Investigation on different oxides as candidates for nano-sized ODS particles in reduced-activation ferritic (RAF) steels

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Contents

- Introduction
- Production of ODS alloys
- Mechanical tests
- EBSD measurements
- Outlook
Introduction

What are ODS alloys?

Oxide Disperions-Strengthened alloys

Nano-sized oxide-particles 10-20nm

+ good corrosion-resistance
+ excellent high-temperature properties
++ improved creep-strength
- material tends to be brittle
- high production costs

(TEM High-Angle-Annular-Dark-Field Image)
Production of ODS alloys

Fe13Cr1W0.3Ti

Y2O3

mechanical alloying
Production of ODS alloys

Fe13Cr1W0.3Ti  Y2O3  

mechanical alloying  

compressing into capsules
Production of ODS alloys

Fe13Cr1W0.3Ti

Y2O3

mechanical alloying

compressing into capsules

100 Mpa

1100°C

hot consolidating (HIP)
Production of ODS alloys

Fe13Cr1W0.3Ti  Y2O3

100 Mpa  1100°C

compressing into capsules

mechanical alloying

hot rolling / heat treatment

hot consolidating (HIP)
Production of ODS alloys

Compacting of the powders containing the different oxides:

- HIP at 1100°C / 100 MPa for 2 hours
- Hot-rolling at 1100°C
- Reduction from 45 mm diameter to 6 mm thickness
- 5 passes needed for final shape, with reheating after each pass

TU Clausthal
### Production of ODS alloys

**Production-parameters:**

<table>
<thead>
<tr>
<th>No.</th>
<th>composition</th>
<th>milling-speed</th>
<th>milling-time</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fe13Cr1W0.3Ti + 0.3La$_2$O$_3$</td>
<td>1200 / 800</td>
<td>80h</td>
</tr>
<tr>
<td>2</td>
<td>Fe13Cr1W0.3Ti + 0.3Ce$_2$O$_3$</td>
<td>1200 / 800</td>
<td>80h</td>
</tr>
<tr>
<td>3</td>
<td>Fe13Cr1W0.3Ti + 0.3MgO</td>
<td>1200 / 800</td>
<td>80h</td>
</tr>
<tr>
<td>4</td>
<td>Fe13Cr1W0.3Ti + 0.3ZrO$_2$</td>
<td>1200 / 800</td>
<td>80h</td>
</tr>
<tr>
<td>5</td>
<td>Fe13Cr1W0.3Ti + 0.3Fe$_2$Y</td>
<td>1200 / 800</td>
<td>80h</td>
</tr>
<tr>
<td>Ref.</td>
<td>Fe13Cr1W0.3Ti + 0.3Y$_2$O$_3$</td>
<td>1200 / 800</td>
<td>80h</td>
</tr>
</tbody>
</table>

- milling in argon-atmosphere
- ball to powder ration 10:1 (2000g : 200g)
- complete produktion in argon (glove-box)
Characterization (FIB/TEM)

Fe$_{13}$Cr$_1$W$_{0.3}$Ti + Y$_2$O$_3$

Fe$_{13}$Cr$_1$W$_{0.3}$Ti + La$_2$O$_3$ rolled

*GRC - Physical Metallurgy 2011, August 3., Boston, USA
clearly visible rolling texture

- grain size approx. 400 to 800 nm width, but micrometer-sized length
Characterization (oxides)

- formation of ODS particles with La$_2$O$_3$
- fine distribution of the oxides (inside grains$^1$ and on GB$^2$)
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- formation of ODS particles with La$_2$O$_3$
- fine distribution of the oxides (inside grains$^1$ and on GB$^2$)
EBSD Measurements

Fe13Cr1W0.3Ti + Y₂O₃

Color Coded Map Type: Inverse Pole Figure [001]
Iron - Alpha
EBSD Measurements

Results from EBSD Measurements

- A bimodal grain size distribution is visible
- Grains with <110> parallel to the rolling direction
- Predominance of \{001\} <110> rotated cube (α-fiber) (typical texture in bcc metals)
EBSD Measurements

ODS-13Cr-1W-0.3Y₂O₃-KIT
hot-rolled @ 1100°C

Area fraction vs. Grain size (μm)
EBSD Measurements

KAM – Kernel Average Misorientation Map

Fine recrystallized grains are surrounded by coarser elongated ones.
Mechanical tests (charpy-impact test)

All specimens in LT- orientation
Mechanical tests (charpy-impact test)

Y$_2$O$_3$

MgO

RT
Mechanical tests (charpy-impact test)

Y$_2$O$_3$ MgO

RT 300°C
Mechanical tests (tensile tests – yield strength)

- Y-containing alloys show the best results
- Most alloys perform in a similar way
Comparison of 12Cr, 13Cr and 14Cr ODS

- Performance in tensile tests is comparable to alloys produced at other facilities.
Conclusion and Outlook

Alternative oxides for ODS steels

- Formation of nano-oxides is possible with alternative oxides
- Tensile properties of different oxides are comparable to yttrium-alloys
- Improved charpy-impact properties for Ce₂O₃ and MgO

Outlook

- Detailed TEM Characterization of nano-oxides is still in progress
- EBSD mappings of selected oxides (other than Y₂O₃)
Thank you for your attention!
Mechanical tests (tensile tests – total elongation)

![Graph showing the total elongation of various materials at different test temperatures.](chart.png)
Mechanical tests (charpy-impact test)

Impact energy (J) vs. temperature (°C)

- Ce2O3
- La2O3
- base-1
- Y2O3 rolled
- Y2O3-HIP
- FeY

All specimens in LT- orientation