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BACKGROUND

- $FeCO_3$ layers play a key role in corrosion processes affecting carbon steels used in flexible pipe tensile armor wires.
- Local rupture of corrosion products is associated with CO_2-assisted stress corrosion cracking (SCC-CO_2).
- The role of steel microstructure in $FeCO_3$ layer evolution remains poorly understood.
- **Objective:** to evaluate the influence of microstructure and carbon content on $FeCO_3$ layer evolution under substitute ocean water confined CO_2 conditions.

EXPERIMENTAL SETUP

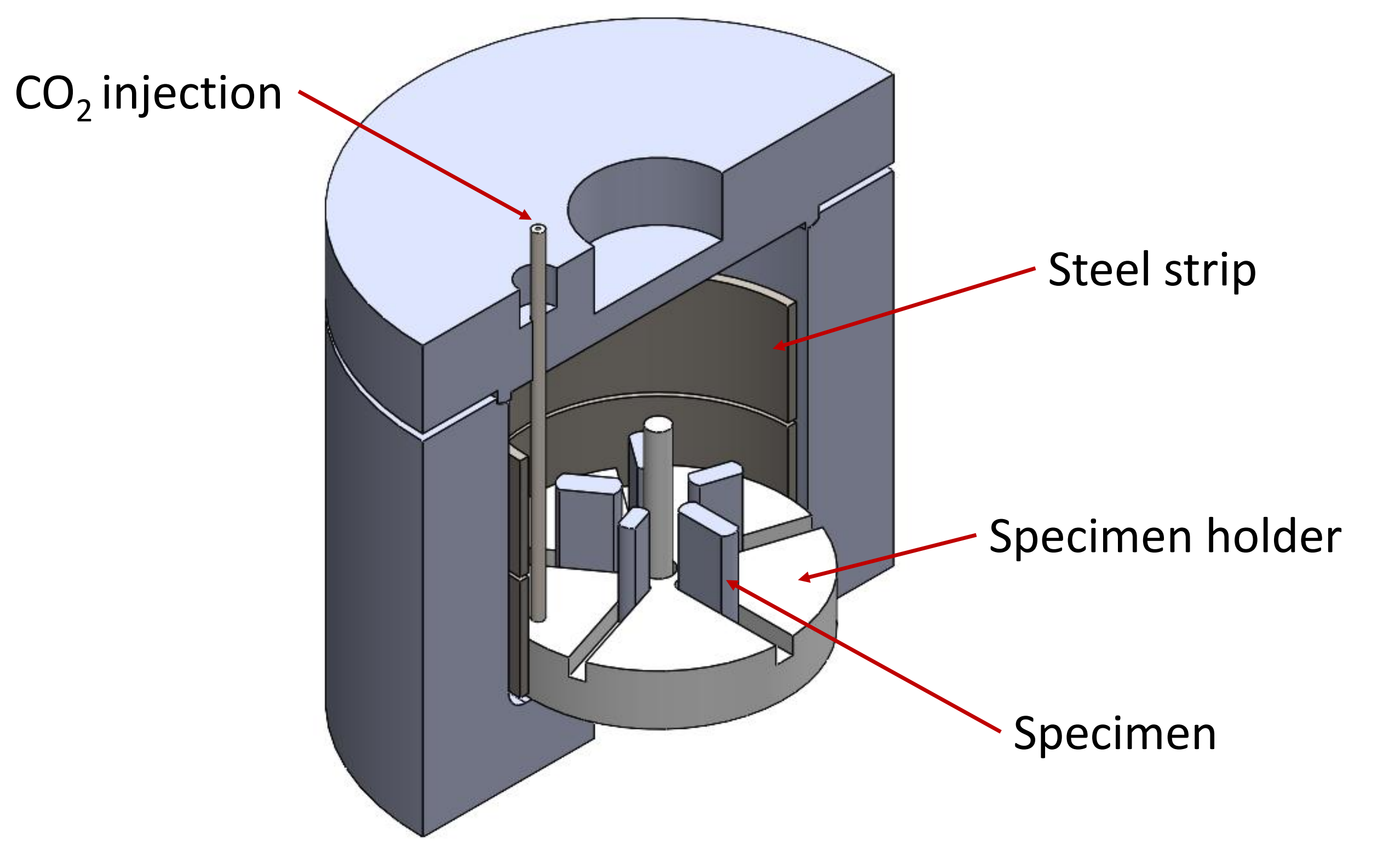

Figure 1 – Schematic of the autoclave setup used for CO_2 corrosion experiments.

- Substitute ocean water without heavy metals (ASTM D1141)
- CO_2 pressure: 10 bar(g)
- Temperature: 40 °C
- Volume/area (V/A): 1 mL/cm²
- Duration: 1, 7, 14, 21, 28, 35, 42, and 47 days
- 5 steels:
  - Steel A with 0.33 wt.% C
  - Steel B and C with 0.68 wt.% C, but the first presents spheroidized cementite, and the second, lamellar cementite.
  - Steel D and E with 0.74 wt.% C. D spheroidized and E lamellar.

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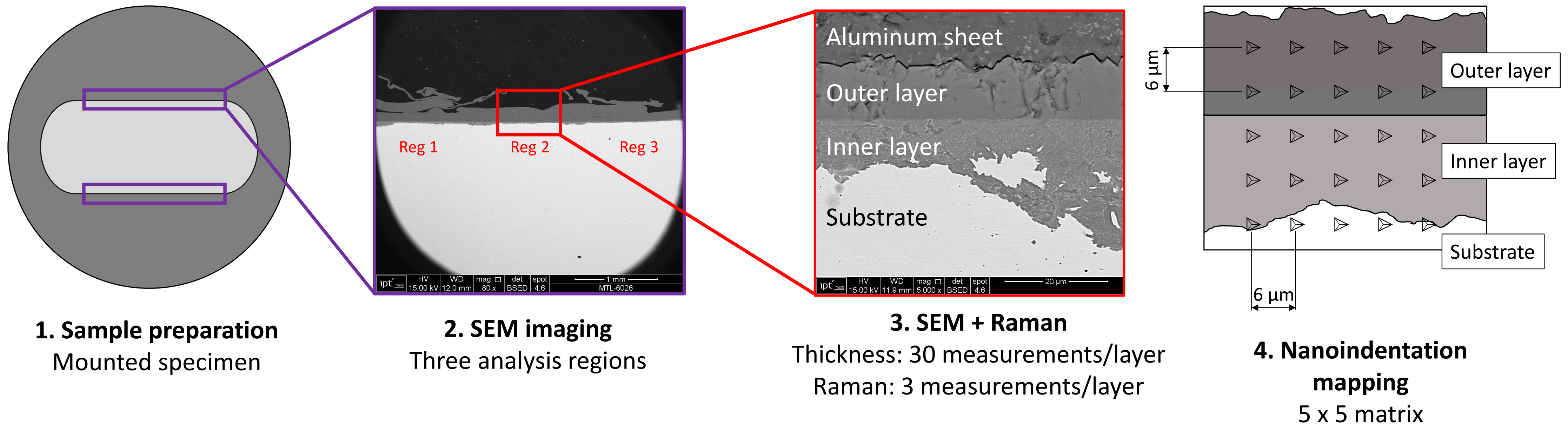
CORROSION PRODUCTS CHARACTERIZATION



1. Sample preparation
Mounted specimen

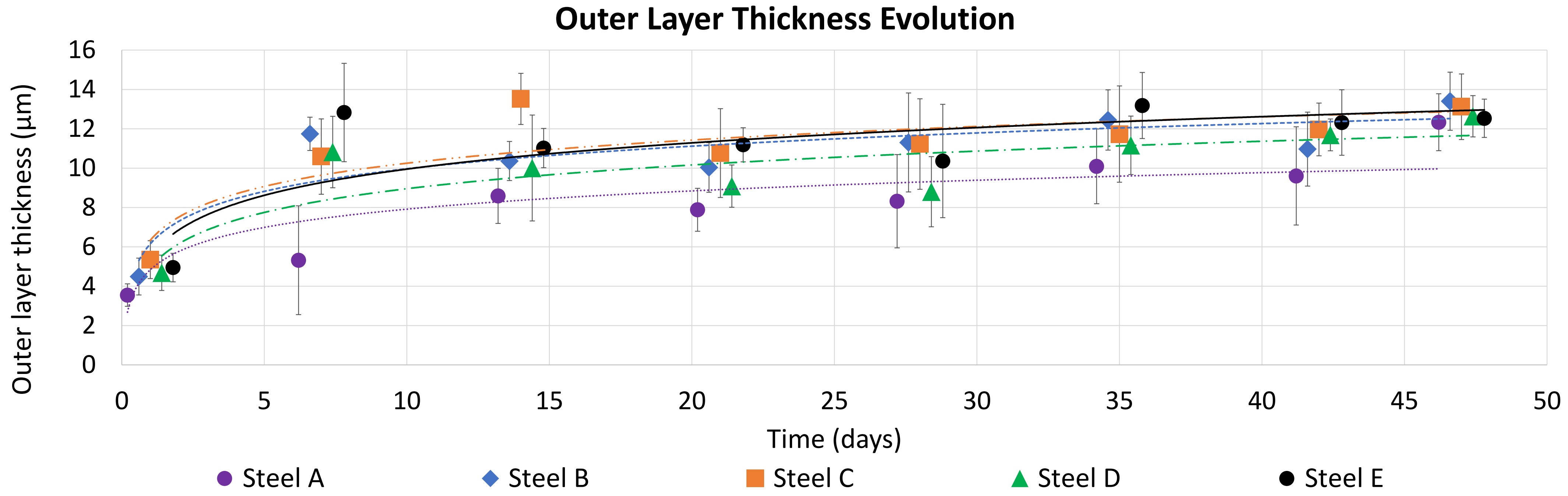
2. SEM imaging
Three analysis regions

3. SEM + Raman
Thickness: 30 measurements/layer
Raman: 3 measurements/layer

4. Nanoindentation mapping
5 x 5 matrix

Figure 2 – Workflow for specimen analysis and corrosion product characterization.

THICKNESS RESULTS



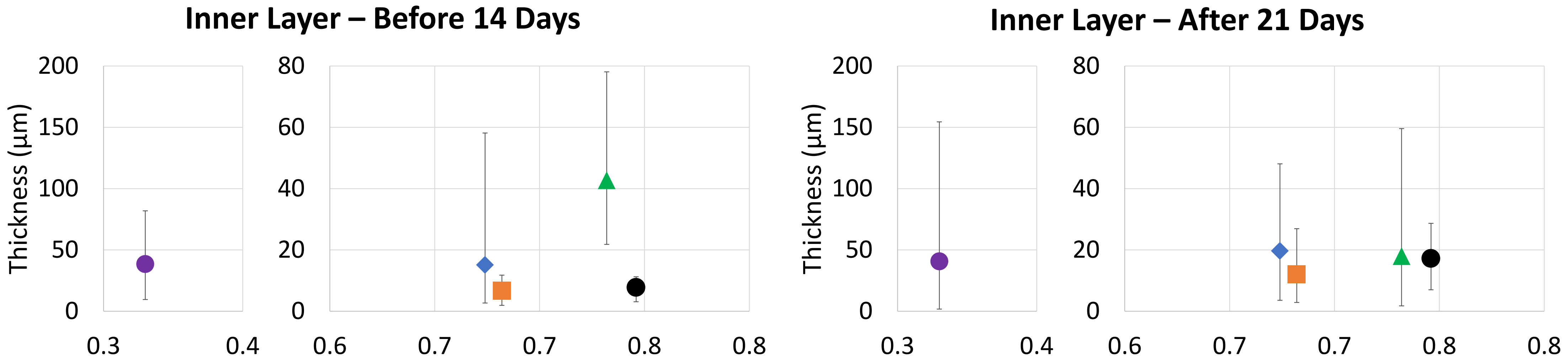


Figure 3 – Measured thickness of inner corrosion product layers as a function of carbon content and exposure time.

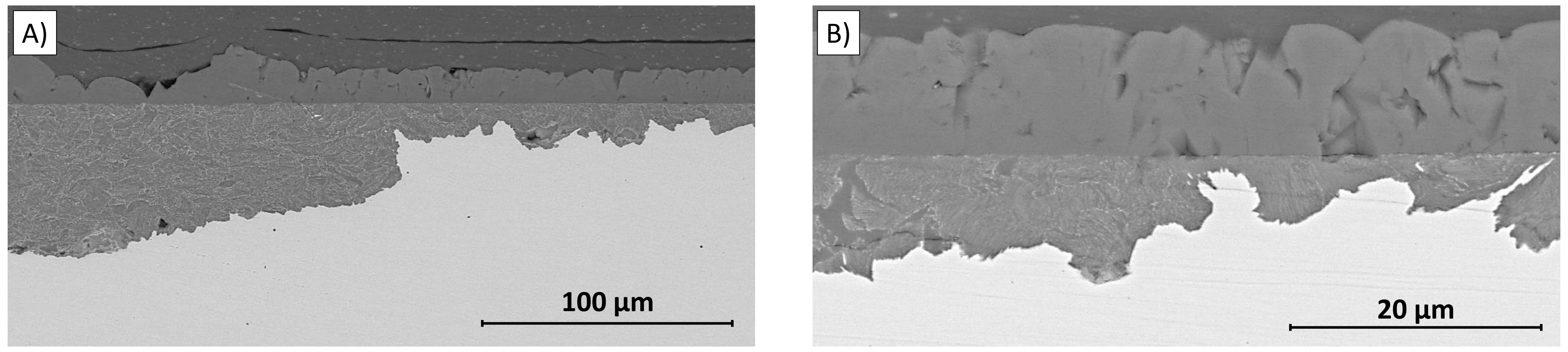


Figure 4 – Cross-section of corrosion product layers after 14 days of exposure: (A) Steel B and (B) Steel C.

Steel microstructure controls early $FeCO_3$ layer evolution in CO_2 corrosion environments

Lamellar cementite restrains inner-layer growth, while spheroidized cementite promotes thicker layers

CHARACTERIZATION RESULTS

- Outer layer phases: siderite and hematite
- Inner layer phases: siderite, cementite, goethite, and hematite

ANOVA analysis of nanoindentation data showed no statistically significant difference ($p > 0.05$) in hardness and elastic modulus between the outer and inner layers.

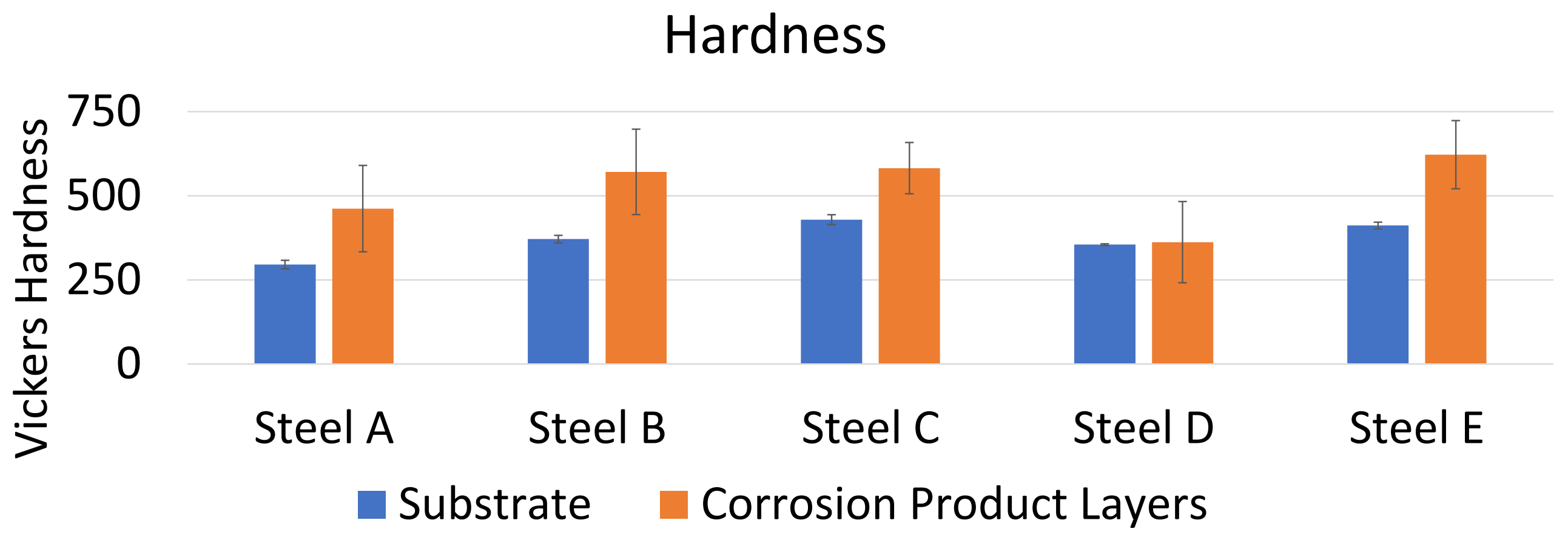


Figure 5 – Comparison of substrate hardness and nanoindentation hardness of corrosion products.

CONCLUSIONS

- Steel microstructure significantly influences inner $FeCO_3$ layer growth during the first 21 days of exposure.
- Lamellar cementite microstructures restrain corrosion product layer growth, whereas spheroidized cementite promotes thicker internal layers.
- The low-carbon steel exhibited the highest internal layer thickness and the largest variability in measured values.
- The pipeline material that failed in service exhibited the lowest hardness values in corrosion products.
- Despite differences in corrosion layer evolution, nanoindentation revealed no statistically significant variations in hardness or elastic modulus across most materials between the inner and outer layers.

Understanding how steel microstructure influences the evolution of corrosion product layers helps clarify the mechanisms associated with SCC-CO_2 in flexible pipe armor steels.