Lab testing to qualify Ti Gr 12 (UNS R53400) for potential ballot for Inclusion in NACE MR0175 / ISO15156-3

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Introduction to Exova

Exova

KEY CUSTOMER SECTORS

- Aerospace
- Oil & Gas and Industrials
- Construction
- Fire and Building Products
- Transportation
- Food and Pharmaceuticals
- Environmental

KEY TECHNICAL DISCIPLINES

- Polymers and composites
- Corrosion and protection
- Metal technology
- Fire
- Structures and systems
- Calibration
- Chemistry and microbiology

- c. 25,000 customers
- More than 90 years’ experience
- c. 4,500 experts
- 143 laboratories and offices in 32 countries

INNOVATION. TEAMWORK. PERFORMANCE. INTEGRITY.
Sour Service Standard, MR0175

• Following valve failures in 1970s, NACE (National Association of Corrosion Engineers) developed a standard for materials recommendations to avoid sulphide stress cracking issued in 1975

• Continuously updated, over time standard expanded to additional applications and alloys were added by balloting data.
Sour Service Standard, MR0175 / ISO15156

• “CRA rewrite” issued March 2003
• US and European approaches merged into MR0175/ISO15156 in late 2003
• Changes
  – Test parameters for alloys changed (but existing alloys retained/grand-fathered)
  – Broader range of damage mechanisms
  – Move towards User responsibilities for selection of appropriate material etc.
• Latest edition ISO15156-2015
• Included without restriction on sour service conditions (ISO15156-3 A.11, Table A.41 for any equipment or component), but:

  – UNS R53400 shall be in the annealed condition. Heat treatment shall be annealing at \((774 \pm 14) ^\circ C [(1425 \pm 25) ^\circ F]\) for 2 h followed by air-cooling. Maximum hardness shall be 92HRB;

  – Hardness has not been shown to correlate with susceptibility to SSC/SCC. However, hardness has been included for alloys with high strength to indicate the maximum testing levels at which failure has not occurred.
The problem...

- Manufacturing limitation
  - Arose from original ballot for specific products which were batch annealed
  - This restriction precludes manufacturing with continuous processing
- Data needed to provide basis of ballot to add new entry
  - It cannot be “unrestricted service” so the NACE Level VII conditions have been selected
Production Process for Titanium Plate

1. Electron Beam Cold Hearth Melt
2. Forge Ingot to Slab
3. Bloom Slab to Plate
4. Hot Roll Plate to Final Gauge
5. Anneal and Level Plate
6. Surface Condition and Pickle Plate
7. Inspect, Line Mark & Cut Plate to Final Size
8. Test and Certify
Balloting

- ISO15156-3:2015 Annex B provides clear guidance
  - Three heats of material
  - Material in final product form
  - Assessment including
    - Microstructural evaluation
    - Chemical composition
    - Hardness
    - Mechanical properties
Testing Requirements

• Testing for both primary and secondary damage mechanisms (SSC, GHSC, SCC)
• Use of methods acceptable per Annex B
  • Note: required loads of 100% AYS / 0.2%PS in contrast to earlier requirements for SCC
  • Hardness not expected to correlate to SCC resistance but still cited in Table A.41
  • Test results on parent materials only will be submitted as are all data in ISO15156-3…
    » Note Seam welds from tube may be assessed later for comparison as hardness > parent

Test Geometries

• Parent materials
  • Triplicate uniaxial tensile specimens per sample

• Weldments if tested
  • Triplicate C-ring specimens per sample
Conditions for testing

- **SSC per TM0177 Method A**
  - Parent material
  - 1bara P[H₂S], 90%AYS, 75F(24°C) – 30days

- **GHSC per TM0177 Method A**
  - Parent material
  - 1bara P[H₂S], 90%AYS, 75F(24°C) – 30days
  - Specimens coupled to C-steel

- **SCC**
  - Parent (Method A) 100%AYS at 205°C (400F)
  - NACE Level VII conditions with elemental sulphur added
  - 90 days
### Test Conditions “NACE Level VII”

#### Table E.1 — Test conditions

<table>
<thead>
<tr>
<th>Environmental factor</th>
<th>Level I</th>
<th>Level II</th>
<th>Level III</th>
<th>Level IV</th>
<th>Level V</th>
<th>Level VI</th>
<th>Level VII</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature °C (°F)</td>
<td>25 ± 3 (77 ± 5)</td>
<td></td>
<td></td>
<td>90 ± 5 (194 ± 9)</td>
<td>150 ± 5 (302 ± 9)</td>
<td>175 ± 5 (347 ± 9)</td>
<td>205 ± 5 (401 ± 9)</td>
</tr>
<tr>
<td>$P_{CO2}$ MPa (psi)</td>
<td></td>
<td></td>
<td></td>
<td>0,7 (100)</td>
<td>1,4 (200)</td>
<td>3,5 (500)</td>
<td>3,5 (500)</td>
</tr>
<tr>
<td>$P_{H2S}$ MPa (psi)</td>
<td></td>
<td></td>
<td></td>
<td>0,003 (0,4)</td>
<td>0,7 (100)</td>
<td>3,5 (500)</td>
<td>3,5 (500)</td>
</tr>
<tr>
<td>NaCl minimum percentage mass fraction</td>
<td>Test conditions defined and documented by the user</td>
<td>Test in accordance with B.4</td>
<td>Test in accordance with B.4 and B.8</td>
<td>15</td>
<td>15</td>
<td>20</td>
<td>25</td>
</tr>
<tr>
<td>Calculated Cl$^{-}$ milligrams per litre</td>
<td>101 000</td>
<td>101 000</td>
<td>139 000</td>
<td>180 000</td>
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<td>pH</td>
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<td>$S^0$</td>
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<td>Galvanic coupling to steel</td>
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<tr>
<td>Other</td>
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</tr>
</tbody>
</table>

See B.3.5.1 and B.3.5.2

Optional (see B.7)

Optional; see Clause B.8

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*The equivalent mg/l concentration for ambient temperature used in Tables A.1 to A.42 was calculated from the corresponding percentage mass fraction value.*

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Outcome

• Post test evaluation
  – Detailed visual and metallographic assessment for presence of cracks
• Reported to ITA
• If appropriate, prepare ballot to ISO15156/MR0175 Maintenance Panel to allow continuous processing
  – Potential presentation to ISO15156 at EuroCorr, Montpellier Saturday 10 Sept.
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