Grain size refinement and properties of low nickel austenitic stainless steel

Raekoon hienontuminen ja ominaisuudet matalanikkelisessä ruostumattomissa teräksissä

Materials engineering laboratory, Anna Kisko, 29.05.2012
Presentation overview

• Introduction
• Experimental
• Results
  – Microstructure
  – Grain size
  – Mechanical properties
• Summary
• Acknowledgement
Differences between 200 and 300 series

<table>
<thead>
<tr>
<th>200-series</th>
<th>300-series</th>
</tr>
</thead>
<tbody>
<tr>
<td>+ Cheap price</td>
<td>+ Corrosion properties</td>
</tr>
<tr>
<td>+ Higher work hardening rate</td>
<td>+ Surface quality</td>
</tr>
<tr>
<td>• due lower Ni and higher Mn contents</td>
<td></td>
</tr>
<tr>
<td>+ (Yield) strength</td>
<td></td>
</tr>
</tbody>
</table>

Nickel monthly price US dollars per metric ton

21.05.2012
Ni price: 16.775 USD/kg
Applications

• Structural applications
• Indoor and outdoor applications
• Car industry
• Pipes
Alloying philosophy of austenitic stainless steels and 200-series

<table>
<thead>
<tr>
<th>AISI</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>N</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>301</td>
<td>0.093</td>
<td>1.12</td>
<td>1.22</td>
<td>16.7</td>
<td>6.4</td>
<td>0.67</td>
<td>0.074</td>
<td>0.25</td>
</tr>
<tr>
<td>204Cu</td>
<td>0.079</td>
<td>0.40</td>
<td>9.00</td>
<td>15.2</td>
<td>1.1</td>
<td>0.03</td>
<td>0.115</td>
<td>1.68</td>
</tr>
</tbody>
</table>

Instead of Cr and Ni, 200-series steels are alloyed with Cr, Mn and N, and sometimes with Cu

- **Cr**: Oxide layer
- **Ni**: Stabilize austenite
- **C**: Increases strength
- **Mo**: Acid and corrosion resistance
- **Si**: Increases high temperature oxidation resistant
- **Mn**: Stabilizes austenite, increases WHR
- **N**: Stabilize austenite, increases pitting corrosion resistant and strength
- **Cu**: Stabilizes austenite, improves corrosion resistant, improves drawability and elongation, increases SFE
  - With Cu-additions, N additions can be reduced => softer grades
Introduction to the study

• Austenitic stainless steels have very good ductility and formability but their yield strength is quite low.

• There are various methods to improve the strength, e.g. solid solute strengthening, work hardening and grain refinement.

• Many austenitic steel grades are metastable at room temperature, during cold working formation of strain-induced martensite occurs readily increasing their strength.

• In a subsequent proper heat treatment martensite reverts back to austenite.

• As a result of this grain size refinement, excellent combinations of yield strength and elongation have been achieved to Types 304L, 316L, 301 and 301LN.

• This investigation describes
  – the reversion in two metastable austenitic Cr-Mn stainless steels, namely 204Cu and 201.
  – possible grain refinement achievable by reversion annealing as well as its effects on microstructure and mechanical properties.
Materials and Procedures

<table>
<thead>
<tr>
<th>AISI</th>
<th>Thickness [mm]</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>204Cu</td>
<td>1.00</td>
<td>0.079</td>
<td>0.40</td>
<td>9.00</td>
<td>15.2</td>
<td>1.1</td>
<td>0.03</td>
<td>1.68</td>
<td>0.115</td>
</tr>
<tr>
<td>201</td>
<td>1.50</td>
<td>0.052</td>
<td>0.34</td>
<td>7.05</td>
<td>17.2</td>
<td>3.6</td>
<td>0.18</td>
<td>0.25</td>
<td>0.245</td>
</tr>
<tr>
<td>301</td>
<td>1.27</td>
<td>0.093</td>
<td>1.12</td>
<td>1.22</td>
<td>16.7</td>
<td>6.4</td>
<td>0.67</td>
<td>0.25</td>
<td>0.074</td>
</tr>
<tr>
<td>301LN</td>
<td>1.50</td>
<td>0.030</td>
<td>0.50</td>
<td>1.23</td>
<td>17.4</td>
<td>6.6</td>
<td>0.19</td>
<td>0.17</td>
<td>0.168</td>
</tr>
</tbody>
</table>

- Cold rolling to 60% thickness reduction
- The cold rolled samples were annealed on a Gleeble:
  - Heating rates 1-1000°C/s
  - Annealing temperatures 700°C, 800°C, 900°C, 950°C and 1000°C
  - Soaking times 1s, 10s, 100s, 200s and 1000s
  - Cooling rate 200°C/s down to 400°C
Research Methods

• **Microstructural examination**
  – Ferritescope (Helmut Fisher FMP 30)
  – Optical microscope (Nikon Eclipse MA 100)
  – FE-SEM (Zeiss Ultra Plus)
  – SEM-EBSD (Oxford Instrument HKL, Channel5 software)
  – FE-TEM (LEO 912)

• **Mechanical properties**
  – Microhardness (CSM Instruments)
  – Tensile test (Zwick/Z100)
Cold deformed stage

<table>
<thead>
<tr>
<th>Steel</th>
<th>$\alpha^-\text{-martensite content}$ [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>204Cu</td>
<td>33</td>
</tr>
<tr>
<td>201</td>
<td>23</td>
</tr>
</tbody>
</table>

- Lath-type martensite
- Retained austenite

204Cu 60% CR

$B_\alpha = [\bar{1}11]$  
$B_\gamma = [011]$
Dilatometry for 204Cu

Relative change in length as a function of temperature for various heating rates

The derivate as a function of temperature for various heating rates.
As and Af temperatures for 204Cu

Heating rate effects to the reversion mechanism
### $\alpha'$-martensite contents after reversion treatments

<table>
<thead>
<tr>
<th>Steel - Ann. temp. [°C]</th>
<th>Annealing time [s]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
</tr>
<tr>
<td>204Cu - 700</td>
<td>14</td>
</tr>
<tr>
<td>204Cu - 800</td>
<td>0</td>
</tr>
<tr>
<td>201 - 700</td>
<td>13</td>
</tr>
<tr>
<td>201 - 800</td>
<td>1</td>
</tr>
</tbody>
</table>

![Graph](image)
Microstructure and microhardness for 204Cu

Materials engineering laboratory, Anna Kisko, 29.05.2012
Microstructure and microhardness for 201
Microhardness after 1s soaking

![Graph showing microhardness vs. annealing temperature for different conditions.]

Materials engineering laboratory, Anna Kisko, 29.05.2012
The impact of reversion and recrystallization on grain structure
Microstructures after reversion treatment

Nucleation of new equiaxed grains in martensite.

Subgrains and small angle grain boundaries in recovered austenite.

Defect free equiaxed austenite grains

Defect free equiaxed austenite grains
The impact of reversion and recrystallization on grain structure

Relative frequency $F(x)$ as a function of grain size without retained austenite

204Cu 900° C10s  204Cu 800° C10s  201 800° C10s
Grain sizes after reversion treatment

<table>
<thead>
<tr>
<th>Steel</th>
<th>Original grain sizes [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>204Cu</td>
<td>18</td>
</tr>
<tr>
<td>201</td>
<td>13</td>
</tr>
<tr>
<td>301LN</td>
<td>14</td>
</tr>
</tbody>
</table>
Mechanical properties

Hall-Petch relation

\[ y = \sigma_0 + k d^{1/2} \]

- \( y \) = yield strength
- \( \sigma_0 \) = friction stress
- \( k \) = constant
- \( d \) = grain diameter

Yield strength
- 304: \( k = 495 \text{ MPa } \mu \text{m}^{-0.5} \) [1]
- 301LN: \( k = 221 \text{ MPa } \mu \text{m}^{-0.5} \) [2]

Tensile strength
- 304: \( k = 295 \text{ MPa } \mu \text{m}^{-0.5} \) [1]
- 301LN: \( k = 350 \text{ MPa } \mu \text{m}^{-0.5} \) [2]

Summary

- The fraction of strain-induced martensite remains quite low, as at the 60% thickness reduction, the fractions were 33% and 23% for 204Cu and 201.
- Based on dilatometer observations, the reversion mechanism for the 204Cu with low heating rate (1-150°C/s) seems to be diffusional controlled reversion. For faster heating rate (200-1000°C/s) the reversion mechanism seems to change to martensitic shear.
- The results indicate that reversion from martensite to austenite reduces effectively the grain size for both 204Cu and 201 steels.
- The ultrafine grain size results in significantly enhanced strength with good ductility, the combination exceeding the values of conventional annealed and temper rolled austenitic stainless steels.
Acknowledgements

The financial support of The Finnish Funding Agency for Technology and Innovation (Tekes) is gratefully acknowledged as well as Finnish Metals and Engineering Competence Cluster (FIMECC Oy).

Outokumpu is also thanked for the materials and support for the research.
Questions?