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
Single cycle anodic polarization and repassivation properties

Halide film → Oxide film at pit bottom

Letter E (V) Mz+ O2- Metal

Letter E (V) H2O Metal

Metal

Solution

Halide film

Oxide film at pit bottom

Letter E (V) I (A) Solution

Single cycle anodic polarization (Pitting Scan, PS)

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

iptp ∝ rate hydrolysis equilibrium at [Al³⁺]_crit

2Al³⁺ + H₂O + OH⁻ ↔ 2Al(OH)²⁺ + H⁺

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

Effective anodic charge transfer coefficient αₜₐₑffe * estimated from the steepness by the equation:

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

Increasing steepness → αₑffective → 0 (α → λ) → Accelerating action of Cl⁻

αeff = α - λ.

α - anodic charge transfer coefficient

λ - the effective kinetic order of metal dissolution with respect to [Cl⁻]
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 with multiple SSRT machine (4 load cells, 30 kN each)

Smooth tensile test specimen

<table>
<thead>
<tr>
<th>Alloy</th>
<th>7075-T6</th>
<th>2024-T3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (mm)</td>
<td>2.0</td>
<td>1.5</td>
</tr>
<tr>
<td>Elastic Modulus E (GPa)</td>
<td>74.5</td>
<td>75.7</td>
</tr>
<tr>
<td>Yield strength YS, Rp02 (MPa)</td>
<td>510</td>
<td>354</td>
</tr>
<tr>
<td>Ultimate tensile strength UTS (MPa)</td>
<td>583</td>
<td>499</td>
</tr>
<tr>
<td>Gauge section area (mm²)</td>
<td>40</td>
<td>32</td>
</tr>
</tbody>
</table>
Slow strain rate test and pitting scan in combination

Electrochemical setup

Gamry Interface 1000 multipotentiostat

Separate two compartments (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

Working electrodes (WE)
Opposite surfaces of the tensile specimen
(geometrical area 2 cm² each)
Slow strain rate test and pitting scan in combination

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

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

- $E_i = E_{corr}$
- $i_{rev} = 2.5 \text{ mA/cm}^2$
- $E_f = -1.1 \text{ V (vs SCE)}$

Two-electrode configuration
OCP monitoring during straining

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

Output Data
- Load (t)
- OCP ($\Delta t$)
- $E, i, Q (\Delta t)$
- OCP (t)

Stagnant NaCl (pH 6.5)
Room T ($\geq 25 ^\circ C$)
Al 7075-T6
(5.8 Zn-2.6Mg-1.7Cu)
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 \( \dot{\varepsilon} \leq 10^{-6} \text{ s}^{-1} \) with OCP/PS sequence

\[ \dot{\varepsilon} = 10^{-6} \text{ s}^{-1} \quad 0.6 \text{ M NaCl} \]

\[ \dot{\varepsilon} = 10^{-7} \text{ s}^{-1} \quad 0.1667 \text{ mV/s} \]

\( \Delta\sigma \approx 5 \text{ MPa} \)
\[ \dot{\varepsilon} = 10^{-6} \text{s}^{-1} \quad 0.6 \text{M NaCl} \quad 0.1667 \text{mV/s} \]

Resolved spikes upon derivation of the stress – time curves
Load drop in correspondence with the anodic polarization cycle
Al 7075-T6

\[ \dot{e} = 10^{-7} \text{ s}^{-1} \quad 0.1 \text{ M NaCl} \quad 0.1667 \text{ mV/s} \]

Spikes in \( \sigma/\text{dt} - t \) curves better resolved for \( \dot{e} = 10^{-7} \text{ s}^{-1} \) and with dilution of NaCl solution

The correspondence with anodic dissolution/repassivation shown in \( \log I - t \) curves

\( \sigma/\text{dt} \) decreases once localized corrosion onsets at \( E_{\text{pit}} \)
Al 7075-T6

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

0.1 M NaCl  0.1667 mV/s

0.6 M NaCl  0.1667 mV/s

Chloride ions concentration influences the time interval during which $d\sigma/dt$ increases along wise with the repassivation response.
Al 7075-T6

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

0.1 M NaCl  0.1667 mV/s

$\varepsilon = 10^{-7}$

s

Al 7075

- T6

0.6 M NaCl  0.1667 mV/s

OCP

3 h

LT surface

Sharp cracks prevalently developed in 0.6 M NaCl
Al 7075-T6

FFT— from time to frequency domains

\[ \dot{\varepsilon} = 10^{-6} \text{ s}^{-1} \]

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

0.6 M NaCl

\[ \varepsilon = 10^{-6} \text{ s}^{-1} \]
\[ \varepsilon = 10^{-7} \text{ s}^{-1} \]

0.1667 mV/s

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

TISA – time interval square amplitude

\[ \Delta t \left( Re^2 + Im^2 \right) \]
\[ n \]

Re, Im – real and imaginary parts of the transform data
n – length of the input sequence
\( \Delta t \) – sampling interval
Al 7075-T6

Comparison of OCP variation with time

0.6 M NaCl

\[ \dot{\varepsilon} = 10^{-6} \text{ s}^{-1} \]

OCP tends to increase with time during dynamic straining at \( 10^{-6} \text{ s}^{-1} \).

The trend is opposite for \( 10^{-7} \text{ s}^{-1} \) 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

\[ \dot{\varepsilon} = 10^{-6} \text{ s}^{-1} \]

Typical pitting scan curves (with and without straining)
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
Typical pitting scan curves (with and without straining)

\[
\text{Typical pitting scan curves (with and without straining)}
\]

\[\log |\text{ipit}| - \log t \text{ plots (blue symbols)} \]
linear relationships
slope \(\approx 1\) rehardless \(\dot{\varepsilon}\)

\[\dot{\varepsilon} = 10^{-6} \text{ s}^{-1}\]

\[\dot{\varepsilon} = 10^{-7} \text{ s}^{-1}\]

log \(|\text{icL}|\) - log t plots (pink symbols)
linear relationships
slope \(\approx 1\) all cases
No cathodic corrosion but \(H_2\uparrow\)

\[H_2O + e^- = \frac{1}{2} H_2 + OH^-\]
Kinetic properties of repassivation

\[
\dot{\varepsilon} = 10^{-6} \text{ s}^{-1}
\]

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

\[
\dot{\varepsilon} = 10^{-7} \text{ s}^{-1}
\]

Spikes in \( d\sigma/dt \) plots

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

Non monotonic and similar variation of \( i_{\text{ptp},\varepsilon} \) and \( \alpha_{\text{eff},\varepsilon} \) by decreasing the strain rate
Al 7075-T6  

Kinetic properties of repassivation  

0.6 M NaCl  0.1667 mV/s

ratios $\frac{i_{\text{ptp}, \varepsilon}}{i_{\text{ptp}, 0}}$ and $\frac{\alpha_{\text{eff}, \varepsilon}}{\alpha_{\text{eff}, 0}}$ as a function of $\varepsilon$ ($\varepsilon$ – 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 $\frac{i_{\text{ptp}, \varepsilon}}{i_{\text{ptp}, 0}}$ and $\frac{\alpha_{\text{eff}, \varepsilon}}{\alpha_{\text{eff}, 0}}$

Decreasing $i_{\text{ptp}, \varepsilon}/i_{\text{ptp}, 0}$ and $\alpha_{\text{eff}, \varepsilon}/\alpha_{\text{eff}, 0}$  

$\sigma_{\text{nom}}$ (MPa)  

SSRT 0.1 M  

SSRT 0.6 M  

SSRT-OCP/PS 0.6 M  

SSRT-OCP/PS 0.1 M  

Spikes in $d\sigma/dt$ plots  

mean $\varepsilon$ (mm/mm)
Al 2024-T3 (some results)
(4.4Cu-1.6Mg)
Al 2024-T3

0.6 M NaCl 0.1667 mV/s

\[ \dot{\varepsilon} = 10^{-6} \text{ s}^{-1} \]

\[ \dot{\varepsilon} = 10^{-7} \text{ s}^{-1} \]

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

«Serrations» better resolved for \( \dot{\varepsilon} = 10^{-7} \text{ s}^{-1} \)
Resolved spikes upon derivation of the stress – time curves in correspondence with the anodic polarization cycle for both $\dot{\varepsilon} = 10^{-6}$ 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