Control of Defects and Microstructure in ODS Alloys

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Control of Defects and Microstructure in ODS Alloys

- Recrystallisation of PM2000
  - torsionally deformed tube
  - extruded bar
  - sheet samples subjected to bending
  - sheet samples deformed torsionally
- Torsional forming trials on ASTM 446 ferritic steel tube
- Evolution of Oxide Dispersions during Secondary Recrystallisation
- Replacement ODM751
- Selective Laser Melting (SLM) of PM2000 alloy powder
Recrystallisation of torsionally deformed PM2000 tube

PM2000
- fine grained
- 2 metre length
- 25.8 mm O.D.
- 2.9 mm wall

Torsion trial parameters
- 1.25 rpm torsion
- traverse rate, 0.01ms⁻¹
- lead length 200mm
- 720°C
- 20° twist

End-section of 2 meter length of PM2000 tube, twisted at Forecrêu at 750°C.
Recrystallisation of torsionally deformed PM2000 tube

PM2000 tube ‘end stock’ + SR 1380°C/1h. Transverse section, SEM.

PM2000 tube torsionally deformed ~20% + SR 1380°C/1h. Inner wall. Transverse section, SEM.
a) channelling contrast SEM
b) Boundary map.

PM2000 tube torsionally deformed ~20% + SR 1380°C/1h. Transverse section, SEM.
Recrystallisation of torsionally deformed PM2000 tube
Recrystallisation of torsionally deformed PM2000 tube

a) torsionally deformed ~20%
Plan section. TEM.

d) Deformed + SR 1380°C/1h
Transverse section. SEM.

c) Deformed + SR 1380°C/1h.
Plan section. TEM.

e) Deformed + SR 1380°C/1h
Plan section. SEM.
Recrystallisation of extruded PM2000 bar

PM2000 bar (can/HIP/hot extrude (1000°C). Fine-grained as-extruded state. SEM longitudinal sections

Predominance of labg's near bar surface
Recrystallisation of extruded PM2000 bar

As–extruded + 900°C/1h. SEM transverse section

As–extruded + 1100°C/1h. SEM transverse section

As–extruded + 1200°C/1h. a) SEM transverse section
   b) SEM longitudinal section

As–extruded + 1100°C/1h. SEM transverse section
Recrystallisation of extruded PM2000 bar

As–extruded + 1380°C/1h. Transverse section. SEM.
PM2000 sheet subject to bending/twisting +SR

Rolling

Bent samples

Twisted samples

120° → 90° → 45°

360° → 270° → 180° → 90°

Samples deformed (bend/twist then SR (1380°C /1h).
PM2000 sheet subject to bending/twisting +SR

120°

90°

45°

Fine grained samples deformed in bending + SR (1380°C/1h). MA957/□-section from plate/ 30° final bend angle + 1300°C/1h (after Capdevila and Bhadeshia, 2000)
PM2000 sheet subject to bending/twisting +SR

PM2000 ‘bar’ sample cut from sheet, twisted 360° + SR (1380°C /1h).
Torsional forming trials: ASTM 446 steel tube

- Torsion trials: Forecreu, France → Kennametal Inc, Evans GA (twist drill manuf.)

- Kennametal trials unfunded; tube supplied free issue ex-UoL

- PM2000 replaced by Sandvik 4C54 (ASTM 446) heat resisting seamless Cr ferritic steel tube

Sandvik 4C54 chemical composition wt.%

<table>
<thead>
<tr>
<th>C max</th>
<th>Si</th>
<th>Mn</th>
<th>P max</th>
<th>S max</th>
<th>Cr</th>
<th>N</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2</td>
<td>0.5</td>
<td>0.8</td>
<td>0.03</td>
<td>0.015</td>
<td>26.5</td>
<td>0.2</td>
<td>Balance</td>
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</tbody>
</table>

- tube 21.3mm diameter, 2.7mm wall; arrangement supported by internal steel mandrel
Torsional forming trials: ASTM 446 steel tube

• Sandvik 4C54 (ASTM 446) tube + internal steel mandrel (15.5mm diam.)
• Initial parameters:
  torsion at $\sim 1000^\circ$C/ 0.55 r.p.m/ mm.s$^{-1}$ hot zone
• outcomes:
  60$^\circ$ torsion achieved/wall thinning+thickening/
tube dynamic recrystallisation

Longitudinal section through a ‘ridge’. SEM channelling contrast. Original tube wall thickness 2.7mm.

Transverse section showing 60$^\circ$ inclination of stringers in microstructure.
Torsional forming trials: ASTM 446 steel tube

Trial 2:
- Fixed mandrel
- 950°C
- 0.55 r.p.m
- 7mm traverse speed
- 60°helix
Torsional forming trials: ASTM 446 steel tube

**Outer Stainless Steel Tube:**
- OD = 21.3mm
- ID = 16.0mm

**Internal Mandrel 1:**
- Diameter = 15.0mm

**Internal Mandrel 2:**
- Diameter = 15.0mm

**Fillet weld**

**Trial 5:**
- Split mandrel
- < 900°C
- 0.3 r.p.m
- 7.2 mm traverse speed
- ~30° helix

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[Image of a steel tube with dimensions and trials details]
Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation

- PM2000 sheet in fine-grained (KKL4) condition + friction stir weld (TWI)
- FS joint + SR 1380°C/1h
- Carbon extraction replicas from parent sheet and joint
Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation

Cubic \( \text{Y}_2\text{O}_3 \)

Extraction replica from friction stir weld in PM2000 sheet + SR 1380\( ^\circ \text{C} \)/1h
Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation

$Y_3Al_5O_{12}$ Yttrium-Aluminium-Garnet (YAG)

Extraction replica from friction stir weld in PM2000 sheet + SR 1380°C/1h
Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation

YAIO₃ Yttrium-Aluminium-Perovskite (YAP)

Extraction replica from friction stir weld in PM2000 sheet + SR 1380°C/1h
Evolution of PM2000 Oxide Dispersions during Secondary Recrystallisation

Friction Stir Weld

As-received Alloys

Recrystallization Treatment (1380°C, 1hr)

Y₂O₃ or YAG

Y₂O₃ or YAG

Y₂O₃ - 34%
YAG - 50%
YAP - 16%

Y₂O₃ - 16%
YAG - 66%
YAP - 16%

Y₂O₃ - 10%
YAG - 5%
YAP - 85%

Y₂O₃ - 20%
YAG - 70%
YAP - 10%
Replacement ODM751

ODM 751 powder – Dour Metal SA, Belgium

ODM751 MA powder particles, Dour Metal SA.
Optical micrograph, etched transverse section.

ODM751 MA powder. SEM image un-sieved powder
Replacement ODM751

- Powder supplied by Dour Metal, s.r.o.
- Powder sample dispersed in H₂O/surfactant.
- Laser diffraction measurements (5/sample).
- Results averaged over five tests.
- Powder size range: ~10-110µm.
- Mean size: ~35 µm.

Polishing relief: un-etched optical section

Etched sample: optical section
• Energy dispersive X-ray analysis (EDX) of trial samples of replacement ODM751 MA powders.

• Blue spectrum: region exhibiting MA structures contains elements expected in ODM 751 (nominal composition below).

• Red spectrum: region remaining un-etched. Significant levels of Cr and some Fe.

<table>
<thead>
<tr>
<th>Element</th>
<th>Cr</th>
<th>Al</th>
<th>Mo</th>
<th>Ti</th>
<th>Y₂O₃</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt%</td>
<td>16</td>
<td>4.5</td>
<td>1.5</td>
<td>0.6</td>
<td>0.5</td>
<td>Balance</td>
</tr>
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ODM 751 Nominal Composition (wt%)
Replacement ODM751

- EDX measurements from trial sample of replacement ODM751 MA powder (as shown in SEM image of polished/etched sample).
- Tabulated values normalised without the carbon contribution.

<table>
<thead>
<tr>
<th>Element</th>
<th>Sample</th>
<th>ODM751 (nominal)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>16.4 ± 1.6</td>
<td>16</td>
</tr>
<tr>
<td>Al</td>
<td>4.8 ± 0.8</td>
<td>4.5</td>
</tr>
<tr>
<td>Mo</td>
<td>1.6 ± 0.1</td>
<td>1.5</td>
</tr>
<tr>
<td>Ti</td>
<td>0.6 ± 0.1</td>
<td>0.6</td>
</tr>
<tr>
<td>O</td>
<td>3.4 ± 0.9</td>
<td></td>
</tr>
<tr>
<td>Y$_2$O$_3$</td>
<td></td>
<td>0.5</td>
</tr>
<tr>
<td>Fe</td>
<td>73.2 ± 1.4</td>
<td>bal</td>
</tr>
</tbody>
</table>

Error bounds are one standard deviation.
Selective Laser Melting (SLM) of PM2000 alloy powder

PM 2000 Thermocouple Sleeves
Application: e.g. in Gas Turbines; temperature measurement close to combustion chamber

PM 2000 Burner Nozzles
Fuel injection in diesel engines (test parts)

Micro-heat exchanger by SLM (20mm cube)
Selective Laser Melting (SLM) of PM2000 alloy powder

PM2000 Particle size analysis

- The PM2000 MA powder exhibited a bi-modal particle size distribution.
- Powders were sieved to remove large particles that would impede flow/disturb the powder bed.
Selective Laser Melting (SLM) of PM2000 alloy powder

Selective Laser Melting (SLM) of PM2000 alloy powder

- Walls ≤ 200μm thickness
- Columnar grain structure
- Some porosity; sensitive to build parameters

50W, 0.1m/s wall

PM2000 SLM wall, side view, 50w, 0.1m/s. Electron channelling contrast images.
Selective Laser Melting (SLM) of PM2000 alloy powder

Wall integrity as a function of laser power (W) and scan speed (m.s\(^{-1}\))
Selective Laser Melting (SLM) of PM2000 alloy powder

PM2000 tube (SR 1380°C/1h)
Nominal oxide particle diameter = 30.8 ± 15.1nm

Evidence of:
Retention of oxide particles in PM2000 after SLM fabrication process.
An increase in mean particle size following 50W/0.2m.s⁻¹ SLM.

PM2000 SLM Wall, 50W, 0.2m.s⁻¹
Nominal oxide particle diameter = 45.8 ± 18.6nm
Summary

Recrystallisation of PM2000 tube, bar and sheet

(i) SR (1380°C/1h) of torsionally deformed tube results in helical grain structures. Grain size varies:
  • across the wall
  • around the circumference
  • along the tube axis.
Isolated regions of PR material remain.

(ii) Grain size across SR product forms (tube, extruded bar and sheet samples subject to bending and torsion):
  • Can be inhomogeneous
  • Varies surface to bulk
  • Is influenced by cumulative processing history

Torsional forming trial

(i) Torsion trials on ASTM 446 supported by Kennametal Inc
  • 60° twist achieved
  • Extensive plasticity/dynamic recrystallisation
  • Tube ‘ribbing’ with local wall thickening/thinning
  • Macroscopic straightness with fixed/split-mandrel
Summary

Evolution of oxide dispersions during secondary recrystallisation of PM2000

(i) Yttria/YAG/YAP present in sheet and friction stir weld before and after SR
(ii) YAG replaced by YAP as dominant oxide type during SR

Replacement ODM751

(i) Dour Metal, s.r.o., Slovakia producing initial batches of replacement ODM751
(ii) Composition produced is close to legacy ODM751
(iii) Incomplete MA of residual amounts of Cr-rich precursor

SLM of PM2000 alloy powders

(i) Sieved PM2000 powders can be fabricated using SLM techniques
(ii) ‘fully dense walls’ can be built using a combination of laser power (W) and scan speed
(iii) Initial trials suggest ODS particles can be retained, though with some coarsening