Effect of Heat Treatment on the Hydrogen Embrittlement Response of Electroless Ni-P-Al_2O_3 Nanocomposite-Coated Steel

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PRESENTATION OUTLINE

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ii. Statement of the Problem
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v. Hydrogen Charging and Characterisation
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Background: Hydrogen embrittlement in steel

- Hydrogen can enter steel during processing or service and reduce ductility, promote premature cracking and compromise structural integrity.
- Hydrogen embrittlement susceptibility depends on the steel, hydrogen exposure conditions, stress state and surface condition.
- Protective surface coatings may reduce hydrogen entry, but coating integrity and thermal history can influence performance.
- Electroless Ni-P offers a relatively uniform deposit; Al_2O_3 incorporation is investigated as a route to modify coating properties.

Aim and specific objectives

Aim:
To evaluate how heat treatment influences the hydrogen-embrittlement response of steel protected with electroless Ni-P-Al_2O_3 nanocomposite coatings.

Objectives:

- Produce electroless Ni-P and Ni-P-Al_2O_3 coatings on low-carbon steel.
- Heat-treat the nanocomposite-coated specimens at selected temperatures.
- Compare tensile behaviour before and after hydrogen charging using a strain-based embrittlement index.
- Relate mechanical response to hardness, corrosion behaviour and coating/microstructural observations

Materials and coating deposition

- **Substrate:** Low-carbon steel recovered from decommissioned LPG cylinders. Approx. 0.15–0.25 wt.% C. Specimens prepared and ground/polished before deposition
- **Surface Preparation Sequence**: grind 240-1200 grit, acetone degrease, 10% HCl pickle, $SnCl_2$ sensitise, $PdCl_2$ activate
- Both baths: 30 g/L $NiSO_4 \cdot 6H_2O$, 20 g/L $NaH_2PO_2 \cdot H_2O$, 15 g/L sodium citrate, 0.2 g/L SDS
- **Nanoparticle addition:** : 3 g/L γ-Al_2O_3 and 2 mg/L lead acetate
- 85 ± 2 °C, pH 4.8 ± 0.2, 150 rpm, 60 min deposition









Specimen matrix and heat-treatment conditions

Sample ID	Condition
S1	Uncoated steel
S2	As-deposited Ni-P
S3	As-deposited Ni-P-Al_2O_3
S4	Ni-P-Al_2O_3, 200 °C, 1 h
S5	Ni-P-Al_2O_3, 300 °C, 1 h
S6	Ni-P-Al_2O_3, 500 °C, 1 h

Exposure: 20 wt.% NH_4SCN, 50 °C, 48 h, then SSRT

Hydrogen charging and characterisation

- **Charging condition:** 20 wt.% NH_4SCN solution; 50 °C; 48 h

Uncharged specimens served as the reference condition.

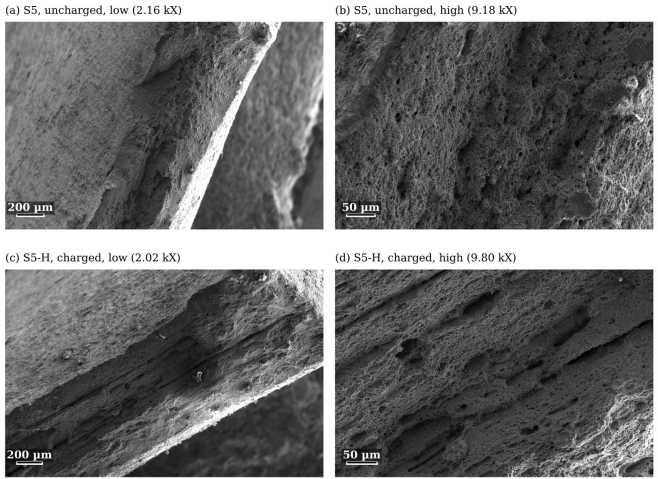
- **Mechanical response:** Slow strain rate tensile testing (SSRT); Tensile strength and elongation/strain response before and after charging.
- **Microhardness:** HV0.1

Immersion mass loss / corrosion rate

- XRD, SEM/EDS and fracture-surface examination
- **Strain-based embrittlement index:**

$$EI(\%) = \frac{\varepsilon_{uncharged} - \varepsilon_{charged}}{\varepsilon_{uncharged}} \times 100 \ ;$$

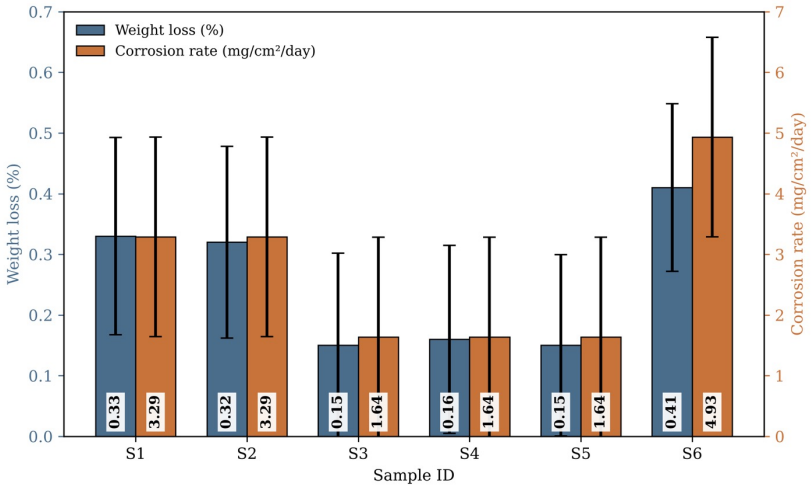
where ε is strain at break

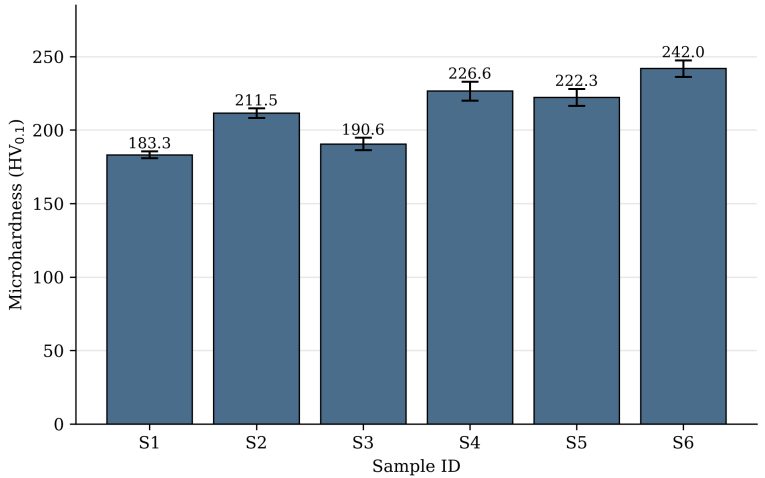


Positive EI = ductility lost. Negative EI = strain increased.

Hydrogen-embrittlement response

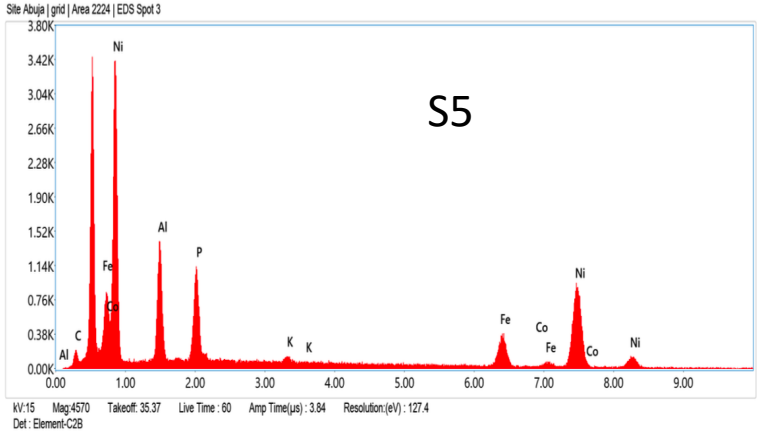
- **S5 embrittlement index,** EI = 1.26%
- A relatively small strain-based loss was recorded for the 300 °C furnace-cooled condition.
- The result is consistent with a comparatively favourable ductility-retention response under the applied charging condition
- The 300 °C Ni–P–Al_2O_3-coated steel (S5) emerged as a promising condition for mitigating hydrogen-induced ductility loss in this study.

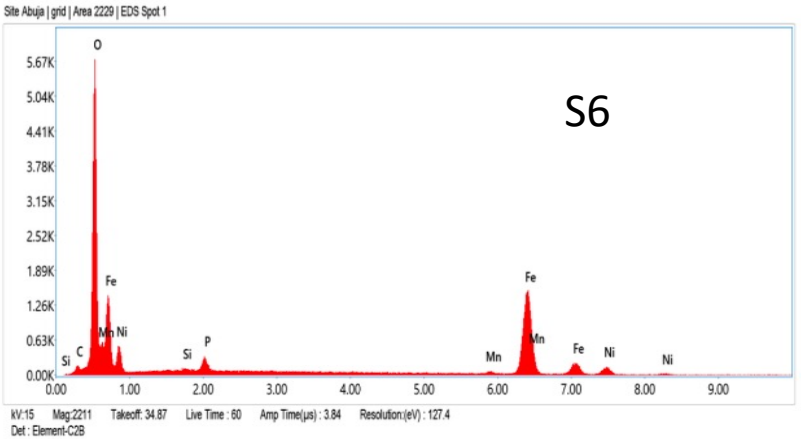




Microstructural and phase characterisation

- XRD was used to examine changes in the coating-related diffraction response following heat treatment.
- The strongest XRD response was observed for the 500 °C condition.
- SEM/EDS provided surface and compositional evidence, while fracture-surface examination supported assessment of tensile failure features.
- These observations were considered alongside mechanical and corrosion measurements rather than used as stand-alone proof of hydrogen blocking.





Hardness peaks at 500 °C

Sample	Hardness (HV0.1)	SD	Change vs S1
S1	183.26	2.35	—
S2	211.50	3.67	+15.41%
S3	190.62	4.30	+4.02%
S4	226.60	6.19	+23.65%
S5	222.34	5.56	+21.33%
S6	**241.96**	**5.50**	**+32.04%**

Hardness: S6 (500 °C) recorded the highest hardness: 241.96 HV0.1.

Higher hardness did not correspond automatically to the best overall response.

Mass loss / corrosion: S5 recorded a corrosion rate of 1.64 mg cm^{-2} day^{-1}, approximately 50% lower than the uncoated reference (S1).

S6 showed higher mass loss than S3–S5, despite its higher hardness.

The 300 °C condition offered a more balanced combination in the results.

S3–S5 lost about half the mass of uncoated steel

Sample	Mass loss (g)	Mass loss (%)	Rate (mg cm^{-2} day^{-1})
S1	0.02	0.33	3.29
S2	0.02	0.32	3.29
S3	0.01	0.15	1.64
S4	0.01	0.16	1.64
S5	0.01	0.15	1.64
S6	**0.03**	**0.41**	**4.93**

Balance resolution 0.01 g: interpret as a screening indicator, not a standardised corrosion rate

Tensile response: 300 °C gave the lowest ductility loss

Sample	UTS unexposed (MPa)	UTS exposed (MPa)	Strain at break unexp.	Strain at break exp.	EI (%)
S1	431.31	453.72	0.13626	0.13051	4.22
S2	455.96	498.63	0.15051	0.14292	5.04
S3	499.69	n/a	0.12459	n/a	n/a
S4	478.25	460.09	0.12503	0.12252	2.01
S5	**507.75**	**499.26**	**0.13417**	**0.13248**	**1.26**
S6	460.91	477.89	0.15127	0.16067	−6.21

- **S5: 300 °C,** Uncharged UTS: 507.75 MPa; Highest UTS among the coated conditions
- **S1: uncoated steel;** UTS: 431.31 MPa (uncharged); UTS: 453.72 MPa (after charging).

What the tensile results suggest:

- The 300 °C furnace-cooled nanocomposite condition (S5) showed a promising strength response.
- The uncoated steel showed a higher measured UTS after charging.

Integrated result: 300 °C is promising

Sample	Hardness (HV0.1)	Mass-loss rate (mg cm^{-2} day^{-1})	UTS unexposed (MPa)	EI (%)
S1 uncoated	183.26	3.29	431.31	4.22
S5 (300 °C)	**222.34**	**1.64**	**507.75**	**1.26**
S6 (500 °C)	241.96	4.93	460.91	−6.21

- Heat treatment altered the measured response, but the effect depended on temperature and the property considered.
- The 300 °C condition (S5) was promising because it combined a high uncharged UTS, a low strain-based EI and favourable corrosion behaviour.
- The 500 °C condition produced the highest hardness value, but its higher mass loss shows why hardness cannot be used as the sole performance criterion.
- Overall, the findings support the potential of heat-treated Ni–P–Al_2O_3 coatings to mitigate hydrogen-induced ductility loss under the conditions tested.

Conclusions and study limitations

Conclusions

- Heat treatment influenced the hydrogen-embrittlement response of electroless Ni–P–Al_2O_3-coated steel.
- S5 (300 °C, 1 h,) was the most promising condition in the combined assessment of tensile strength, strain-based EI and corrosion response.
- The highest hardness occurred at 500 °C, but this condition did not provide the best overall balance of properties.
- The coating/heat-treatment route shows potential for mitigating hydrogen-induced ductility loss; direct hydrogen-transport claims are not established.

Limitation

No direct hydrogen permeation or hydrogen-content measurements.

Thank you. Questions?

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