

# STEEL FASTENERS FAILURE BY HYDROGEN EMBRITTLEMENT

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**ABSTRACT:** his paper details the analysis carried out on two failed bolts to find out the reason for failure, using macrofractography, optical metallography and scanning electron microscope (SEM) techniques. Usual measures to prevent such failures are also referred. Similar fractures occurred at the head to shank transition of the bolts during fastening, or afterwards, while waiting for delivery. Since failure occurred in a region where foreseen stresses were quite below yield strength and there was no evidence of mechanical deformation, embrittlement was suspected. SEM observation of the fracture surface crack initiation region showed areas of intergranular crack propagation indicating brittleness, and higher magnification observation showed hydrogen embrittlement features. The bolts, which microstructure was typically that of hardened and tempered steel, had been zinc electroplated and it is well known that hydrogen release concurs with zinc deposition. Moreover, the diffusion and accumulation of the hydrogen in metals is favoured by cold working, as is the case of the head to shank transition region.

**Keywords:** Hydrogen embrittlement, failure, steel, fractography, scanning electron microscopy

**RESUMO:** Este artigo apresenta a análise efectuada em dois parafusos fracturados, com o objectivo de determinar as causas da fractura, usando técnicas de macrofractografia, metalografia óptica e microscopia electrónica de varrimento. Também são referidas as precauções habituais para evitar este tipo de falha. As fracturas, semelhantes entre si, ocorreram na transição entre a cabeça e o corpo do parafuso, durante ou após a operação de aperto. Uma vez que as fracturas apareceram numa região em que as tensões previstas estariam bastante abaixo da tensão de cedência e não se observava deformação mecânica, a fragilização foi desde logo uma possível suspeita. A observação da superfície na zona de iniciação da fractura, por microscopia electrónica de varrimento, revelou zonas de propagação intergranular de fissuras, apontando para uma fragilização, sendo possível observar aspectos típicos de fragilização por hidrogénio a ampliações mais elevadas. Os parafusos, cuja microestrutura correspondia à de um aço temperado e revenido, tinham sofrido um tratamento superficial de zincagem. Sabe-se que, durante estes tratamentos, a libertação de hidrogénio compete com a deposição do zinco e, além disso, a difusão e acumulação do hidrogénio nos metais é favorecida pela deformação a frio, o que é precisamente o caso da zona de transição cabeça/corpo de um parafuso.

**Palavras chave:** Fragilização por hidrogénio, falha, aço, fractografia, microscopia electrónica de varrimento

## 1. INTRODUCTION

Zinc electroplated steel fasteners are widely used for assembling threaded joints, since zinc is anodic to steel and offers both good protection to atmospheric corrosion and good appearance, even when applied in thin films in order to obey the dimensional tolerances imposed to fasteners. Moreover, acid zinc plating baths developed since the 1970's made the process low-cost and easily applied in barrel or continuous procedures [1].

However, when high strength steel fasteners are required, such as ISO 898-1 grade 10.9 or higher, for which nominal tensile strengths over 1000 MPa are specified, bolts are made of quenched and tempered steel [2], and electroplating presents a high risk of hydrogen embrittlement. It is well known that during zinc electroplating hydrogen release concurs with zinc deposition. Besides selecting plating

solutions and conditions to produce high cathode efficiency in order to minimize the amount of hydrogen generated on the metal, a proper elevated-temperature (180-230 °C, lower to tempering temperature) baking heat treatment is generally required for hydrogen removal. The treatment must be applied for several hours, generally a minimum of 4 hours is specified (because the metallic coating plated on metal often acts as barrier to hydrogen diffusion [3]), and in a few hours, or immediately, after electroplating and before any further treatment is applied.

Hydrogen embrittlement is a well-known phenomenon in which a metal is weakened by the incorporation of hydrogen in or below its surface, e.g. during plating or etching. Hydrogen diffuses easily into the metal crystalline structure either as atoms or protons. Non-metallic inclusions such as sulphides, as well as phosphorus, favour hydrogen adsorption, acting as catalysts. Grain boundaries and other

lattice defects are also regions where hydrogen diffuses and accumulates easily, hence preferential for hydrogen embrittlement to occur. Hydrogen diffusion is also easier in all regions where the crystalline structure may be distorted as a result of work hardening or cold deformation. The accumulation of hydrogen restricts ductility and promotes brittleness.

Hardened and tempered steels need only a few p.p.m. of hydrogen to cause embrittlement [3, 4].

Failure induced by hydrogen is favoured or caused by applied stresses, even static or of low intensity, and is often referred to as “delayed” or “retarded”, since it may not take place immediately after tensile stresses are applied. This type of embrittlement is not detectable by hardness determinations, but tensile tests will show considerable reduction of the expected tensile strength of the material [5].

This work presents a case study where premature fragile failure of several bolts occurred, during assembly of components, or afterwards while waiting for delivery (delayed failure), at low stresses, quite below the specified proof stress.

## 2. FAILURE ANALYSIS

The fractures occurred at the head to shank transition of several steel fasteners used to fix an engine to its base. The following items and information were provided for failure analysis:

- Photographs of a component with a fractured bolt inside and of two fractured bolts (Fig. 1);
- two fractured bolts (head and shank);
- bolts were supposed to be class 10.9 (ISO 898-1) and zinc electroplated.



**Fig. 1.** General view of two fractured bolts

Preliminary visual inspection of the fractured bolts showed that:

- All bolts failed in a similar way;
- fractured surfaces were similar and the rupture had occurred apparently without necking (plastic

deformation) in the transition zone between head and shank;

- there was no general oxidation and/or corrosion;
- fracture surfaces were quite smooth and normal to the direction of the applied loads, except for the central part corresponding to the end of fracture;
- fractures seemed to have had its origin all around the circular external bolt surface, where several initiating zones were observed.

The following tests were carried out:

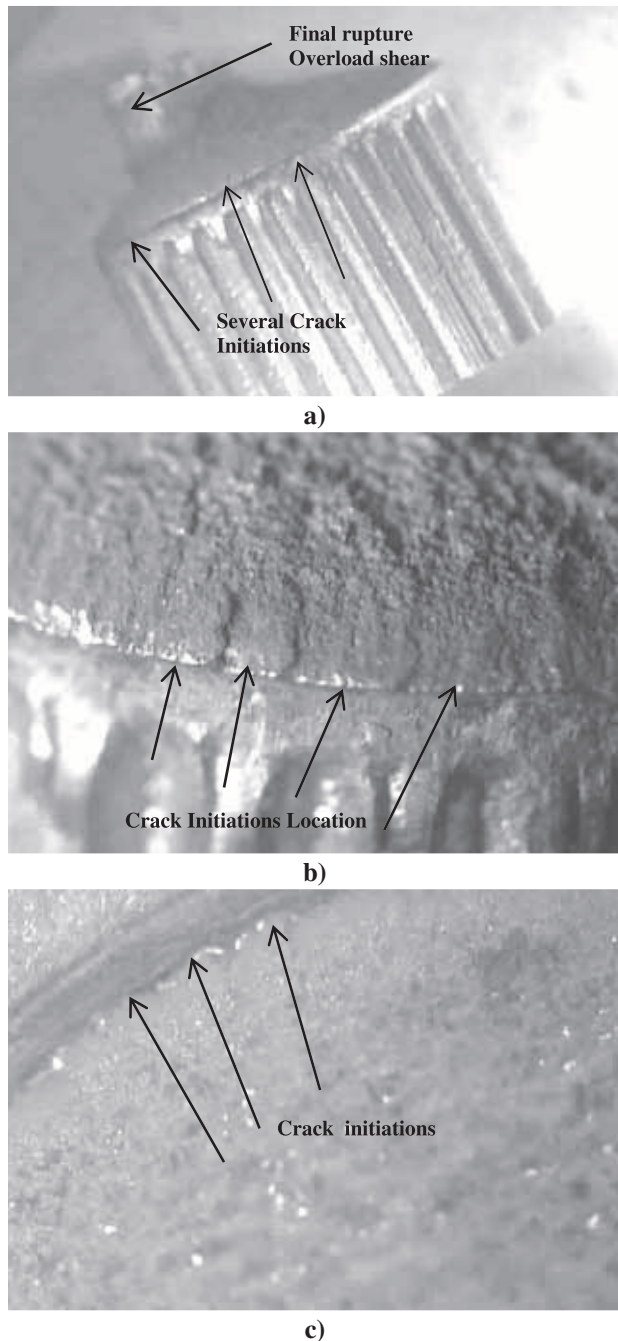
- Fractographic examination of both bolts fracture surfaces at low magnifications (3 to 20 times) with an optical stereomicroscope and also at higher magnification with a SEM (20 to 10000 times) after ultrasonic cleaning in an ethylic alcohol bath.
- Metallographic examination by optical microscopy of the fractured bolts microstructure on longitudinal sections from the head and shank part nearest to the head taken through the bolt axis.
- Rockwell C hardness determinations on a cross section of the shank.

## 3. RESULTS

### 3.1. FRACTOGRAPHY

Observations at low magnification (Fig. 2) showed that:

- Fracture characteristics were similar in both bolts indicating that failure must have occurred by the same causes;
- the fractures were almost parallel to the cross section, and occurred with no evidence of deformation or distortion near the origin;
- the fracture surfaces were nearly flat with several crack initiation zones all around the external bolt surface;
- origins were located at slightly different levels and after a short propagation cracks joined together in a common front;
- the fracture surface area corresponding to this first stage of propagation showed the smoothest surface on fig. 2 b) and 2 c);
- cracks continued to propagate towards the centre, and fracture surfaces became rougher;
- final rupture occurred by overload shear and showed a change in crack propagation direction towards the top of the bolt head;
- both fractures occurred almost instantaneously after cracking initiation due to the applied stresses.



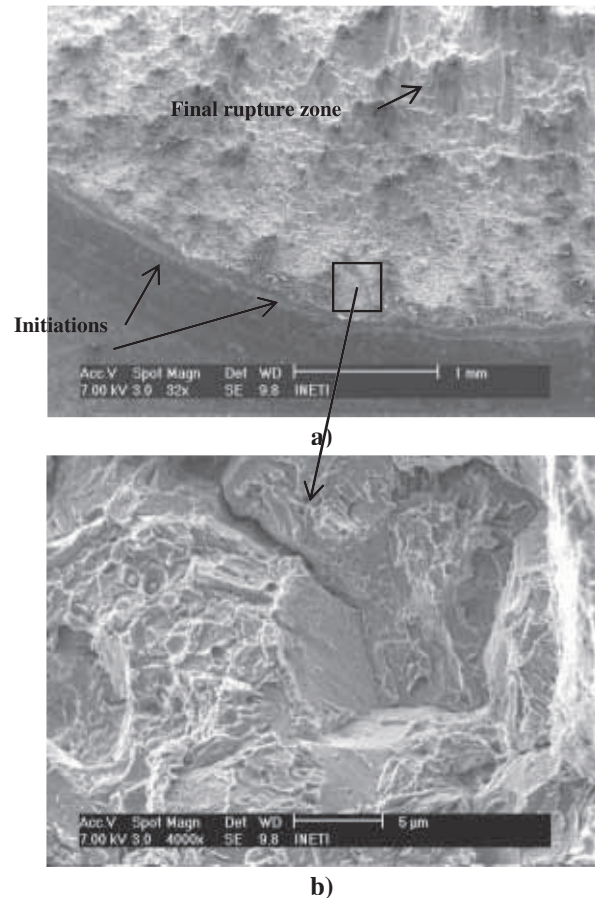
**Fig. 2.** Fractured bolt: a) General side view of fracture surface, shank side; b) details of fracture initiation zones at head-shank transition surface, shank side; c) details of fracture initiation zones at head-shank transition surface, head side.

SEM allowed further information about surface fracture features (Fig. 3, 4 and 5):

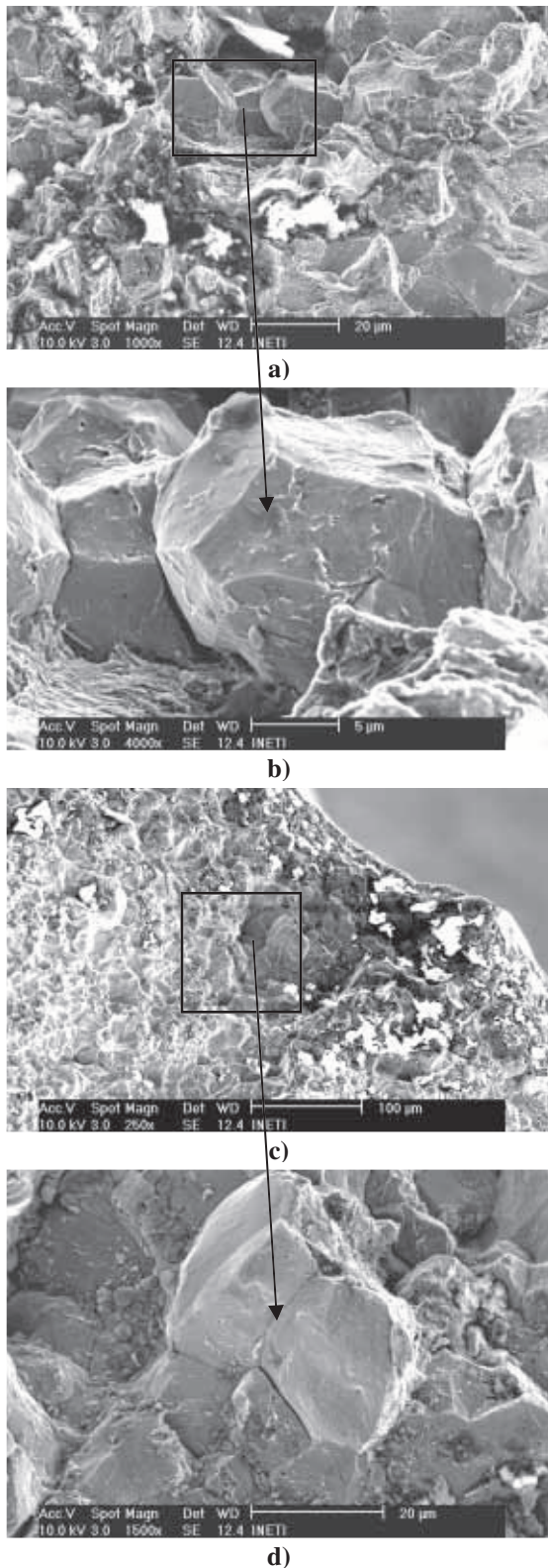
- Cracking initiation zones and zones near the surface presented both intergranular and transgranular crack propagation;
- crack growth zones showing intergranular features also presented intercrystalline subsidiary cracking (or yawning grain boundaries), and micro-pores and ductile hair lines, often branched forming crow feet marks, at prior austenite grain boundaries [6];
- intergranular fracture surfaces zones showed some small areas, looking like nests, where intercrystalline

characteristics are more pronounced as grains seemed more individualized;

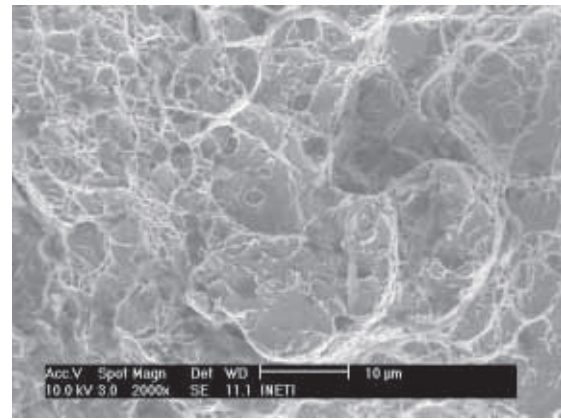
- at the end of the propagation failure process, close to the centre of the bolts, the fracture surface showed a typical dimpled configuration with elongated cavities (fig. 5);



**Fig. 3.** SEM observation of one of the bolts fracture surfaces: a) overall view of initiation zones near shank external surface; b) detail from marked area, fracture propagation partially intercrystalline and transcrystalline, intergranular zones showing intercrystalline subsidiary cracking, micro-pores and ductile hair lines at the former austenite grain boundaries.



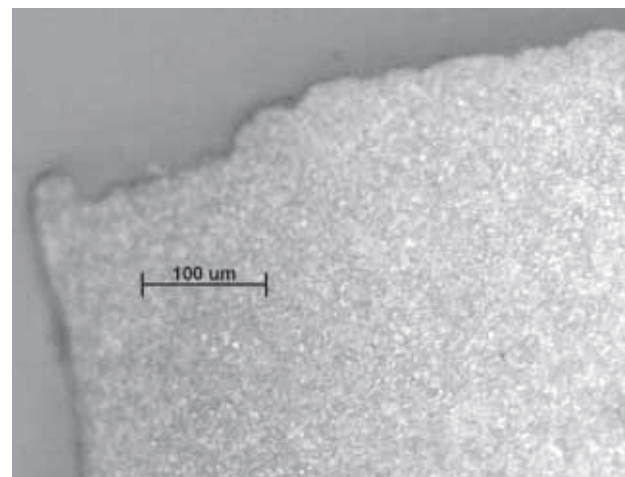
**Fig. 4.** SEM observation of the other bolt fracture surfaces: a) and c) general view at the initiation zone near the external surface of the shank; b) and d) details from marked zones with pronounced intercrystalline characteristics showing grain surface features like micro-pores and ductile hair lines at the former austenite grain boundaries.



**Fig. 5.** SEM observation of fractured surfaces: dimpled rupture fracture surface at final rupture zone near bolt centre

### 3.2. OPTICAL MICROSCOPY

Metallographic examination of the longitudinal sections from the head and the shank showed the typical microstructure of hardened and tempered steel, with no visible external surface decarburization (Fig. 6) or continuous carbide precipitation along grain boundaries (Fig. 7).

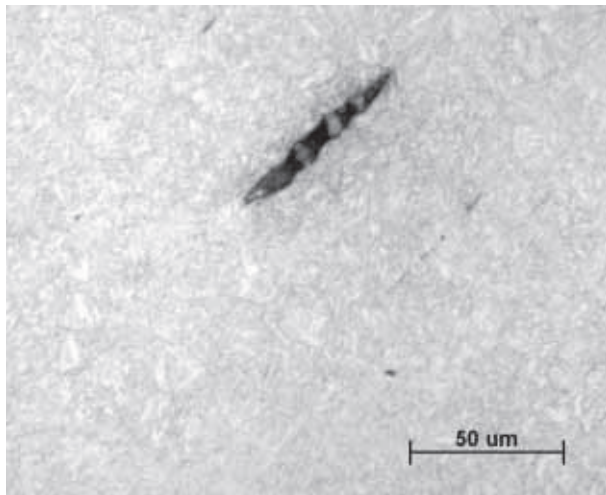


**Fig. 6.** Metallography of the shank cross section: external surface and initiation of fracture zone from the fracture surface

At the initiation zone, fracture surface was flat and rather smooth and there was no fracture branching neither oxides deposits nor decarburization over the fracture surface. Some small intercrystalline subsidiary cracking was detected on fracture surface grains.

There were several inclusions of the sulphide (type A) and oxide (type D) types, but always below the 2 ½ index of the JK chart (plate I ASTM E45) for type A inclusions and below 1 ½ for type D.

Few non-metallic inclusions appeared cracked or debonded from the matrix, indicating lack of ductility. Fig. 7 exemplifies one agglomerate of inclusions, cracked and debonded from the matrix, and seeming to originate a micro-crack, almost 50 µm long, with rather sharp tips.



**Fig. 7.** Metallography of the shank cross section just below fracture surface: sulphide and oxide non metallic inclusions and a set of cracked and/ or debonded inclusions, resulting into a micro crack.

### 3.3. ROCKWELL HARDNESS

The determinations were carried out at a cross section cut at approximately 1,5cm from the end of the thread. The average value obtained was  $36,2 \pm 3,1$  HRC (95% mean confidence level), value which is within the range specified for grade 10.9 bolts (32 to 39 HRC, after ISO 898-1 [2]).

## 4. DISCUSSION

The failure analysis here reported involves high strength steel fasteners grade 10.9 [2], heat treated (quenched and tempered), in order to achieve the specified high strength levels. The hardness values determined were in the range specified for this class of bolts (class 10.9, 32 to 39 HRC).

The flatness of fracture surfaces, the serial failures and the fact that failure process took place in a region where, when in service, expected tensile stresses are considerably lower (at least twice to three times lower) than those expected after fastening in the threaded shank zone at the first thread inside the nut, pointed out to a manufacture or material defect or to an embrittlement phenomenon as causes for failure.

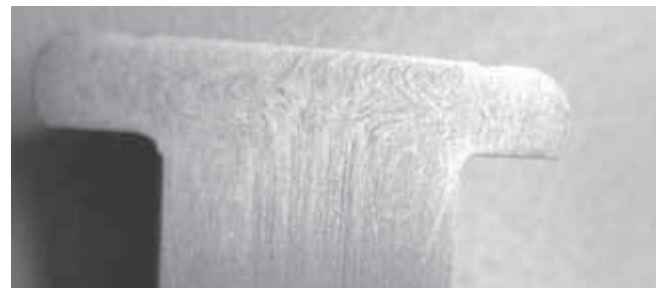
The microstructure observed corresponded to hardened and tempered steel, without detectable preferential carbide precipitation along previous austenite grain boundaries, so carbide embrittlement was excluded. Surface decarburization was not detected either.

Inclusion content of the steel was far below the maximum limits accepted for this kind of application. Forging defects, cracks or other discontinuities, that could have acted as stress concentrators or raisers were also not observed. Finally, there was not oxidation of the fracture surface, or crack branching along grain boundaries, which would indicate quenching cracking or a stress corrosion crack process.

The observation of the fracture surfaces carried out in the SEM showed that, starting at the external surface, crack

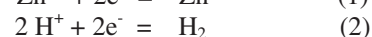
growth occurred intergranularly, indicating embrittlement. Typical features of hydrogen embrittlement were observed in the fracture surfaces of both bolts, as described above.

Bolts manufacturing process can produce highly non-uniform deformation, as shown in Fig. 8. The head region and in particular the head/shank interface can undergo significant cold work hardening raising the tensile strength in these regions considerably in excess of the 1000 MPa [4] specified. At these high strength levels, subsequent electroplating brings a serious risk of hydrogen embrittlement. The deformed microstructure which resulted from the manufacturing process, highly distorted and having high concentration of lattice defects like vacancies and dislocations, favours the diffusion and concentration of the hydrogen released during electroplating.



**Fig. 8.** Bolts cross section macrostructure showing the deformation lines produced by head forging due to non-uniform deformation

The diatomic hydrogen molecule is too large to enter the surface of a solid metal and diffuse interstitially. However, in the dissociated form, single atoms are relatively mobile, even at room temperature [4]. One way to produce atomic hydrogen is the cathode reaction competing with metal deposition in an electroplating process (equations (1) and (2)):



Therefore, the electrolytic process must be controlled, namely high efficiency procedures must be used, current intensity and bath impurities controlled and good agitation provided in order to liberate the hydrogen bubbles formed over the cathode surface. The control may not always be efficient in order to prevent hydrogen from concentrating in microstructure defects and some hydrogen may be inevitably present.

The solubility of hydrogen decreases with temperature and hydrogen accumulated in lattice defects may precipitate in gaseous form, originating a pressure high enough to produce small cracks. This can however be avoided by a post plating baking treatment to remove hydrogen prior to service [3,4]. Zinc electroplated components must undergo the treatment at temperatures around 200°C, for several hours and immediately or shortly after plating.

Internal hydrogen embrittlement has been known for years, but is far away from being banished or even controlled. Some components may require longer baking times due to

more gas evolution during plating or to thicker and less permeable coatings. To prevent risk of failure due to improper baking after electroplating, standardized tests on samples may be conducted [7,8]. Another alternative is to choose a coating procedure without hydrogen embrittlement risk.

## 5. CONCLUSIONS

Hydrogen embrittlement remained as the only probable cause of the failure observed. Unlike stress corrosion cracking and quenching cracks, cracks caused by hydrogen embrittlement usually do not branch [3] neither show oxidized surfaces. Typical features of hydrogen embrittlement were observed on the fracture surfaces of both bolts.

Bolts had been zinc electroplated, which is one way to introduce hydrogen into metals, and baking treatment after plating may not have been well conducted.

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