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

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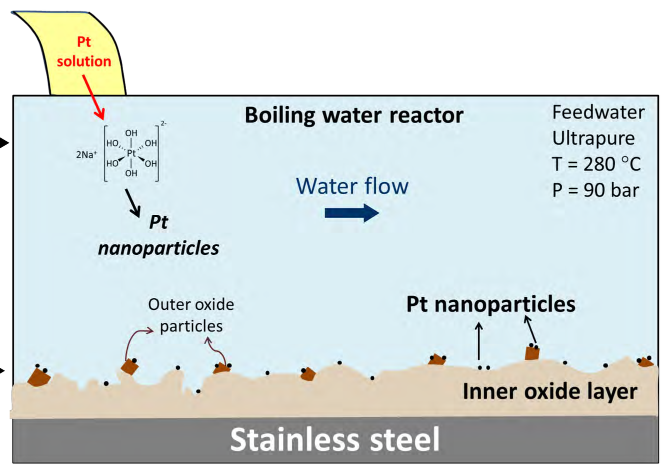
Addition of noble metal (Pt)

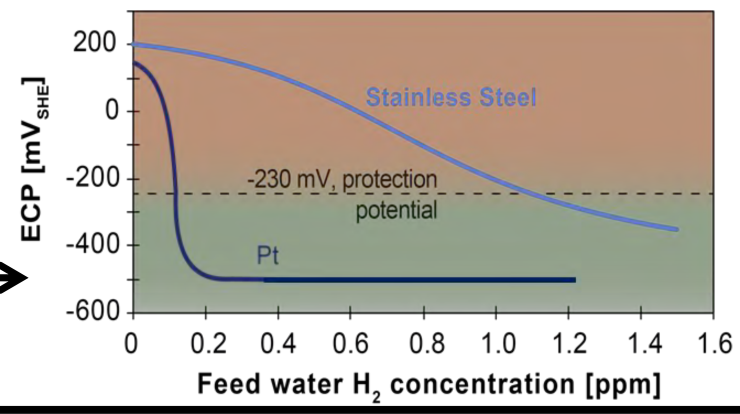
Pt catalyst

$O_2 + 4H^+ + 4e^- \longrightarrow 2\,H_2O$

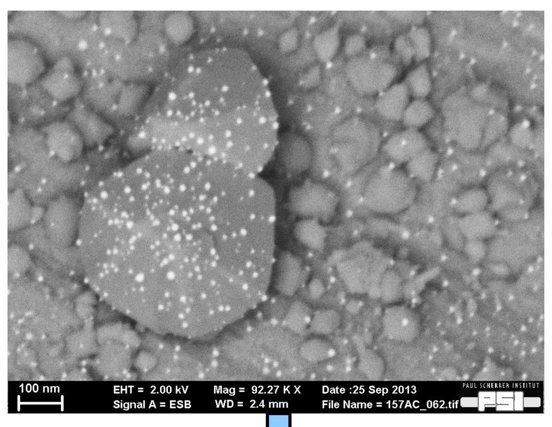
↓

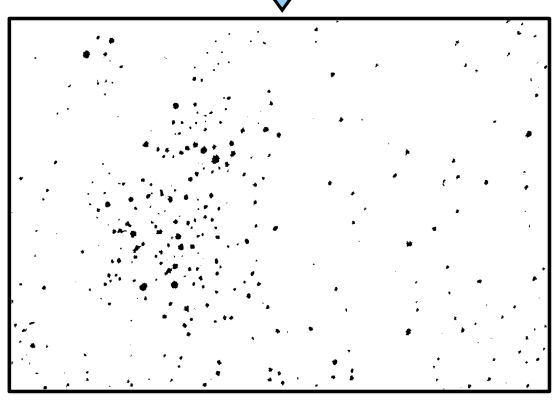
Pt decreases the susceptibility to stress corrosion cracking (SCC)





Heterogeneous distribution of Pt particles





2 Objectives

To quantify non-destructively:

- Pt surface loadings → $\frac{Pt\ mass\ deposited}{total\ area}$
- Pt spatial distributions (interparticle distances)
- Establish relations between:
  - (a) Pt surface loadings vs. ECP
  - (b) Pt interparticle distances vs. ECP

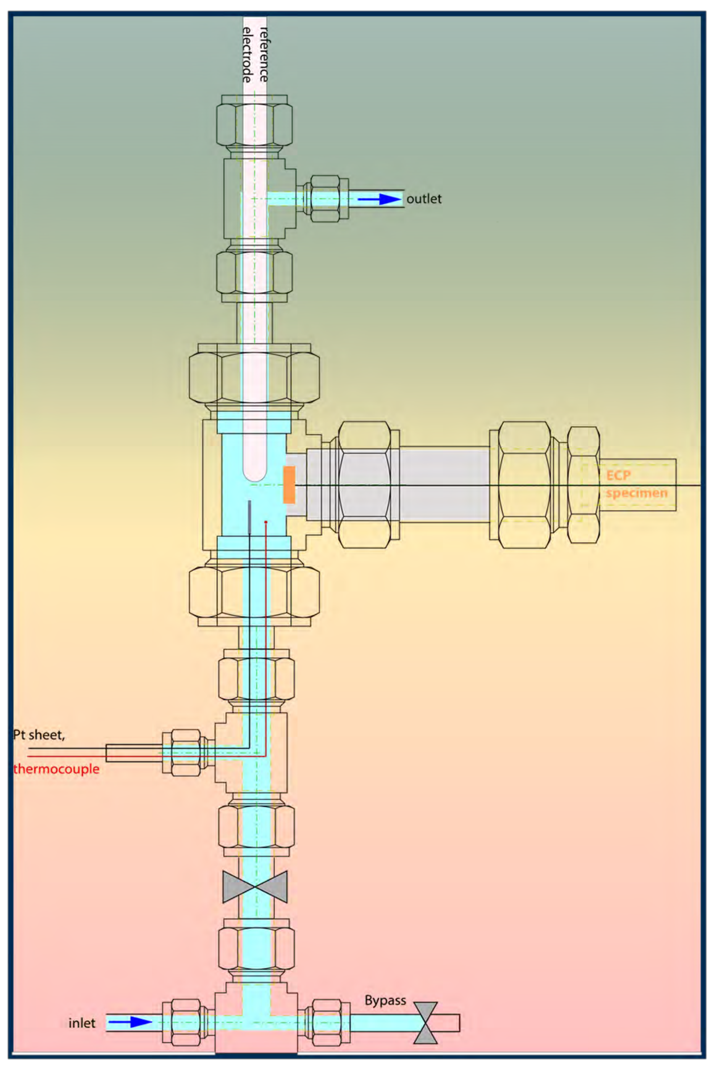
Routes → **Electrochemical Corrosion Potential (ECP) & Electrochemical Impedance Spectroscopy (EIS)**

3.1 Electrochemical Corrosion Potential (ECP) ⟷ Pt Spatial Distribution

Experimental setup and details

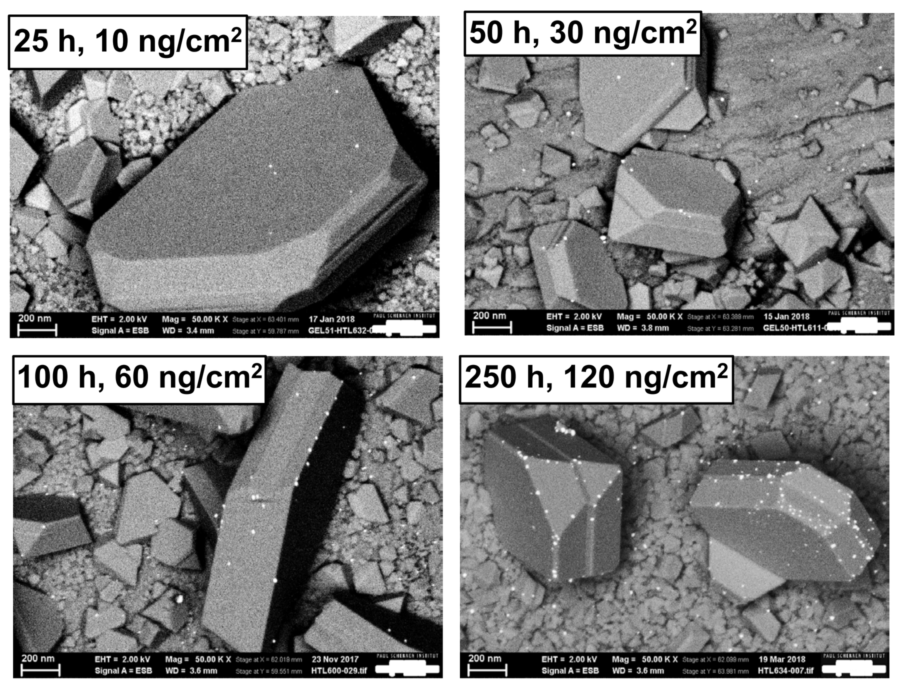
Scheme of ECP cell

high-purity water, 270 °C, 90 bar



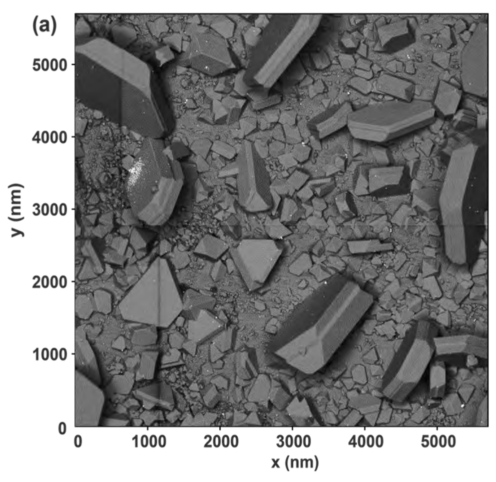
Specimens with varying Pt loadings and interparticle distances

Pt injection duration, nominal surface loading

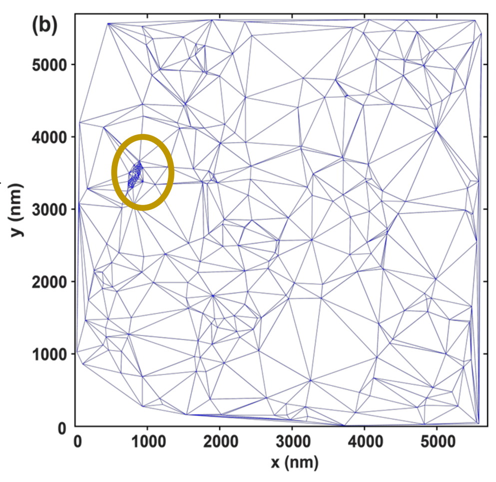


Duration of Pt injection (h)	Pt amount (µg)	Mean particle size (nm)	Avg. Pt surface loadings (ng/cm²)	Mean interparticle distance ± 1SD (nm)
25	57	8.5 ±3.8	9 ±5	563 ±351
50	113	14.4 ±8.7	36 ±30 142 ±133	344 ±272
100	207	10.1 ±4.8	44 ±30	290 ±232
250	553	10.9 ±5.7	123 ±36	201 ±162

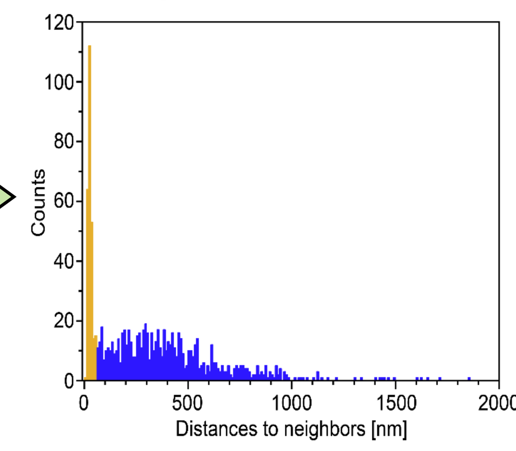
Stitched SEM pictures



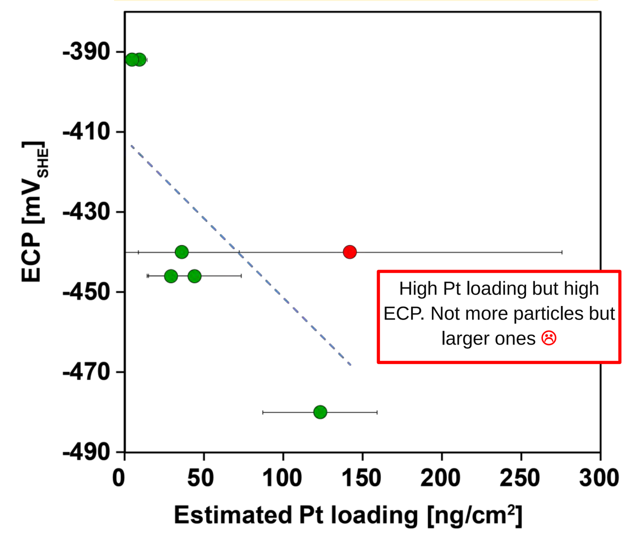
Delaunay triangulation



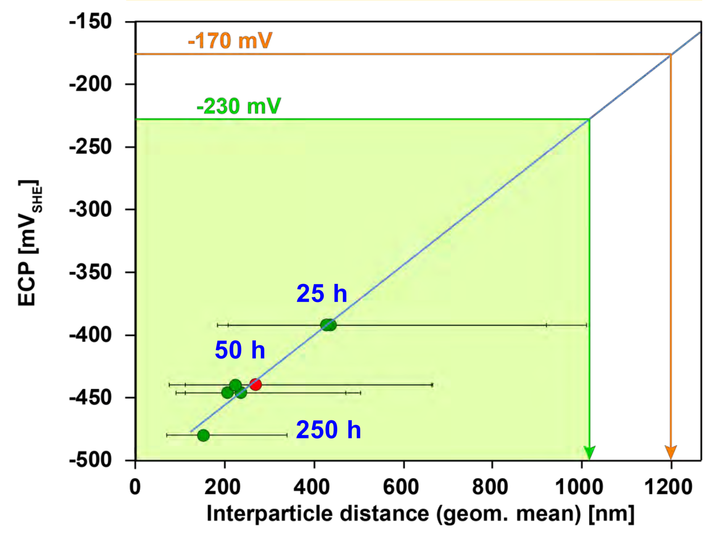
Interparticle distances



Pt loading vs. ECP



Interparticle distance vs. ECP



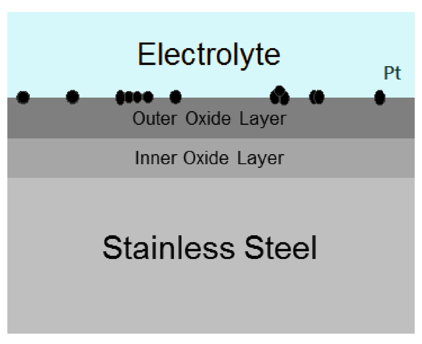
✓ **Interparticle distances are better predictors of ECP as compared to loadings**

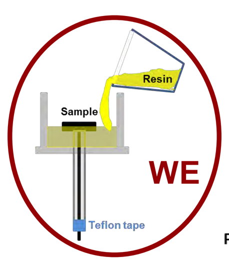
P. V. Grundler et al., Assessment of the SCC Mitigation Capabilities of the Noble Metal Chemical Application Technology in a Simulated BWR Environment, 19th International conference on Environmental Degradation of Materials in Nuclear Power Systems, Aug 18-22, 2019, Boston, USA

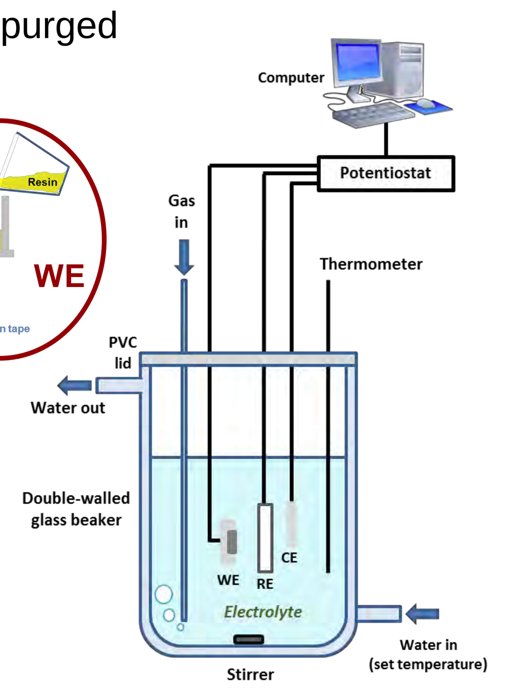
3.2 Electrochemical Impedance Spectroscopy (EIS) ⟷ Pt Surface Loading

Experimental setup and details

25 °C, 0.5M Na_2SO_4, atm. pressure, Ar-purged







$$\text{Impedance } Z(f,t) = \frac{V_o \sin(2\pi f t)}{I_o \sin(2\pi f t - \theta)}$$

$$Z = Z_{real} + iZ_{imag}$$

$$|Z| = \sqrt{Z_{real}{}^2 + Z_{imag}{}^2}$$

Frequency (f) = 20,000-0.005 Hz

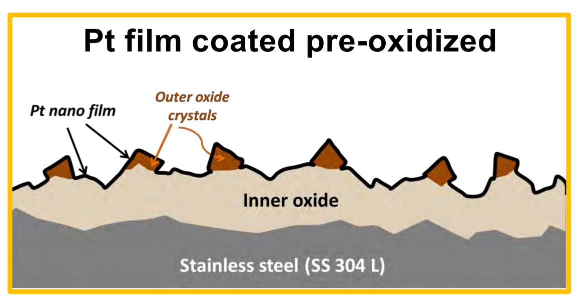
V_o = 10 mV, 10 points/decade

WE = Working electrode (specimen)
RE = Reference electrode (Hg/HgSO$_4$)
CE = Counter electrode (Pt foil)

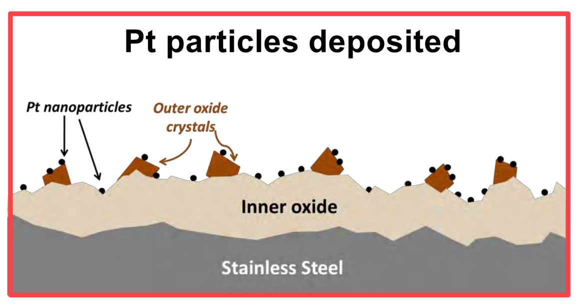
Specimen-oxide-Pt configurations

As-received

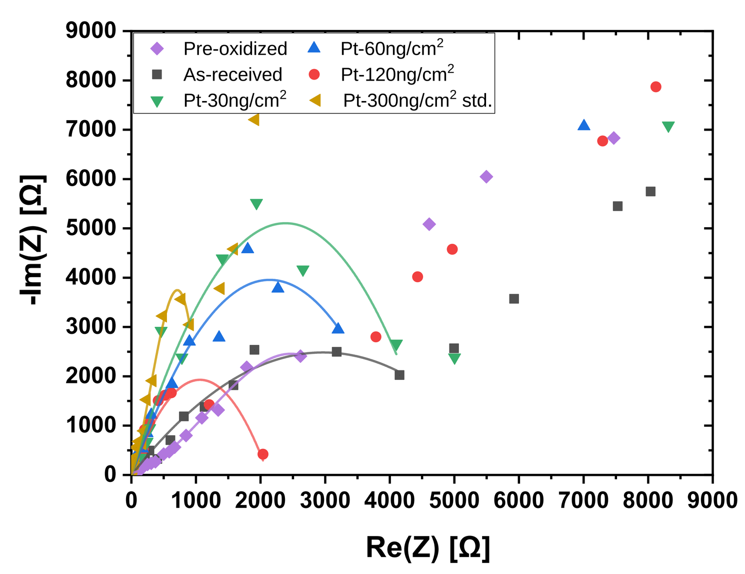
Pt film coated pre-oxidized



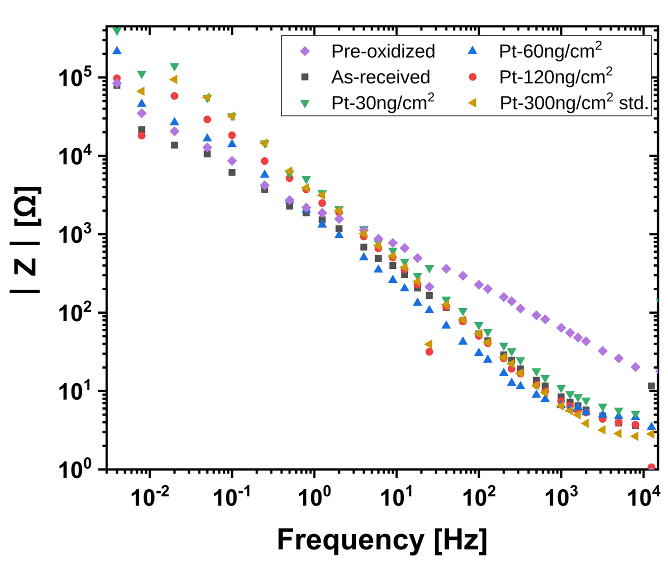
Pt particles deposited



Nyquist Plots



Bode plots



Re(Z): Pre-oxidized ≈ As-received > Pt-30 > Pt-60 > Pt-120 > Pt-300 (std.)

✓ **Qualitative differences are showcased for varying Pt amount and type (particles/coating) in these preliminary measurements**

4 Takeaways and Outlook

- ✓ **Mean interparticle distances are better predictors of ECP values compared to Pt loadings** ➡ *Predicted a max. 1000 nm Pt interparticle distance for "protective" ECP of < -230 mV$_{SHE}$*
- ✓ **EIS method seems promising, revealing qualitative differences for varying amounts of Pt quantities and distributions** ➡ *Further investigations ongoing to confirm results*

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