

# “Clean” Military Hardware - Webinar

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June 16, 2022



# Salim Brahim P.Eng., Ph.D.

- ❖ IFI Technical Director
- ❖ President and Principal Engineer - IBECA Technologies
- ❖ Adjunct Professor - McGill University, Co-Director McGill HE Facility
  - ➔ Over 33 years of experience in the fastener industry

## Highlights of Current and Past Contributions to Industry Standards Development

- Chair - Research Council on Structural Connections (RCSC)
- Chair - ASTM F16 (Fasteners) and ASTM F07 (Aerospace & Aircraft)
- Developed ASTM F1941/F1941M and ISO/TR 20491
- Head of Delegation for Canada - ISO TC2 Committee on Fasteners
- ASME Committee B18 (Fasteners), and B01 Threads
- SAE Fastener Committees
- Recipient of ASTM Fred F. Weingruber Award
- Recipient of IFI Soaring Eagle Technology Award

# INDUSTRIAL FASTENERS INSTITUTE



IFI Headquarters  
6363 Oak Tree Blvd  
Independence, OH 44131 USA  
[www.indfast.org](http://www.indfast.org)

IFI is ***the*** association of North American ***fastener manufacturers***

Mandate to actively engages with:

- ✓ **Suppliers**
- ✓ **Customers**
- ✓ **Government**
- ✓ **Public at large**



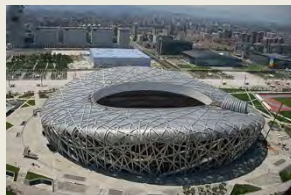
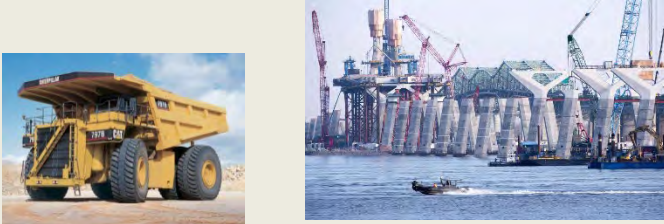
to advance the ***competitiveness, products,*** and ***innovative technology*** of its members and North American fastener manufacturing in a global marketplace.

**IFI currently has ~ 160 member companies and is continuing to grow!**

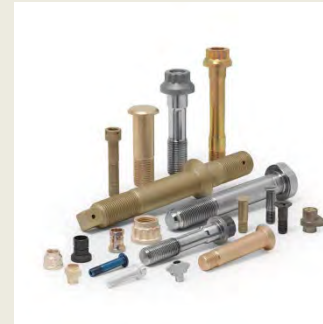


# Membership by Industrial Sector

## Division I - Industrial Products

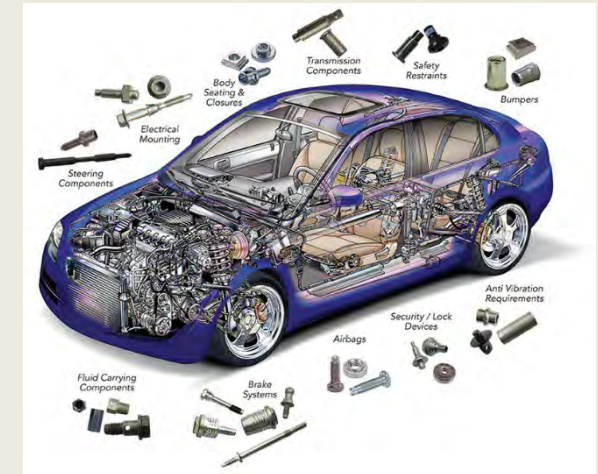


## Division II – Aerospace



Copyright 2022 Industrial Fasteners Institute

## Division III - Automotive



# Membership by Industrial Sector

## ASD: Associate Suppliers Division

Materials, machinery, equipment, and/or services





# Technical Activities

Fastener **standards** development

- IFI Standards

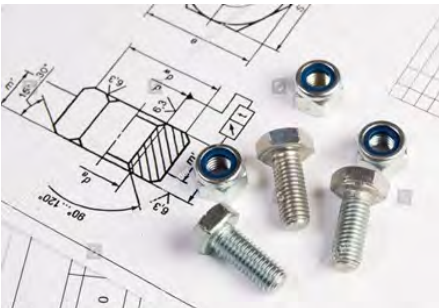
Active in **consensus standards organizations**:

- ISO, ASTM, ASME, SAE, RCSC

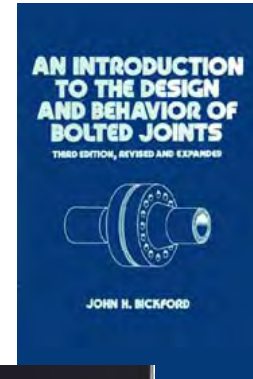
Provision of **technical resources**

- Standard
- Publications
- Books
- Technical bulletins

Technical and engineering **support**



IFI STANDARD®	QUALITY ASSURANCE REQUIREMENTS FOR FASTENER TESTING LABORATORIES	IFI-139 Page 1 of 13
Published and issued by the Industrial Fasteners Institute of Independence, OH		Issued: January 1991 Revised:



TECHNICAL  
REPORT

ISO/TR  
20491

First edition  
2019-02

**Fasteners — Fundamentals of  
hydrogen embrittlement in steel  
fasteners**

*Fixations — Principes de la fragilisation par l'hydrogène pour les  
fixations en acier*

# Training & Education



Members Only  
Training

Technical classes for members only ([IFI MOT](#))

Education and training for industry at large

➤ Collaboration: [Fastener Training Institute \(FTI\)](#)

Emphasis on **practical** and **hands-on** training

- Workshops
- Case studies
- How to read the standards
- Testing and measurement
- Plant visits

Other sponsored programs

- Fastener Education Foundation
- [Rock Valley College - Cold Forming Apprenticeship](#)
- [ECC Compton Center – Compton College](#)

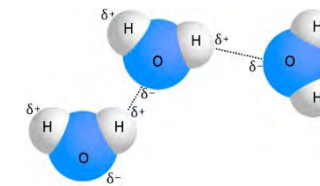


**Rock Valley College**

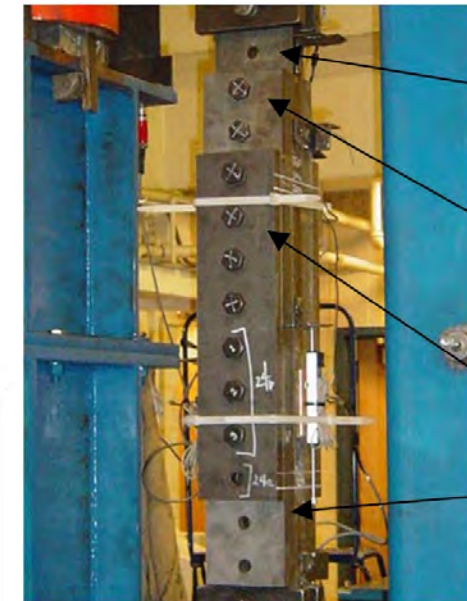
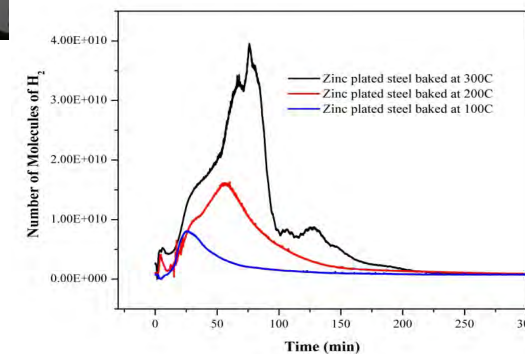
# Research & Development

## Sponsorship of **Research & Development**

- Bolting Technology Council (ASTM F16.96)
- Research Council on Structural Connections (RCSC)
- McGill Hydrogen Embrittlement Facility



## McGill Hydrogen Embrittlement Facility





# Technical Presentation

## Why replace cadmium (Cd) ?

Cadmium is used as a sacrificial coating for steel applications (fasteners, aerospace, military)

- Several beneficial properties, e.g, OCP close to steel.
- Electroplating: was predominantly based on Cyanide chemistry – less today.
- Long term exposure & cadmium poisoning →
  - Complications include cough, anemia, and kidney failure and increases risk of cancer

## Why hexavalent chromium (Cr<sup>6+</sup>) ?

Hex chrome used as a passivation layer to protect sacrificial coatings (e.g., Zn, AL, Cd)

- Hexavalent chromium compounds are genotoxic carcinogens.

**Environmental regulations e.g., EU RoHS (2003) and REACH (2007)**

**→ Hard deadlines for replacing Cd with more environmentally friendly options, such as Zn-Ni and Al and eliminating Cr<sup>6+</sup>**



# McGill



## REPORT

Joint Projects  
funded by

NATURAL SCIENCES AND ENGINEERING RESEARCH COUNCIL OF CANADA  
(NSERC)

### REPLACEMENT OF CADMIUM COATINGS FOR AEROSPACE APPLICATIONS

(CRDPJ 424990 – 11)

Héroux-Devtek  
Pratt & Whitney Canada  
Safran Landing Systems\*

\* formerly Messier-Bugatti-Dowty

### EFFECT OF MATERIAL CHARACTERISTICS OF ZINC BASED COATINGS ON HYDROGEN EMBRITTLEMENT OF STEEL FASTENERS

(CRDPJ 428422 – 11)

Boeing  
Canadian Fasteners Institute

Principle Investigator  
Prof. Stephen Yue

Department of Mining and Materials Engineering

McGill University

## 2012-2017

- Tribological studies: comparisons between Cd, Al and Zn-Ni coatings
- Corrosion studies: Cd, Al and Zn-Ni coatings in neutral, acidic and alkaline environments
- Effect of baking on aerospace grade electroplated Zn and Cd on high strength steel
- Quantification of hydrogen uptake in steels due to corrosion of sacrificial coatings

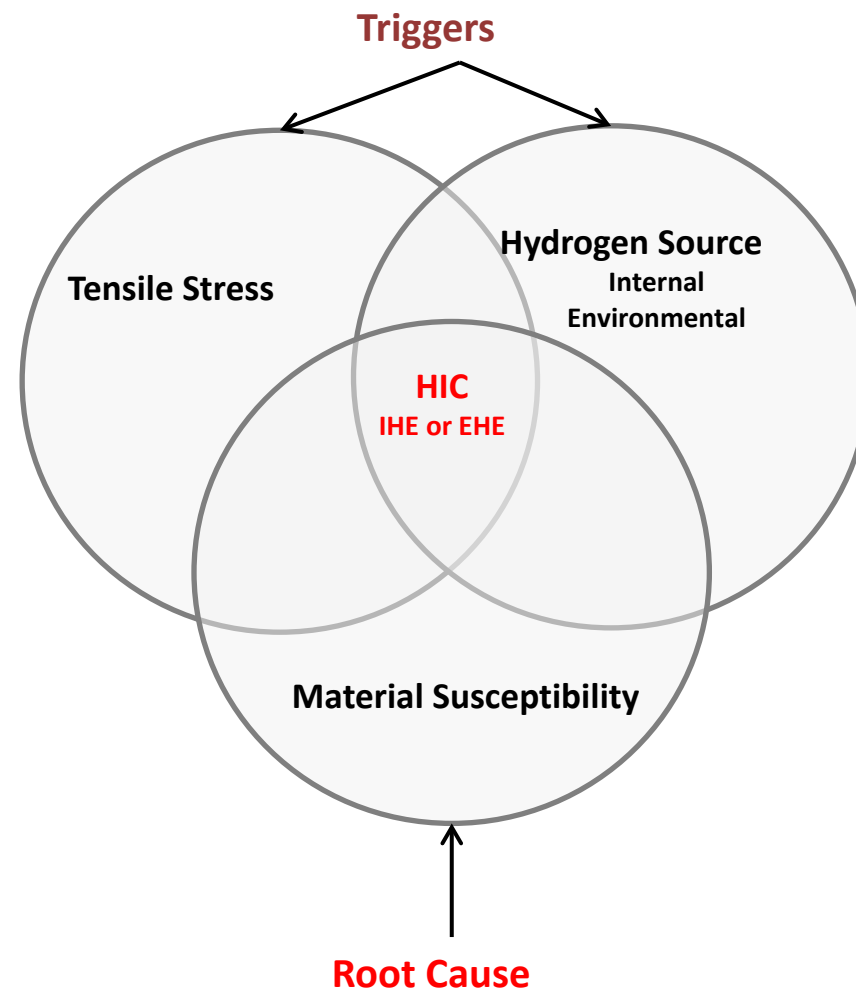
# Hydrogen Embrittlement defined

Hydrogen Embrittlement (HE) — a permanent loss of ductility in a metal or alloy caused by hydrogen in combination with stress, either externally applied or internal residual stress.

Source: ASTM F 2078

1. Susceptibility
2. Hydrogen
3. Stress

**TIME!**





# Sources of hydrogen

## IHE: Internal Hydrogen Embrittlement

Residual H from processing

Electroplating

Acid Pickling



## EHE: Environmental Hydrogen Embrittlement

H absorption during service –  
principally **corrosion**

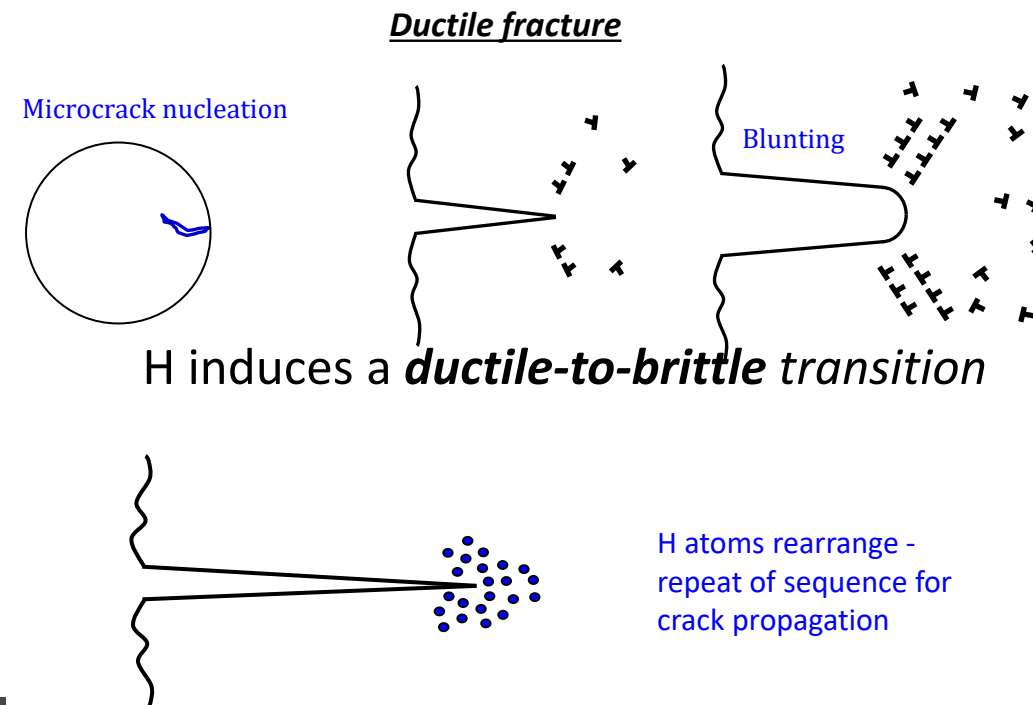
Stress corrosion cracking (SCC)

Cathodic hydrogen absorption (CHA)

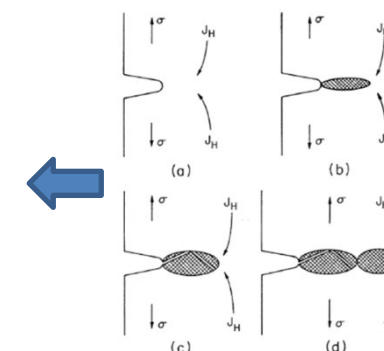


# HE damage mechanism

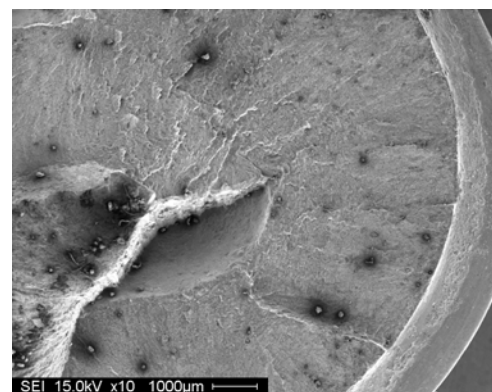
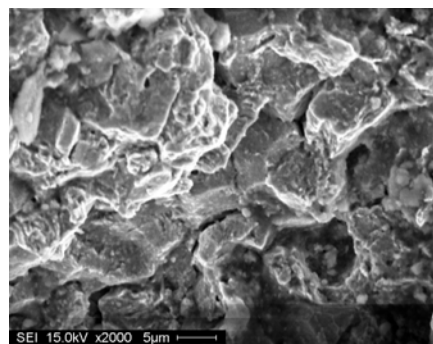
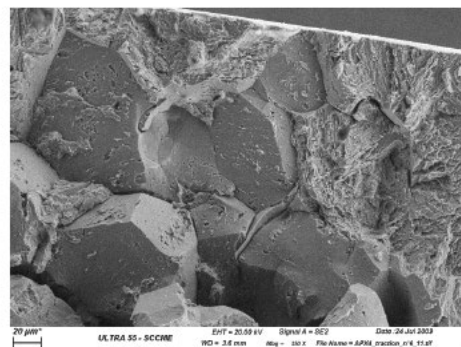
- **Stress concentration** gradient
- **Transport & trapping** affected by microstructural characteristics
- **Hydrogen damage** → crack initiation/growth
  - **Decohesion of atomic bonds (HEDE)**
  - **Hydrogen Enhanced Local Plasticity (HELP)**
  - **Dislocation pinning**



**Sequential brittle fracture!**

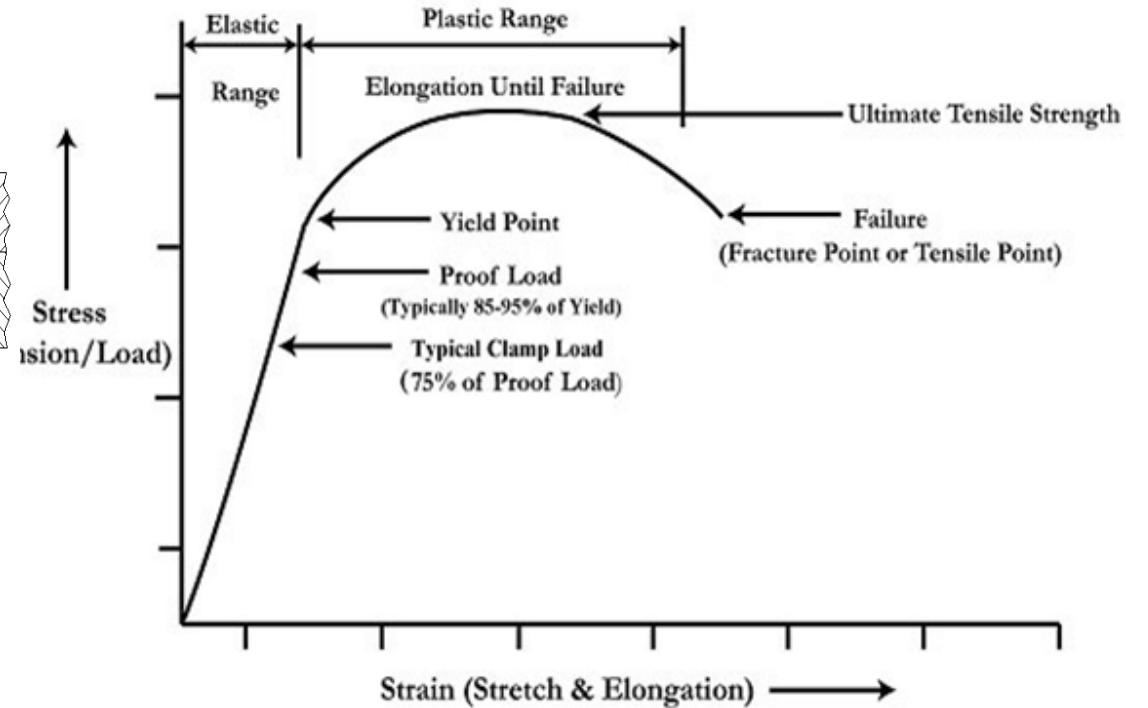
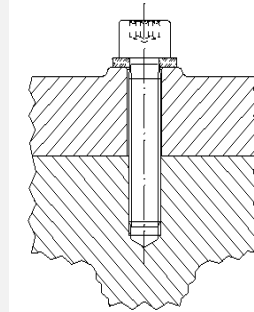


Crack propagation mechanism

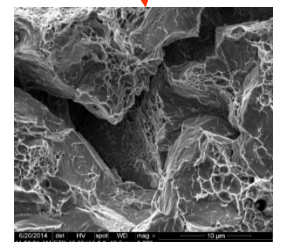
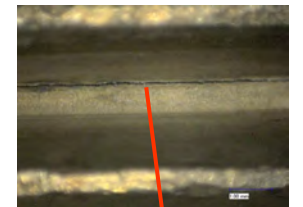
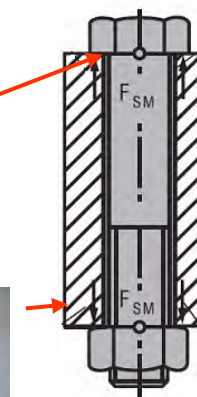


# Bolt strength levels and mechanical behavior

- **400 MPa (PC 4.6) - 60 ksi (Gr. 2) -**
  - Plain carbon steel
  - Not heat treated
- **800 MPa (PC 8.8) - 120 ksi (Gr. 5)**
  - Plain carbon steel or alloy steel
  - Heat treated
- **1000 MPa (PC 10.9) - 150 ksi (Gr. 8)**
  - Alloy steel (or carbon boron steel for 10.9)
  - Heat treated
- **1200 MPa (PC 12.9) - 180 ksi**  
**(39 HRC, 390 HV)**
  - Alloy steel
  - Heat treated



Tensile Stress-Strain Diagram



Rajagopalan, Brahimi, Yue, 2010

High strength (>1200 MPa) → high HE **susceptibility**

Critical applications → high **stresses**

Coated → potential for residual **hydrogen**

Used in corrosive environments → **hydrogen** generation

Built-in stress concentration areas

- Threads
- Underhead fillet radius

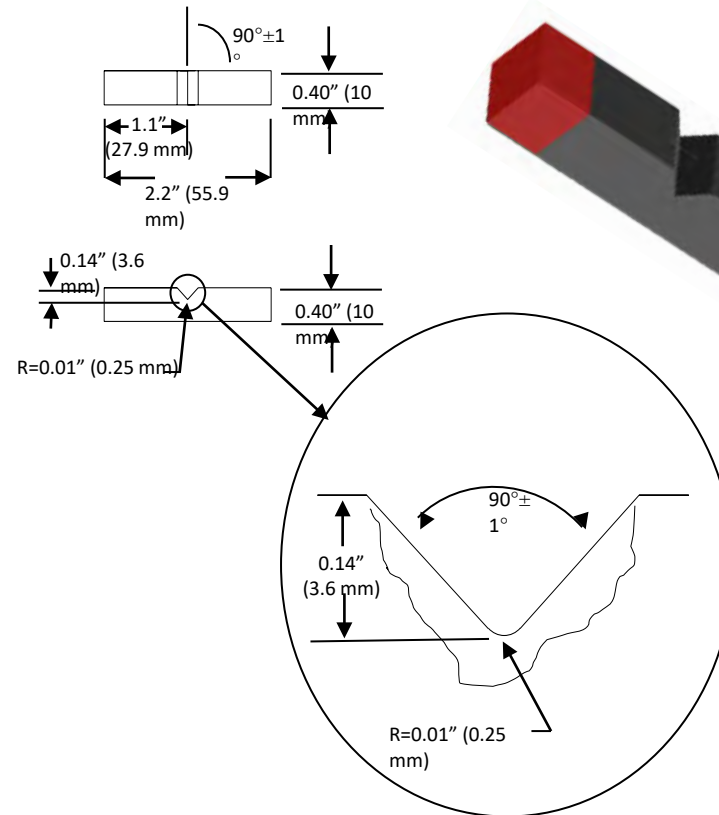


# Coating process evaluation – test in air

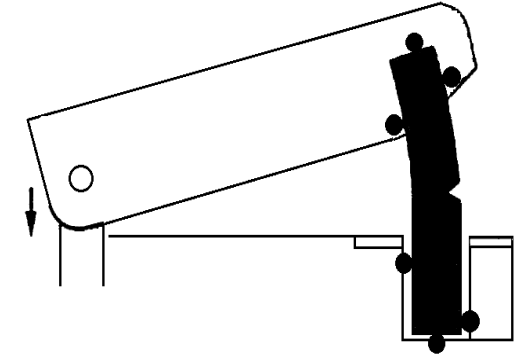
~60 process & processing conditions were evaluated

- Zn – acid chloride – barrel
- Zn – alkaline (non cyanide) – barrel
- Zn/Ni – acid chloride – barrel
- Zn/Ni – alkaline (non cyanide) – barrel
- Zn/Ni – alkaline (non cyanide) – rack
- Zn/Fe – alkaline (non cyanide) – barrel
- Cadmium – cyanide – barrel
- Zn phosphate – barrel
- Mechanical zinc – bulk drum
- Magni 555® – bulk dip spin
- Dacromet® – bulk dip spin

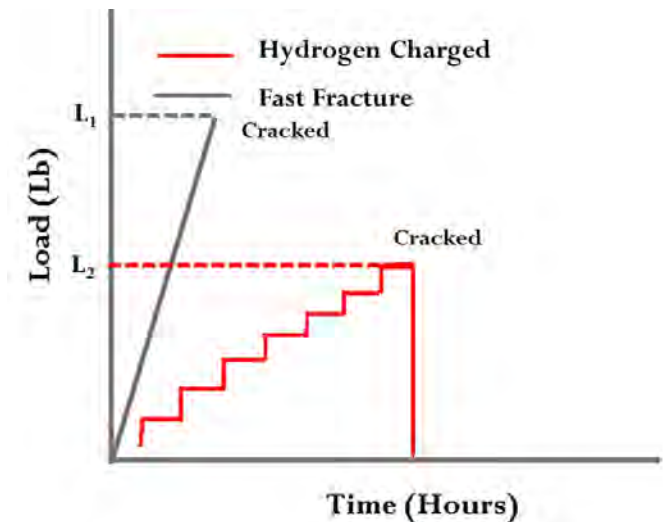
**Notch bar → 53 HRC → worst case**



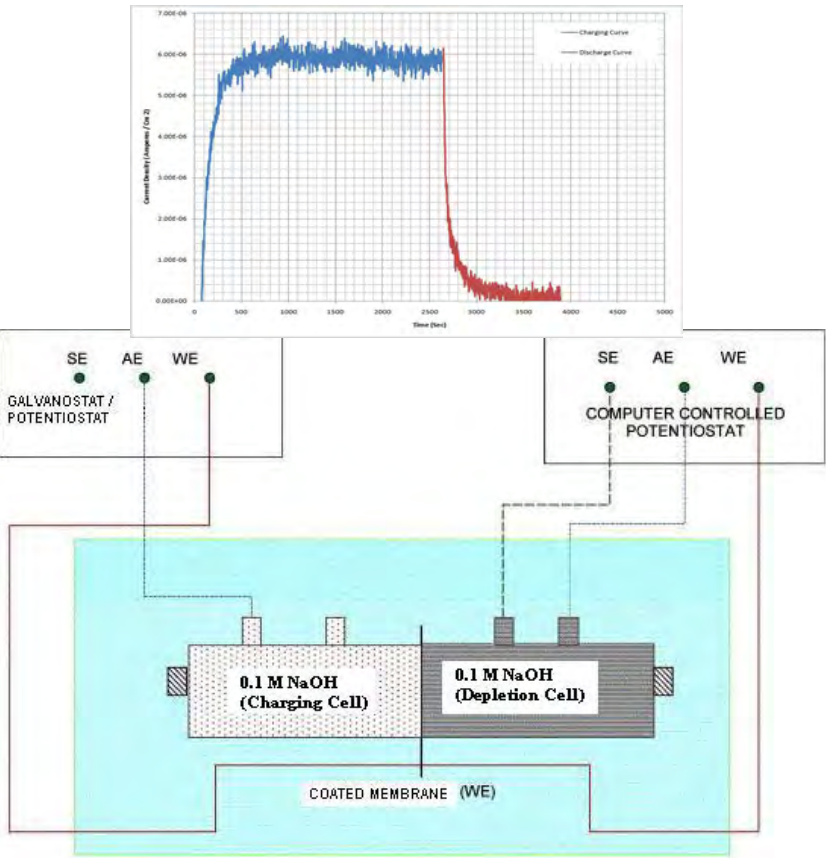
Notched square bar – ASTM F519 type 1e  
H introduced by electroplating (IHE)



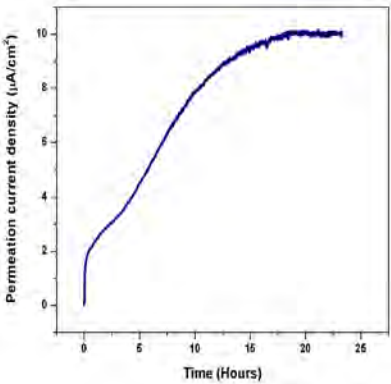
ASTM F1940 Loading protocol  
24 h test



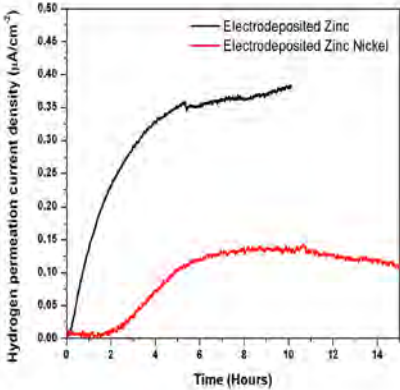
# Other analytical techniques – Electrochemical permeation (EP)



Calculation of effective diffusion coefficient of the coating material



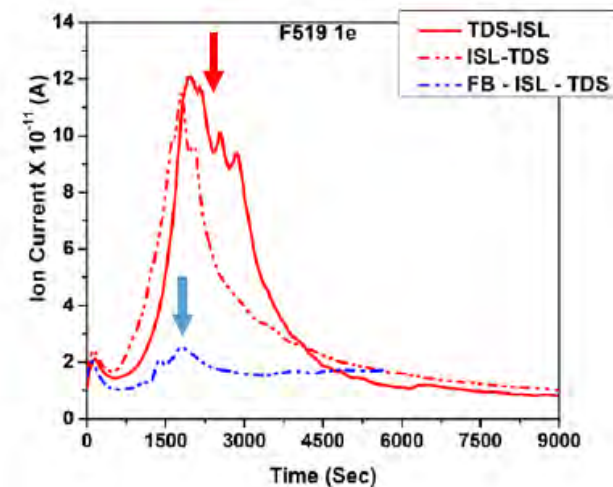
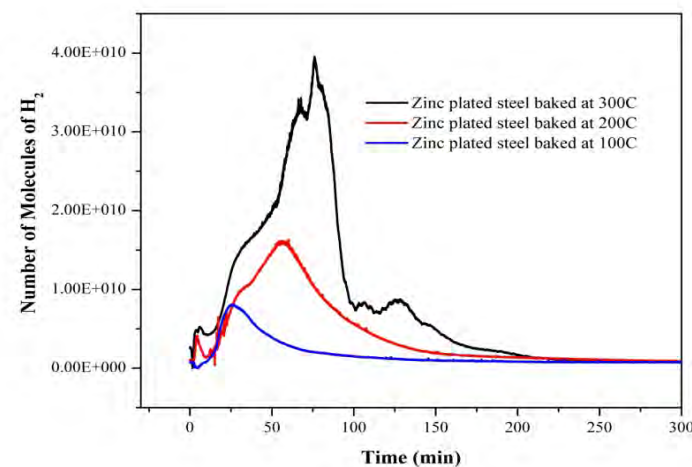
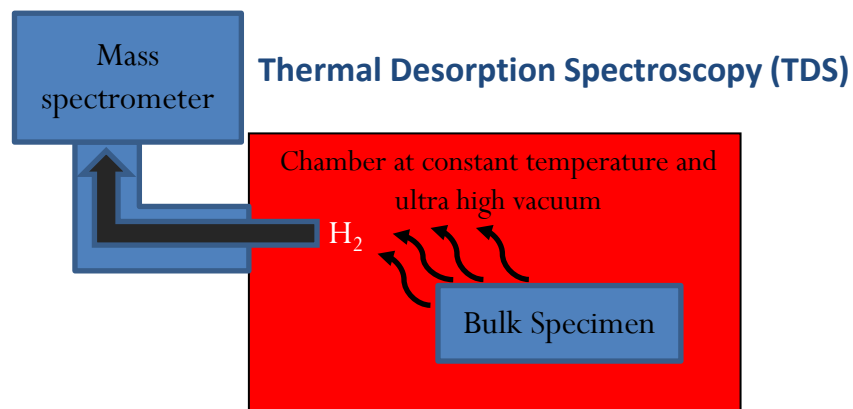
Permeation transients of low carbon steel shim



Permeation transients of Zn and Zn –Ni coatings on low carbon steel shim

Material	Diffusion Coefficient D (cm <sup>2</sup> s <sup>-1</sup> )	Subsurface hydrogen conc. C <sub>0</sub> (Mol cm <sup>-3</sup> )	Permeation flux J <sub>ss</sub> (mol s <sup>-1</sup> cm <sup>2</sup> )
Bare steel	3.86E-07	2.70E-06	1.05E-10
Electrodeposited Zn	8.20E-09	5.38E-06	3.94E-12
Electrodeposited Zn-Ni	3.91E-09	2.89E-06	8.6E-13

## Other analytical techniques – Thermal desorption spectroscopy (TDS)



**Measure H discharge as a function of time and temperature**

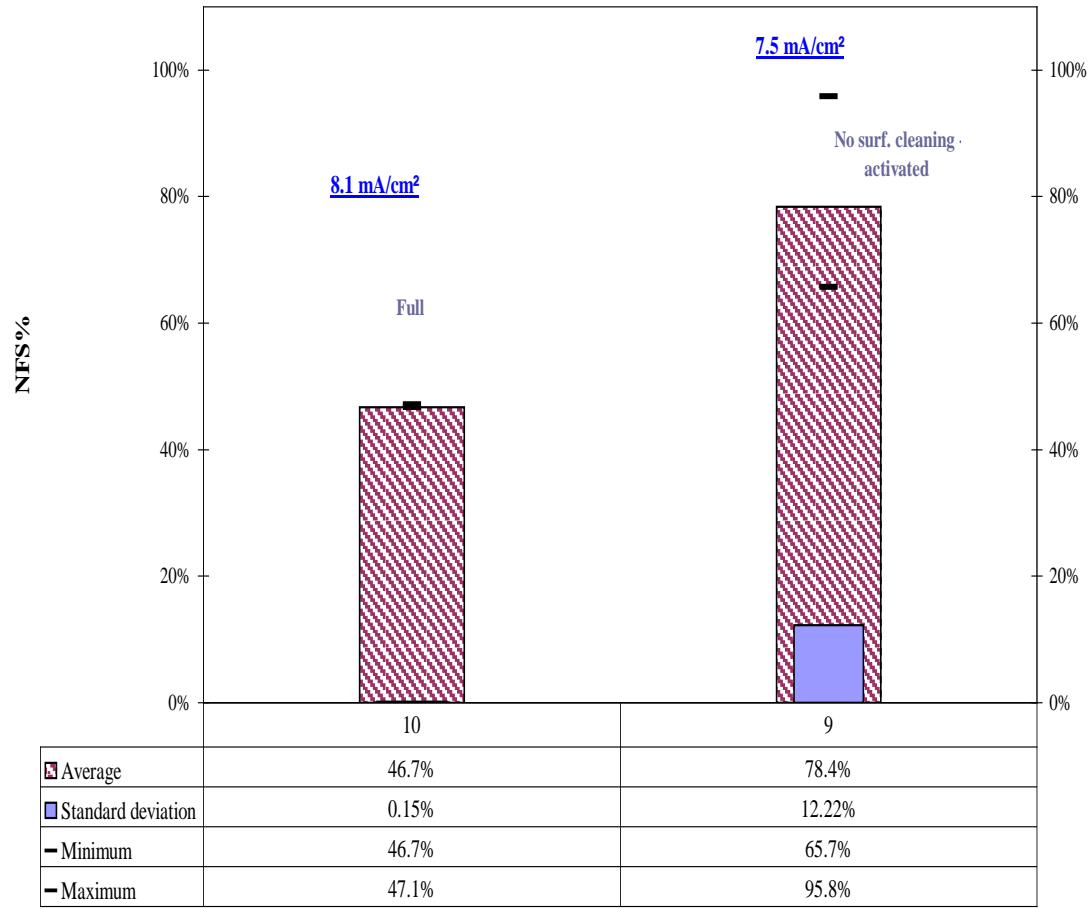
Rajagopalan, Brahimi, Yue, 2010



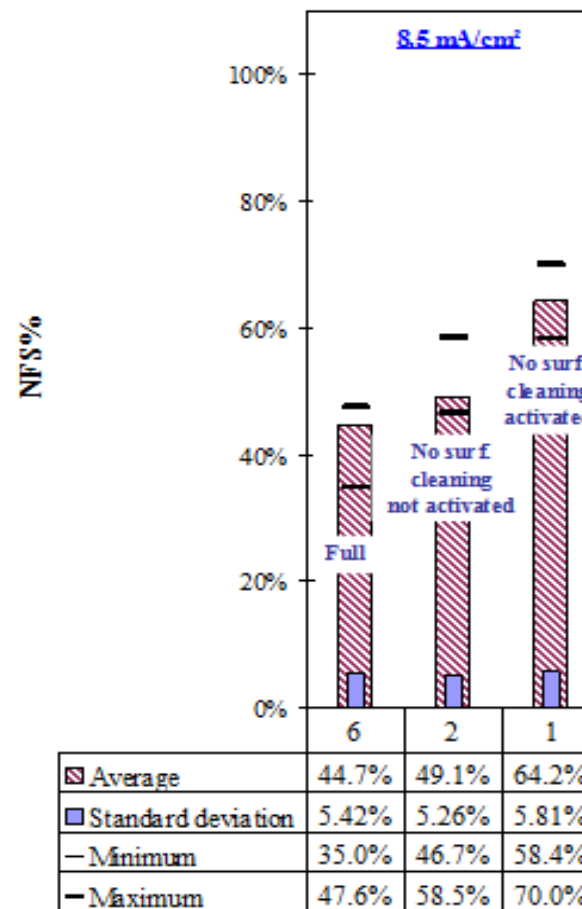
# Zinc electroplating

**Effect of baking  
200 °C (~400 °F)**

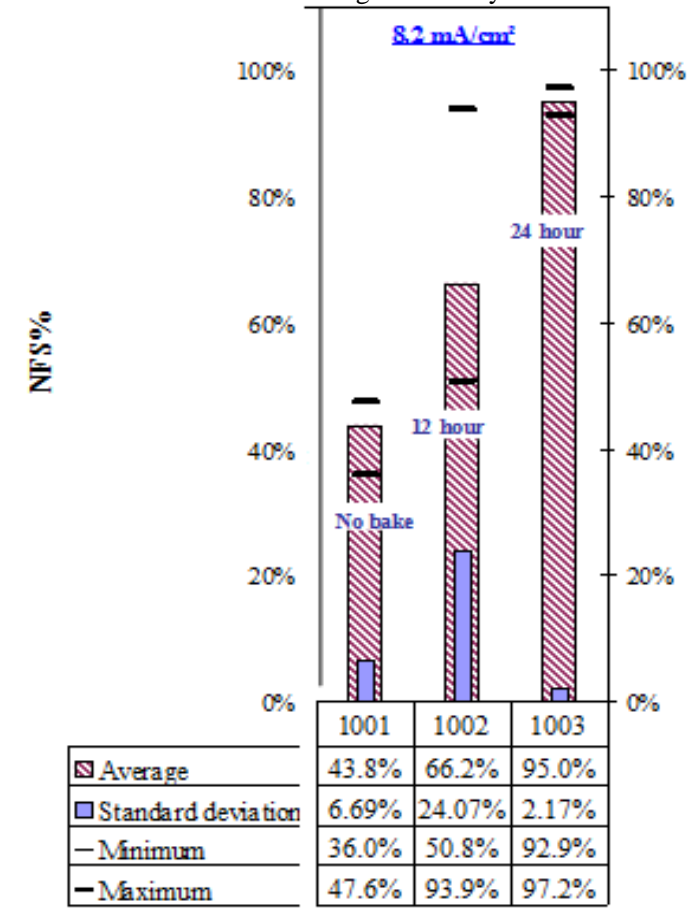
**Alkaline zinc**  
High efficiency 80-90%



**Zinc - acid chloride**  
High efficiency 90-95 %



**Zinc - acid chloride**  
High efficiency 90-95 %

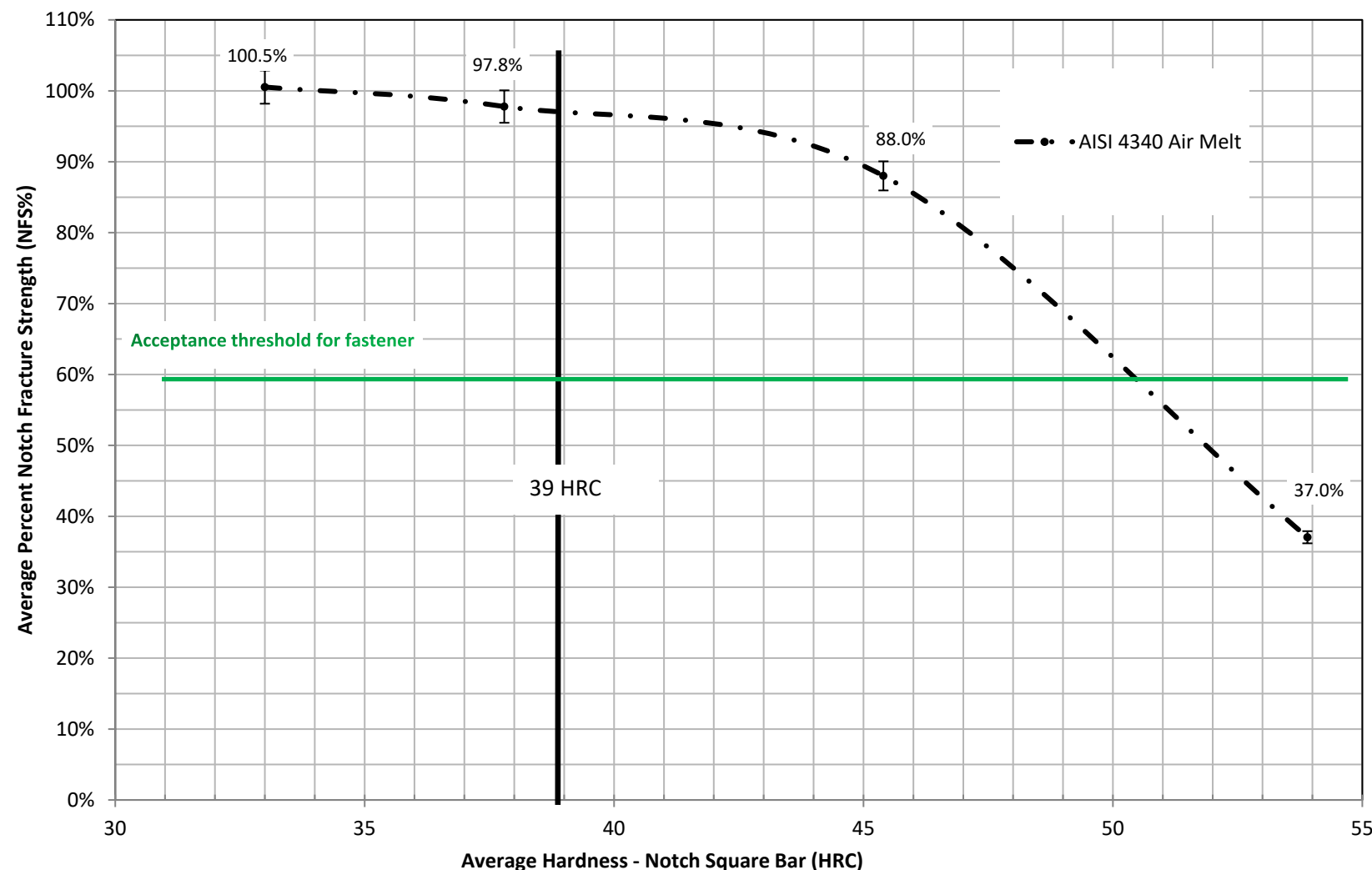


Brahimi, Yue, 2008

# IHE – Worst case: Zn Electroplating

The susceptibility of steel fasteners increases significantly when the specified hardness is above 39 HRC (380 HV).

In other words, they can tolerate the presence of higher concentrations of hydrogen without any delayed degradation of their mechanical strength



# Confusing electroplating technical standards

## **IHE avoidance – requirement for “baking”**

Some standards have defined critical hardness limits for baking that are lower, ranging from 31 to 35 HRC. Examples: ASTM B633/ASTM B850 and ISO 2081/ISO 9588 – **these standards should NOT be used for electroplating fasteners.**

These values are largely unsupported by data → adopted primarily as a precaution against manufacturing errors.

It is appropriate to classify susceptible fastener products as those having minimum specified hardness above 39 HRC (390 HV).

This classification is based on both scientific research and longstanding fastener industry practice.

### **Standards suited for fasteners:**

→ **ASTM F1941/F1941M**

→ **ISO 4042**

# Summary findings

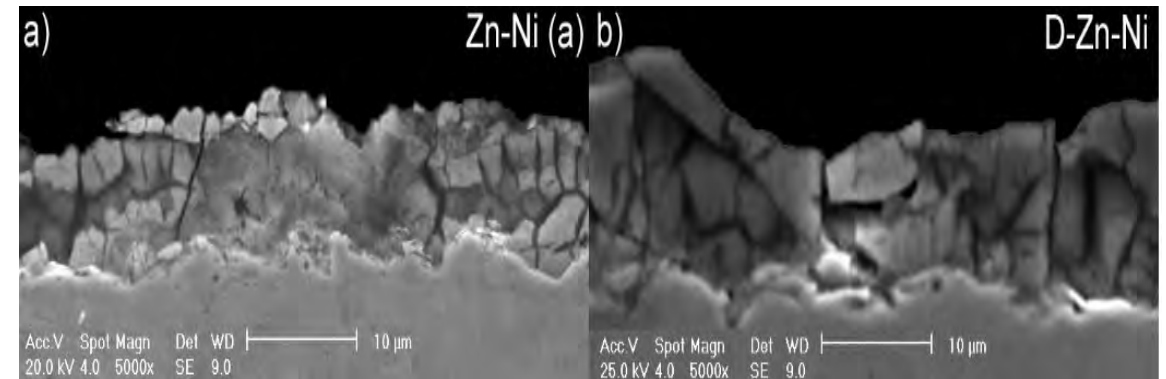
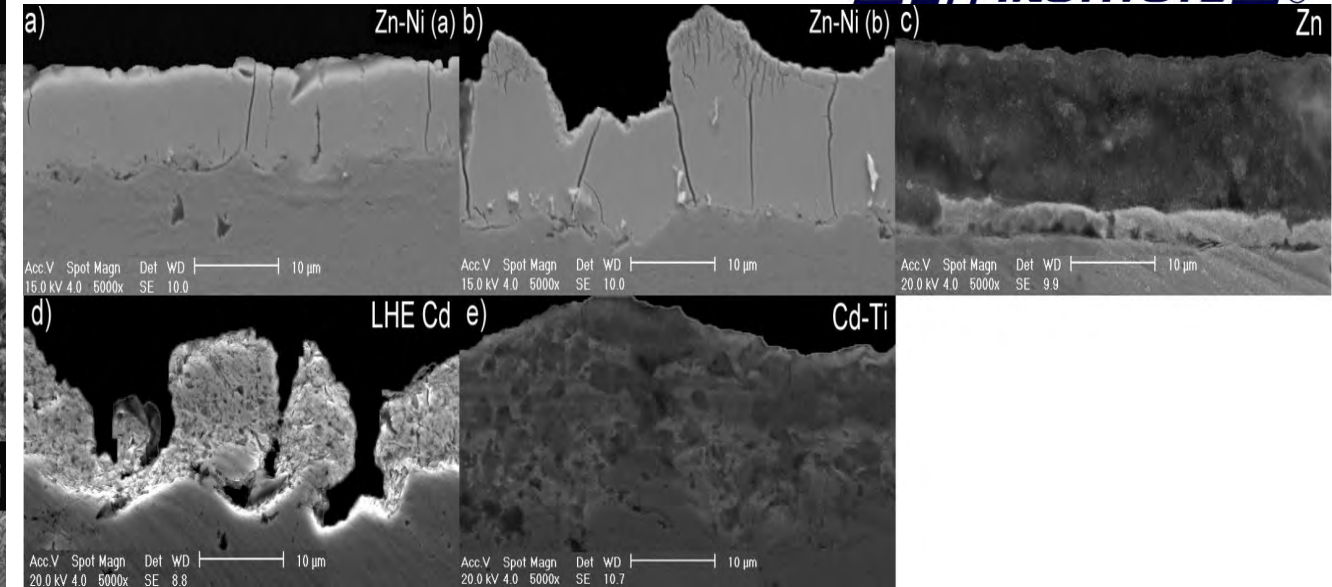
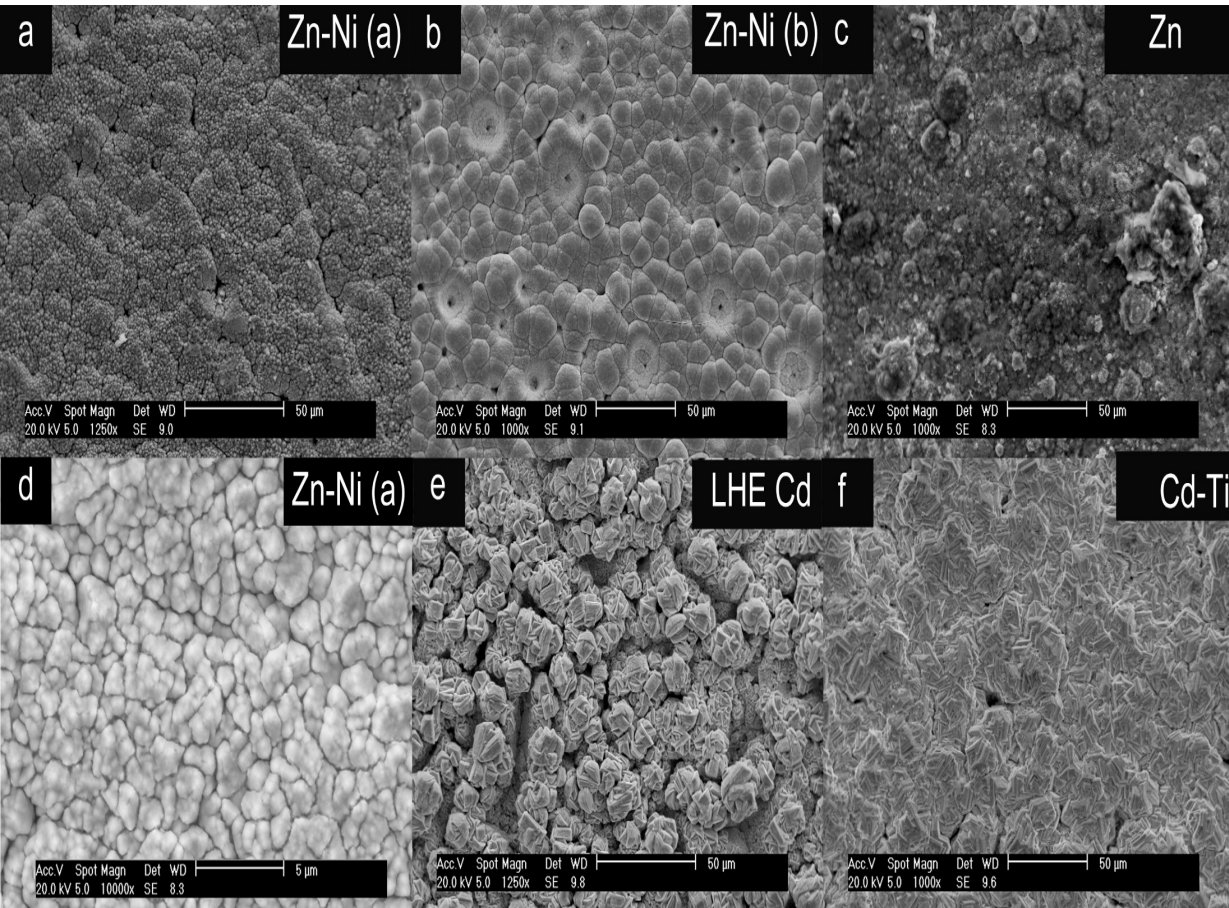
- Zinc acid chloride → most embrittling
- Alkaline zinc → slightly less embrittling
- Alkaline zinc-iron process → moderately embrittling (86%)
- The least embrittling processes → LHE zinc-nickel, alkaline and acid

Results support grouping into two basic parameters affecting IHE:

- (i) **Coating permeability** - first order effect,
- (ii) **Quantity of hydrogen** introduced by the process - second order effect

- Baking at min. 400 ° F (204 °C) can fully restore ductility
- However... baking response depends upon the permeability of the coating  
→ baking time must be adapted to the coating type
- **For Zn plated coatings, 4 h bake is a waste of time and money!**
- Zn-Ni Coatings will require less baking time than Zn → **to be determined!**
- **Parts below 39 HRC are not embrittled –no baking required! ref. ASTM F1941**

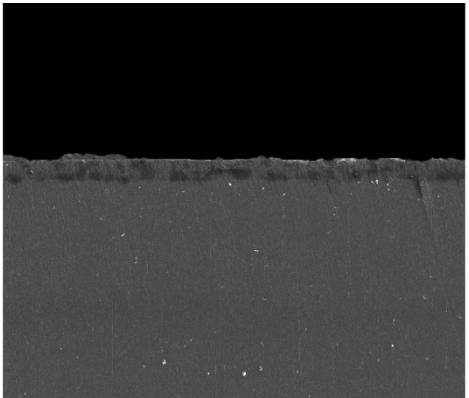
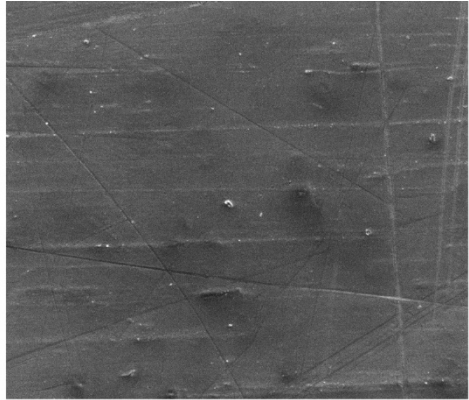




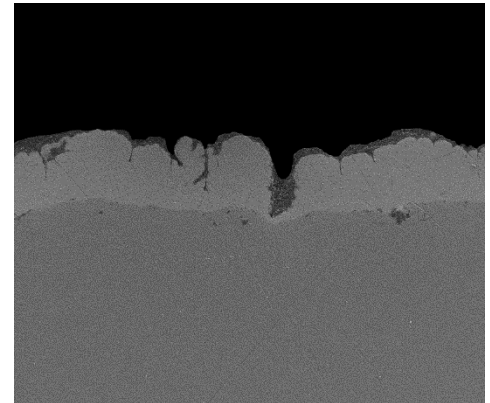
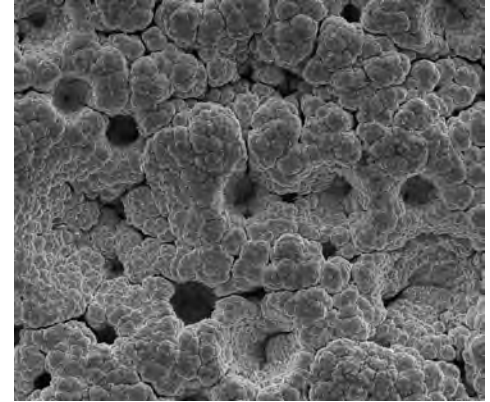
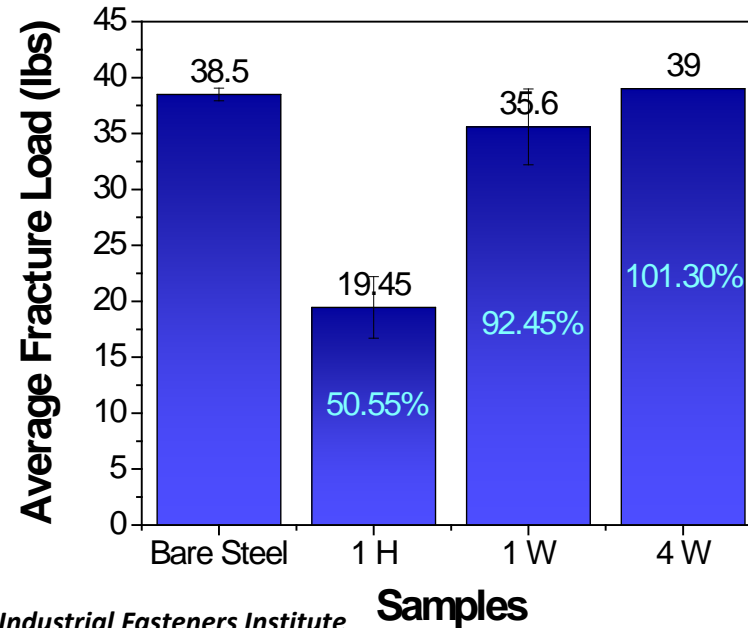
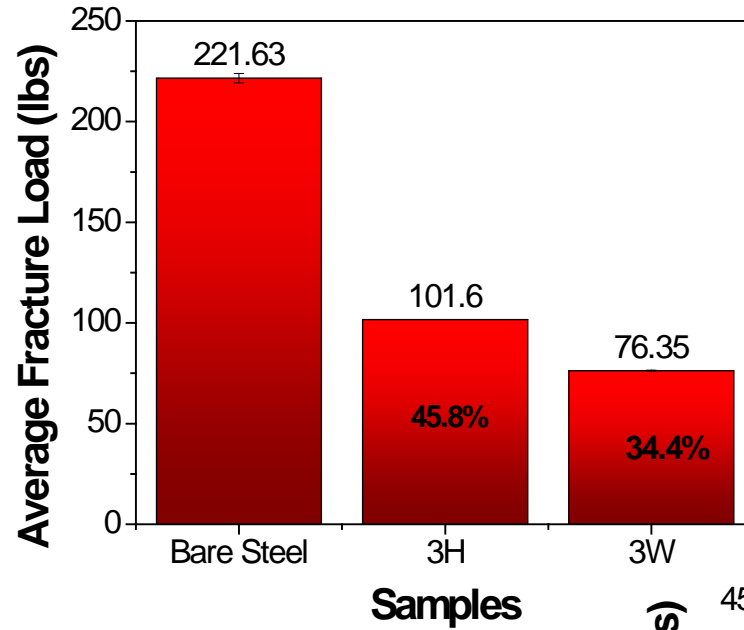
*Sriraman, K. R., S. Brahimi, J. A. Szpunar, J. H. Osborne and S. Yue,*  
 Characterization of corrosion resistance of electrodeposited Zn-Ni, Zn  
 and Cd coatings, *Electrochimica Acta*, **105**, 314-323, 2013

# Comparison Zn vs Zn-Ni

## Zn Coating



50  $\mu$ m  
5.9  $\pm$  0.6  $\mu$ m



50  $\mu$ m  
14.2  $\pm$  3.8  $\mu$ m

# What is “LHE” Zn-Ni?

## **LHE Zn-Ni**

- Low Hydrogen Embrittlement
- High H permeability
- Ni 12-16%
- Gamma Zn-Ni deposit
- No brighteners – dull appearance
- Cauliflower – columnar structure
- Coating has through cracks
- Cracks more with baking

## **Bright Zn-Ni**

- Low H permeability
- Less than 10% Ni
- Brighteners - shiny and esthetic
- Platelet structure
- Uniform and continuous (defect free)



# Zinc Nickel Specifications

## Aerospace

- **AMS2461** Plating, Zinc-Nickel Alloy (12 to 16% Ni) Issued 2020-11 **(LHE) (Fasteners and Standard Parts)**
- **MIL-DTL-32648** (USAF) ZINC NICKEL Plating, Low Hydrogen Embrittlement Electro Deposition **(LHE)**
- **prEN 4826** (ASD-STAN STANDARD) Aerospace series Zinc-nickel (12-16% Ni) plating of steels with specified tensile strength  $\leq 1\,450$  MPa, copper, copper alloys and nickel alloys and aluminium alloys for parts and fasteners **(LHE)**

## Non-Aerospace

- **MIL-PRF-32647** Performance Specification ZINC-NICKEL Electroplating for Fasteners **(Fastener specific)**
- **ASTM B841-18** Standard Specification for Electrodeposited Coatings of Zinc Nickel Alloy Deposits
- **ASTM F1941 / F1941M** – 16 Standard Specification for Electrodeposited Coatings on Mechanical Fasteners, Inch and Metric **(Fastener specific)**
- **ISO 4042 Fasteners** — Electroplated coating systems **(Fastener specific)**

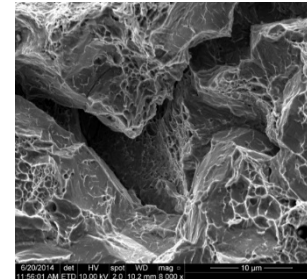


# JUST RELEASED! ISO TR 20491:2019



## *Fundamentals of hydrogen embrittlement in steel fasteners*

- 4-5 years of development in ISO TC 2
- Significant milestone for the fastener industry
- Will serve as basis for discussions related to fastener hydrogen embrittlement
- Will inform the evolution of fastener standards and practices around the world
- Available at the [IFI Web Store](#). Or ISO Online Collection



TECHNICAL  
REPORT

ISO/TR  
20491

First edition  
2019-02

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**Fasteners — Fundamentals of  
hydrogen embrittlement in steel  
fasteners**

*Fixations — Principes de la fragilisation par l'hydrogène pour les  
fixations en acier*

# Publications

- Priyadarshi Behera P., Rajagopalan, S.K., Brahimi, S.V., Venturella C.A., Gaydos S.P., Straw R.J., Yue S., 2021, *Effect of brush plating process variables on the microstructures of Cd and ZnNi coatings and hydrogen embrittlement*, *Surface and Coatings Technology*, Volume 417, 2021, 127181, ISSN 0257-8972, <https://doi.org/10.1016/j.surfcoat.2021.127181>.
- ISO/TR 20491:2019 *Fasteners - Fundamentals of hydrogen embrittlement in steel fasteners*. International Organization for Standardization (ISO), Switzerland, 2019.
- Melo, E.B., Behera, P., Sriraman, K.R., Brahimi, S.V. and Yue, S., 2017. Hydrogen Permeation and TDS Quantification Following Corrosion of Sacrificial Coatings. In International Hydrogen Conference (IHC 2016): Materials Performance in Hydrogen Environments. ASME Press.
- Behera, P., Sriraman, K.R., Lee, L., Chromik, R.R., Yue, S. and Brahimi, S., 2015, May. Corrosion Behaviour of Ion Vapor Deposited (IVD) and Electroplated Al Coatings On Steel Substrates. In CORROSION 2015. NACE International.
- Behera, P., Sriraman, K.R., Lee, L., Brahimi, S., Yue, S. and Chromik, R.R., 2015, May. Hydrogen Permeability of Ion Vapor Deposited (IVD) and Electroplated Al Coatings by Electrochemical Permeation Cell Technique. In CORROSION 2015. NACE International.
- Brahimi, S., *Fundamentals of Hydrogen Embrittlement in Steel Fasteners*, IBECA Technologies – Industrial Fasteners Institute, [www.indfast.org/shop](http://www.indfast.org/shop), July, 2014.
- Sriraman, K.R., Brahimi, S., et al., *Hydrogen embrittlement of Zn, Zn-Ni and Cd coated high strength steel*. *Journal of Applied Electrochemistry*, 2013. **43**(4): p. 441-451.
- Sriraman, K.R., Brahimi, S., et al., *Characterization of corrosion resistance of electrodeposited Zn-Ni, Zn and Cd coatings*. *Electrochimica Acta*, 2013. **105**: p. 314-323.
- Sriraman, K.R., Brahimi, S., et al., *Tribocorrosion behavior of electrodeposited Zn, Zn-Ni Cd and Cd-Ti coatings on low carbon steel substrates*. *Surface & Coatings Technology*, 2013. **224**: p. 126-137.
- Sriraman, K.R., Brahimi, S., et al., *Tribological behavior of electrodeposited Zn, Zn-Ni, Cd and Cd-Ti coatings on low carbon steel substrates*. *Tribology International*, 2012. **56**: p. 107-120.
- Sriraman, K.R., Brahimi, S., and Yue, S., *Characterization of hydrogen embrittlement in Zn, Zn-Ni, Cd and Cd-Ti coated steel*, in *Hydrogen 2012, International conference on Hydrogen-Materials interaction 2012*: Jackson Hole, Wyoming.



**NSERC  
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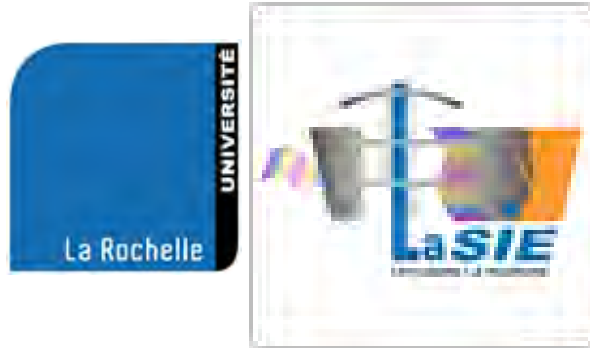
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# Academic collaborations



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THANK YOU!