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AESF Heritage: The 2002 Hydrogen Embrittlement Seminar:
4. Hydrogen Embrittlement – A Personal View

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ABSTRACT

When atomic hydrogen enters steel and some other metals, such as aluminum and titanium alloys, it can cause loss of ductility or load carrying ability, cracking (usually as sub-microscopic cracks) or sudden catastrophic failures well below the yield strength or even the normal design strength for the alloys. This phenomenon often occurs in alloys that show no significant loss in ductility when measured by conventional tensile tests, and is frequently referred to as hydrogen induced delayed brittle failure, hydrogen stress cracking or hydrogen embrittlement. Hydrogen embrittlement is also observed in very clean iron and steel. Even mild steel and dead mid steel are susceptible under certain conditions. However, because of the complexity of the problem, the results of research work differ considerably from batch to batch of the same material, from material to material, from laboratory to laboratory and even from one test to another. Considerable research work has been carried out in the last 40 – 50 years producing inconclusive and often confusing results. The potential hazards of hydrogen embrittlement from the electro- and autocatalytic plating processes of high strength carbon steels have long been recognized in the plating industry and pre- and post- coating heat treatments as well as some test methods are specified in International Standards. These standards, based on research and field work, merit recognition from the plating industry.

Introduction

Hydrogen as a gaseous environment, or when trapped as a solute constituent in the matrix, is destructive to the mechanical properties of most metals and alloys, typically leading to a reduction in ductility and promoting sudden and premature catastrophic brittle failures, in presence of residual or applied tensile stress. The failures may be instantaneous or may take years after exposure to atomic hydrogen during fabrication, for example due to breakdown of lubricants, cleaning, acid pickling, conversion coatings such as phosphating and black oxide coating, autocatalytic and electroplating processes, as well as by corrosion during service and cathodic protection. Failure may occur at stresses considerably lower than the yield strength or the design strength of the material. It is normally encountered in steels of tensile strength of 1000 MPa (310 HV, 31 HRC) and above that strength level. Figure 1 shows the relationship between ductility, as expressed as percentage reduction of area, and hydrogen content.¹

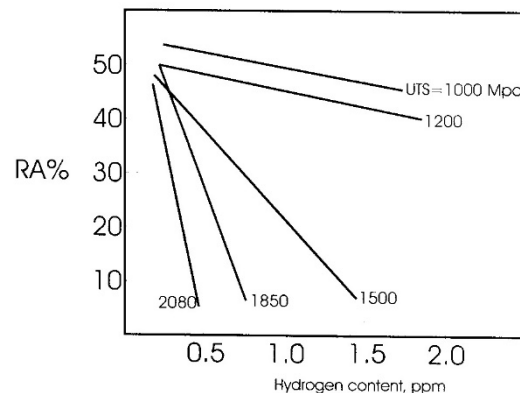


Figure 1 - The effect of hydrogen content and strength level on the tensile ductility of a quenched and tempered alloy steel (A.S. Tetelman and A.J. Mc Evily Jr, *Fracture of Structural Materials*, J. Wiley & Sons, Inc, 1967; p.461.)

Background

Theories

Hydrogen embrittlement in steel has been extensively studied and has been the subject of numerous papers, reports and books. A number of theories have been proposed to explain the phenomenon and its various aspects.²⁻¹²

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The hydrogen can enter steel as atomic hydrogen from a variety of sources. The absorption of hydrogen from any one single source may not be significant enough by itself to cause hydrogen damage but the cumulative effect of hydrogen absorbed has detrimental consequences on the mechanical properties. Hydrogen tends to accumulate at regions of high stresses and reach critical concentration during the processing procedures, as a precursor to the initiation of sub-microscopic cracks. It is known that a concentration of 0.1 ppm of hydrogen in the material may produce embrittlement. Hydrogen embrittlement can also be observed in very clean iron and steel; even mild steel and dead mild steel may be susceptible under certain conditions. The presence of sulfur not exceeding 100 – 150 A° at grain boundaries can cause hydrogen embrittlement problem.

Experience of service failures over the decades has led to the belief of material strength, as defined by hardness, characterizing the susceptibility of a given steel to hydrogen embrittlement. However, hardness alone is not the only parameter for the susceptibility of a given steel; the microstructural variations have a profound influence in hydrogen embrittlement. Elements, *e.g.*, grain boundaries, dislocation tangles, voids and pores, solid-solid interfaces such as inclusion-matrix and carbide matrix boundaries, also play a significant role in giving rise to the trapping of hydrogen atoms. Additionally, the size, shape and location of the inclusions in the structure play a critical role. The relationship between the type of traps and the form of hydrogen embrittlement, however, is fraught with considerable uncertainty.

The susceptibility of a material to hydrogen embrittlement is related to the characteristics of its trap population. Mobile hydrogen atoms attracted to the various trap sites tend to become immobile. These traps can be reversible or irreversible. In a reversible trap, hydrogen is attracted by attractive forces such as stress fields, temperature gradients etc., but can readily overcome the low binding energy barrier associated with the trap to become mobile. These traps can act both as a source and as a sink for hydrogen. The irreversible traps, such as high angle boundaries, inclusions, voids etc. are associated with high binding energy and are not capable of releasing hydrogen atoms so readily. Diffusion is controlled by the rate of escape of hydrogen from the traps; nature and density of the traps, trap mobility and the energy barrier associated with each trap affect the rate of diffusion of hydrogen.

Work with transition metals appeared to suggest that the total amount of hydrogen that could be absorbed rapidly decreased with increasing sulfur concentration. Sulfur appeared to block dissociation sites for hydrogen absorption and recombination sites for hydrogen desorption. Apparently, the degree of embrittlement was observed to be independent of the amount of non-metallic inclusions.

In another work involving two different types of carbon steels, one with low sulfur (0.003 wt%) and the other with high sulfur (0.024 wt.%), it was noted that the high sulfur steel because of its higher trap density absorbed greater amount of hydrogen than the low sulfur steel under identical charging condition,¹³ and the trapping energy of inclusion-matrix interfaces was greater in low sulfur steel than in high sulfur steel. The weak traps can, therefore, act as sources, while strong traps act as a sink for hydrogen.

Szklarska-Smialowska and Lunarska¹⁴ noted that the non-metallic inclusions, particularly manganese containing sulfides, occurring at the metal surface tend to promote hydrogen entry into metal. In high strength steels, internal non-metallic inclusions act as nucleation sites for hydrogen induced cracks. However, sulfides exhibiting a higher thermal expansion coefficient, may, in some cases, favorably affect the resistance of the steel to hydrogen induced cracking by trapping hydrogen in microvoids present around the inclusions.

Pound¹⁵ using a potentiostatic technique, applied to AISI 4340 steel and Monel, observed that the principal irreversible traps identified on the basis of the irreversible trapping constants appeared to be manganese sulfide inclusions for the steel, and, with some uncertainty, sulfur and phosphorus clusters at grain boundaries in the nickel containing alloys. Traps such as Fe₃C and TiC interfaces appeared to be not as effective as the MnS interface based on trap density calculations.¹⁶ The binding energy of hydrogen at the MnS interface was found to be higher, *i.e.*, the trap to be irreversible, than that for the ferrite-carbide interface, dislocations, microvoids. However, hydrogen embrittlement is not always related to trapping of hydrogen. Materials, such as Hastelloy, do not exhibit hydrogen trapping but are well known to be susceptible to hydrogen embrittlement. The author concluded that the type of cracking found in AISI 4340 steel, therefore, might not be directly correlated with sulfide inclusions; other factors, such as size and shape of inclusions, could play a critical role.

It is accepted that nascent hydrogen enters the steel and diffuses through it until it encounters a defect or a void where it can recombine into molecular hydrogen, thus increasing the pressure and causing growth and further separation of the flaw. Gross

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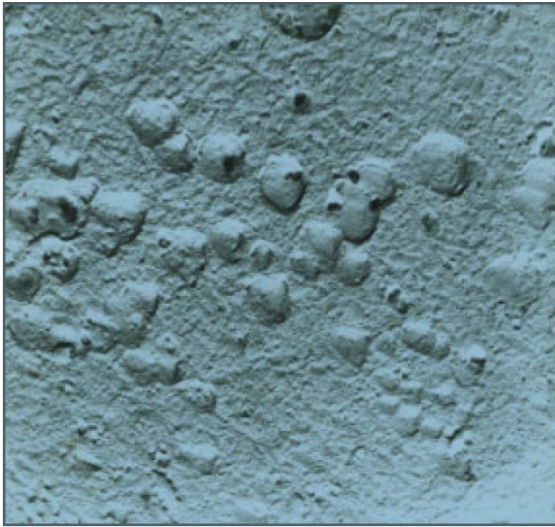


Figure 2 - Transmission electron micrograph of a carbon replica of the surface of a corrosion fatigue tested mild steel specimen exhibiting formation of hydrogen blisters. $S_{max} = 198$ MPa and $R = -1$ after 2000 reversals. 20,000X.

inclusions are examples of such defects. The internal flaw growth eventually manifests as a blister on the surface. Figure 2 shows formation of hydrogen blisters on a corrosion fatigue tested mild steel specimen.¹⁷

It is also well known that although the quantity of hydrogen induced shows some indication with the degree of embrittlement, it is not the sole factor involved. For example, if hydrogen is generated in a cavity or on a sulfide inclusion close to the surface, it can produce sufficient pressure to force up the surface layer causing crack initiation. Ultimate failure may occur due to a completely different failure mode, *e.g.*, fatigue, corrosion fatigue etc. However, if hydrogen is generated at an isolated cavity or inclusion far away from the surface, and if a tensile stress is applied, then the combined force can initiate a crack even though the tensile stress may be insufficient for breakage. Additionally, if there is a large number of uniformly distributed traps, the hydrogen concentration at potential crack sites may not reach the critical level for cracks to initiate or if the cracks initiate, they may not proceed far depending on the service condition. Hence, the distribution of hydrogen is also considered to be significant.

Pre- and post- processing treatments

The various treatments proposed in a range of standards and specifications related to hydrogen embrittlement relief procedures could be attributed to a less than adequate understanding of the problem involved. Processes involving aqueous solution codeposit hydrogen and therefore have the potential to embrittle steel. Whether the items are embrittled or not will depend on what steps are taken to prevent the hydrogen from reaching the critical concentration.¹⁸

The deleterious effects of hydrogen absorbed during processing, such as cleaning, pickling etc., can be minimized by pre- and post- processing heat treatments.

In a research work¹⁹ on sustained load, fracture toughness and bend tests to determine the susceptibility to hydrogen embrittlement of cadmium plated 3% Cr-Mo-V steels, of varying tensile strengths (1700 MPa to 2230 MPa), carbon and impurity content, it was noted that:

- (i) extremely small amounts of hydrogen, the greatest amount extracted from any specimen was 0.20 mL/100gm, assuming an even distribution in the steel, could cause embrittlement of high strength steels. Concentration at traps and highly stressed areas would result in localized areas with considerably higher hydrogen content,
- (ii) stripping of cadmium from plated specimen did not reduce the degree of embrittlement unless the specimens were subsequently given embrittlement relief heat treatment,
- (iii) the effect of carbon content and impurities on the 200-hr sustained load life of low alloy steels was small in comparison with the effect of tensile strength. Low carbon steels, however, were more susceptible to hydrogen embrittlement than higher carbon steels annealed to the same tensile strength. Most impurities were detrimental, but 0.016 percent sulfur appeared to have a beneficial effect possibly because of entrapment of hydrogen at voids associated with manganese sulfide inclusions. Small grain size maraging steels appeared to be resistant to hydrogen embrittlement possibly because of similar entrapment of hydrogen at precipitates and the locking of dislocations in the structures,
- (iv) the parameter affecting the sustained load life of low alloy steels was the tensile strength level. An increase in tensile strength from 1700 MPa to 1850 MPa resulted in a marked reduction in sustained load life. However, increasing the embrittlement relief heat treatment over 24 hours at 200°C extended the sustained load life of low alloy high strength steels but did not necessarily eliminate failures.

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The author concluded that the Allison instrumented bend test,²⁰ notched tensile tests and fracture toughness tests appeared to be unsuitable due to the short time nature of the tests, *i.e.*, there was insufficient time for hydrogen diffusion to the highly stressed area.

In a study²¹ on the tendency towards brittle failure, embrittlement test strips, which were fabricated from SAE 1075 spring steel, hardened and tempered to approximately 1600 MPa, and zinc plated and stressed in the form of U-bends, were subjected to 200°C embrittlement relief treatment for 4 hours. The author concluded that the four-hour treatment after electroplating was ineffective in providing relief from embrittlement.

Sell and Shreir²² investigated the restoration of ductility, which was assessed by bend tests, of cadmium plated bainitic steels (0.75%C, 1620 MPa) in relation to embrittlement relief heat treatment. The authors concluded that:

- (i) restoration of ductility can be achieved by heating at 200°C for approximately 12 hours.
- (ii) the decrease in hydrogen content due to the relief treatments corresponded approximately with the restoration of ductility, although complete removal of hydrogen introduced during cadmium plating required a somewhat greater time than complete restoration of ductility as assessed by bend tests.

These authors also observed that in the case of mild steel (0.2%C, 0.8 Mn), removal of hydrogen from unplated specimens, which had been saturated with hydrogen, could be achieved in approximately 10 hours at 200°C, the hydrogen content falling from 1.5 ppm to 0.0 ppm. In the case of cadmium plated bainitic steel, the hydrogen content after plating was 1.0 ppm compared to 1.5 ppm for the mild steel. The decrease in hydrogen content of the bainitic steel after the relief treatment, approximating to the original hydrogen content of the steel, was obtained after 12 to 16 hours.

Environmental sensitive fracture investigation of steels (0.06%C, 17%Cr, 1200 MPa) by these authors showed that on nominally smooth surfaces polishing scratches could act as sites for nucleation of microcracks in the presence of hydrogen.

Reynolds²³ investigated the time – temperature relationship of the post plating heat treatment processes using steels (0.81%C, 0.47% S and 0.98%C, 0.009%S, tensile strength 1700 MPa), zinc plated and subsequently given post plating heat treatments for varying length of time at temperatures around 200°C and 220°C. The author suggested that the effectiveness of the treatment appeared to fall off rapidly with the reduction of time, the relationship being essentially linear and heavily biased towards 24-hour region.

With hardened and tempered carbon spring steel (1.03%C, 0.010S, tensile strength 1800 MPa), he observed that:

- (i) hydrogen content of the bulk strip, with either zinc *in situ* or with the plating removed, could not be correlated with the level of ductility as represented by the angle of bend at fracture during slow bend test,
- (ii) the plated strip with lower zinc thickness (0.009 mm) exhibited a significant improvement in ductility after 18 hours at 190°C /200°C, as assessed by the bend test. With higher zinc thickness (0.036 mm), the strip did not exhibit a significant improvement after 18 hours at that temperature,
- (iii) irrespective of zinc thickness, a number of the plated strips still exhibited severe hydrogen embrittlement after post plating treatment at 200°C for 18 hours.

Lukacs²⁴ investigated the effect of post plating heat treatments²⁴ on hydrogen embrittlement of 8 to 10 mm thick zinc and cadmium plated SAE 1022 and 1083 steels, which were tested under static load conditions and torqued to the prescribed loading of the Australian Fasteners Institute recommendation. He concluded that there were definite relationships between:

- (i) the time allowed from the end of plating cycle to the time the parts were placed into the furnaces or ovens, and
- (ii) the duration of the embrittlement relieving treatment and temperature.

The parts, associated with his work, were treated for periods of 2, 4 and 8 hours, respectively, and were placed in the oven within 1 hour of plating at 200°C ± 5°C. A further batch were allowed to stand for 16 hours prior to the commencement of the embrittlement relief treatment. He noted that, for both types of steels, failure rates of the parts, which were given embrittlement relief treatment within 1 hour of the end of plating cycle, were considerably lower than those which were allowed to stand for 16 hours. Additionally, failure was still observed even after 8-hour embrittlement relief treatment.

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Commenting on a service failure, Fliege,²⁵ in a communication to the Editor of the *Product Finishing Journal*, wrote that the blanked and stamped end plates or end washers in a bearing assembly for 747 aircrafts showed radial fracturing after cadmium and chromium plating. As a result of that experience, the author instituted a 24 hour post plating heat treatment of all parts, both chromium and cadmium plated, which appeared to solve further hydrogen embrittlement problem.

Considerable evidence now exists that the delay period prior to the heat treatment following application of coatings, is critical and should be as short as possible. Once the internal damage – initiation of sub-microscopic cracks – has occurred, no subsequent heat treatment will be able to repair the damage already sustained. Evidence also exists that higher temperature, around 220°C, and longer duration, improve the effectiveness of the heat treatment.

There is also a widely held belief that hydrogen naturally diffuses out of conversion coated or porous plated steel article. Tables 1 and 2 show that the storage of phosphated items at room temperature did not remove hydrogen from the items.²⁶ Even after one year of storage at room temperature, the hydrogen content appeared to be similar in the steels. Additionally, all the components with higher hydrogen content, without heat treatment as well as with shorter duration and lower temperature, failed in service. Furthermore, failed components, which were heat treated at 210°C for 12 hours, were found to contain about 1 ppm or less of hydrogen. Failure of components in service was not observed when the duration of heat treatment was increased to 24 hours and the hydrogen content was further reduced.

Table I Results of hydrogen analysis of phosphated components		
	0.35 C low steel	0.45 C low alloy steel
Tensile strength, MPa	1000 to 1300	1000 to 1300
After phosphating, ppm	7.3	7.1 to 7.4
After 1 year storage at room temperature	7 to 9	7

Table II Results of post-phosphating heat treatment vs hydrogen analysis of phosphated components					
0.3C low alloy steel, Tensile strength 1440 to 1800 MPa					
	No heat treatment ^A	100 ⁰ C 5 h ^A	180 ⁰ C 10 h ^A	210 ⁰ C 12 h ^B	220 ⁰ C 24 ^C
Hydrogen content ppm	6.5 to 10	6 to 6.5	3.7 to 4.6	0.8 to 1.0	0.5
A : Parts failed in service B : Hydrogen content after failure of parts C : No failure reported in service					

Similar belief that the application of surface barrier, such as a flash coating followed by a thicker coating, would prevent ingress of hydrogen into the metal, also does not appear to work, considering that around 0.1 ppm of hydrogen may be required to cause hydrogen embrittlement problem.

Hydrogen embrittlement is known to involve a time dependent diffusion process. For steels containing hydrogen in which the tensile test is performed sufficiently slowly the hydrogen has time to diffuse to areas, such as grain boundaries, where the steel can become embrittled causing premature failure. Thus, a slow strain rate test method^{27,28} can be used to assess the severity of

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embrittlement. ASTM 200 hour sustained load tests have also been widely used. However, there have been reports of failure as well, of test specimens immediately after the completion of 200-hour tests. Additionally, the results of hydrogen embrittlement tests exhibit considerable scatter, possibly, due to various dimensions, nature and sites of stress raising defects which lead to crack initiation. Depending on the varied range of statistical techniques, the test methods, such as delayed failure, uniaxial tensile test, fracture mechanics, bend test etc., can show a steel component to be either compatible or very susceptible to hydrogen embrittlement. Furthermore, successful testing of coupons or specimens manufactured from a so-called metallurgically equivalent material may not assure satisfactory component. Because of the complexity of the problem, the results of research work differ considerably from batch to batch of the same material, from material to material, from laboratory to laboratory and even from one test to another. As a consequence, it is considered that there is no standard test method available to guarantee complete freedom from the susceptibility or the degree of susceptibility to hydrogen degradation. Considerable research work has been carried out in the last 40 – 50 years producing inconclusive and often confusing results.

Development of ISO Standards

During the development of the ISO Standards,^{29,30} it was noted that at least fifty or so stress relief and embrittlement relief treatments were used commercially, as well as invoked in many countries national standards and regional specifications. The treatments centered on various tensile strength levels, varying thicknesses of parts, a plethora of temperature ranges – from heating in boiling water to above the tempering temperature of the material – and varying duration periods of the embrittlement relief treatments. Therefore, it was deemed relevant to simplify these parameters, following nearly two decades of discussion with various organizations, establishments and countries as well as with commercial processors.

However, to avoid ambiguity and regional variations in terminology, it was considered essential, at first, to harmonize some of the definitions of terms used in the ISO Standards. As a result, following definitions were commonly agreed:

“Hydrogen embrittlement: type of embrittlement of a metal or alloy caused by absorption of atomic hydrogen, for example, during electroplating, autocatalytic plating, cathodic cleaning, or pickling, and manifested by delayed fracture, brittle fracture or a reduction of ductility, in the presence of tensile stress, either externally applied and/or internal residual stress”.

“Stress relief heat treatment (SR): thermal process carried out over a temperature range and for a duration of time such that no alteration of metallurgical structures, such as recrystallization, of the basis metal occurs, but at which stress relief of the parts to be plated is achieved”.

“Embrittlement relief heat treatment (ER): thermal process carried out over a temperature range and for a duration of time such that no alteration of metallurgical structures, such as recrystallization, of the basis metal occurs, but at which embrittlement relief of the plated articles is achieved.”

It is generally agreed that the degree of embrittlement may vary over a range in a given lot and is a function of the concentration of atomic hydrogen in each part within the lot. If hydrogen entry or charging follows a normal distribution curve, then it may be possible for varying amount of mobile hydrogen to remain in items, for example, in barrel plating processes. Hence, it is not possible to guarantee that all the items in a barrel will be completely free from hydrogen embrittlement. Consequently, the ISO Standards suggest that the heat treatment procedures, which shall be deemed to commence at the time at which the whole of each part attains the specified temperature, do not guarantee complete freedom from the adverse effects of hydrogen degradation.

The removal of residual or induced stresses due to cold forming, straightening, grinding, machining etc., is essential to deter mobile hydrogen, generated during cleaning or surface preparation processes, from migrating to the areas of high stress. Consequently, for pre-coating stress relief (SR), the heat treatments originally centered on four tensile strength levels: 1000 MPa to below 1400 MPa, 1400 MPa to below 1800 MPa, and from and above 1800 MPa, and for surfaced hardened parts below 1400 MPa. However, because of contractual requirements specified in the “traditional treatments” in some countries’ national standards, additional classes in the categorization of steels were included in the document.²⁹

For the pre-coating stress relief heat treatments, the temperatures were originally specified in the range of $210^{\circ}\text{C} \pm 10^{\circ}\text{C}$ for a minimum period of 3 hours for 1000 MPa to below 1400 MPa, minimum period of 18 hours for 1400 MPa to below 1800 MPa and

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minimum 24 hours for 1800 MPa and above, tensile strength. Minimum duration of 8 hours, at 130°C to 160°C, was required for surface hardened parts of tensile strength below 1400 MPa. The existing disparities in the temperature range and the minimum duration period incorporated in ISO 9587 also relate to the “traditional treatments” referred to in the national standards of some countries.

Combinations of shorter time at appropriate higher temperatures were also encouraged provided that they were shown not to be detrimental. However, if stress relief heat treatment was given after shot peening in accordance with ISO 12686 31, or other cold working processes, to introduce beneficial compressive stresses, the heat treatment temperature would be restricted to 230°C. Additionally, for tempered steels, the maximum temperature of heat treatments, for both pre- and post – coating procedures, was limited to at least 50°C below the tempering temperature.

The removal of hydrogen, absorbed during cleaning, preparation and coating processes, is also essential to reduce the effects of hydrogen degradation. It was observed that, for the post coating heat treatments, the effectiveness of the heat treatment tends to fall off rapidly with the reduction of time, the relationship being effectively linear and veered toward, at least, 24-hour period. As a result, consideration was given to calculate the minimum duration of heat treatment according to a simple formula, $t = 0.02R_m - 12$, where t is the minimum duration, in hours, and R_m is the actual tensile strength, expressed in megapascals.

Therefore, as long as it is possible to identify the actual tensile strength, or of hardness value which can be converted to tensile strength, of a particular article, the time for post coating heat treatment can be calculated easily. Alternatively, from the table given in the standard, the duration of heat treatment may be estimated according to the appropriate ER (embrittlement relief) classes and tensile strength. Appropriate reduction in temperature for articles, which would suffer unacceptable reduction in hardness because of heat treatments in accordance with the table given in the standard, was also incorporated. Classes for unpeened items and for engineering chromium plated articles, as well as for parts of greater than 25 mm thick and articles with threads or sharp notches, had been added. The low temperature heat treatment procedures, 210°C ± 10°C, thus established³⁰ appear to be effective in reducing the susceptibility to hydrogen embrittlement, although it is recognized that the procedures do not guarantee complete freedom from the adverse effects of hydrogen embrittlement.

Again the inconsistencies in incorporating additional classes, tensile strengths, duration and temperatures of heat treatments in ISO 9588 relate to the “traditional treatments” referred to in some national standards. It is hoped that in the process of time these inconsistencies will gradually fade away.

Additional classes, such as SR = 0²⁹ and ER = 0³⁰, were added to provide purchasers with a choice of specifically stipulating no heat treatment of the parts. Furthermore, in absence of any heat treatment procedure in the drawing or contract, or if the processor was not in possession of the necessary information, then, according to ISO 9588, the articles would be treated as if classes designating SR = 1 or ER = 1, as appropriate, was specified.

In ISO 9588, the post coating heat treatment temperature was limited to 220°C because of the possibility of toxic gases evolving from cadmium plated articles at or around 230°C and formation of blisters when zinc electroplated parts were subjected to the embrittlement relief heat treatment around 230°C (Fig. 3). Additionally, the delay period between the end of the plating cycle and the commencement of the heat treatment was specified as to be as short as possible, preferably within 1 hour but not later 3 hours.



Figure 3 - Formation of blisters on zinc plated parts, subjected to embrittlement relief heat treatment around 230°C for 24 hours.

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To determine the effectiveness of the embrittlement relief heat treatments, test methods, such as ISO 10537³¹ and ISO 15724³², are being developed. Freedom from failure of articles or test samples may demonstrate the effectiveness of the heat treatment procedures, depending on the number of samples that are tested.

The inclined wedge method³¹ was initially developed at the IBM Corporation³³ as an effort to refine the Flat Plate test of MIL-STD-1312. In this test method, the test samples, after hydrogen embrittlement heat treatment, are subjected to stress by tensioning with a mating nut after insertion through a clearance hole, in a hardened rectangular wedge of steel. Varying lengths of the threaded articles are accommodated with filler plates. The articles are torqued to the desired value, then held for a minimum specified number of hours and checked to determine if the initial torque has been maintained. They are then examined for embrittlement failures. The greater the test hours used, the higher the confidence level that can be assumed. The test method can be used for acceptance test when used with a sampling plan which is also specified in the ISO Standard.

ISO 15724³² describes a method in which hydrogen containing steel part, after the coating is removed, is made the anode in an electrochemical cell containing an alkaline solution as the electrolyte, with a nickel-nickel oxide electrode forming the cathode. The anodic current measured as a function of time is attributed to the concentration of mobile hydrogen. This technique is an adaptation of the electrochemical permeation method for hydrogen diffusion.

This method does not measure actual hydrogen concentration and it is assumed that the hydrogen is uniformly distributed throughout the steel. It is limited to flat specimens of carbon and alloy steels, excluding austenitic stainless steels. The method may be used as a quality control procedure as it provides a quick means of measuring the effectiveness of pre- and/or post-coating heat treatments, or of monitoring hydrogen uptake during plating or due to corrosion.

Many of the International Standards related to metallic coatings now contain clauses seeking information from the purchaser, as a mandatory requirement, related to the tensile strength of the parts, requirements for stress and embrittlement relief heat treatments, as well as requirements for hydrogen embrittlement tests to determine the effectiveness of the heat treatment procedures.

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