Embrittlement Relief Baking of Steel Fasteners After Cleaning and Electroplating Processes

Procedure

INTRODUCTION

Hydrogen embrittlement (HE) is caused by the introduction of hydrogen into steel that can initiate fracture due to residual stress or in service when external stress is applied. The hydrogen can be generated during manufacture, cleaning or plating processes, or the exposure of cathodically protected steel parts to a service environment including fluids, cleaning treatments, or maintenance chemicals that may contact the surface of steel components.

The production of hydrogen will occur due to an electrode reaction in *either* acid or alkaline solutions.

It must also be noted that high strength metals are at most risk from hydrogen embrittlement. The susceptibility and sensitivity of metals to HE will be dependant upon the alloying elements and their dispersion in the material, the hydrogen content of the metal, the tendency of the material to absorb hydrogen, the hydrogen metal interaction, and the applied stress to the component.

The absorption of hydrogen by metal was first observed in 1864 when an iron specimen was immersed into dilute sulphuric acid. Furthermore, the amount of hydrogen that can be absorbed by iron could be varied by the application of a cathodic current.

This discovery in 1922 coupled with the earlier observation demonstrates that hydrogen atoms produced electrochemically may enter the metallic lattice and permeate through the metal structure. It is the entry of these hydrogen atoms into the lattice of the metal that causes embrittlement, and as a result, possible catastrophic failure of components in service.

If large amounts of hydrogen are absorbed into the metal then a loss of ductility may occur, however, the loss of ductility may not be evident through normal mechanical testing of fasteners.

Internal pockets of hydrogen may occur in localised areas, which could explain subsurface cracking. Small amounts of hydrogen that are dissolved within the metal may also react with the micro structural features of metal alloys. This will produce catastrophic failures of components at applied stresses far below the yield strength of the component.

1: Scope

1.1 This specification covers the Hydrogen De-Embrittlement of all Heat Treated Fasteners, which are cleaned or coated with a tensile strength of 800MPa (hardness 320Hv) and above. This will also include the De-Embrittlement of fasteners with property classes of 8.8 Grade and above.

1.2 This specification will cover the Embrittlement, including the following:

- Definitions.
- Categorisation of steels.
- Information to be submitted to the supplier.
- Cleaning Processes.
- Baking temperature.
- Duration of baking
- Commencement process

1.3 All procedures are Intafast Ltd plus our ISO certified partners and, unless stated otherwise, are recognized to be 'Industry Normal' practice.

1.4 Unless stated otherwise the methods and practices given here have no association with any National or Internationally recognized Standards Organizations.

1.5 The information contained within this procedure applies to all propitiatory or development products, manufactured by or for Intafast Ltd when needed.

1.6 This procedure may be adopted for use with any product where by the need to De-Embrittle arises. However, with regard to case hardened and through hardened products, if the products would suffer an unacceptable reduction in hardness by undergoing Baking as given in table 1, then advice will be sought from the relevant engineering bodies.

1.7 The implementation of this standard may involve the use of hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of whosoever uses this standard to consult and establish appropriate health and safety practices and determine the applicability regulatory limitations prior to use.

1.8 This specification attempts to reduce all possible risk of hydrogen embrittlement of fasteners. However, it must be noted that complete elimination of hydrogen embrittlement cannot be guaranteed. If the risk of hydrogen embrittlement is to be completely avoided then the use of mechanical plating/cleaning or other non-embrittling cleaning and coating processes must be used.

2: Referenced Documents

Reynolds, L., (2000) ***"Examination of Self Tapping Screws 805046MOT902",*** Institute of Spring Technology, Report No.2827Q. BS7371:Part 1:1991***:"Coatings on metal fasteners".***

BS1706:1990: ***"Electroplated Coatings of Zinc and Cadmium on Iron and Steef'.***

BS3382:1961***:"Specification for Electroplated Coatings on Threaded Components".***

BS EN 1403:1998: ***"Corrosion protection of metals*** - ***Electrodeposited coatings*** - ***Method of specifying general requirements".***

BS ISO 9587:1999: ***"Metallic and other inorganic coatings*** - ***Pre-treatment of iron or steel to reduce the risk of hydrogen embrittlement'.*** BS ISO 9588:1999: ***"Metallic and other inorganic coatings*** - ***Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement'.***

BS EN 12540:2000: ***"Corrosion Protection of Metals. Electroplated coatings of nickel, nickel plus chromium, copper plus nickel and copper plus nickel plus chromium".*** DEF 03-4 /. Issue 3:Feb 1991: ***"The pre-treatment and protection of steel items of specified maximum tensile strength exceeding 1450 MPa".***
DEF 03-2 / Issue 2:Mar 1991:
"Cleaning and preparation of metal surfaces".

JIS H 8610:1991: ***"Electroplated coatings of zinc on iron or steef'.*** ASTM B 633: ***"Standard Specification for Electrodeposited Coatings of Zinc on Iron and*** Steef'. Raymond, L., Ed., (1988) ***"Hydrogen Embrittlement: Prevention and Controf',*** ASTM STP 962, Philadelphia, USA.

3: Definitions

3.1 Baking. A low temperature baking of theproduct given after processing to reduce or eliminate hydrogen embrittlement by expelling the absorbed hydrogen,or by diffusing it through the product.

3.2 Baking time. The duration of the heat treatment shall commence from the time at which the whole of each part attains the specified temperature. This temperature must be achieved within 4 Hours (maximum) after completion of the electroplating process.

4 :Categorization of steels

4.1 With the exception of surface hardened products, the Baking conditions shall be determined upon the tensile strength if known. If only the minimum tensile strength is known, then categorization will be done by relating either known or measured hardness values to the equivalent tensile strengths. Approximate figures for the relationship between hardness and tensile strength are given in table 1.

4.2 Products which have been surface hardened shall be considered to be in the category appropriate to the hardness of the surface layer.

NOTE: The risk of hydrogen embrittlement failure of parts with hardness greater than 390HV including 12.9 grade product, cannot be excluded, and is considered high risk. Coating processes which do not involve electrolytic reactions are recommended. Where possible mechanically applied coatings, non-electrolytically applied zinc flake coatings, or organic coatings should be used.

5: Information to be submitted to the supplier

5.1 The type of protective coating. That will be applied to the product.

5.2 The tensile strength and / or hardness of the product to be processed. If the product is case hardened, then the case hardness must be specified. Categorisation of hardness can be either by known / specified or measured hardness values.

6: Cleaning Processes

6.1 Processes using cathodic treatments inalkalineoracidsolutionsmustbeavoided.

6.2 Parts with a tensile strength up to 1255MPa (hardness 390Hv), but not including 12.9 grade product, shall be cleaned with an inhibited acid, alkaline or mechanical process.

***Note:** The use of inhibitor in acid cleaning baths does not necessarily guarantee avoidance of hydrogen embrittlement.*

The time of which parts are immersed in inhibited acid increasesthe risk of hydrogen embrittlement. Therefore, immersion time should be kept to a minimum.

6.3 Parts with a tensile strength greater than 1255MPa (hardness390Hv), including 12.9 Grade product), shall be cleaned using non-acidic methods.

- **De-greasing in organic solvent.**
- **De-greasing in aqueous alkaline solution with applied anodic direct current if required. Cathodic or alternating current treatments shall not be used.**
- **Alkaline de-rusting.**
- **Abrasive cleaning. Care must be taken to as the product will be especially sensitive to corrosion after this method. Protective treatment should be undertaken without delay.**

6.4 Parts heat treated to a tensile strength greater than 1400MPa (hardness 440Hv) shall be cleaned using one or more of the following methods. All processes in the cleaning sequence shall occur concurrently without delay, an should be followed immediately by the protective treatment. Other processes may be involved in special circumstances.

7: Electroplating

7.1 Where Electroplating is carried out in conventional plating solutions, steps shall be taken to ensure the solution is working at a typical efficiency for the process. After any electroplating process of susceptible fasteners, the de-embrittlement baking requirements must be complied with.

8: Baking Temperature

8.1 For all Hydrogen De-Embrittlement processes, the Baking temperature shall be between 190°C and 210°c.

8.2 The Baking time and it's measurement shall be in accordance with section 10.

9: Commencement of Baking Process

9.1 The heating process shall commence within 1 hour after the completion of electroplating. The oven will be at the temperature specified as in section 7 at the commencement of the Baking process.

9.2 For zinc and cadmium plated products, baking shall be carried out before chromate treatment.

10: Duration

10.1 The duration of the Baking according to product hardness is given in table 1.

10.2 The baking time shall commence when the components attain the baking temperature.

11: Certification

Full traceable records of Hydrogen De-Embrittlement process must be maintained for a minimum of 5 years These records shall contain the minimum information as follows:-

1. **Batch Number**
2. **Order Number**
3. **Record of cleaning process employed**
   Oven Temperature
4. **Record of commencement of Baking, including time when electroplating finished.**
5. **Baking Time**

Table 1. Duration of the Baking according to product tensile strength.

Specified Minimum Tensile Strength MPa. (Hardness, Hv 10Ka)	Baking Duration (Hrs)	Application Examples of Products
800-1400 (250-440)	**8**	**8.8-12.9 Grade Hardened**
1400-1800 (440-560)	**18**	**Case Hardened Product/Spring Steel**
Above 1800 (Above 560)	**24**	

INDUSTRIAL FASTENERS INSTITUTE

REFERENCE

Identifying Hydrogen Embrittlement Failures

Cases of hydrogen embrittlement in screws and bolts.

Experience has shown that many of suspected hydrogen embrittlement failures were eventually proven to have causes completely unrelated to hydrogen embrittlement.

It is important for fastener suppliers to learn how to quickly determine whether or not a reported failure could be attributable to hydrogen embrittlement.
True Failures Have Common Characteristics

The wide understanding and experience related to confirmed cases of hydrogen embrittlement failures in fasteners indicate that the following elements must all be true:

Fasteners must be core hardened to at least Rockwell C32.

The parts must have come into contact with acid at some point in their processing.

The failures must occur some time after installation, usually between one and twenty four hours. Parts must have a nonporous finish (usually electroplated). The most common finish associated with hydrogen embrittlement failures in screws and bolts is electroplated zinc.

The parts must be under stress when failure occurs.

If any of these factors are not present, the chances of the failure being confirmed as hydrogen embrittlement are unlikely.

Unhardened fasteners or those of Grade 5 or Property Class 8.8 or lower do NOT fail due to hydrogen embrittlement.

Fasteners with phosphate an-a 011 do NOT fail due to hydrogen embrittlement. Parts that are cleaned "by mechanical processes instead of acid are highly unlikely to fail due to hydrogen embrittlement. Failures that occur while parts are being installed are NOT due to hydrogen embrittlerment.

Head to Body Junction is the Most Common Failure Hydrogen embrittlement failures occur where the stress in the screw or bolt is most highly concentrated when installed in an application.
The most common location of hydrogen embrittlement failures in parts is where the head joins the body of the screw or bolt.

Frequently, it looks like material has been scooped out of the underside of the head. The second most common failure location is immediately above where the external thread is engaged with the internal thread in the application. If the failure is somewhere else on the part, the cause is probably something other than hydrogen embrittlement.

Hydrogen Embrittled Parts Exhibit Intergranular Failures . Metallurgists refer to hydrogen embrittlement failures as intergranular brittle failures, as opposed to ductile failures.

The understanding of this is that when parts fail from hydrogen embrittlement, the connections between the material's grains let go very abruptly from one another when they are put under stress. They leave sharp edges on the surface of the fracture with an appearance much like rock candy. There is usually no evidence of any tearing ac-tion on the surface of the fracture and the fractures are relatively straight across the part transverse to its axis.
This is different from failures that look like the part has pulled apart like clay where there is some "necking down• before total failure occurs. When a failed surface looks pulled or twisted it is called a ductile failure instead of a brittle or intergran-ular fracture.