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IMPACT OF HYDROGEN CHARGING TECHNIQUES ON HYDROGEN EMBRITTLEMENT BEHAVIOR OF S960MC AHS STEEL

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Abstract: The study explores the influence of hydrogen embrittlement on advanced high-strength steel S960MC, focusing on the role of different hydrogen charging techniques. Hydrogen embrittlement poses a critical challenge in high-strength steels, compromising their structural integrity and limiting their applications in demanding environments. The findings indicate that S960MC steel demonstrates intrinsic resistance to hydrogen embrittlement when exposed to a hydrogen-supersaturated environment without external factors like electric current or elevated temperature. However, cathodic hydrogen charging significantly enhances hydrogen diffusion into the material, leading up to a 60% decrease in ultimate tensile strength. In contrast, immersion hydrogen charging showed a minimal effect on the mechanical properties. Fractographic analysis showed that cathodic charging led to severe embrittlement, characterized by mixed transcrystalline quasi-cleavage, intercrystalline fractures, and extensive secondary cracking. Conversely, immersion charging resulted in negligible embrittlement, with minimal changes in fracture morphology. These results highlight the critical role of hydrogen charging methods in the embrittlement behavior of S960MC steel, emphasizing the substantial impact of cathodic charging on material degradation.
Keywords: hydrogen charging, high-strength steels, cathodic hydrogen charging, immersion hydrogen charging

1. INTRODUCTION

Hydrogen embrittlement (HE) results from a series of complex processes whose nature varies depending on the metal type and external factors, such as temperature, exposure to hydrogen environments, and mechanical stress. Numerous theoretical mechanisms have been proposed to explain the interaction between hydrogen and metal host, however, no consensus has been reached regarding a single, universal mechanism. In recent years, it has become increasingly evident that multiple mechanisms can operate concurrently, contributing to the embrittlement process. Current theories generally categorize the HE mechanisms into three main groups: (i) mechanism that enhance plasticity (Lynch, 1988; Lynch, 2009; Huang et al., 2023), (ii) mechanisms that enhance decohesion (Troiano, 2016), and (iii) synergistic action and interplay of multiple HE mechanisms that combine both (i) and (ii) (Djukic et al., 2019).

The development of experimental techniques capable of measuring hydrogen concentration at specific nanoatomic sites is essential for advancing research in this field. These techniques should include in situ capabilities to monitor microstructural changes during hydrogen exposure, considering key factors such as concentration, exposure time, and temperature (Tunes et al., 2024). To effectively study and address this phenomenon, reliable hydrogen charging methods are also critical. The immersion-type method, although time-consuming, provides a simple approach with minimal equipment and ensures uniform hydrogen absorption. In contrast, the cathodic method is faster but requires power supplies and electrodes and may lead to inconsistent hydrogen absorption due to variations in current density (Takagi and Toji, 2012).

The present work investigates the HE behaviour of S960MC advanced high-strength steel, focusing on the effects of different hydrogen charging methods, looking at mechanical behaviour under various conditions. In order to do this, cathodic-and immersion hydrogen charging technique were used, comprising tensile testing immediately after hydrogen exposure and fracture surface analysis.

2. EXPERIMENTAL MATERIAL AND METHODOLOGY

2.1 Experimental material, metallographic analysis and tensile testing

The experimental material used in this study was advanced high-strength steel S960MC, provided as a 3 mm thick sheet. The steel underwent thermomechanical treatment, specifically cold rolling, to achieve its high-strength properties. The chemical composition of the material is provided in Table 1. The metallographic analysis was performed using a NEOPHOT 32 optical microscope.

Table 1

Chemical composition of S960MC steel [wt.%]

C	**Si**	**Mn**	**P**	**S**	**Al**	**Nb**	**V**	**Ti**	**Mo**	**B**	**Ni**	**Cr**
0.085	0.205	1.09	0.012	0.01	0.029	<0.004	0.014	0.019	0.012	0.003	0.06	1.17

Tensile testing was conducted using an Instron 5985 testing machine, in accordance with the STN EN ISO 6892-1 standard. The loading rate was set to 0.5 mm/min. The dimensions of the tensile test specimens are provided in Fig. 1. After testing, the fracture surfaces were examined to assess the failure modes and fracture characteristics using a TESCAN VEGA LMU II scanning electron microscope (SEM), enabling detailed fractographic analysis.

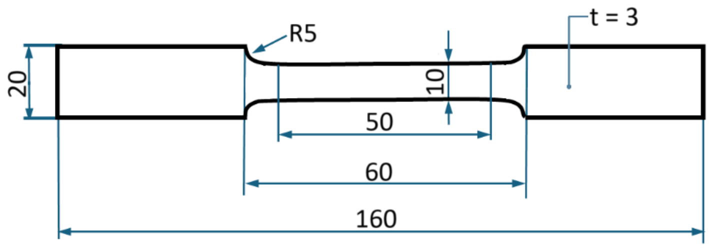

Fig. 1. Tensile test specimen [mm]

2.2 Cathodic hydrogen charging

The cathodic hydrogen charging of samples was performed using a 0.05 M H_2SO_4 solution with the addition of 1 g KSCN per liter of solution, prepared in accordance with ISO FDIS 17081. Prior to testing, specimens were degreased with acetone, rinsed with ethanol, and dried with warm air. Each prepared experimental specimen (no. 3) was mounted into the experimental setup (Fig. 2. a) no. 1 – DC source) and connected as the cathode while platinum electrodes, selected for their high hydrogen absorption capacity (approximately 100 times their volume), were connected as anodes (no. 4). Platinum anodes were securely mounted on a PTFE electrode body (no. 2), enhancing hydrogen permeation stability at temperatures up to 90°C, with the electrode distance from the specimen set to approximately 1.5 cm. All tests were conducted at a controlled laboratory temperature of 21°C. After hydrogen charging, specimens were rinsed in distilled water, dried with warm air and immediately subjected to tensile testing; therefore, the sample geometry was designed to accommodate both the charging and subsequent tensile testing requirements.

Table 2
Parameters for Cathodic Hydrogen Charging technique

Sample No.	**Current Density [A/cm²]**	**Current [A]**	**Voltage [V]**	**Temperature [°C]**	**Time [h]**
1	0.1	0.66	9	29.5	24
2					48
3					72

2.3 Immersion hydrogen charging

Before testing, specimens were cleaned with acetone, rinsed with ethanol, and dried using warm air. The hydrogen charging followed an immersion method (Fig. 2 b)) in a 20% ammonium thiocyanate (NH_4SCN) solution (no. 3), prepared by dissolving 500 g of ammonium thiocyanate in 2000 ml of distilled water. The solution volume was 500 ml per specimen, and the solution volume to specimen area ratio was 7.8 ml/cm². Specimens (no. 2), with dimensions corresponding to those used for tensile testing (as shown in Fig. 1), were mounted on a holder (no. 1) and immersed in the solution for 1 and 2 weeks at 21°C, with each specimen serving as the working electrode in the experimental setup following the procedure outlined by Takagi and Toji (2012). After immersion, the specimens were rinsed with distilled water to remove residual NH_4SCN, dried with warm air, and prepared for further testing. Immediately following hydrogen exposure, the specimens were subjected to tensile testing.

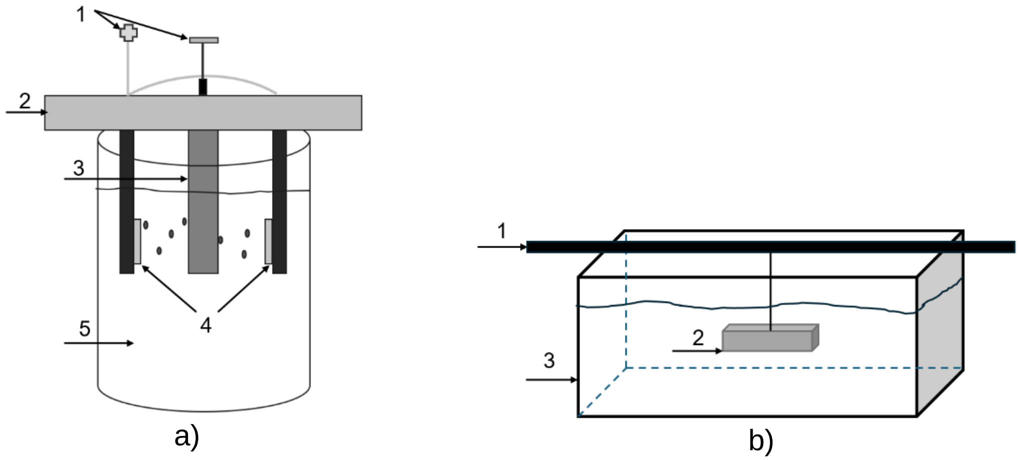

Fig. 2. Schematic procedure setups of hydrogen charging methods: a) cathodic and b) immersion

3. RESULTS AND DISCUSSION

3.1 Metallographic Analysis and Tensile testing

The metallographic analysis of S960MC steel in as-received state, observed in Fig. 3., revealed a fine martensitic microstructure. Sulphides, deformed during hot rolling, retained a flattened shape aligned with the rolling direction after solidification.

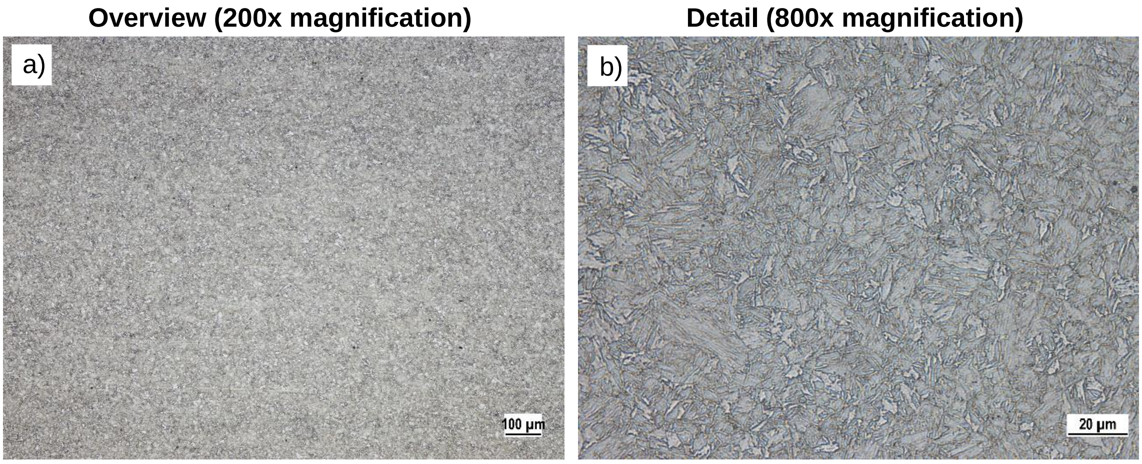

Fig. 3. As-received microstructure of S960MC steel observed using optical microscopy: a) martensitic microstructure overview; b) detail of a)

Cathodic hydrogen charging induced significant embrittlement in the material, as evidenced by the tensile test results presented in Table 3 and the stress-strain curve in Fig. 4 (left), when compared to the as received state (non-charged). Specimen no. 1 was charged for 24 hours, specimen 2 for 48 hours, and specimen 3 for 72 hours. The results clearly indicated significant reductions in both yield strength and ultimate tensile strength (UTS), with UTS decreasing by 35% for specimen 1, 38% for specimen no. 2, and up to 61% for specimen no. 3. Additionally, ductility was markedly reduced, characterized by a sharp decline in plastic deformation, while elastic deformation increased, indicating a significant loss of ductility primarily attributed to hydrogen embrittlement.

In contrast, immersion hydrogen charging had minimal impact on the mechanical properties of the test specimens (Table 3, Fig. 4). Specimen no. 1, exposed to hydrogen for 2 weeks, showed a modest 4.6% reduction in UTS, while specimen no. 2, charged for

1 week, experienced a 3.3% decrease in UTS compared to the as received state. Overall, immersion hydrogen charging caused negligible changes to the mechanical properties of the specimens.

Table 3
Mechanical properties after hydrogen exposure

	Specimen No. (charging time)	Yield Strength [MPa]	UTS [MPa]	E [%]
	As received	1034	1083	7.33
Cathodic hydrogen charging	1 (24 h)	691	705	5.2
	2 (48 h)	654	663	3.74
	3 (72 h)	425	426	2.81
Immersion hydrogen charging	1 (1 week)	1009	1047	7.51
	2 (2 weeks)	999	1033	6.56

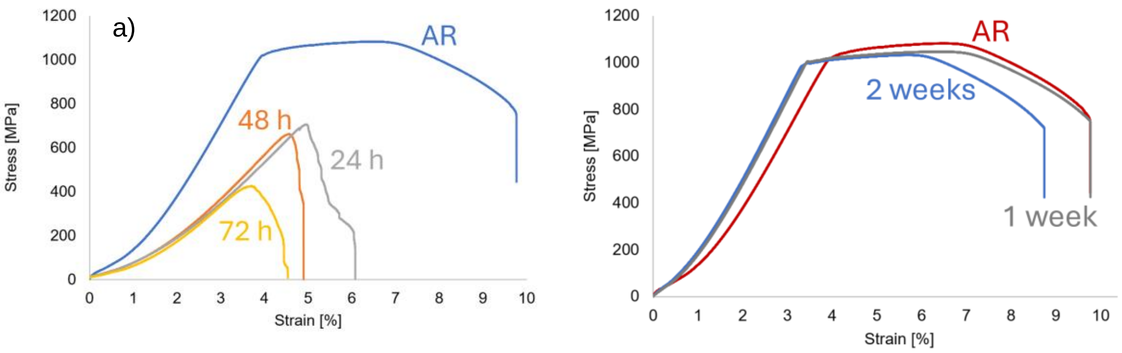

Fig. 4. Stress-Strain curves after: a) cathodic hydrogen charging; b) immersion hydrogen charging.

3.2 Fractographic analysis

The fracture surface after tensile testing of as received specimens exhibited a ductile character, characterized by a fine dimple morphology, as observed in Fig. 5 a)-b).

After 24 hours of cathodic hydrogen charging, the fracture surface exhibited a mixed fracture mode, with both transcrystalline and intercrystalline failure, along with the presence of secondary cracking (Fig. 5c). Additionally, a fish-eye morphology, indicative of inclusion-related fracture, was observed (Fig. 5d).

Even after two weeks of immersion hydrogen charging, no significant changes were observed on the fracture surfaces compared to the as received state. Fractographic analysis showed only minor changes, with no signs of brittle fracture characteristics, such as transcrystalline facets, intercrystalline fractures, or secondary cracking.

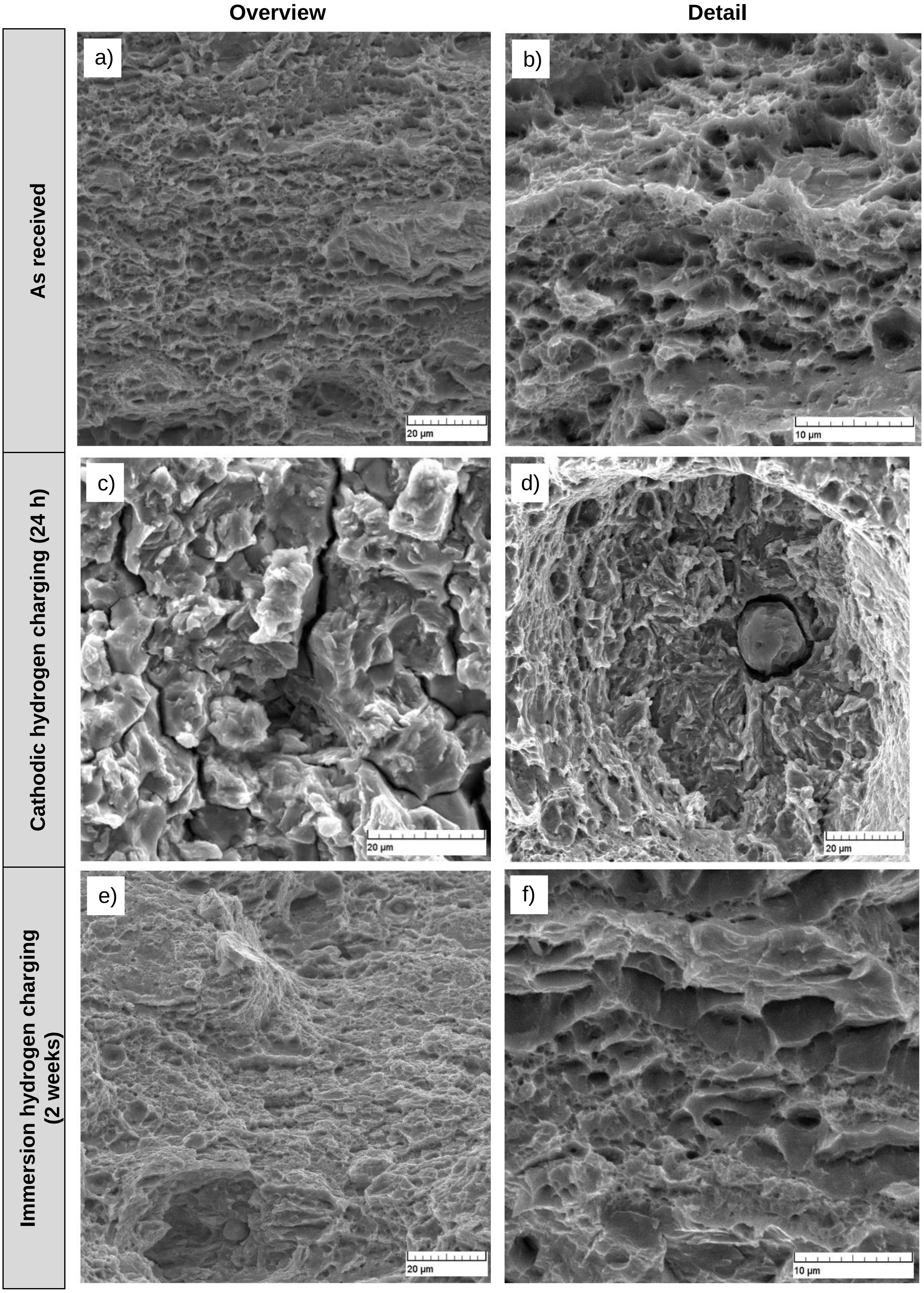

Fig. 5. SEM images of fracture surfaces after tensile testing: a)-b) as received; c)-d) cathodic hydrogen charged for 24 h; e)-f) immersion hydrogen charged for 2 weeks

4. CONCLUSIONS

In the present work, hydrogen embrittlement has been analysed through tensile testing and fractography analysis. Key conclusions are as follows:

- S960MC steel exhibited minimal sensitivity to a hydrogen-supersaturated environment without additional factors like electric current or elevated temperature, suggesting an intrinsic resistance to hydrogen embrittlement under non-catalysed conditions.
- Cathodic hydrogen charging markedly enhanced hydrogen diffusion in the steel, resulting in substantial embrittlement. This method caused a notable 60% reduction in mechanical properties, including tensile strength, yield strength, and ductility.
- Fractographic analysis showed minimal embrittlement in specimens subjected to immersion hydrogen charging, while cathodic charging led to pronounced embrittlement, characterized by mixed failure of transcrystalline quasi-cleavage and intercrystalline, and extensive secondary cracking. This fracture morphology further supports the embrittling effect of cathodic hydrogen charging.

The findings underscore the substantial susceptibility of S960MC steel to hydrogen embrittlement, predominantly via cathodic hydrogen charging, which significantly degrades mechanical properties and alters fracture behavior. This susceptibility highlights the need for further research into hydrogen embrittlement in differently processed samples, particularly focusing on the effects of hydrogen on prestressed and welded joints, as these investigations could ultimately contribute to improved safety and reliability in structural applications.

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REFERENCES

Djukic, M. B., Bakic, G. M., Zeravcic, V. S., Sedmak, A., Rajicic, B., 2019. *The synergistic action and interplay of hydrogen embrittlement mechanisms in steels and iron: Localized plasticity and decohesion*, Engineering Fracture Mechanics, 216, 106528, DOI: 10.1016/j.engfracmech.2019.106528.

Huang, L., Chen, D., Xie, D., Li, S., Zhang, Y., Zhu, T., Raabe, D., Ma, E., Li, J., Shan, Z., 2023. Quantitative tests revealing hydrogen-enhanced dislocation motion in α-iron, Nature Materials, 22(6), 710–716, DOI: 10.1038/s41563-023-01537-w.

Lynch, S., 2009. *Comments on "A unified model of environment-assisted cracking"*, Scripta Materialia, 61(3), 331–334, DOI: 10.1016/j.scriptamat.2009.02.031.

Lynch, S., 1988. *Environmentally assisted cracking: Overview of evidence for an adsorption-induced localised-slip process,* Acta Metallurgica, 36(10), 2639–2661, DOI: 10.1016/0001-6160(88)90113-7.

Takagi, S., Toji, Y., 2012. *Application of NH4SCN Aqueous Solution to Hydrogen Embrittlement Resistance Evaluation of Ultra-high Strength Steels*, ISIJ International, 52(2), 329–331, DOI: 10.2355/isijinternational.52.329.

Troiano, A. R., 2016. *The role of hydrogen and other interstitials in the mechanical behavior of metals,* Metallography Microstructure and Analysis, 5(6), 557–569, DOI: 10.1007/s13632-016-0319-4.

Tunes, M. A., Uggowitzer, P. J., Dumitraschkewitz, P., Willenshofer, P., Samberger, S., Da Silva, F. C., Schön, C. G., Kremmer, T. M., Antrekowitsch, H., Djukic, M. B., Pogatscher, S., 2024. *Limitations of hydrogen detection after 150 years of research on hydrogen embrittlement*, Advanced Engineering Materials, Volume, Issue, page number, DOI: 10.1002/adem.202400776.