Process Management to Control Risk Of Hydrogen Embrittlement

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For decades, the world as we know it has been plagued with the fear of *Hydrogen Embrittlement*. "Fear" is probably not too strong a word when we realize that this 'hydrogen bug' can infect our products with catastrophic results and liability claims. To further complicate the situation, our knowledge of the phenomenon is very limited and, until recently, we did not have reliable and effective tools to measure and monitor our efforts to harness this potential monster.

Hydrogen embrittlement concerns are not limited to fasteners alone! Every hardened metal part is a candidate for this "disease." Cams, pins, rivets, springs, hardened stampings, and hardened screw machined parts are also potential victims, just to name a few. And since almost every known industry uses at least some, if not a substantial quantity of susceptible parts, the problem becomes enormous.

Preliminary research suggests that processors now have a tool and a method by which they can monitor their process to identify and eliminate, or at least control potential sources of detrimental hydrogen. Appropriate documentation will minimize potential liability and instill a new level of confidence between the producer and the purchaser. Until now, a bake period following a chemical or electrochemical process, has been considered to be the normal procedure to guard against embrittlement. Costly baking has been mandated by specifications, without due consideration of the necessity of the process. In many cases, the baking process has been proven to be ineffective or in some instances, actually exacerbate embrittlement.

"Process control" is the undisputed way of the future. Now that we have uncovered a reliable measuring method, we have opened opportunities to develop, operate, and refine better processes providing better product at lower costs.

The Insidious Nature of Hydrogen Embrittlement

Hydrogen embrittlement occurs at the atomic level, *within* a metallic material; therefore, there are no visible, exterior signs of potential failure of a part, product, or structure. The consequences are much more devastating than corrosion because failures are unexpected, occurring with no warning because the crack initiates at the atomic level, within the metal, below the surface, whereas corrosion occurs on the surface of the metal and can be visually detected. Similar to blood pressure in humans, there is no external indication that a problem exists with hydrogen embrittlement. Only by indirect methods can the problem be monitored.

By comparison, corrosion or stress corrosion cracking failures are evidenced by the presence of corrosion products (e.g., rust, etc.). Hydrogen embrittlement failures can occur in the absence of any corrosion products. No external evidence of corrosion products causing rust or discoloration needs to exist.

Interestingly, attempts to alleviate corrosion problems with different finishes can actually introduce hydrogen embrittlement problems, especially those that use cathodic protection systems to prevent corrosion—to keep parts nice and shiny—either by using anodic coatings or platings, sacrificial anodes or imposed voltages during plating processes.

Technical Challenge:

Hydrogen Initiates Cracks at Atomic Level

Controlling hydrogen embrittlement is technically challenging because of the requirement of trying to measure the interaction of the smallest atom in the periodic table, hydrogen, on an atomic level within metals used in large structures to very small structural elements such as screws. With time in service under stress, subsurface hydrogen cracks initiate and grow until a time-dependent catastrophic failure occurs. A hydrogen embrittlement failure can occur, while sitting in storage from residual stress or even sooner in service when an external stress is applied. The hydrogen can come from cleaning or plating steel parts or from exposure of cathodically protected steel parts to a service environment that includes fluids, cleaning treatments, or maintenance chemicals that may contact the surface of steels. The critical parameter that delineates the stress at which a failure will occur is the threshold stress.

The Need for an Accelerated Test Method To Measure Threshold

The combined residual and applied stress above which timedelayed fracture will occur (Finite Life) or below which fracture will never occur (Infinite Life) is referenced as the threshold stress. Classically, time-to-failure sustained load tests are conducted to measure the threshold stress.

The reason that the threshold has not been used to measure the stress for the onset of a stress- induced hydrogen crack in production is that an economical and reliable method to measure the threshold in a reasonable amount of time did not exist.

For high-strength steels (>175 ksi), 12-14 specimens and several high-load capacity machines are required to measure the threshold, which can take as much as 3-4 months. For lowstrength steels (<175 ksi), the run-out can take as much as 4-5 years per U.S. Navy requirements for steels at 33-35 HRC. Even for steel specimens with cracks already introduced and tested per ASTM E1681,>10,000 hr (>1 year) is specified for run-out. None of these testing protocols are practical for a quality assurance or process control specification.

Technological Breakthrough

A new procedure was developed that resulted in an accelerated, economical, quantitative, reproducible test method that measures a *threshold stress*. The test method clearly defines the susceptibility of a material to hydrogen embrittlement and to time-delayed cracking once the part or structure has been put into service. This research was initially conducted at LRA Labs, under an Army contract from Milt Levy at Watertown in 1988-90, to study hydrogen embrittlement from plating of high-strength steels. Additional research was carried out by means of a Small Business Innovative Research (SBIR) contract in 1990-93, under Dr. Alexis I. Kaznoff at NAVSEA in Washington, DC, to develop an accelerated hydrogen embrittlement test method for welded HY-130 steels at 33-35 HRC.

This new method, commercially identified as the Rising Step LoadTM (RSLTM)¹, provides a means to measure the threshold stress or threshold stress intensity for the onset of hydrogen stress cracking in steel in one week on only one machine. This new test method was incorporated in new standards to control the risk of hydrogen embrittlement.

Hydrogen Embrittlement Detected Before the Part Fails

The new standard, ASTM F1624 entitled, "Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique" detects hydrogen embrittlement before the test specimen breaks. It measures the critical stress at which a crack is formed. By staying below this stress, designated the threshold stress, during installation, a part will never fail from the time-delayed fracture mode commonly referred to as hydrogen assisted stress cracking. This practice is far superior to the current practice of selecting an arbitrary sustained load and an arbitrary time-to-failure to create an arbitrary pass/fail criterion.

Process Control Assures Quality

Because the F1624 standard test method is accelerated, it lowers the cost of determining a threshold stress, and also permits quantitative monitoring of the embrittling tendencies of plating baths with mechanical tests on a regular basis. This capability to measure long term effects in real-time provides the basis for using process control to assure quality instead of performing static load plate tests on a statistically significant sample size from each lot of plated fasteners.

Joint Effort Generates Solution

It was not until the spring of 1995, when engineers and scientists from Aerojet, Thiokol, KLM, United Airlines, Boeing/Douglas, Northrop-Grumman, the US Air Force, US Navy, Ford, General Motors, and Daimler Chrysler focused on this critical issue and united under the auspices of ASTM Subcommittee F07.04. This group recognized the commonality of the problem at the First International Technology Transfer Conference held in Denver, CO, May 17-19, 1995, entitled, *Hydrogen Embrittlement: Problems & Solutions*. Any progress made toward a technically sound, reliable, reproducible and economically feasible test method was a direct outgrowth of the problems addressed at this conference.

Interlaboratory Testing Demonstrates Repeatability & Reliability

Once the test method was developed and standardized as ASTM F1624, interlaboratory testing was performed to de-

termine the repeatability and reliability by a group of fastener companies and testing laboratories from Canada and the United States. They formed a consortium to apply the results of the research programs to formulate an accelerated, reliable, low-cost process control verification standard for fasteners. The group, known as CEGAF, consisted of Camcar Textron, Elco Textron, Galvano, Acadian, Fracture Diagnostics Inc. (FDI) and the RSLTM Technology Center. The group has now expanded and is active as an ASTM F16.3 Task group. Funding is being sought for additional formal research programs.

At the ASTM Meeting at Norfolk, VA, in November of 1998, ASTM Committee F16 on Fasteners approved a new standard, ASTM F1940, entitled, *Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners*. This standard incorporates ASTM F1624 with the concepts of an aerospace industry process control standard, ASTM F519 entitled, *Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments* to provide a process control standard for the plating and finishing industry that represents the first significant improvement in hydrogen embrittlement testing in 40 years.

These new standards will permit platers to:

- 1. Verify that the plating or coating process is in control;
- 2. Verify that the plating or coating process is not causing internal hydrogen embrittlement;
- 3. Eliminate the need to test plated fasteners or other components for internal hydrogen embrittlement;

This new test method will also:

- 4. Provide a means of determining the effects of a process change;
- 5. Provide a means of determining the effects of baking;
- 6. Assure the purchaser that the processed parts will pass any static load plate test;
- 7. Require only one test in one day (24hr) on one specimen on one desktop machine to monitor one plating line.

By incorporating this new technology and taking advantage of the benefits of accelerated testing with reproducibility and reliability, our "fear" of unanticipated hydrogen embrittlement failures and subsequent litigation, can now be alleviated in a cost-effective manner with a technically correct testing method.

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¹*Rising Step Load*TM(*RSL*TM) *is a trademark of Fracture Diagnostics Inc., Denver, CO. (www.FractureDiagnostics.com).*

² For procedure information, see www.LRA-FDI-BSL.com; for equipment information, see www.FractureDiagnostics.com.

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About the Authors



Jerry Schmidt's career in metal finishing spans more than 37 years and includes active employment in production, management and sales positions with prominent Canadian electroplating organizations, including Marsland Engineering, Rauscher Plating and Acadian Platers. In 1978 he was elected international president of the AESF. In February of 1997, he

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"Powder Paints Live Up to New Chal-

R. Bruce Meade joined Camcar Textron in December of 1978, after six years at National Lock Fasteners, leaving as Assistant Chief Metallurgist. At Camcar, he initially served as a laboratory supervisor at one of Camcar's manufacturing operations, moving to the Headquarters' Metallurgical Service in 1980. He has supervised that laboratory since 1982. The laboratory's main functions include raw material and heat treatment examination, as well as fracture analysis. He is active in the ASTM committee on fasteners (F16). He recently helped develop an ASTM standard, F1940: Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners.



Dr. Louis Raymond, P.E. (CA), FASTM, FIAE, is owner and principal consultant of L. Raymond & Assoc., Newport Beach, CA, a professional consulting corporation that specializes in failure analysis, fracture mechanics, hydrogen embrittlement, stress corrosion, and fatigue. He is chairman of Committee F07 on Aerospace and Aircraft and Subcommittee

F07.04 on Hydrogen Embrittlement. Dr. Raymond is a recipient of the 1993 ASTM Charles B. Dudley Award, and in 1994 he was selected as Engineer of the Year by the Institute for the Advancement of Engineering (IAE).

Dr. Raymond has been very active in writing standards for hydrogen embrittlement on material susceptibility and process control with ISO, ASTM. AIS/NAS, and other standardswriting organizations. He has completed a book for ASTM that consists of a compilation of standards on Mechanical Test Methods for Hydrogen Embrittlement: Evaluation & Control with Emphasis on Fasteners.

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