Formation and development of nanoclusters in ODS steels and their influence on mechanical properties

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APT&M 2016 - Gyeongju
Motivation

- ODS steels
  - Nanoclusters
  - Ultrafine-grained
  - Radiation resistance
  - Corrosion resistance
  - Creep resistance

> How are the mechanical properties influenced by the microstructure?

- Expectations
  - High thermal stability of Y-Ti-O particles
  - Pinning of grain boundaries by particles
  - Little grain growth

Materials

- Analysis of ferritic and austenitic ODS steels
- Improved high temperature properties of austenitic (fcc) ODS steel expected

<table>
<thead>
<tr>
<th>In wt. %</th>
<th>Fe</th>
<th>Cr</th>
<th>Ni</th>
<th>Ti</th>
<th>Y$_2$O$_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>FNC 14</td>
<td>Bal14</td>
<td></td>
<td>0.4</td>
<td>0.25</td>
<td></td>
</tr>
<tr>
<td>ANC 25/20</td>
<td>Bal25</td>
<td>20</td>
<td>0.4</td>
<td>0.25</td>
<td></td>
</tr>
</tbody>
</table>

- Decreasing Y$_2$O$_3$ particle size by titanium additions
- Formation of Y-Ti-O-containing nanoclusters (~4 nm)

Processing

- Attritor or planetary ball mill
- Elemental powders + Y₂O₃
- Argon atmosphere

FAST
- Field Assisted Sintering Technique
- Pressure of 50 MPa
- 5 min at 1000 °C
- Fast heating and cooling rate (100 K/min)

Atom probe measurements

- Tips produced by SEM/FIB
- LEAP 4000X HR by Cameca®

FNC 14: mainly voltage mode
- detection rate: 0.3 %
- pulse rate: 200 kHz
- pulse fraction: 19 to 20 %
- temperature: 50 to 60 K

ANC 25/20: laser mode
- detection rate: 0.3 %
- pulse rate: 200 kHz
- pulse energy: 100 pJ
- temperature: 50 K
Reconstruction of a FNC 14 (FAST) tip

- Small Y-Ti-O nanoclusters
- Impurities from processing
- 5 nm thick, 2-dimensional longitudinal section of the reconstruction
- Proof of cluster formation in FNC 14
- Identification of clusters with maximum separation method

Comparison of ANC 25/20 and FNC 14

ANC 25/20 (FAST)
- Cluster size: 4.9 nm
- Number density: $1.2 \times 10^{23} \text{ m}^{-3}$

FNC 14
- Similar volume fraction
- Smaller clusters
- Higher number density
Composition of nanoclusters

- Removal of matrix
- Modification of mass spectrum
- Decomposition of peaks
- Fe content artificially set to 0
- Cr (and Ni) content reduced proportionally
- Average composition of all identified nanoclusters

50 nm

Composition of nanoclusters

- No significant changes in FNC 14 during annealing
- N seems to substitute O in FNC 14
- Ni seems to substitute Y in ANC 25/20

Ratio of (Y, Ti, Cr, Ni) to (O, N) is around 3:1 in both alloys
Grain size

- Determination of grain size
- Orientation mappings from EBSD measurements
- Area weighted averaging

FNC 14
ANC 25/20

- Comparable grain size of all ODS steels after consolidation (between 0.4 and 0.5 µm)
- No grain growth even after 1000 h at 1000 °C
- Nanoclusters effectively pin grain boundaries and prevent grain growth
- No thermally induced changes in microstructure up to 1000 °C!

Mechanical properties – Compression tests

- Compression tests between room temp. and 1000 °C
- Constant strain rate: $10^4$ s$^{-1}$

- Strength of FNC 14 > ANC 25/20 at all temperatures
- Influence of smaller clusters and higher amount of clusters
- Drop of strength for ANC 25/20 at higher temperature
Modeling of strength

\[ \sigma_{ys} = \sigma_0 + \sigma_{HP} + \sigma_{OW} + \sigma_{SS} \]

\[ \sigma_{HP} = \frac{k_{HP}}{\sqrt{d}} \]

Orowan strengthening

- Calculation of strengthening contributions in the low temperature range

- Calculated strength can depict results from compression tests very well.

Summary

- Production and analysis of ferritic and austenitic ODS steels
- APT for characterization of nanoclusters

- Large number of nanoclusters (< 5 nm) in both steels
- Ratio of Y, Ti, Cr, Ni to O and N was 3:1

- Nanoclusters and grain size (around 0.5 \( \mu \)m) stable during long-term annealing at 1000 °C

- Strength of FNC 14 higher at all temperatures
- Drop of strength at higher temperature for ANC 25/20
- Calculation of strengthening contributions possible
Thank you very much for your attention!