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### Possibility of Hydrogen Embrittlement Occurrence After Cadmium Plating According to Industry-Specific Standards

# Małgorzata Rutkowska-Gorczyca<sup>1\*</sup>, Mateusz Jaroszewski<sup>2</sup>, Saule Abdulina<sup>3</sup>

<sup>1</sup>Faculty of Mechanical Engineering, Wroclaw University of Science and Technology, Wroclaw, Poland 
<sup>2</sup>Scientific Circle Named After R. Haminn, Faculty of Mechanical Engineering, 
Wroclaw University of Science and Technology, Wroclaw, Poland 
<sup>3</sup>D.Serikbayev East Kazakhstan Technical University, Ust-Kamenogorsk, Kazakhstan

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#### **ABSTRACT**

Hydrogen embrittlement is a complex phenomenon that remains difficult to systematize. Despite years of research, the impact of hydrogen on metallic materials is still not fully characterized. Highstrength steels are particularly vulnerable, which has serious implications for the aerospace, automotive, shipbuilding, and energy industries. With increasing global focus on climate neutrality, hydrogenbased technologies are gaining attention, further highlighting the need for materials that combine mechanical strength with resistance to hydrogen-induced degradation. Hydrogen ingress can occur at nearly every stage of manufacturing, making planned and systematic research into hydrogen embrittlement essential for quality control. This study examines the susceptibility of AISI 4340 high-strength steel to hydrogen embrittlement after cadmium electroplating at three cathodic current densities: 0.7 A/dm<sup>2</sup>, 2.25 A/dm<sup>2</sup>, and 7.5 A/dm<sup>2</sup>. Twelve notched specimens underwent sustained load tensile (SLT) testing over 200 hours. Surface morphology was characterized using scanning electron microscopy (SEM), and fracture surfaces were analyzed for embrittlement features. Results showed significant morphological differences in the cadmium coatings and confirmed brittle fracture zones across all conditions. The study highlights the role of current density in influencing coating integrity and susceptibility to embrittlement. The combination of mechanical and fractographic analysis provides valuable insight into how embrittlement initiates and progresses. Findings confirm that cadmium plating, regardless of current density, promotes hydrogen embrittlement in AISI 4340 steel. Thus, precise control of electroplating parameters is critical to minimize risk in industrial applications.

<sup>1\*</sup> Corresponding author. E-mail address: malgorzata.rutkowska-gorczyca@pwr.edu.pl https://doi.org/10.24191/jmeche.v22i3.5668

#### INTRODUCTION

In connection with the current pursuit of climate neutrality, the concept of hydrogen and hydrogen fuels as energy carriers of the future is developing. Hydrogen is considered the key to implementing changes in energy policy. The pace of these changes is causing a rapidly growing demand for research on the impact of hydrogen on metallic materials (Jürgensen & Pohl, 2023; Sozańska, 2006; Yu et al., 2013). Due to the small mass and size of the hydrogen atom, hydrogen easily penetrates the crystal lattice of metals, causing changes in their microstructure. The hydrogen-induced change in microstructure directly affects the material's properties. One of the effects of such hydrogen interaction is the phenomenon of hydrogen embrittlement. Hydrogen embrittlement is a process of reducing plastic properties (such as necking and elongation) or the material's resistance to cracking, which occurs because of contact with atomic hydrogen (Caskey, 1977; Lai et al., 2013; Li et al., 2020; Paatsch & Hodoroaba, 2002; Saborío-González & Roias-Hernández, 2018; Song & Curtin, 2013; Symons, 2001). The effects of hydrogen embrittlement in structural materials are one of the most common causes of machinery and equipment failures. High-strength steels are the materials most susceptible to the destructive effects of hydrogen. Failures can occur within relatively short time intervals, resulting in enormous time and financial losses in industries such as chemical, shipbuilding, or nuclear energy. Despite the catastrophic consequences that hydrogen embrittlement can cause, this issue is rarely discussed outside of aerospace or advanced engineering journals. Despite numerous studies, the phenomenon of hydrogen embrittlement has not been fully resolved both theoretically and practically. The complexity of the problem, due to the variety of factors influencing hydrogen interaction and the different working conditions of materials, makes it difficult to determine a universal method for studying hydrogen embrittlement (Miller et al., 2021; Caskey, 1977; Churchill et al., 2019; Rogers, 1968). The phenomenon of hydrogen embrittlement occurs because of contact between metal and hydrogen, when individual hydrogen atoms penetrate the surface of the metal. At high temperatures, the solubility of hydrogen increases, facilitating its diffusion into the material's structure. Individual hydrogen atoms in the metal gradually transform into hydrogen molecules, creating internal pressure within the metal. This pressure can rise to a level that reduces the material's plasticity, hardness, and tensile strength, eventually leading to hydrogen-induced cracking (HIC) (Das et al., 2020; Das et al., 2021; Khanchandani & Gault, 2023; Moshtaghi & Safyari, 2023; Pietkun-Greber & Janka, 2010; Pietkun-Greber, 2018; Safyari et al., 2023; Safyari et al., 2024). Hydrogen can penetrate metallic materials at various stages of production, processing, and use, especially in electrochemical, high-temperature processes, and in corrosive or high-pressure environments. Hydrogen absorption leads to hydrogen embrittlement, which poses a significant threat to the mechanical strength of metals and their long-term durability (Kengesbekov et al., 2022; Rakhadilov et al., 2024; Toleuova et al., 2020).

One classification of hydrogen embrittlement is the division into external and internal hydrogen embrittlement. External embrittlement occurs under the influence of applied external load. During such loading, there is a possibility of defects occurring on or near the surface. Internal embrittlement occurs under the influence of internal stress. Additionally, hydrogen activity can be classified as strong or weak. Strong activity occurs when the volume of hydrogen that can be introduced into the material is significant. Weak activity refers to cases where the hydrogen volume is negligible (Beachem, 1972; Gerberich et al., 1988; Komatsu et al., 2021; Liu et al., 2023; Pietkun-Greber, 2018; Sofronis & Birnbaum, 1995; Mohamad Nor Azli et al., 2021; Tenckoff, 1988; Yeshiwas & Krishniah, 2021). Currently, companies use standards created by ASTM International (American Society for Testing and Materials). There are several standards related to identifying hydrogen embrittlement; however, the most used is the ASTM F519-18 standard - Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments. The aim of this work is to determine the suitability of research methods for detecting hydrogen embrittlement in metallic materials in the context of materials science research. The scope of the work includes the analysis of relevant standards, the assessment of product quality, and the conducting of pilot tests on hydrogenated elements, considering materials science research.

#### METHODOLOGY AND RESEARCH MATERIAL

The subject of the study was cadmium-coated notched samples (Fig 1) made of AISI 4340 steel without the dehydrogenation process. The AISI 4340 steel was supplied as standard samples according to ASTM F519 (Table 1). AISI 4340 steel is a high-strength alloy steel, commonly used in the aerospace, automotive, and defense industries. This steel is used for classification studies of electrochemical processes and the potential for hydrogen embrittlement after quenching. The material, prepared as samples for strength testing, exhibited a distinct texture after machining.

The samples were subjected to a cadmium coating process using various cathodic current densities (Table 2). For each selected current density level, four samples were prepared and tested, in accordance with the requirements of the ASTM F519 standard. Standardized samples were subjected to strength tests for the potential occurrence of hydrogen embrittlement. The SLT (Sustained Load Test) procedure, which is the most widely used testing method and is suited to the selected sample type, was applied in the conducted tests.

The testing of the samples involved a creep test to apply tensile stresses equal to 75% of the notched breaking strength. The test result is considered positive if none of the 4 tested samples fail within 200 hours. If one sample fails, the remaining 3 test samples must be further loaded in three stages after 200 hours, with each stage lasting 2 hours and the load increased to 80%, 85%, and 90% of the notched breaking strength, respectively. If no sample fails, the test result is also considered positive, and the process is qualified as not causing hydrogen embrittlement (Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments, 2018). An analysis was performed on the samples that did not pass the strength test, which, according to the standard, indicates the occurrence of hydrogen embrittlement.

Fractographic studies of the fractures and the registration of the coating morphology were conducted using a Phenom ProX scanning electron microscope (SEM) with SED and BSE detectors.

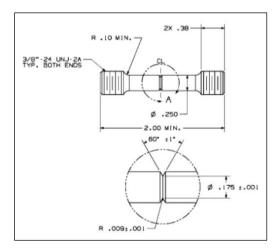


Fig. 1. Dimensional requirements for round notched tensile samples according to ASTM F519-18 standard. Values in inches

Table 1. Strength parameters of AISI 4340 steel

Tensile strength	Yield strength	Herdness	Elongation	Modulus of elasticity
930-1080 MPa (hardened), up to 1400-1600 MPa (heat-treated)	470-725 MPa (hardened), up to 1200 MPa (heat- treated)	55 HRC	12-15%	200 GPa

Table 2. Cathodic current density parameters used during the cadmium plating process

	Low	Medium	High
I [A/dm <sup>2</sup> ]	0.7	2.25	7.5

#### RESULTS AND DISCUSSION

To comprehensively evaluate the quality of cadmium coatings and their susceptibility to hydrogen embrittlement, surface morphology and fracture characteristics were investigated using scanning electron microscopy (SEM). The study focused on the effect of varying cathodic current densities on coating structure and failure mechanisms in cadmium-plated high-strength steel. Morphological analysis enabled the identification of surface features that changed with plating parameters, while fractographic observations revealed the presence of brittle fracture zones, indicative of hydrogen-induced damage. These complementary studies provide critical insight into how processing conditions influence both the integrity of the coating and the underlying substrate's mechanical response. The observations were consistent across four replicate samples for each current density tested, as required by ASTM F519. This increases the interpretative depth of the results and more closely aligns the discussion with established HE mechanisms such as HELP and HEDE.

#### **Examination of Cadmium Coating Surfaces**

To analyse the differences in coating morphology produced at different current density values, scanning electron microscopy (SEM) methods were applied. Surface examinations of the samples before the cadmium electroplating process revealed the presence of surface topography from the finishing treatment of the samples made according to the standard. Parallel lines from the grinding process are visible on the surface (Fig 2). Surface studies of cadmium-coated samples showed varied surface morphology depending on the applied current density. Coatings formed at the lowest current density of 0.7 A/dm<sup>2</sup> exhibited a finegrained, granular structure. The resulting crystallites are approximately 15 μm in size and show a polygonal grain structure (Fig 3), reflecting the surface of the substrate material. Increasing the current density to 2.25 A/dm<sup>2</sup> also produced crystallites of a similar size. The previously revealed texture of the steel surface in its supplied state is also visible (Fig 4). The coating reflects the substrate's surface topography, as evidenced by the width of the lines present. The morphology shows a more irregular grain shape. Applying a high current density of 7.5 A/dm<sup>2</sup> resulted in the most irregular morphology of the cadmium coating (Fig 5). The study revealed the formation of flake-like cadmium deposits. These flakes form both the matrix, consisting of fine flake precipitates, and larger precipitates with a diameter of approximately 30 µm. The large flakes protrude above the coating surface, in some areas separating and bending. The irregular morphology of the coating and its flake structure may reduce the parameters of sealing and corrosion resistance. The morphology may also affect the ability of hydrogen, stored during electrolytic processes, to escape during heat treatment in a vacuum atmosphere.

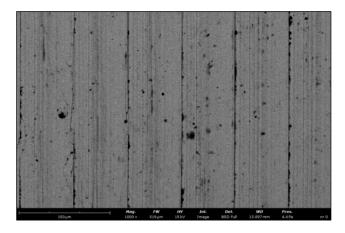


Fig. 2. Surface of AISI 4340 sample in the as-received condition.

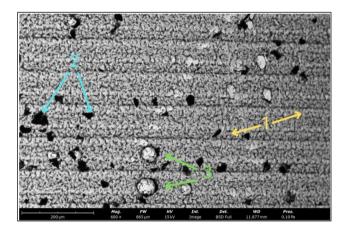


Fig. 3. Surface of the cadmium coating of the sample created at  $0.7 \text{ A/dm}^2$  - (1) Visible parallel bands approximately  $100 \mu m$  wide (2) unevenly distributed porosities and (3) coagulated precipitates.

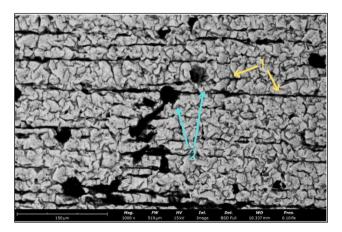


Fig. 4. Surface of the cadmium coating of the sample created at  $2.25~\text{A/dm}^2$  - (1) Visible developed surface texture of the coating with irregularly spaced bands and (2) contaminants approximately  $100~\mu m$  in length. https://doi.org/10.24191/jmeche.v22i3.5668

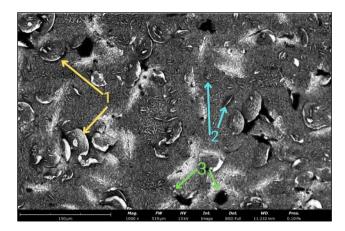


Fig. 5. Surface of the cadmium coating of the sample created at 7.5 A/dm² - (1) Visible embedded flake-like areas approximately 50 μm in diameter (2) flake-shaped precipitates embedded in the coating matrix and (3) pores.

#### Fractographic Studies

To evaluate the nature of the fractures formed in cadmium-coated samples created at different current densities, studies were conducted using scanning electron microscopy (SEM) methods (Fig 6). In the asreceived samples, the fracture showed the presence of a ductile fracture, which is a typical response of this material under tensile loading (Fig 7). In contrast, in the fractures of cadmium-coated samples, both ductile and brittle areas were observed (Figs 8 - 10), indicating the presence of mixed-mode failure mechanisms.

These zones exhibited characteristic features of cleavage or intergranular fracture, commonly associated with hydrogen embrittlement. Importantly, fractographic analysis revealed that brittle fracture areas were present in all tested samples, regardless of the applied cathodic current density. This consistent occurrence strongly suggests the diffusion and localization of hydrogen within the material microstructure during or after the plating process. The presence of brittle zones, even in cases where mechanical testing may not indicate failure, serves as a primary microstructural indicator of hydrogen embrittlement. In high-strength steels such as AISI 4340, hydrogen tends to be localized at internal stress concentration sites, such as inclusions, grain boundaries, or surface defects introduced during electroplating. These microstructural anomalies act as initiation points for crack propagation under reduced ductility, leading to premature fracture in otherwise ductile materials.

From a diagnostic perspective, the identification of brittle fracture morphology, such as river patterns, transgranular or intergranular cracks, and quasi-cleavage surfaces, offers compelling evidence of hydrogen activity within the steel matrix. As such, the presence of brittle areas is not merely a supplementary observation but rather a critical tool for verifying the occurrence of hydrogen embrittlement. Their appearance, even in the absence of complete sample failure, underscores the limitations of mechanical-only assessment methods, such as those defined in ASTM F519-18, and highlights the indispensable role of fractography in comprehensive material evaluation. The observations of brittle fracture features were consistent across all replicate samples, confirming the repeatability and strengthening the reliability of the findings. Although microscopic analysis is inherently qualitative, the coherence between the observed fracture morphologies and the expected effects of hydrogen exposure clearly reinforces the validity of the results. The logical alignment of these findings with well-established mechanisms of hydrogen embrittlement confirms their interpretative accuracy and highlights the essential role of fractographic analysis in evaluating hydrogen-induced damage.

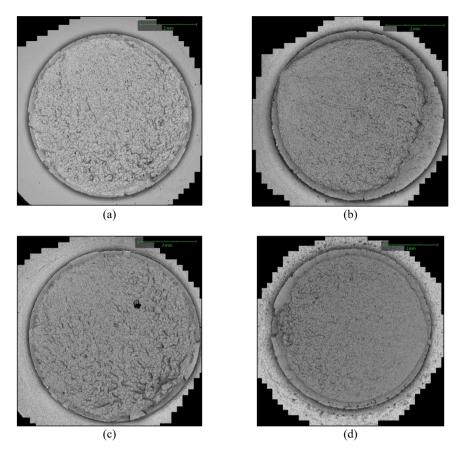


Fig. 6. Macroscopic image of the fracture surfaces of the samples (a) as-received condition (b) with coating created at  $0.7 \text{ A/dm}^2$  (c) with coating created at  $2.25 \text{ A/dm}^2$  and (d) with coating created at  $7.5 \text{ A/dm}^2$ .

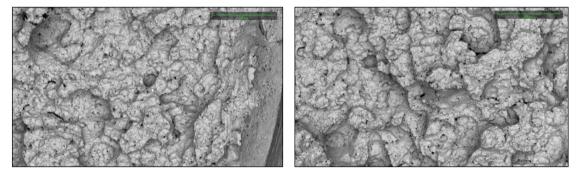


Fig. 7. Fracture surface of the sample without coating. Visible ductile fracture.

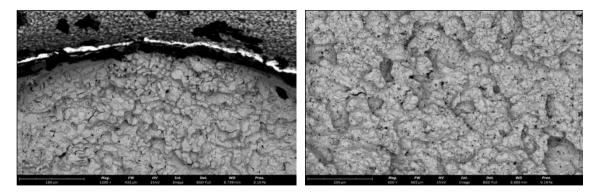


Fig. 8. Fracture surface of the sample with coating created at 0.7 A/dm². Both ductile and brittle fractures are visible.

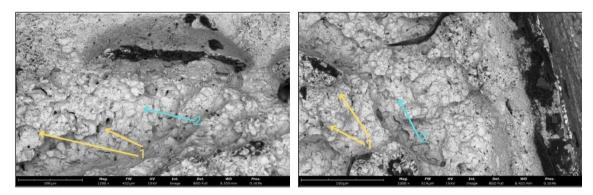


Fig. 9. Fracture surface of the sample with coating created at  $2.25 \text{ A/dm}^2$  - (1) Visible ductile area and (2) small brittle

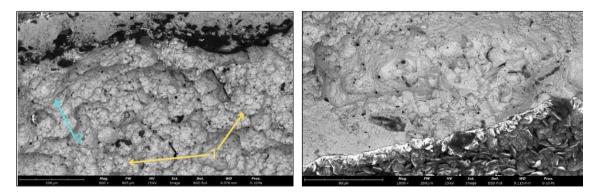


Fig. 10. Fracture surface of the sample with coating created at  $7.5~\text{A/dm}^2$  - (1) Visible ductile area and (2) small brittle area.

#### CONCLUSIONS

This study provides a comprehensive investigation into the susceptibility of cadmium-plated AISI 4340 high-strength steel to hydrogen embrittlement (HE), with particular emphasis on the influence of varying cathodic current densities during the electroplating process. By examining both surface morphology and fracture characteristics, the research offers valuable insights into how processing parameters affect the structural integrity of electroplated components and their exposure to hydrogen-induced degradation.

A key observation was the consistent presence of brittle fracture zones in all coated samples, regardless of the applied current density. These zones were especially prominent near the outer surface in the shear region, an area most exposed to electrolyte interaction, indicating site-specific hydrogen accumulation (Au, 2003; Caskey, 1977; McMahon, 2001). This observation provides direct microstructural evidence of HE, validating the interpretation that brittle areas are not incidental, but rather diagnostic of hydrogen ingress. Such zones are indicative of crack initiation due to hydrogen trapping at microstructural defects like dislocations and inclusions.

The implications are significant: these brittle regions, even when not leading to full mechanical failure, undermine the ductility and reliability of high-strength steels. Their consistent occurrence reinforces the argument that reliance solely on mechanical testing, as stipulated in ASTM F519-18, may fail to capture early signs of hydrogen damage. As such, integrating fractographic analysis into standard testing protocols is essential for a complete assessment of hydrogen susceptibility.

Another crucial finding pertains to the relationship between surface morphology and HE. High cathodic current densities resulted in flake-like, non-uniform coatings that disrupt the barrier effect of the cadmium layer and facilitate hydrogen entrapment. While a quantitative correlation between morphology and HE was not established in this study, the visual and structural evidence suggests a strong qualitative link. These irregularities can act as preferential hydrogen entry points and inhibit desorption, particularly in post-processing environments lacking heat treatment.

Furthermore, the absence of a dehydrogenation step in this study allowed an unmitigated evaluation of hydrogen's effect. However, this also limits direct comparison with industrial practices that incorporate post-baking. Future work should explore the mitigating influence of such treatments and include permeability tests and hydrogen mapping to better define the relationship between coating parameters and HE progression (Barth & Steigerwald, 1970; Miller et al., 2021; Voort, 1990; Oriani, 1978; Park et al., 2024; Trautmann et al., 2020; Westlake, 1969).

In summary, this research confirms that cadmium electroplating, especially under high current densities, can promote microstructural conditions conducive to hydrogen embrittlement. It emphasizes the need for multi-method diagnostics, including fractography, to ensure the structural integrity of critical components. These findings contribute to a more refined understanding of HE and advocate for updates to current standards and engineering protocols in hydrogen-sensitive applications.

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#### CONFLICT OF INTEREST

The authors declare no conflicts of interest.

#### **AUTHOR CONTRIBUTIONS**

Conceptualization, M.R.-G., Methodology, M.R-G., Investigation, M.R-G., M.J., S.A.; Writing—review & editing, M.R.-G.; Visualization, S.A.; Supervision, M.R.-G. All authors have read and agreed to the published version of the manuscript.

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