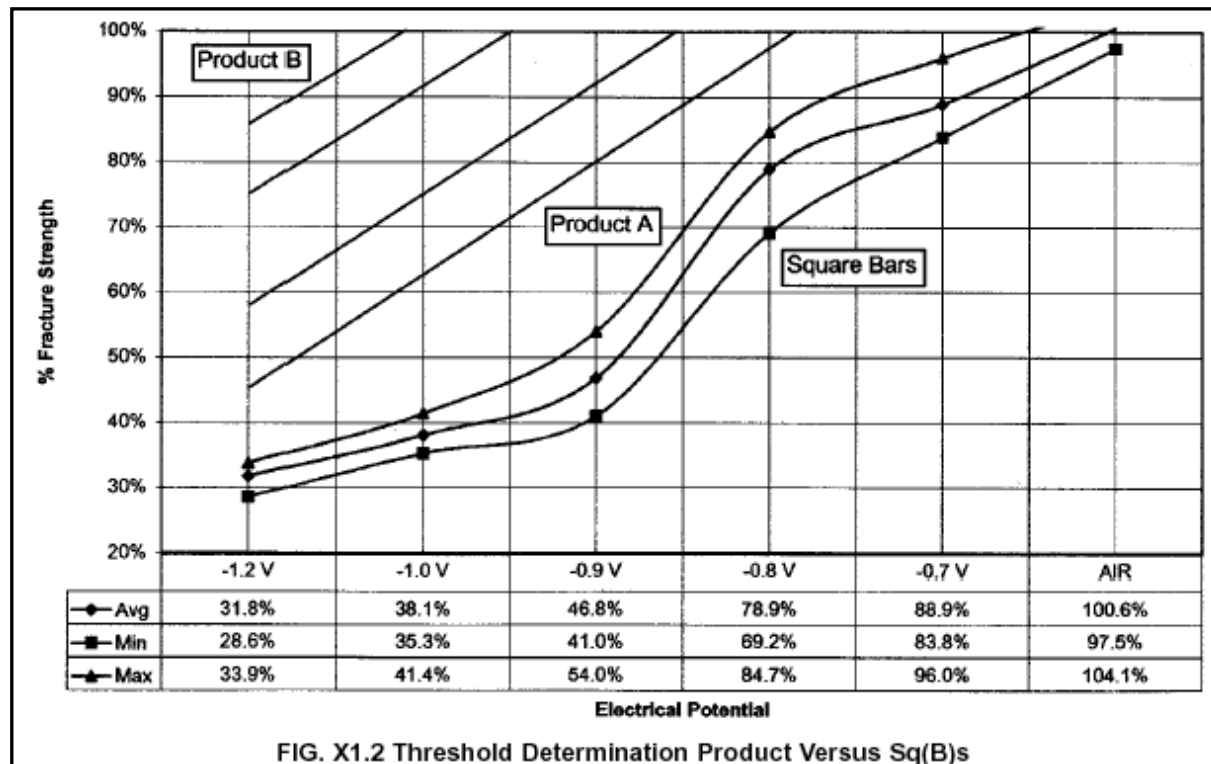


Method of Analysis

- Change parameter on **F1940** vertical axis
(%NFS => **Hsr**)
- Obtain similar data at lower hardness levels by conducting **RSL threshold** test on notched square bars of bare steel at imposed potential vs SCE to obtain threshold as **f(Vsce)**
- Overlay measured **OCP** of coating
- Extend vertically; intersection is effect of coating on threshold stress
- Determines acceptability of coating (**Hsr** ≥ **1.2**) as a function of the material and hardness

Steel susceptibility to EHE

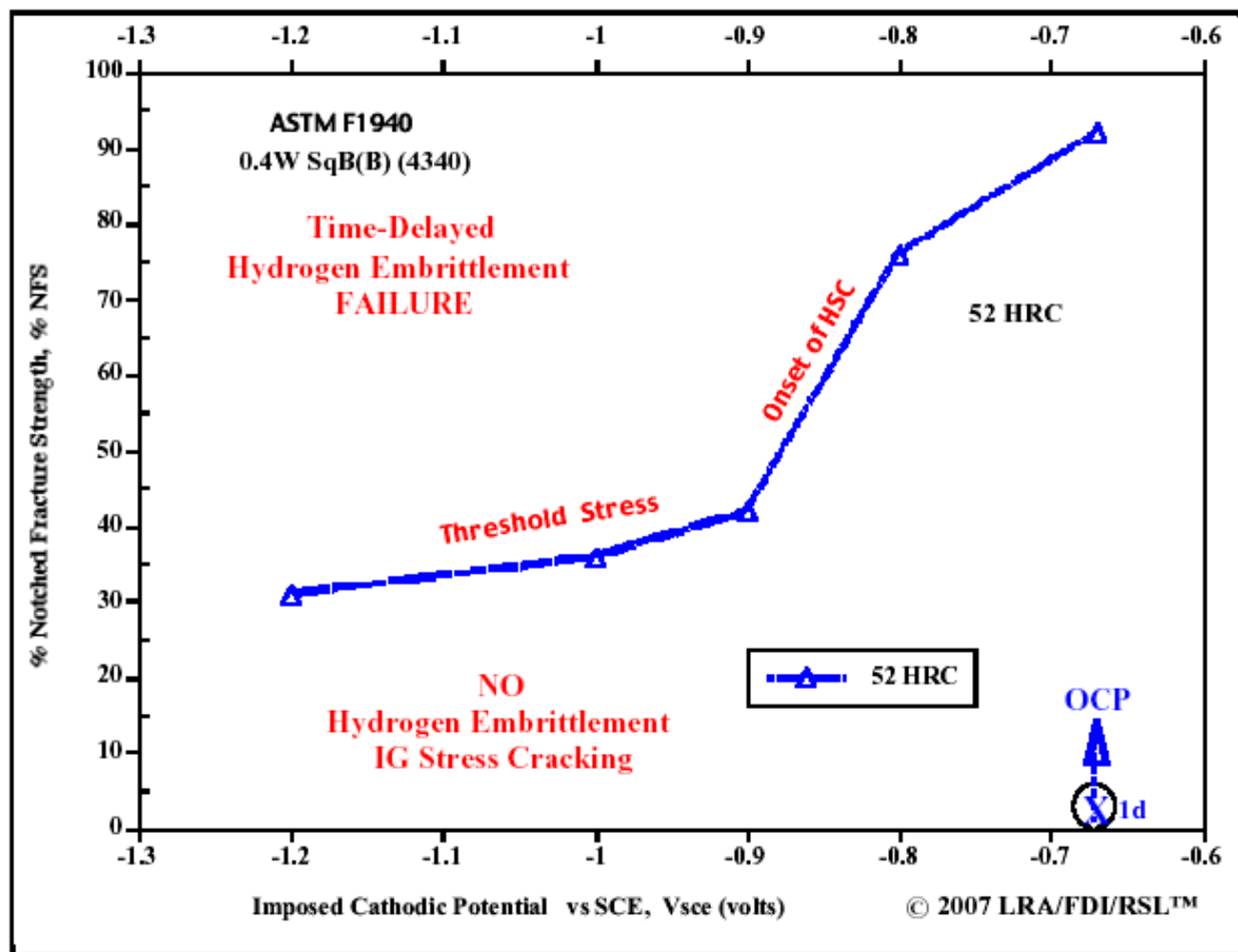
- Ref: **ASTM F1940** ("Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners") as a starting point



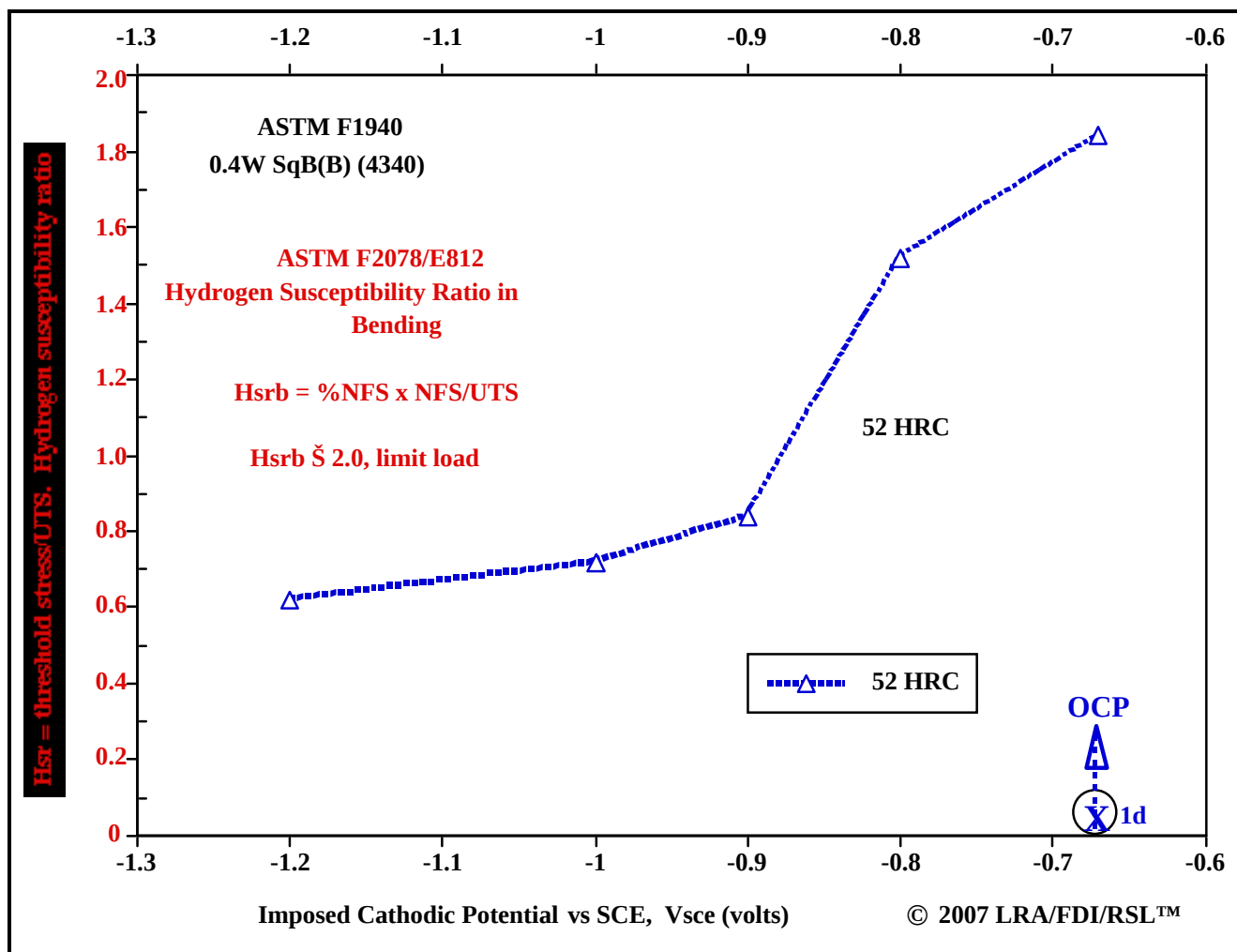
Key Items:

- Imposed cathodic potential, V_{sce} , on 4340 steel at 50-52 HRC;
- Curve is threshold stress for HSC per **ASTM F1624** as %NFS (%FS);
- Above $\Rightarrow$ HSC,
- Below $\Rightarrow$ NO Fail

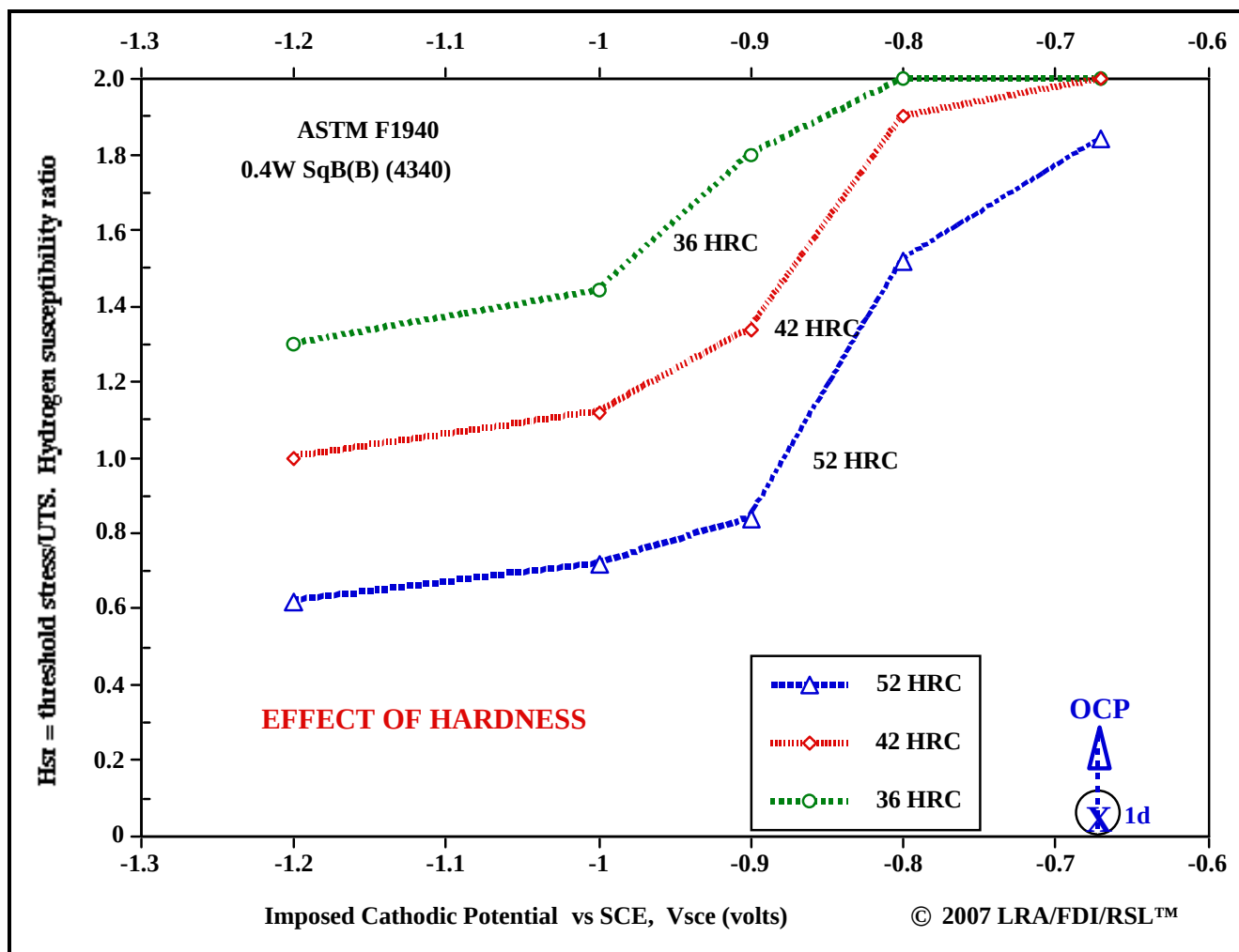
Method of Analysis



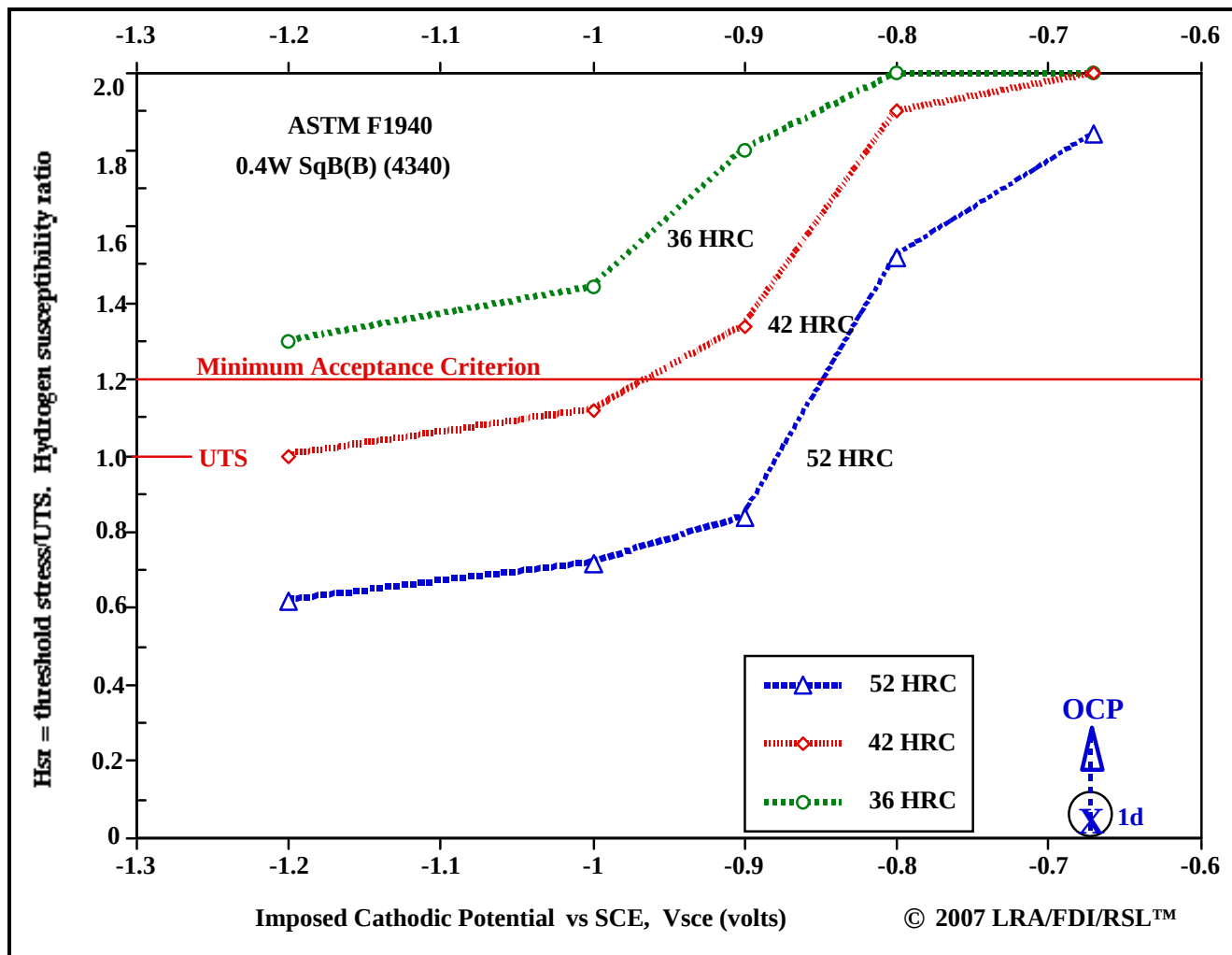
Method of Analysis



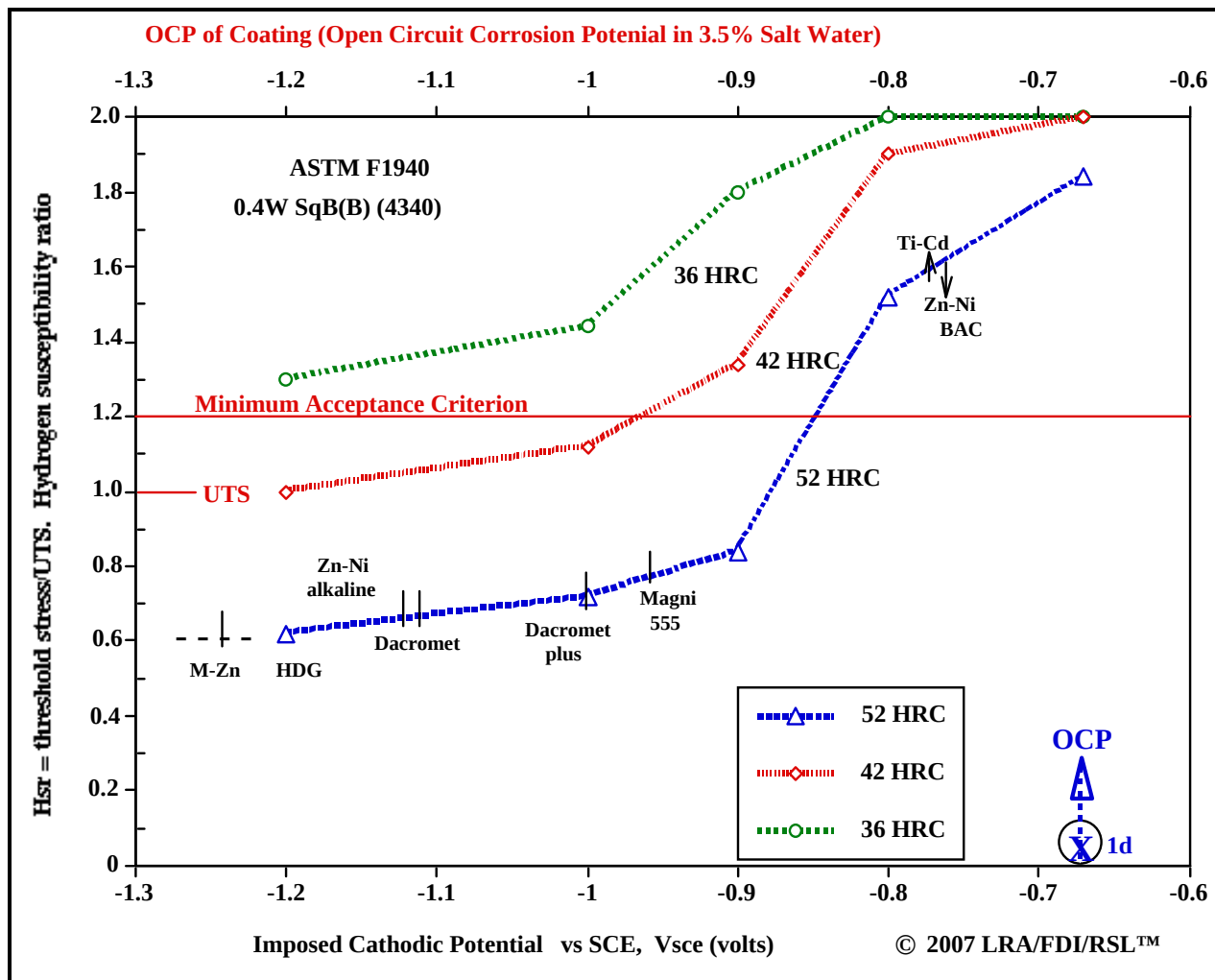
Method of Analysis



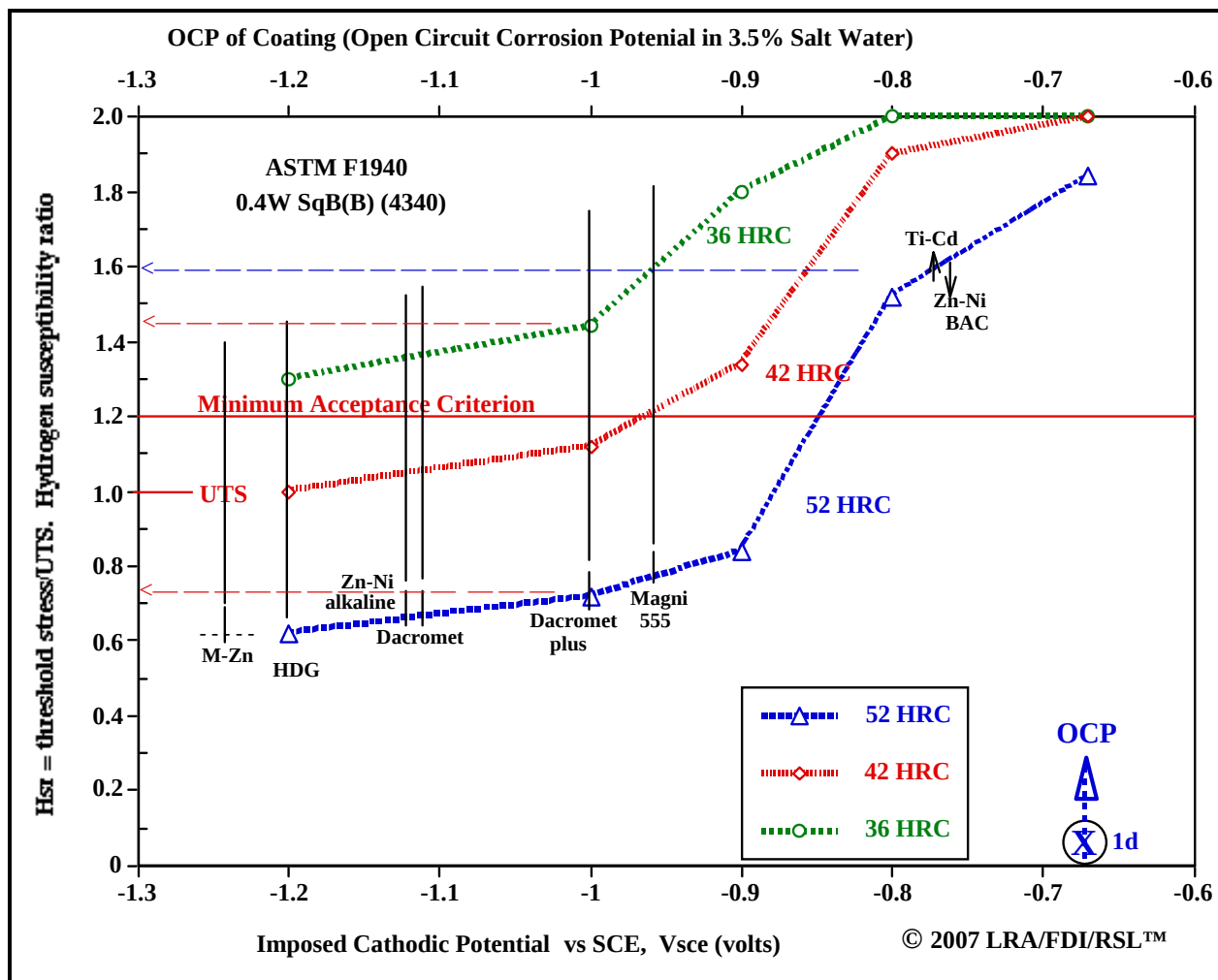
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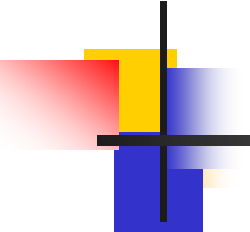


Method of Analysis



Method of Analysis

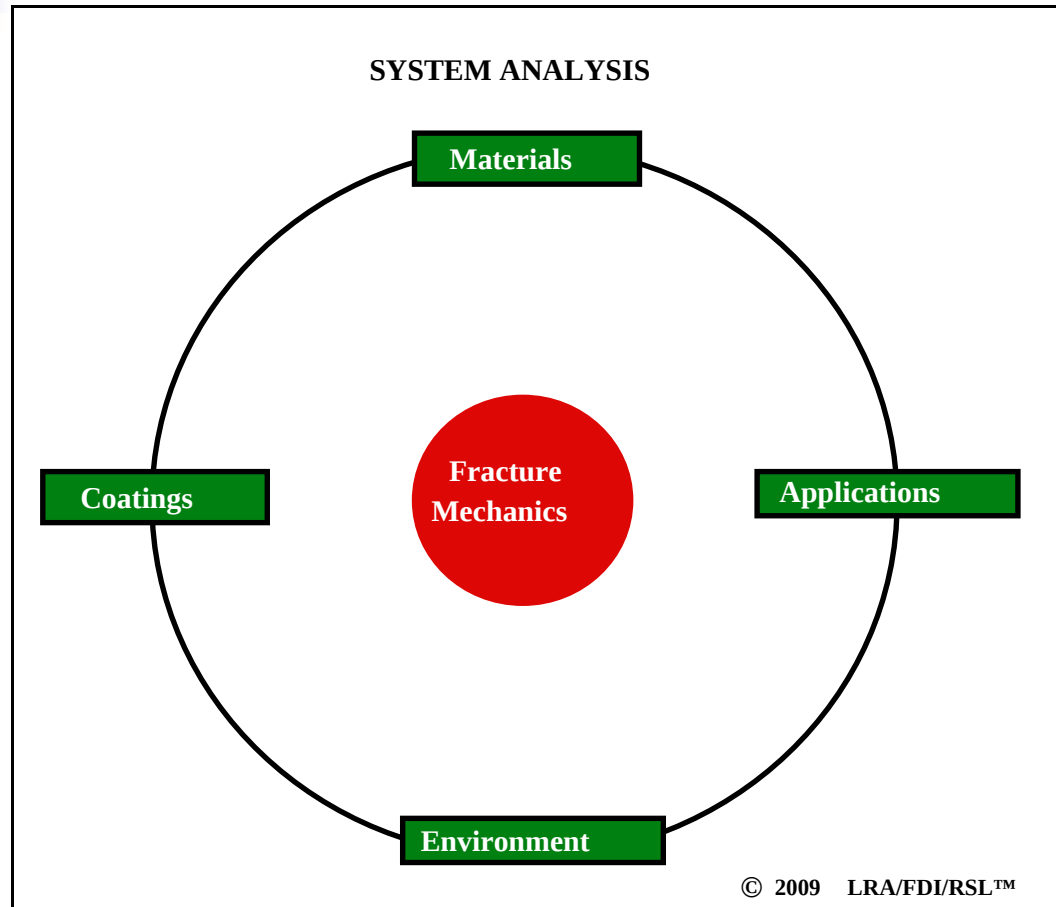




Conclusions

- **Neither coating nor alloy should be evaluated separately**; but instead, they should be examined from a **system analysis** perspective such that both material as **f(HRC)** and coating are evaluated together.
- **Ex:** Coating adequate for steel at 42 HRC might not be adequate for steel at 52 HRC
- Using the proposed acceptance criterion of **$H_{sr} \geq 1.2$** for hydrogen embrittlement testing of coated fasteners will minimize the risk of service failures because it includes a margin of safety in bending.

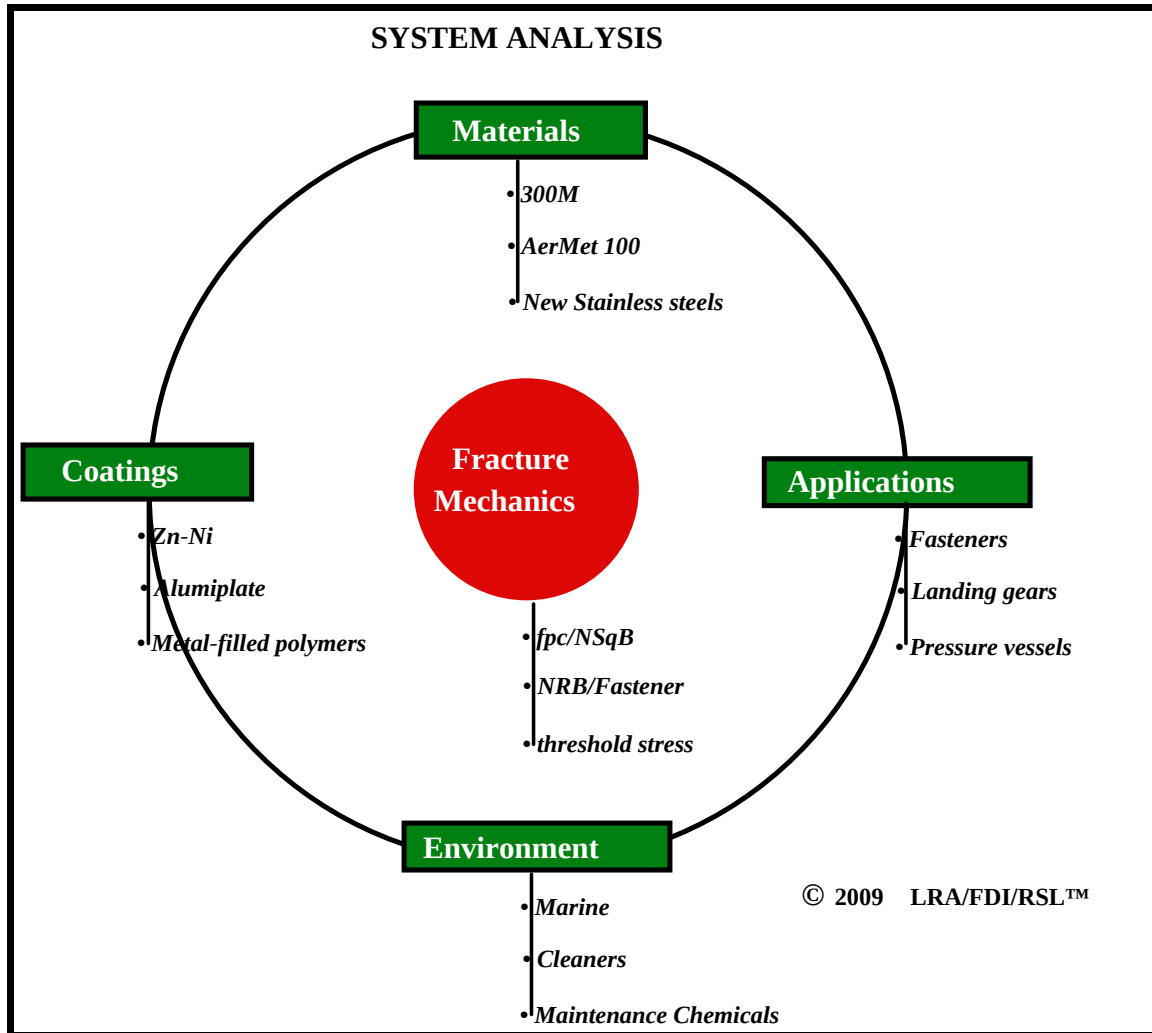
System Analysis



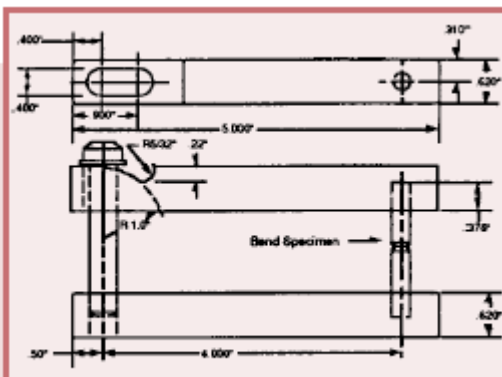
- Cannot examine each item separately

- The results on one item, affects the behavior of the one, **linked** together by **Fracture Mechanics**

System Analysis



IHE RSL™ Air Test Machine



ASTM F519,
Type 1c self-
loading frame.



Side view of RSL™
computer controlled
Type 1c test frame.



Photograph of
RSL™ test frame
showing adaptor
with notched round
bar specimen in
fixture ready for
testing.



Type 1c Notched
Round Bar plated
specimen set into
adaptor fixture for
24-hour RSL™
Accelerated Hydrogen
Embrittlement Test.

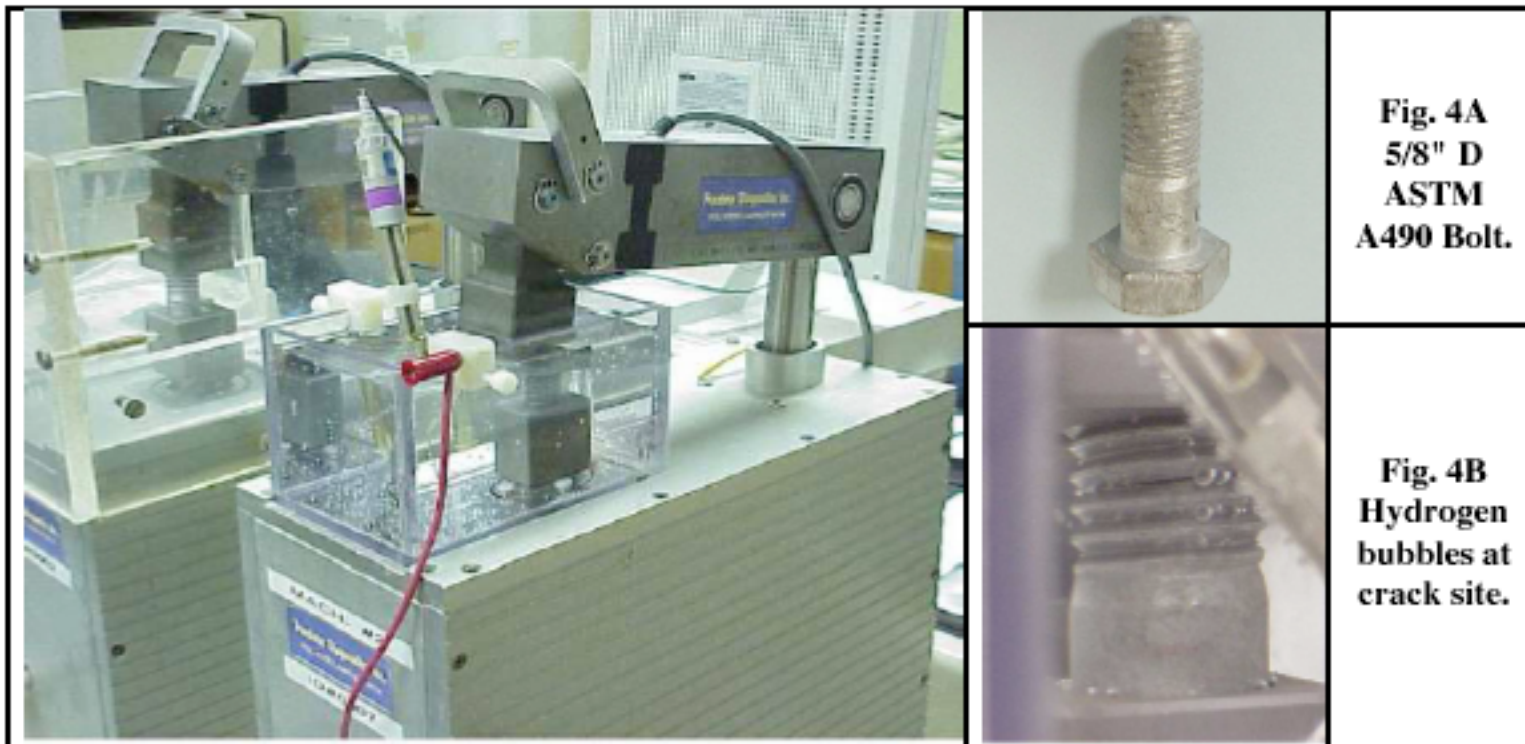
EHE Computer controlled RSL™ Environmental Test Machine

Fig. 4C Fracture Diagnostics International RSL™ (Bend) Test Machine for determination of environmental threshold stress for crack initiation of a zinc coated 5/8" D A490 bolts in hydrogen charging environment.

Background

Since 1965, I have dedicated myself, while at the Aerospace Corporation, on developing **rapid inexpensive small specimen testing techniques that quantify results**,

driven by working on failure analyses of small remnants requiring quick turn around time, such that they can be used as a quantitative cost effective solution by the designer instead of costly "trial & error" engineering solutions. The results are in terms of stress and stress intensity so that the designer can also establish a margin of safety/Risk.

Small Charpy-sized specimens have been modified to measure the fracture toughness, K_{Ic} (E399) K_{Ictod} (E1280), K_{Isc} (F1624) in 1-to-5 days as compared to 3-to-5 years run-out times for Sustained Load CB-Tests or 5,000hrs to 10,000hrs (7-to-14 months) for ASTM E399 specimens per E1681.

The techniques have been applied the more rigorous **MSP & FTRP** testing requirements of the Navy to also include an accelerated, inexpensive measurement of the dynamic tear modulus, **Td**, and **threshold stress intensity** under galvanic corrosion, cathodic charging potential of coatings on steel substrates in a marine environment. Work on these methods initiated in 1993 as an accelerated test methods for hydrogen cracking of welds and currently have been used for **accelerated corrosion fatigue** threshold measurements and a rapid test for new alloys being and coatings for Cd-replacement.



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