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Investigation of SCC of high strength aluminum alloys by means of slow strain rate test and cyclic anodic polarization in combination

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Investigation of SCC of high strength Aluminum Alloys by means of Slow Strain Rate test and Cyclic Anodic Polarization in combination

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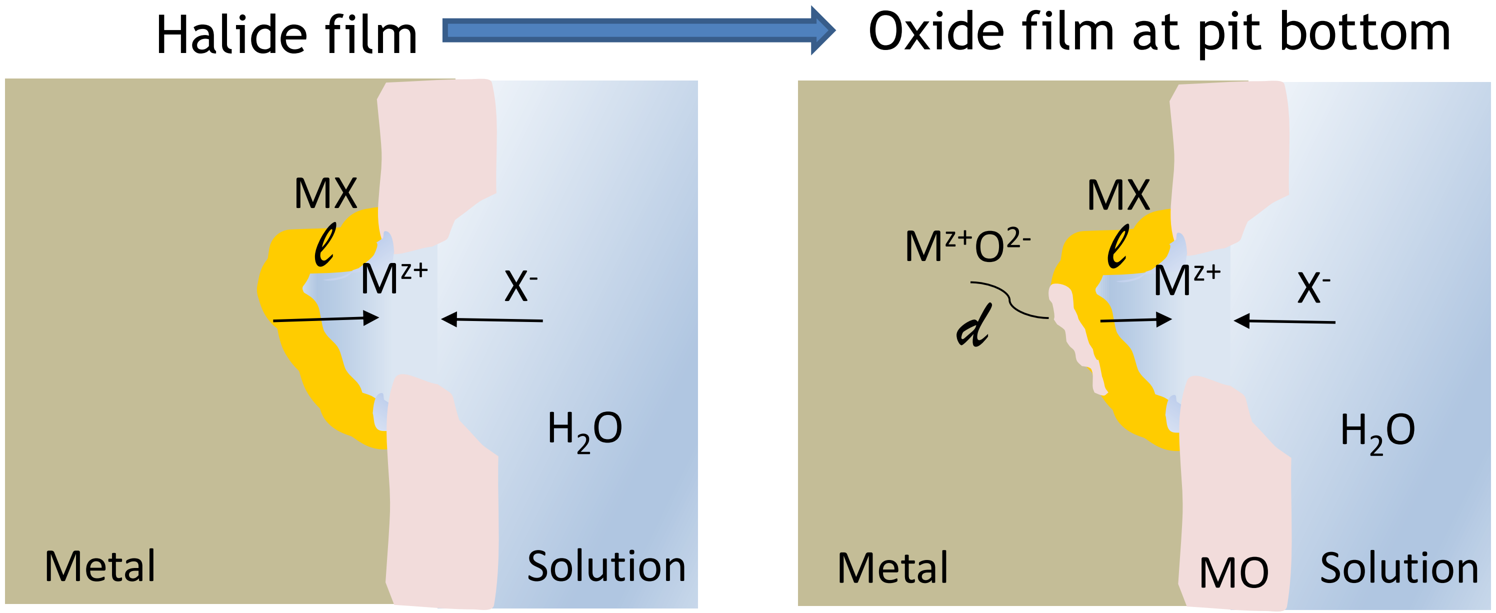
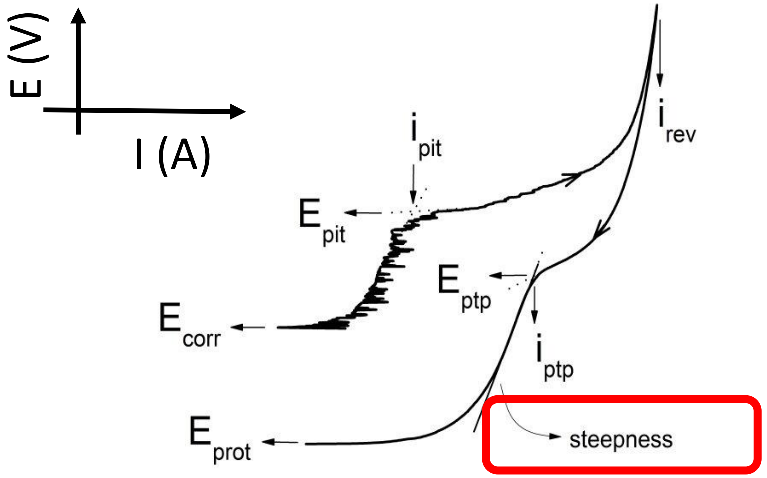
Hernstein, 2018

✓ Singe cycle anodic polarization and repassivation properties



Single cycle anodic polarization (Pitting Scan, PS)



Eptp - thermodynamic driving force of Al dissolution on freshly created (filmed) surface

iptp $\propto$ rate hydrolysis equilibrium at $[Al^{3+}]_{crit}$

$2Al^{3+} + H_2O + OH^- \leftrightarrow 2Al(OH)^{2+} + H^+$

High currents driving a potential drop:
compensation by Cl^- electromigration of local electrodissolution processes

Effective anodic charge transfer coefficient α_{eff} *
estimated from the steepness by the equation:

$$\ln\left(\frac{i}{iptp}\right) = \frac{a_{eff}\,F}{RT}(E - E_{ptp})$$

$\alpha_{eff} = \alpha - \lambda$
α - anodic charge transfer coefficient
λ - the effective kinetic order of metal dissolution with respect to [Cl-]

Increasing steepness ➔ $\alpha_{eff} \rightarrow 0$ ($\alpha \rightarrow \lambda$) ➔ Accelerating action of Cl^-

✓ Singe cycle anodic polarization and repassivation properties

Previous studies- permanent load in bending

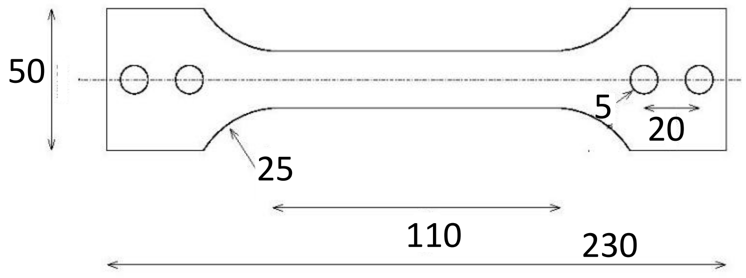
anodic processes localization

This work – dynamic straining

✓ Slow strain rate test and cyclic anodic polarization (pitting scan, PS) in combination

Multiple SSRT machine (4 load cells, 30 kN each)

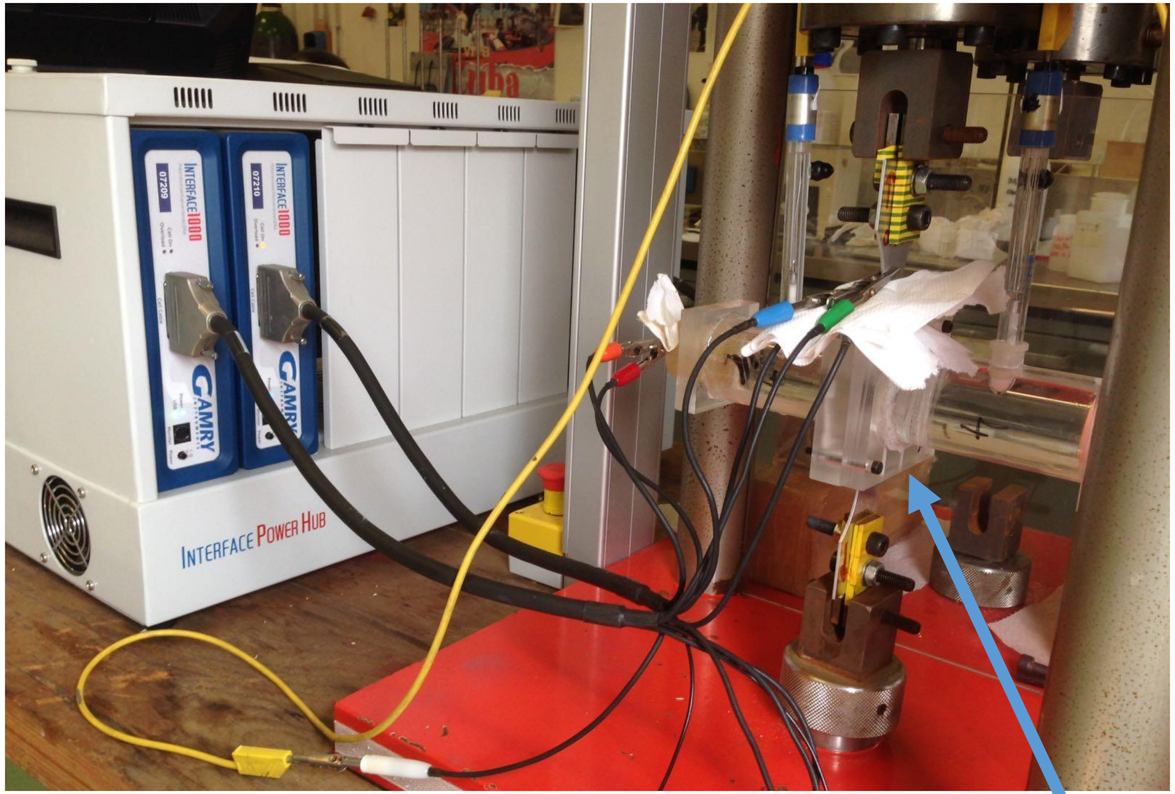
Smooth tensile test specimen



Alloy	7075-T6	2024-T3
Thickness (mm)	2.0	1,5
Elastic Modulus E (GPa)	74.5	75,7
Yield strength YS, Rp02 (MPa)	510	354
Ultimate tensile strength UTS (MPa)	583	499
Gauge section area (mm^2)	40	32

✓ Slow strain rate test and pitting scan in combination

Electrochemical setup



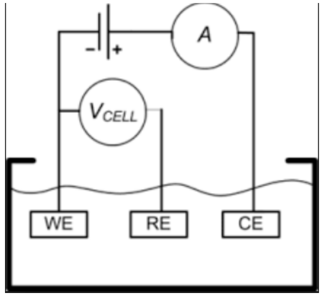
Gamry
Interface 1000 multipotentiostat

Working electrodes (WE)
Opposite surfaces of the tensile specimen
(geometrical area 2 cm^2 each)

Separate two compartents (Plexiglass cell)

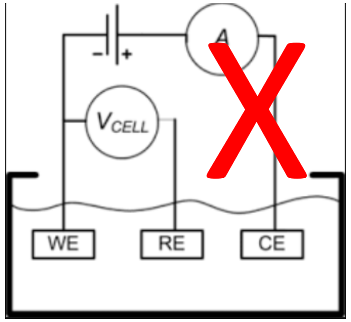
Compartment 1: Three-electrode configuration
DC polarization

WE – tensile specimen
RE – SCE
CE – Ir-coated Ti



Compartment 2: Two-electrode configuration
Open circuit

WE – tensile specimen
RE – SCE



✓ Slow strain rate test and pitting scan in combination

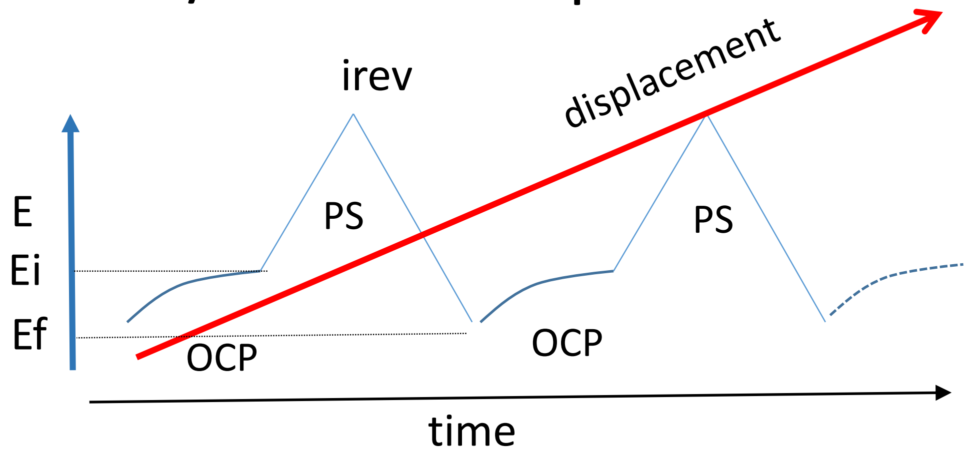
Stagnant NaCl (pH 6.5)
Room T (≅ 25 °C)

Combined experiment SSRT-OCP/PS at a constant extension rate

Three-electrode configuration
Pitting scans with OCP in between during straining

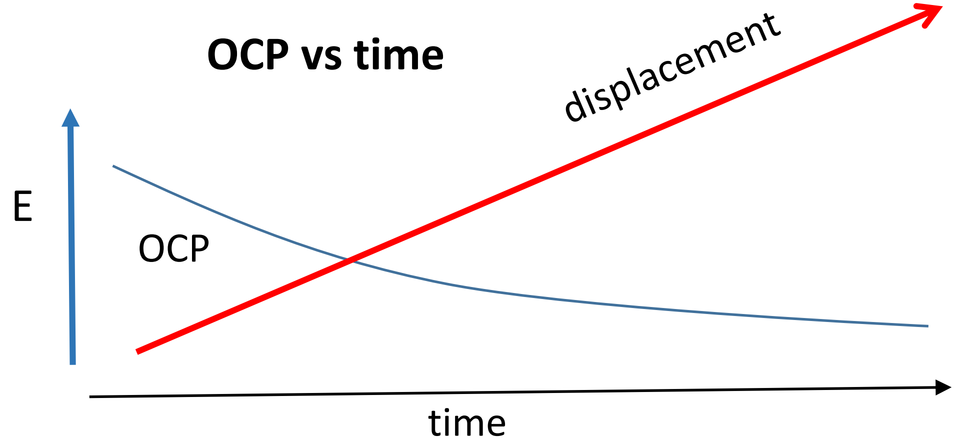
Ei = Ecorr
i_{rev} = 2.5 mA/cm^2
Ef = -1,1 V (vs SCE)

OCP/PS consecutive sequences



Two-electrode configuration
OCP monitoring during straining

OCP vs time



Output Data

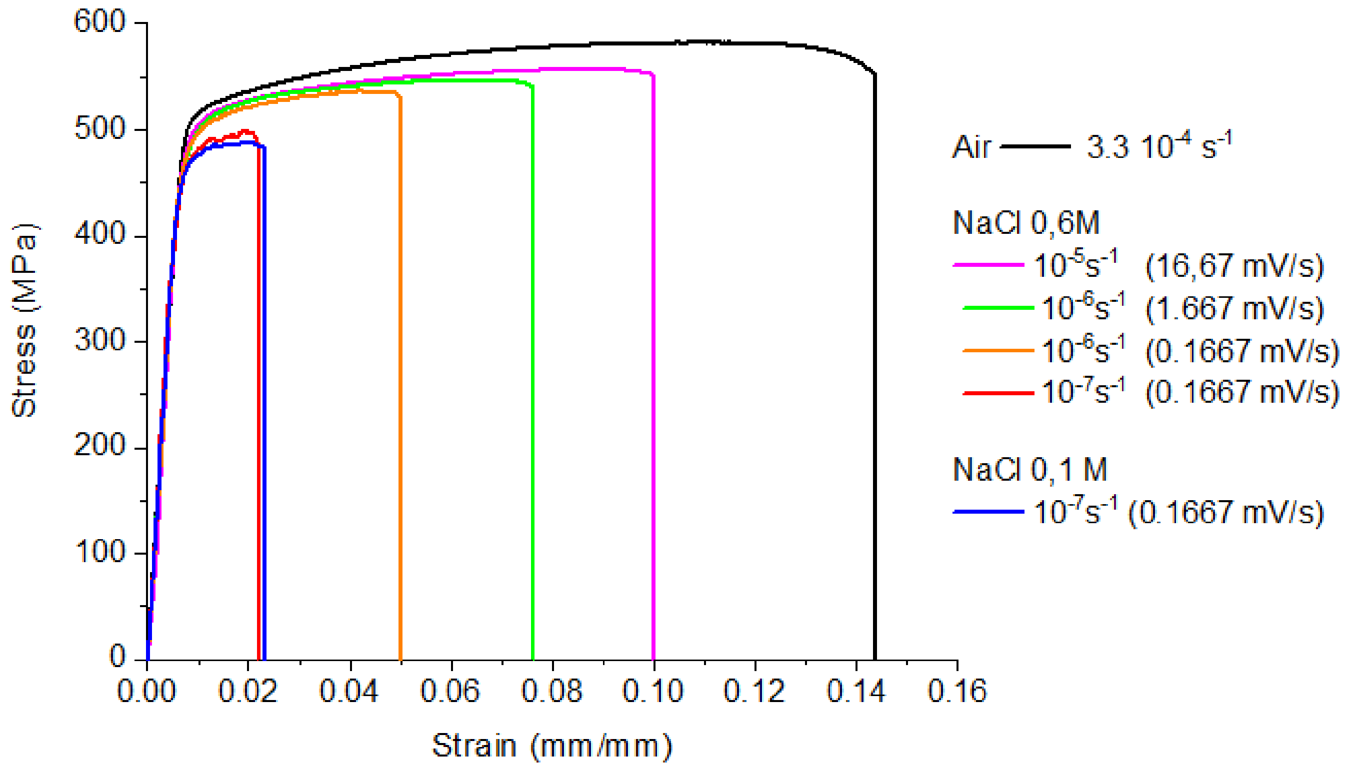
Load (t)
OCP (Δt)
E,i,Q (Δt)
OCP (t)

❖ **Control tests: PS (no straining), OCP (SSRT, no PS), OCP (noPS, no SSRT)**

Al 7075-T6

(5.8 Zn-2.6Mg-1.7Cu)

Al 7075-T6

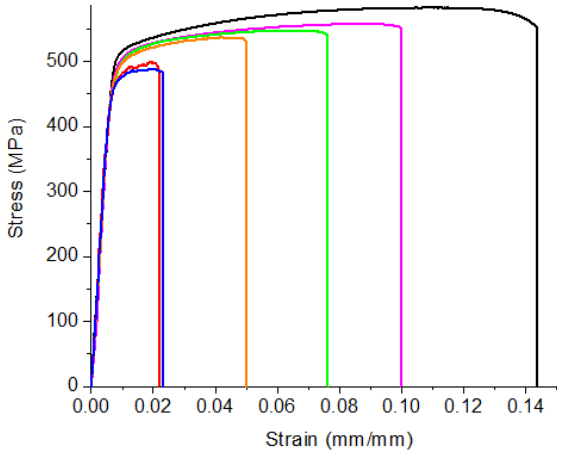


NaCl (M)	$\dot{\varepsilon}$ (s^{-1})	ν (mVs^{-1})	OCP
0.6	10^{-5}	16.67	110 s
0.6	10^{-6}	1.667	18 min
0.6	10^{-6}	0.1667	18 min
0.6	10^{-7}	0.1667	3 h
0.1	10^{-7}	0.1667	3 h

The strain at break and the yield strength decrease with decreasing strain rate, regardless the electrochemical perturbation

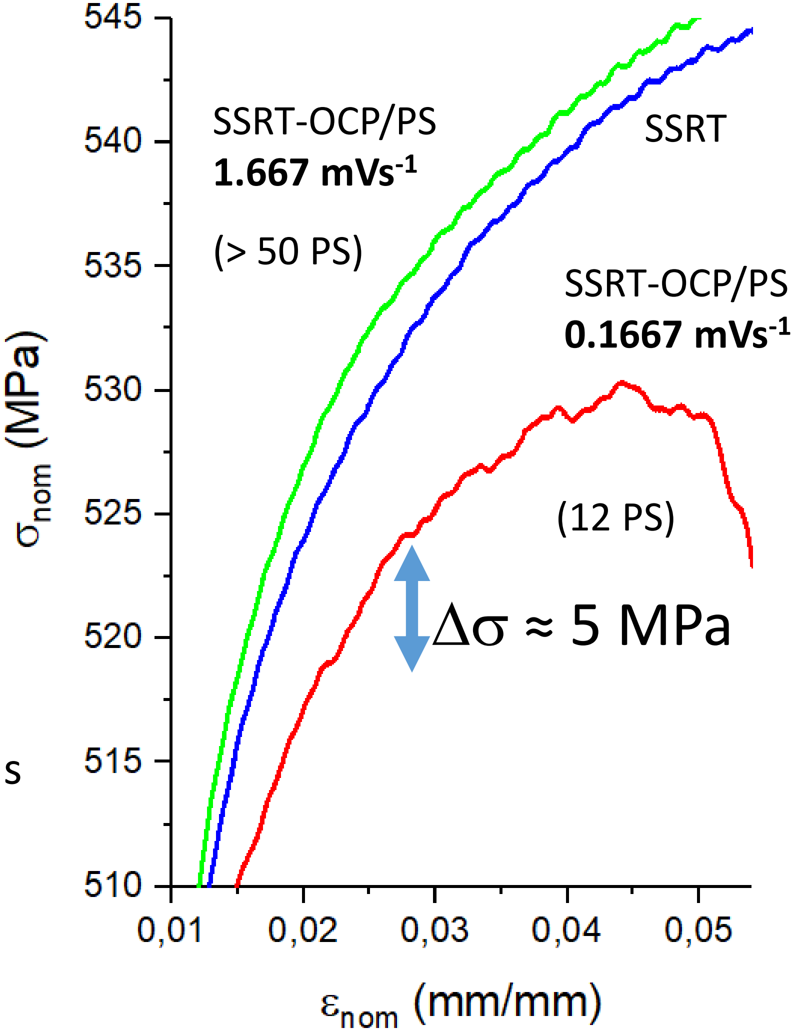
Al 7075-T6

A closer look at

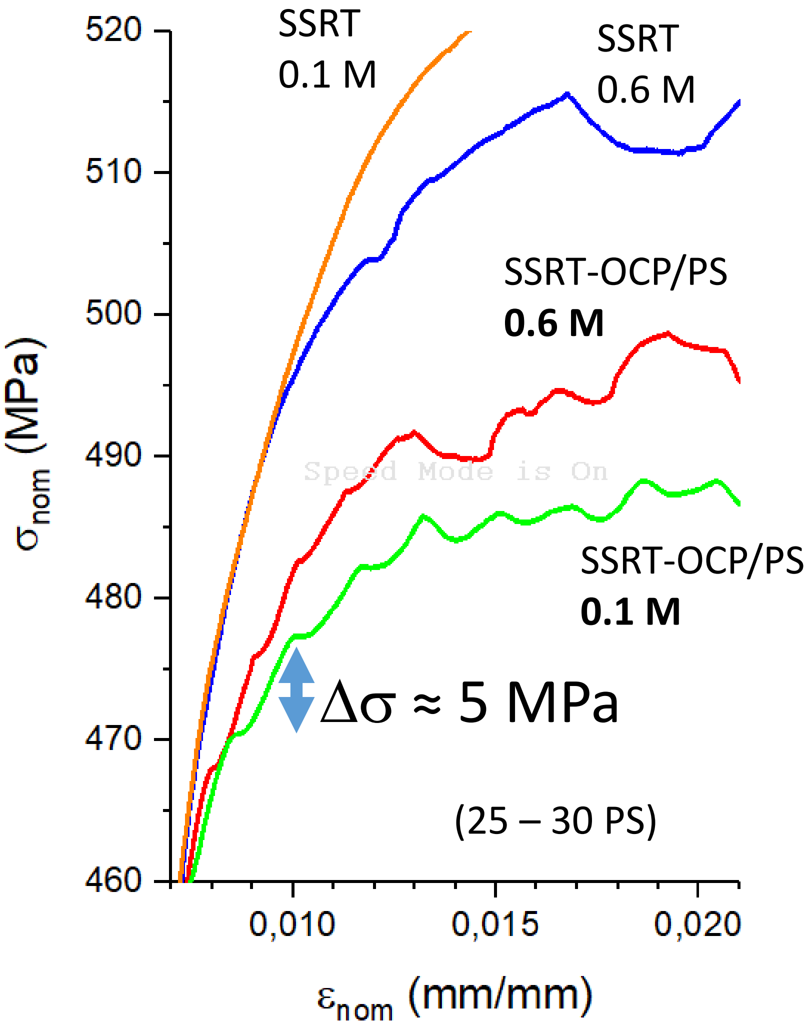


Stress relaxation/recovery events
ONLY in elasto-plastic and plastic regions
for $\dot{\varepsilon} \leq 10^{-6}$ s^{-1} with OCP/PS sequence

$\dot{\varepsilon} = 10^{-6}$ s^{-1} 0.6 M NaCl

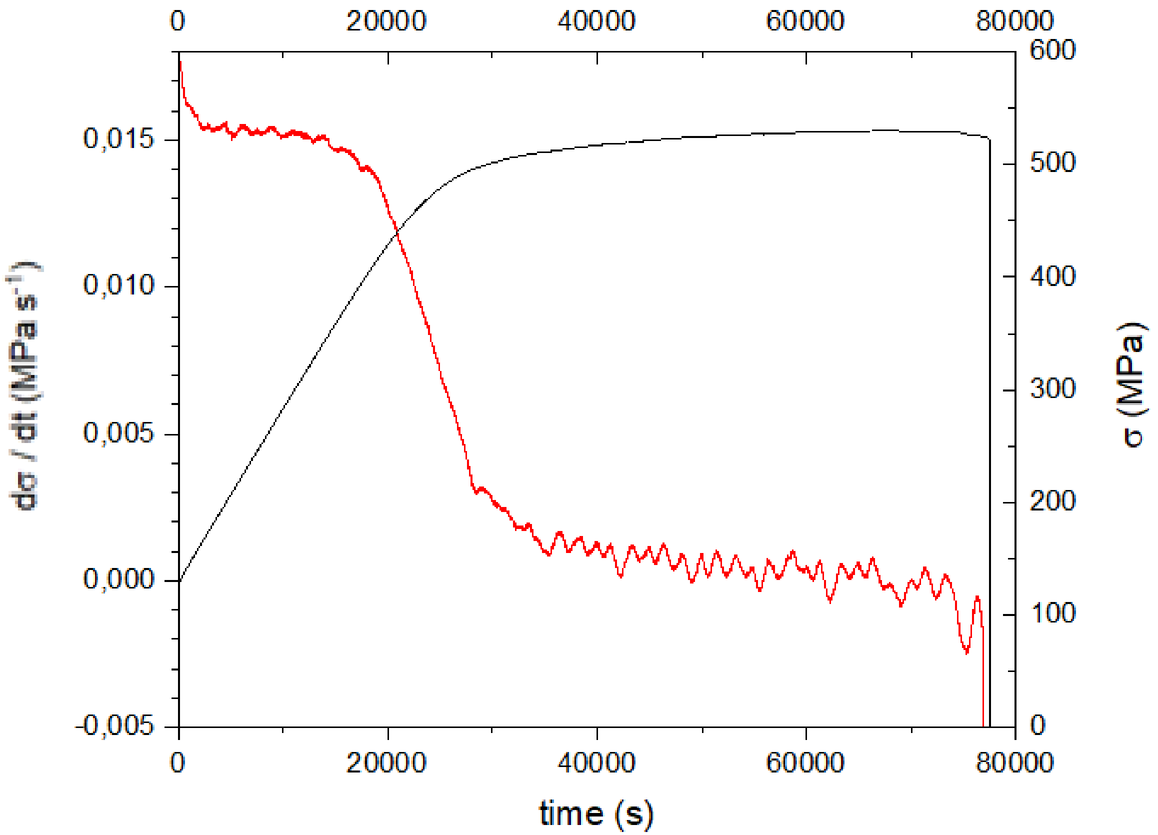


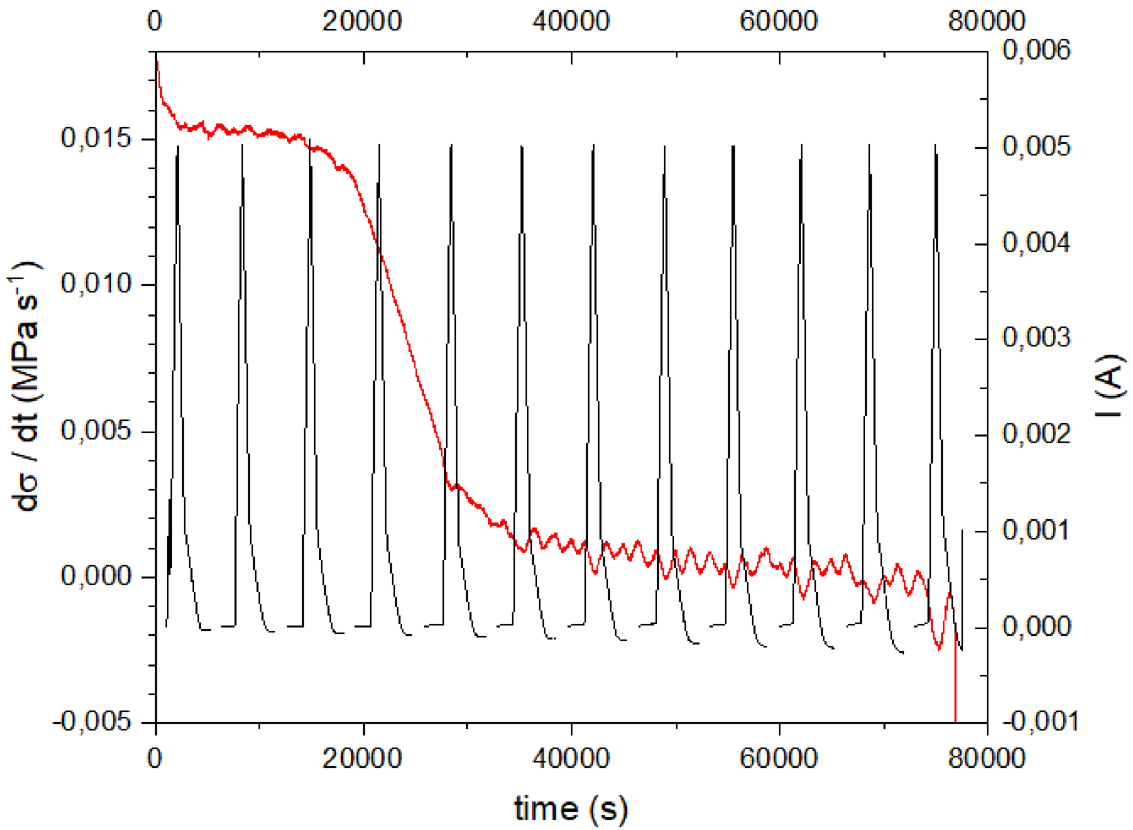
$\dot{\varepsilon} = 10^{-7}$ s^{-1} 0.1667 mV/s



Al 7075-T6

$\dot{\varepsilon} = 10^{-6}\ s^{-1}$ 0.6M NaCl 0.1667 mV/s



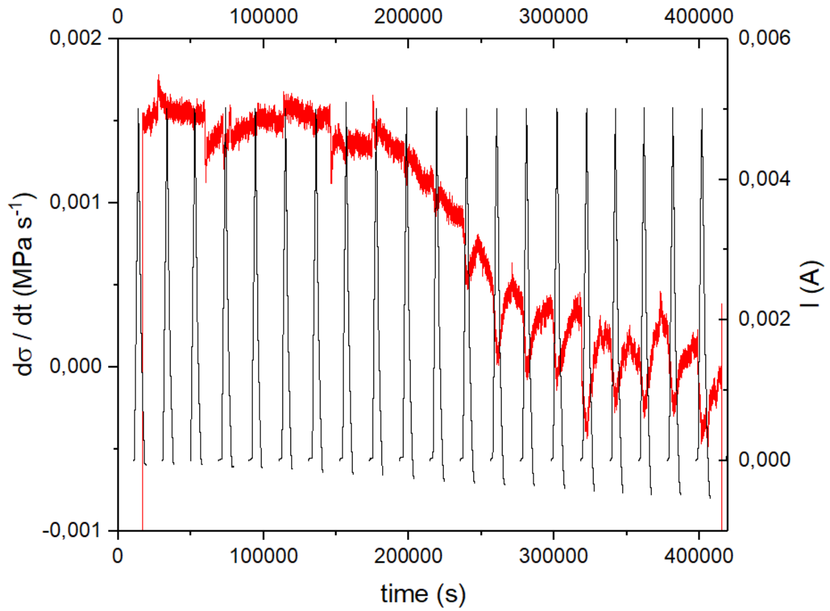


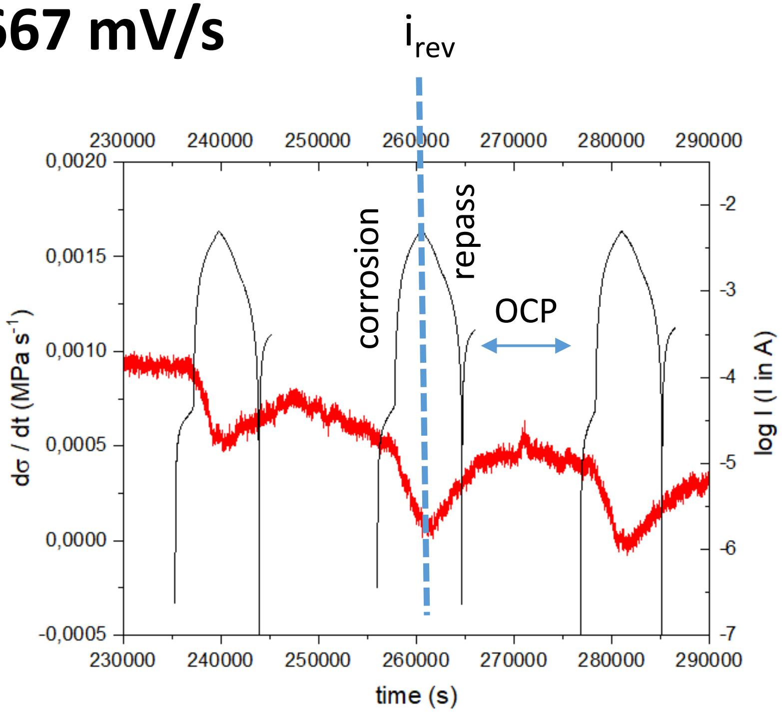
Resolved spikes upon derivation of the stress – time curves

Load drop in correspondence with the anodic polarization cycle

Al 7075-T6

$\dot{\varepsilon} = 10^{-7}\ s^{-1}$ 0.1 M NaCl 0.1667 mV/s

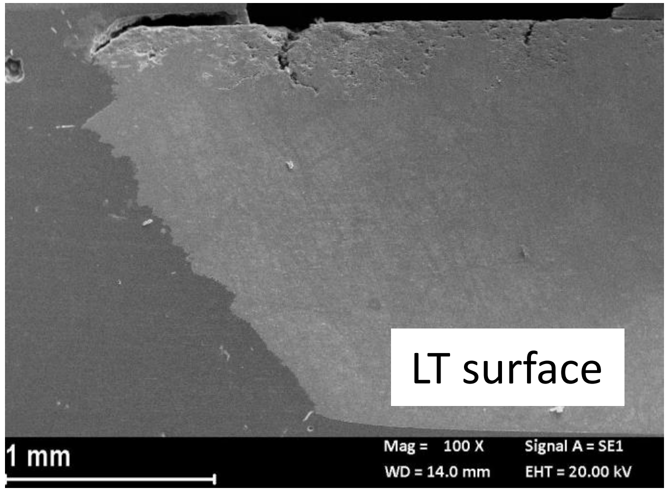


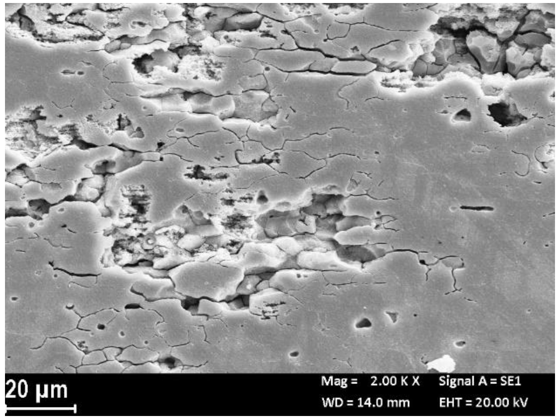


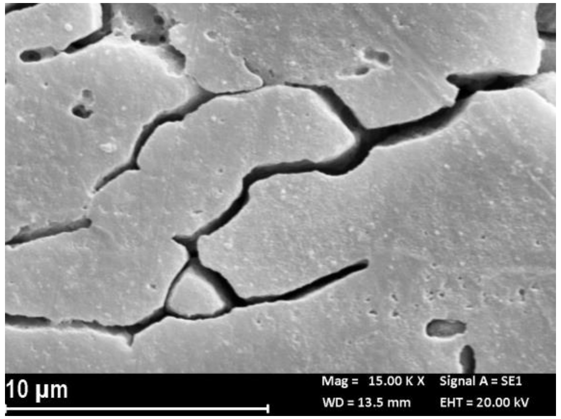
Spikes in dσ/dt – t curves better resolved for $\dot{\varepsilon} = 10^{-7}\ s^{-1}$ and with dilution of NaCl solution

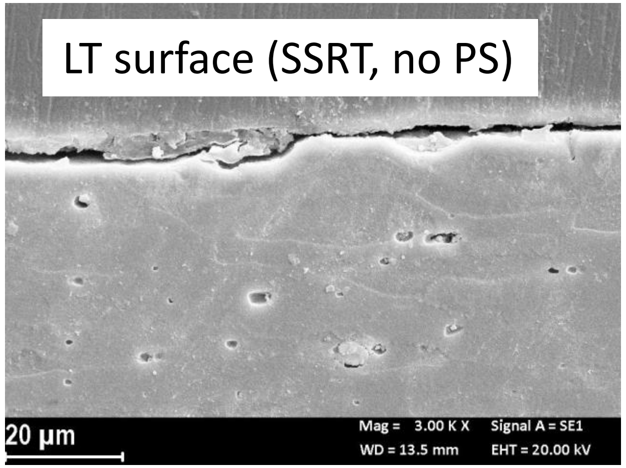
The correspondence with anodic dissolution/repassivation shown in log I – t curves

dσ/dt decreases once localized corrosion onsets at Epit



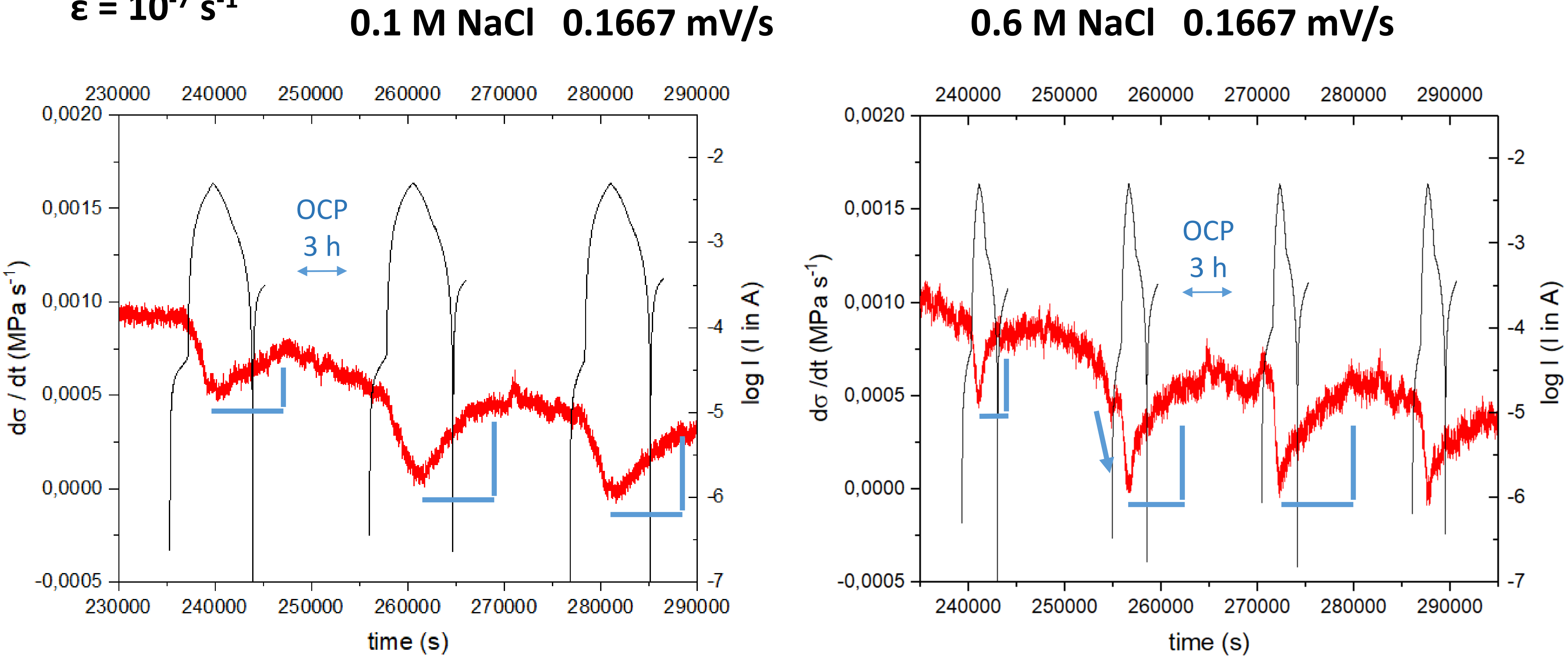






Al 7075-T6

$\dot{\varepsilon} = 10^{-7}\ s^{-1}$

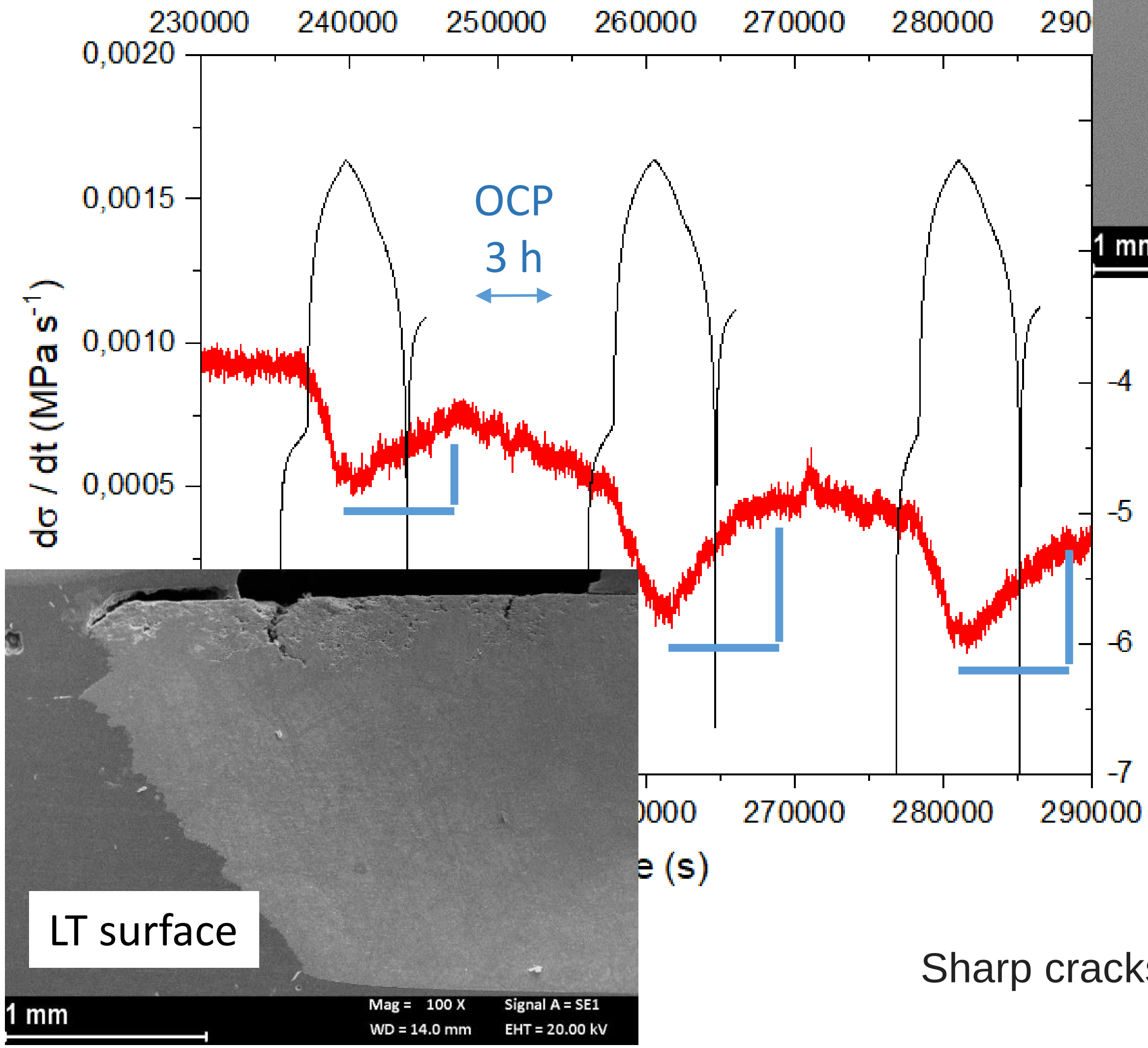


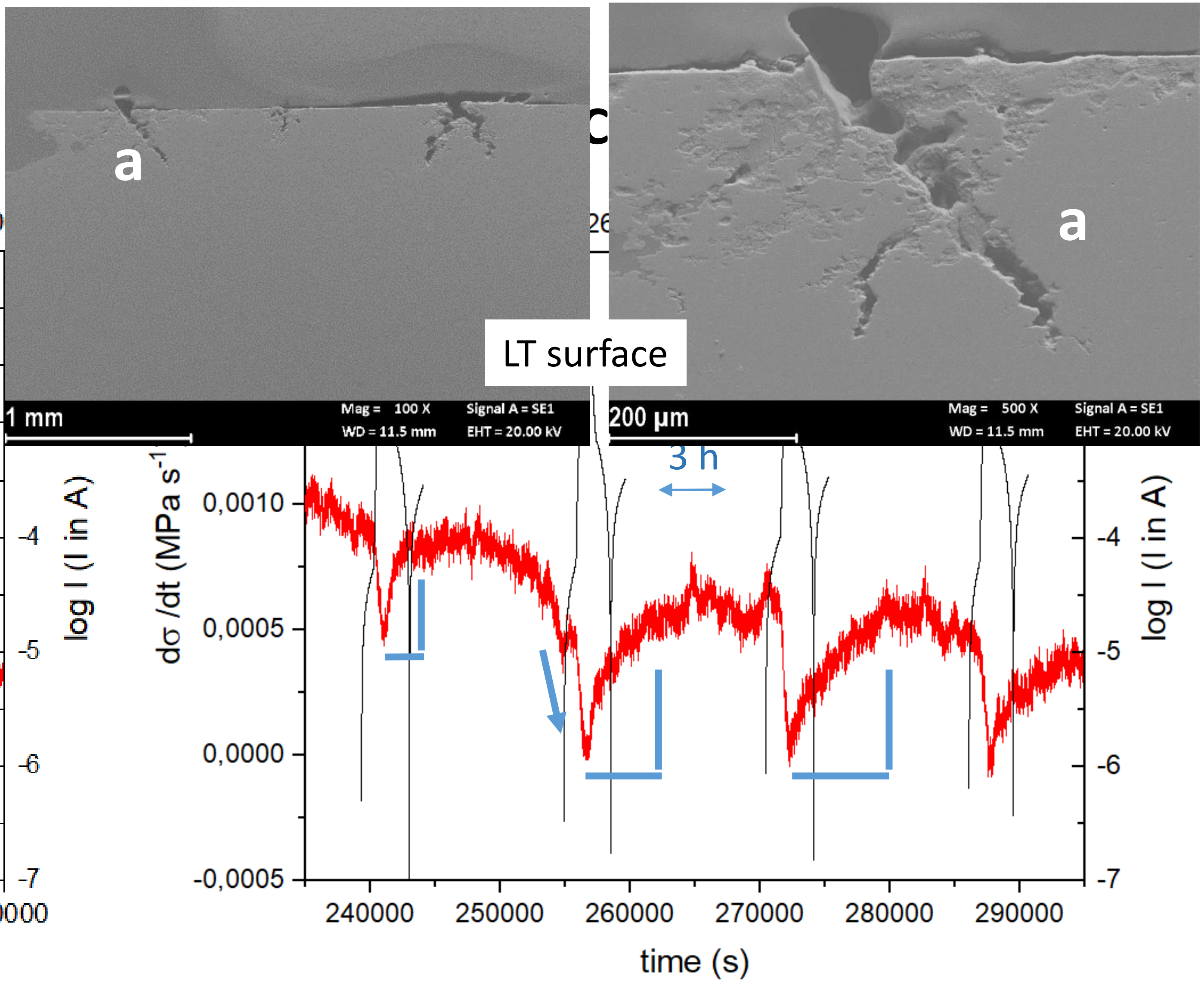
Chloride ions concentration influences the time interval during which dσ/dt increases along wise with the repassivation response

Al 7075-T6

$\dot{\varepsilon} = 10^{-7}\ s^{-1}$ **0.1 M NaCl 0.1667 mV/s**

0.6 M NaCl 0.1667 mV/s

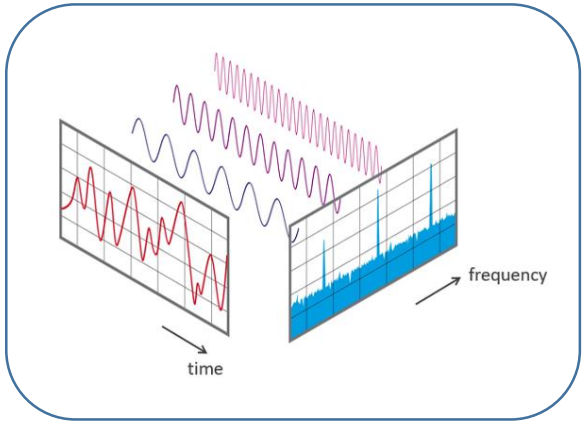




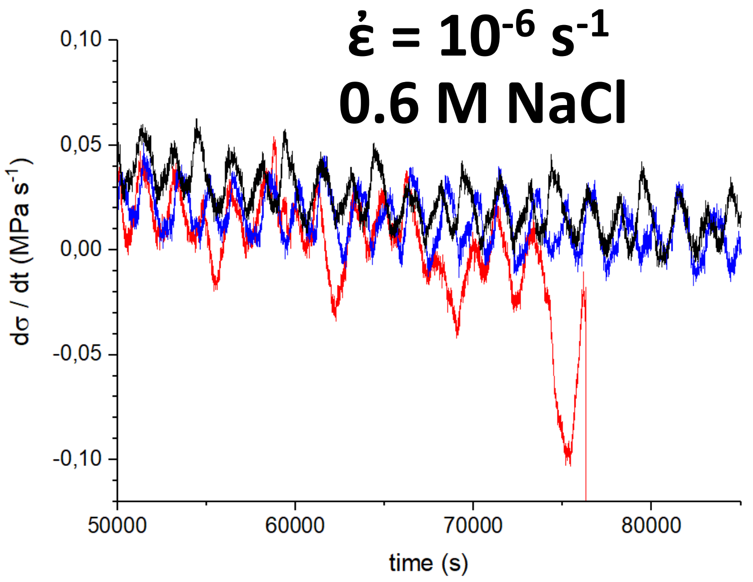
Sharp cracks prevalently developed in 0.6 M NaCl

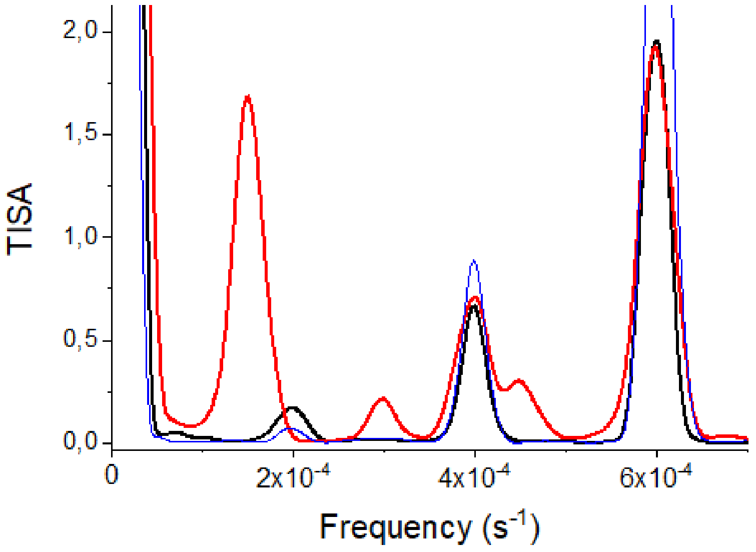
Al 7075-T6

FFT– from time to frequency domains



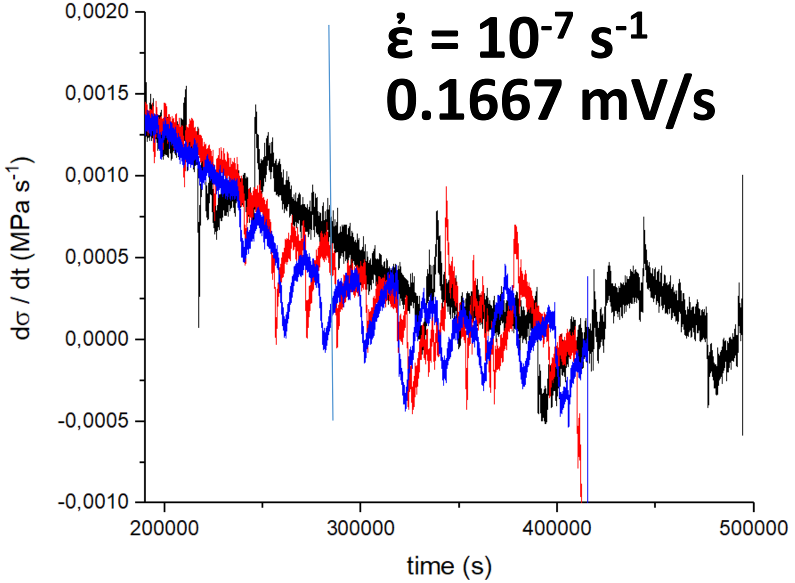
- 1.667 mV/s
- 0.1667 mV/s
- no PS

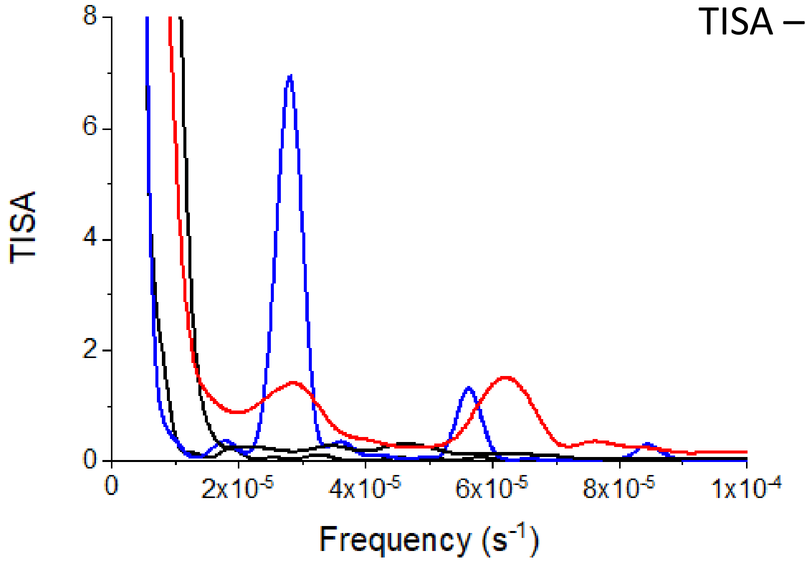




Trace interpolation B-spline
FFT – Hanning window
(satisfactiry in 95% of cases)

- 0.1 M NaCl
- 0.6 M NaCl
- no PS (0.6 & 0.1 M NaCl)





TISA – time interval square amplitude

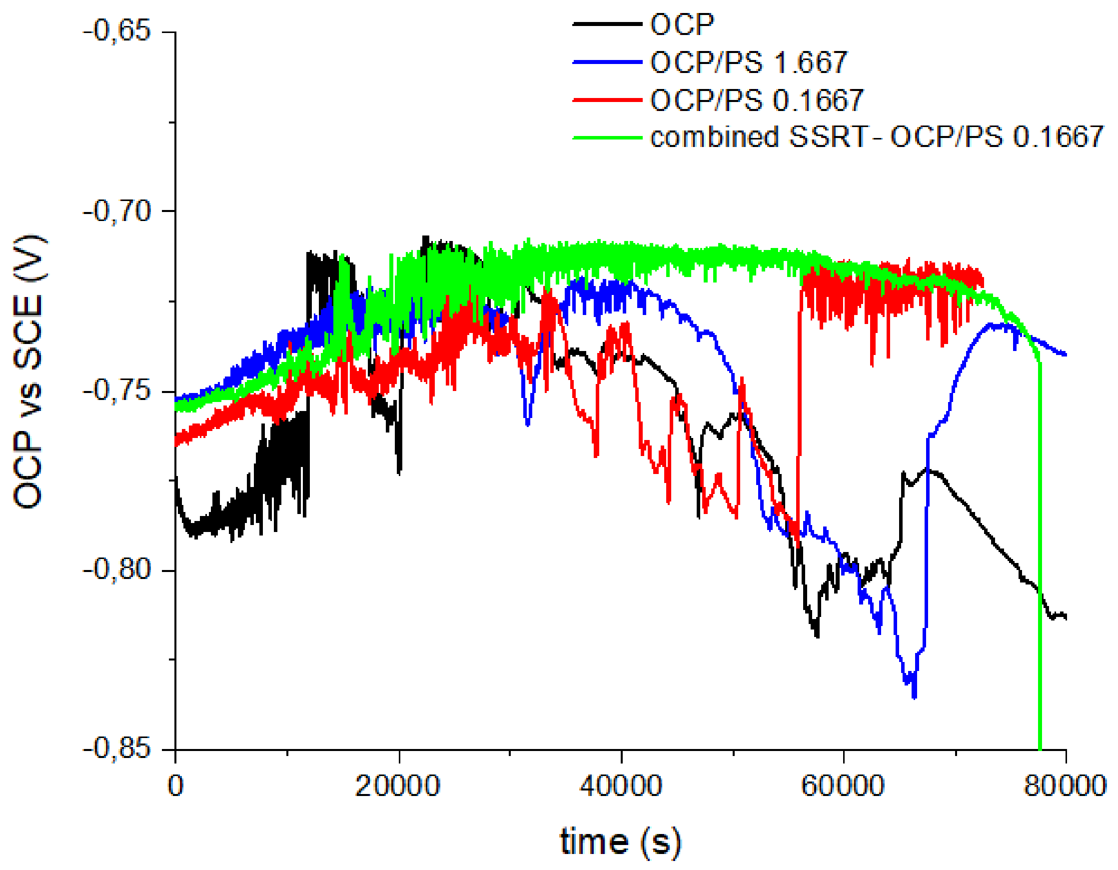
$$\frac{\Delta t(Re^2 + Im^2)}{n}$$

Re, Im – real and imaginary parts of the transform data
n – length of the input sequence
Δt – sampling interval

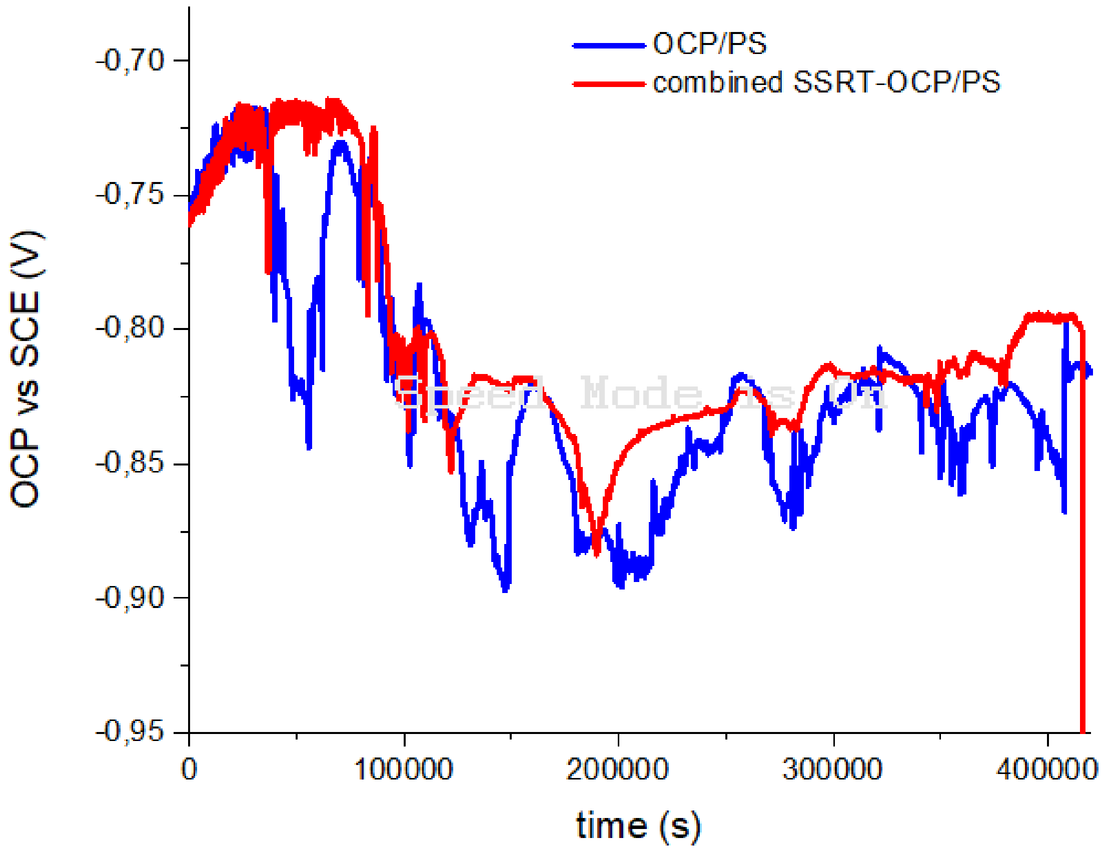
Al 7075-T6

Comparison of OCP variation with time **0.6 M NaCl**

$\dot{\varepsilon} = 10^{-6}\ s^{-1}$



$\dot{\varepsilon} = 10^{-7}\ s^{-1}$



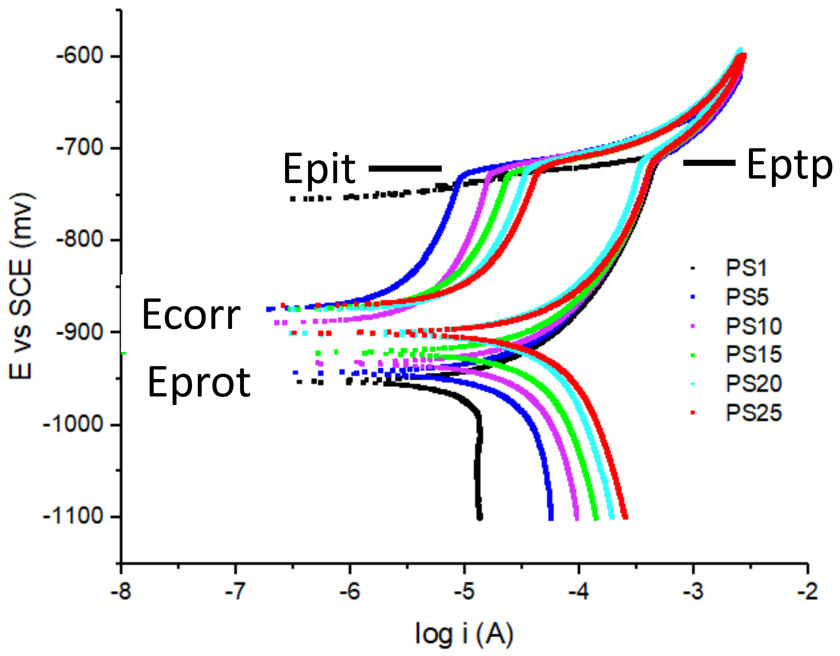
OCP tends to increase with time during dynamic straining at $10^{-6}\ s^{-1}$

The trend is opposite for $10^{-7}\ s^{-1}$ but the negative transients are less significant in comparison to the results with no straining

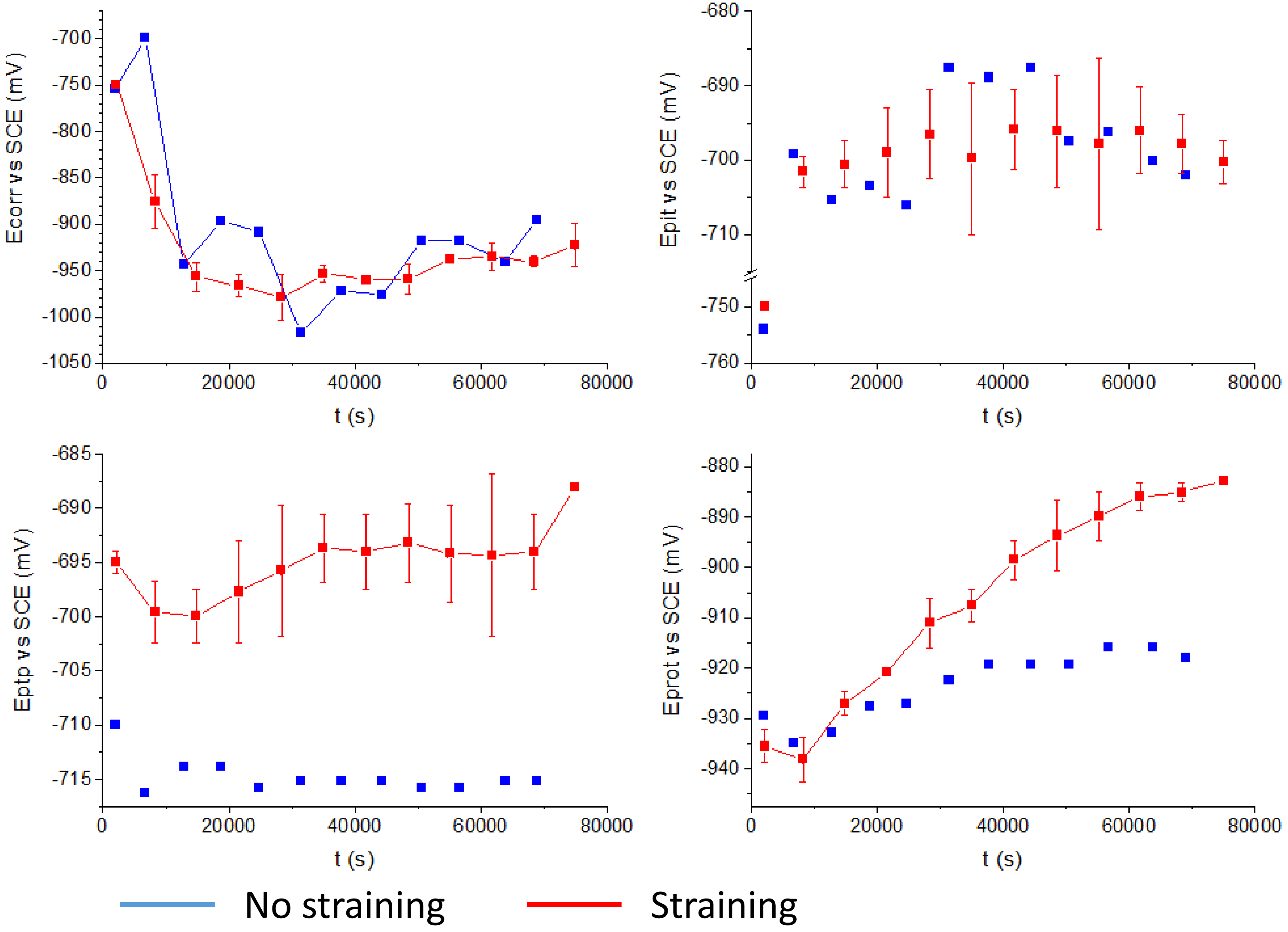
Al 7075-T6

Electrochemical potentials **0.6 M NaCl 0.1667 mV/s**

Typical pitting scan curves
(with and without straining)



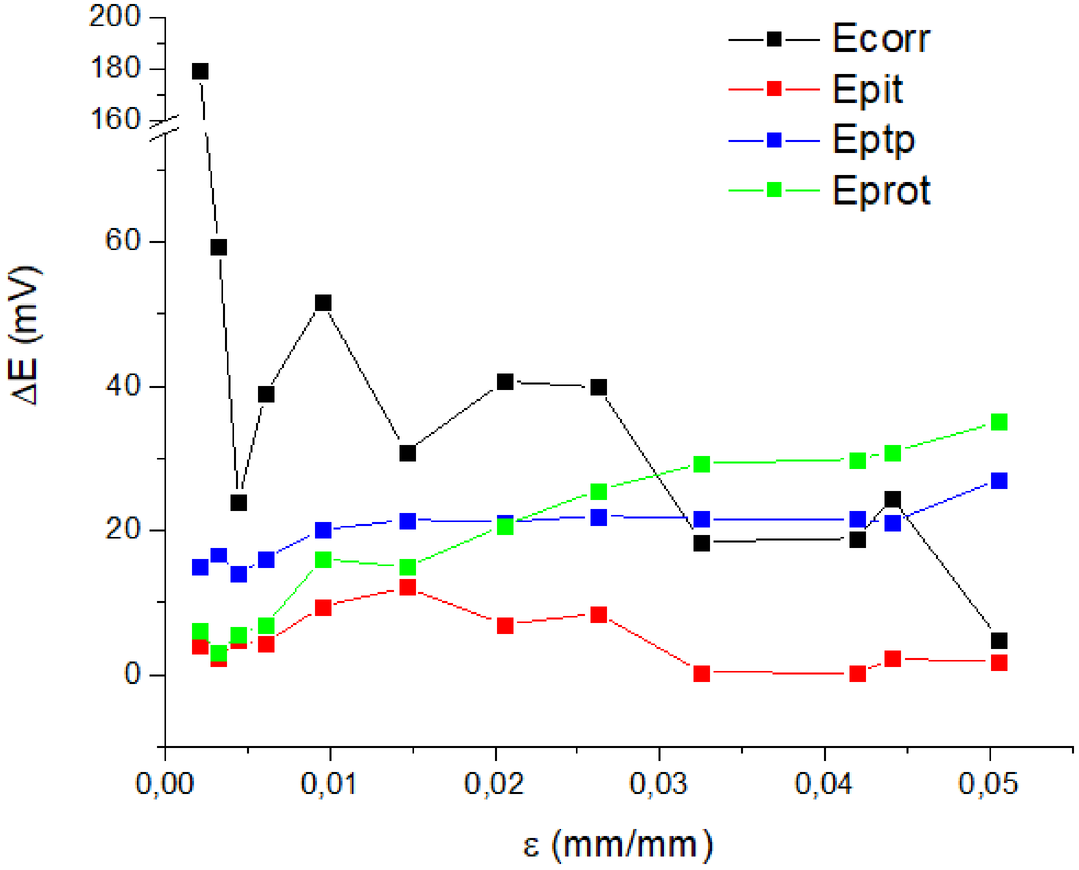
$\dot{\varepsilon} = 10^{-6}\ s^{-1}$



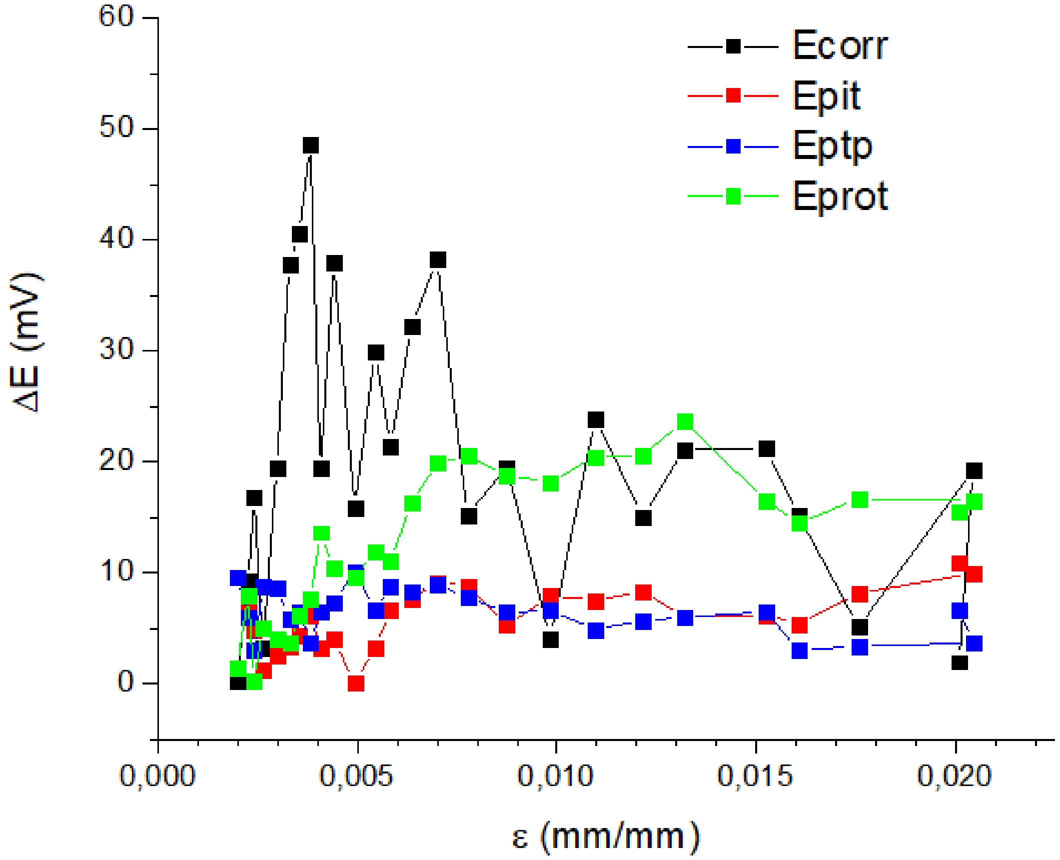
Al 7075-T6

Electrochemical potentials **0.6 M NaCl 0.1667 mV/s**

$\dot{\varepsilon}$ = 10^{-6} s^{-1}



$\dot{\varepsilon}$ = 10^{-7} s^{-1}



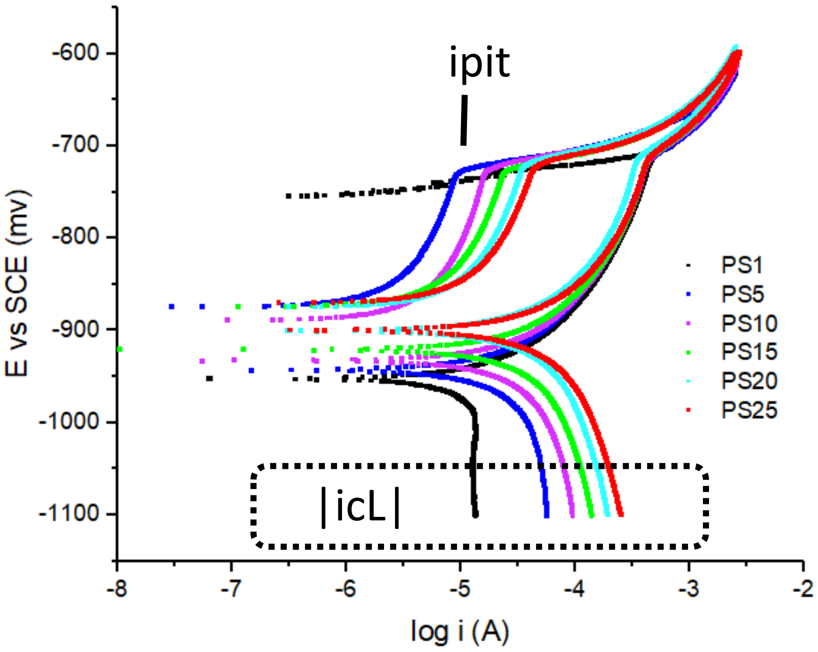
ΔE – absolute difference between a given E with and without straining

The difference tends to be less important as the strain rate decreases - nearly stationary conditions

Al 7075-T6

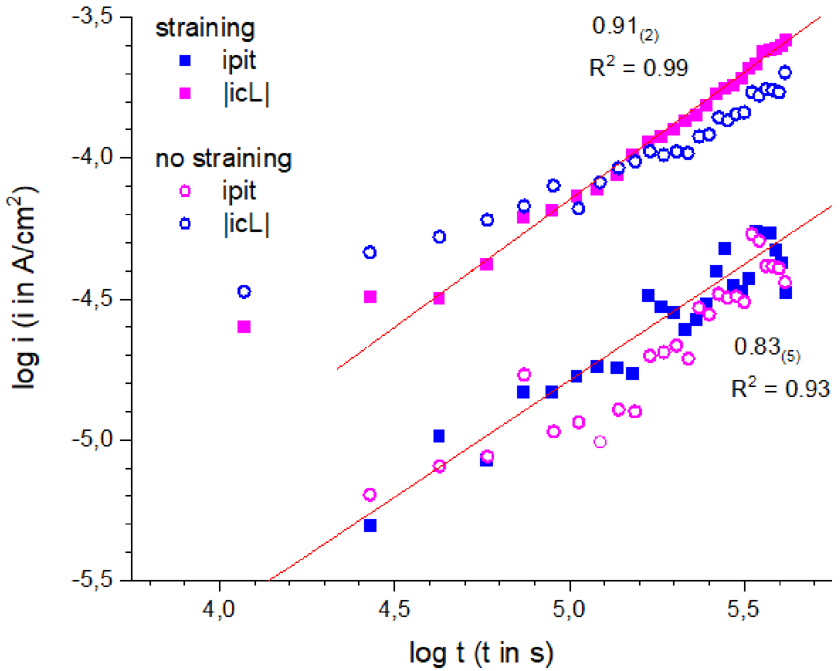
Current densities **0.6 M NaCl 0.1667 mV/s**

Typical pitting scan curves
(with and without straining)

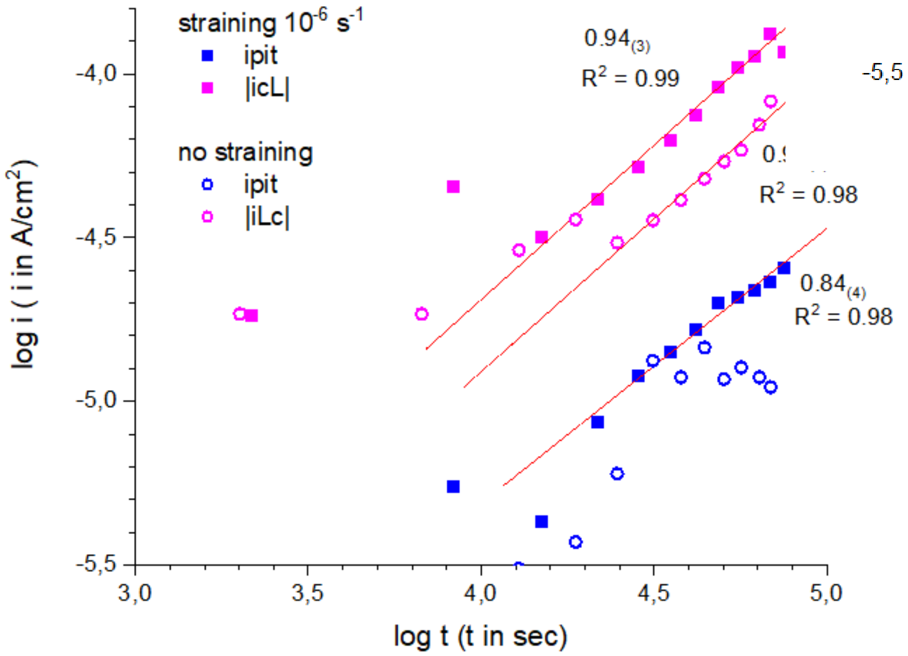


log |ipit| - log t plots (blue symbols)
linear relationships
slope ≅ 1 rehardless $\dot{\varepsilon}$

$\dot{\varepsilon} = 10^{-7}\ s^{-1}$



$\dot{\varepsilon} = 10^{-6}\ s^{-1}$



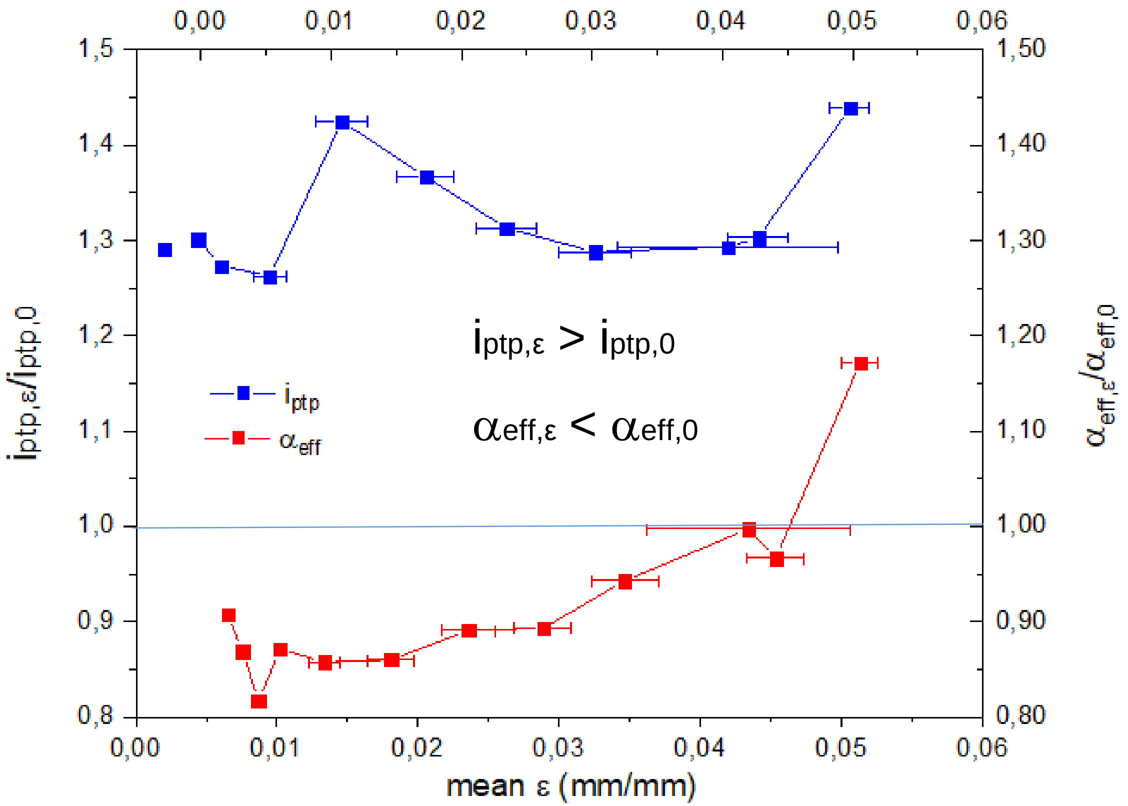
log |icL| - log t plots (pink symbols)
linear relationships
slope ≅ 1 all cases
No cathodic corrosion but H_2↑

$$H_2O + e = \frac{1}{2}H_2 + OH^-$$

Al 7075-T6 Kinetic properties of repassivation **0.6 M NaCl 0.1667 mV/s**

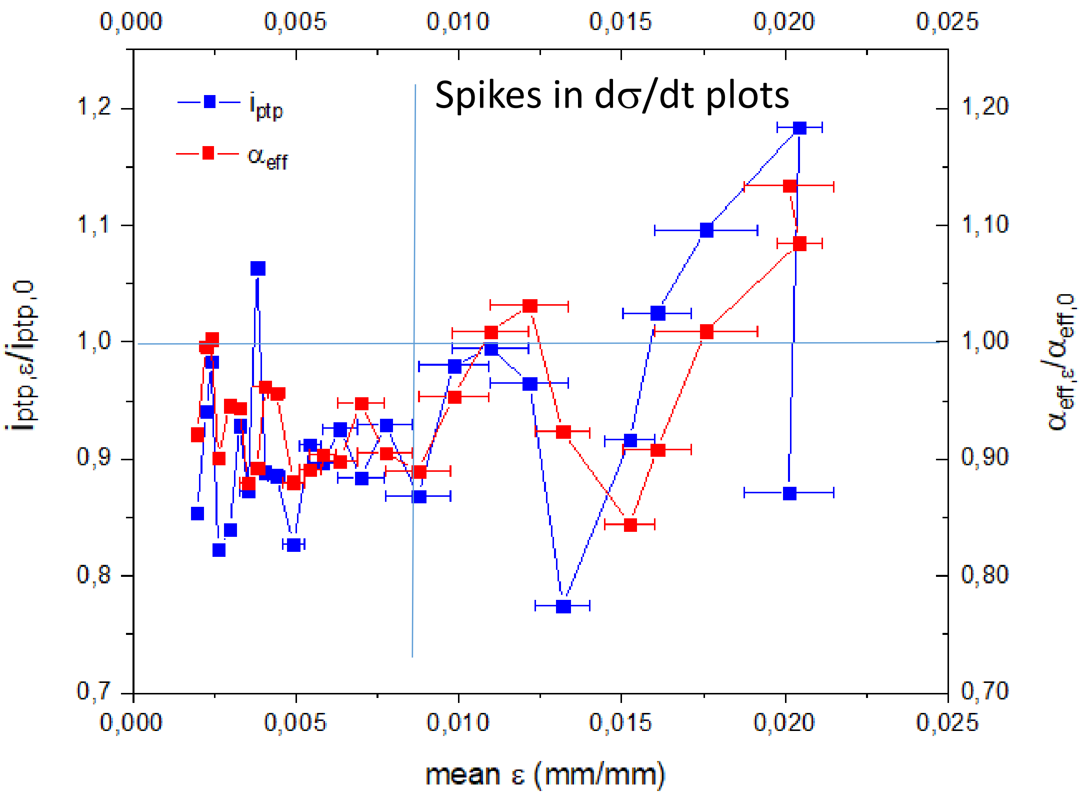
ratios $i_{ptp,\varepsilon}/i_{ptp,0}$ and $\alpha_{eff,\varepsilon}/\alpha_{eff,0}$ as a function of ε (ε – straining, 0 – no straining)

$\dot{\varepsilon} = 10^{-6}$ s^{-1}



Corrosion and repassivation promoted with creation of fresh surfaces due to straining

$\dot{\varepsilon} = 10^{-7}$ s^{-1}

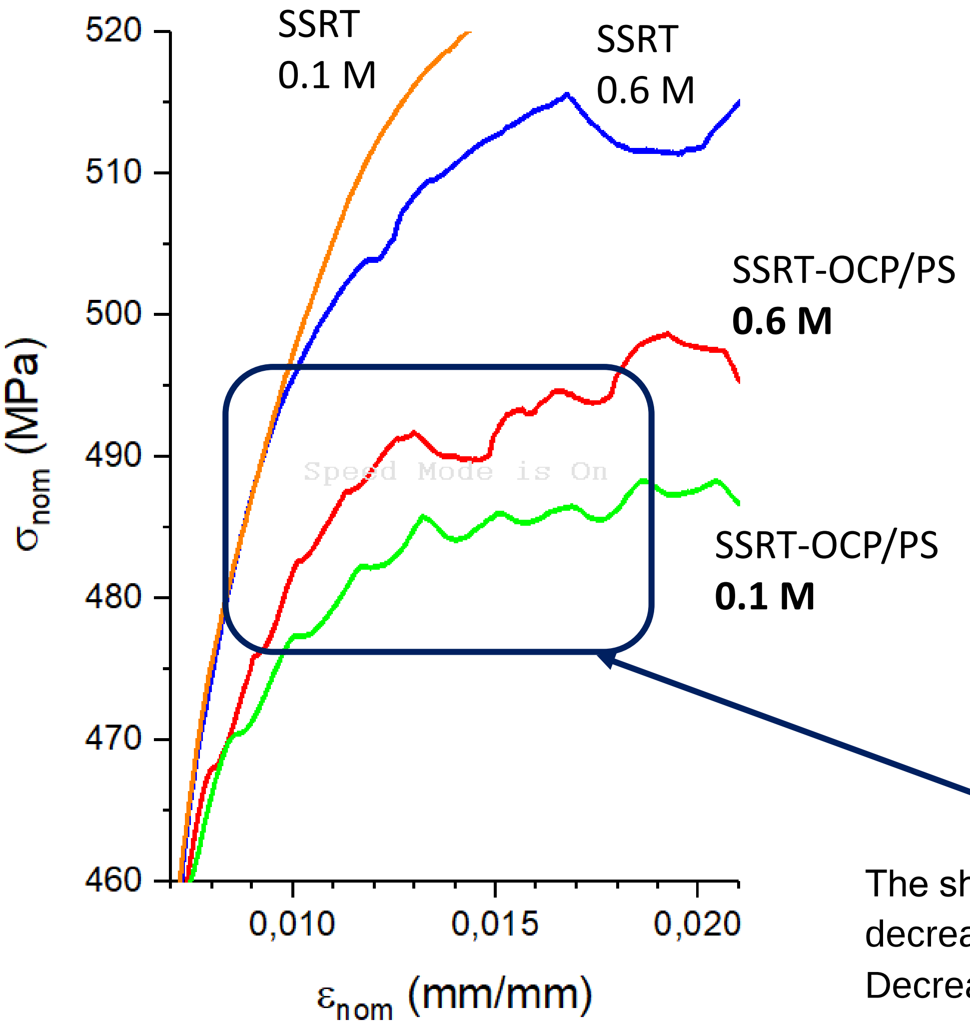


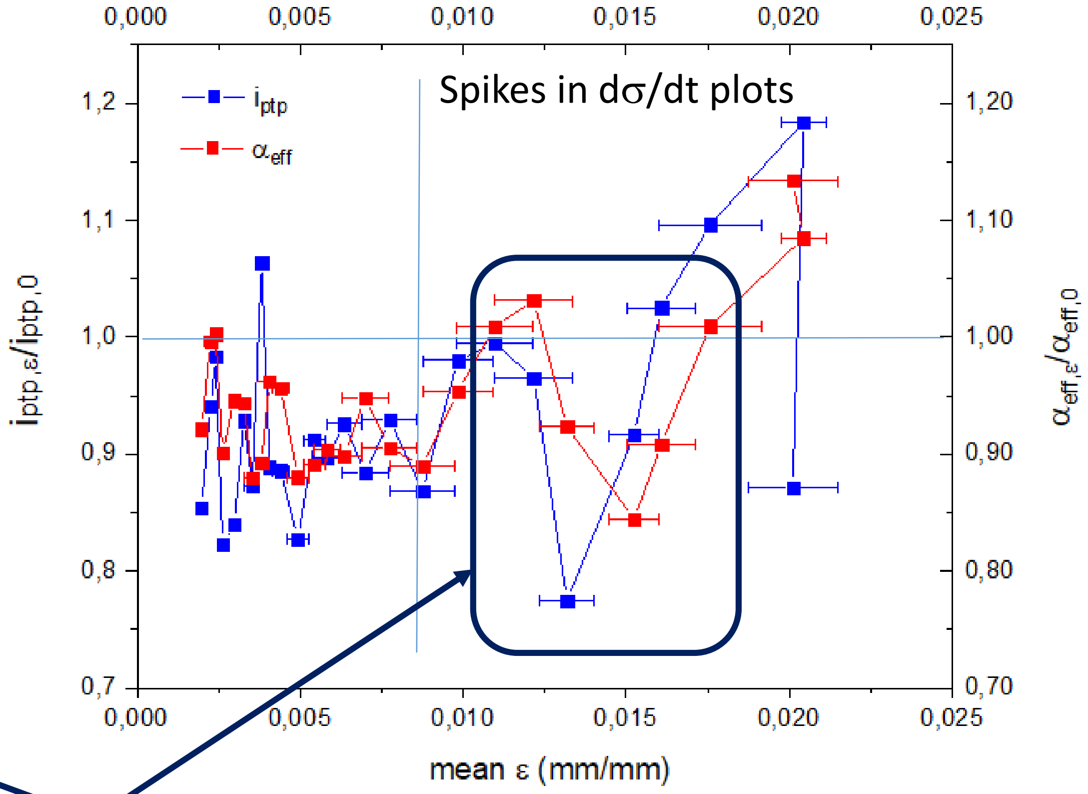
Non monotonic and similar variation of $i_{ptp,\varepsilon}$ and $\alpha_{eff,\varepsilon}$ by decreasing the strain rate

Al 7075-T6 Kinetic properties of repassivation **0.6 M NaCl 0.1667 mV/s**

ratios $i_{ptp,\varepsilon}/i_{ptp,0}$ and $\alpha_{eff,\varepsilon}/\alpha_{eff,0}$ as a function of ε (ε – straining, 0 – no straining)

$$\dot{\varepsilon} = 10^{-7}\ s^{-1}$$





The shape of the "serrations" changes in correspondence with a transition from decreasing to increasing $i_{ptp,\varepsilon}/i_{ptp,0}$ and $\alpha_{eff,\varepsilon}/\alpha_{eff,0}$

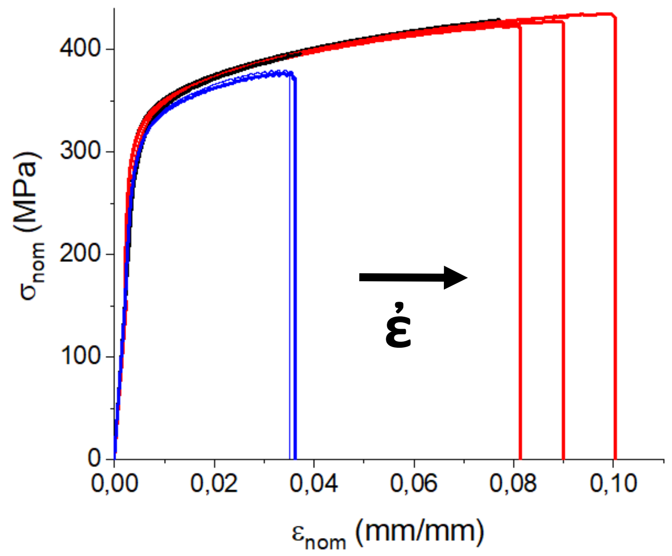
Decreasing $i_{ptp,\varepsilon}/i_{ptp,0}$ and $\alpha_{eff,\varepsilon}/\alpha_{eff,0}$ ➡ $d\sigma/dt \cong 0$

Al 2024-T3 (some results)

(4.4Cu-1.6Mg)

Al 2024-T3

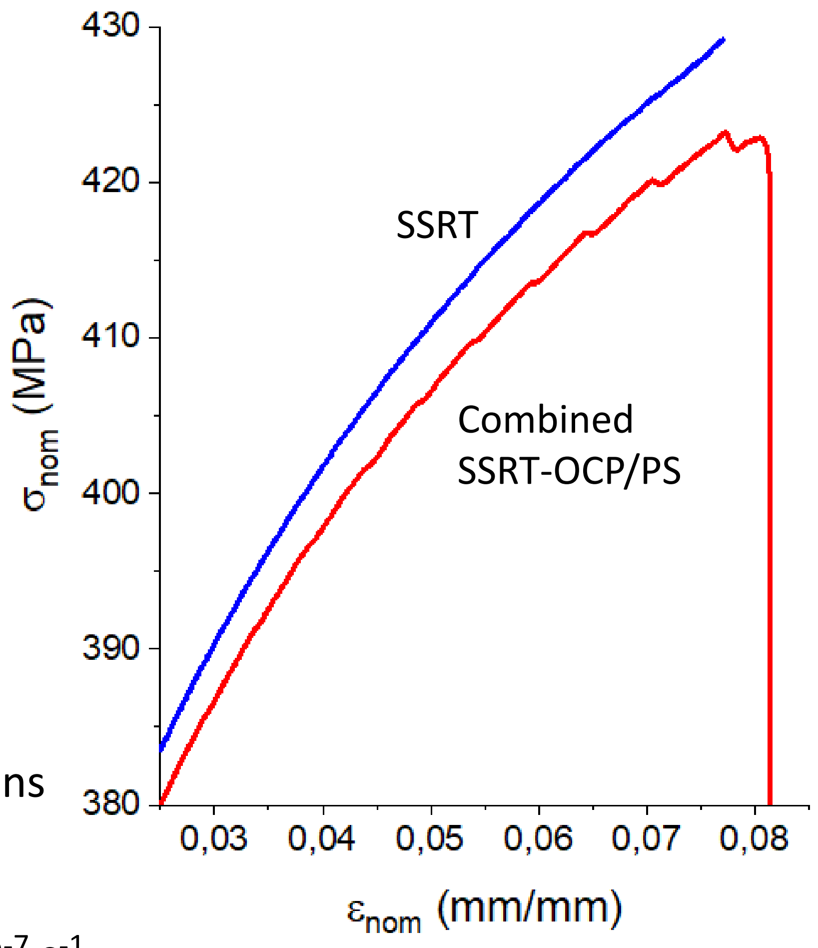
0.6 M NaCl 0.1667 mV/s



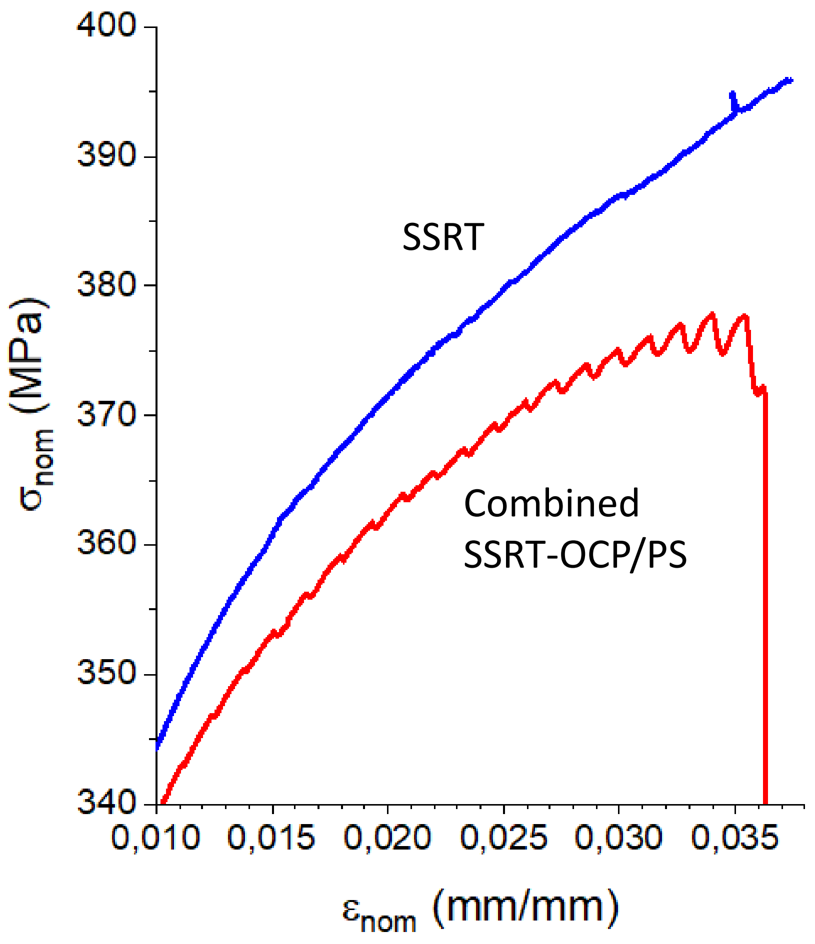
— **10^{-7} s^{-1}** (2 replications)

— **10^{-6} s^{-1}** (3 replications)

$\dot{\varepsilon} = 10^{-6}$ s^{-1}



$\dot{\varepsilon} = 10^{-7}$ s^{-1}



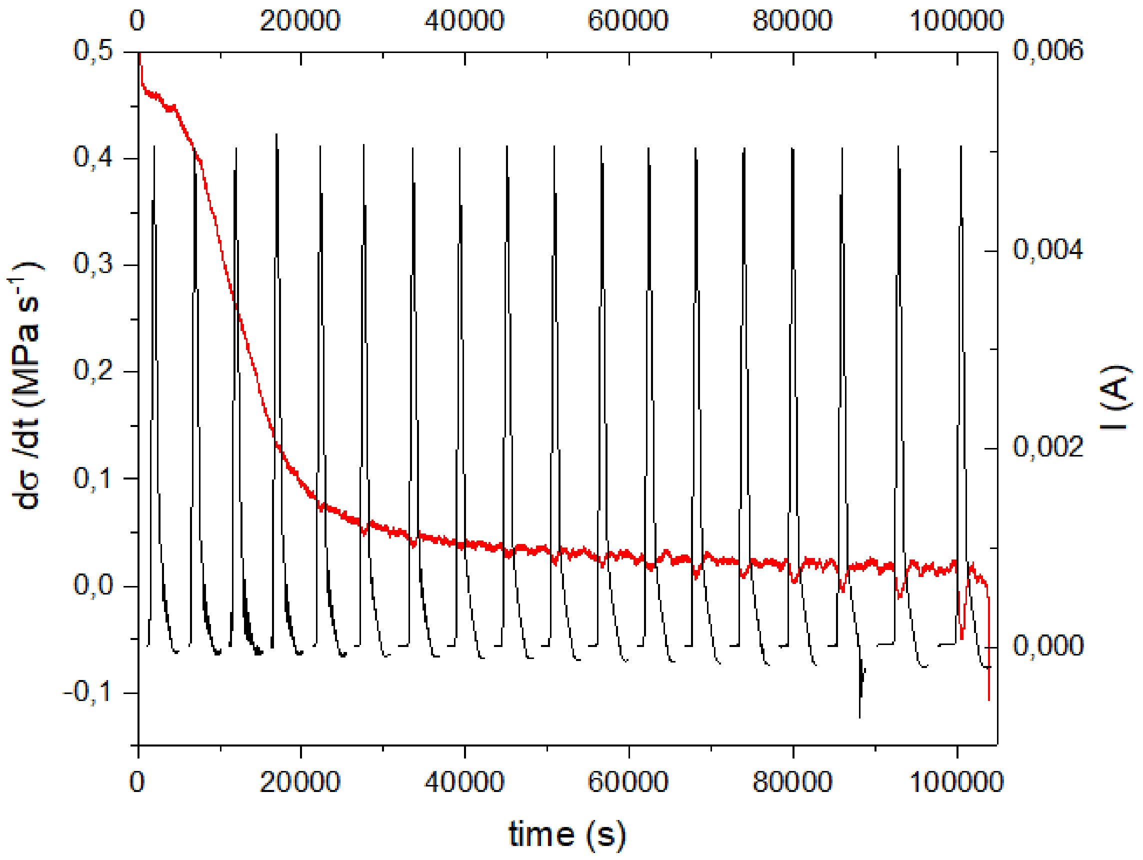
Stress relaxation/recovery events
ONLY in elasto-plastic and plastic regions for $\dot{\varepsilon} \leq 10^{-6}$ s^{-1} with OCP/PS sequence

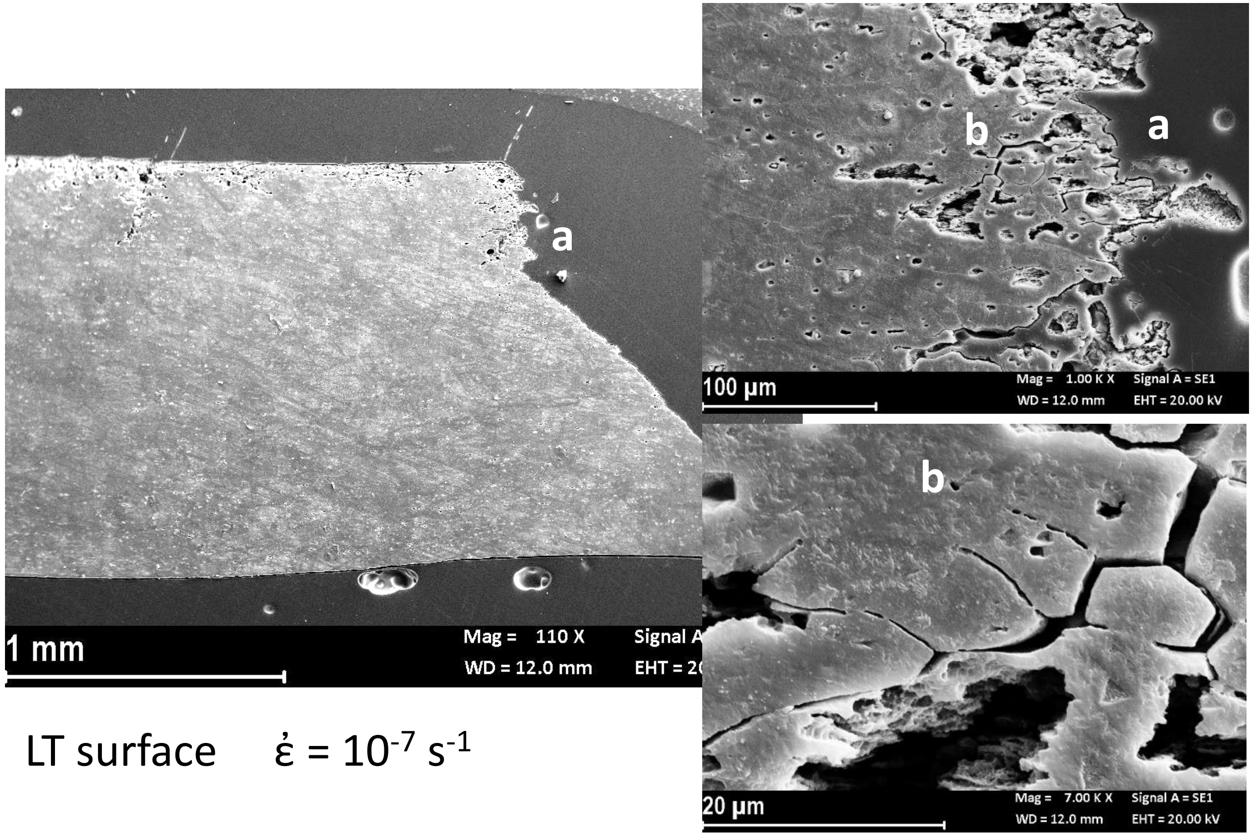
«Serrations» better resolved for $\dot{\varepsilon} = 10^{-7}$ s^{-1}

Al 2024-T3

0.6 M NaCl 0.1667 mV/s

ε̇ = 10^{-6} s^{-1}





LT surface ε̇ = 10^{-7} s^{-1}

Resolved spikes upon derivation of the stress – time curves in correspondence with the anodic polarization cycle for both ε̇ = 10^{-6} s^{-1} and 10^{-7} s^{-1}

Final remarks

From the combination of SSRT and corrosion/repassivation sequences:

- ❑ Decrease of the strain rate and of the yield strength regardless the electrochemical perturbation
- ❑ Stress relaxation/recovery events induced with anodic polarization cycle

Under nearly-stationary conditions (slow strain and potential scan rates):

- ❑ Crack nucleation and propagation enhanced during anodic dissolution
- ❑ Stress recovery time dependent on crack morphology
- ❑ Correlation between the repassivation behavior and the characteristics of stress relaxation/recovery events

Thank you for the attention