IBECA

IBECA Technologies Corp.

Perspectives on Hydrogen Embrittlement on Coated High Strength Fasteners

Salim Brahimi ing.
IBECA Technologies / McGill University

RCSC Annual Meeting
Estes Park, Co
June 6, 20124

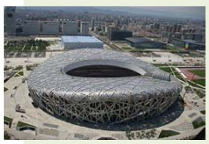
Outline

- Background
- HE Theory
- Research Highlights
- SFOBB rod failure case
- Coatings and Hot Dip Galvanizing and high strength bolts
- Outlook and future direction with coatings

Perspective: Fastener Engineering

- Fastener manufacturing
- Bolting and joint engineering
- Fastener standard specifications (ASTM, SAE, ISO)

- 25 years of experience, case studies, failure investigations

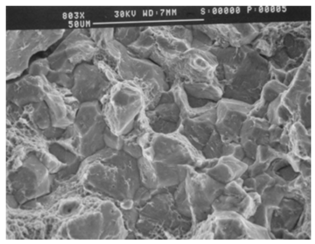


3

IBECA

IBECA Technologies Corp.

Some Hydrogen Embrittlement Theory



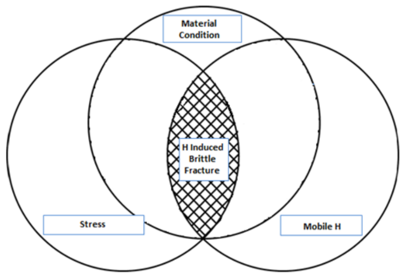
Definition

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

Courtesy IBECA

Conditions for HE Failure

1. **Susceptibility**
2. **Hydrogen**
3. **Stress**



4. TIME!!

Page 3

IHE: Internal Hydrogen Embrittlement

Residual H from **processing**

➔ Electroplating
➔ Pickling

4

EHE: Environmental Hydrogen Embrittlement

H from external sources, e.g. **Corrosion** ➔ H rich environment

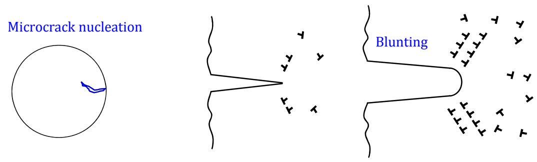
Stress corrosion cracking **(SCC)**

Cathodic hydrogen absorption **(CHA)**

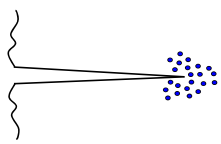
HE Mechanism

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

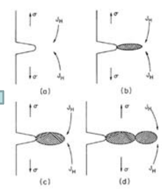
Ductile fracture



H induces a ***ductile-to-brittle*** *transition*

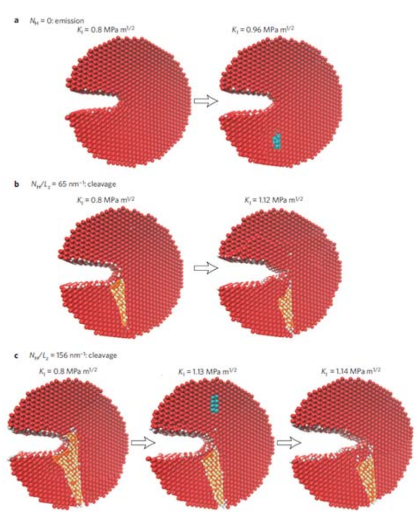


Sequential brittle fracture!



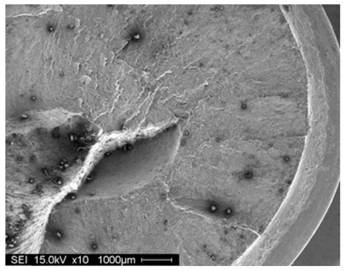
Crack propagation mechanism

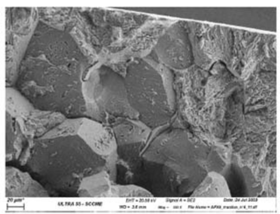
Modelling Atomic Scale Mechanism

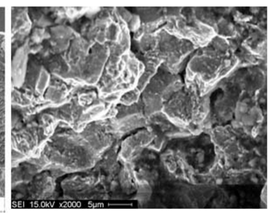


Jun Song and W. A. Curtin Nature Materials 2012

Fracture Morphology





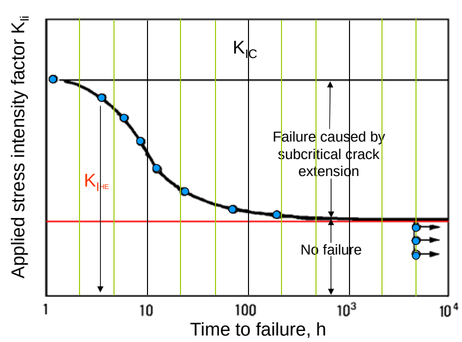


Courtesy IBECA

Quantifying Susceptibility

Characterized by: **Threshold Stress**

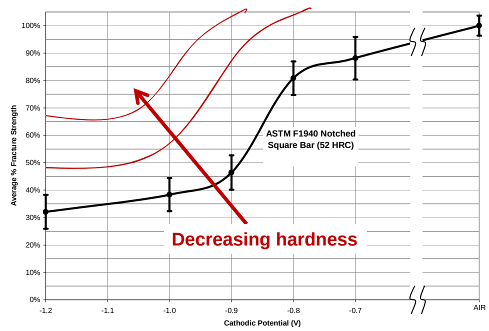
K_{IHE} or K_{ISCC}



Source: ASM Handbook Vol.13

Given amount of H^+

HE Threshold Curve



ASTM F1940
Brahimi, AESF 1999

Threshold stress is affected by:

- **Strength** ↑
- Ductility ↑
- Toughness ↑

Threshold Stress ↓ ↑ ↑ ⟺ **Stress intensity**

- Composition
- Microstructure
- Temperature

⟺ **H transport kinetics**

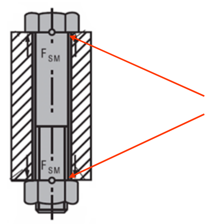
K_{IHE} must be measured

High Strength Fasteners

- High strength ➔ 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 filet radius

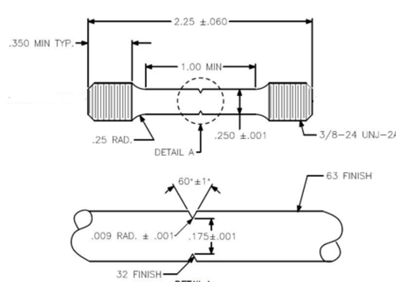


Page 13

IBECA

IBECA Technologies Corp.

Hydrogen Embrittlement Test Methods



Basic Principles

The basic premise for HE testing is to allow "**TIME**":

- H Transport
- H assisted cracking

Purpose →

- Production parts, or
- Witness specimens for process control

Environment →

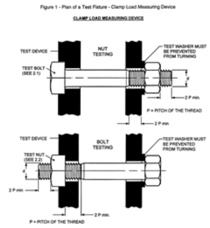
- Air (IHE)
- Solution, e.g. 3.5% NaCl (EHE)

HE Test Loading Methods

- **Sustained Load (SL) Tests**
- **Slow Strain Rate (SSR) Tests**
- **Incremental Step Load (ISL) Tests**
- **Hybrid Methods**

Sustained Load (SL) Test

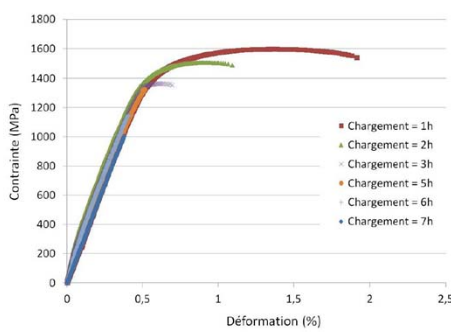
- Tighten the fastener and hold it!
- 24-200 hours depending on the test spec.
- Pass/fail method (not quantitative)
- Good method for production parts



Courtesy IBECA

Slow Strain Rate (SSR) Test

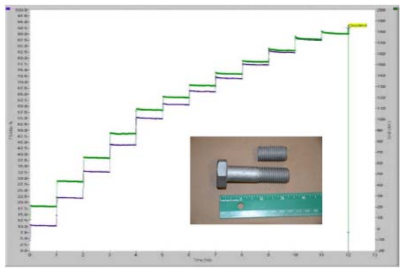
- <u>Slowly </u>increase the load
- Quantitative method
- Measures loss of ductility
- Good research Tool



Flurentin et al. Cetim 2013

Incremental Step Load (ISL) Test

- Modified version of sustained load test.
  - Tensile load
  - Bending load
- Measures fracture strength
- Not suitable for soft materials



Courtesy IBECA

Product Testing

- **NASM 1312-12** *Fastener Test Methods – Part 5: Stress Durability (200 h)*
- **ASTM F606 (Section 7)** *Standard Test Methods for Determining the Mechanical Properties of Externally and Internally Threaded Fasteners, Washers, Direct Tension Indicators, and Rivets (24-48 h)*
- **ISO 15330** *Fasteners -- Preloading test for the detection of hydrogen embrittlement -- Parallel bearing surface method*
- **ASTM B839** *Standard Test Method for Residual Embrittlement in Metallic Coated, Externally Threaded Articles, Fasteners, and Rod—Inclined Wedge Method (24-48h)*

Process Verification

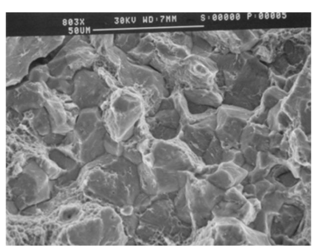
- **ASTM F519** *Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments*

- **ASTM F1940** *Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners*

IBECA

IBECA Technologies Corp.

Research Highlights: Coatings and Coating Processes



What can be done to manage the risk of HE?

- Applications engineering → develop robust design
- **Minimize hydrogen from coating processes**
- **Design better coatings**
- **Design and select materials with a low susceptibility to HE failure**

Research Objectives: Coatings

Investigate the effects of coating **process variables** and **coating characteristics** on **IHE** of steel fasteners

Long term objective:

Develop guideline for industry to optimize coating processes

Develop low HE coatings and coating processes

Research Objectives: **Materials**

Correlate HE **susceptibility** with metallurgical and microstructural characteristics in HS steels

- Rank the HE **susceptibility** of **materials** used to make **fasteners**
- Better understand the roles of **composition**, **microstructure** and **strength** on **H trapping and transport**, and ultimately on susceptibility
- Design **new high strength steels** with **lower HE susceptibility**

Page 25

McGill University – History of Excellence

James McGill

McGill University, Montreal

1813 James McGill, a Scottish immigrant who prospered in Montreal, bequeathed his 46-acre estate and 10,000 pounds to "the Royal Institution for the Advancement of Learning."

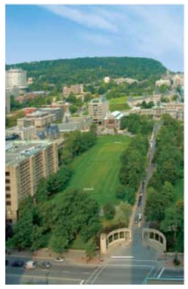
1829 McGill College (now McGill University) was inaugurated in Burnside Place, James McGill's country home.

1843 the University constructed its first buildings, the central and east wings of the Arts Building.

1884 the first women students were admitted

26

Quick Facts



- The downtown campus has 104 buildings on 80 acres. Twenty-four of these are heritage properties.
- ~ 37,000 students
- ~ 8000 degrees granted every year
- Most Rhodes Scholars of any Canadian university

McGill is Canada's best-known university, ranked among the world's finest universities

Notable Firsts

Nature of radioactivity (**Ernest Rutherford**)

Epilepsy researcher and world famous neurosurgeon **(Wilder Penfield)**

First artificial cell (**Thomas Chang**)

First Internet Search Engine (**Peter Deutsch, Alan Emtage, Bill Heelan**)

Inventor of the Charge Coupled Device used in digital cameras and photocopiers (**Willard Boyle**)

First game of organized hockey March 3, 1875, at the Victoria Skating Rink in downtown Montreal

McGill vs. Harvard: First intercollegiate football game was played on May 14, 1874

RESEARCH PARTNERS

McGill

NSERC CRSNG

CFI Canadian Fasteners Institute

IFI Industrial Fasteners Institute

IBECA

INFASCO

BOEING

boltcouncil
Research Council on Structural Connections
rcsc

ASTM

NASF
National Association for Surface Finishing

Subommittee F16.96

Department of the Navy
United States of America

NUCOR
FASTENER DIVISION

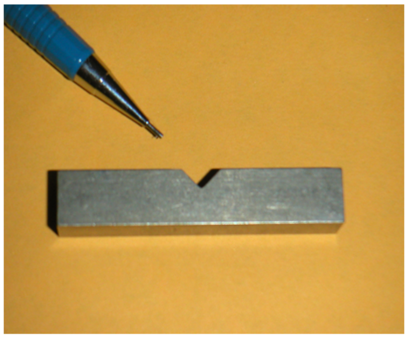
29

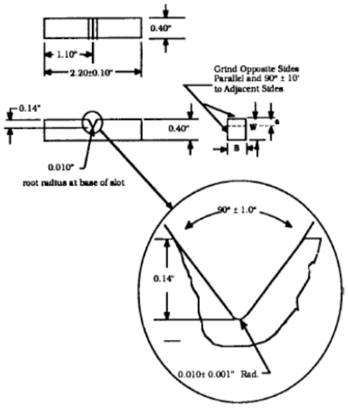
Comparing susceptibility of fastener steel grades

RESULTS

1

Specimen type



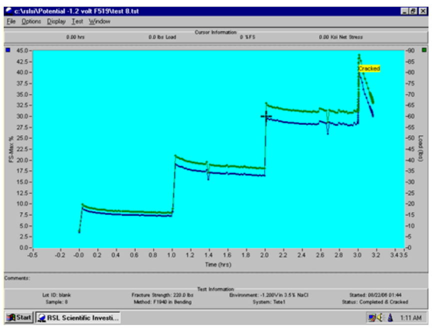


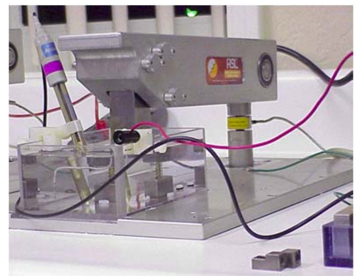
Notched square bar – ASTM F519 type 1e

2

Environmental testing

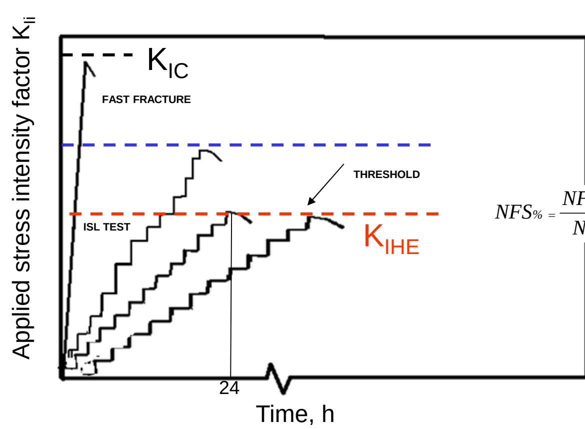
ISL test at -1.2 V cathodic potential





Threshold determination

Incremental Step Load (ISL) Test
Accelerated method to measure **threshold stress**



$$NFS_{\%} = \frac{NFS_{F1624}}{NFS_{(B)}} \times 100$$

Notch Fracture Strength = Threshold

$$NFS_{\%} = \frac{NFS_{F1624}}{NFS_{(B)}} \times 100$$

- ⇨ $NFS_{\%}$ = Percent notch fracture strength
- ⇨ NFS $_{(B)}$ = Fast fracture load of specimen in air (baseline)
- ⇨ $NFS_{(W)F1624}$ = Lowest notch fracture load of specimen under imposed potential

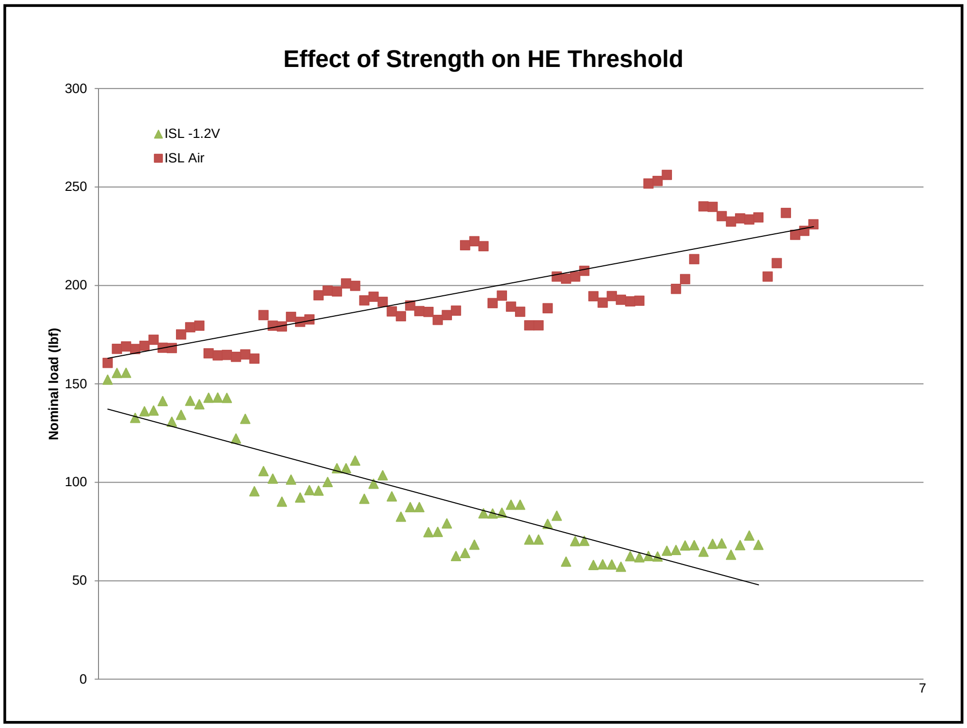
5

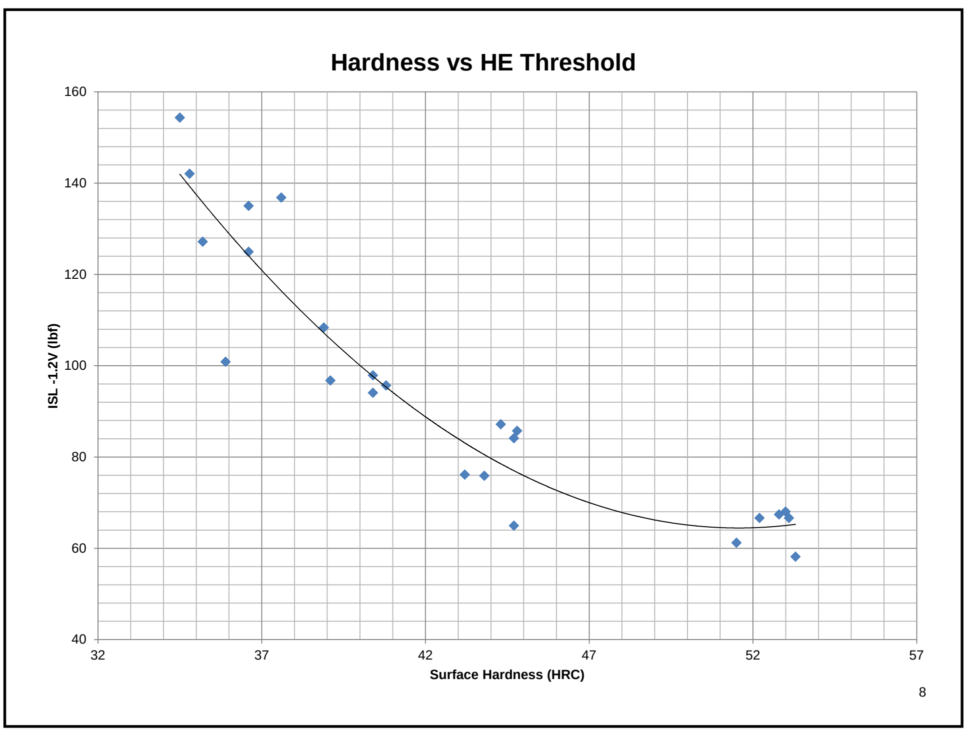
Materials and specimens

- Select list of materials to be tested
- Acquire materials
- Manufacture test specimens: 35, 39, 44, 53 HRC
- > 3000 specimens ➔ 2 + years

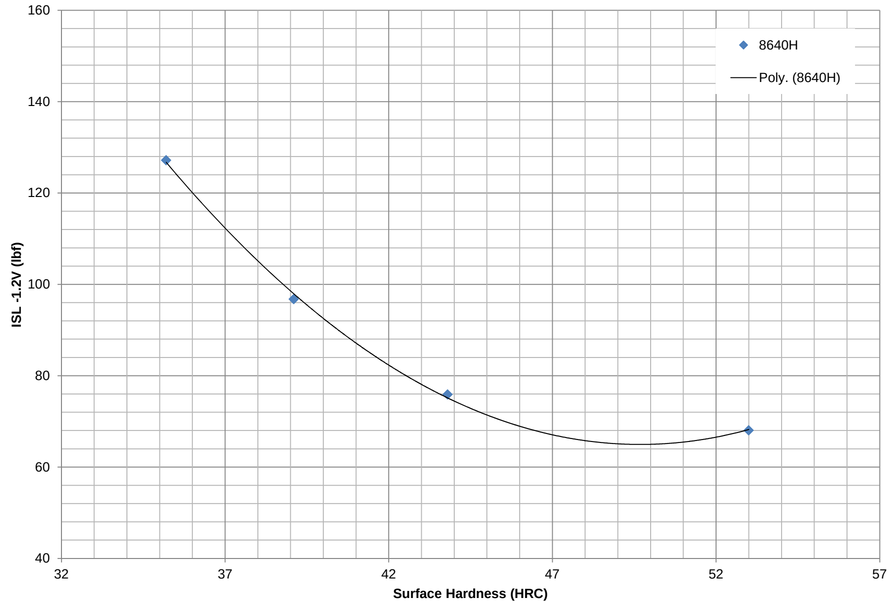
Plain carbon steels	Alloy steels	Boron steels	Stainless steels
1039	4340 air melt	10B21	A286
1541	4340 vac melt	10B38	PH13-8Mo
	4140		Aermet 100
	8637		
	5140		
	4042		
	4135 sph ann		
	4135 as rolled		

6


Effect of Strength on HE Threshold
300
250
200
150
100
50
0
Nominal load (lbf)
ISL -1.2V
ISL Air
7

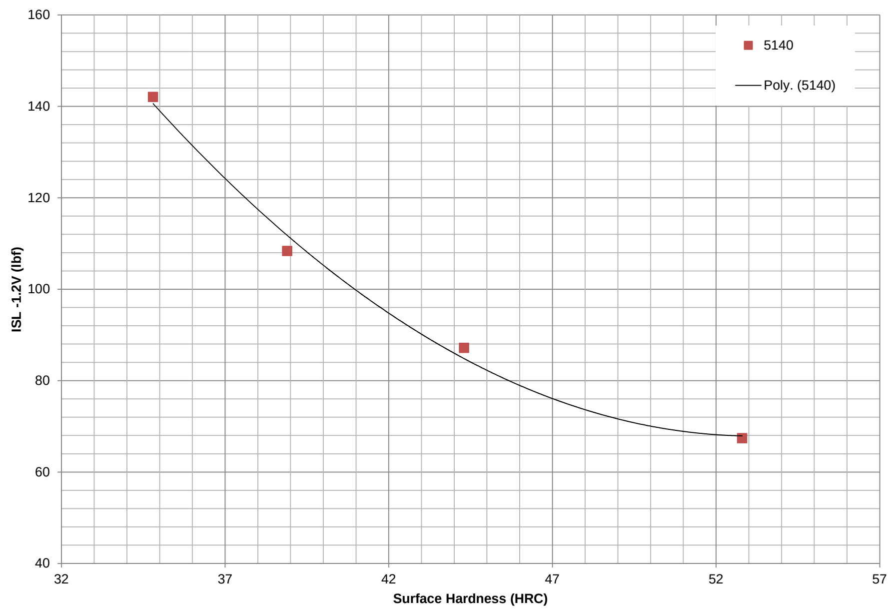

Hardness vs HE Threshold
160
140
120
100
80
60
40
ISL -1.2V (lbf)
32
37
42
47
52
57
Surface Hardness (HRC)
8

Hardness vs HE Threshold

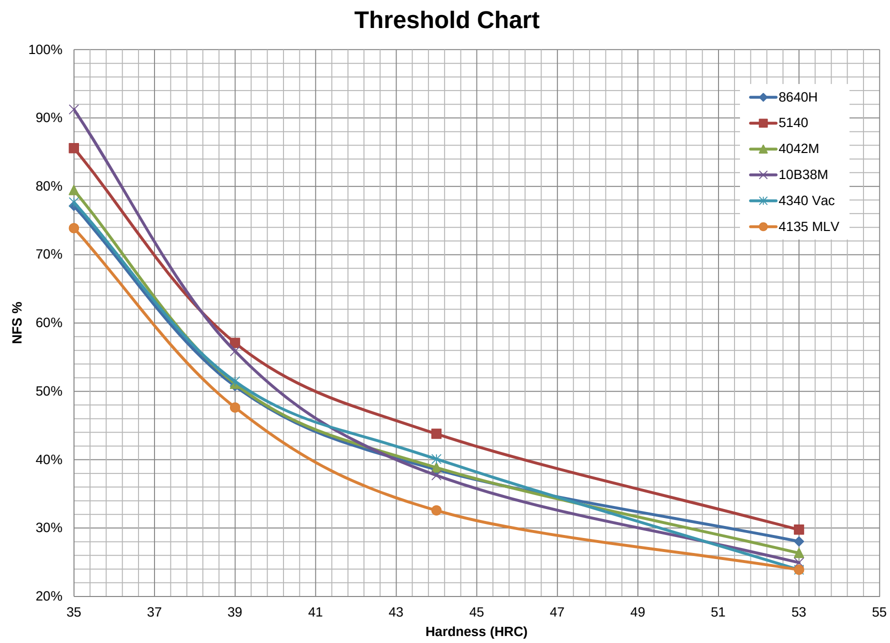


9

Hardness vs HE Threshold

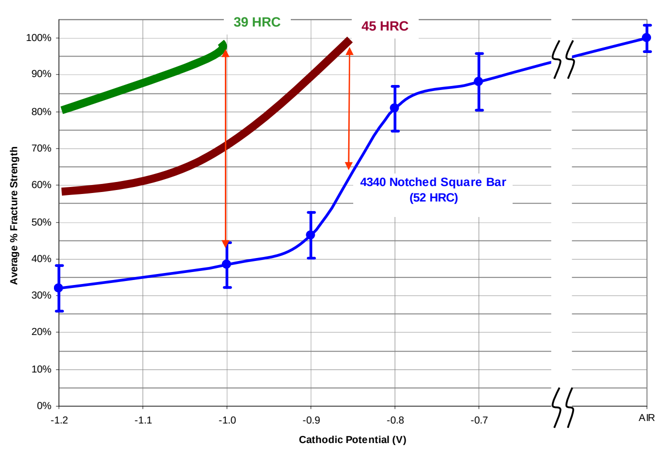


10



11

Threshold curve



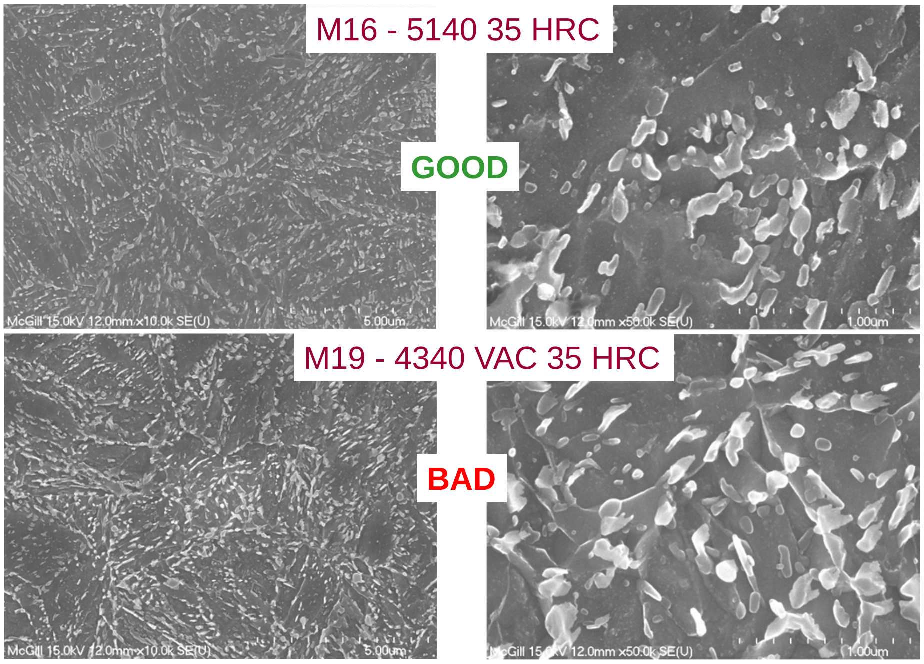
12

M16 - 5140 35 HRC

GOOD

M19 - 4340 VAC 35 HRC

BAD



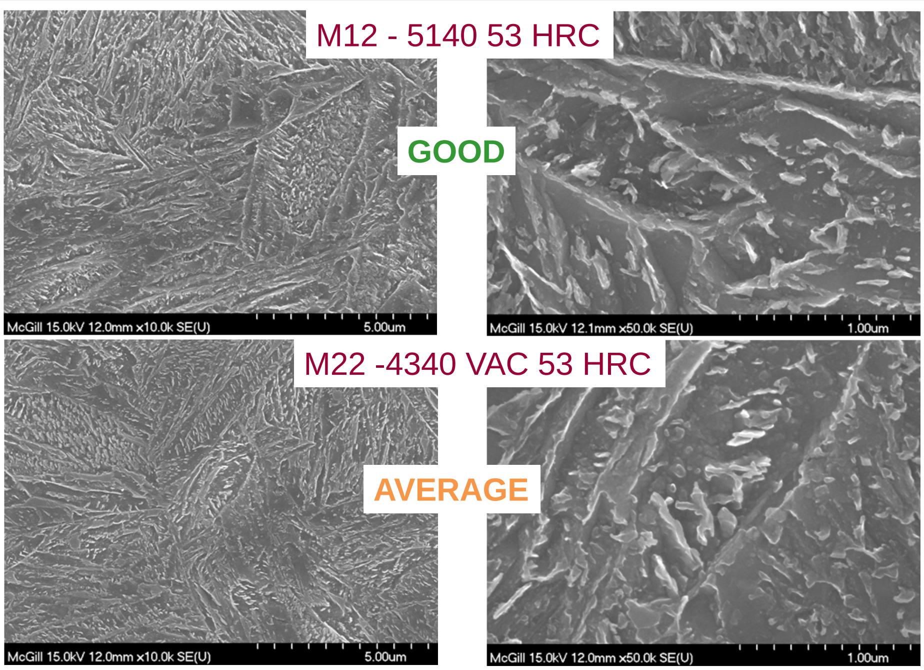
13

M12 - 5140 53 HRC

GOOD

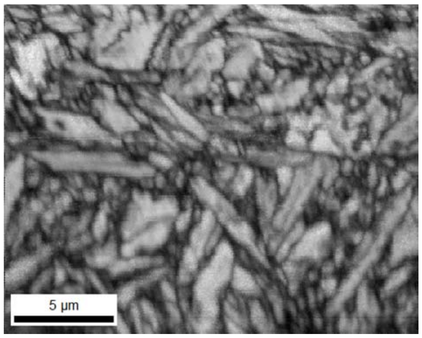
M22 -4340 VAC 53 HRC

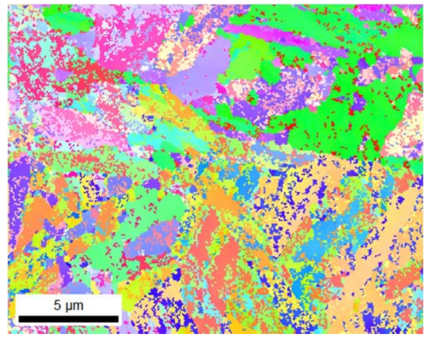
AVERAGE

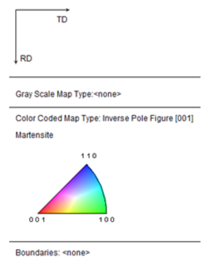


14

EBSD- Texture and GB Orientation

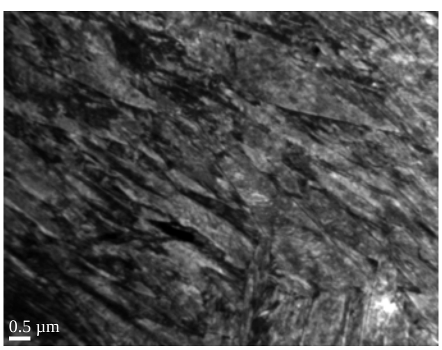


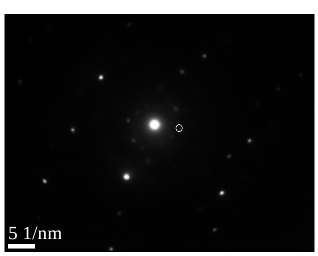


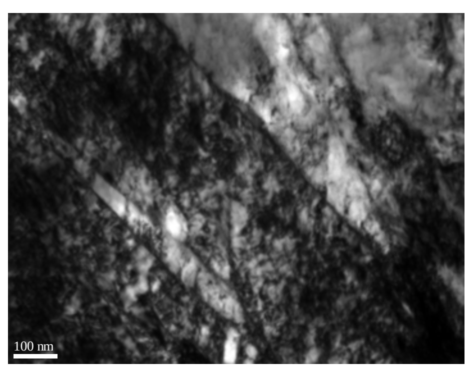


15

Transmission Electron Microscopy





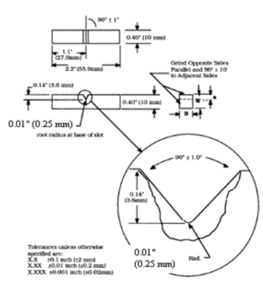


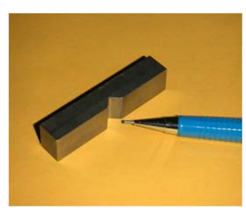
16

Coatings: processes and materials
Some Research Results

RESULTS

Standard test specimen (ASTM F519)





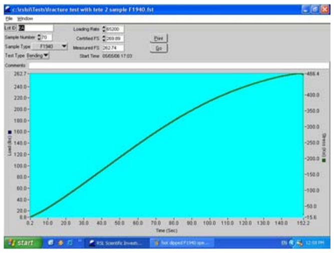
Hardness: 51-53 HRC

- ***Preheated*** *at 538 °C for 2 hours*
- ***Austenitised*** *at 816 °C for 45 minutes at temperature*
- ***Quenched*** *in oil at 70 °C*
- ***Air tempered*** *at 246 °C for 2 hours at temperature followed by air cooling*
- ***Double air tempered*** *at 266 °C for 2 hours at temperature followed by air cooling*
- *Manufacturer certified average surface hardness:* ***51.4 HRC***

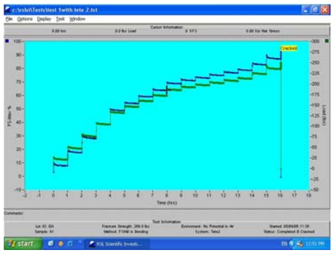
Final machining

- ***Stress Relief*** *at 190 °C for 4 hours*

Testing Protocol



Fast fracture in air



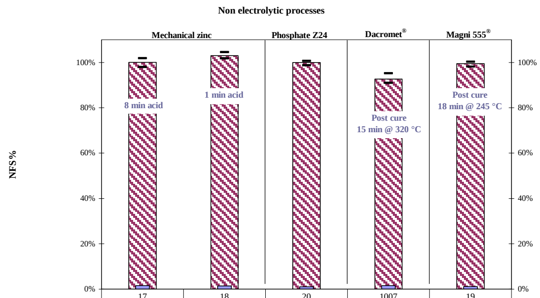
ISL fracture in air

Coating processes evaluated

~60 processes/conditions tested

- 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

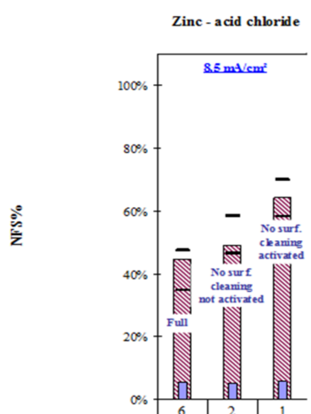
Non Electrolytic Processes



	17	18	20	1007	19
Average	100.1%	103.0%	100.0%	92.7%	99.5%
Standard deviation	1.54%	1.40%	1.00%	1.54%	1.12%
Minimum	98.0%	101.8%	98.8%	91.0%	98.2%
Maximum	101.9%	104.6%	100.7%	95.2%	100.3%

Brahimi S., Yue S., 2008

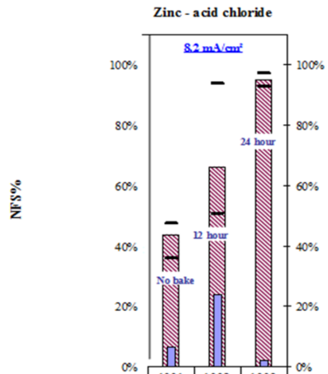
Zinc – Acid Chloride



	6	2	1
Average	44.7%	49.1%	64.2%
Standard deviation	5.42%	5.26%	5.81%
Minimum	35.0%	46.7%	58.4%
Maximum	47.6%	58.5%	70.0%

Brahimi S., Yue S., 2008

Effect of baking time at 200 °C (~400 °F)

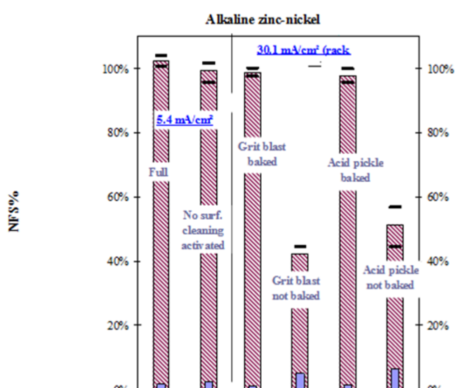


	1001	1002	1003
Average	43.8%	66.2%	95.0%
Standard deviation	6.69%	24.07%	2.17%
Minimum	36.0%	50.8%	92.9%
Maximum	47.6%	93.9%	97.2%

Brahimi S., Yue S., 2008

Zinc-Nickel Alkaline

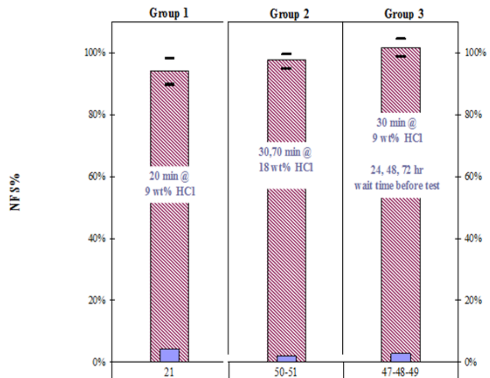
Very low to low efficiency 30-50%



	8	7	36	37	38	39
Average	102.4%	99.3%	98.7%	42.2%	97.7%	51.4%
Standard deviation	1.73%	2.61%	1.04%	4.98%	1.53%	6.35%
Minimum	100.5%	95.6%	97.6%	33.3%	95.6%	44.4%
Maximum	103.9%	101.7%	100.1%	44.5%	99.9%	56.8%

Brahimi S., Yue S., 2008

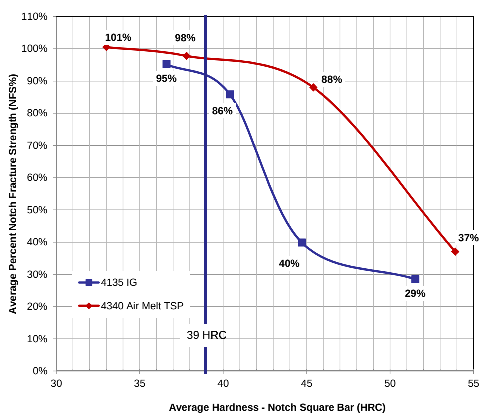
Acid Exposure Without Barrier Coating



	21	50-51	47-48-49
Average	94.2%	97.7%	101.7%
Standard deviation	4.22%	2.06%	2.88%
Minimum	89.8%	95.0%	98.8%
Maximum	98.2%	99.6%	104.5%

Brahimi S., Yue S., 2008

HE Susceptibility of Zn Electroplated Steel



Brahimi S., 2013

Summary Findings
Processes

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

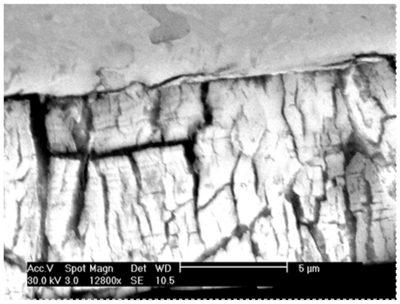
Results support grouping into two basic parameters affecting IHE:

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

Summary Findings
Baking

- Baking at min. 400 ° F (204 °C) can fully restore ductility
- However… baking response depends upon the permeability of the coating
  ➔ Therefore 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**

More Results: Coating Characterization



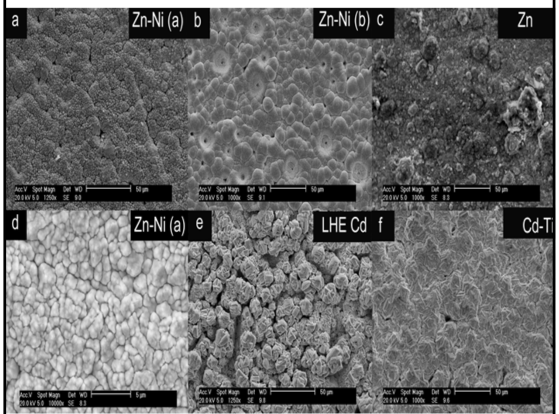
Defect characterization Zn-Ni

Interface shows large inter-connected cracks ➔ path for hydrogen diffusion

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**

Page 13

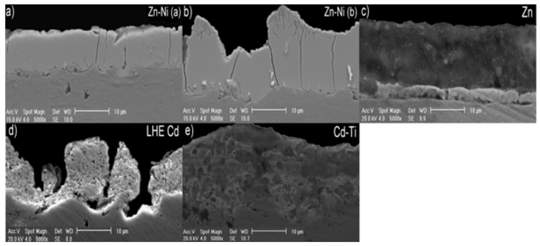
Surface Morphology



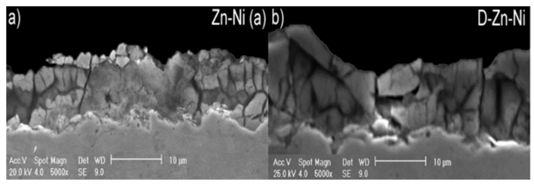
Sriraman, K. R., S. Brahimi, J. A. Szpunar, J. H. Osborne and S. Yue, Characterization of corrosion resistance of electrodeposited Zn-Ni, Zn and Cd coatings, ***Electrochimica Acta,* 105, 314-323, 2013**

14

Interface morphology



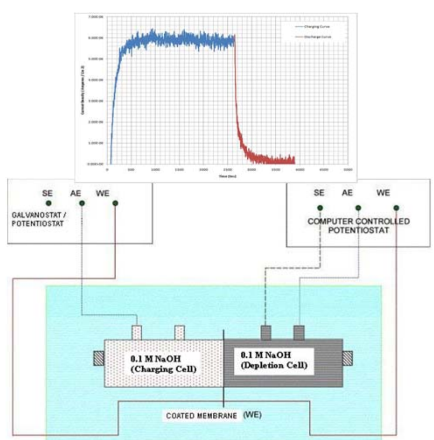
As plated coatings



Baked coatings

Sriraman, K. R., *et al*, ***Electrochimica Acta*, 105, 314-323, 2013** 15

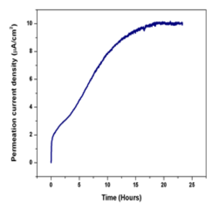
Electrochemical Permeation



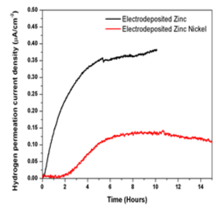
Electrochemical permeation studies

Calculation of effective diffusion coefficient of the coating material

Hydrogen permeation characteristics of steel, Zn and Zn-Ni



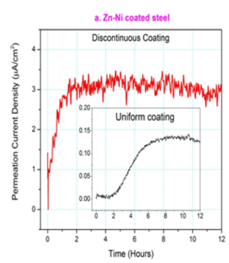
Permeation transients of low carbon steel shim

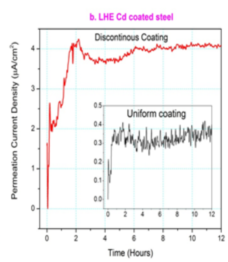


Permeation transients of Zn and Zn –Ni coatings on low carbon steel shim

Material	Diffusion Coefficient D (cm^2s^{-1})	Subsurface hydrogen conc. C_0 (Mol cm^{-3})	Permeation flux J_{ss} (mol $s^{-1}cm^2$)
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

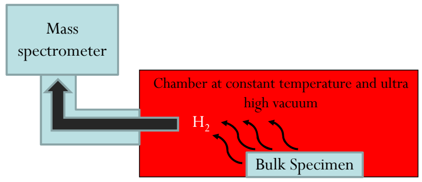
Comparing hydrogen permeability for uniform & discontinuous coatings

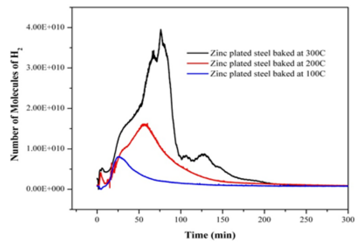




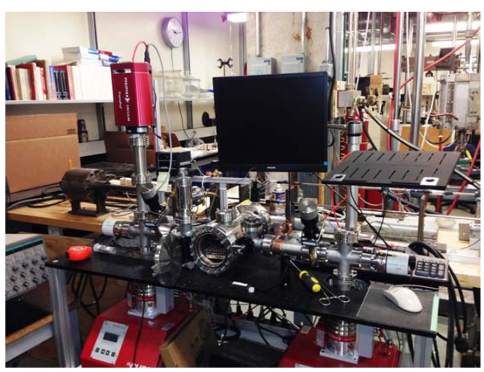
Material	Diffusion Coefficient D X 10^{-08} (cm^2s^{-1})	Subsurface hydrogen conc. C_0 X 10^{-06} (Mol cm^{-3})	Permeation flux J_{ss} X 10^{-11} (mol $s^{-1}cm^{-2}$)
Zn-Ni	0.391	2.711	0.087
Zn-Ni with microcracks	5.778	6.707	3.177
Cd	4.543	1.041	0.378
Cd with discontinuities	25.135	2.080	4.183

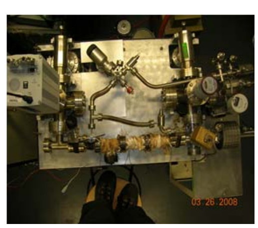
Thermal Desorption Spectroscopy (TDS)





Measure H discharge as a function of time and temperature





Summary

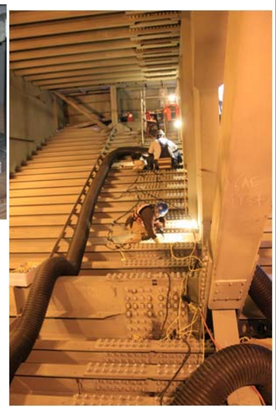
- The Zn-Ni plating process induced microcracks and defects in the coating microstructure.
  Defects expanded during the baking treatment, acting as hydrogen escape pathways.

- Permeability experiments showed that the defect free Zn-Ni coating had superior resistance to hydrogen diffusion

- During the initial stages of plating, the defect free Zn-Ni layer acts as a barrier to hydrogen absorption. During the baking process the defects present in the coating act as a pathway for any hydrogen to diffuse out of the coated steel thereby further minimizing the risk of embrittlement due to plating process.

- From the hydrogen re-embrittlement studies it was shown that Zn-Ni coatings pose minimal risk of hydrogen embrittlement during sacrificial corrosion of the coatings

Some Engineering Implications Metallic Coated A490 Bolts



Courtesy IBECA

Page 22

East Span San Francisco Oakland Bay Bridge (SFOBB)

baybridge360

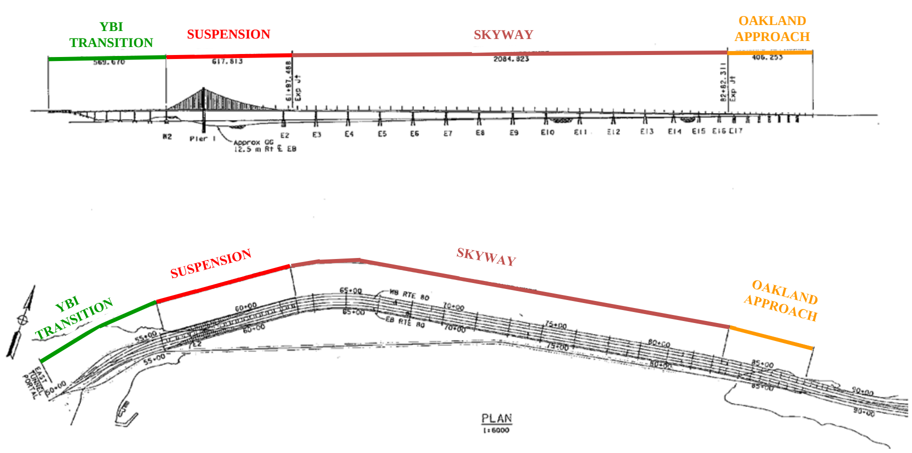
Must understand the material condition

- Is the failure "**normal/common**": ***YES*** / ***NO*** ?
- If YES:
  - ⇨ reduce or eliminate H,
  - ⇨ reduce stress,
  - ⇨ choose less susceptible (i.e. lower strength material
- If No:
  - ⇨ **abnormal** material condition.
  - ⇨ In my experience 10.9 or Gr8 failure is always related to poor material

➔ **Material susceptibility is the key!**

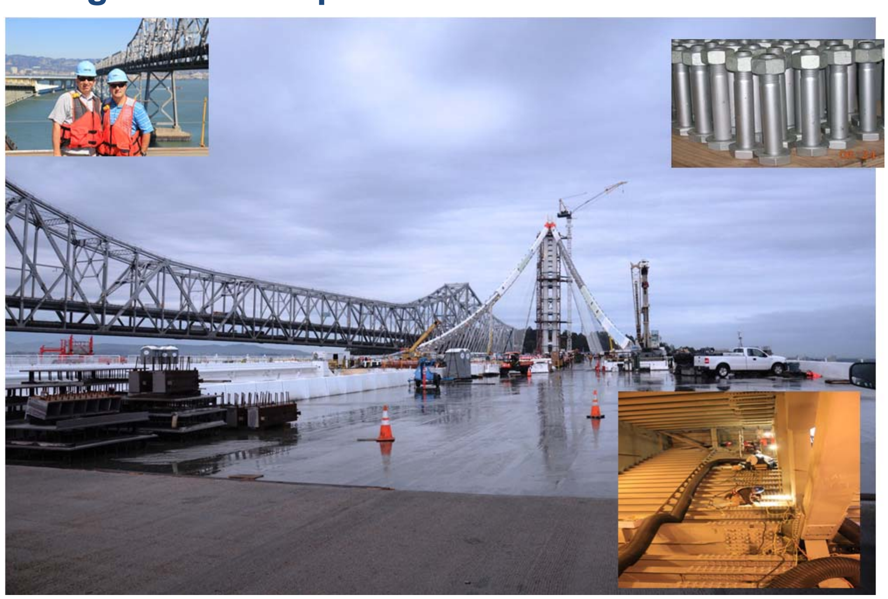
2

SFOBB East Span Segments

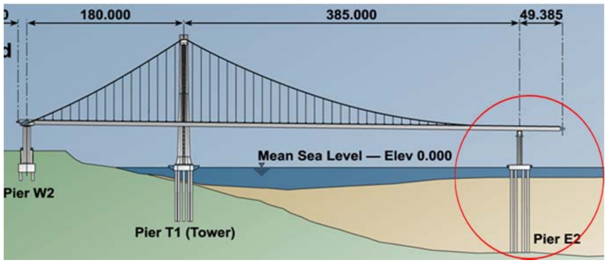


Courtesy Brian Maroney
ASTM F16 Meeting
Jacksonville, Nov. 2013

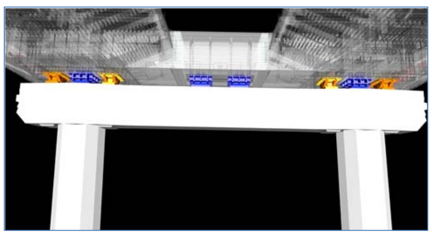
Bridge Near Completion – March 2013



Pier E2 Anchor Rods



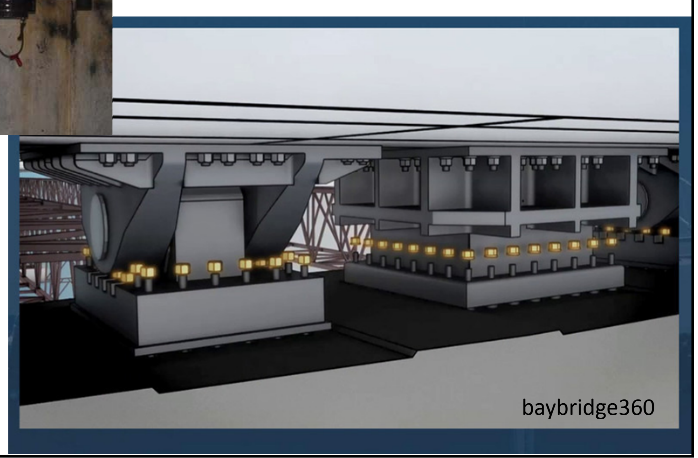
Courtesy Brian Maroney
ASTM F16 Meeting
Jacksonville, Nov. 2013



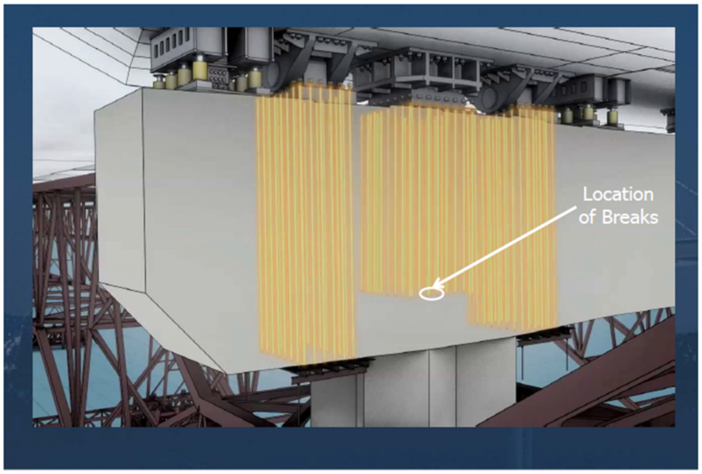
Hydraulic Tensioning of Rods – March 2013

Target Tension

0.75 Fu
➔ 0.70 Fu



Anchor Rod Failures S1/S2



baybridge360

Nut separation days after tensioning

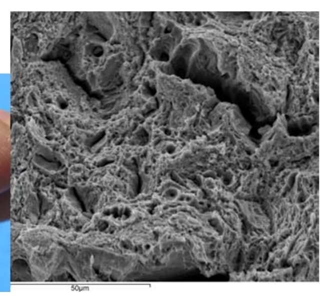
32 of the 96 (2008) A354 BD anchor rods on shear keys S1 /S2 on Pier E2 failed after being tightened to specified tension levels

Chemical / mechanical properties

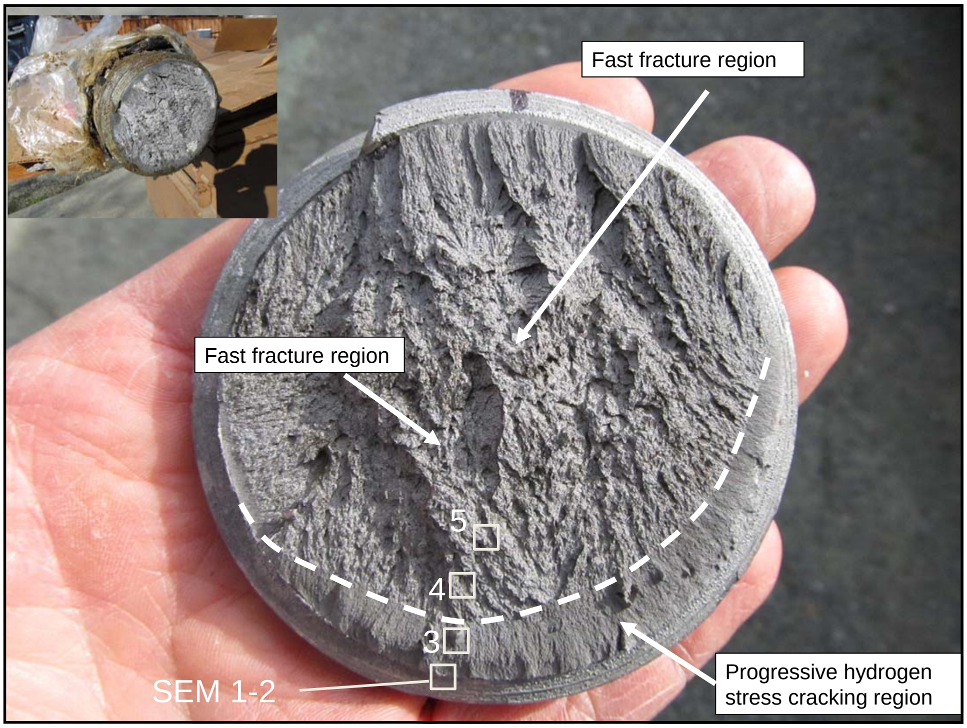
Spectrochemical Analysis (Reported as Wt. %)				
		Mill Test Report(1)	Mill Test Report(2)	Requirement ASTM A354 Gr BD
Aluminum	Al	0.001	0.001	
Carbon	C	0.41	0.41	0.33 -0.55
Chromium	Cr	0.98	0.98	
Cobalt	Co	0.007	0.007	
Copper	Cu	0.20	0.20	
Iron	Fe			
Manganese	Mn	0.92	0.92	0.57 min.
Molybdenum	Mo	0.16	0.16	
Nickel	Ni	0.10	0.10	
Phosphorus	P	0.014	0.014	0.040 max.
Silicon	Si	0.23	0.23	
Sulfur	S	0.034	0.034	0.045 max.
Titanium	Ti	0.002	0.002	
Tungsten	W			
Vanadium	V	0.030	0.030	
Zirconium	Zr			

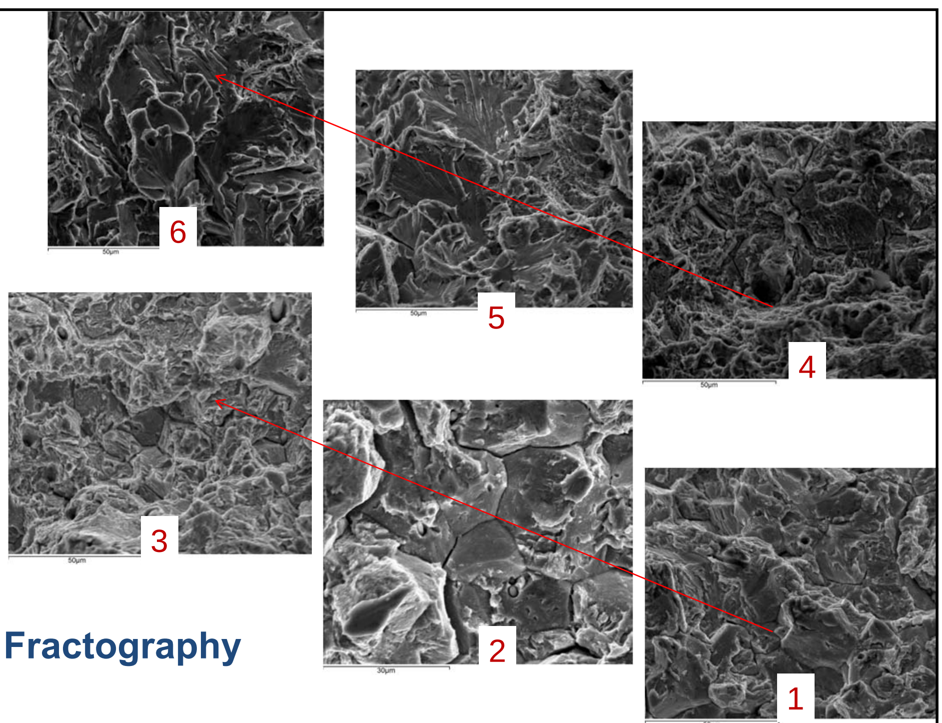
ASTM A354 Gr BD Mechanical Properties	
Yield Strength	115 psi min.
Tensile Strength	140 psi min.
Elongation in 2 inches	14% min.
Reduction in Area	40% min.
Hardness Rockwell C	31 -39

Tensile Testing



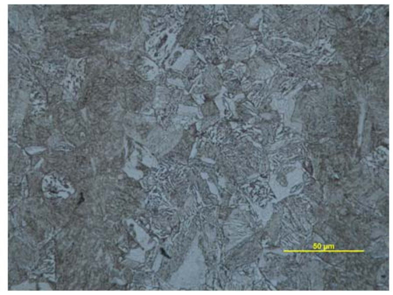
- Material meets **yield** strength, **tensile** strength and **elongation** requirements for A354 grade BD,
- Note: elongation was marginally above the minimum of 14%.





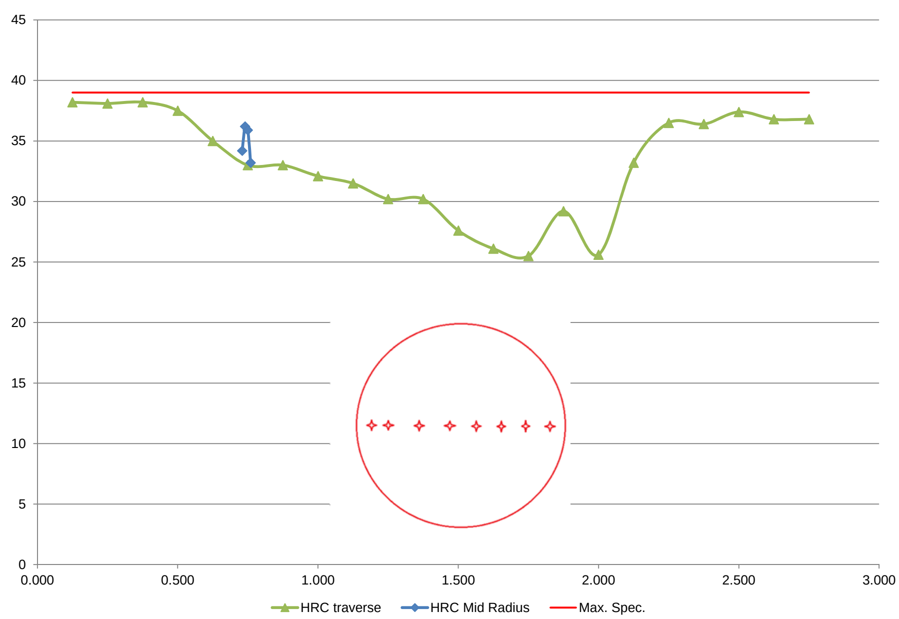
Fractography

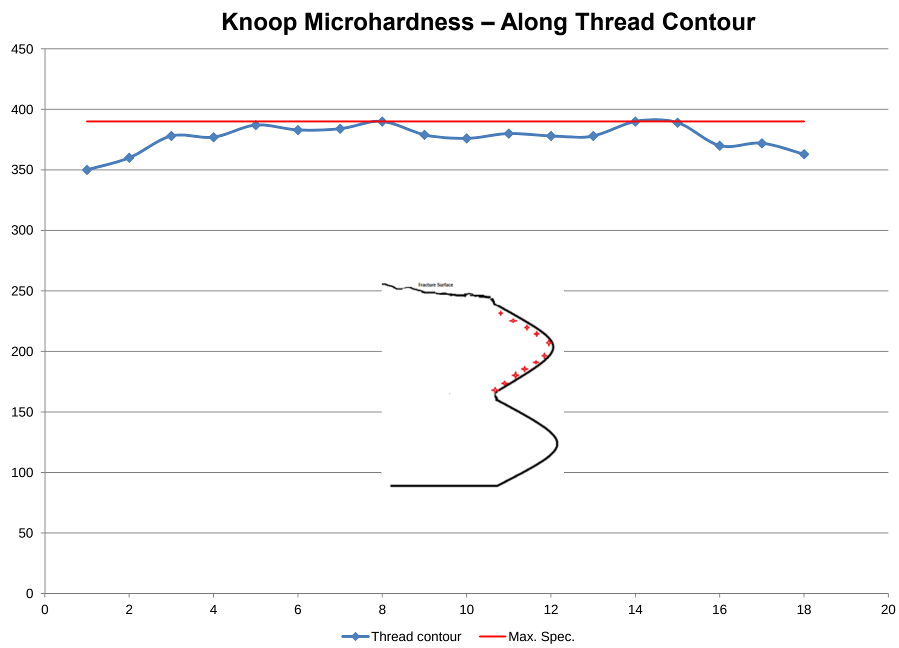
Poor Microstructure



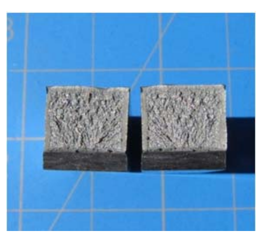
13

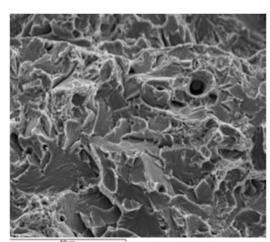
Cross-sectional Macro Rockwell C Hardness Profile





Charpy V-notch Impact Testing





- Results (18-24 J at 25 °C) indicate the material lacks toughness, even when tested at room temperature

Note: Charpy v-notch impact testing is not a requirement of ASTM A354.

Stabdard fastener requirement 27J @ -20 °C

Conclusions of failure investigation

- The metallurgical condition of the steel was poor
  - ⇨ microstructure inhomogeneous
  - ⇨ large difference in hardness from center to edge,
  - ⇨ high local hardness near the surface
  - ⇨ Additional consequence, the material exhibits low toughness and marginal ductility
- The mechanism ➔ hydrogen embrittlement, resulting from:
  - ⇨ Applied tensile load and
  - ⇨ Hydrogen that was already present and available in the rod material as they were tensioned
- The root cause of the failures is attributed to **higher than normal susceptibility of the steel to hydrogen embrittlement.**

Outcomes

- The steel rods comply with the basic mechanical and chemical requirements of ASTM A354 grade BD
  - ➔ A354 is being revised to include better safeguards
- Procurement of future A354 grade BD anchor rods for the SFOBB project to include supplemental requirements to assure against HE failure
- Revision of ASTM A354 is currently underway to include such supplementary requirement
- New Design: Shear Keys S1 and S2 reinforcement

Brahimi, S., Aguilar, R., and Christensen, C., *Metallurgical analysis of Bay Bridge broken anchor rods S1-G1 & S2-A6*, Joint Report, California Department of Transportation – American Bridge/Fluor, May 7, 2013.

Comparison of 3" Diameter E2 Rods

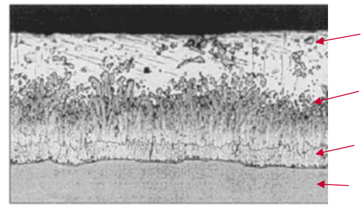
		Manufacturing	Mechanical Testing: Elongation (%)	Mechanical Testing: Reduction of Area (%)	Mechanical Testing: Fu (ksi)	Hardness Range (+/-)	CVN @ 4C (J)
	ASTM A354BD	-	14% min	40 min	140 min[1]	31-39[4]	-
Fabrication Year	2008	-	12.5%-15%	40-50	159-171	26-39	18-24 @ Surface
Fabrication Year	2010	Vacuum Degassing	14%-17%	40-53	153-158	27-38	49-53 @ Surface
Fabrication Year	2013 (4340)	Vacuum Degassing	19%-21%	55-59	160-163	30-37	>68 @ Center 64–67 @ Surface

What happens during hot dip galvanizing?

- Apparent lower tolerance for poor material condition
- The recent example of the Bay Bridge
- But a few others over the years as well
- In all cases they would not have satisfied more stringent standards such as ISO 898-1

20

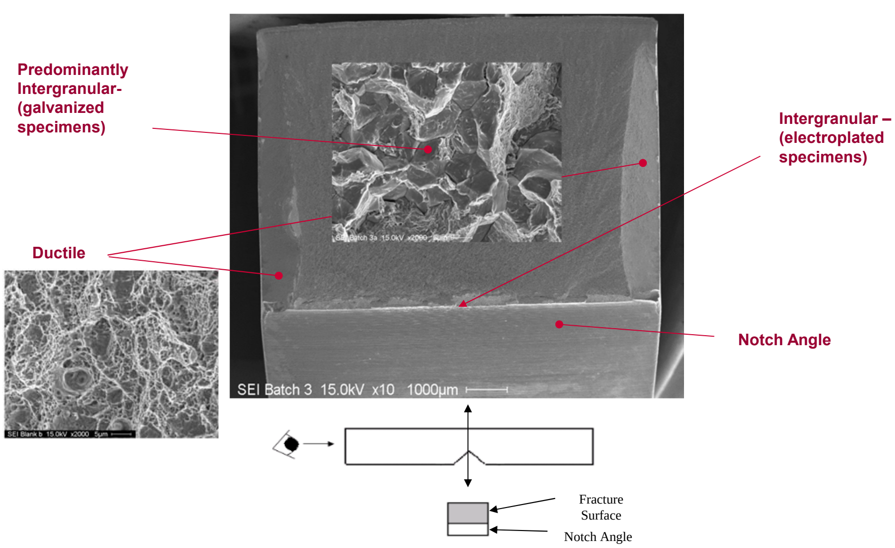
Hot dip zinc - galvanizing



Alkaline cleaning
16 min
25% KOH
80 °C
→
Water rinse
2 min
→
Acid pickling
16 min
10 wt % HCl
moderately heated to 27 °C
→
Water rinse
2 min
→
Flux solution
2 min
ZnCl + NH_4Cl
in solution
78 °C
↓
Drying
16 min.
forced Hot Air
40 °C
→
Transfer
basket
→
Molten zinc immersion
3.0 min @ 450 °C
~99% Zn, ~0.7% Pb, ~0.08% Al
→
Centrifuge
5 sec
to remove excess zinc
↓
Transfer
→
Water cool
30 sec.
→
Drying
air blow

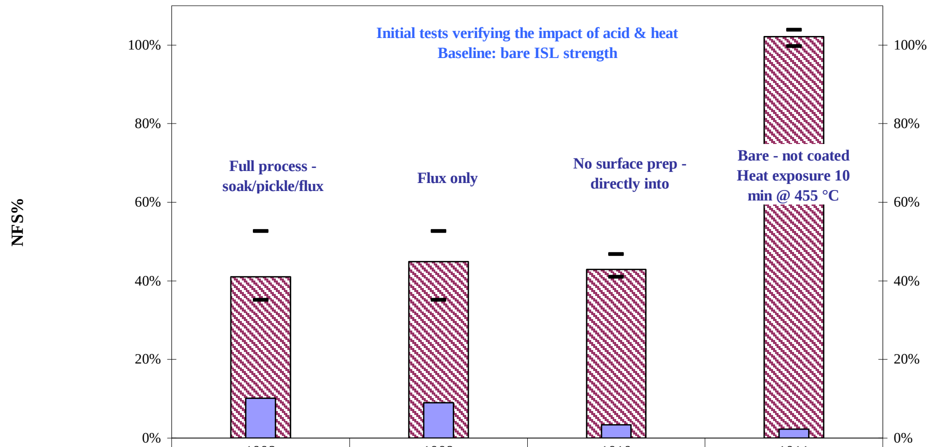
21

Fractography – fracture surface map



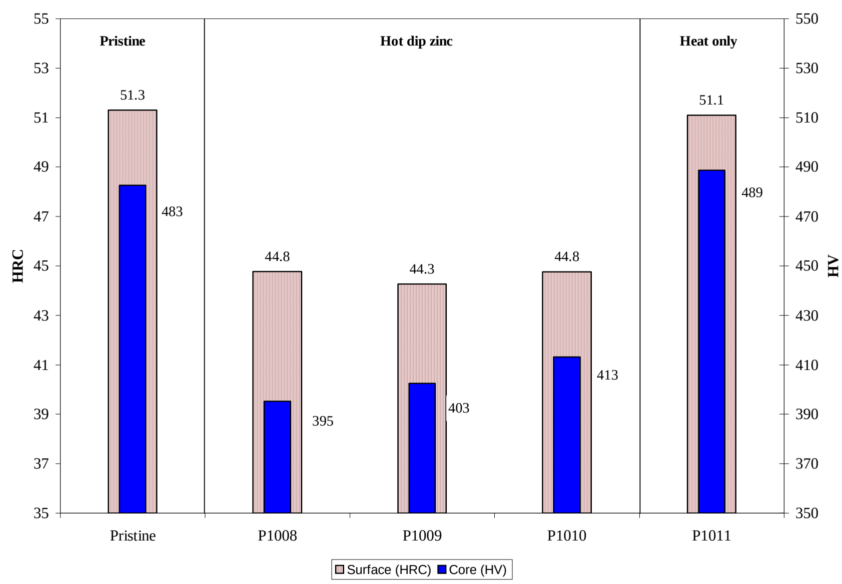
22

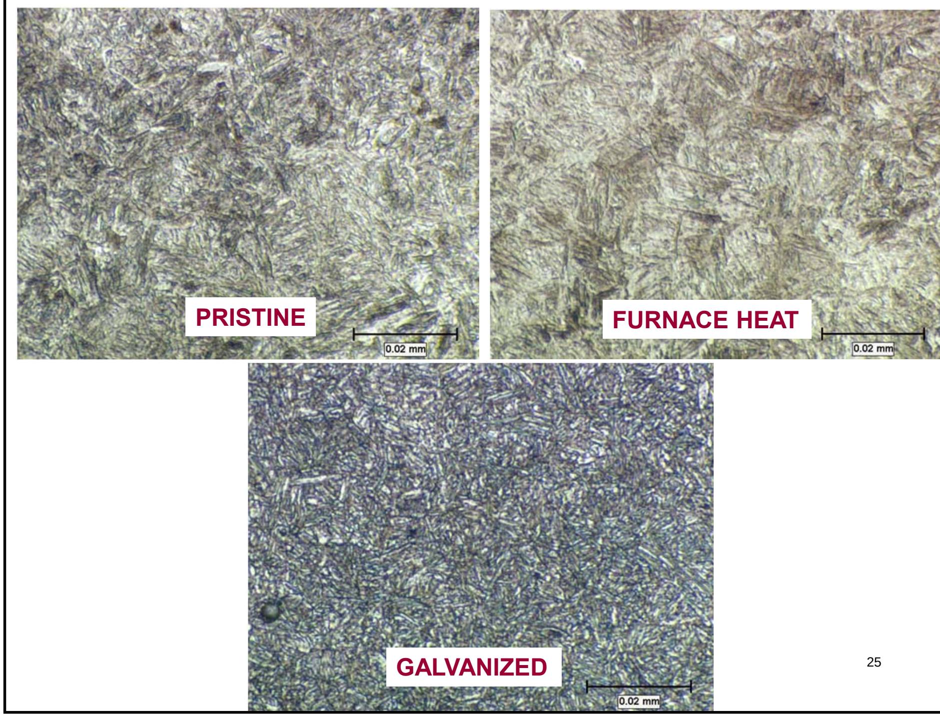
Effect of acid pickling



	1008	1009	1010	1011
Average	41.0%	44.9%	42.9%	102.1%
Standard deviation	10.12%	8.94%	3.35%	2.21%
Minimum	35.1%	35.1%	41.0%	99.7%
Maximum	52.7%	52.7%	46.8%	103.9%

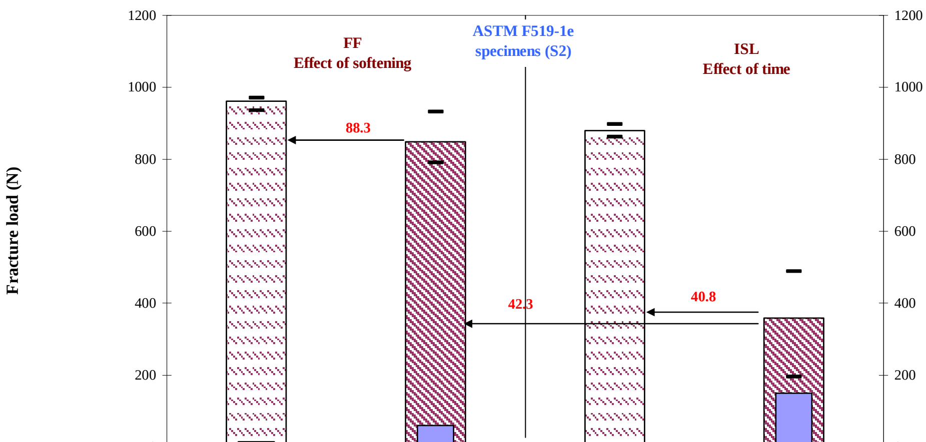
Hardness





25

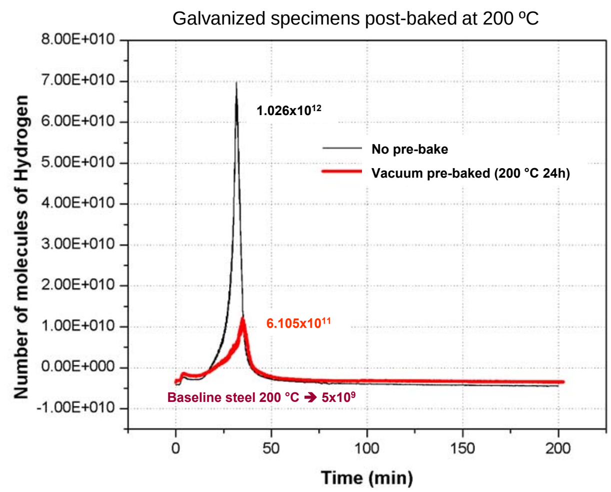
Softening vs. embrittlement



	FF-Baseline	FF-P27	ISL-Baseline	ISL-P27
Average	962	849	880	359
Standard deviation	14.9	60.6	12.2	149.5
Minimum	936	791	863	196
Maximum	971	932	898	489

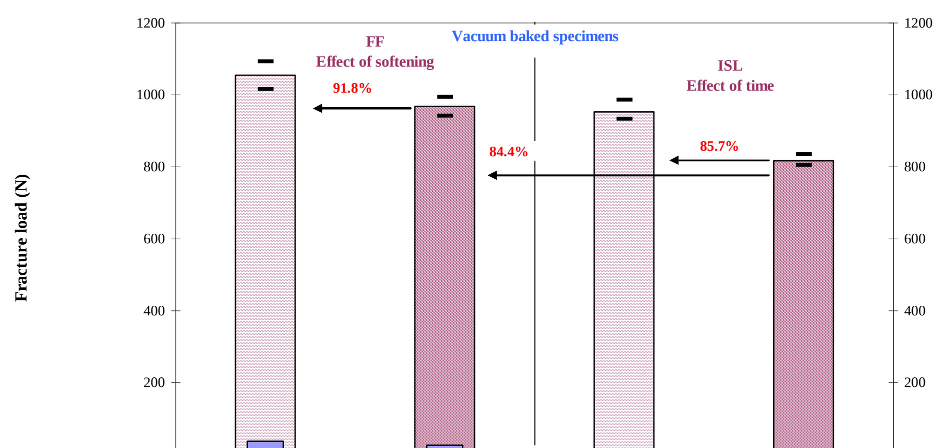
26

Effect of pre-baking in vacuum



27

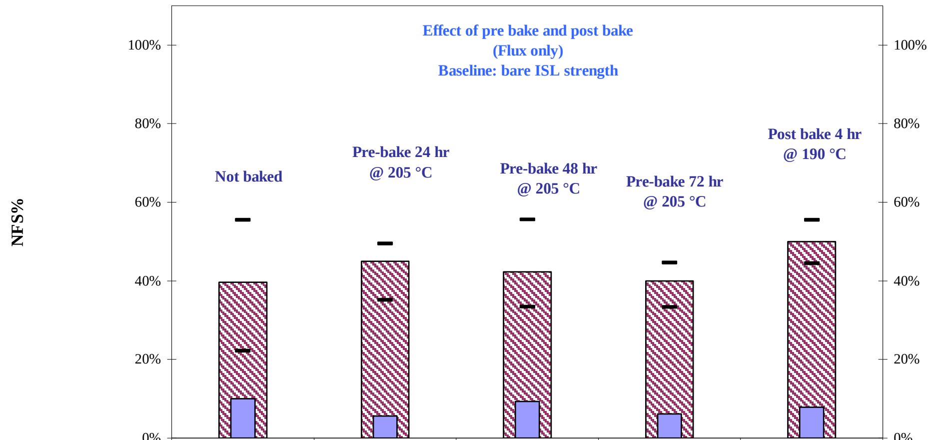
Effect of pre-bake in vacuum (24 h @ 200 °C)



	FF-Bare	FF-P31	ISL-Bare	ISL-P31
Average	1055	968	953	817
Standard deviation	38.0	26.0	15.8	17.0
Minimum	1016	942	934	806
Maximum	1093	994	986	835

28

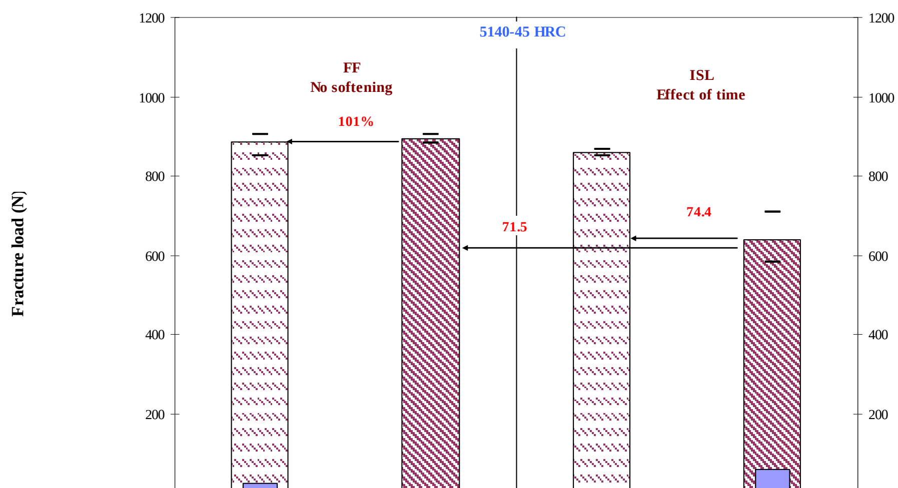
Effect of pre and post-bake in atmosphere (Ar or air)



	24&27	23	25	26	28
Average	39.7%	45.0%	42.3%	40.0%	50.0%
Standard deviation	10.02%	5.65%	9.30%	6.11%	7.82%
Minimum	22.2%	35.1%	33.4%	33.3%	44.5%
Maximum	55.5%	49.5%	55.6%	44.6%	55.5%

29

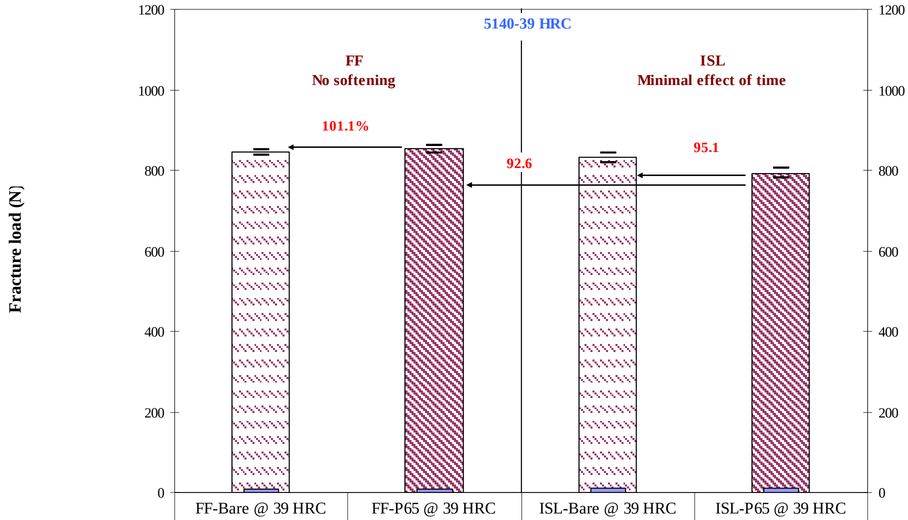
Effect of initial specimen hardness



	FF-Bare @ 44 HRC	FF-P65 @ 44 HRC	ISL-Bare @ 44 HRC	ISL-P65 @ 44 HRC
Average	886	895	860	640
Standard deviation	23.0	9.0	9.0	60.0
Minimum	852	883	850	583
Maximum	905	905	866	709

30

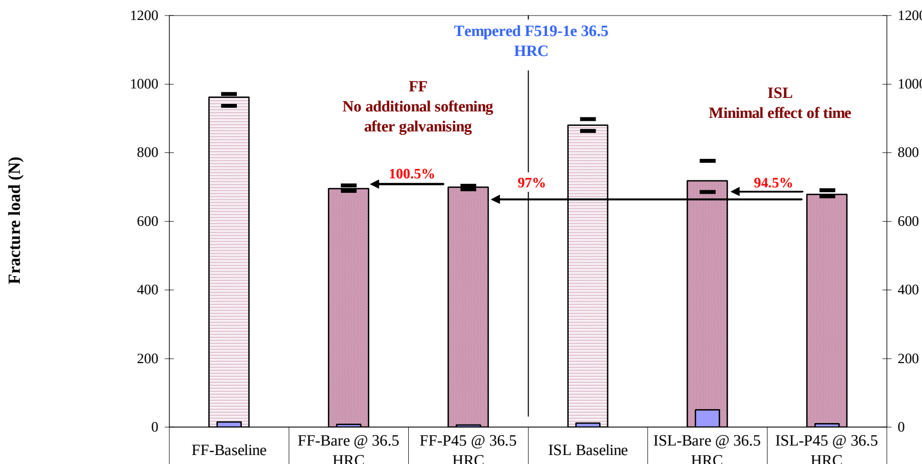
Effect of initial specimen hardness



	FF-Bare @ 39 HRC	FF-P65 @ 39 HRC	ISL-Bare @ 39 HRC	ISL-P65 @ 39 HRC
Average	845	854	832	791
Standard deviation	7.0	8.0	10.0	11.0
Minimum	837	843	820	780
Maximum	851	863	844	806

31

Effect of initial specimen hardness



	FF-Baseline	FF-Bare @ 36.5 HRC	FF-P45 @ 36.5 HRC	ISL Baseline	ISL-Bare @ 36.5 HRC	ISL-P45 @ 36.5 HRC
Average	962	696	700	880	718	679
Standard deviation	14.9	8.0	5.8	12.2	50.3	10.0
Minimum	936	688	693	863	686	673
Maximum	971	704	703	898	776	690

32

Conclusions

- **Hydrogen embrittlement** is the cause of brittle failures in test specimens ➔ **time dependence**
- Embrittlement triggered by **up-quenching** (thermal shock)
  - ➔Hydrogen in **reversible traps**, released by **up-quench** upon immersion. Thick zinc coating **prevents hydrogen escaping**, instead causing it to redistribute (accumulate at **grain boundaries?)**
- Problem goes away if:
  - ➔ Material **hardness is lowered** (i.e. lower susceptibility)
  - ➔ **Hydrogen is removed** (pre-baked in vacuum)

Brahimi, S., et al. *Effect of surface processing variables on hydrogen embrittlement of steel fasteners Part 1: Hot dip galvanizing.* Canadian Metallurgical Quarterly, 2009. **48**(3): p. 293-301.

33

McGill

NSERC CRSNG

CFI Canadian Fasteners Institute

Industrial Fasteners Institute

INFASCO

IBECA

BOEING

NUCOR FASTENER DIVISION

ASTM INTERNATIONAL

Subommittee F16.96

NASF National Association for Surface Finishing

boltcouncil Research Council on Structural Connections rcsc

	ASTM A354 Grade BD	ASTM A490	SAE J429 Grade 8	ISO 898-1 PC 10.9
Size Range	1⁄4 and greater	1⁄2 to 1-1⁄2	1/4 thru 1-1/2	M1,6 to M39 (~1/16 - 1-1/2 in)
Chemistry	Alloy Steel	Alloy Steel	Anything	Anything
Core Hardness	1⁄4 - 2-1/2, =< 2-1⁄2 33-39 HRC, 31-39 HRC	33-38 HRC	33-39 HRC	320-380 HV 32-39 HRC
Min Tensile	1⁄4 - 2-1/2, =< 2-1⁄2 150 ksi , 140 ksi	150 ksi	150 ksi	1040 MPa (~150.9 ksi)
Max Tensile		173 ksi		
Min Yield	1⁄4 - 2-1/2, =< 2-1⁄2 130 ksi, 115 ksi	130 ksi	130 ksi	940 MPa (~136.4 ksi)
Proof stress	1⁄4 - 2-1/2, =< 2-1⁄2 120 ksi, 105 ksi	120 ksi	120 ksi	830 MPa (~120.5 ksi)
Min. Elongation	14%	14%	12 %	9%
Min Red area	40%	40%	35 %	48%
Charpy Impact			27 J at −20 °C	
X sect. hard. range				
Surface Hardness			58.6 30N (~38.5 HRC)	390 HV (~39.8 HRC)
Carb		F2328, 12.2		Section 9.11
Decarb		F2328, 12.2	ASTM F2328, Class 2	Yes
90% martensite			Yes	Yes
Min Temp temp	800 °F	800 °F (427°C)	425 °C (800 °F)	425 °C
Ref. Temper test			Y	Y
Surface Discont.		ASTM F788 + Mag particle + 100 %	ASTM F788	ISO 6157-1 (or 6157-3)

Comparison of 3" Diameter E2 Rods

		Manufacturing	Mechanical Testing			Hardness Range (+/-)	CVN @ 4C (J)
			Elongation (%)	Reduction of Area (%)	Fu (ksi)		
	ASTM A354BD	-	14% min	40 min	140 min[1]	31-39[4]	-
Fabrication Year	2008	-	12.5%-15%	40-50	159-171	26-39	18-24 @ Surface
	2010	Vacuum Degassing	14%-17%	40-53	153-158	27-38	49-53 @ Surface
	2013 (4340)	Vacuum Degassing	19%-21%	55-59	160-163	30-37	>68 @ Center 64–67 @ Surface

ASTM A354

NOTE 2 —Quenched and tempered alloy steel bolts for structural steel joints up through 1-1⁄2 in. in diameter are covered in Specification A490. Alloy steel bolts, studs, and other externally threaded fasteners (that is, heavy hex-structural bolts over 1-1⁄2 in., hex bolts, anchor bolts, and countersunk bolts) exhibiting similar mechanical properties to bolts conforming to Specification A490 shall be covered by Grade BD of this specification.

When bolts of Grade BD of this specification are considered for pretentioned applications in excess of 50 % of the bolt tensile strength, the additional requirements of head size, maximum tensile strength, nut size and strength, washer hardness, tests, and inspections contained in Specification A490 should be carefully considered.

NOTE 4 —Research conducted on bolts of similar material and manufacture indicates that hydrogen-stress cracking or stress cracking corrosion may occur on hot-dip galvanized Grade BD bolts.

37