Enhancing grain refinement of austenitic steel with Ti additions by melt treatment sequence optimization

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**Objective**

Grain size control is one of the most important aspects in the cast alloys processing.

**Objective:** To develop an advanced solidification process for controlling the crystallization structure of austenitic grades of cast steels in heavy sections.

- High alloyed austenitic steels are used in both the as-cast condition and heat treated conditions.
- The as cast grain size is important for all applications as it determines the minimum grain size achievable in these cast alloys.
Potential heterogeneous nuclei in base steel

Factor stability in the melt

19Cr18Ni superaustenitic stainless steel
Calculated stability of potential nuclei

- Ti > 0.2
- Zr > 0.2
- Hf > 0.25
- Nb > 2%

Phases, wt % vs Temperature, °C

Austenite
Liquid

- Oxides
Effect of additional de-oxidation

- What is the sequence of reactions during processing?
- How sequence of treatment will change composition of reaction products?
- What will be optimal sequence of melt treatment?

One step
- Refiner

Complex
- De-ox + Refiner

Sequential
- De-ox
- Refiner

[S] [Al]
[O] [Ca]
[N] [Refiner]
Simulated cases and experimental heats

<table>
<thead>
<tr>
<th>Heat #</th>
<th>Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>First</td>
</tr>
<tr>
<td>B</td>
<td>-</td>
</tr>
<tr>
<td>T1</td>
<td>Ti</td>
</tr>
<tr>
<td>T2</td>
<td>Al, Ca</td>
</tr>
<tr>
<td>T3</td>
<td>Ca</td>
</tr>
</tbody>
</table>
Effect of treatment sequences on precipitations

B0 (Al+Ca)

T1 (Ti+Al, Ca)

T2 (Ti+Al, Ca)

T3 (Ca+Al, Mg, Ti)
Experimental procedure: heavy section casting
Macrostructure

B2 base

T1 (IF: Ti, Ladle: deox)

T2 (Ladle: deox + Ti)
Chemistry of precipitates

<table>
<thead>
<tr>
<th>Element</th>
<th>Diameter Range</th>
<th>% of Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.5 - 2 µm</td>
<td>11.38 %</td>
</tr>
<tr>
<td></td>
<td>2 - 5 µm</td>
<td>43.43 %</td>
</tr>
<tr>
<td></td>
<td>5 - 10 µm</td>
<td>33.07 %</td>
</tr>
<tr>
<td></td>
<td>All</td>
<td>87.88 %</td>
</tr>
</tbody>
</table>

- B2 base
- T1 (Ti + deox)
- T2 (Deox + Ti)
Co-precipitation grain refinement approach

<table>
<thead>
<tr>
<th>Compound</th>
<th>Lattice parameter $a_0$ at 2800 °F, Å</th>
<th>Planar disregistry with TiN, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiN</td>
<td>4.308</td>
<td>-</td>
</tr>
<tr>
<td>MgAl$_2$O$_4$</td>
<td>4.098</td>
<td>4.9</td>
</tr>
<tr>
<td>MgO</td>
<td>4.310</td>
<td>0.0053</td>
</tr>
<tr>
<td>Ti$_2$O$_3$</td>
<td>5.225</td>
<td>16.2</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>4.825</td>
<td>17.48</td>
</tr>
</tbody>
</table>

Experimental heat T3 for concept verification

<table>
<thead>
<tr>
<th>Heat #</th>
<th>Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>IF</td>
</tr>
<tr>
<td></td>
<td>Ladle</td>
</tr>
<tr>
<td>T3</td>
<td>Ca</td>
</tr>
<tr>
<td></td>
<td>Al, Mg, Ti</td>
</tr>
</tbody>
</table>

[Image of microstructure]
Precipitations - Heat T3

MgAl$_2$O$_4$ with TiN

Diameter Range
- 0.5 - 2 µm
- 2 - 5 µm
- 5 - 40 µm
- All 39%

Component Concentrations:
- Mg: 65.97222%
- Al: 62.98701%
- Ti: 63.81579%
- N: 62.33766%
- O: 58.33333%

Alloy Composition:
- Mg: 61.80556%
- Al: 64.17911%
- Ti: 58.57143%
- N: 51.89874%
- O: 56.94444%
- Mn: 53.33333%

Diameter Range:
- 0.5 - 2 µm: 6.84%
- 2 - 5 µm: 26.36%
- 5 - 40 µm: 5.8%
- All: 39%
TEM of complex precipitate

1, 3, 5 Spinel

9, 10 Ca-Si modified spinel

FeFCC matrix

2, 6, 7 TiN

4 MnS
Spinel – TiN boundary

Bulk:
- Constant zone axes angles
- The same [011] planes in beam direction
- Very close orientation

Boundary:
- Flat topology
- Almost epitaxial
High resolution Matrix-TiN-Spinel line scan

Step size – 5 nm, spot size ≈ 10 nm
Comparison of achieved grain refinement

<table>
<thead>
<tr>
<th></th>
<th>R</th>
<th>Equiaxed grains, ( \mu \text{m} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>G2 (base)</td>
<td>0.57</td>
<td>2.4</td>
</tr>
<tr>
<td>T2 (N+Deox+Ti)</td>
<td>0.78</td>
<td>2.2</td>
</tr>
<tr>
<td>T3 (Al+Mg+Ti)</td>
<td>0.82</td>
<td>0.5</td>
</tr>
</tbody>
</table>
Findings

- Optimized sequence of de-oxidation and refinement provides increase grain refinement efficiency.
- Co-precipitation of targeted nucleation sites by inclusion engineering is the effective way to enhance heterogeneous nucleation.
- The well-refined as-cast structure was achieved in experimental heavy section castings.
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