Hydrogen Induced Cracking in X70 and X52 Pipeline Steels

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Garrett Angus
Outline

• Hydrogen induced cracking overview
  – Hydrogen trapping
  – Effect of microstructure morphology, inclusions and hard bands

• Current work
  – Intergranular crack characterization
  – Comparison of as-received vs damaged boundaries

• Future work
  – Maintain chemistry, change thermo-mechanical processing
Project Motivation

- Pipeline steels subjected to HIC through “sour” service environments
- As strength increases, HIC susceptibility considered to increase
  - Factors affecting HIC include inclusions and regions of high hardness
  - What strengthening mechanisms are leading to this HIC susceptibility at higher hardness?

Y. Nakai et al., Technical Report, Kawasaki Steel, 1980
Hydrogen Trapping

- **Reversible**
  - Dislocations
  - Coherent precipitate interfaces

- **Irreversible**
  - Some grain boundaries
  - Voids
  - Cracks
  - Incoherent precipitate interfaces

J. Takahashi et al., *Scripta Met.*, 2012
Effect of Inclusions Hardness

- Elongated MnS particles are detrimental to HIC
- Clusters of oxides also increase susceptibility

J. Nieto et al., MS&T Proceedings, 2012

Brown and Jones., Corrosion, 1984
Possible Cracking Mechanism

- Crack initiation occurs when hydrogen diffuses to irreversible traps and recombines to form molecular hydrogen.
- Crack propagation is limited by hydrogen diffusion to crack.


Findley et al., *Mat Sci and Tech*, 2015
Research Objectives

✓ Evaluate HIC of four different alloys and microstructures

• Evaluate the microstructure in the vicinity of HIC
  – Grain boundary types
    • High angle, low angle, coincident site lattice?
  – Banding
  – Secondary microconstituents

• Design, produce and test experimental steels
## Experimental Background

<table>
<thead>
<tr>
<th>wt pct</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Ti</th>
<th>Nb</th>
<th>V</th>
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</thead>
<tbody>
<tr>
<td>X52</td>
<td>0.067</td>
<td>1.03</td>
<td>0.24</td>
<td>0.10</td>
<td>0.06</td>
<td>0.0007</td>
<td>0.044</td>
<td>0.007</td>
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<tr>
<td>X70</td>
<td>0.050</td>
<td>1.59</td>
<td>0.3</td>
<td>0.01</td>
<td>0.26</td>
<td>0.013</td>
<td>0.066</td>
<td>0.005</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>wt pct</th>
<th>Mo</th>
<th>Al</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>Cu</th>
<th>Ca</th>
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<tbody>
<tr>
<td>X52</td>
<td>0.03</td>
<td>0.031</td>
<td>0.0082</td>
<td>0.0007</td>
<td>0.010</td>
<td>0.35</td>
<td>0.0027</td>
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<tr>
<td>X70</td>
<td>0.09</td>
<td>0.026</td>
<td>0.0081</td>
<td>0.003</td>
<td>0.010</td>
<td>0.01</td>
<td>-</td>
</tr>
</tbody>
</table>

- Low carbon microalloyed steels
- X52 and X60 are approved for sour service use
As-Received Microstructures

IQ maps with grain boundaries overlaid

X52

X70

IQ maps with grain boundaries overlaid
Identifying Features

- **Intergranular**
- **Transgranular**
- **High < GB**
- **Low < GB**
- **Crack Plane**
- **M/A**
Crack Propagation

- Crack propagation both transgranular and intergranular
- May indicate some boundaries preferentially crack over others
  - Therefore, a grain boundary study was initiated to look at intergranular cracking
• 5 independent parameters necessary to fully characterize boundary $\lambda(\Delta g, n)$
  – 3 to describe misorientation matrix $\Delta g = g_1^{-1} g_2$
  – can be described as rotation $\omega$ about axis $[uvw]$
5 independent parameters necessary to fully characterize boundary $\lambda(\Delta g, n)$
- 2 to describe grain boundary plane ($n$)
Damaged Boundary Analysis

Over 1200 misorientation pairs analyzed
Crack Propagation

- Image quality maps with grain boundary misorientations overlaid might show plastic deformation near crack
  - Need to be sure that these clusters of low angle boundaries were not present before testing
X70 MacKenzie Plots

- X70 As-Received
- X70 NACE
- Average of NACE and Cathodic
- X70 Cathodic

Number Fraction vs. Misorientation Angle (Degrees)
Misorientation Space

- Rodrigues-Frank space
- $R = uvw \tan(\omega/2)$
- Components of $R$ ($r_1, r_2, r_3$)

\[\begin{align*}
r_3 &= 0.0000 \\
0.0238 & \quad 0.0476 & \quad 0.0714 \\
0.1190 & \quad 0.1429 & \quad 0.1667 & \quad 0.1905
\end{align*}\]
Rodrigues-Frank space can allow for identification of special coincident site lattice boundaries.
Coincident Site Lattice Theory

$\Sigma 5$

36.9°

CSLs might have lower energy configurations than random high angle boundary
X70 As-Received Microstructure

Σ29a → 46.3°∥<100>  Σ27a → 31.2°∥<110>
X52 NACE Mackenzie Plot

![Graph showing misorientation angle vs. number fraction for X52 NACE and X52 As-Received samples.](image-url)

- **X52 NACE**
- **X52 As-Received**
X52 As-Received vs. Damaged RF

Low angle

\[ r_3 = 0.0000 \]

\[ \Sigma 13b \]

\[ \begin{align*}
0.1190 \\
0.1429 \\
0.1667 \\
0.1905 \\
0.2143 \\
0.2381 \\
0.2619 \\
0.2857 \\
0.3095 \\
0.3333 \\
\end{align*} \]

\[ \max = 12.529 \]

\[ \begin{align*}
8.221 \\
5.394 \\
3.540 \\
2.323 \\
1.524 \\
1.000 \\
0.656 \\
\end{align*} \]
Experimental Heats
**Experimental Heats**

<table>
<thead>
<tr>
<th>Approximate X65 Chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>wt pct</strong></td>
</tr>
<tr>
<td>Exp. Target*</td>
</tr>
</tbody>
</table>

| **wt pct** | Mo | Al | N | S | P | Cu | Ca |
| Exp. Target* | - | 0.040 | 0.0050 | 0.0020 | 0.012 | - | 0.0010 |

*Courtesy of ArcelorMittal

- **Purpose**: examine effect of varying microstructure due to different TMP paths while maintaining same composition.
Thermomechanical Processing

![Diagram showing temperature and strain with key stages: Rough Rolling at ~1004°C, Finish Rolling at ~794°C, and Air Cool at ~645°C.]

- Rough Rolling at ~1004°C
- Finish Rolling at ~794°C
- Air Cool at ~645°C

Key temperatures:
- $T_{NR}$: ~1004°C
- $Ar_3$: ~794°C
- $Ar_1$: ~645°C
Thermomechanical Processing

- **Accelerated cooled microstructure**
  - Ferrite/ possible bainite or acicular ferrite

- **Air cooled microstructure**
  - Ferrite and pearlite

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<table>
<thead>
<tr>
<th>Yield Strength (ksi)</th>
<th>Tensile Strength (ksi)</th>
</tr>
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<tbody>
<tr>
<td>69.8</td>
<td>99.5</td>
</tr>
<tr>
<td>73</td>
<td>82</td>
</tr>
</tbody>
</table>

Preliminary mechanical properties provided by Arcelor Mittal
Summary

• Cracking occurs both transgranularly and intergranularly

• $\Sigma3$ boundaries do not appear resistant to cracking in either X52 or X70

• $\Sigma70$ NACE and cathodically charged
  – $\Sigma29a$ boundaries might be resistant to cracking

• $\Sigma52$ NACE charged
  – No high MRD boundaries with high HIC resistance

Thank you
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