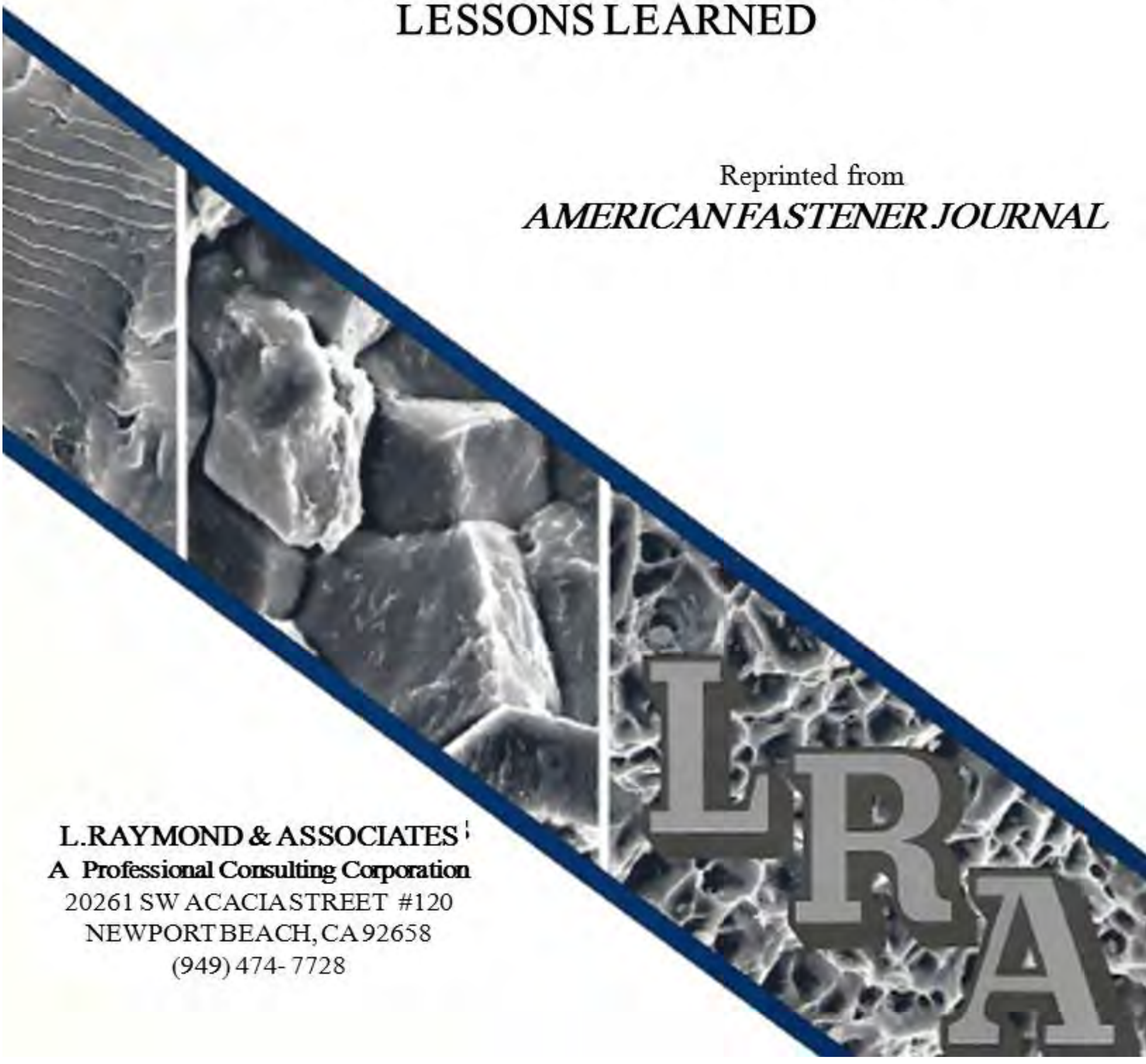
FAST FACTS

FAILURE ANALYSIS of FASTENERS: LESSONS LEARNED



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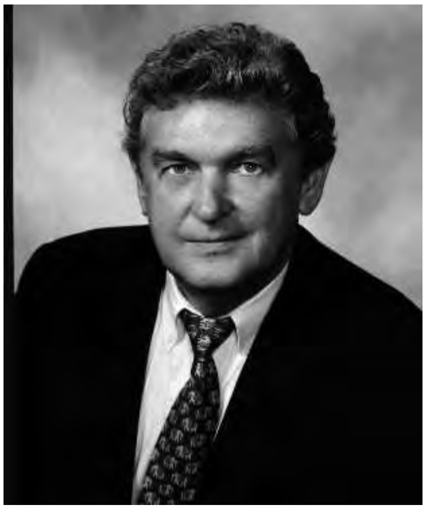

Louis Raymond, Ph.D.

Ph.D., P.E. (CA), FASTM, FIAE

Dr. Louis Raymond has spent 40 years on research of hydrogen embrittlement failure mechanisms and accelerated fracture mechanics testing protocols in support of government and industry.

* **Society Affiliations:** During this time, he served as Chairman of **ASTM F07 on Aerospace & Aircraft** for about six years, and Chairman of ASTM F07.04 on **Hydrogen Embrittlement** for over 40 years. Dr. Raymond is very active working with various other coating and fastener standard organizations, including **ISO**, Federal, Military, **NAS, SAE /AMS** and **IFI**.

* **Advisory Board:** Dr. Raymond served as a Special Consultant to the **Scientific Advisory Board** on the **F-111** Failure Analysis Program. He first became involved with hydrogen embrit tlement for the Aerospace Corporation working on the failure of stainless steel bolts for the **Titan** Launch Vehicle Systems. Since then, he has served on the Nat**ional Academy of Sciences** and the National Materials Advisory Board, helping to develop accelerated test methods for Hydrogen Embrittlement, Fatigue, and Fracture Toughness within the frame work of Fracture Mechanics.

***NASA:** Dr. Raymond continues his consulting work in failure analysis, working on such manufactures products as gears for Apache, Blackhawk helicopters, hydraulic actuators and pistons for aircraft, and **MP35N** locknuts for NASA space launch vehicle systems. All of these analyses are under taken using accelerated **RSL™** testing techniques. Dr. Raymond has also worked on behalf of NASA in failure analysis of the space shuttle Challenger, and is the recipient of the **NASA Inventor's Award for Space Processing (1978).**

***Non-aerospace Applications:** At LRA, Dr. Raymond is very active in applying his testing methodology to non-aerospace and aircraft applications, such as with propeller blades for supertankers, windmills for the **DOE**, trains for the **DOT**, offshore platforms for the **DOI**, icebreakers for the Coast Guard. More recently, under another SBIR contract, Dr. Raymond had been applying these accelerated test methods in support of the Navy's Fracture Toughness Review Program (**FTRP**) and Material Selection

***Teaching:** Concurrent to his consulting and advisory work, Dr. Raymond served as an Adjunct Professor at California State University Long Beach. In directing graduate research programs in these areas, he developed a very active industry-university student co-op graduate study program. While at the university, he received research funding from the David Taylor Naval Ship Research Development Center for developing accelerated test methods for hydrogen embrittlement of welded structures.

* **Fracture Mechanics:** Over the past 20 years, Dr. Raymond has been president and director of technical operations for his own company, **LRA** Consulting and R&D Laboratories. Here he continues work on the fields of fracture mechanics. He received a research grant form the Army (**AMMRC**) to verify the use of an imposed potential to simulate galvanic coupling. And, in a cooperative study with Aerojet and Standard Pressed Steel, he received a grant from **NASA** for evaluating the use of Multiphase **MP 159** alloy for fastener applications on the ASRM. Dr. Raymond served as a consultant for the **Aerospace Corporation** and worked on a study with Boeing Space Division Seattle, on Custom 455 for use as a torsion beam in the **Interim Upper Stage of the Shuttle**.

* **Patents:** Dr. Raymond has developed and patented a computer-controlled digital displacement hydrogen embrittlement accelerated testing system under contract from a Navy Small Business Innovative Research Grant (**SBIR**). This research led to the development and commercialization of the Rising Step Load™ (**RSL™).** RSL™ testing system is currently manufactured by Fracture Diagnostics International (**FDI**). Process (**MSP**) being applied to all new structurally critical marine designs.

***Fastener Technology Center:** Dr. Raymond is active in failure and life analysis of fasteners taking into account the effects of fatigue and exposure to the environment both during and after service. He has established LRA Laboratories as a "**Technology Center for the Fastener System and Design Analysis**". In 2006 Dr. Raymond received the Industrial Fastener Institute (**IFI**) **Science Award**. Recently, he has initiated a series of articles on The Structural Integrity of **Aerospace Fasteners** in www.fastenerjournal.com.

***Publications:** Dr. Raymond actively lectures and writes articles on the effects of processing, including the effects of heat treatment and coatings on the service performance of fasteners. The studies incorporate the most recent and advanced concepts of fatigue and fracture mechanics, as well as the newest technological advances, for use in the aerospace and aircraft industries. His articles commonly appear in **The American Fastener Journal**, **ASTM Standardization News**, and **Corrosion** (a publication of **NACE**.)

***Consulting:** Dr. Raymond works as a consultant with various fastener manufacturers, distributors and users. He has trained fastener-testing labs for certification by major corporations. He organizes and teaches a variety of Short Courses, such as on "Hydrogen Embrittlement: Its Prevention and Control", which discussing new, existing and emerging test methods to Identify and evaluate plating and coatings as potential sources of hydrogen embrittlement.

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This compilation summarizes 50 years of research on Hydrogen Embrittlement that includes but is not limited to an in-depth analysis of fastener failures, fastener engineering, and fastener design applications. The Fast Facts provide data, images, and graphs to ultimately further the infrastructure of fastener analysis. The mission for the prior is to educate and inform readers on the technical aspects of fasteners and fastening applications, reduced to the basic lessons learned from each of the technical issues of extensive failure analyses reports. The Fast Facts Journals were published bi-monthly in the American Fastener Journal. Suggestions and or comments in regards of this compilation shall be addressed to LRA Laboratories.

For more information on the entire spectrum of RSL-ISL test machines, standards and doing basic research on hydrogen embrittlement, start with Dr. Raymond's bio-link (www.LouRaymond.com) under his photo and follow through with the additional inks at the bottom of that page, on through,

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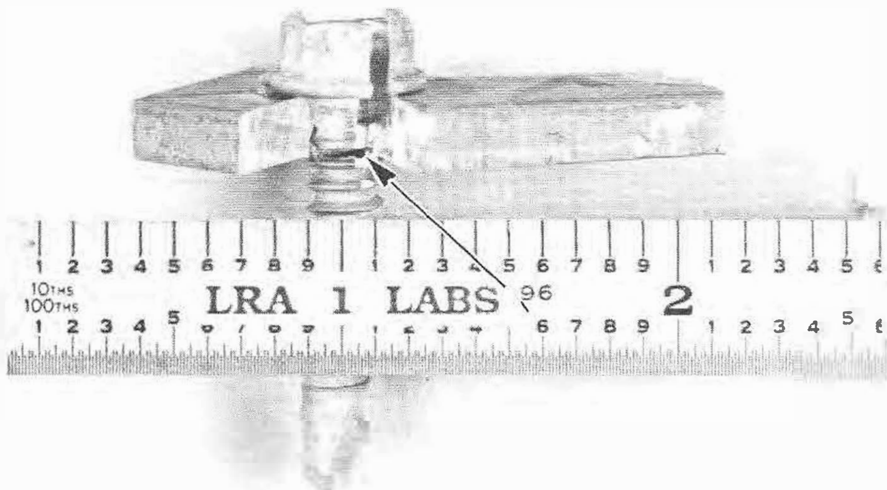
Fast Fact . . . No. 1

Fastener Failure Analysis

SEM Photographic "Fingerprints" Identify "Cause" of Failure in Fasteners!

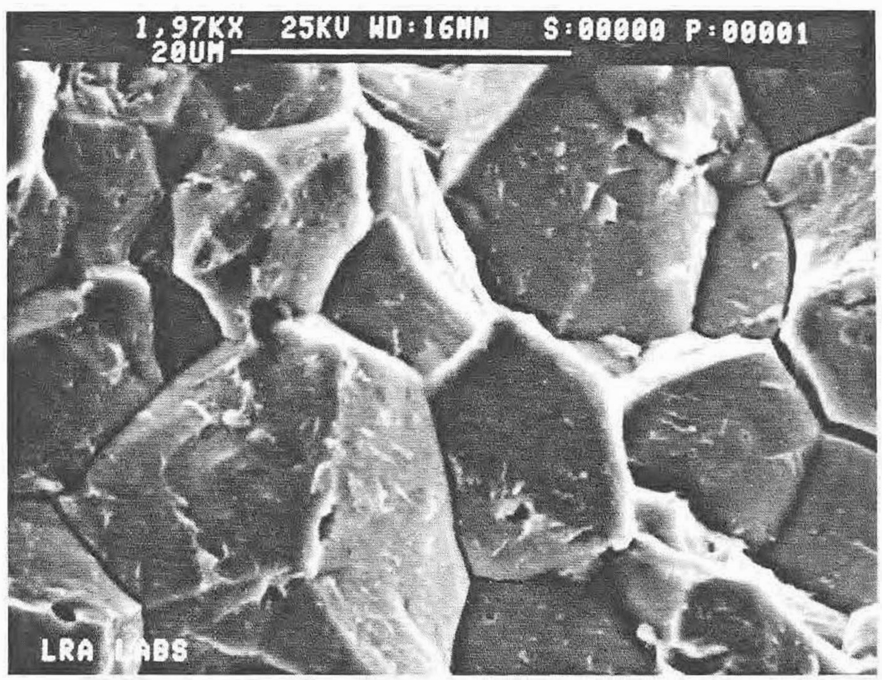
LRA Failure Case #90271



Arrowpoints to stress crack in Type 410 stainless steel Cd-plated (1/4-14) TEK* in 3/16" steel (84 HRB) plate exposed to water.

SEM

Scanning Electron Microscope Photographic "Fingerprint" of intergranular cracking on fracture surface identifies "cause" as hydrogen embrittlement.



* TEK fasteners are commonly used in building construction (window frames, curtain walls).

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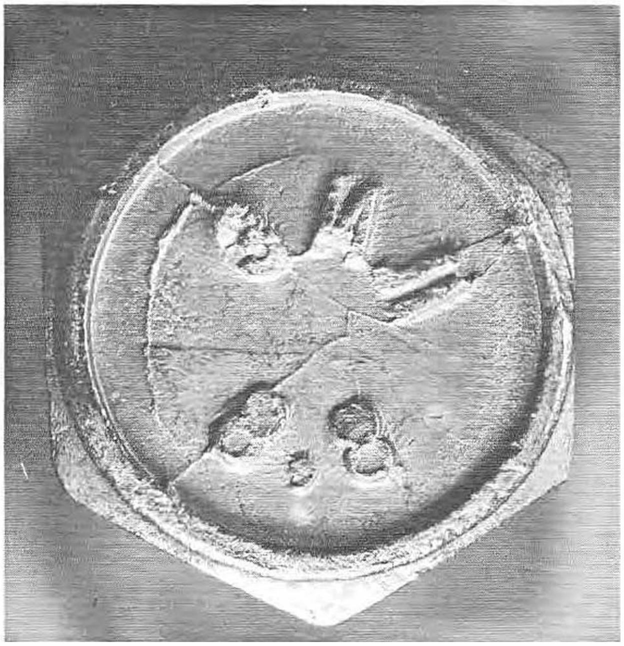
Fast Fact . . . No. 2

Russian Made Fastener

SEM X-ray "Fingerprints" Identify the Coating on Fastener



Head/Grade Marking

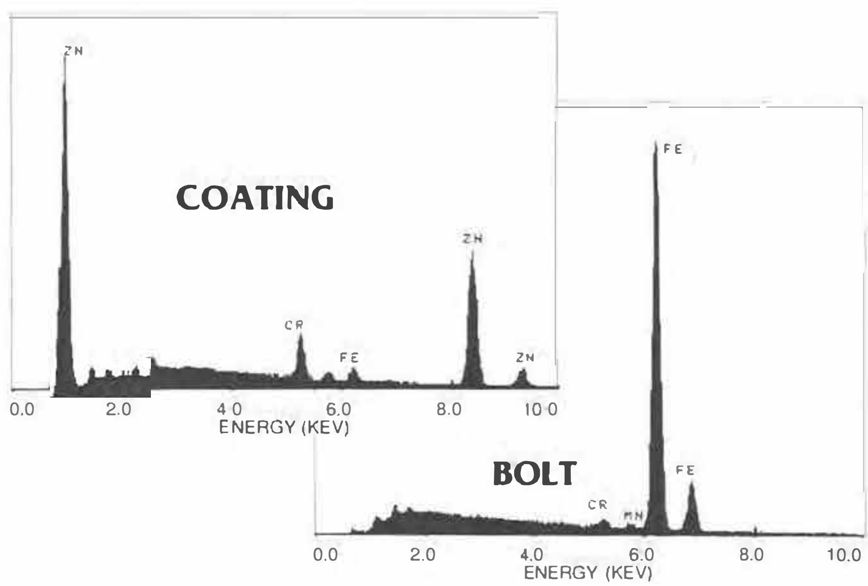
Three Russian letters (top) identify the manufacturer as the Zil plant in Moscow. The two numbers (bottom) identify the metric grade 8.8, equivalent to an SAE grade 5 bolt.

The measured hardness of 258HB for the 12mm bolt is consistent with a minimum tensile strength of 120ksi (240HB) and a maximum hardness of 302HB.

SEM

The SEM X-ray spectrum of the bolt is consistent with a medium-carbon steel heat treated to produce tempered martensite, which was verified by microstructural analysis.

The SEM X-ray spectrum of the coating is consistent with a Type II zinc with a chromate conversion coating (ASTM B633).



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Fast Fact . . . No. 3

Fastener Failure Analysis

X-Ray Microprobe With Scanning Electron Microscope Identifies the Cause of Stress Corrosion Failure in Bolt

LRA Failure Case #72641

Failed Bolt

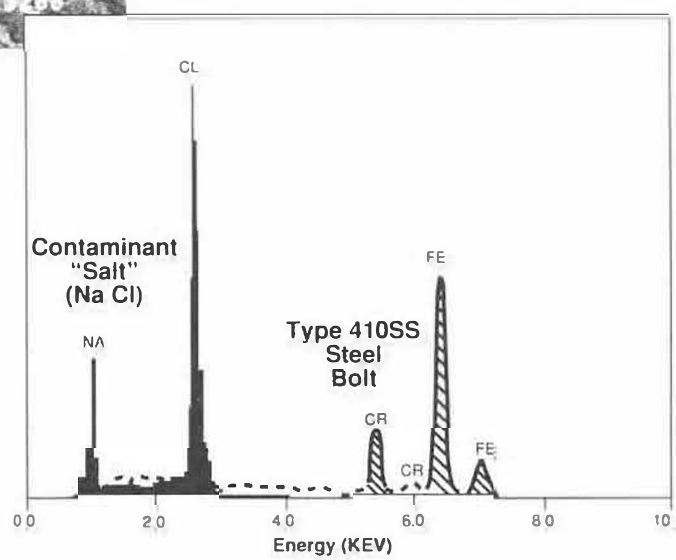
A failed bolt* was found with contaminants on the fracture face. The x-ray spectrum identifies the type of contaminant, and double exposure photographic technique identifies the location relative to the origin by selective fluorescence.

SEM

Double exposure photographic technique identifies location of contaminant. Selective fluorescence of chlorine appears as white spots on fracture face.

X-Ray Spectrum

Identifies source of contamination as salt water.



* Failed bolt is Type 410 stainless steel Cd-plated (1/4-14) TEK self-drilling fastener.

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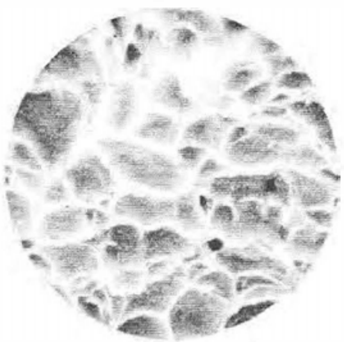
Fast Fact... No. 4

Fastener Failure Analysis

SEM Photographic Finger Prints Identify the "Cause"!!

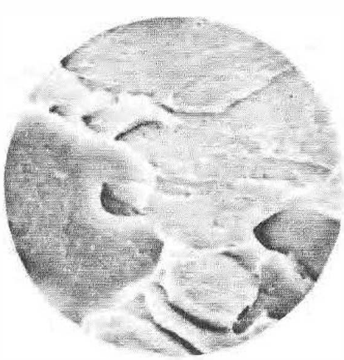
Failure Modes Typically found in SAE Grade 5 Bolts

(Medium-carbon alloy steel, heat treated to a maximum of about 300 BHN)



Dimpled Ruture Typical of a Ductile Overload Fracture.

Intergranular Cracking Typical of Subcritical or Stable Crack Growth by Hydrogen Embrittlement.



Quasi-cleavage Typical of Brittle Failure at a Fracture Stress below the yield strength.

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Fast Fact No. 5

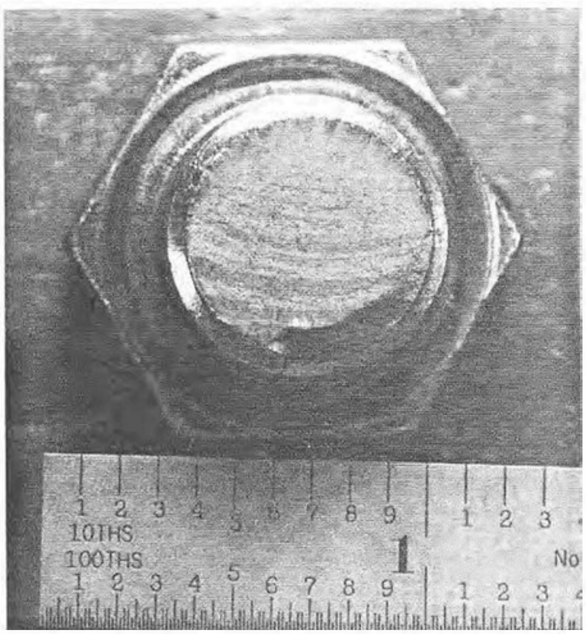
Fastener Failure Analysis

SEM ““Fingerprint” Identifies Fatigue Failure in a Grade 8 Bolt

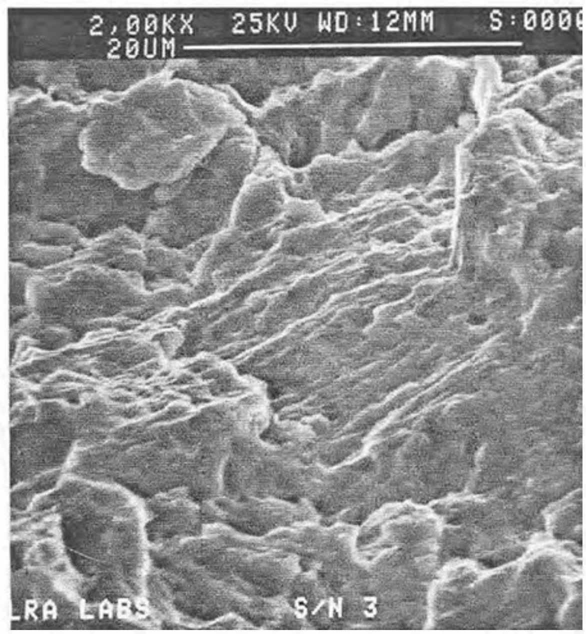
Problem: A 3/4"-10 UNC Grade 8 bolt from a 400KW generator turntable failed in service.

Question: Was failure due to overload (dimple rupture) or fatigue (striations)?

Results: SEM photographic “fingerprint” shows striations near fracture origin, identifying fatigue as the cause of failure.



Fractured Surface of Bolt
(Beach Marks)



SEM: Fatigue Striations
Near Origin (2KX)

Conclusion: Vibration caused bolt to work loose. Bolt absorbs entire cyclic load amplitude.

Recommendation: Employ a nut locking system on the bolt to inhibit the loosening process. Replacement with exotic and costly bolts of higher fatigue strength is not required.

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Fast Fact No. 6

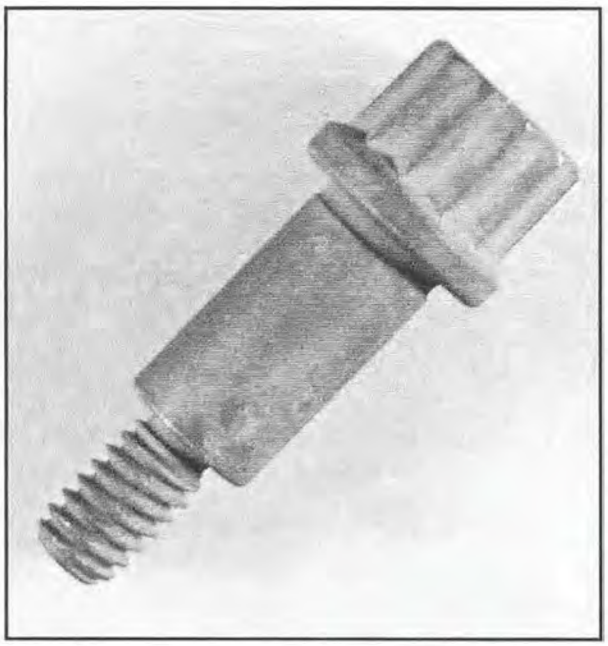
Fastener Failure Analysis

MIL STD 1312-5A inadequate to identify potential of Hydrogen Embriitlement Failure

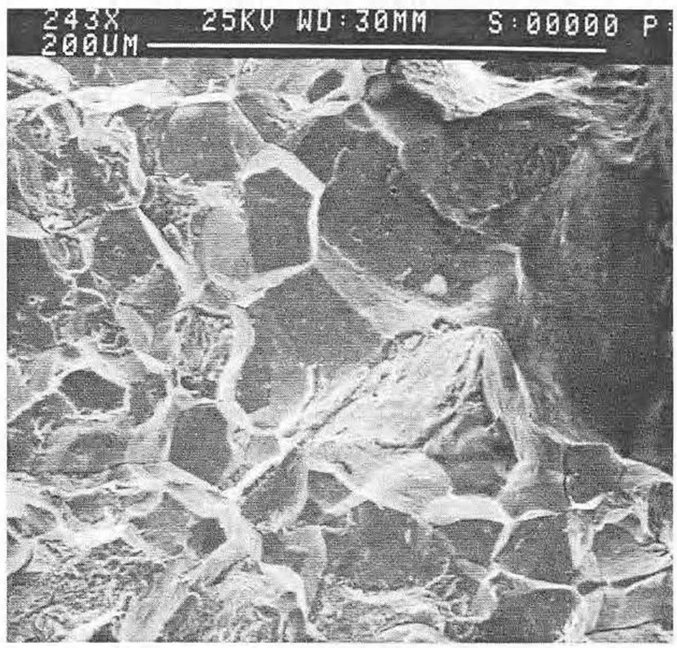
Problem: A cadmium plated H-11 engine bolt (46HRC) experienced a time delay failure in the thread relief area shortly after installation although the lot had been qualified per MIL STD 1312-5A, the Stress Durability test.

Question: Was the failure due to Hydrogen Embrittlement?

Results: SEM photographic "fingerprint" shows intergranular cracking near the fracture origin, suggesting Hydrogen Embrittlement as the cause of failure. These results were verified by a slow strain rate tensile test.



H-11 Engine Bolt



SEM: Intergranular Fracture Near Origin (240X)

Conclusion: The time delay failure was by hydrogen embrittlement. Local bending on installation produced a higher stress than 75% of the tensile strength (220ksi) applied during the Stress Durability test.

Recommendation: Use a wedge under the head of the bolt during the Stress Durability test to insure hydrogen embrittlement relief due to processing.

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Fast Fact No. 7

Fastener Failure Analysis

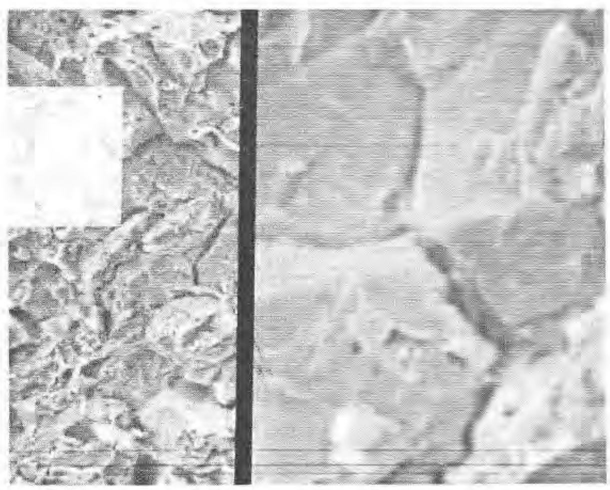
IFI-113 did not identify potential of Hydrogen Embrittlement Failure in Pan-Head Fasteners

Problem: Several zinc-plated, 10-24, self-tapping, pan-head steel fasteners (39/53 HRC, core/case hardness) experienced a time delay failure in the threads shortly after installation although the lot had been qualified in accordance with IFI-113, Section 3.9, the Hydrogen Embrittlement Test.

Question: Was the failure due to Hydrogen Embrittlement?

Results: SEM photographic "fingerprint" shows intergranular cracking originating at the thread root and progressing from the case into the core.

Pan-Head Fastener



Intergranular Fracture throughout Case (53 HRC) and Core (39 HRC) 500X/2,000X

Conclusion: Hydrogen embrittlement was the cause of failure, which only occurred in the pan-head fasteners when applied stress exceeded the threshold stress due to additional bending stresses during installation. The hydrogen embrittlement test described in IFI-113 does not take these stresses into account.

Recommendation: To insure against hydrogen embrittlement cracking due to processing, use a wedge under the head of the fastener during the IFI-113, Section 3.9, Hydrogen Embrittlement Test.

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Fast Fact No. 8

Fastener Failure Analysis

Requirement for baking of zinc-plated Grade 8 bolts

Problem: According to ASTM B633 "Specification for Electrodeposited Coatings of Zinc on Iron and Steel," a hydrogen relief treatment (i.e., baking) is only required if the strength level of the steel is higher than 175 ksi (38 HRC), implying that Grade 8 bolts (32±1 HRC) are immune to hydrogen embrittlement (HEM).

Question: Are zinc-plated Grade 8 bolts (32±1 HRC) really immune to HEM?

Results: Testing of Grade 8 bolts revealed potential for HEM.

Unplated Fastener

ASTM E-8 tensile test revealed 100% dimple rupture

Zinc-Plated Fastener Baked 3 hours at 375° F

LRA slow strain rate test revealed intergranular fracture (bright area)

Conclusion: There is a potential risk for HEM failures in zinc-plated Grade 8 fasteners at 32±1 HRC, contrary to current opinions. In some cases, even a 3 hour bake at 375±25° F is not adequate.

Recommendation: Re-examine: (1) the effectiveness of hydrogen bake-out treatments, (2) the adequacy of plating bath controls, and (3) the adequacy of test methods for HEM in Grade 8 bolts.

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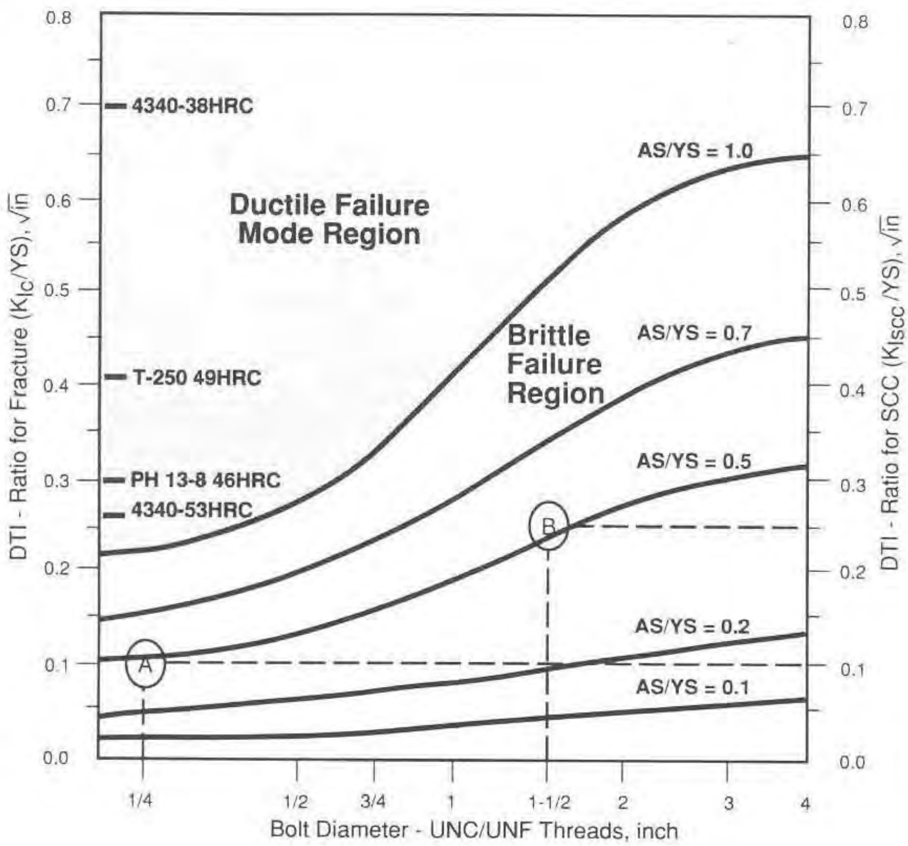
Fast Fact **No. 9**

Selection of Maximum Installation Torque

Based on K_{Iscc} test results of plated fasteners

Approach: Measure KIscc of the steel in the condition used in the fastener… Test in the environment at a potential duplicating the open circuit potential of the coating… Calculate K_{Iscc}/YS… Determine maximum AS/YS (applied stress/yield stress) from chart below for given bolt diameter… Calculate maximum [conservative (μ = 0.1)] installation torque from:

$$T_{i_{max}} \text{ (in-lbs)} \approx 110 \text{ (AS/YS)} \cdot D_{pitch}^3 \text{ (in}^3\text{)} \cdot \text{YS (ksi)}$$



Examples:

(A) Assume K_{Iscc} = 20 ksi$\sqrt{in}$ (zinc-coated Type 410SS), YS = 200 ksi, diameter = 0.25-in. Calculate K_{Iscc}/YS = 0.1 and determine AS/YS = 0.5. Therefore, $T_i \approx$ 110 in-lbs, max.

(B) Assume K_{Iscc}/YS = 70 ksi$\sqrt{in}$ (zinc-coated Type MP159), YS = 280 ksi, diameter = 1.5-in. Calculate K_{Iscc}/YS = 0.25 and determine AS/YS = 0.5. Therefore, $T_i \approx$ 3,500 ft-lbs, max.

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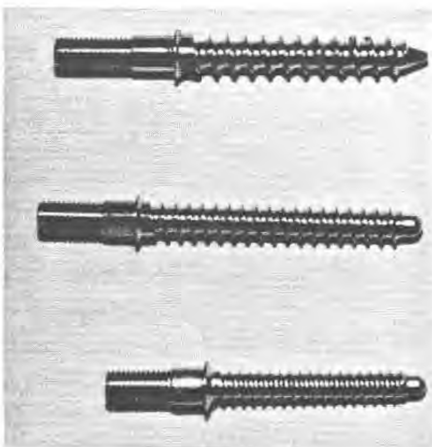
Fast Fact No. 10

Fastener Design

Altering Thread Profile to Minimize Stress Concentration

Design Goal: Reduce the potential for cortical bone screw breakage in a biomechanical implant device subjected to tension, bending, and torsion fatigue stresses.

Approach: Minimize stress concentration at root of threads of cortical bone screws by providing a full radius thread profile. The reduction in stress concentration when compared to a standard Whitworth radiused thread profile is substantial.



9 Threads/inch (0.111" pitch)
Major Diameter: D = 0.253"
Root Diameter; d = 0.163"
Root Radius: r = 0.052"

12 Threads/inch (0.083" pitch)
Major Diameter: D = 0.226"
Root Diameter; d = 0.162"
Root Radius: r = 0.038"

16 Threads/inch (0.063" pitch)
Major Diameter: D = 0.195"
Root Diameter; d = 0.148"
Root Radius: r = 0.029"

Table I: Full Radius Thread Profile

Threads Per Inch	Major Dia (in)	Root Dia (in)	Root Radius (in)	Stress Concentration (K_t)* Tension	Bending	Torsion
9	0.253	0.163	0.052	1.43	1.27	1.20
12	0.226	0.162	0.038	1.56	1.37	1.26
16	0.195	0.148	0.029	1.64	1.42	1.30

Table II: Whitworth Thread Profile

Threads Per Inch	Major Dia (in)	Root Dia (in)	Root Radius (in)	Stress Concentration (K_t)* Tension	Bending	Torsion
9	0.305	0.163	0.015	2.19	1.82	1.53
12	0.269	0.162	0.011	2.50	2.20	1.65
16	0.228	0.148	0.009	2.60	2.25	1.70

* Peterson, R.E., "Stress Concentration Design Factors," John Wiley & Sons, Inc., New York, 1953.

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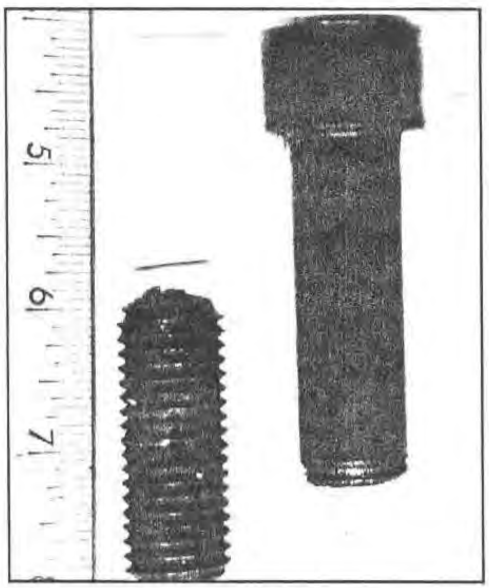
Fast Fact No. 11

Fastener Failure Analysis

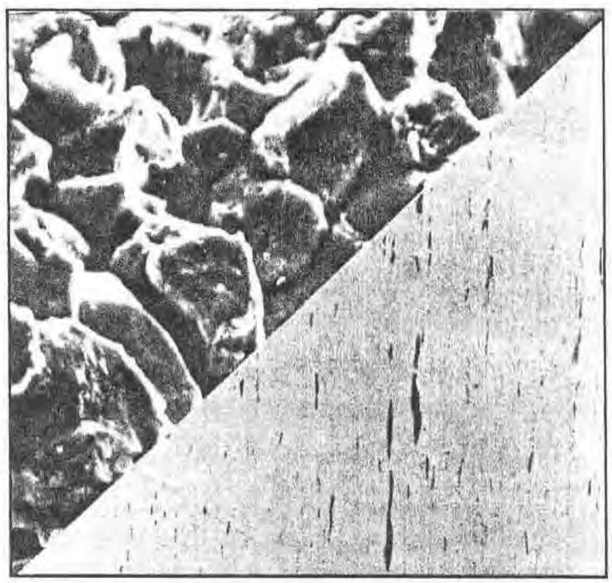
"Free Machining" Grade Type 410

Problem: Numerous failure occurred in 3/4 inch bolts reportedly conforming to Type 410 stainless steel per ASTM A193 Grade B6 for "Stainless Steel Bolting Materials for High-Temperature Service". The bolts were used to attach a housing seal to a pump case.

Results: The tensile properties exceeded the minimum requirements per specification. The hardness was closer to 40HRC instead of 20HRC. Apparently, the manufacturer did not adhere to the minimum specified tempering temperature of 1100°F. Chemical analysis showed the material to be a "free machining" Grade, Type 416. which primarily differs in chemical composition from Type 410 by the higher sulfur and manganese content.



Failed Tensile Fastener.



Intergranular fracture surface and Mn-sulfide stringers.

Conclusion:

The bolt failures were due to hydrogen embrittlement from the combination of too high a hardness and the high sulfur content of the "free machining" grade of Type 410 stainless steel, contributing to the susceptibility of the Type 416 stainless steel.

Recommendation:

Avoid using "free machining" grades of steel for tensile fasteners that approach or exceed a hardness of 38HRC.

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Fast Fact No. 12

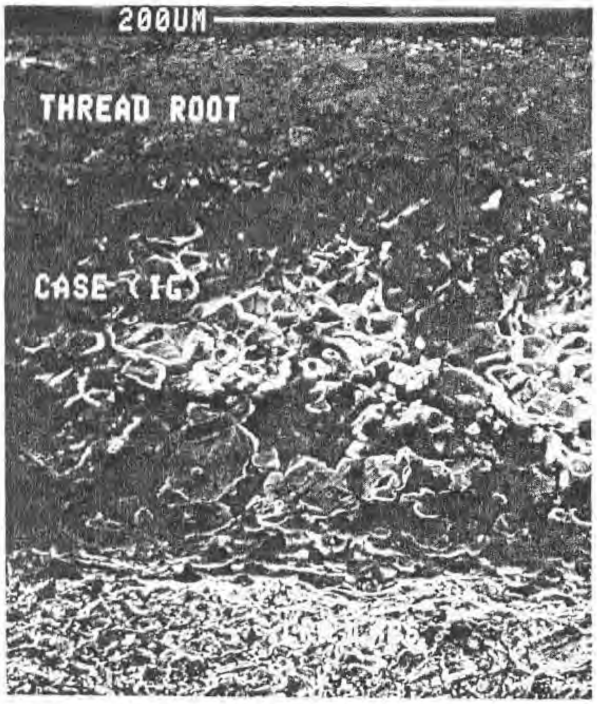
Fastener Failure Analysis

Intergranular Fracture Surface on Case Hardened Surface

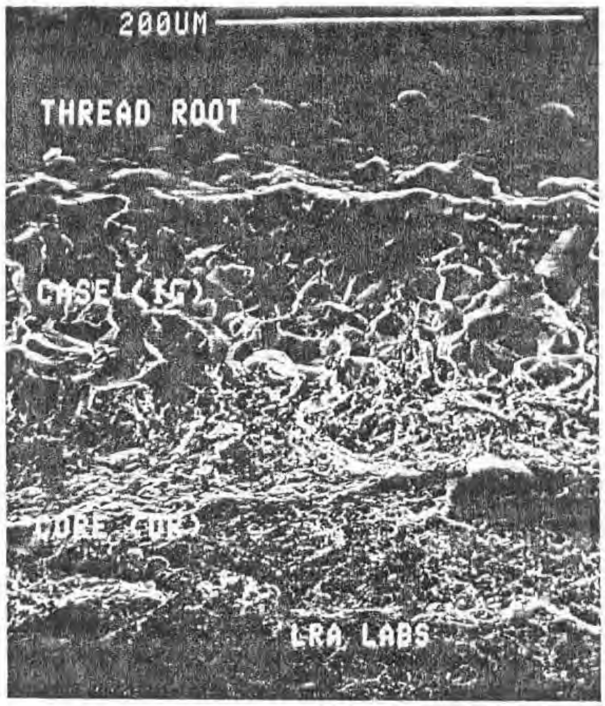
Problem: Failure of a tensile fastener in service was immediately attributed to hydrogen embrittlement because of the presence of an intergranular fracture surface on the outer diameter.

Question: Does an intergranular fracture surface in a case hardened steel fastener always mean that the failure was caused by hydrogen embrittlement?

Approach: A fastener from the same lot, with no thread root cracks was ruptured under impact loading in the lab and then characterized in the SEM.



Failed in-service tensile fastener. Hydrogen embrittlement suspect.



Tested in lab. Fracture caused by impact overload.

Results:

The fracture surface was identical with the exception of some contamination products on the surface of the failed fastener.

Conclusion:

All hydrogen embrittlemnt failures in high-strength steels are intergranular, but not all intergranular fractures in high-strength steels are caused by hydrogen embrittlement.

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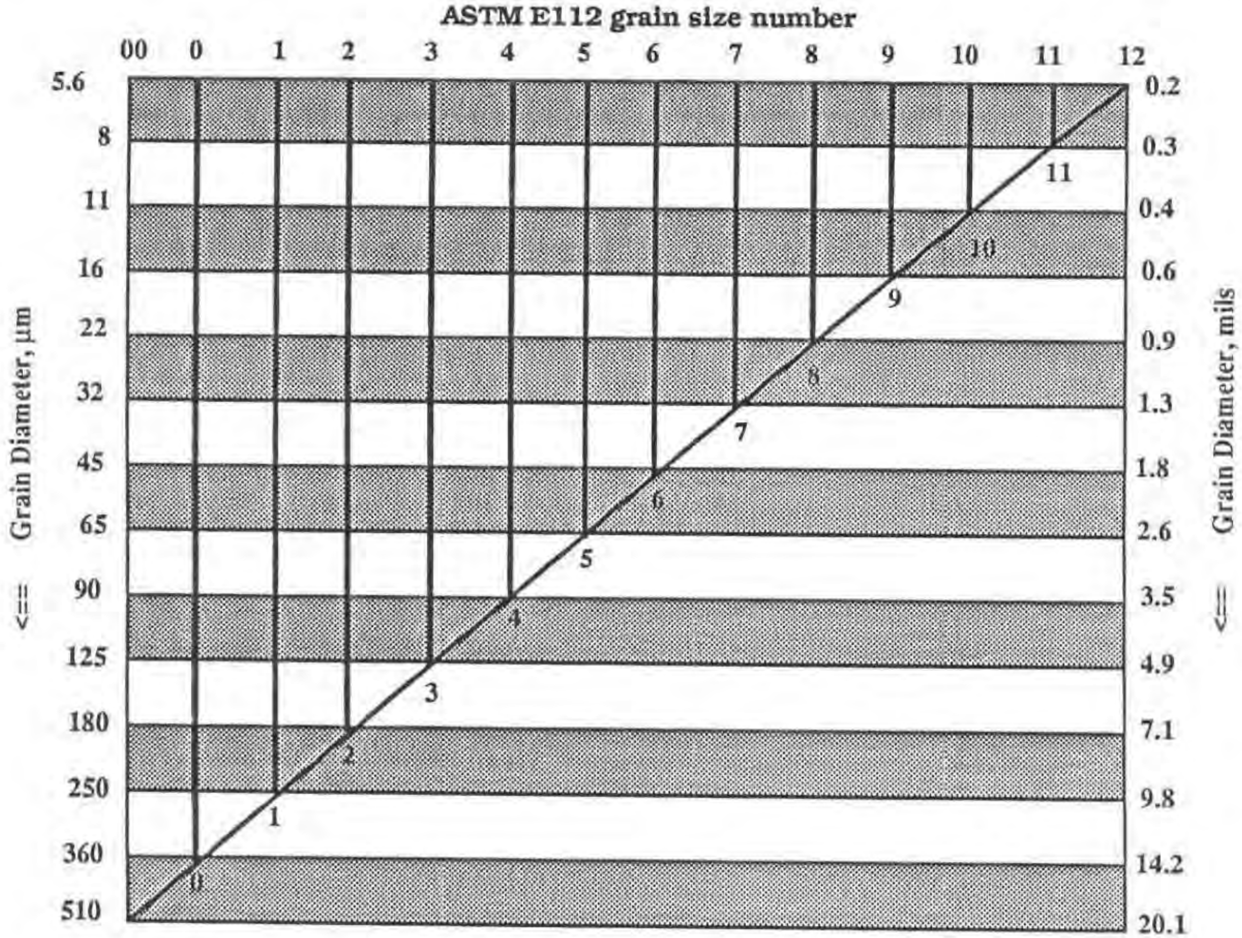
FAST FACT . . .

No. 13

Nomogram for Determining ASTM Grain Size



NOTE: 1µm = 40 micro-inch, and 1 mil = 0.001 inch = 25 µm

This chart was developed by Dr. Raymond at LRA Failure Analysis and Corrosion Research Laboratories for estimating the ASTM grain size number directly from photomicrographs taken with the light microscope or Scanning Electron Microscope (SEM). The approach is consistent with ASTM E112-80, "Standard Methods for Estimating the Average Grain Size of Metals," and Supplement E930-83, "ALA Grain Size". First, determine the grain diameter in micrometers from the SEM photomicrographs, or use a ruler to measure it in millimeters or inches. If the latter method is used, the measurement must be converted to micrometers or mils by dividing by the appropriate magnification. The ASTM grain size number can then be read directly from the chart.

Example 1: A 20µm diameter grain from an SEM photomicrograph would have an ASTM grain size number slightly larger than 8.

Example 2: A 2-inch grain at 500X would actually be 4 mils in diameter and have an ASTM grain size number between 3 and 4.

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Fast Fact No. 14

Fastener Failure Analysis

Intergranular Fracture of a Ductile Low-Carbon Steel Fastener

Problem: A tensile test of a fastener at -65°F in a low-carbon steel per ASTM A545 (AISI 1006) exhibited a lot-to-lot variation in fracture strength and fractographic features. At room temperature, the tensile strength, ductility and failure modes were similar.

Question: Why are the fracture surface features vastly different at -65°F even though they are similar at room temperature and meet the mechanical and chemical composition requirements of ASTM A545.

Anticipated Quasi-cleavage fracture surface for AISI 1006 steel at -65°F.

Observed Intergranular fracture surface for AISI 1006 steel at -65°F.

Results: Microstructural differences were metallographically observed corresponding with differences observed with the fracture surfaces. Precipitation of a second phase was noted in the grain boundaries.

Conclusion: Test materials, even plain, low-carbon steels, at the Lowest Anticipated Service Temperature (LAST) at maximum service loading rates. The precipitation morphology and the resulting fracture modes @ -65°F can be controlled with heat treatment.

Acknowledgement: This Fast Fact was submitted by Sadiq Ghias of Cherry-Textron Metallurgical Lab.

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Fast Fact No. 15

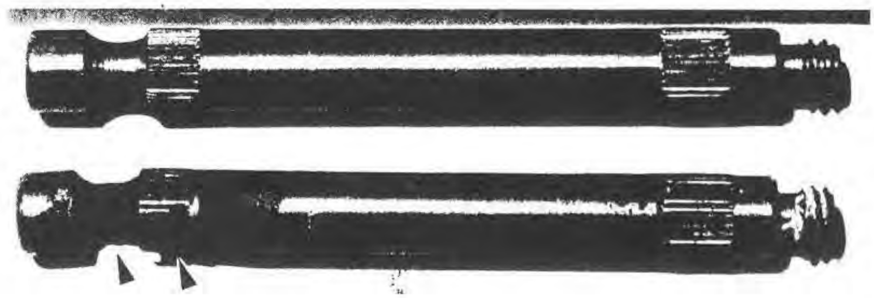
Fastener Failure Analysis

Improper Choice of Stainless Steel for Pins

Problem: Excessive corrosion of stainless steel pins causes failure of component in as little as three months operating time.

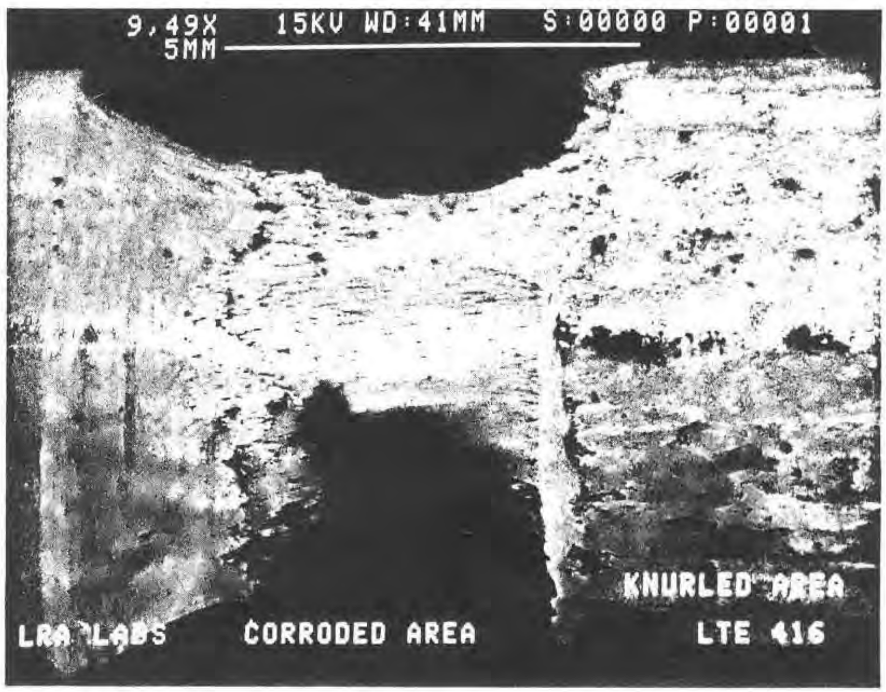
Question: Why did severe corrosion of the stainless steel pins occur?

A new and a three month old stainless steel pin are shown. The bottom sample removed from service exhibited extensive localized corrosion attack, marked with arrows.



SEM
Scanning electron microscope shows the "woody, stringer" like appearance of severe localized corrosion attack.

Energy dispersive x-ray analysis shows the steel to be Type 303 free machining grade stainless steel. The high sulfur content that aids in machinability also increases the Type 303 stainless steels susceptibility to localized corrosion attack when exposed to aqueous environments.



Recommendations: Use a more corrosion resistant grade of stainless steel that is free of harmful manganese sulfide stringers.

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Fast Fact No. 16

Fastener Failure Analysis

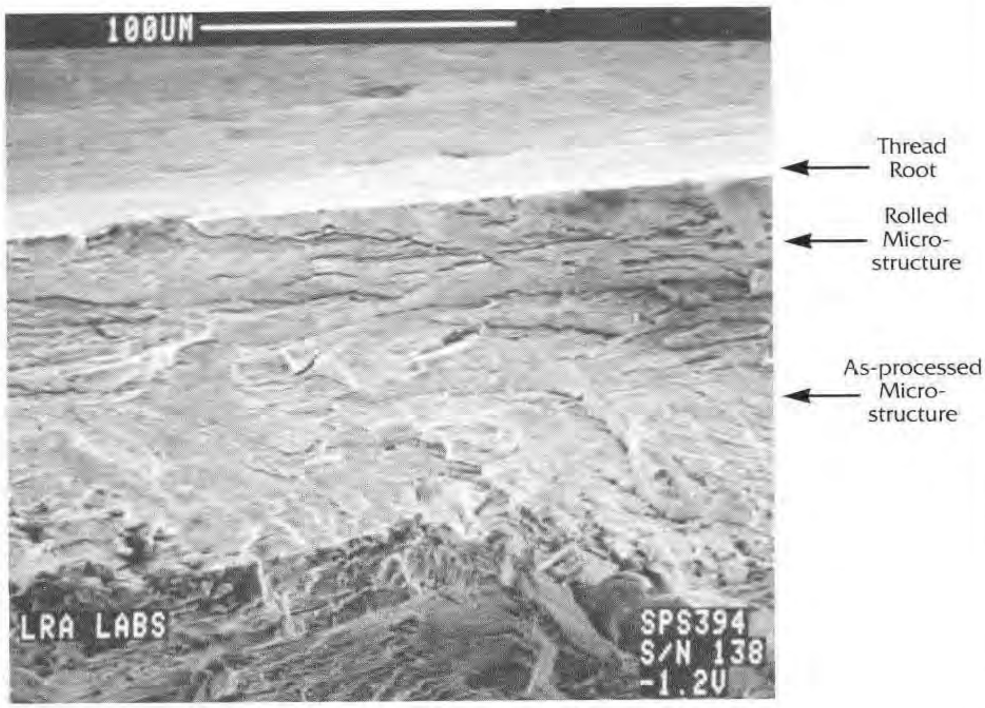
Rolled Threads and Hydrogen Embrittlement

Question: Does the presence of rolled threads benefit the fasteners resistance to hydrogen stress cracking in service?

Approach: Use the LRA accelerated stress corrosion testing system and measure K_{Iscc} of the base metal as compared to the processed threads under conditions of cathodic protection of the fastener.

SEM

Scanning electron microscope shows the existence of 35μ m compression layer at the root of a rolled thread that is exposed on a fracture surface produced by the LRA stress corrosion test under conditions of cathodic protection.



Results: Presence of rolled threads raises the resistance to hydrogen stress cracking in service (K_{Iscc}) by 50%; i.e., the installation torque can be increased by 50% above the threshold of a machined thread without inducing hydrogen embrittlement in service when used in a steel structure.

Recommendations: Use rolled threads in processing of fasteners. Initiate a Quality Control K_{Iscc} testing program to insure processing controls relative to resistance to hydrogen embrittlement are maintained. K_{Iscc} is much more sensitive to subtle microstructural variations than are mechanical properties such as tensile, yield, elongation and R.A.

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Fast Fact No. 17

Fastener Failure Analysis

Rising Load Identifies Hydrogen Embrittlement in Plated Fasteners

Problem: In-service failures leads to suspicion of Hydrogen Embrittlement (HEM) due to processing in fasteners. Sustained load test at 75 percent of UTS produces no failures even after 72 hours.

Solution: Perform standard E8 tensile test on fasteners before and after baking to establish base line data. Perform slow rising load tensile test over 24 hour time period to detect HEM. Presence of HEM is signified by fracture occurring below E8 fracture load.

ASTM E8 Tensile Test (No Bake) Fracture @ 5600 pounds	ASTM E8 Tensile Test (Baked) Fracture @ 5700 pounds
Slow Rising Load Test (No Bake) Fracture @ 2500 pounds	Slow Rising Load Test (Baked) Fracture @ 4200 pounds

Conclusion: Slow rising load test is discriminating in the detection of HEM in fasteners. Large intergranular fracture zone and low fracture loads under slow rising load testing confirms the presence of HEM.

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Fast Fact

No. 18

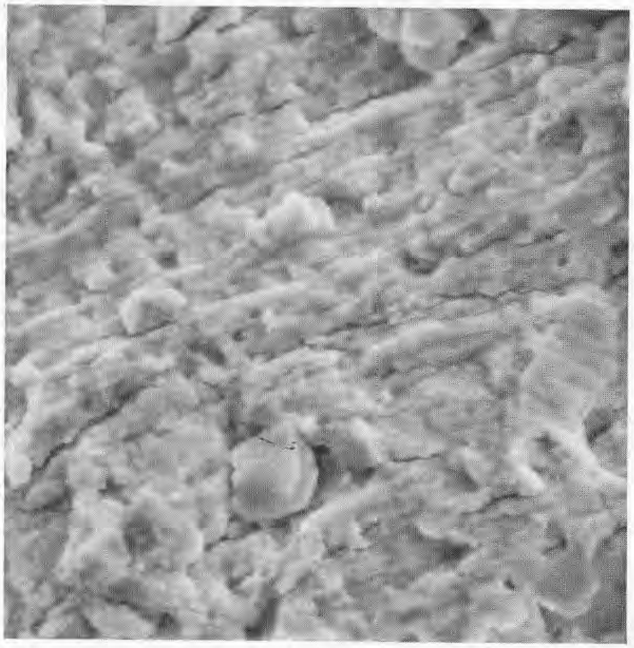
Fastener Failure Analysis

Optical and SEM examination identifies cause of Bolt Failure

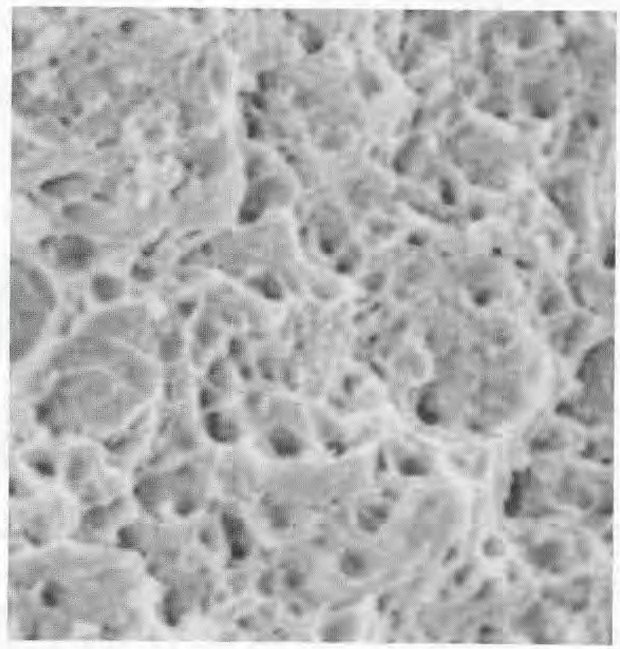
Problem: A 1/2-13 Type 5140 steel retaining bolt for the lower timing gear on a marine engine failed, resulting in permanent damage to the engine. The marine engine was in service for 13 years. The engine was serviced 2 years prior to the bolt failure.

Question: What caused the retaining bolt to fail after 13 years?

Question Results: Optical examination of the retaining bolt revealed corrosion (rust) at the root of the thread. A SEM examination revealed fatigue striations over 2/3 of the fracture surface of the retaining bolt. The remaining fracture surface consisted of dimples, typical of a ductile overload failure.



SEM photograph (1200X) showing fatigue striations.



SEM photograph (1220X) showing dimples.

Conclusion: The protective paint on the retaining bolt was removed during servicing, thereby exposing it to the environment. A pit caused by corrosion in the root of the thread over a period of 2 years, eventually initiated a fatigue crack during operation of the engine. The crack grew to over 2/3 of the cross sectional area of the retaining bolt by fatigue until it failed by overload.

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Fast Fact No. 19

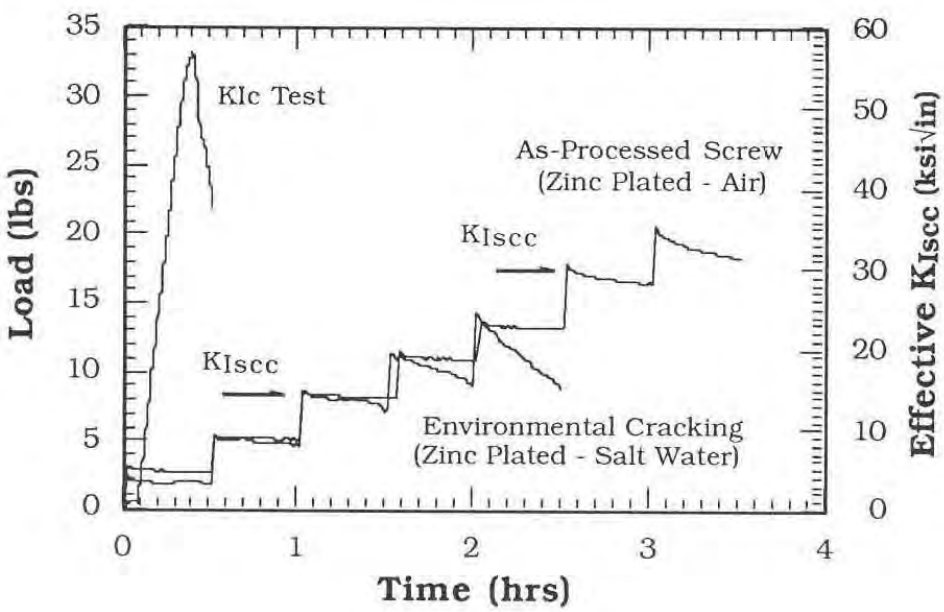
Fastener Failure Analysis

Evaluating the effects of processing and the environment on screws using accelerated LRA RSL-B SCC test system

Problem: The performance of fasteners can be affected by processing and the environment. For example, a fastener improperly coated can experience hydrogen embrittlement while a fastener in a salt water environment can experience stress corrosion cracking.

Question: How do you evaluate the effect of processing and the environment on the performance of a fastener.

Results: The accelerated rising step load in bending (RSL-B) technique, developed at LRA Laboratories, Inc., was used to evaluate the performance of zinc plated 1/4-28 self tapping screws in air and in salt water. The results are shown below on a plot of applied load versus time. A drop in load was the method used to determine when the screw failed. The screw tested using a constant loading rate (KIc test) failed at 33 pounds. The KIc test represents the maximum performance of the screw. The screw tested using the RSL-B K_{Iscc} test in air (Zinc Plated - Air) failed at 18 pounds. This test showed the zinc plating degraded the performance of the screw by 45%. The screw tested using the RSL-B K_{Iscc} test in salt water (Zinc Plated - Salt Water) failed at 9 pounds. This test showed the zinc plating and the salt water environment degraded the performance of the screw by 73%. Note that all the tests were done in less than four hours.



Conclusions: The RSL-B SCC test demonstrated the effect of processing and the environment on the structural performance of zinc plated 1/4-28 self tapping screws.

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Fast Fact No. 20

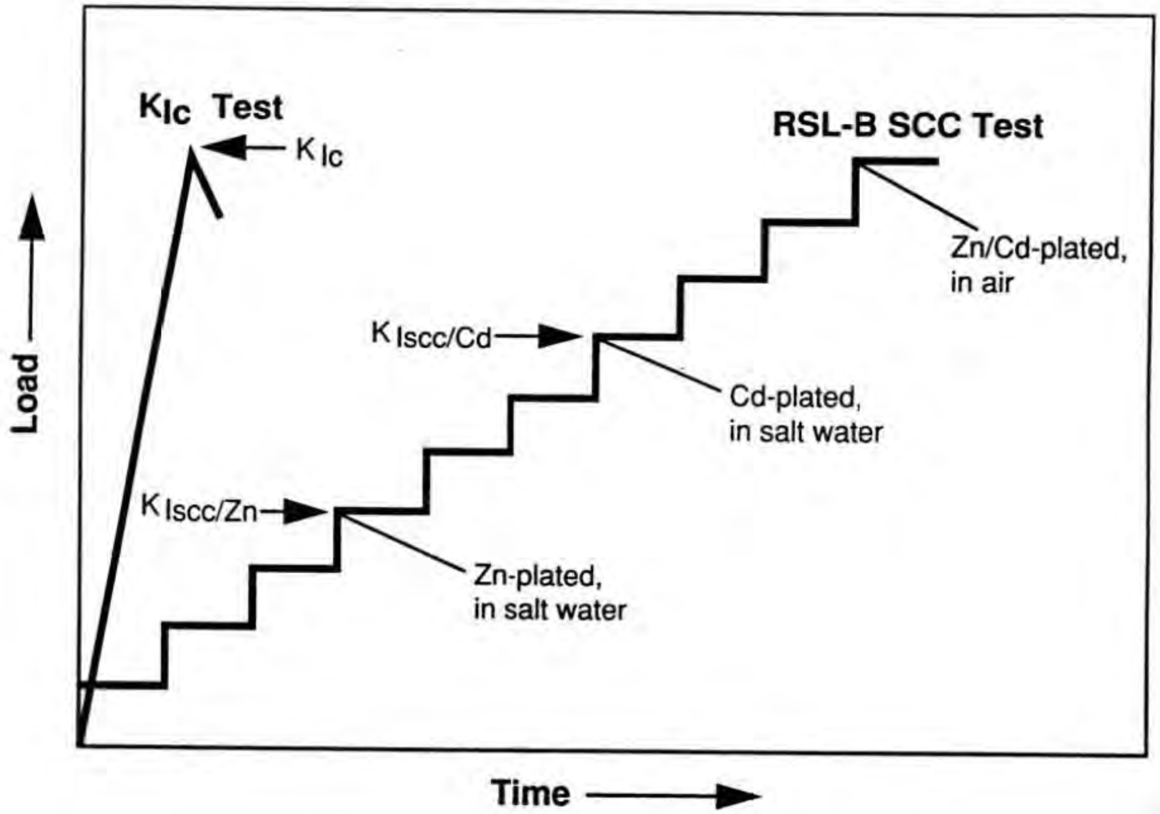
Fastener Failure Analysis

Difference of Coating on SCC of Fastener under Service Conditions

Problem: Zinc plated bolts were found to break by hydrogen assisted crack growth in a marine environment.

Approach: Compare zinc-plated to cadmium-plated bolts with regard to resistance to SCC. Use LRA RSL-B SCC test to evaluate bolts resistance to SCC.

Effect of Coatings or Platings on K_{Iscc} in a Marine Environment



Conclusion: Zinc-plated bolts were found to be more susceptible to SCC in an aqueous environment than cadmium-plated bolts under the same test conditions.

Solution: Change plating from zinc to cadmium, or lower the installation torque/stress to $K_{applied} < K_{Iscc,zinc}$.

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Fast Fact No. 21

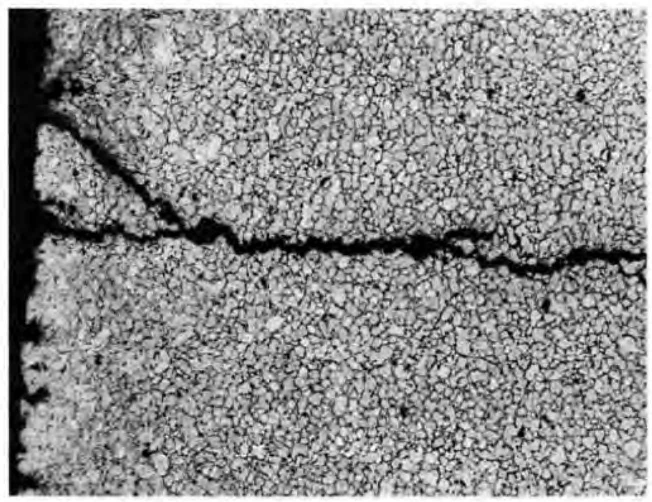
Brass Nut Failure Analysis

Problem: Seventy brass nuts used in an air conditioning system exhibited longitudinal cracks approximately nine weeks after installation.

Question: What caused the brass nuts to fail in a time delay manner?

Results: Visual and metallographic examinations revealed that the cracks were originating on the nut inner diameters as shown in the macro photograph below. Metallographic examination clearly shows the intergranular nature of the through wall cracks. The observed cracking is typical of stress corrosion cracking found in some brass alloys.

Photograph showing the through-crack on the polished transverse cross section of a failed nut.



Photograph (100X) showing the start of the intergranular through-crack at the I.D. of the nut.

Conclusion: A careful review of the aqueous mixture flowing through the system revealed the presence of one component that contained nitrites and nitrates. The additive containing nitrites and nitrates flowing through the inner pipe diameters caused the formation of intergranular stress corrosion cracks resulting in delayed failure of the brass nuts.

Recommendation: Avoid exposing brass components to aqueous environments that are known to cause stress corrosion cracking.

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Fast Fact

No. 22

Head Failure in Tension Bolt

Problem: A tensile fastener that passed the ASTM F606/E8 wedge tensile test (Fig.1), which requires failures in the thread (Fig.2), exhibited failures in the head when installed in service.

Question: What caused the change in failure location?

Approach: An ASTM F606 wedge tensile test conducted at the normal (ASTM E8) tensile loading rate was compared to a slow strain rate ASTM F606 wedge tensile test.

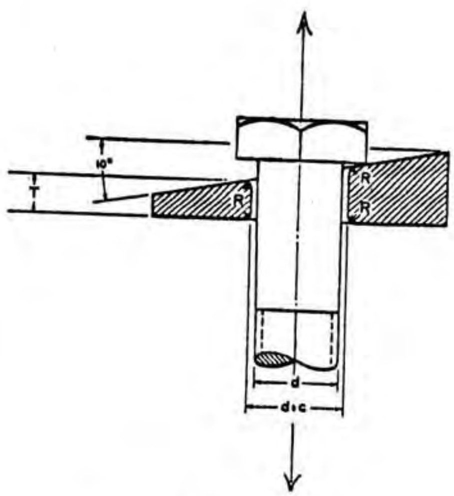

Figure 1. ASTM F606 Detailed Drawing Wedge Tensile Test

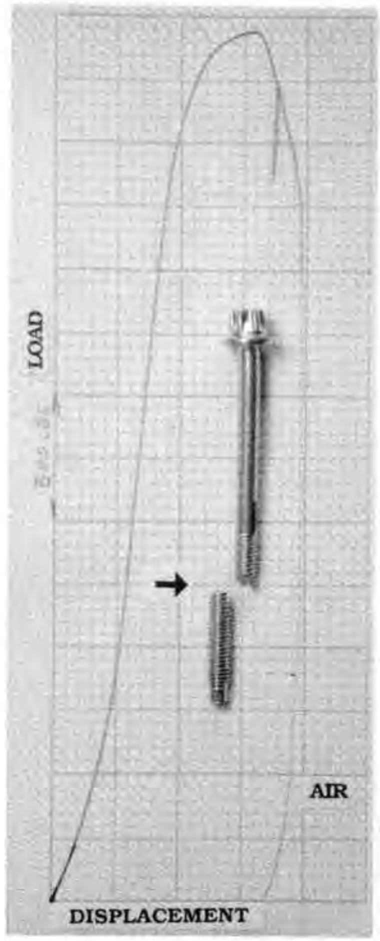

Figure 2. Actual Test Data ASTM F606/E8 Wedge Tensile Test

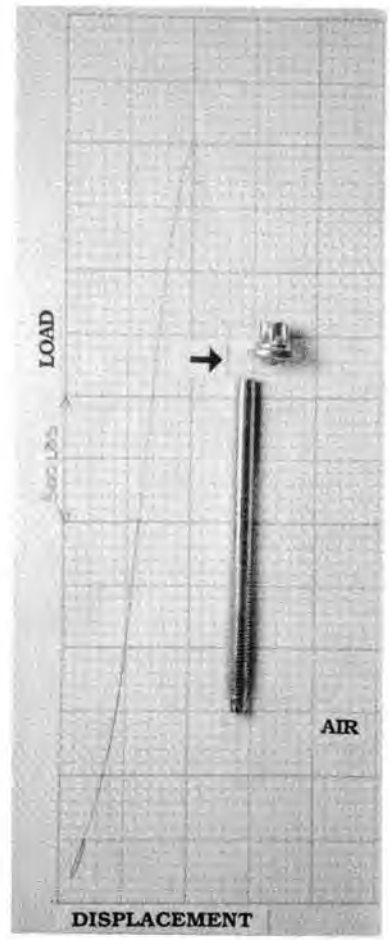

Figure 3. Actual Test Data Slow Strain Rate Wedge Tensile Test

Results: The change in loading rate caused a change in the failure location of the tensile bolt (Fig.3) and the failure mode changed from dimpled to intergranular.

Conclusion: Slow strain rate test conditions producing high local bending stresses under the head induced time delay hydrogen assisted fracture of the head not observed under normal tensile test loading rates.

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Fast Fact No. 23

Optimization of Damage Tolerance of Fasteners to Hydrogen Embrittlement

Problem: How can one compare the damage tolerance of fastener materials at different strength levels to hydrogen embrittlement?

Approach: Conduct a tensile test of a tempered martensitic steel at different hardness and measure the corresponding resistance to hydrogen embrittlement.

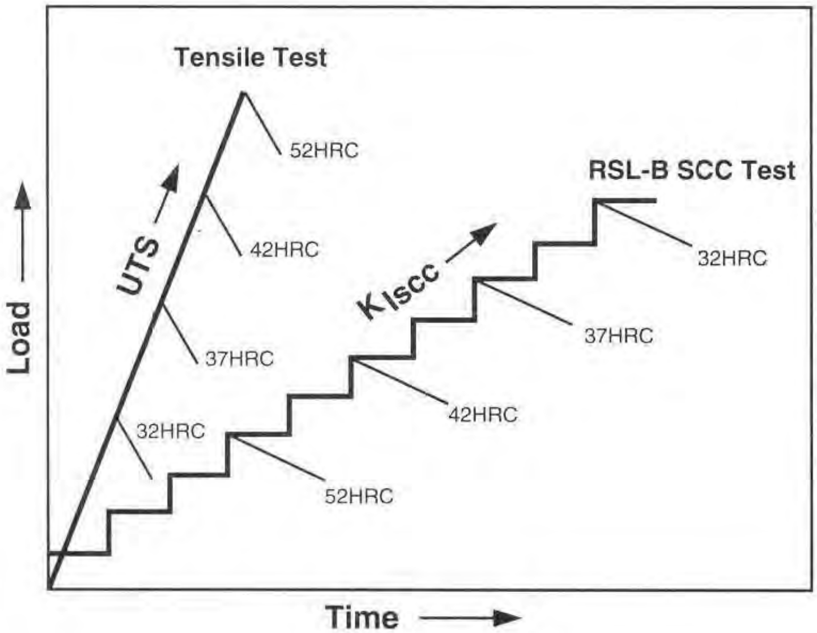

Figure 1.
Tensile strength and the resistance to hydrogen embrittlement vary in an opposite direction — the harder or stronger the more susceptible.

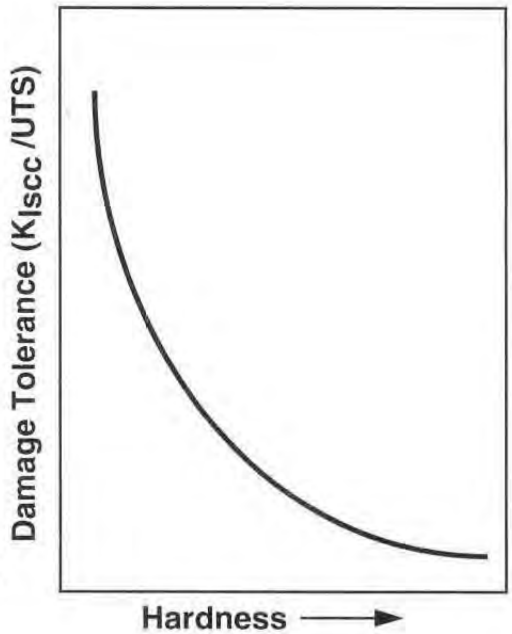

Figure 2.
Damage tolerance decreases with an increase in hardness.

Results: As anticipated, the tensile strength (UTS) increases with hardness as shown in Fig. 1. The susceptibility to hydrogen stress cracking as measure with the rising step load is also seen to increase (Fig. 1). Opposite to the tensile behavior, the threshold for hydrogen stress cracking (K_{Iscc}) is seen to decrease as the hardness increases.

Conclusion: The resulting damage tolerance to hydrogen as measured by the ratio of threshold for hydrogen stress cracking to the tensile strength is seen to decrease as the hardness increases (Fig. 2). To select the minimum damage tolerance for any application must be analyzed from "fitness for purpose" considerations. For fasteners, the diameter, thread radius, and installation stress are the important variables.

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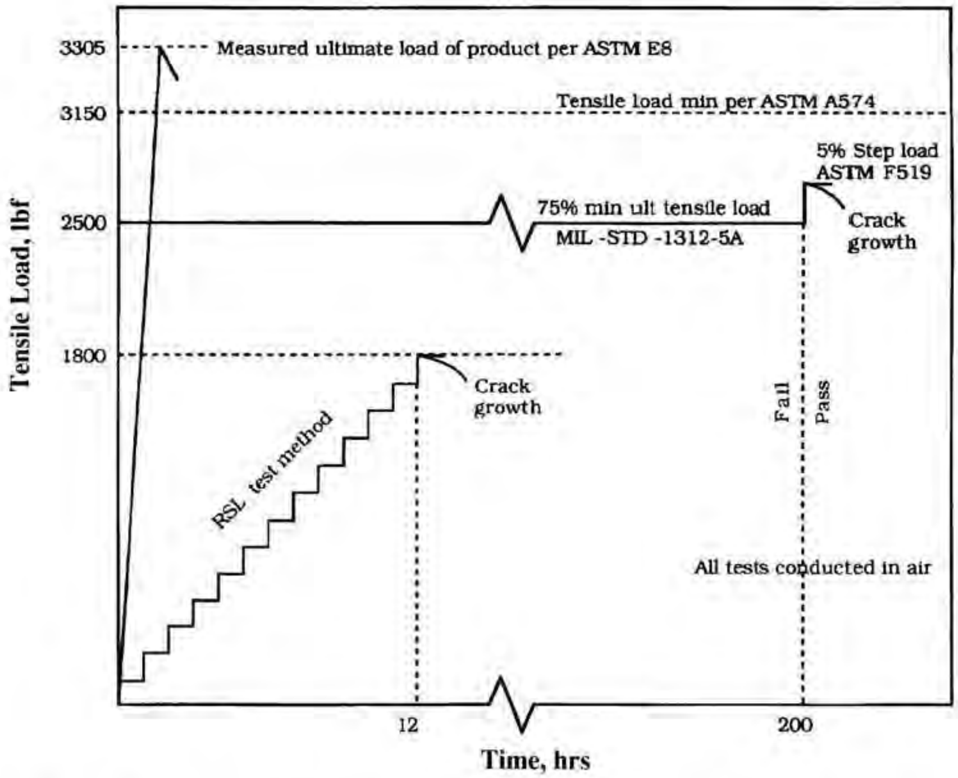
Fast Fact No. 24

Rising Step Load Accurately Identifies Potential for Hydrogen Embrittlement

Problem: Evaluate the potential for hydrogen embrittlement in as-processed zinc plated 10-24 alloy steel socket head cap screws (ASTM A574).

Approach: Compare MIL-STD-1312-5A to the Rising Step Load (RSL) technique (ASTM F7.04 Draft STD D02N16) to measure the resistance to internal hydrogen embrittlement (IHE) of as-processed zinc-plated fasteners.



Results: All tests were conducted in air. The tensile test per ASTM E8 qualified the fasteners by exceeding the minimum rupture load of 3,150 pounds (The actual rupture load was 3,305 pounds). Relative to hydrogen embrittlement, the fastener passed a 200 hour sustained load test at 75% minimum ultimate tensile strength of the product per MIL-STD-1312-5A or 2500 pounds, but failed the RSL test in 12 hours by exhibiting crack growth at 1800 pounds. Using the test protocol of ASTM F519 to discriminate the failure potential of the sample after it successfully passed the 200 hour sustained load, a subsequent 5% step load increase immediately identified the potential for hydrogen embrittlement by initiating crack growth.

Conclusion: The use of an incremental loading or rising step load technique, which is a modified slow strain or constant extension rate test, provides a more accurate and accelerated method of identifying the true potential for hydrogen embrittlement potential of zinc plated fasteners.

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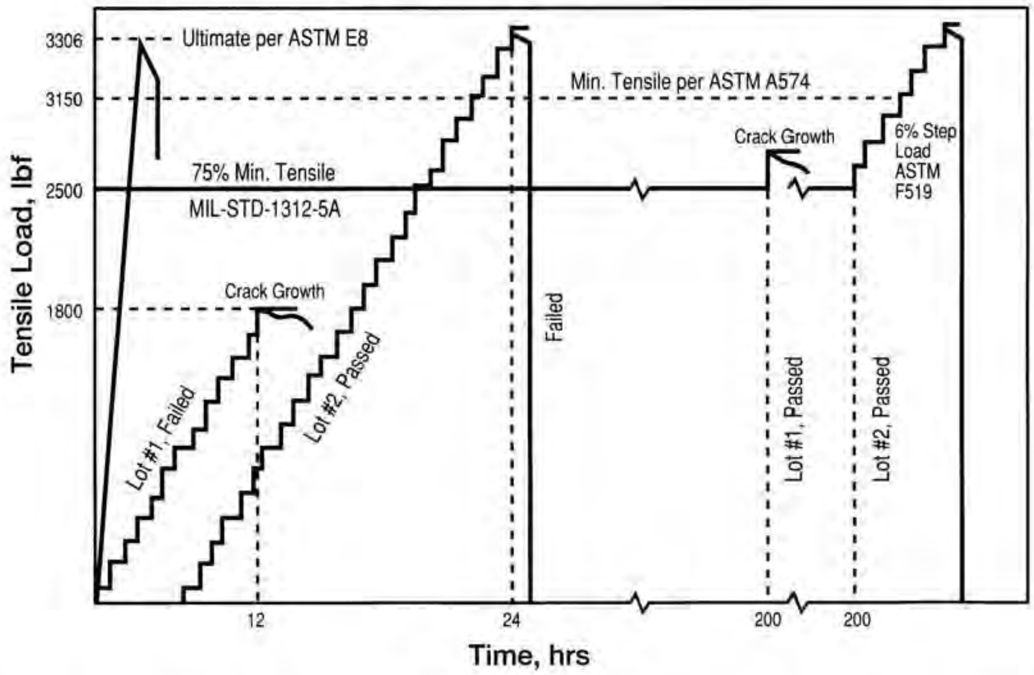
Fast Fact **No. 25**

Rising Step Load Accurately Identifies Potential for Hydrogen Embrittlement — Part 2

Problem: In Fast Fact #24, the potential for hydrogen embrittlement in as-processed zinc plated 10-24 alloy steel socket head cap screws (ASTM A574) was evaluated by the RSL-CERT(T) test (ASTM F7.04 Draft STD D02N16) and compared to MIL-STD-1312-5A and ASTM F519 for embrittled screws. How would the test results differ for screws with no residual hydrogen due to plating?

Approach: The resistance to internal hydrogen embrittlement (IHE) of another lot of as-processed zinc-plated screws was examined and found to be free of residual hydrogen.



Results: All tests were conducted in air. Relative to hydrogen embrittlement, both lots passed a 200 hour sustained load test at 75% minimum ultimate tensile strength of the product per MIL-STD-1312-5A or 2500 pounds, but only the 1st lot failed the RSL test in 12 hours by exhibiting crack growth at 1800 pounds. Using the test protocol of ASTM F519 to discriminate the failure potential of the sample after it successfully passed the 200 hour sustained load, subsequent 5% step load increase immediately identified the potential for hydrogen embrittlement by initiating crack growth in the 1st lot and could be loaded to rupture in the 2nd lot. These results are completely consistent with the RSL-CERT(T) test, which was completed in much less time; 12 hrs. for embrittled Lot #1 and 24 hrs. for embrittlement relieved Lot #2.

Conclusion: The use of an incremental loading or rising step load technique, constant extension rate test RSL-CERT(T) provides a more accurate and accelerated method of identifying the true potential for hydrogen embrittlement of zinc plated fasteners.

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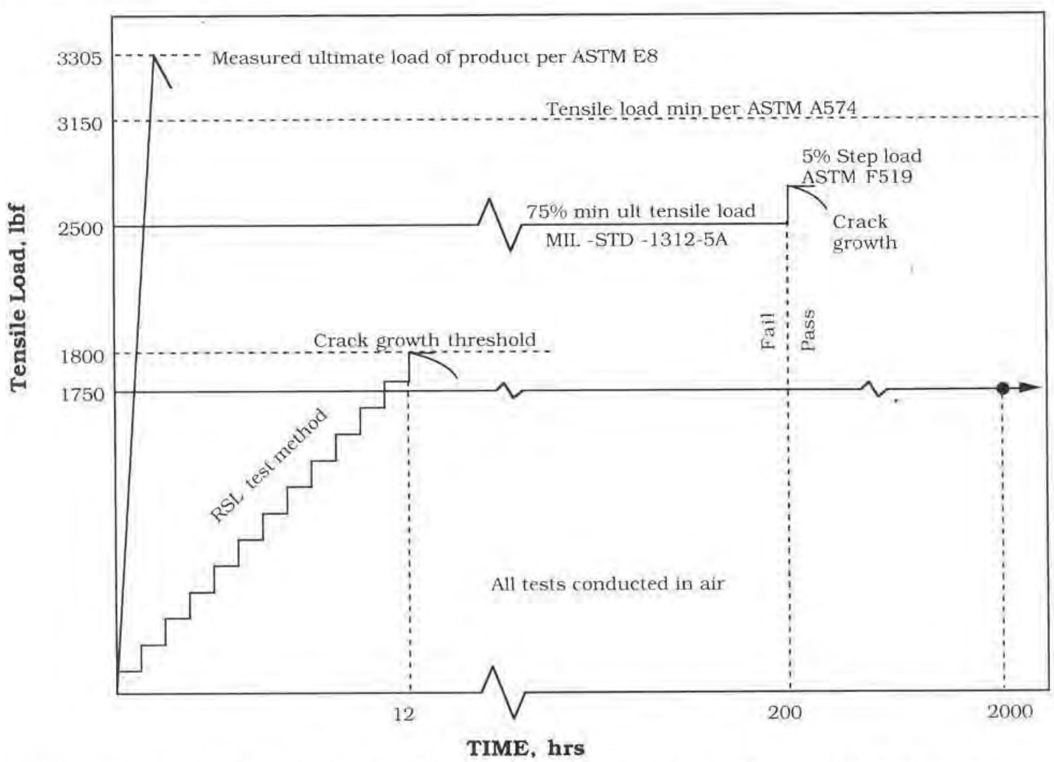
Rising Step Load Accurately Identifies Potential for Hydrogen Embrittlement - Part 3

Problem: In Fast Fact #24 (Part 1) & FF #25 (Part 2), the potential for hydrogen embrittlement in as-processed zinc plated 10-24 alloy steel socket head cap screws (ASTM A574) was evaluated by the RSL(T)-CERT test (ASTM F7.04 Draft STD D02N16) and compared to MIL-STD-1312-5A and ASTM F519. Consistency in the results of the various test methods was found between embrittled screws (FF#24) and non-embrittled screws (FF#25). The final issue is to determine the significance of the threshold stress for embrittled screws.

Approach: Measure the threshold stress to internal hydrogen embrittlement (IHE) per ASTM F7.04 Draft STD D02N16 of the as-processed lot of zinc-plated screws from FF#24 that were found to be embrittled due to residual hydrogen from plating. Establish the physical significance of the threshold load by applying and maintaining a constant load for at least 2000h just below the threshold measured by the RSL-CERT test method to verify that no time delay rupture will occur.



Results: All tests were conducted in air. Loading a screw in air at 50 pounds below the measured threshold did not induce a time delay rupture in the embrittled screws, even after 2000h.

Conclusion: The significance of a threshold stress measured by the RSL-CERT method, employing ASTM F7.04 Draft STD D02N16, is that it defines a clamping load below which no time delay hydrogen embrittlement stress cracking will occur in an installed fastener. Again, the use of an incremental loading or rising step load technique, constant extension rate test, RSL(T)-CERT, can be used to measure a threshold stress and provide a more accurate and accelerated method of identifying the true potential for hydrogen embrittlement of zinc plated fasteners.

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Fast Fact No. 27

Rising Step Load Accurately Identifies Effectiveness of Hydrogen Relief Treatments on Embrittlement due to Zinc Phosphate

Problem: A prescribed bake-out treatment for a zinc phosphate coating is given in terms of time at room temperature or time at 200°F-225°F. Federal Specification TT-C-490 calls for 200°F-210°F for 15 min and DOD-P-1623F calls for 210°F-225°F for 8 hrs or a room temperature bake out for 120 hrs (5 da). Others standards in the automotive industry call for 239±18°F for 1 hr or a room temperature bake out for 48 hrs (2 da). There is an implied equivalence between storage at room temperature and time at elevated temperature. This equivalence nor the effectiveness of a hydrogen embrittlement relief treatment has never been proven.

Approach: The Rising Step Load (RSL) technique provides an precise method to quantitatively measure the effectiveness of the various plating and coating hydrogen embrittlement relief treatments much in the same way as a thermometer is used to precisely measure the body temperature to determine if a person has a fever. To demonstrate this capability, low-alloy steel specimens at 50 HRC to 52 HRC were ZnP plated per TT-C-490 and stored for various times prior to test. The RSL test was used to quantify the percent recovery.

Results: All RSL tests were sequentially conducted in air in 24 hrs. The rupture stress of the plated specimens divided by the breaking or fracture stress of the unplated specimen tested in air per ASTM E8 was the measure of percent recovery.

Condition of Test Specimen	Storage Time	% Recovery
ZnP plated per TT-C-490	0da	60
ZnP plated per TT-C-490	1da	70
ZnP plated per TT-C-490	3da	81
ZnP plated per TT-C-490 + 190°F for 5 min.	7da	97
Unplated – ASTM E8 Tensile	0da	100

Conclusion: Storage at room temperature is an hydrogen relief treatment for ZnP coated high-strength steel specimens. An elevated temperature bake out treatment might be required to remove all of the residual hydrogen. The RSL test method can quantitatively measure the effectiveness of a bake out treatment or the percent recovery to the unplated condition.

NOTE: These results cannot be used as general guidelines without conducting more tests for a specific plating procedure. DOD-P-1623F, for example, is a much heavier ZnP coating than TT-C-490 and therefore would require a more extended bake out time. Other factors like the use of an acid bath during pickling would affect the amount of hydrogen introduced into the steel due to processing and therefore the storage or bake out requirements. Residual stress due to case hardening the part, for example, would also be an important consideration.

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Fast Fact

No. 28

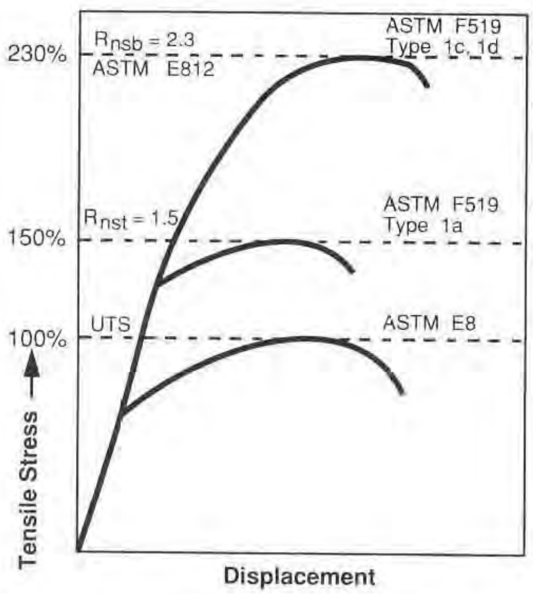
Notch Effect in Fasteners

Problem: A tensile fastener has helical threads. The question becomes, what is the effect of the notch depth and radius at•the root of the thread on the strength and life of a fastener?

Solution: Material strength is characterized by a smooth round bar tensile tested in accordance with ASTM E8. A typical load-displacement curve is shown in the figure below.

Notched Strength/Tension: Fastener strength is more accurately related to the notched round bar tensile strength, which can be much higher than the material strength, by as much as a factor of 1.5 to 1.6. The notched tensile strength of Grade 8 steel is about 225 ksi as compared to the ASTM E8 smooth bar tensile strength of 150 ksi. For this reason, a larger, fictitious stress area is often used instead of net section area in order for the calculation of the strength of a tensile fastener to be the same as the material tensile strength. A comparison of the load curves for the two types of specimens is shown in the figure to demonstrate the effect of a notch on the tensile strength. Stated differently, the tensile stress produced in a notched tensile bar such as in ASTM F519 Type 1a, can be 150% higher than the ultimate tensile strength of a smooth tensile bar.

Notched Strength/Bending: Unfortunately, all of preceding discussion is based on the assumption that the load is perfectly axial and the stress is a pure tensile stress. In fact, most of our stress durability standards such as MIL-STD-1312-5A have the faces of the bearing surface parallel within 1° and perpendicular within 0.5° in order to insure a pure axial loading. The problem then magnifies, because in the real world or in an actual service installation, all of the clamping load is not perfectly axial and more often than not, a bending load is introduced into the fastener.



The effect of a notch on a bar in bending is again superimposed in the attached figure. As noted, the notched round bar bend strength can be much higher than the material strength by as much as a factor of 2.3. The notched bend strength of Grade 8 steel is about 350 ksi (see AFJ Jan/Feb 95, pg 7) as compared to the ASTM E8 smooth bar tensile strength of 150 ksi. Stated differently, the tensile stress produced in a notched bend bar, such as in ASTM F519 Type 1c, or 1d, can be 230% higher than the ultimate tensile strength in a smooth tensile bar.

The notched strength in bending is calculated analogous to ASTM E812 for a precracked specimen and is designated R_{nsb}. By analogy, the notched strength in tension is designated R_{nst}. Compared to the smooth bar tensile strength, $R_{nst} \leq 1.5$ and $R_{nsb} \leq 2.3$.

Life: The importance of the notched tension or notched bend strength to the life of the part is that depending on the geometry and the type of loading on a fastener, local stresses can be produced that are as much as twice as high as the maximum tensile strength as measured with a tensile test. This means that much less hydrogen is required to induce a hydrogen stress cracking failure in a fastener in bending than one loaded in pure tension.

Summary: The presence of a notch plays a very important role in fasteners. The purpose of this Fast Fact is to illustrate and quantify the magnitude of the change in the maximum local stress produced by a notch depending on whether the type of load is tension or bending and the subsequent effect the resulting stress has on minimizing the amount of hydrogen required to induce an intergranular time delay failure.

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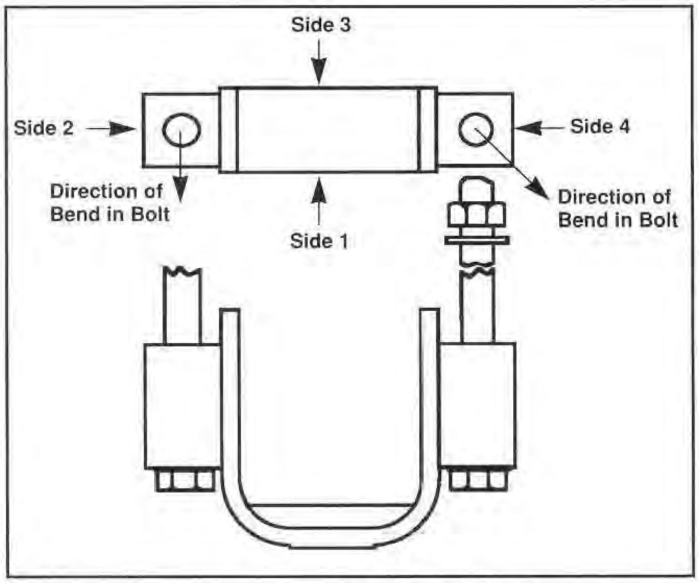
Fast Fact No. 29

Failure Analysis of Front Axle Lateral Rod Bracket and U-Bolts

Problem: Two fractured lateral rod bracket bolts (5/8-11) and a fractured U-bolt (5/8-18) were provided for a failure analysis to determine the cause of the fractures. The hardware was obtained from the front axle of a bus that was reportedly involved in a front-end collision with an automobile. The objective was to determine if the failed bolts caused the accident or were a result of the accident.



Schematic illustration of lateral rod bracket and fractured bolts.

Approach: The chemistry of the fractured parts was analyzed and the hardness of the materials was measured. A visual examination and a scanning electron microscope (SEM) examination analysis of the fracture surfaces were conducted. In addition, the Charpy V-notched (CVN) impact energy of the U-bolt material was measured to estimate the fracture toughness and to provide an exemplar fracture surface for impact overload conditions.

Results: The chemistry of the bracket bolt and the U-bolt was found to be consistent with the requirements of an SAE Grade 8 fastener. The hardness values of the fractured parts were also found to be 37 HRC, which is within the required range for a Grade 8 fastener of 33 HRC to 39 HRC.

The SEM evaluation of the fracture surfaces revealed that the bracket bolts failed as a result of a tensile stress overload in a bending mode and the U-bolt failed as a result of a shearing mode overload. No evidence of prior defects was found on any of the fractured parts.

The impact test conducted on the CVN specimen machined from one leg of the fractured U-bolt revealed that the U-bolt material had adequate fracture toughness, estimated to be 170 ksi $\sqrt{\text{in}}$ (50 ft-lbs). In addition, the fracture surfaces produced by the CVN impact test were examples of tensile stress overload in the central region, while the slanted surfaces along the sides and the bottom of the fracture surface are due to shear stress overload. At higher magnification in the SEM, the dimpled rupture morphology typical of the tensile stress overload was essentially identical to the fracture surface morphology of the bracket bolt, but is distinctly different from the fracture surface features of the U-bolt. However, an examination of the fracture surface morphology of the shear lip region of the CVN specimen at high magnification in the SEM fractograph was essentially identical to the fracture features observed on the fracture surfaces of the U-bolt. Thus, it is apparent that the fracture of the U-bolt occurred as a result of a shearing type of failure.

Conclusion: The lack of any defects in these materials, the good toughness exhibited by the impact test on the U-bolt material, the fact that failure occurred as a result of an overload stress, and the fact that the failed parts were discovered following a front end collision with an automobile, indicates that the failure of the bracket bolts and the U-bolt occurred as a direct result of the accident and were not the cause of the accident.

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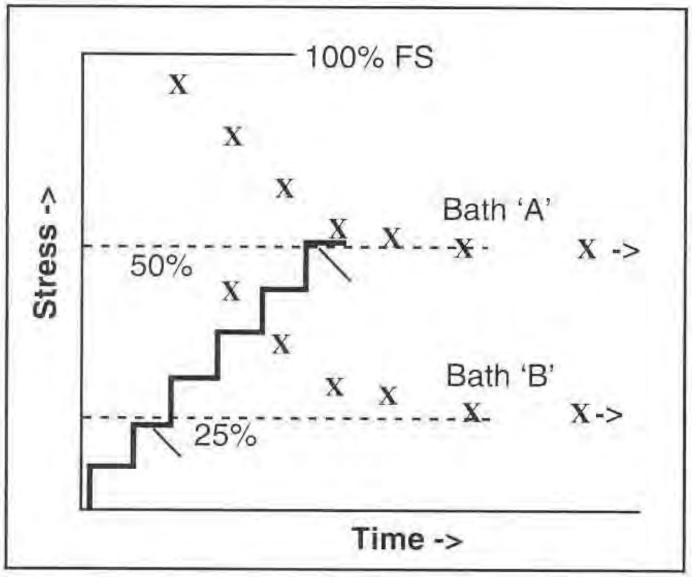
Fast Fact No. 30

Quantifying Plating Baths Relative To Hydrogen Embrittlement

Problem: The amount of residual hydrogen that is introduced into a fastener during electroplating and its effect on the service performance of the product is not currently measured. Pass/fail sustained load tests are used to indirectly imply if a product is free of hydrogen. The conditions on a pass/fail sustained load test represent only one point of a series of tests used to measure the threshold for hydrogen assisted stress cracking and therefore do not quantify the threshold.



*Schematic illustration of rising step load (RSL) test compared to a sustained load time-to-failure test (**x**) to measure the threshold for hydrogen embrittlement of two different plating baths.*

Approach: Measurement of the threshold for hydrogen embrittlement in a specimen can be used to quantify the amount of residual hydrogen in a plated fastener. The stress below which no hydrogen assisted stress cracking will occur after a specimen is plated can be used as a direct measure of the quality of a plating bath. The higher the threshold, the better the quality of a plating bath and its processed parts.

By ratioing the threshold to the fracture strength (FS) of the specimen, a quantitative measure of the amount of degradation in the specimen due to residual hydrogen is calculated that can be used to compare the quality of different plating baths.

By utilizing ASTM F1624 and a specimen at 52 HRC, thresholds for hydrogen assisted cracking can be detected in less than 8h. If standardized specimens are used, it will be possible to compare one plating bath to another.

Results: To insure that the test will be conducted in ≤24h as a quality control test, specimens were used at 52 HRC to represent a "worst case" condition of the fasteners in accordance with ASTM F519. From the attached Figure, plating bath 'A' with a ranking of 50% would be less embrittling than plating bath 'B' with a threshold of 25%.

If either specimen had achieved 100% of the fracture strength in an RSL test, it could be concluded that the plating process is free of hydrogen. Since both baths embrittle the specimens, the product should be tested in accordance with ASTM F1624 to determine if the residual hydrogen was enough to cause embrittlement of the product. If the product threshold is 100%, it means that the product will have tolerance for the residual amount of hydrogen. The quantitative correlation between the test specimen and the product can be used in future quality control testing of the product with the specimen and RSL test method.

Conclusion: The use of the rising step load test per ASTM F1624 provides an accelerated and cost effective method of measuring the threshold for the onset of stress cracking due to hydrogen embrittlement that can be used to quantify the quality of a plating bath.

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Fast Fact No. 31

Similarities/Differences Between Hydrogen Embrittlement and Stress Corrosion Cracking

Hydrogen Embrittlement	Stress Corrosion Cracking
Time-delay-fracture in Air after exposure to environment.	Time-delay-fracture in Environment after exposure to stress.
Requires $[H]^+$ critical from processing (plating, acid cleaning) for a given stress above Hydrogen Stress Cracking threshold.	Requires $[H]^+$ critical from corrosion reaction in environment for a given stress above Hydrogen Stress Cracking threshold.
Plating, acid cleaning, etc., considered environment in which "corrosion reaction" takes place to generate $[H]^+$, while stress is below threshold.	"Corrosion reaction" takes place in environment to generate $[H]^+$, while stress is above threshold.

The only difference between hydrogen embrittlement and stress corrosion cracking is the sequence of applying the stress and exposure to hydrogen.

Phenomenon is the same!

Mechanism is the same!

The only difference is sequencing of stress and introduction of $[H]^+$ into the part.

Hydrogen Embrittlement: $[H\}^+$ + Stress --> Rupture

Stress Corrosion Cracking: Stress + $[H]^+$ --> Rupture

Note: If part was bent or plastically deformed to introduce residual stress before being put into a plating bath, it would have cracked during the plating process and have been identified as Hydrogen Embrittlement although having all the ingredients of Stress Corrosion Cracking.

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Fast Fact No. 32

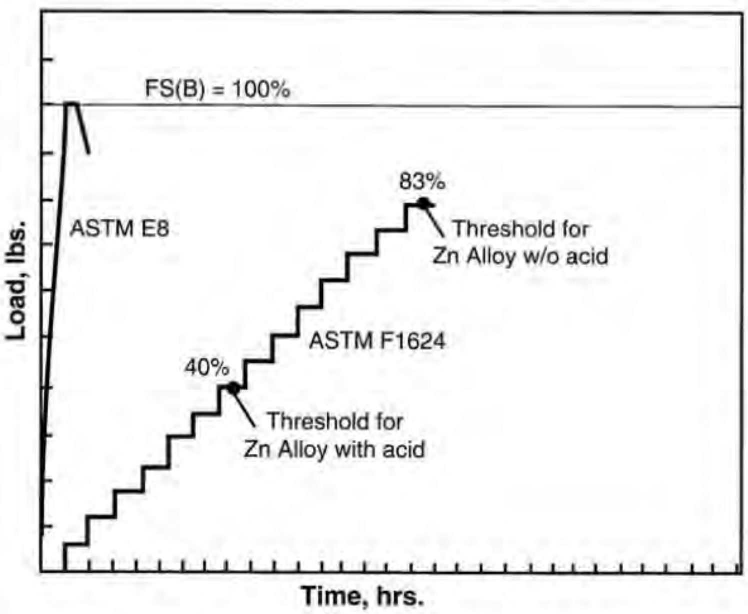
Fastener Failure Analysis

Identifying the source of hydrogen in M10 fasteners using the Rising Step Load (RSL™) accelerated testing technique.

Problem: Within 24 hours of their installation, failures of zinc alloy plated M10 fasteners were detected. Subsequent failure analysis of the fasteners revealed the parts were hydrogen embrittled by the finishing process, but was undetected by conventional hydrogen embrittlement plate testing.

Question: Can the primary source of the hydrogen contamination be identified for this zinc alloy plating process?

Approach: Evaluate the residual hydrogen produced by processing using certified ASTM F519 square bar bend test specimens that are processed exactly the same way as the fasteners. To evaluate the contribution of a specific processing step, the step is omitted upon subsequent specimen processing. The difference in degradation in the test specimen is directly attributable to that processing step. In this M10 failure, an acid descaling process was being utilized prior to the zinc alloy plating. Since the suspected hydrogen generating steps were the acid bath and the zinc alloy deposition process, specimens were processed with and without the acid descaling step to isolate the primary source of residual hydrogen.



Results: The fracture strength (FS) of the square bar was measured to be 250 pounds at a relatively rapid loading rate in accordance with ASTM E8, which is independent of any residual hydrogen. The Rising Step Load (RSL™) technique, ASTM F1624, at a slower rate measured the degradation of the test specimens after zinc alloy plating with and without the acid descaling process. The results are illustrated on the plot of applied load versus time. A drop in load was the method used to determine the threshold.

The specimen plated without the acid bath descaling process failed at 208 pounds. The RSL test showed the zinc plating degraded the fracture strength of the specimen to approximately 83% FS of the maximum. The specimen tested after plating with the acid bath failed at 100 pounds, 40% FS. The RSL tests demonstrated that the acid descaling process was the primary source of hydrogen responsible for the M10 fastener failures.

Conclusion: The primary source of hydrogen contamination in finishing processes can be identified using ASTM F519 test specimens and the RSL technique per ASTM F1624.

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Fast Fact No. 33

Hydrogen Susceptibility Ratio - H_{sr}

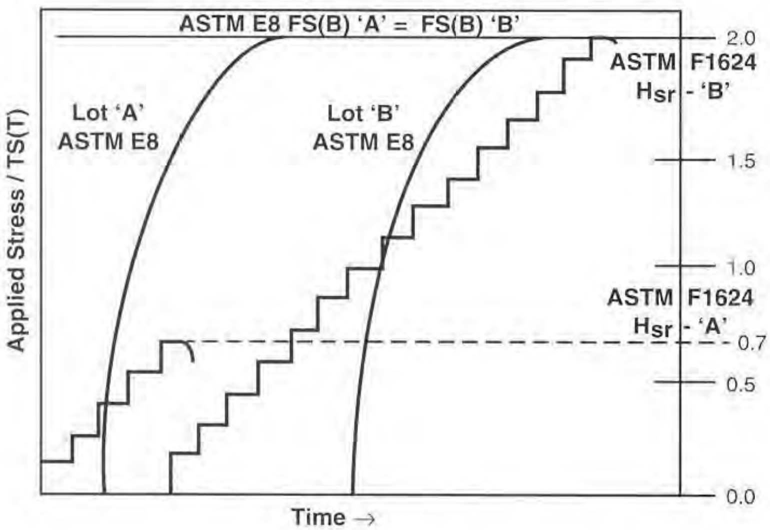
A method of identifying if lots of fasteners are susceptible to hydrogen stress cracking induced by processing and/or the environment prior to their use.

Problem: Multiple lots of M10 Grade 8 fasteners were finished in the same zinc cyanide processing line. Within days of their installation, one lot of these fasteners began to exhibit in-service hydrogen stress cracking (HSC) failures. The lots of fasteners all passed the ASTM F606 hydrogen embrittlement test.

Question: How can the susceptibility of a lot of fasteners to a service environment be detected prior to their processing and subsequent use?

Approach: Compare the baseline breaking load tested in tension. FS(T) or bend, FS(B) in air at ASTM E8 loading rates to the threshold stress measured with the Rising Step Load accelerated testing technique per ASTM F1624 in an aggressive hydrogen charging environment for the lot of fasteners failing in service and for a lot which was not failing in service.

The results at the faster rate are independent of the amount of residual hydrogen in the bolt because sufficient time is not available for the hydrogen to diffuse. At the slower rate, sufficient time exists for the diffusion of hydrogen.



H_{sr} is a measure of the product's susceptibility to hydrogen stress cracking regardless of the source of hydrogen, whether it is from a plating bath or from the environment. H_{sr} is the threshold stress normalized by dividing by the minimum specified tensile strength of the fastener, TS(T). The maximum value that can be measured is 1.5 in tension and 2.0 in bending. Anything less is a measure of the susceptibility to hydrogen assisted stress cracking.

Results: The results are illustrated on the plot of applied stress versus time. The baseline, fracture strength in bending, FS(B), of the two lots was measured to be the same (2.0) at a relatively rapid loading rate in accordance with ASTM E8, which is independent of any residual hydrogen. By comparison, the RSL-H_{sr} tests indicated a significant difference in susceptibility to hydrogen stress cracking. Lot 'A', the fasteners failing in service, was found to have an Hsr of 0.7. Lot 'B' was found to be immune to HSC with an H_{sr} of 2.0.

Conclusion: The H_{sr} can be used as a quality assurance test to evaluate lot-to-lot variations in hydrogen stress cracking susceptibility using the RSL technique per ASTM F1624.

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Fast Fact No. 34

Hydrogen Uptake Distribution From Barrel Electrodeposited Zinc

Using ASTM F519 Type 1e Test Specimens and ASTM F1624 Test Protocol

Problem: The residual hydrogen concentration introduced into fasteners during an electrolytic barrel plating process has been claimed to follow a normal distribution or bell-shaped curve. In addition, it has been claimed that baking of fasteners does not eliminate this random hydrogen distribution and therefore is believed to be responsible for the randomness of failures in the plate test.

Question: Is the distribution of the residual hydrogen concentration from a barrel plating process bell shaped or uniform?

Approach: Evaluate the residual hydrogen produced by processing using ten (10) certified ASTM F519 square bar bend test specimens that are processed with a batch of fasteners in an acid-chloride zinc barrel plating process. Compare the breaking loads when tested in bending, FS(B), in air as measured with the Rising Step Load accelerated testing technique per ASTM F1624. The degradation in breaking strength is the measure of the residual hydrogen in the test specimen as a result of the plating process.

Specimen Number	RSL™ Fracture Load, lbs.
51	227.7
52	226.0
53	238.0
54	224.8
55	228.6
56	225.6
57	222.8
58	229.7
59	224.1
56	226.0
Average ==> 227.3 ±4.1 lbs (±1.8%)	

Table 1. *Hydrogen distribution in standard specimens represented by RSL™ fracture load.*

Results: The measured fracture loads of the ten (10) certified ASTM F519 square bar bend test specimens are listed in Table 1. Based upon these results, the distribution of hydrogen uptake for the barrel plating process was found to be uniform within ±1.8%. The variation observed for the plate tests is therefore due to another source. The most likely source would be from the variation in the torque-tension relationship for a given torque resulting in a bell-shaped distribution of maximum local tensile stresses in the fasteners in the plate.

Conclusion: The barrel plating process does not produce a random or bell-shaped distribution of residual hydrogen in plated parts. The amount of residual hydrogen co-deposited during the plating process is uniform. Rising Step Load (RSL™) technique, ASTM F1624, quantitatively measured the degradation from an acid-chloride zinc barrel plating line.

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Fast Fact

No. 35

Fastener Failure Analysis

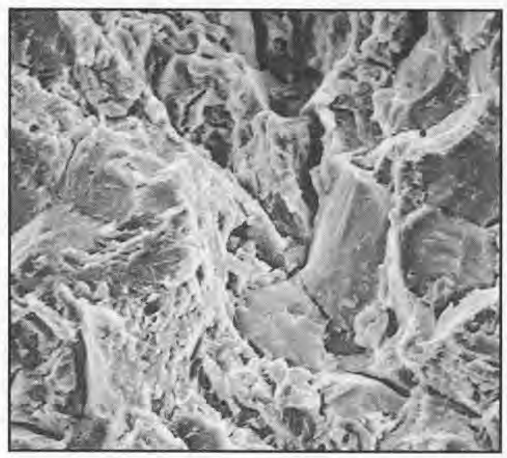
In-service failure mechanism for case hardened M6 fasteners is duplicated using the Rising Step Load (RSL™) accelerated testing technique

Problem: Within days of installation, failures were observed for organic/zinc fill plated M6 self tapping screws. The fracture surface morphology in the core was primarily intergranular (IG) throughout with some patches of IG with dimple features. Subsequent failure analysis of the screws revealed the parts failed by time delayed hydrogen assisted crack growth.

Question: Could the fracture topography/failure mode of the failed screws in the core be duplicated with exemplar RSL™ hydrogen embrittlement testing?

Approach: Since the source of the hydrogen responsible for the failures was not known, a scanning electron microscopy (SEM) analysis was performed on screws from the failed lot subjected to RSL™ testing in air and under a galvanic hydrogen charging environment. The air tests evaluated the effect of residual hydrogen produced during processing of the screws, internal hydrogen embrittlement (IHE). The external hydrogen embrittlement (EHE) tests were conducted at the electrochemical potential of the plating material to simulate the galvanic interaction between the base metal and the coating in service. All of the tests were conducted in bending per ASTM F1624 using the FDI Rising Step Load test system, RSL™ 1000S1-B.

Core Fracture Morphology for IHE RSL™ Test Dimple Overload Features. 1KX



Core Fracture Morphology for EHE RSL™ Test — IG with Dimple Features. 1KX

Results: The fracture morphologies for the IHE and EHE RSL™ exemplar testing are shown in the above Figures. The IHE RSL™ tests were all found to reach 100% of the fracture strength and exhibited only dimple overload features in the core. The EHE RSL™ tests exhibited significant reductions in fracture strength, and revealed a fracture surface primarily IG with patches of IG with dimples in the core, which is identical to the core of the in-service failures.

Conclusion: The in-service failure fracture morphology was duplicated using RSL™ exemplar testing per ASTM F1624. The source of the hydrogen contamination responsible for the M6 failures was not from processing, but was attributed to the galvanic interaction between the base steel and the coating material in the service environment.

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Fast Fact No. 36

Selection of a Coating for Hydrogen Sensitive Products

Based on Rising Step Load (RSL™) accelerated testing results

Problem: How can one evaluate the Internal Hydrogen Embrittlement (IHE) potential of various coatings and specific processes especially for application on a cold-worked, high-strength product with residual stresses.

Approach: Evaluate the residual hydrogen produced by candidate coating processes using certified ASTM F519 square bar bend test specimens that are processed exactly the same way as the product form. Compare the coated specimen breaking loads when tested in bending in air per ASTM F1624 as measured with the FDI Rising Step Load test machine, RSL™ 1000S1-B. The degradation in breaking strength, with respect to the baseline of 225.0 pounds, is the measure of the residual hydrogen in the test specimen as a result of the coating process.

Coating Process	RSL™ Fracture Load, lbs.	% Fracture Strength
ZnP/Oil Coating	225.0	100
ZnP/Oil Coating	216.0	96
Zn-Alloy Plating	166.5	74
Zn-Alloy Plating	177.8	79
Zn-Alloy Plating w/Acid Descale	101.3	45
Zn-Alloy Plating w/Acid Descale	114.8	51

Table 1. *IHE potential of coating processes represented by RSL™ fracture load.*

Results: The measured fracture loads of the processed ASTM F519 square bar bend test specimens are listed in Table 1. Based upon these results, a clear delineation between processes in terms of IHE potential is observed. The zinc-alloy plating process which included a mineral acid descale severely degraded the fracture strength of the test specimens. Eliminating the acid descaling step reduced the absorbed hydrogen, but still resulted in a 20% to 25% loss in fracture strength. Only the ZnP/oil coating process did not induce IHE in the test specimen.

Conclusion: The potential for IHE for various coatings or finishing processes can be identified using the ASTM F519 standard square bar test specimens and the RSL™ technique per ASTM F1624. To minimize the potential for IHE cracking during processing, these hydrogen sensitive, cold-worked, high strength products should be coated using the ZnP/Oil process.

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Fast Fact No. 37

Selection of Baking Time and Temperature

Baking parameters identified for thin dense chrome plating process using Rising Step Load (RSL™) accelerated testing protocol.

Problem: Multiple post-plating bake cycles (times/temperatures) were proposed to eliminate the deleterious effects of hydrogen produced during a thin dense chrome plating process.

Question: Which baking time and temperature combination is required to eliminate the potential for internal hydrogen embrittlement (IHE)?

Approach: Evaluate the residual hydrogen produced by processing using certified ASTM F519 square bar bend test specimens that are processed exactly the same way as the parts in the thin dense chrome plating process. Compare the Rising Step Load (RSL™) threshold loads of unprocessed bare, as processed, and processed and baked specimens when RSL™ threshold tested in bending in air per ASTM F1624 as measured with the FDI RSL™ 1000S1-B test machine. The degradation in threshold load is the measure of the residual hydrogen in the test specimen as a result of the plating process and the post-plating baking cycles.

Table 1. *Effectiveness of Baking Cycles in Terms of IHE Represented by RSL™ Threshold Load.*

Specimen Condition	RSL™ Threshold Load,* lbs.	% of Baseline Threshold
Bare - Baseline	214.5 ± 5.1	100
As-processed - No Bake	85.4 ± 9.7	40
Baked 8 hrs @ 275F	102.9 ± 4.8	48
Baked 12 hrs @ 275F	115.8 ± 5.6	54
Baked 8 hrs @ 375F	202.8 ± 5.0	94
Baked 12 hrs @ 375F	211.5 ± 0.2	99

* Average and Std. Deviation of four tests.

Results: The measured threshold loads of the processed ASTM F519 square bar bend test specimens are listed in Table 1. Based upon these results, the effectiveness of the two baking temperatures is clearly observed. The post-plating bake cycle at 275°F reduced the potential of IHE slightly after times of 8 and 12 hours. The baking cycle at 375°F, though, nearly eliminated the embrittlement after 8 hours and completely eliminated it after 12 hours. The 275°F baking cycle should not be used as a post-plating bake unless longer baking time is found to be more efficient in the removal of the residual hydrogen from the processing.

Conclusion: To insure the parts are free from the potential for IHE, baking should be conducted at 375ºF for 12 or more hours. The effectiveness of post-plating bake cycles can be identified using standard ASTM F519 test specimens and the RSL™ technique per ASTM F1624.

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No. 38

Fast Fact

Fastener System Design and Analysis

KI_{scc} of zinc plated alloy steel determined using Rising Step Load (RSL™) accelerated testing protocol.

PROBLEM

Zinc electroplated 3/4"-16 Grade 8 alloy steel fasteners were found to be failing in service from environmental hydrogen embrittlement (EHE).

QUESTION

Why were the fasteners failing under normal service conditions?

APPROACH

Evaluate the stress corrosion cracking (SCC) threshold, $K_{Iscc,}$ for the fastener material, size, and coating using the FDI Rising Step Load test machine, RSL™ 1000S1-B. To evaluate the SCC threshold, $K_{Iscc,}$ of the steel, single edge-notched bend specimens, SEN(B) per ASTM E1290-93, were EDM machined from the 3/4"-16 fasteners. The specimens were fatigue precracked to an a/W of approximately 0.5. Each specimen was tested at decreasing effective strain rates per ASTM 1624-95, using the Incremental Step Loading protocol at an applied electrochemical potential equal to that of the coating material in an aqueous 3.5% NaCl solution. The threshold is the point when the stress intensity became invariant with decreasing strain rate.

Table 1.
Stress Corrosion Cracking Threshold, K_{Iscc}, for Zinc Plated Alloy Steel 3/4"-16 Fasteners

Test Number	Effective Strain Rate, sec^{-1}	Stress Intensity Factor, K_I, ksi $\sqrt{in.}$
1	6.6×10^{-8}	34.3
2	3.3×10^{-8}	30.9
3	1.3×10^{-8}	26.7
4	6.6×10^{-9}	25.9
5	3.3×10^{-9}	26.1

RESULTS

The alloy steel when tested at the electrochemical potential of zinc was observed to have an extremely low stress intensity factor. The test run at the strain rate of 6.6×10^{-8} sec^{-1} was shown to initiate crack growth at a stress intensity of 34.3 ksi $\sqrt{in.}$ Decreasing the effective strain rate lowered the KIscc to a threshold value of 26.0 ksi $\sqrt{in.}$ The slight variation in the threshold numbers between Test Number 4 and Test Number 5 is attributable to the range of hardnesses typically observed in fasteners. Because of its extremely low SCC threshold, this 3/4"-16 Grade 8 alloy steel fastener is susceptible to in-service failures when galvanic corrosion occurs between the fastener and coating at stresses greater than 50% of the yield strength based upon a Damage Tolerance Index, DTI, analysis (AFJ FF#9).

CONCLUSION

The SCC threshold for the Zinc electroplated 3/4"-16 Grade 8 alloy steel fasteners was found to be extremely low at 26.0 ksi $\sqrt{in.}$ To alleviate the in-service failures, either a coating that will generate less hydrogen under galvanic corrosion conditions or a material with a higher SCC threshold should be used.

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No. 39

Fast Fact

Process Control Verification to Prevent IHE in Plated or Coated Fasteners

An Introduction to a Draft Standard

BACKGROUND

An ASTM F16.93 task group, made up of industry experts, was established to investigate the use of an alternate process control method using the incremental step loading (ISL) technique per ASTM F1624. This process control method will allow the quantitative statistical evaluation of finishing processes for damage from internal hydrogen embrittlement (IHE).

SCOPE

The purpose of this test is to prevent IHE of fasteners by monitoring the plating or coating process. The process will be quantitatively evaluated on a periodic basis with specimens as compared to qualifying each lot of fasteners being plated or coated.

Trend analysis will be utilized to insure quality as compared to statistical sampling analysis of each lot of fasteners. This test method consists of a mechanical test for the evaluation and control of the potential for IHE that may arise from various sources of hydrogen in a process.

SUMMARY

Specimens of fixed geometry, certified to have been heat treated to a hardness range of 50-52 HRC and have been certified to exhibit sensitivity to embrittlement from trace amounts of residual hydrogen in steel will be processed with actual parts.

The unstressed test specimen is processed in accordance with the plating or coating process being qualified. The specimen is then tested under incremental step load in four point bending to measure the threshold stress for IHE.

SIGNIFICANCE

The fundamental assumption is that if certified specimens with demonstrated sensitivity to IHE, processed with the fasteners have a threshold $\geq 75\%$ of the incremental step load notched bend fracture stress, $NFS(B)_{F1624}$, of an unprocessed specimen, then it is assumed that all fasteners processed the same way during the period will also pass any sustained load IHE test.

It is intended to be used as a qualification test for new or revised plating or coating processes and as a periodic inspection audit for the control of a plating or coating process.

INTERPRETATION

If the threshold in air of the specimen is $\geq 75\%$ $NFS(B)_{F1624}$, then the process is considered as to not produce sufficient hydrogen to induce time delayed IHE failures in the plated or coated fasteners. If the threshold in air of the specimen is $< 75\%$ $NFS(B)_{F1624}$, then the process is considered potentially embrittling.

Since embrittlement related to hydrogen content varies with hardness, actual fasteners made of low-strength steel might have more tolerance for residual hydrogen due to the process and might not need the rigorous requirement set forth in this standard for threshold. Therefore, adjustments in threshold requirements can be made once a correlation has been established or through an agreement between manufacturer and purchaser.

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No. 40

Fast Fact

Fastener System Design and Analysis

Coating system selection based upon KI_{scc} of an alloy steel determined using Rising Step Load (RSL™) accelerated testing protocol

PROBLEM

The stress corrosion cracking (SCC) threshold, KI_{scc}, for zinc electroplated 3/4"-16 Grade 8 alloy steel fasteners was found to be extremely low, (AFJ FF #38). To alleviate the observed in-service failures, it was recommended a new coating material be selected that would increase the threshold, KI_{scc}, under galvanic corrosion conditions.

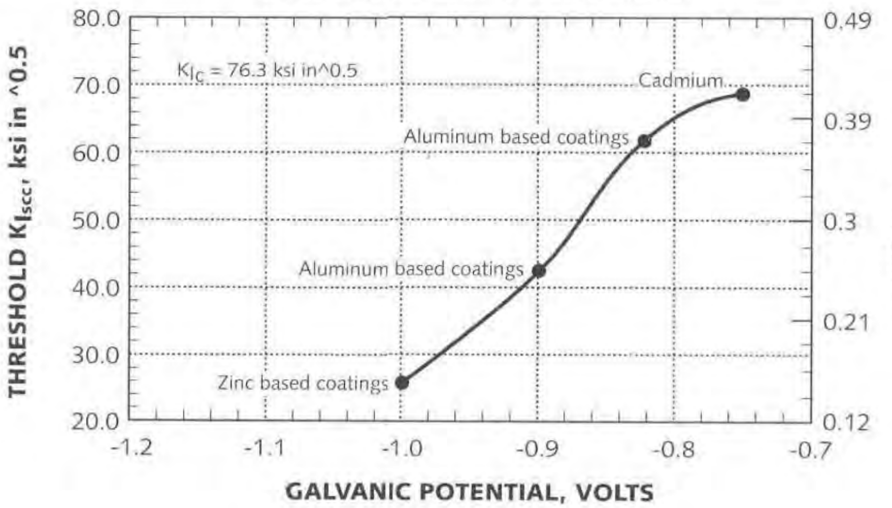
APPROACH

To evaluate the KI_{scc} of the steel, single edge-notched bend specimens, SEN(B) per ASTM E1290-93, were EDM machined from the 3/4"-16 fasteners. The specimens were fatigue pre-cracked to an a/W of approximately 0.5. Each specimen was tested at decreasing effective strain rates per ASTM 1624-95, using the Incremental Step Loading protocol at an applied electrochemical potential equal to that of the coating material in an aqueous 3.5% NaCl solution. The potentials used were -0.750V for cadmium, -0.825V and -0.900V for aluminum based coatings, and -1.0V for other zinc based coatings. The threshold is the point when the stress intensity became invariant with decreasing strain rate.

RESULTS

The alloy steel when tested at the electrochemical potential of the zinc based coatings was again observed to have an extremely low threshold of 26.0 ksi $\sqrt{in}$. The tests conducted at the two aluminum potentials of -0.9V and -0.825V resulted in threshold values of 42 ksi $\sqrt{in}$ and 63 ksi $\sqrt{in}$ respectively. For cadmium, the threshold was measured to be 69 ksi $\sqrt{in}$. Based upon a Damage Tolerance Index, DTI, analysis (*AFJ*, FF #9) for this fastener size and strength, an installation torque (applied tensile stress) equal to its yield strength could be applied without risk of hydrogen assisted service failure for both the cadmium and the aluminum coating at the more noble potential of -0.825V.

STRESS CORROSION CRACKING THRESHOLD, KI_{scc}, FOR ALLOY STEEL 3/4"-16 FASTENERS



CONCLUSION

The threshold for SCC, KI_{scc}, was measured for an alloy steel as a function of coating material using the RSL™ testing protocol. Based upon these results, either the cadmium or the noble aluminum coating should be selected for this alloy. If cadmium is selected, care must be taken to insure hydrogen absorbed during plating is removed. This Grade 8 alloy steel is sensitive to hydrogen from any source and will fail from internal hydrogen embrittlement (IHE) from processing.

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No. 41

Fast Fact

Hydrogen Assisted Stress Cracking in Fasteners

The Three Required Elements for Cracking

PROBLEM

With every failure of a steel fastener or part a familiar quote is always heard, "These parts have been manufactured the same way for the last 20 years without failure, why are they failing now?" The answer to this quandary is also always the same, "Something has changed in the manufacturing, installation, or service environment to result in these failures!"

QUESTION

What are the key elements that are required for hydrogen assisted stress cracking from internal or external hydrogen embrittlement (IHE or EHE) to occur in steel fasteners and assemblies?

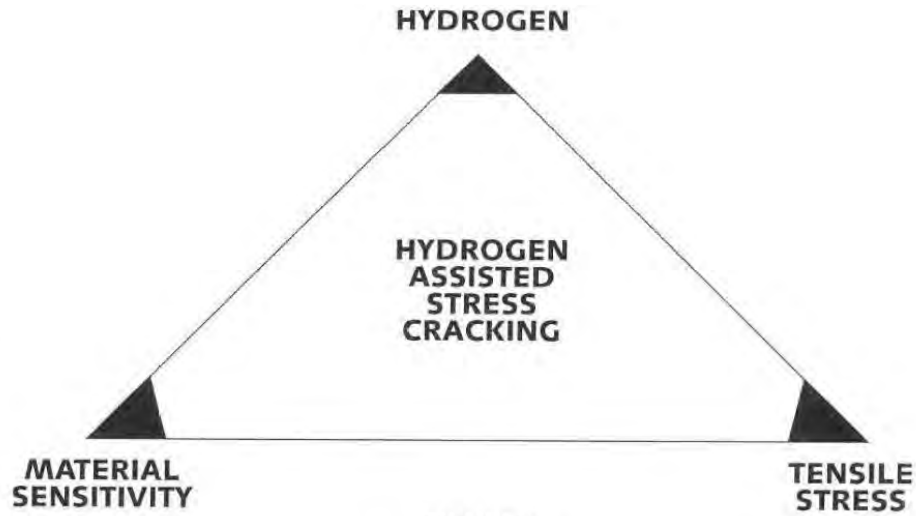

FIGURE 1
The Hydrogen Assisted Stress Cracking Triangle

DISCUSSION

To aid in this discussion, the Hydrogen Assisted Stress Cracking Triangle is presented in Figure 1. The three required elements for this form of cracking are: material sensitivity; hydrogen concentration; and tensile stress. If any element is missing, cracking will not occur.

Material Sensitivity: Many factors, some known, most not, determine a steel's sensitivity to hydrogen. These factors include: composition (base & tramp), melting practice, heat treatment, mechanical work, and surface treatments. Typically hardness is a good indicator for sensitivity, but improperly processed steels at HRC 32 and less can be extremely sensitive to hydrogen. A sensitive steel, with a low threshold, requires only a small amount of hydrogen and stress to fail and vice versa.

Stress: Tensile stresses are a combination of residual stresses and applied stresses, both from axial clamping loads and local bending from installation misalignment. Singularly or in combination, when the stress exceed the steel's threshold for a fixed concentration of hydrogen, cracking will occur.

Hydrogen: The source of hydrogen in a steel can be from processing (IHE), environmental exposure (EHE – galvanic corrosion), or a combination of both. Hydrogen concentration in a plated part is cumulative. A plated part with residual hydrogen may not fail at installation, but if exposed to hydrogen produced from a galvanic reaction, will fail when the critical concentration is exceeded.

CONCLUSION

To determine the root cause for hydrogen assisted failures and identify "What has changed," the RSL™ testing method is the only technique available for determining a material's sensitivity to cracking, the threshold stress required for cracking to occur, and the source(s) of the hydrogen responsible for the failure.

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No. 42

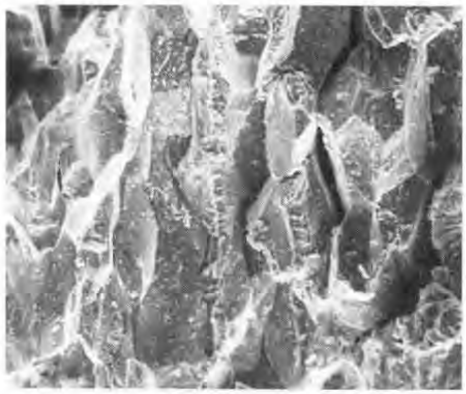
Fast Fact

Stainless Steel Washer Failure Analysis

Root cause of failure identified using RSL™ accelerated testing protocol

PROBLEM

Cold rolled and formed AISI 301 stainless steel Belleville washers (46 HRC) were experiencing time delayed failure in a steam/condensate environment up to one year after installation. The system design had these washers in contact with both bare and zinc plated steel surfaces. Scanning electron microscopic (SEM) examination of the fracture surfaces revealed the morphology to be primarily intergranular (IG) throughout.



(a)

(b)

FIGURE 1
Scanning Electron Micrographs @ 350X of (a) the In-Service Fracture Surface and (b) the EHE Fracture Surface, generated in the RSL™ test.

QUESTION

What was the root cause of the in-service failures?

RESULTS AND DISCUSSIONS

(1) It was speculated, because of the washer forming processes, that the washer's microstructure had transformed from an austenitic to a martensitic structure. This transformation was verified using a magnet, where martensite is magnetic and austenite is not.

(2) Since a martensitic structure at this hardness is very susceptible to hydrogen assisted cracking and the washers were in contact with zinc plated parts, environmental hydrogen embrittlement (EHE) from the galvanic couple was suspected. To verify this mechanism, (EHE) tests were conducted at the electrochemical potential of the plating material in contact with the washers to simulate the galvanic interaction between the two in service. All of the tests were conducted in compression per ASTM F1624 using the FDI Rising Step Load test system, RSL™ 1000S1-B. The RSL™ tests resulted in crack formation at loads of 25% to 35% less than the flattening load for the Belleville washers tested in air. SEM examination of the EHE test fracture surfaces revealed a duplication of the in-service fracture morphology. The only visible difference between the two was corrosion products on the in-service fracture surface, Figure 1, that formed after the washer cracked.

CONCLUSION

The root cause of the failure is the strain induced transformation of the stainless steel to untempered martensite resulting in susceptibility to EHE. The galvanic interaction between the washers and the zinc plated parts was the source of the environmentally produced hydrogen. The in-service failure fracture morphology was duplicated using RSL™ exemplar testing per ASTM F1624.

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No. 43

Fast Fact

ASTM F1940

Standard Test Method for Process Control Verification to Prevent IHE in Plated or Coated Fasteners

BACKGROUND

In a previous article (*American Fastener Journal* Fast Fact #39), an introduction was made to a process control method to eliminate lot testing by using the incremental step loading (ISL) technique per ASTM F1624. This process control method will allow the quantitative statistical evaluation of finishing processes for damage from internal hydrogen embrittlement (IHE).

CURRENT STATUS

Since then, the draft standard has been accepted as a standard test method by ASTM's committee F16 on Fasteners. The new standard has been given the numeric designation ASTM F1940. With the acceptance of this standard, the ASTM F16.93 task group has been charged with developing guidelines for its usage in the fastener/plating industry. As part of this task group's mission both seminars and symposia have been planned to promote awareness to the standard and guidelines for its application. The following is a listing of the currently planned events:

SUNDAY, APRIL 18, 1999

A seminar entitled **"HYDROGEN EMBRITTLEMENT: PROCESS CONTROL & FAILURE ANALYSTS of PLATED or COATED PARTS"** will be presented by Dr. Louis Raymond & Dr. J. Barton Boodey at the Prime F. Osborn III Convention Center, Jacksonville, FL.

This seminar has been coordinated with **ASTM F07.04 SUBCOMMITTEE SPRING MEETING** focusing on Hydrogen Embrittlement Standards; **ASTM F07.07 SUBCOMMITTEE MEETING** on Maintenance Chemicals and Cleaners Standards; the **"35th Aerospace/Airline Plating & Metal Finishing Forum & Exposition"** sponsored and organized by AESF; and the **SAE AMS Committee "J" Spring Meeting on Aircraft Maintenance Chemicals and Materials.**

SUNDAY, MAY 16, 1999

A seminar entitled **"THE USE OF ASTM STANDARDS TO CONTROL HYDROGEN EMBRITTLEMENT AND STRESS CORROSION CRACKING IN FASTENERS"** will be presented by Dr. Louis Raymond & Dr. J. Barton Boodey at the Westin Hotel in Seattle, WA.

This seminar has been coordinated with **ASTM F16 COMMITTEE SPRING MEETING** focusing on Fasteners; **ASTM E28 COMMITTEE SPRING MEETING** focusing on Mechanical Testing; and the **SECOND SYMPOSIUM ON STRUCTURAL INTEGRITY OF FASTENERS,** sponsored by ASTM E8 on Fatigue and Fracture.

TUESDAY, JUNE 22, 1999

A series of presentations on **ASTM F1940 IN THE QUALITY IN METAL FINISHING SESSION OF AESF SUR/FIN '99** to be held at the Cincinnati Convention Center, Cincinnati, OH.

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No. 44

Fast Fact

Threshold Stress

Fracture Morphology Relationship in M10-1.5 Socket Head Cap Screws

BACKGROUND

Hydrogen induced crack growth occurs when a critical combination of applied/residual tensile stress and hydrogen concentration are attained. The greater the hydrogen concentration, the lower the critical stress or the lower the hydrogen concentration, the higher the critical stress. From either perspective, this definition puts a new meaning on the concept of threshold. What has been shown by the Rising Step Load (RSL™) test method is that physically, the threshold is the instant that the critical combination of stress and hydrogen concentration are attained at a sufficiently slow loading rate. Therefore, the threshold serves as a measurement of residual hydrogen and this stress can be increased or decreased by varying the concentration of hydrogen in the part.

QUESTION

By varying hydrogen concentration in a fastener, will the fracture morphology change as a function of threshold stress?

ANSWER

Yes, the fracture morphology will change dramatically from a brittle intergranular IG surface to a mixture of IG and ductile dimpled features by decreasing the hydrogen concentration. To demonstrate this effect, two (2) Grade 12.9 socket head cap screws were electrochemically charged with hydrogen during separate RSL™ tests at different potentials. The electrochemical potentials produce two different concentrations of hydrogen in the fasteners. The resulting fracture morphologies are shown in Figures 1 and 2. Figure 1 shows the fracture morphology for the fastener with the higher concentration of hydrogen that failed at a threshold stress of 140 ksi. Figure 2 illustrates how increasing the threshold to 260 ksi by reducing the amount of hydrogen in the fastener produces a mixed IG and dimple fracture morphology.

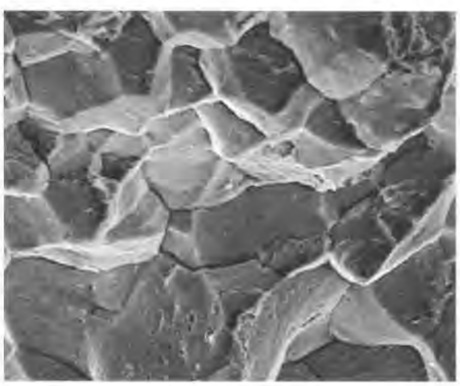

FIGURE 1.
High (H)/140 ksi threshold
100% IG

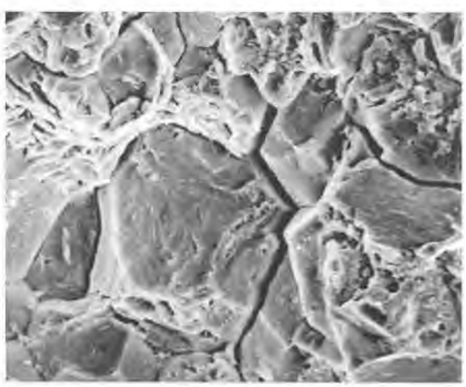

FIGURE 2.
Low (H)/260 ksi threshold
Mixed IG & Dimple

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Fast Fact

What is RSL™ Testing All About?

The Quantitative Measurement of Threshold

BACKGROUND

In Fast Fact #44, see *American Fastener Journal* May/June 1999, it was clearly shown that by changing the amount of hydrogen in a fastener not only did the fracture morphology of the failure change, but so did the stress required for hydrogen assisted cracking to occur. This critical stress for cracking is defined as the threshold. Physically, the threshold is the instant that the critical combination of stress and hydrogen concentration are attained at a sufficiently slow loading rate. Below the threshold stress no time dependent cracking will occur (infinite life), but above this stress, subcritical cracking leading to time delay fracture will occur (finite life), Figure 1.

QUESTION

What are some of the different ways that threshold can be quantitatively measured for standard test specimens and fasteners using the RSL™ testing method?

INTERNAL HYDROGEN EMBRITTLEMENT (IHE) EVALUATION USING STANDARD SPECIMENS

Certified test specimens, per ASTM F1940, are used to evaluate finishing processes for damage from hydrogen or to isolate the effects of a specific process in generating or removing hydrogen. The threshold stress determined in air serves as a quantitative measure of residual hydrogen in the specimen. A higher threshold corresponds to less residual hydrogen in fasteners processed the same way, see *American Fastener Journal* Fast Fact #37.

INTERNAL HYDROGEN EMBRITTLEMENT (IHE) EVALUATION USING PRODUCT

Actual fasteners are tested in air after processing to quantify their threshold stress for internal hydrogen embrittlement. The threshold stress determined again serves as a quantitative measure of residual hydrogen in the fastener. In addition, the threshold is used to determine the maximum service stress in air for this lot of fasteners. The applied service stress must be below the measured threshold to ensure infinite life.

HYDROGEN ASSISTED CRACKING THRESHOLD EVALUATION

Fasteners are taken prior to processing or service and are evaluated for their threshold. Testing is conducted in an aqueous solution under an imposed electrochemical potential to measure their threshold stress for hydrogen assisted cracking. The threshold results predict the response of the fastener to hydrogen introduced during a finish processing, service exposure under galvanic corrosion conditions, or a combination of both. Again, the applied service stress must be below the measured threshold to ensure infinite life. ■

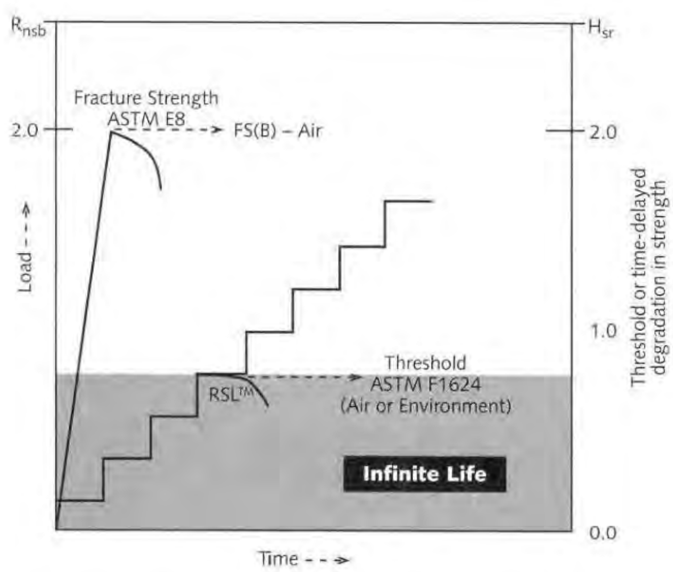

FIGURE 1.
The new short term or accelerated method to measure the threshold is the Rising Step Load (RSL)™ technique per ASTM F1624.

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Fast Fact

No. 46

Quantitative Measurement of Threshold K_{Iscc} as a Function of Hydrogen Concentration

BACKGROUND

In Fast Fact #44, see AFJ May/June 1999, an increase in hydrogen concentration in a fastener was shown to reduce the stress required for hydrogen-assisted cracking to occur and to change the corresponding fracture morphology. This critical stress for cracking was defined as the threshold and is the instant that the critical combination of stress and hydrogen concentration is attained at a sufficiently slow loading rate. To demonstrate threshold variation and crack arrestment with changes in hydrogen concentration, a rising step load (RSL) test was conducted on a pre-cracked single-edge notched bend, SEN(B), bar at two hydrogen-charging potentials. These potentials were used to produce significantly different hydrogen concentrations at the crack tip of the specimen.

RESULTS

Figure 1 shows the test and how it was broken into five (5) specific regions. In region A, the specimen was polarized to -1.2V and then step loaded until crack propagation began as shown in region B. The threshold stress intensity for cracking, $K_{Iscc,}$ at this hydrogen concentration (-1.2V) was 14.3 MPa $\sqrt{m}$. After an increment of crack growth occurred in region B, the potential was changed to -0.8V. This decrease in hydrogen charging potential reduced the amount of hydrogen at the crack tip and the cracking was observed to arrest almost immediately. The stress intensity was now below the threshold for cracking at this new hydrogen concentration. The specimen was then held for 18 hours at the stress intensity of 14.3 MPa $\sqrt{m}$ with no further crack advance. In region D, the specimen was further step loaded until crack propagation began again, region E. The threshold stress intensity for cracking, $K_{Iscc,}$ at this hydrogen concentration (-0.8V) was found to be much higher at 26.4 MPa $\sqrt{m}$. This is why no cracking occurred in region C at a stress intensity of 14.3 MPa $\sqrt{m}$.

CONCLUSION

From this experiment it has been clearly shown that the threshold stress intensity for cracking, $K_{Iscc,}$ is a direct function of the hydrogen concentration at the crack tip. By decreasing the amount of hydrogen in a material/fastener, the threshold for cracking can be dramatically increased.

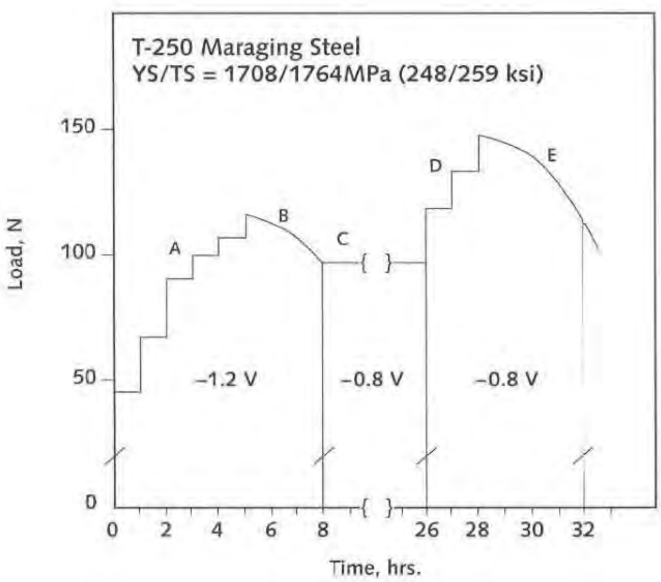

FIGURE 1.
Loading schedule for the crack arrestment investigation and threshold measurements of T-250 Maraging Steel.

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No. 47

Fast Fact

An Application Guideline for ASTM F1940 - Process Control Verification to Prevent IHE in Plated or Coated Fasteners

BACKGROUND

In previous articles (AFJ FF#39 and FF#43) an introduction to this alternate process control method using the incremental step loading (ISL) technique per ASTM F1624 was made. This process control method will allow the quantitative statistical evaluation of finishing processes for damage from internal hydrogen embrittlement (IHE). The development of guidelines for its application in the fastener plating community was deemed paramount. An application guideline has been incorporated into F1940 and has received acceptance by ASTM Committee F16 on Fasteners. Highlights of this Annex are contained herein.

SCOPE

This application guideline is targeted to the general fastener plating industry and was designed to be used as a template for the application of ASTM F1940. It does not specify any mandatory requirements, however it should serve as a checklist for anyone who wishes to use the (ISL) test method for process verification to prevent IHE in plated or coated fasteners.

TESTING CRITERIA

Testing of an individual plating process shall be conducted at the highest specified pickling acid concentration *and the longest pickling duration* for a given line. In addition, testing shall be conducted at the highest operational current density for an electroplating cell.

A minimum of three square bar specimens shall be placed in a single processing unit. A processing unit can be a barrel, a rack, a drum or a basket depending on the nature of the process being tested. The average of three results within a unit shall represent a single data point for statistical evaluation. Variation within each unit must be within ±10% of the measured average threshold for the group of three specimens. This is a benchmark for the validity of the results within a single unit.

Variation of results from one unit test to the next must be within ±10% of the measured average threshold for the population of units, in addition to meeting the minimum threshold requirement, in order to meet process control objectives.

SAMPLING SCHEDULE

Stage 1: Test three specimens in one unit daily for a minimum of one operational week. If variation of the test results remains within the acceptable range, go to stage 2. If not, testing must continue in order to determine and eliminate the cause of variation and IHE.

Stage 2: Test three specimens in one unit weekly for a minimum of four weeks. If variation of the test results remains within the acceptable range, go to stage 3. If not, testing must continue in order to determine and eliminate the cause of variation and IHE. It might be necessary to return to stage 1.

Stage 3: Test three specimens in one unit monthly for as long as process stability has been established by achieving and maintaining acceptable variation of results. In case of unacceptable variation, testing must continue in order to determine and eliminate the cause of variation and IHE. It might be necessary to return to stage 1 or stage 2.

It is possible to further reduce the testing frequency through the establishment of operating limits for the process control variables. In order for this to be accomplished multilevel experimentation must be conducted in order to determine the impact of each variable on process performance.

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No. 48

Fast Fact

Failure Analysis: Liquid Metal Embrittlement (LME) of Steels
Another Intergranular Cracking Mechanism

BACKGROUND

Failure by Liquid Metal Embrittlement (LME) of steel is distinct and more easily recognized than that brought about by stress corrosion cracking, hydrogen embrittlement, or temper embrittlement. The distinctive features of LME include: a significant degradation in mechanical properties; high velocity crack propagation resulting in fast fracture; a fracture morphology that is typically intergranular (IG) with little crack branching; and the fracture surface is usually wetted with the liquid metal. The fracture surface need not be coated with the liquid, but the thin layers of metal responsible for the degradation are easily detected using energy dispersive x-ray analysis in the scanning electron microscope. LME causes cracking and subsequent failure in stressed parts. The threshold stress required for cracking typically decreases with increasing temperature and strength of the base steel component. Cracking occurs in a brittle manner at extremely low stresses/strains.

EMBRITTLEMENT COUPLES

LME of steels is typically associated with cadmium but has also been reported for a variety of other materials. These materials include: copper, antimony, zinc, lead, tin, and indium. Each of these materials has been found to form brittle intergranular cracking in steels during processing or service exposure where elevated temperatures and stresses are combined. The materials can be in the form of alloys or singular elements.

EXAMPLE OF AN IN-SERVICE LME FAILURE

The failure of a hardened (44 HRC to 48 HRC) AISI 1050 conical washer on a KEPS nut was originally attributed to environmentally induced hydrogen embrittlement since the failure mode was intergranular and did not occur until after the assembly was exposed to the service environment. Further examination of the fracture surface revealed the presence of the coating material on the intergranular facets. This observation led to the speculation that the crack had formed as a result of the quench and was filled in by the tin-zinc (Sn-Zn) plating process thus explaining the coating material on the fracture surface. The service application was then determined to be a connection point between the exhaust manifold and the catalytic converter. The temperatures in this region are known to exceed 500°F. With the combination of an elevated service temperature, low melting point coating materials, a relatively high stress in the washer, and the washer being a high strength steel, the potential for LME was evident. Subsequent testing of the washers under stress at an elevated temperature resulted in liquid metal pools on the surface of the washer and the formation of LME cracks.

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No. 49

Fast Fact

Failure Analysis: Effect of Plating Thickness on the Potential for Hydrogen Embrittlement

BACKGROUND

To reduce the potential for internal hydrogen embrittlement (IHE) of plated fasteners, post-process baking of these parts is performed. Typically these thermal treatments are conducted within 1 hour of plating at temperatures of 375°F to 400°F for 4 to 23 hours. The residual hydrogen concentration in these parts is reduced by diffusing the hydrogen through the coating material. Interstitial diffusion is based upon a time-temperature relationship and the following general equation relates diffusion distance, x, to time, t, and the diffusion coefficient of the hydrogen atoms in the coating material, D.

$$\left(\frac{x^2}{D}\right) = t \qquad \textbf{equation 1}$$

From this equation it can be seen that increasing the distance for the hydrogen to diffuse from 0.1 mil to 0.3 mil would require a nine (9) fold increase in time. Clearly, coating thickness becomes the primary controlling factor for reducing the potential for IHE in a fixed time and temperature post-plating bake.

EXAMPLE OF COATING THICKNESS EFFECTS

Grade 10.9 M5-0.8x151mm fasteners were being plated in a zinc-chloride barrel process followed immediately by a five (5) hour 400°F bake. In-service failures under the heads of these fasteners were being observed in a high stress (bending) application. To identify the mechanism responsible for these failures, RSL™ testing was conducted on both the threaded section of the fastener in bending and the entire fastener in tension with a 6° wedge under the head. These RSL™ test methods allowed each region of the fastener to be evaluated for its potential for IHE at local stresses at or above the tensile strength of the material.

The bend RSL™ testing revealed that there was no potential for IHE in the threaded section of the fastener up to local stresses of two (2) times the tensile strength of the material. The residual hydrogen level in this part of the fastener was low since the threshold stress for cracking was not exceeded. The tensile RSL™ testing resulted in IHE failures at stresses just above the tensile strength of the material. This reduction in threshold stress for IHE corresponded directly to an increased amount of residual hydrogen beneath the head of the fastener.

X-ray examination of the fasteners revealed that the threaded and head fillet regions had coating thicknesses of 0.2 to 0.3 mils and 0.5 to 0.7 mils, respectively, produced by current variations along the length of the fastener. These coating thickness variations resulted in differing residual hydrogen concentrations after the bake with the thicker coating being responsible for the in-service failures. ■

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No. 50

Fast Fact

Hydrogen Assisted Cracking Threshold

The Role of Hydrogen Concentration

BACKGROUND

In recent Fast Facts, #44 (May/June '99), #45 (July/Aug '99), and #46 (Nov/Dec '99), it has been shown that a threshold stress for hydrogen assisted cracking, HAC, exists and that no time dependent cracking will occur (infinite life) below this stress, but above this threshold stress, subcritical cracking leading to time delay fracture will occur (finite life). This statement holds true for fixed combinations of material sensitivity and hydrogen concentration. If either of these conditions is changed, the threshold stress for HAC will also change. This concept is the basis for all hydrogen embrittlement relief baking treatments conducted after the plating or coating of fasteners. During these baking treatments, the residual hydrogen concentration is reduced, thereby increasing the threshold stress required for HAC.

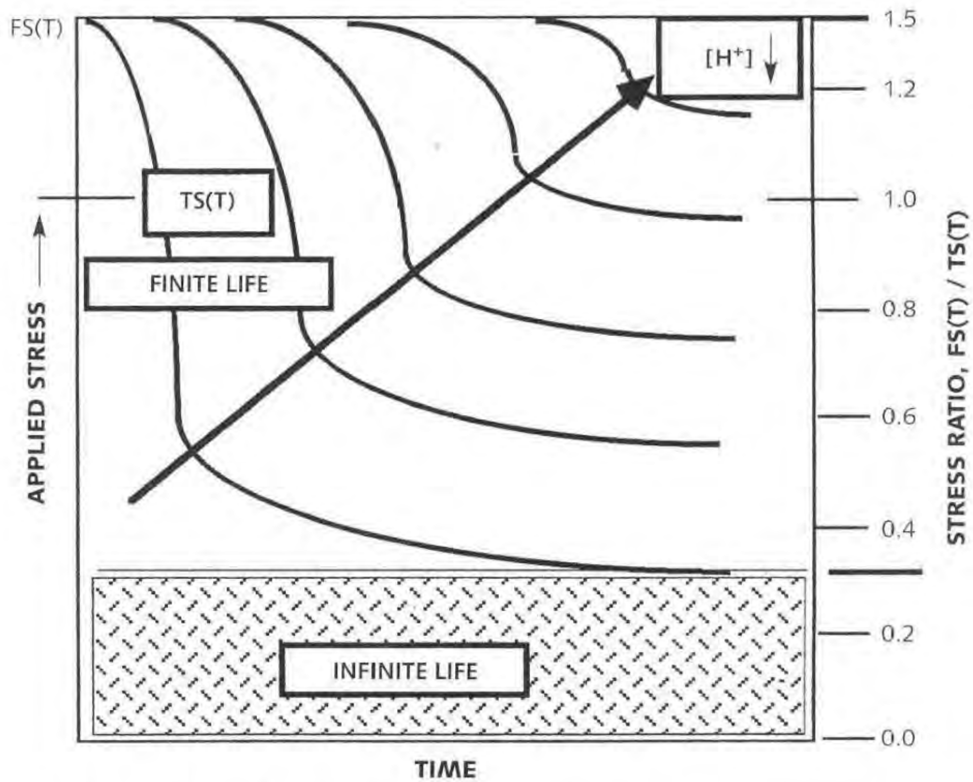

FIGURE 1. The effect of hydrogen concentration [H+] on the time to failure curves of steel showing that the threshold stress increases as the hydrogen concentration decreases.

EXAMPLE OF THRESHOLD CHANGE AS A FUNCTION OF HYDROGEN CONCENTRATION

Figure 1 is a schematic representation of threshold stress change with decreasing hydrogen concentration. From this figure a minimum threshold is observed at a stress ratio of Tensile Strength in Tension, TS(T), to Fracture Strength in Tension, FS(T), of approximately 0.3. As the residual hydrogen concentration is reduced, moving from the lower left to the upper right hand corner of the figure, the applied stress at threshold increases. In the limit, the hydrogen concentration reaches a level where it has no adverse effects. As long as the part does not have additional hydrogen introduced from its service environment, it can now be stressed to a high level without HAC occurring. If hydrogen is generated in-situ from an environmental source, such as a galvanic couple between the base steel and the coating material, the threshold required for HAC will decrease. The reduction in threshold will be directly related to the new residual hydrogen concentration in the part. ■

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Fast Fact

Internal Hydrogen Embrittlement
The Pitfalls of Using a Soak Pickle on High-Strength Parts

BACKGROUND

In the automotive industry, many high-strength steel parts, including fasteners, conical washers, flat washers and self tapping screws, with hardnesses above 39 HRC are zinc electroplated. Since the majority of the zinc plating lines exhibit efficiencies greater than 90%, the question of where the residual hydrogen that resulted in internal hydrogen embrittlement (IHE) of these parts is inevitably asked when a failure occurs. Recent RSL™ testing of zinc-chloride plating lines has conclusively shown that the acid descaling step is the primary source of residual hydrogen in these plated parts. Figure 1 illustrates the quantitative difference in IHE for standard square bar specimens, tested per ASTM F1940-99, that were zinc electroplated with and without a Muriatic acid descaling step. The specimens plated without the soak pickle process failed at 225 pounds, 90% of the fracture strength. The RSL™ tests showed the zinc plating by itself only degraded the strength of these specimens by 10%. The specimens tested after plating with the soak pickle failed at 113 pounds, or 45% of their fracture strength. The soak pickle process reduced the fracture strength of the standard test bars by 55%. A similar loss would be expected for high-strength hydrogen-sensitive steel parts.

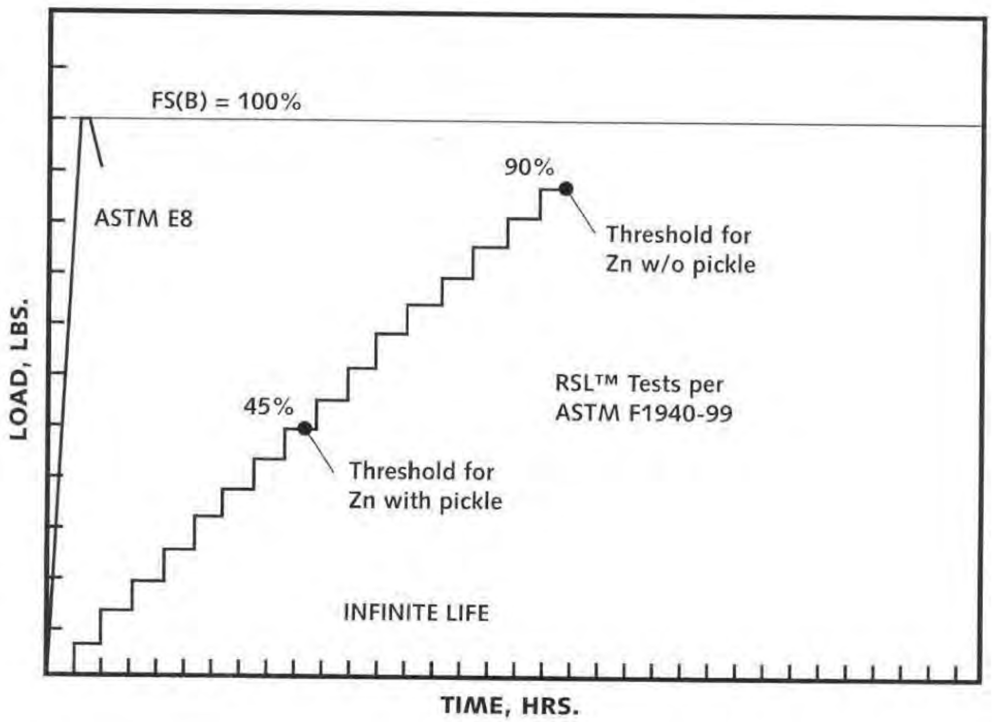

FIGURE 1. The effect of a soak pickle on the RSL threshold for zinc plated ASTM F1940 test specimens.

ALTERNATIVES FOR HIGH-STRENGTH STEEL PARTS

In other industries, the use of a soak pickle for parts with hardnesses of 40 HRC and above is prohibited without Material & Processes group approval. Alternative methods such as grit and vapor blasting, anodic or periodic reverse electrocleaning and pickling, alkaline descaling, or the use of less aggressive acids such as citric and glycolic are known to lessen or eliminate hydrogen absorption into these parts. In discussions with various platers, the number one concern for using these alternate descaling/surface activation methods is cost. The cost of using Muriatic acid is less compared with these alternate methods in terms of expendable materials, process time, and up front capital costs. The bottom line question then becomes: "Is the reduced cost of using a Muriatic soak pickle worth the risk of an IHE failure that could result in a multi-million dollar recall debit?". The appropriate decision can only be made based upon the quantitative measurement of the IHE potential for a specific plating line. The results of RSL™ testing conducted per ASTM F1940 will provide this quantitative data that is required to make this decision. ■

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Fast Fact
Internal Hydrogen Embrittlement
How long can I wait before
I bake an electroplated part?

BACKGROUND fracture strength, test coupons

The previous Fast Fact #51 focused on machined from the part must be tested miniput into mizing _ an electroplated the amount part. of hydrogenIt is easi- they and examined have been for fractured. IG with the If no SEM micro-after er to *avoid* introducing hydrogen into cracks are present, the parts can still be the plated part as compared to remov- salvaged, ing it from the plated part, which is accomplished by baking. Now the issue not only becomes how long to bake but **EXAMPLE**

how soon after electroplating should Electroplated steel projectile bodies at the part be put into the baking furnace? 46-48 HRC were in storage for two The conventionally accepted answer is years when it was realized that they had that the delay time between plating and not been baked. The parts were suspect baking should not exceed one hour. because they were exposed to an acid In theory, the time delay before a crack bath during the plating process. forms is a function of the amount of the Test coupons were cut from the projecresidual stress and the amount of resid- tile bodies. ASTM E8 loading rate fracual hydrogen in the part. The higher the ture strength tests were conducted and residual stress and the higher the then examined in the SEM. No IG was amount of residual hydrogen, the short- present; therefore, no microcracks were er the time required for microcracks to present due to the two year delay time. form. If there is no residual stress, the The projectile bodies could still be salpart can be stored indefinitely prior to *vaged.* If microcracks had been present baking without any microcracks form- due to the long delay time between ing. While in storage, the thermo- plate and bake, the fracture surface dynamic driving force is to remove would have shown IG cracks on the hydrogen from the part. fracture surface.

If residual stresses and hydrogen do To determine if any residual hydrogen exist in the part, intergranular (IG) still existed in the projectile bodies microcracks could possibly begin to Rising Step Load (RSL ™) tests pe form with time with no externally ASTM F1624 were conducted in air on applied stress. To determine if no micro- test coupons cut from the parts. cracks were formed in parts stored an Residual, detrimental hydrogen was excessive amount of time before bak- shown to exist since the fracture stress

ing, a fracture strength test should be was below that measured with the iniperformed prior to baking. The pres- tial fracture test at the ASTM E8 loading

ence of microcracks can be detected by rate and IG cracks were present as veria significant reduction in the fracture fied with the SEM. strength of ASTM F519 test coupons@ 51-53 HRC. At a lower hardness, even if no degradation is observed in the

CONCLUSION

The conventionally accepted delay time between plating and baking of less than one hour, is a conservative estimate based on a worst case assumption that some residual stresses do exist in the electroplated parts. The only way to verify that the parts have been baked soon enough to avoid hydrogen embrittlement is to test the parts per ASTM E8 and ASTM F1624 as illustrated.

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The remaining parts were then baked and RSL tests were again conducted in air. The degradation in fracture strength was no longer observed; and therefore, it was concluded that all of the residual hydrogen had been removed and the parts were ready for service.

No. 53

Fast Fact

FIRST IN A SERIES...

Hydrogen Embrittlement Testing

Time-to-Failure/Sustained Load Test (TTF/SLT) What Does It Tell Us About Product Safety?

ISSUE:

The sustained load test, often referred to as the stress durability or hydrogen embrittlement relief test, is conducted in many forms, from a fastener plate test to a notched round bar tensile test under sustained or constant load. It is used to test actual hardware in ASTM F606, NASM1312-5 or with specimens in ASTM F519 or in Federal plating specifications. The time used to establish the pass/fail conditions varies from 24h to 200h to sometimes as much as 6 months for NASA. The question becomes: "*Why does the type and time of the test vary so much?...* and *What does it tell us about product safety?.... Do all the tests give us the same information?...*

The answer to these questions is long and involved and therefore, the answers will be presented in a Series of Fast Facts, one step at a time.

The purpose of any of the tests is to prevent a time-delayed, hydrogen embrittlement service failure of a structural component, which in our case is a fastener, under a sustained applied and/or residual stress. They should serve as a proof test that guarantees service life.

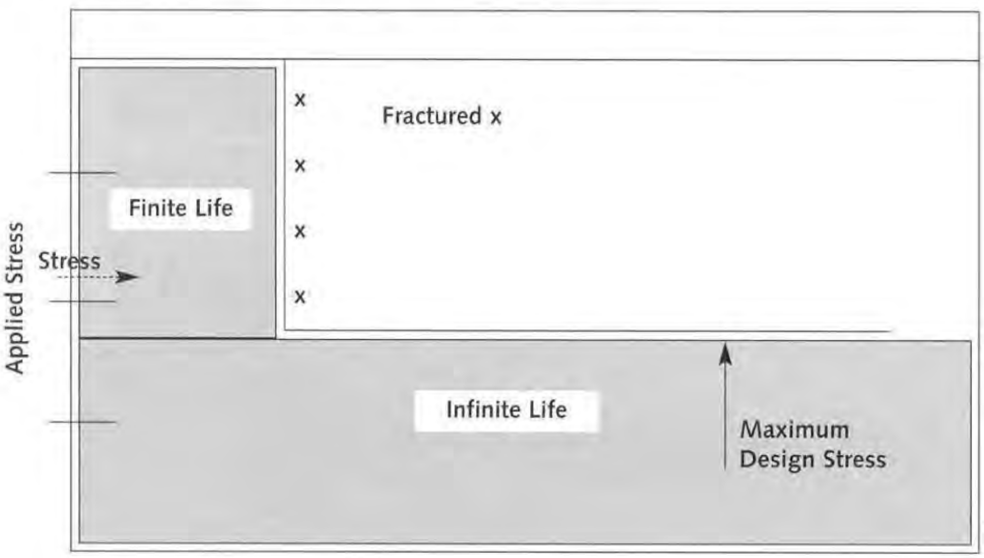

FIGURE 1. Schematic delineating zones of a TTF/SLT Curve

The Threshold is the Stress below which NO time dependent or delayed fracture occurs (region of Infinite Life) and, above which, time dependent or time delayed fracture does occur (region of Finite Life). The design objective is that the maximum anticipated sustained residual design or service stress is less than the threshold stress for the onset of subcritical crack growth. The existence of a threshold is verified with sustained load tests conducted for times far in excess of 10,000 hours (over 1year).

HOW IS THE OBJECTIVE MET?... KEYWORD: THRESHOLD

The concept of threshold is illustrated in Figure 1 that delineates different zones that relate to the life of a fastener under a sustained load test (SLT). The stress is plotted on the vertical axis. The time-to-failure (TTF), which is the measure of life, is plotted on the horizontal axis. ■

info

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No. 54

Fast Fact

SECOND IN A SERIES...

Hydrogen Embrittlement Testing

Time-to-Failure/Sustained Load Test (TTF/SLT) What Does It Tell Us About Product Safety?

Fast Fact #53 identified the different failure zones of a TTF/SLT curve and established the **"threshold"** as the line delineating the "Finite/Infinite Life" zones in hydrogen embrittlement. To answer the question: *"Why does the type and time of the test vary so much?* ...and *"Do all the tests give us the same information?*...we must understand how the threshold is related to (1) the residual hydrogen, (2) the hardness, and (3) the geometry of the part or specimen.

How is this accomplished? The basis for all TTF/SLT is the work of Troiano, wherein he demonstrates the phenomenon of time-delayed failure in notched round bar steel specimens at about 52 HRC that were charged with different amounts of hydrogen. The results of his classic experiment are schematically shown in Figure 1.

The test specimens were loaded in air to various percentages of the notched fracture strength, NFS(T) measured at ASTM E8 loading rates. The load remained constant until the specimen broke (fractured, designated by an "x" in Figure 1) and the time-to-fracture (TTF) recorded—or, continued to run (designated by •→ in the figure) until a threshold stress or stress below which no fracture occurred was established.

Even using charged specimens, the run-out times were exceptionally long, often exceeding 1,000h (>1mo) or as much as 10,000h (>1yr).

The conclusions drawn from his work are that both the **Finite Life** and **Threshold** (see Figure 1 in Fast Fact #53) increase as the amount of residual hydrogen decreases for Type AISI 4340 steel at a given hardness level, and the lower the hydrogen concentration, the longer the time required to conduct the test.

Another issue to be noted from Troiano's test results is that the test was conducted in pure axial tension where NFS(T) of the 1/4-inch notched round bar of AISI 4340 steel at about 52 HRC had a fracture strength based on the net cross-sectional area that is about 1.5 times the ASTM E8 smooth bar tensile strength. At an applied load of 75% of the notched tensile fracture strength, the resultant applied net stress is then equal to about the ASTM E8 smooth bar tensile strength.

In the next Fast Fact, #55, incorporating these test results into a quality assurance test for product safety for parts of different hardness and geometry will be addressed. ■

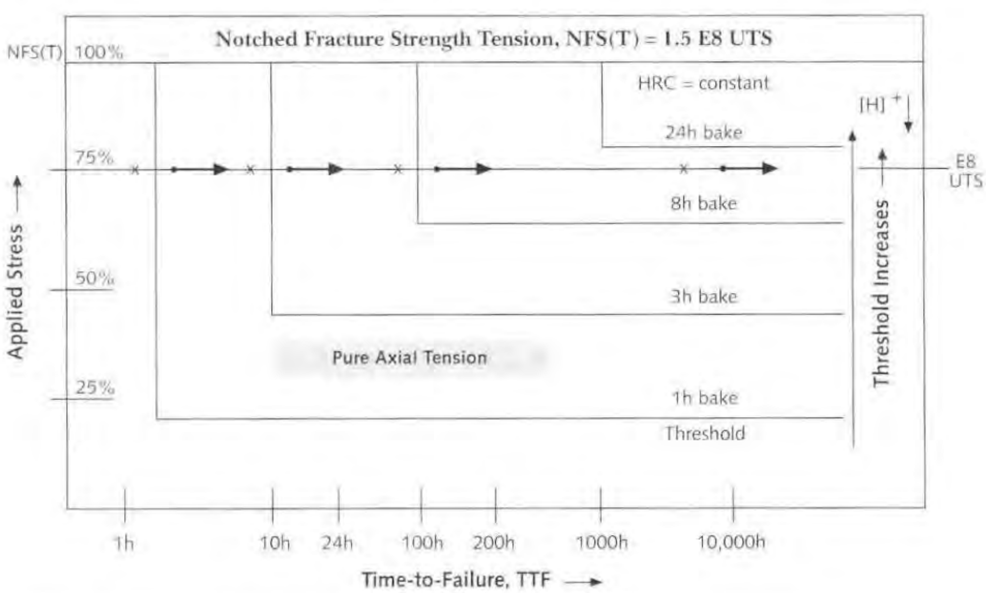

FIGURE 1. Effect of Residual Hydrogen Levels on TF/SLT Curve, © 2001 LRA

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No. 55

Fast Fact

THIRD IN A SERIES...

Hydrogen Embrittlement Testing

Time-to-Failure/Sustained Load Test (TTF/SLT) What Does It Tell Us About Product Safety?

To answer the question: *Why does the type and time of a hydrogen embrittlement proof test vary so much?* and *Do all of the proof tests give us the same information?* We must understand the relationship of the **"threshold"** to (1) the residual hydrogen, (2) the hardness, and (3) the geometry of the part or specimen.

Fast Fact #53 identified the different failure zones of a TTF/SLT curve and established the threshold as the line delineating the "Finite/Infinite Life" zones in hydrogen embrittlement. Fast Fact #54 illustrated that for a given hardness, the threshold increases as the amount of residual hydrogen in the steel decreases. Correspondingly, The TTF in the zone of Finite Life also increases.

This Fast Fact #55 addresses the logic behind incorporating hydrogen embrittlement proof test results into assuring product safety for parts of different hardness and geometry.

The purpose of any hydrogen embrittlement **proof test** is to prevent a time delayed service failure. In theory, this is accomplished by exposing the hardware to a higher stress than it will see in service and for a time longer than it will see in service. Obviously, holding it for a longer time than is will see in service is often impractical and becomes the limiting factor to the proof test.

There are two approaches to resolving these problems. One faction believes in testing the actual hardware as in NASM13125 and the IFI plate test; whereas, the other uses a "worst case" approach with specimens as in ASTM F519.

What are the differences in the approaches regarding the quality assurance of the product?

The "worst case" approach of ASTM F519 is based on the premise that if the specimen, which is harder than the part and therefore more sensitive to hydrogen embrittlement than the part, passes the test, then all of the parts should pass the test.

What is the significance of "passing the ASTM F519 test?"

In ASTM F519, the specimens are loaded to 75% of their notched fracture strength. The Pass/Fail criterion is that no fracture occurs in 200 hrs or TTF $\geq$ 200 hrs. (Pass); and TTF < 200 hrs. (Fail). As noted in Fast Fact #54, the longer the test time, the more closely the threshold of 75% of the notched fracture strength is approached, which means that the specimen has less hydrogen than one with a lower threshold, say 50%; i.e., (1) the longer the sustained load test time, the more closely the specimen approaches a threshold equal to the applied stress and (2) the higher the threshold, the less hydrogen in the part.

For a given hydrogen concentration, the attached Figure illustrates that using the "worst case" condition of ASTM F519 and maintaining a sustained load on Type 4340 steel at 54 HRC for 200 hrs, approaches the threshold of 75% the notched fracture strength, which was shown to be about 100% of the smooth bar tensile strength.

As illustrated in the Figure, for product at a lower hardness, the threshold for the same amount of hydrogen would be higher than 75% and would require much longer times than 200 hrs to produce fracture. Therefore, to measure the actual threshold for the lower hardness specimen would take a significantly longer time than at the higher hardness. ■

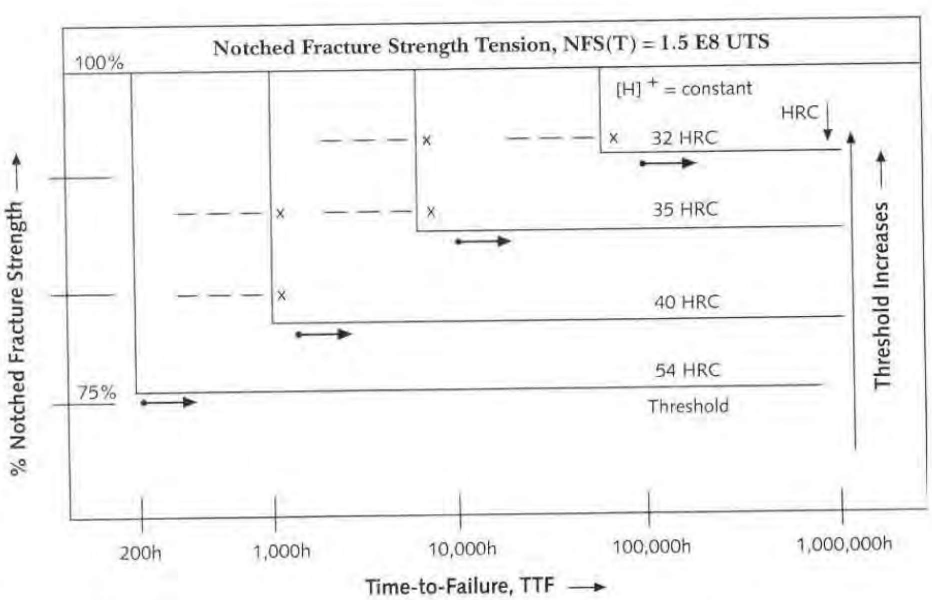

FIGURE 1. Effect of Hardness on the TTF/SLT Curve, © 2001 LRA/RSL™

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Fast Fact

FOURTH IN A SERIES...

Hydrogen Embrittlement Testing

Time-to-Failure/Sustained Load Test (TTF/SLT) What Does It Tell Us About Product Safety?

To answer the question: *Why does the type and time of a Hydrogen Embrittlement proof test vary so much?* and *Do all of the proof tests give us the same information?* we must understand the relationship of the **"threshold"** to (1) the residual hydrogen, (2) the hardness, and (3) the geometry of the part or specimen.

This Fast Fact #56 addresses testing actual product or specimens at the hardness of the product.

At illustrated in Figure 1, at the same threshold stress of 75% of the notched fracture strength, the tolerance for an amount of residual hydrogen in a steel increases, as the hardness of the steel decreases.

As time-to-fracture increases, the threshold approaches the applied stress. Testing lower-hardness steel product requires longer times to establish that the threshold is near the proof load.

Short time sustained load tests for low-hardness product like fasteners at 35-38 HRC, increases the probability of service failures even though the proof test is successfully passed by both re-torqueing or non-destructive inspection, because oftentimes the time is not sufficient for a crack to nucleate in the finite life region, which again takes a longer time-for-crack nucleation than a higher hardness steel with the same amount of residual hydrogen, as graphically illustrated in Figure 1 by the symbol, — — — x.

Testing actual product of lower hardness to the same criterion of 75% NFS for 24h instead of 200h is in the exact opposite direction based on technical evidence — oftentimes it does not prove anything. It is technically incorrect. It can be totally insensitive to the existence of residual hydrogen that could cause failure of the product. To make the test meaningful, run out times should be increased. If 200h is adequate for a 52-54 HRC steel, then longer times are required for a lower hardness steel. NASA uses NASM 1312-5 to run out times of six months for fasteners at 50-52 HRC.

The most popular IFI plate test on actual product at a lower hardness is to load the fastener in a plate at 80% of the limiting torque. Wait for 24 hrs, and then attempt to retorque to the original value. There are several problems with this approach: (1) there is no guarantee that in the actual installation the tension produced by a lower torque does not result in a higher stress in the fastener than in the plate test, and (2) 24 hrs might not be adequate to initiate a crack, especially in the lower hardness steel fastener.

It should be noted that much longer times than 200 hrs would be required to produce a fracture in the lower hardness specimens.

To overcome the issue of stress, wedges are often used to add a bending stress to the axial tensile stress. At 52-54 HRC, the test time in ASTM F519 allowed for a crack to form is specified as ≥200 hrs, so 24 hrs might not be adequate to nucleate a crack in lower hardness fastener in a plate test. For this reason, parts often pass a 24 hr plate test but fail in service. ■

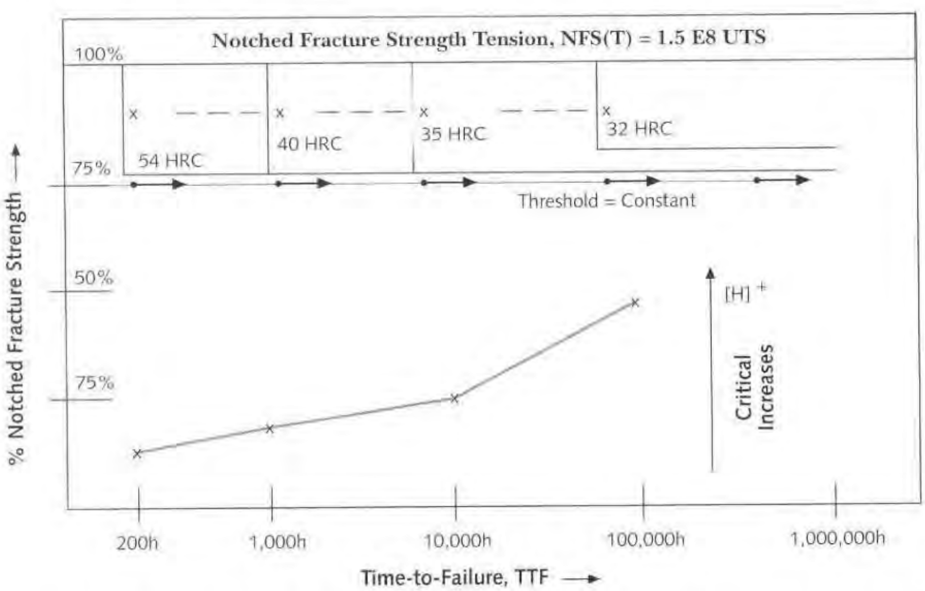

FIGURE 1. The tolerance for hydrogen increases as the hardness decreases, © 2001 LRA/RSL™

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History of LRA/FDI/RSL™

Company Performance and Accomplishments summary: LRA is a small R&D business founded in 1983 in Newport Beach, CA. The vision of LRA is to leverage military technologies, by minimal modification, to meet commercial and space requirements and effectively transfer into the product marketplace, with a special focus hydrogen embrittlement (HE) and stress corrosion cracking (SCC). In support of failure analysis, LRA has developed rapid, inexpensive testing techniques that use miniature specimens for evaluating SCC and HE. The focus of LRA is developing methods for use in failure analysis, where small specimens and accelerated methods are essential. Normal test methods for evaluating material for KIscc required more material than was available for most failure analyses, and required far too much time to produce results within the time limits demanded of a failure analysis.

These unique, accelerated test methods have a long history of development. The approach was introduced by Dr. Raymond to the National Academy of Sciences and published under **NMAB 328**, in 1976 entitled, *"Rapid Inexpensive Tests for Determining Fracture Toughness"*. These methods were further developed for *"Microstructural Analysis and Indexing of HY-130 Steel Weld Metals for SCC, for the High-Strength Steel Weldments"* in support of the Subcritical Cracking Program, David W. Taylor Naval Ship Research and Development Center, DTNSRDC/SME-80/76, Sept '80. These methods were then extended and found to be successful for HY-130 weld metal studies for **NAVSEA** under a previous **SBIR** *"Accelerated Stress Corrosion Cracking Screening Test Method for HY-130 Steel"*, (PHASE I & Phase II - Final Reports: SBIR Topic N88-84: Naval Sea Systems Command Contract No. N00024-89-C-3833, 10 December 1989 and 29 Sept 1993, respectively).The accelerated test methods were then applied to *"MP 159 Fastener Characterization Program using Accelerated Small Specimen Test Methods for (1) Stress Corrosion Initiation, (2) Crevice Corrosion, (3) Hydrogen Embrittlement, and (4) Fracture Toughness Testing of 1-1/4 Bar Stock and 1-1/4 Finished Bolts"*, (Final Report as Subcontractor to Standard Pressed Steel, Jenkintown, PA under contract to NASA, Huntsville, AL MSFC, 20 Aug 1993).

After completion of the Phase II (Naval Sea Systems Command Contract No.N00024-89-C-3833) the test method was commercialized in Phase III by creating **FDI** to manufacture the test machines. The 1991 SBIR resulted in three patents (Patent No. 5585570, 5505095 and 5549007). In addition, Dr. Raymond received the Los Angeles City Council of Engineers and Scientists *George Washington, 1994 Engineer of the Year* "for development of an innovative accelerated environmental test machine", followed by a Phase I Navy SBIR Topic N99-199, Contract N00024-99-C4095 entitled, *"Accelerated, Small Specimens Screening of High-Strength Corrosion Resistant Fastener Materials for Critical Naval Marine Applications"*. In 2006 after completion of the SBIR in 2000 he received the *Industrial Fastener Institute Technology Award* for "a significant contribution towards the understanding of hydrogen embrittlement, through years of research into accelerated methods for measuring the threshold stress, and development of the incremental step load technique as a practical means for qualifying and controlling hydrogen embrittlement in fasteners."

Since completing the Phase I award in 2000, LRA has worked to develop standards covering the Rising Step Load test method resulting in **ASTM F1624** *"Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique"*. The test method has been inserted into **ASTM F519** and **NASM 1312/5** and has been adapted to testing fasteners resulting in it being incorporated into a **ICC/AC193B** standard for measuring the hydrogen embrittlement threshold stress of concrete anchor bolts. Recently, the latest standard that incorporate the RSL™ Testing Protocol is **ASTM F2660** *"Standard Test Method for Qualifying Coatings for Use on A490 Structural Bolts Relative to Environmental Hydrogen Embrittlement"* has been released. Now working with ASTM Committee E08 on adapting the RSL protocol to modify **ASTM 1681** on **KIscc**.

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