Assessment of the Pt nanoparticle distribution on oxidized stainless steel surfaces by electrochemical techniques

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1 Background

Online NobleChem (OLNC) technology in boiling water reactors

Addition of noble metal (Pt) Pt catalyst

O₂ + 4H⁺+4e⁻ → 2 H₂O

Pt decreases the susceptibility to stress corrosion cracking (SCC)

Heterogeneous distribution of Pt particles

2 Objectives

To quantify non-destructively:
- Pt surface loadings → Pt mass deposited total area
- Pt spatial distributions (interparticle distances)
- Establish relations between:
  1) Pt surface loadings vs. ECP
  2) Pt interparticle distances vs. ECP

Routes → Electrochemical Corrosion Potential (ECP) & Electrochemical Impedance Spectroscopy (EIS)

3.1 Electrochemical Corrosion Potential (ECP) ↔ Pt Spatial Distribution

Experimental setup and details

Scheme of ECP cell

high-purity water, 270 °C, 90 bar

Specimens with varying Pt loadings and interparticle distances

Pt injection duration, nominal surface loading

25 h, 10 ng/cm²

250 h, 120 ng/cm²

100 h, 60 ng/cm²

Duration of Pt injection (h) Pt amount (nm) Mean particle size (nm) Avg. Pt surface loadings (ng/cm²) Mean interparticle distance < 150 (nm)

25 57 8.5 ±3.8 9 ±5 563 ±351

50 113 14.4 ±8.7 36 ±30 344 ±272

100 207 10.1 ±4.8 44 ±30 290 ±320

250 553 10.9 ±5.7 129 ±36 201 ±162

Interparticle distances are better predictors of ECP as compared to loadings

3.2 Electrochemical Impedance Spectroscopy (EIS) ↔ Pt Surface Loading

Experimental setup and details

25 °C, 0.5M Na₂SO₄, atm. pressure, Ar-purged

Specimen-oxide-Pt configurations

Nyquist plots

Bode plots

Impedance Z(f, t) = Z_{ uncomp} + Z_{comp} = Z_{real} + jZ_{imag}

Frequency (f) = 20,000 - 0.005 Hz

Vₚ = 10 mV, 10 points/decade

WE = Working electrode (specimen)

RE = Reference electrode (Hg/HgO₂)

CE = Counter electrode (Pt foil)

Re(Z) vs. ECP

Predicted a max. 1000 nm Pt interparticle distance for "protective" ECP of < -230 mV SCE

Further investigations ongoing to confirm results

4 Takeaways and Outlook

- Mean interparticle distances are better predictors of ECP values compared to Pt loadings
- EIS method seems promising, revealing qualitative differences for varying amounts of Pt loadings and distributions
- Qualitative differences are showcased for varying Pt amount and type (particles/coating) in these preliminary measurements

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