Friction Stir Welding of ODS Steels for Future Generation Nuclear Reactors

A thesis submitted to the University of Manchester for the degree of Doctor of Philosophy in the Faculty of Science & Engineering

2017

Huw Dawson

School of Materials
Contents

Lists of Acronyms .................................................................................................................. 6
List of Figures ......................................................................................................................... 8
List of Tables .......................................................................................................................... 14
Abstract .................................................................................................................................. 16
Declaration ............................................................................................................................... 17
Copyright Statement ............................................................................................................... 18
Acknowledgements ................................................................................................................ 19

1. Introduction ......................................................................................................................... 20

1.1. Scope of thesis .................................................................................................................. 20

1.2. Nuclear power .................................................................................................................. 20
  1.2.1. Energy generation ....................................................................................................... 20
  1.2.2. Radiation damage to materials .................................................................................. 23
  1.2.3. Gen IV fission reactors ............................................................................................... 25
  1.2.4. Fusion reactors ........................................................................................................... 26
  1.2.5. Operating conditions ................................................................................................. 26
  1.2.6. Future reactor candidate materials ............................................................................ 28
  1.2.7. Reduced-activation steels ......................................................................................... 29

1.3. Oxide Dispersion-Strengthened (ODS) steels ................................................................. 30
  1.3.1. Introduction to ODS steels ....................................................................................... 30
  1.3.2. Production ................................................................................................................ 31
  1.3.3. Microstructure .......................................................................................................... 33
  1.3.4. ODS Strengthening mechanisms ............................................................................... 37
  1.3.5. Radiation damage resistance .................................................................................... 40

1.4. Friction stir welding ....................................................................................................... 41
  1.4.1. Introduction .............................................................................................................. 41
1.4.2. Friction stir welding (FSW) process ..................................................................... 42
1.4.3. FSW of steel ........................................................................................................ 44
1.4.4. Recrystallization due to FSW ............................................................................ 45
1.5. Abnormal grain growth ......................................................................................... 47
1.5.1 Types of grain growth ......................................................................................... 48
1.5.2 Factors affecting grain growth ............................................................................ 49
1.5.2 Factors leading to AGG ....................................................................................... 49
1.6. Residual stresses .................................................................................................... 51
1.6.1. The significance of residual stresses ................................................................. 51
1.6.2. Origins of residual stress .................................................................................... 52
1.6.3. Measuring residual stresses by neutron diffraction ........................................... 52
1.6.4. Advantages of using neutrons .......................................................................... 54
1.6.5. Residual stresses in friction stir welds ............................................................... 55
2. Impact of friction stir welding on the microstructure of ODS steel ....................... 73
2.1. Introduction ............................................................................................................ 75
2.2. Experimental .......................................................................................................... 76
  2.2.1. Base Material .................................................................................................. 76
  2.2.2. Friction Stir Welding ....................................................................................... 77
  2.2.3. Structural characterization .............................................................................. 77
2.3. Results .................................................................................................................... 78
  2.3.1. Base material .................................................................................................. 78
  2.3.2. Friction stir welding ....................................................................................... 80
  2.3.3. Grain size distribution .................................................................................... 81
  2.3.4. Nano-particle size distribution ....................................................................... 82
2.4. Discussion ............................................................................................................... 84
2.5. Conclusions ............................................................................................................ 89
3. Mechanical properties and fracture behaviour of ODS steel friction stir welds at variable temperatures ......................................................................................... 93
5.2.2. Welding and post-weld heat treatment ...................................................... 136
5.2.3. Characterization of weld microstructures .................................................. 137
5.3. Results.............................................................................................................. 138
  5.3.1. As-welded structure characterization ....................................................... 138
  5.3.2. Post-weld heat treated structure ............................................................... 144
5.4. Discussion........................................................................................................ 145
  5.4.1. As-welded structure .................................................................................. 145
  5.4.2. Abnormal grain growth behaviour ......................................................... 146
5.4. Conclusions..................................................................................................... 149
6. Final conclusions and future work ................................................................. 154
  6.1. Conclusions.................................................................................................. 154
  6.2. Future work................................................................................................... 155
  6.3. Publication of research ............................................................................... 158
  6.4. Contributions of authors ............................................................................ 158
7. Appendix ........................................................................................................... 160

Final word count: 36748
## Lists of Acronyms

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Meaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>Austenitic S.S.</td>
<td>Austenitic stainless steel</td>
</tr>
<tr>
<td>bcc</td>
<td>Body centred cubic</td>
</tr>
<tr>
<td>BNC</td>
<td>Budapest Neutron Centre</td>
</tr>
<tr>
<td>CDRX</td>
<td>Continuous dynamic recrystallization</td>
</tr>
<tr>
<td>dpa</td>
<td>Displacements per atom</td>
</tr>
<tr>
<td>DDRX</td>
<td>Discontinuous dynamic recrystallization</td>
</tr>
<tr>
<td>DRX</td>
<td>Dynamic recrystallization</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron Backscatter Diffraction</td>
</tr>
<tr>
<td>EDM</td>
<td>Electrical Discharge Machining</td>
</tr>
<tr>
<td>F-M</td>
<td>Ferritic-martensitic</td>
</tr>
<tr>
<td>fcc</td>
<td>Face centred cubic</td>
</tr>
<tr>
<td>FSW</td>
<td>Friction stir welding</td>
</tr>
<tr>
<td>GAR</td>
<td>Grain aspect ratio</td>
</tr>
<tr>
<td>GDRX</td>
<td>Geometric dynamic recrystallization</td>
</tr>
<tr>
<td>Gen II</td>
<td>Generation II</td>
</tr>
<tr>
<td>Gen IV</td>
<td>Generation IV</td>
</tr>
<tr>
<td>GFR</td>
<td>Gas-cooled fast reactor</td>
</tr>
<tr>
<td>HAZ</td>
<td>Heat affected zone</td>
</tr>
<tr>
<td>HI</td>
<td>Heat input</td>
</tr>
<tr>
<td>HIP</td>
<td>Hot isostatic pressing</td>
</tr>
<tr>
<td>IFC</td>
<td>Inertial fusion confinement</td>
</tr>
<tr>
<td>ILL</td>
<td>Institut Laue-Langevin</td>
</tr>
<tr>
<td>IPF</td>
<td>Inverse Pole Figure</td>
</tr>
<tr>
<td>LD</td>
<td>Longitudinal direction</td>
</tr>
<tr>
<td>LLB</td>
<td>Laboratoire Léon Brillouin</td>
</tr>
<tr>
<td>LN</td>
<td>Longitudinal-Normal</td>
</tr>
<tr>
<td>MA</td>
<td>Mechanical alloying</td>
</tr>
<tr>
<td>MFC</td>
<td>Magnetic fusion confinement</td>
</tr>
<tr>
<td>MSR</td>
<td>Molten salt reactor</td>
</tr>
<tr>
<td>ND</td>
<td>Normal direction</td>
</tr>
<tr>
<td>ODS</td>
<td>Oxide dispersion-strengthened</td>
</tr>
<tr>
<td>OPS</td>
<td>Oxide polishing-suspension</td>
</tr>
<tr>
<td>Pb-LFR</td>
<td>Lead-cooled fast reactor</td>
</tr>
<tr>
<td>PCBN</td>
<td>Polycrystalline cubic boron nitride</td>
</tr>
<tr>
<td>PhD</td>
<td>Doctor of Philosophy</td>
</tr>
<tr>
<td>PKA</td>
<td>Primary knock-on atom</td>
</tr>
<tr>
<td>RAFM</td>
<td>Reduced-activation ferritic/martensitic</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Definition</td>
</tr>
<tr>
<td>--------------</td>
<td>------------</td>
</tr>
<tr>
<td>SANS</td>
<td>Small-angle neutron scattering</td>
</tr>
<tr>
<td>SCWR</td>
<td>Supercritical-water-cooled reactor</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td>SFE</td>
<td>Stacking fault energy</td>
</tr>
<tr>
<td>SFR</td>
<td>Sodium-cooled fast reactor</td>
</tr>
<tr>
<td>STEM</td>
<td>Scanning transmission electron microscope</td>
</tr>
<tr>
<td>SZ</td>
<td>Stir zone</td>
</tr>
<tr>
<td>TD</td>
<td>Transverse direction</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscope</td>
</tr>
<tr>
<td>TL</td>
<td>Transverse-Longitudinal</td>
</tr>
<tr>
<td>$T_m$</td>
<td>Melting temperature</td>
</tr>
<tr>
<td>TMAZ</td>
<td>Thermo-mechanical affected zone</td>
</tr>
<tr>
<td>TWI</td>
<td>The Welding Institute</td>
</tr>
<tr>
<td>UTS</td>
<td>Ultimate tensile strength</td>
</tr>
<tr>
<td>UVeFSW</td>
<td>Ultrasonic vibration enhanced friction stir welding</td>
</tr>
<tr>
<td>VHTR</td>
<td>Very-high-temperature reactor</td>
</tr>
<tr>
<td>XRD</td>
<td>X-ray diffraction</td>
</tr>
<tr>
<td>YAG</td>
<td>Yttrium aluminium garnet</td>
</tr>
<tr>
<td>YAH</td>
<td>Yttrium aluminium hexagonal</td>
</tr>
<tr>
<td>YAM</td>
<td>Yttrium aluminium monoclinic</td>
</tr>
<tr>
<td>YAP</td>
<td>Yttrium aluminium perovskite</td>
</tr>
<tr>
<td>YAP'</td>
<td>Yttrium aluminium pseudo-perovskite</td>
</tr>
<tr>
<td>YAT</td>
<td>Yttrium aluminium tetragonal</td>
</tr>
<tr>
<td>YS</td>
<td>Yield strength</td>
</tr>
</tbody>
</table>
### List of Figures

<table>
<thead>
<tr>
<th>Chapter</th>
<th>Page</th>
<th>Figure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21</td>
<td>Fig. 1. Binding energy per nucleon versus the mass number, $A$ [2].</td>
</tr>
<tr>
<td>1</td>
<td>22</td>
<td>Fig. 2. Examples of fission and fusion reactions [5].</td>
</tr>
<tr>
<td>1</td>
<td>23</td>
<td>Fig. 3. Cross sections of fusion reactions against energy [6].</td>
</tr>
<tr>
<td>1</td>
<td>25</td>
<td>Fig. 4. Possible damage to a lattice from a displacement cascade [10].</td>
</tr>
<tr>
<td>1</td>
<td>28</td>
<td>Fig. 5. Expected total radiation dose and operating temperatures of Gen IV and fusion reactors compared to Gen II fission reactors [22].</td>
</tr>
<tr>
<td>1</td>
<td>29</td>
<td>Fig. 6. Irradiation levels of common steel alloying elements following exposure to blanket front end irradiation doses. The black line represents the ITER administrative safe irradiation level for hands-on maintenance [26].</td>
</tr>
<tr>
<td>1</td>
<td>32</td>
<td>Fig. 7. Diagram of the first steps of the MA956 production process [32].</td>
</tr>
<tr>
<td>1</td>
<td>33</td>
<td>Fig. 8. Transmission electron micrograph showing the dispersoids in MA956 aligned parallel to the extrusion direction [29].</td>
</tr>
<tr>
<td>1</td>
<td>37</td>
<td>Fig. 9. Binary phase diagram for Fe-Cr, calculated using Thermo-Calc [62].</td>
</tr>
<tr>
<td>1</td>
<td>38</td>
<td>Fig. 10. Left, diagram of a lattice with a substitutional solute atom, in red. Right, lattice with interstitial solute atoms, in black [65].</td>
</tr>
<tr>
<td>1</td>
<td>38</td>
<td>Fig. 11. Diagram showing a dislocation line bowing around an unshearable particle [66].</td>
</tr>
<tr>
<td>1</td>
<td>41</td>
<td>Fig. 12. Illustration of how helium bubbles nucleate and agglomerate around nano-clusters [80].</td>
</tr>
<tr>
<td>1</td>
<td>43</td>
<td>Fig. 13. Diagram showing the microstructural features of a FSW joint [100].</td>
</tr>
<tr>
<td>1</td>
<td>45</td>
<td>Fig. 14. Schematic diagram representing the microstructural evolution occurring during DDRX. The dotted lines represent the prior grain boundaries [111].</td>
</tr>
<tr>
<td>1</td>
<td>46</td>
<td>Fig. 15. Schematic diagram representing recrystallization by progressive lattice rotation [111].</td>
</tr>
<tr>
<td>1</td>
<td>47</td>
<td>Fig. 16. Schematic diagram of the microstructural evolution during GDRX, adapted from [119].</td>
</tr>
<tr>
<td>1</td>
<td>48</td>
<td>Fig. 17. Representative grain structure evolutions with time, along with the associated grain size distributions for top: normal grain growth and bottom: abnormal grain growth [137].</td>
</tr>
<tr>
<td>1</td>
<td>50</td>
<td>Fig. 18. Expected grain growth regimes in a material with an ideal grain assembly. Mean grain diameter against particle dispersion level [140].</td>
</tr>
<tr>
<td>1</td>
<td>51</td>
<td>Fig. 19. Grain growth regimes in a material with an ideal grain assembly. Mean grain diameter against particle dispersion level [111].</td>
</tr>
<tr>
<td>1</td>
<td>53</td>
<td>Fig. 20. Diagram illustrating the origin of Bragg’s law [147].</td>
</tr>
<tr>
<td>Fig.</td>
<td>Page</td>
<td>Description</td>
</tr>
<tr>
<td>------</td>
<td>------</td>
<td>-------------</td>
</tr>
<tr>
<td>21.</td>
<td>54</td>
<td>Schematic showing incident and diffracted beam with changing diffraction angle [148].</td>
</tr>
<tr>
<td>22.</td>
<td>56</td>
<td>Graph of residual stress distribution measured in an MA956 ODS steel plate as measured by X-ray diffraction [145].</td>
</tr>
<tr>
<td>1.</td>
<td>57</td>
<td>(a)-(b) Optical image along the LD/TD and ND/TD planes respectively; (c)-(e) BSE images taken at the representative locations of the plate thickness denoted in (b); and (f) TEM image of the as-received plate material of MA956 ODS steel.</td>
</tr>
<tr>
<td>2.</td>
<td>79</td>
<td>Optical image of the cross section of a representative bead-on-plate ODS steel weld; (b) micro-hardness measurements taken at approx. 2mm from the surface of the weld, as denoted by the dashed line in (a).</td>
</tr>
<tr>
<td>3.</td>
<td>81</td>
<td>(a) EBSD map of the SZ of Weld 4; (b) grain size distribution of the matrix of selected welds; (c) variation of the mean grain diameter with the tool rotation speed and transverse speed.</td>
</tr>
<tr>
<td>4.</td>
<td>84</td>
<td>(a) SANS data collected from an ODS steel weld sample placed under an external magnetic field of 1.5 T; (b) intensity as a function of the scattering vector for the base material and selected welds; (c) oxide particle size distributions derived from the SANS data; (d) variation of the relative volume fraction of particles with the tool rotation speed and transverse speed.</td>
</tr>
<tr>
<td>5.</td>
<td>85</td>
<td>TEM images showing examples of (a)-(b) particle agglomeration and (c) melting; (d) particle size distribution obtained from the TEM images.</td>
</tr>
<tr>
<td>6.</td>
<td>87</td>
<td>(a) Mean grain diameter of the matrix as a function of the relative volume fraction of the oxide particles. The red line corresponds to the estimated Zener pinning limiting grain size (see text); (b) experimental value of the mean grain diameter for each of the studied welds, together with the estimate from Eq. (6).</td>
</tr>
<tr>
<td>1.</td>
<td>98</td>
<td>Friction stir welds of ODS steel produced in this study. The welding parameters used for each of the welds are collected in Table 2. (c) Optical micrograph of the cross section of #2. Advancing side on the right of the cross section.</td>
</tr>
<tr>
<td>2.</td>
<td>99</td>
<td>(a)-(b) BSE images of the TMAZ of #1 and #2, respectively. c) Transmission electron micrograph of the TMAZ of #3.</td>
</tr>
<tr>
<td>3.</td>
<td>100</td>
<td>Hardness maps of the cross sections of the welds. The weld number is indicated in the bottom left corner of each map.</td>
</tr>
<tr>
<td>4.</td>
<td>101</td>
<td>Mean hardness in the TMAZ of the welds as a function of (a) the rotation and traverse speed of the welding tool, and (b) the mean grain diameter of the ferritic matrix.</td>
</tr>
<tr>
<td>5.</td>
<td>102</td>
<td>Mechanical properties of the coarse grained base material as a function of temperature. (a) Tensile strength, where YS and UTS represent yield stress and ultimate tensile stress, respectively. (b) Total and uniform elongation.</td>
</tr>
<tr>
<td>6.</td>
<td>103</td>
<td>Mechanical properties of the welds and base material tested at room temperature as a function of tool traverse speed, using a value of zero for the traverse speed in the case of the base material. (a) Tensile strength and (b) elongation.</td>
</tr>
</tbody>
</table>
Fig. 7. Mechanical properties of the welds and base material tested at 500 °C, as a function of welding parameters. (a) Tensile strength vs. traverse speed. (b) Tensile strength vs. rotation speed. (c) Elongation vs. traverse speed and (d) elongation vs. rotation speed.

Fig. 8. Scanning electron micrographs of the fracture surface of the base material (BM) and two representative welds (#2 and #3) at variable temperatures. The base material specimens shown have been chosen to be representative of the three main fracture modes.

Fig. 1. (a)-(b) Optical images of the base material along the LD/TD and ND/TD planes respectively; (110) and (100) pole figures of the (c) base material in the fine-grained centre line of the plate and (d) in the thermo-mechanical affected zone of the ODS steel welds. The scale is in multiples of random distribution. Data in (a) and (b) taken from Dawson et al. (2017).

Fig. 2. (a) Schematic diagram of the friction stir welding process, adapted from Su et al. (2013); (b) ultrasound image showing the bored thermocouple holes and diagram of thermocouple positions for Weld 1; (c)-(e) temperature profiles during FSW for the three welds; (f) torque measured during welding. Lengths are in units of mm.

Fig. 3. Main components of the SALSA diffractometer at ILL, used during the neutron diffraction experiment to obtain the residual stress maps of ODS steel welds.

Fig. 4. Optical micrographs of the top view and cross section of the three welds: (a) Weld 1, (b) Weld 2, (c) Weld 3. Advancing side is on right hand side of the cross sections.

Fig. 5. Residual stress maps of the three ODS steel welds. Stress in units of MPa.

Fig. 6. Line scan residual stress data at a depth of 1.4 mm from the top surface of the welds, in the (a) longitudinal and (b) transverse orientation; (c) peak tensile residual stress and (d) peak temperature and cooling rate as a function of traverse speed during welding. The peak values are the mean of the 5 greatest values at any 5 measurement locations in each weld.

Fig. 7. Residual stress maps of Weld 2, i.e. 95 mm/min traverse speed, at the three measured locations. Stress in units of MPa. (a) shows the measurement locations in the Transverse-Longitudinal cross section.

Fig. 1. Optical micrographs (left) and hardness maps (right) of the as-welded cross sections. The weld number is indicated in the bottom left corner of each optical micrograph.

Fig. 2. a: EBSD map of the cross section of Weld 2. The colour scale represents the crystallographic orientation relative to the welding direction. b: Grain size distribution for the three butt welds.

Fig. 3. Pole figures of the 3 butt welds with units in multiples times random. Weld number is indicated on the left. WD and TD are the Weld direction and Transverse direction, respectively.
<table>
<thead>
<tr>
<th>Page</th>
<th>Line</th>
<th>Text</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>142</td>
<td><strong>Fig. 4.</strong> Top: optical cross section image of Weld 1. <strong>Bottom:</strong> representative optical micrographs of microstructure from the regions denoted by the inserted boxes in the top optical image.</td>
</tr>
<tr>
<td>5</td>
<td>143</td>
<td><strong>Fig. 5.</strong> Microstructure at the weld boarders. <strong>a:</strong> optical micrograph of bead-on-plate weld, <strong>b</strong> and <strong>c:</strong> back-scattered electron image of Welds 1 and 3, respectively. The top of the weld is towards the top and the advancing side is to the right of all of the micrographs. The red labels in <strong>b</strong> denote <strong>i:</strong> stir zone, <strong>ii:</strong> thermo-mechanically affected zone, <strong>iii:</strong> deformed base material and <strong>iv:</strong> base material.</td>
</tr>
<tr>
<td>5</td>
<td>144</td>
<td><strong>Fig. 6.</strong> Left: Optical micrographs and <strong>right:</strong> hardness maps of the heat-treated weld cross sections. The weld number is indicated in the bottom left corner of each optical micrograph.</td>
</tr>
<tr>
<td>5</td>
<td>145</td>
<td><strong>Fig. 7.</strong> Micrographs of the weld boarders after heat treatment for 1 hour at 1380 °C. <strong>a:</strong> Back scattered electron image of Weld 2 boarder on the advancing side, showing coarse grains at the top and below the thermo-mechanical affected zone that resisted abnormal grain growth. Optical micrographs of the weld boarders: <strong>b:</strong> Weld 2 advancing side, <strong>c:</strong> Weld 3 retreating side, close to top of weld, <strong>d:</strong> Weld 3 advancing side.</td>
</tr>
<tr>
<td>5</td>
<td>147</td>
<td><strong>Fig. 8.</strong> Optical micrograph of the top part of the cross section of Weld 2.</td>
</tr>
<tr>
<td>5</td>
<td>148</td>
<td><strong>Fig. 9.</strong> Optical micrographs showing dense particle build ups close to weld boarders.</td>
</tr>
<tr>
<td>7</td>
<td>161</td>
<td><strong>Fig. 1.</strong> Image of TWI’s MTI RM-2 Precision Spindle FSW machine used for all welds. Located at TWI Technology Centre – Yorkshire. It is a gantry style FSW machine manufactured by Transformation Technologies Inc. (now MTI) of Elkhart, Indiana, USA.</td>
</tr>
<tr>
<td>7</td>
<td>162</td>
<td><strong>Fig. 2.</strong> Image of the type of ceramic tool used for all welds. The tool used was a MegaStir PCBN tool, which uses particles of boron nitride embedded in a tungsten rhenium based matrix. We used a Q70 tool which is 70% boron nitride.</td>
</tr>
<tr>
<td>7</td>
<td>162</td>
<td><strong>Fig. 3.</strong> (a) Setup of FSW and (b) FSW in progress, for butt Weld 3.</td>
</tr>
<tr>
<td>7</td>
<td>163</td>
<td><strong>Fig. 4.</strong> Measured welding parameters during FSW of butt Weld 2 as a function of distance along the weld.</td>
</tr>
<tr>
<td>7</td>
<td>163</td>
<td><strong>Fig. 5.</strong> Image of surface finish following FSW for butt Weld 2.</td>
</tr>
<tr>
<td>7</td>
<td>164</td>
<td><strong>Fig. 6.</strong> Optical micrographs of bead-on-plate Weld 2. (a)-(c) at the TMAZ/BM interface on the advancing side, (d) in the SZ.</td>
</tr>
<tr>
<td>7</td>
<td>165</td>
<td><strong>Fig. 7.</strong> Top: optical cross section image of bead-on-plate Weld 2. Below: representative optical micrographs of the microstructure from regions across the cross sections, indicated by the inserted boxes in the cross section in their respective positions.</td>
</tr>
</tbody>
</table>
**Fig. 8.** Micrographs of the weld boarders. The top of the weld is towards the top and the advancing side is to the right of all of the images. (a) BSE image of butt Weld 1 taken with an accelerating voltage of 10 kV at a 6.4 mm working distance. This image is the same image as Fig. 5, in Chapter 5, however, without the annotations. (b) BSE image of butt Weld 3 taken with an accelerating voltage of 8 kV at a 6.8 mm working distance. (c) BSE image of bead-on-plate Weld 3 taken with an accelerating voltage of 20 kV at a 10.7 mm working distance.

**Fig. 9.** Optical micrographs of the butt welds following 1380 °C heat treatment for 1 hour. (a)-(b) Weld 2 on the advancing side. (c) Weld 3 on the retreating side.

**Fig. 10.** Microstructures of the MA956 base material. (a) BSE image of the fine grained base material taken on an FEI Magellan High Resolution FEG-SEM at 2 kV accelerating voltage. (a)-(b) EBSD maps acquired using an FEI Quanta 650 scanning electron microscope at 20 kV. Maps of (a) fine grained material using a 0.6 µm step size and (b) coarse grained material in the longitudinal-normal plane using a 9 µm step size. Specimen was fully recrystallized by an additional 90 minute heat treatment at 1380 °C using a Lenton tube furnace with an argon atmosphere. Both IPF scales represent the crystallographic orientation with respect to the normal direction (out of plane).

**Fig. 11.** BSE micrographs from the bead-on-plate SZ’s. Micrographs were taken at the centre of the stir zone approximately 1.5 mm from the weld surface, using an FEI Magellan High Resolution FEG-SEM. The associated Weld #’s are inserted in the figure.

**Fig. 12.** EBSD maps of the stir zones of Welds 4 and 5 in the transverse-longitudinal plane, from between 0.5–1.5 mm depth from the top surface at the joint line i.e. the centre of the weld. Maps taken using an FEI Quanta 650 scanning electron microscope on 3 mm-diameter TEM discs were prepared by electropolishing at a temperature of -40 °C, using a Tenupol 5 Jet electropolisher and an electrolyte comprising 90 vol.% methanol and 10 vol.% perchloric acid. The IPF colour scale is with respect to the normal direction, which is the only known direction for these specimens (out of plane).

**Fig. 13.** Etched microstructure of the stir zone of bead-on-plate #5 in the Transverse-Longitudinal plane. The image was taken close to the centre of the weld line on a sample face between 0.5–1.5 mm depth from the original top surface of the weld.

**Fig. 14.** Transmission electron micrographs of the fine grained base material. Micrographs taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.

**Fig. 15.** Various transmission electron micrographs of the SZ of the bead-on-plate Weld 1, taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.
Fig. 16. Various transmission electron micrographs of the SZ of the bead-on-plate Weld 3, taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.

Fig. 17. Various transmission electron micrographs of the SZ of the bead-on-plate Weld 4, taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.

Fig. 18. Grain size distribution of the stir zones of the bead-on-plate welds; provided in addition to Fig. 3b in Chapter 2.

Fig. 19. Inverse pole figures of (a) the fine grained BM from XRD data, b) Butt Weld 1, (c) Butt Weld 2, (d) Butt Weld 3. (b)-(d) Data from EBSD data collected using the same method and location on the sample as was used for Fig. 3(b) in Chapter 2. WD, TD and ND denote Welding Direction, Transverse Direction and Normal Direction, respectively.

Fig. 20. Stress-strain graphs of the tensile specimens tested at room temperature.

Fig. 21. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at RT, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

Fig. 22. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at 150 °C, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

Fig. 23. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at 300 °C, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

Fig. 24. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at 600 °C, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

Fig. 25. Scanning electron micrographs of the fracture surface of the bead-on-plate welds at 500 °C. (a) Weld 1, (b) Weld 4 and (c) Weld 5. Inserted are macroscopic fracture surfaces (top) and optical micrographs of fractured dog bone specimens (bottom). Images taken on a taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

Fig. 26. Fractured tensile specimens: (a) Weld 1 at RT, (b) Weld 1 at 500 °C, (c) Weld 3 at RT and (d) Weld 3 at 500 °C. Welds are bead-on-plate welds as featured in Chapter 3.

Fig. 27. Images of the setup at the SALSA beamline at ILL, France, for measuring the residual stresses of the welded ODS steel plates by neutron diffraction.
List of Tables

<table>
<thead>
<tr>
<th>Chapter</th>
<th>Page</th>
<th>Table</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>27</td>
<td>Table 1. Table of the expected operating conditions for the Gen IV reactors [19].</td>
</tr>
<tr>
<td>1</td>
<td>28</td>
<td>Table 2. Materials under consideration for use in Gen IV reactors [17]. P is a primary considered material and S is a secondary considered material.</td>
</tr>
<tr>
<td>1</td>
<td>32</td>
<td>Table 3. Chemical composition of MA956 ODS alloy (wt.%).</td>
</tr>
<tr>
<td>1</td>
<td>35</td>
<td>Table 4. Y-Al-O compounds found in iron and nickel ODS alloys [36].</td>
</tr>
<tr>
<td>2</td>
<td>77</td>
<td>Table 1. Chemical composition of the studied MA956 ODS steel (wt.%).</td>
</tr>
<tr>
<td>2</td>
<td>77</td>
<td>Table 2. Welding parameters and characteristic parameters of the welded microstructures: mean grain diameter of the ferritic matrix, and relative volume fraction and mean diameter of the oxide nanoparticles. ‘BM’ denotes the base material.</td>
</tr>
<tr>
<td>3</td>
<td>96</td>
<td>Table 1. Chemical composition of the studied MA956 ODS steel (wt.%).</td>
</tr>
<tr>
<td>3</td>
<td>97</td>
<td>Table 2. Welding parameters, i.e. tool traverse and rotation speed, used for the associated weld number.</td>
</tr>
<tr>
<td>3</td>
<td>107</td>
<td>Table 3. Welding parameters and characteristic parameters of the welded microstructures: mean grain diameter of the ferritic matrix, and relative volume fraction and mean diameter of the oxide nanoparticles. ‘BM’ denotes the base material. Data taken from ref. [19]. The table also contains the values of the interparticle spacing (λ) and particle strengthening due to Orowan looping (σP), estimated using Eq. (2) and (3), see text.</td>
</tr>
<tr>
<td>4</td>
<td>118</td>
<td>Table 1. Chemical composition of the studied MA956 ODS steel (wt.%).</td>
</tr>
<tr>
<td>4</td>
<td>123</td>
<td>Table 2. Friction stir welding parameters in the steady state regime, together with the peak tensile residual stress along the longitudinal and traverse directions for the three ODS steel welds produced in this study. The average cooling rate has been determined between the peak temperature and 400 °C. Weld 1 used a mean value of only thermocouples 2–4 in the calculation. Weld 2 and 3 used a mean value of both thermocouples.</td>
</tr>
<tr>
<td>5</td>
<td>136</td>
<td>Table 1. Table 1 Chemical composition of the studied MA956 ODS steel (wt.%).</td>
</tr>
<tr>
<td>5</td>
<td>139</td>
<td>Table 2. Mean grain size of the three studied friction stir welds with varying tool traverse speed, as determined from EBSD maps, together with the measured peak temperature during welding [31].</td>
</tr>
<tr>
<td>5</td>
<td>142</td>
<td>Table 3. Average grain size obtained from optical micrographs at the selected locations of the weld cross section indicated in Figure 4. d is the mean grain diameter with the standard error provided in the parentheses. σ is the standard deviation of the grain diameter distribution. Units are in micrometres.</td>
</tr>
<tr>
<td>7</td>
<td>165</td>
<td></td>
</tr>
<tr>
<td>----</td>
<td>-----</td>
<td></td>
</tr>
</tbody>
</table>

**Table 1.** Measured grain sizes at various locations across the LN plane of the bead-on-plate Weld 2, indicated in Fig. 7. $d$ is the mean grain diameter with the standard error provided in the parentheses. $\sigma$ is the standard deviation of the grain diameter. Units are in micrometres.
Abstract

In this project, we have successfully joined MA956 Oxide Dispersion-Strengthened (ODS) steel plates using Friction Stir Welding (FSW). ODS steels are prime candidate materials for the fuel cladding in Generation IV nuclear fission reactors and as first wall components in nuclear fusion reactors. This is due to their exhibiting excellent high temperature strength and creep behaviour, together with enhanced resistance to radiation-induced void swelling. ODS steels are heavily reliant on a fine dispersion of (Y-Al-O) nano-oxide particles to provide the aforementioned properties. This, however, makes ODS steels particularly problematic to join. Most joining techniques melt the material along the joint line, but this would severely alter or deplete the nano-oxide dispersion and hence be highly detrimental to the material’s performance in a nuclear environment. FSW is a solid-state joining technique, and therefore can join ODS steel without melting the material. Although FSW can potentially alter the microstructure of the base material and affect the distribution of nano-oxide particles, if a sufficient number of nano-sized particles and a sufficiently homogeneous dispersion remain after the welding process, then a major roadblock for the implementation of ODS steels will have been removed.

The research of this thesis focused on the impact of FSW on: i) the microstructure, ii) the mechanical properties, iii) the residual stresses, and iv) the abnormal grain growth behaviour of ODS steels; utilizing a wide array of techniques to assess the micro-to-nano scale structure and the properties of the base material and welds, including optical, scanning and transmission and electron microscopy, X-ray and neutron diffraction, small-angle neutron scattering, tensile testing and micro-hardness measurements. We also produced welds with systematic changes to the tool traverse speed and rotation speed to investigate the impact of changing the welding parameters on the weld microstructure, and therefore optimise the process parameters for enhanced radiation and mechanical performance of the ODS steel welds.
Declaration

I, the author, declare that no portion of the work referred to in the thesis has been submitted in support of an application for another degree or qualification of this or any other university or other institute of learning.
Copyright Statement

I. The author of this thesis (including any appendices and/or schedules to this thesis) owns certain copyright or related rights in it (the “Copyright”) and he has given The University of Manchester certain rights to use such Copyright, including for administrative purposes.

II. Copies of this thesis, either in full or in extracts and whether in hard or electronic copy, may be made only in accordance with the Copyright, Designs and Patents Act 1988 (as amended) and regulations issued under it or, where appropriate, in accordance with licensing agreements which the University has from time to time. This page must form part of any such copies made.

III. The ownership of certain Copyright, patents, designs, trademarks and other intellectual property (the “Intellectual Property”) and any reproductions of copyright works in the thesis, for example graphs and tables (“Reproductions”), which may be described in this thesis, may not be owned by the author and may be owned by third parties. Such Intellectual Property and Reproductions cannot and must not be made available for use without the prior written permission of the owner(s) of the relevant Intellectual Property and/or Reproductions.

IV. Further information on the conditions under which disclosure, publication and commercialisation of this thesis, the Copyright and any Intellectual Property University IP Policy (see http://documents.manchester.ac.uk/display.aspx?DocID=24420), in any relevant Thesis restriction declarations deposited in the University Library, The University Library’s regulations (see http://www.library.manchester.ac.uk/about/regulations/) and in The University’s policy on Presentation of Theses.
Acknowledgements

I would like to thank my supervisor, Enrique Jimenez-Melero, for all his effort and dedication while working with me on this project. I would also like to thank him along with my industrial contacts, Marta Serrano and Stephen Cater, for the initial organising of an interesting project and for their support at important periods during the research.

I would like to extend my thanks to all the people I collaborated with for their help and effort in producing valuable results for this thesis. I also acknowledge the hospitality of all the institutions with which I collaborated. This includes CIEMAT, Spain, and TWI, UK, who both sponsored the project.

I am also grateful to anyone who offered me any help, advice or training, and to anyone throughout my time at the University of Manchester who helped make it a friendly and enjoyable place to work.

Finally, I extend my gratitude to the Engineering and Physical Sciences Research Council (EPRSC) for providing funding through the Centre for Doctoral Training in Advanced Metallic Systems under Grant Agreement EP/L016273/1.
Chapter 1

1. Introduction

1.1. Scope of thesis

This thesis focuses on successfully joining MA956, an Oxide Dispersion-Strengthened (ODS) steel, for use in future generation fission and fusion reactors. If ODS steels are found to be the most desirable material for structural components within a nuclear reactor, then finding a suitable method for joining them that is able to maintain their essential properties is of critical importance. Further details on material requirements and why the selection of a suitable joining method is of such great importance is discussed in Sections 1.2 and 1.4 of this Chapter, respectively. The work aims to both successfully join MA956 via the solid-state technique, friction stir welding (FSW), and investigate the impact that the welding process has on the material. This includes the impact of FSW on the microstructural and mechanical properties along with measuring the residual stresses created by the process. We have also investigated the effect of exposing the welds to a high temperature post-weld heat treatment (PWHT) to investigate induced abnormal grain growth (AGG) and the coarse microstructures produced. Welds using several different parameters have been assessed so that all the material properties of future welds can be optimised for use in nuclear reactors. The remainder of this section continues on to discuss pertinent background information to the thesis including a discussion of nuclear energy, future reactor environments, ODS steel production and behaviour, FSW, grain growth and measuring residual stresses.

1.2. Nuclear power

1.2.1. Energy generation

Nuclear fuels release their energy from the nuclei of their atoms. Combustion of fuels, such as gas and oil, release the energy stored in their chemical bonds. Because of this nuclear energy is able to produce vastly greater amounts of energy per kilogram of fuel. The energy released from 1 kg of oil is ~45 MJ hours whereas 1 kg of uranium can typically release ~50000 MJ [1]. The energy released by nuclear fuels comes from the energy that holds together all the nucleons (protons and neutrons) in a nucleus, known as the binding energy. This is the work that must be done to overcome the nuclear strong force to separate all the nucleons in the nucleus.
Fig. 1 shows a graph of the binding energy per nucleon against the mass number, A. Iron has the highest binding energy per nucleon and so it is the most energetically stable nucleus since the binding energy is a negative potential energy. Therefore it is energetically favourable for the nuclei of elements much larger than iron to split to become smaller nuclei (fission) and for elements much smaller than iron to combine to form larger nuclei (fusion).

Fig. 1. Binding energy per nucleon versus the mass number, A [2].

The binding energy released originates from a mass defect created by the fusion or fission process. The parent nuclei have a higher total mass than the daughter nuclei they create. This deficit in mass is released in the form of energy, since mass and energy are famously related by the equation:

$$\Delta E = \Delta mc^2$$  \hspace{1cm} (1)

In fission, a neutron is absorbed by a large nucleus, such as Uranium-235 creating Uranium-236, which is highly unstable. The unstable nucleus splits into two smaller, similarly sized nuclei, in this case Krypton and Barium, see Fig. 2. The reaction also releases three free neutrons which can themselves be absorbed by another Uranium-235 nucleus to create yet another fission reaction. This process can continue to create a chain reaction. The energy from a controlled chain reaction can be harnessed to produce electricity. The fission of Uranium-235 can also create Barium-144 and Krypton-90 along with 2 free neutrons as decay products. On average the fission of Uranium-235 releases an average of 2.4 free
neutrons [3]. Uranium-235 is a very common nuclear fuel but some other elements with large mass numbers can also be used such as Thorium-232 and Plutonium-239.

In nuclear fusion, single nucleons or very small nuclei, such as deuterium and Helium-3, fuse together releasing energy while in a plasma state. To overcome the Coulomb barrier between the light nuclei, they must have extremely large kinetic energies. The temperature required for a deuterium-tritium reaction (Eq. 2), the most promising fusion reaction, will require temperatures in excess of 100 million degrees Celsius [4]. This is one the major difficulties of producing energy from fusion, along with containment of the plasma.

Fig. 2, showing examples of fission and fusion reactions, also shows the mass of the parent and daughter nuclei. The difference between the two is the energy released by the reaction. The masses in Fig. 2 are given in both MeV and atomic mass units, u.

![Diagram of nuclear reactions](image)

**Fig. 2.** Examples of fission and fusion reactions [5].

Some of the possible fusion reactions are given in Eq. 2-7. Below them, Fig. 3 displays the nuclear cross sections against energy for the given reactions. The cross section of a reaction is a “characteristic area” used to quantify the probability of the nuclear reaction occurring. The energy is given as the centre-of-mass energy since all the particles are moving, at relativistic speeds, in a multi-particle system. The graph illuminates why the deuterium-tritium (D-T) reaction is the most promising reaction for producing fusion energy. The reaction ignites at a lower energy and the reaction has the greatest cross section for much of the energy range given, certainly for energies hoped to be attained in fusion reactors. Therefore the D-T reaction is the most promising to produce a self-sustaining reaction.
D-T: \[ ^2H + ^3H \rightarrow ^4He + ^1n + 17.6\ MeV \] (2)
D-D: \[ ^2H + ^2H \rightarrow ^3He + ^1n + 3.3\ MeV \] (3)
D-D: \[ ^2H + ^2H \rightarrow ^3H + ^1p + 4.0\ MeV \] (4)
D-\(^3\)He: \[ ^2H + ^3He \rightarrow ^4He + ^1p + 18.3\ MeV \] (5)
p-\(^{11}\)B: \[ ^{11}B + ^1H \rightarrow ^4He + ^4He + ^4He + 8.7\ MeV \] (6)
\(^3\)He-\(^3\)He: \[ ^{11}B + ^1H \rightarrow ^4He + ^1p + ^1p + 12.9\ MeV \] (7)

**Fig. 3.** Cross sections of fusion reactions against energy [6].

### 1.2.2. Radiation damage to materials

The neutrons involved with nuclear energy production can also interact with the surrounding structural materials of the reactor. It is very important to limit the effects of radiation damage on structural materials as it can cause void swelling, loss of strength and ductility, and increase the ductile-brittle transition temperature, to mention a few of the potential issues. The mechanisms for radiation damage in matter can be divided into three main categories: scattering collisions, electronic excitations and ionisations, and nuclear reactions – also known as transmutations. Here, only damage caused by neutrons will be discussed but in a nuclear fusion reactor, damage can also be caused by protons and ions.

Transmutation is a nuclear reaction that causes the conversion of an element or isotope into another. Perhaps the most common and damaging are nuclear reactions that lead to the production of helium and hydrogen, \((n,\ \alpha)\) and \((n,\ p)\) reactions, respectively. The production
of these elements is a problem in metals since they can coalesce to form bubbles that grow and can lead to premature failure. Helium is produced when an excited atom decays to an alpha particle which quickly acquires electrons to become elemental helium [7]; the reaction occurs extremely rarely with iron but can occur more commonly with other elements, most notably nickel, which is why nickel ODS alloys are not considered for core nuclear applications. Other reactions can lead to the production of radioactive isotopes, and therefore to active material.

Other possible transmutations are \((n, 2n)\) and \((n, \gamma)\) reactions. In \((n, 2n)\) the nucleus is excited by absorbing an incident neutron. The nucleus can then return to a lower energy state by emitting two neutrons. For this reaction to occur, the incident neutron must excite the nucleus to an energy greater than that of the energy binding the second neutron the nucleus [8]. These additional neutrons can then go on to cause further damage by creating further displacements or transmutations in the material. In the \((n, \gamma)\) reaction the incident nucleus absorbs a neutron and emits a photon. As the nucleus emits the photon it recoils; often with sufficient energy to displace another atom [8].

Neutrons can also interact with nuclei without changing their nature, by scattering. An elastic scattering event is when a neutron scattered by a lattice nucleus experiences no overall change in energy, only in direction. An inelastic scattering event occurs when the incident neutron has a lower energy after the scattering event than before. The energy must be conserved and indeed the change in the neutron’s energy is transferred to the scattering nucleus.

When a neutron is scattered by a nucleus in the lattice, be it via elastic or inelastic scattering, the nucleus must recoil in order to conserve momentum. A neutron with sufficient energy will be able to cause the nucleus to recoil with enough momentum as to become displaced from its lattice site. The displaced nucleus will come to rest in an interstitial position in the lattice away from its original location. This is known as a primary knock-on atom (PKA) [9]. If in turn the PKA has enough energy to displace another nucleus it can produce secondary knock-on atoms. So long as there is enough energy, high order knock on atoms can be created. A large number of nuclei can hence be displaced in what is known as a displacement cascade. These displacement cascades form SIAs and vacancies. On longer time scales, the evolution of these point defects can cause the formation of larger defects, such as voids or dislocation loops.
Fig. 4. Possible damage to a lattice from a displacement cascade [10].

Fig. 4 shows a possible end product of a lattice after a displacement cascade. The lattice is left with a large number of point defects including, voids, vacancies and self-interstitials (Frenkel pairs). All of these locally change the microstructure and therefore change the mechanical properties of the material. Only a small number of the displacements created in the cascade will actually survive; a large proportion of the created self-interstitials will quickly annihilate with the vacancies, in picoseconds timescales. The fraction of defects that survive is known as the displacement efficiency [8]. Radiation damage is usually quantified in units of displacements per atom (dpa). n dpa would represent that, on average within the irradiated volume, an atom has been displaced n times from its equilibrium position [11].

1.2.3. Gen IV fission reactors

There have been six proposed designs of fission reactors that have been selected for further research and development by the Generation IV International Forum on the basis “being clean, safe and cost-effective means of meeting increased energy demands on a sustainable basis, while being resistant to diversion of materials for weapons proliferation and secure from terrorist attacks” [12]. Currently it is hoped that a number of these will be ready for commercial use shortly after 2030. More information on the Generation IV project and each of the proposed reactors can be found on the US Nuclear Energy Foundation website [13] amongst other sources.
1.2.4. Fusion reactors

Nuclear fusion reactors are currently far from being economically viable and are yet to be self-sustaining. Yet the theory behind nuclear fusion is relatively well understood. With further research and further improvement in advanced engineering materials, the scientific community hopes that a commercially viable fusion reactor may be operational in the coming decades. There are two main proposed approaches to nuclear fusion: Inertial Fusion Confinement (IFC) and Magnetic Fusion Confinement (MFC) [14]. MFC uses magnetic fields to confine the plasma spiralling within a toroidal reactor. The plasma needs to be contained for a prolonged period of time so that enough energy is produced from the fusion reactions to raise the temperature to a self-sustaining level. In IFC, a small pellet of several millimetres diameter made from deuterium and tritium is heated rapidly by a single powerful laser. The laser is split and redirected to heat the pellet evenly from all sides. This method is the most common form of ICF but there are several variants on this method. The rapid heating causes the surface to rapidly heat up and become surrounded by a plasma surface layer. The “rocket-like blow off” of the plasma at the surface forces the remainder of the pellet to violently contract in an implosion. As the pellet contracts, the deuterium and tritium reach a critical density for the fusion reaction to be initiated [15, 16].

1.2.5. Operating conditions

While developing or researching alloys for use as structural materials in future nuclear reactors, it is important to consider the operating conditions to which they will be subjected. Generally speaking, future generations of nuclear reactors will be at operating higher temperatures and under higher radiation levels compared with current Generation II (Gen II) reactors, in order to increase the efficiency of energy production. Many currently utilised materials will therefore no longer be fit for purpose, either for safety, economic or practical reasons.

The operating temperatures for the Generation IV reactors will be much higher than that of current Gen II reactors, which generally do not run at temperatures greater than 350-400 °C [17, 18]. Many materials currently used in nuclear reactors will not capable of operating safely at these temperatures because they will become more susceptible to degradation and failure through mechanisms such as creep rupture or hydrogen embrittlement. The temperature can also cause an excessive drop in strength or too large an increase in dimensions at the high temperatures. Along with withstanding the increases in temperatures and pressures, Gen IV structural materials must also show “acceptable resistance” to all
forms of radiation damage at high total neutron doses of between 10-150 dpa [17]. Table 1 shows some of the key operating conditions of the Generation IV reactors [19]. The acronyms in Table 1 stand for Very-High-Temperature reactor, Gas-cooled Fast reactor, Sodium-cooled Fast reactor, SuperCritical Water reactor, Lead-cooled Fast reactor and Molten Salt reactor, from top to bottom. Further information on the specific reactor types can be found in [12, 20].

<table>
<thead>
<tr>
<th>System</th>
<th>Size (MWe)</th>
<th>Operating Temperature (°C)</th>
<th>Fast or Thermal</th>
<th>Pressure (MPa)</th>
<th>Output</th>
</tr>
</thead>
<tbody>
<tr>
<td>VHTR</td>
<td>250</td>
<td>1000</td>
<td>Thermal</td>
<td>7</td>
<td>Electricity and Hydrogen</td>
</tr>
<tr>
<td>GFR</td>
<td>200-1200</td>
<td>850</td>
<td>Fast</td>
<td>7</td>
<td>Electricity and Hydrogen</td>
</tr>
<tr>
<td>SFR</td>
<td>300-1500</td>
<td>550</td>
<td>Fast</td>
<td>0.1</td>
<td>Electricity</td>
</tr>
<tr>
<td>SCWR</td>
<td>1500</td>
<td>510-550</td>
<td>Thermal</td>
<td>25</td>
<td>Electricity</td>
</tr>
<tr>
<td>LFR</td>
<td>50-150, 300-600, 1200</td>
<td>550-800</td>
<td>Fast</td>
<td>0.1</td>
<td>Electricity and Hydrogen</td>
</tr>
<tr>
<td>MSR</td>
<td>1000</td>
<td>700-800</td>
<td>Thermal</td>
<td>0.1</td>
<td>Electricity and Hydrogen</td>
</tr>
</tbody>
</table>

Table 1. Table of the expected operating conditions for the Gen IV reactors [19].

Operating temperatures of fusion reactors are likely to be even higher, around 1000 °C in parts, and with this will come the need for a high pressure coolant [21]. Structural materials in contact with the coolant must also ensure negligible corrosion. The radiation environment will be particularly harsh. The neutrons will have energies of 14.1 MeV, far higher than any current fission reactor (1-2 MeV). The damage level is predicted to be as high as 200 dpa [22]. The facilities to simulate neutron damage at this level do not currently exist and data must be extrapolated from lower levels of damage.
Fig. 5. Expected total radiation dose and operating temperatures of Gen IV and fusion reactors compared to Gen II fission reactors [22].

1.2.6. Future reactor candidate materials

There are several materials of special interest that are being researched and developed for use as structural materials in the future generation of reactors. The harsh environment of the reactor means that many candidate materials will not meet the required specifications. An excellent overview of the most promising candidate materials has been written by Murty and Charit [17], see Table 2. It should be noted that several materials may be required for different reactor components as the conditions and requirements will depend on the purpose and position of the structural component in the reactor.

<table>
<thead>
<tr>
<th>Reactor system</th>
<th>F–M steel</th>
<th>Austenitic S.S.</th>
<th>ODS steel</th>
<th>Ni-base alloys</th>
<th>Graphite</th>
<th>Refractory alloys</th>
<th>Ceramics</th>
</tr>
</thead>
<tbody>
<tr>
<td>GFR</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>–</td>
<td>P</td>
<td>P</td>
</tr>
<tr>
<td>LFR</td>
<td>P</td>
<td>P</td>
<td>S</td>
<td>–</td>
<td>–</td>
<td>S</td>
<td>S</td>
</tr>
<tr>
<td>MSR</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>P</td>
<td>P</td>
<td>S</td>
<td>S</td>
</tr>
<tr>
<td>SFR</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>SCWR</td>
<td>P</td>
<td>P</td>
<td>S</td>
<td>S</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>VHTR</td>
<td>S</td>
<td>–</td>
<td>–</td>
<td>P</td>
<td>P</td>
<td>S</td>
<td>P</td>
</tr>
</tbody>
</table>

Table 2. Materials under consideration for use in Gen IV reactors [17]. P is a primary considered material and S is a secondary considered material.
1.2.7. Reduced-activation steels

Although fusion reactors do not contain a radioactive core, the high energy neutrons produced in the reactor are able to induce highly significant levels of transmutations in the surrounding structural materials. The resultant nuclides may be radioactive and activate the material. The activated material may be radioactive for a very long time, making recycling or disposal of the reactor difficult and expensive, as well as possibly raising safety and environmental concerns amongst the public [23]. To alleviate these concerns, materials that are to be considered for use in fusion reactors must be able to be disposed of or recycled safely after approximately 100 years, i.e. the structural materials must have returned to safe levels of radioactivity in this given period [24].

The decay rates and irradiation levels from activated materials depend on the elements, and isotopes, of the material. Fig. 6, taken from [23] shows the irradiation levels against decay time for elements commonly found in steel alloys following the shutdown of 3.6 GW fusion power reactor with an assumed irradiation flux of \(~10^{19} \text{ m}^{-2}\text{s}^{-1}\) for 5 years [25].

![Fig. 6. Irradiation levels of common steel alloying elements following exposure to blanket front end irradiation doses. The black line represents the ITER administrative safe irradiation level for hands-on maintenance [26].](image)

Many of the common alloying elements in steel, such as Mo and Nb, remain active for 1000s of years. Therefore, the new generation of steels being developed to withstand the higher operating temperatures and irradiation levels must also be composed without, or with very small quantities of, many of the elements included in the previous generation of nuclear grade steels in order to be considered reduced-activation and meet the 100-year safe handling criteria.
The main 3 types of reduced-activation steels are reduced-activation ferritic/martensitic (RAFM) steel, reduced-activation stainless steels and reduced-activation oxide dispersion-strengthened steels. While reduced-activation steels are not a requirement for future fission reactors, many are still being considered and developed for use in Gen IV nuclear fission reactors.

1.3. Oxide Dispersion-Strengthened (ODS) steels

1.3.1. Introduction to ODS steels

ODS steels, the subject of this PhD project, are currently a primary option for use in two of the six Gen IV fission reactors and a secondary option for another two [13, 17] according to the Nuclear Energy Research Advisory Committee; thus their development is of great importance. They are being developed for use as first wall and blanket structural materials in fusion reactors, and as fuel cladding in fission reactors. Currently, zirconium alloys are used for fuel cladding in Gen II reactors but they have significant problems with buckling of the assembly from irradiation growth and creep, and of embrittlement from zirconium hydride formation.

ODS steels are steel alloys that contain a fine dispersion of nano-scale ceramic oxide particles, usually alumina (Al₂O₃) or yttria (Y₂O₃). They are typically alloyed with significant amounts of Cr for corrosion resistance.

In terms of high temperature performance, ODS steels must be shown to be operational at a minimum of 550 °C for use in any of the Gen IV reactors systems and a minimum of 850 °C to be used in the Gas-cooled Fast Reactor (GFR). ODS alloys are also in contention for other non-nuclear applications, such as high temperature turbines and heat exchangers, to be used at temperatures above 1000 °C.

Nickel-base ODS alloys do exist that have excellent high temperature strength but they are in much lower contention for nuclear applications since nickel is not considered as a low-activation material. Nickel nuclei can absorb neutrons making them unstable; they will decay releasing an alpha particle that will quickly become a helium atom [7]. This process will eventually lead to helium embrittlement.

ODS steels are currently considered as a promising candidate due to their improved mechanical performance at higher temperatures, compared to current Gen II reactor materials, and improved radiation resistance. Many of the advantageous properties of ODS
steels stem from their dispersion of nano-scale oxide particles within the matrix. These particles act to hinder the movement of dislocations, which improves creep performance while maintaining good strength and toughness at high temperatures. Those particles also act as effective sinks for the lattice defects induced by radiation and as nucleation sites for helium. These properties will be discussed in more detail in Section 1.3.5.

1.3.2. Production

ODS alloys are manufactured using mechanical alloying (MA) which is a solid-state powder processing technique originally developed for the production of iron and nickel-base ODS superalloys. The main advantages of mechanical alloying are that it can produce a highly homogenous distribution of alloying elements, create an alloy with a controllable microstructure and, vitally, it allows the addition of fine dispersion of ceramic particles, usually oxides such as Y₂O₃, which cannot be achieved by other methods since the oxides cannot be dispersed in the liquid state [27].

The first stage of producing alloys through the MA process is the addition fine powders of the alloying elements and intermetallics, in their relative percentages, into a high energy ball mill. In the ball mill the powder particles repeatedly weld together, fracture, and then weld together again [28]. During this intense and repeated mechanical deformation a very fine, controllable microstructure can be produced, while producing a (near) homogeneous distribution of the constituent elements and intermetallics [29-31]. Bhadeshia [29] showed that the major alloying elements in MA956 are extremely close to having a truly homogenous distribution. He describes the MA process as forcing a random distribution of elements rather than being achieved thermodynamically. The ball milled powder is then consolidated by either Hot Isostatic Pressing (HIP) or, usually, by extrusion at high temperature to produce a consolidated piece with a non-equilibrium distribution.
One ODS steel alloy of particular interest for future nuclear applications, that will be the centre of research for this thesis, is MA956, as previously mentioned, which contains a high percentage of chromium, ~20 wt.%, and ~5 wt.% aluminium in a fully ferritic matrix which allows for effective oxidation and corrosion resistance up to very high temperatures (> 1000 °C).

The chemical composition is shown Table 3. The MA956 sample was manufactured by Special Metals Corporation, Hereford, UK. MA956 is consolidated after mechanical alloying by extrusion at around 1000 °C followed by hot rolling in both the longitudinal and transverse directions [33] at 900-1100 °C. The material was then zone annealed at 1320 °C for one hour followed by air cooling. The MA956 is in the form of a plate with 10 mm thickness.

<table>
<thead>
<tr>
<th>Cr</th>
<th>Al</th>
<th>Y₂O₃</th>
<th>P</th>
<th>Ti</th>
<th>O</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
<th>Ni</th>
<th>Co</th>
<th>N</th>
<th>Cu</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.97</td>
<td>4.44</td>
<td>0.53</td>
<td>0.33</td>
<td>0.21</td>
<td>0.15</td>
<td>0.11</td>
<td>0.05</td>
<td>&lt;0.05</td>
<td>0.04</td>
<td>0.03</td>
<td>0.022</td>
<td>0.009</td>
<td>0.004</td>
<td>bal.</td>
<td></td>
</tr>
</tbody>
</table>

Table 3. Chemical composition of MA956 ODS alloy (wt.%).

Though, overall, the MA process is an excellent method of producing new alloys including ODS steels, there are two particular issues to note when dealing with MA produced materials, namely powder oxidation and porosity. Mechanical alloying involves the use of extremely fine powders which therefore have an extremely large surface area which may become a problem, particularly when using reactive elements such as titanium which MA956 includes in its composition. The powders can oxidise with elements in the air which are
difficult to keep out of the ball mill’s chamber [34]. Gas can be used to purge the chamber, but this can cause fine bubbles to form near dispersoids [35]. During the mechanical alloying process, fresh, rough surfaces are continually exposed to gases which can be absorbed [36]. These gases can later lead to porosity. Porosity is not thought to be a problem in the as-extruded condition [37]. Most porosity in ODS alloys forms during the annealing process, or long exposure to elevated temperatures [37-40].

1.3.3. Microstructure

The high strains, often in the magnitude order of 9 [34], incurred by the material from the mechanical alloying process leaves ODS alloys with exceptionally small grains of the order of nanometers or tens of nanometers [29, 41]. The high temperature of the consolidation process causes the alloys to recrystallize and have larger, but still sub-micron grain sizes. Extrusion and hot rolling in ODS steels normally creates slightly elongated grains and may cause the dispersoids to align in the rolling direction [36, 42].

This alignment of the dispersoids is not seen as strongly in Nickel ODS alloys [42, 43]. As a result, Nickel ODS alloys, such as MA6000, tend to have more equiaxed grains after primary recrystallization. Baloch and Bhadeshia [44] showed some dispersoid alignment but only on the relatively large scale viewed in an optical microscope. The same paper shows that on a much smaller scale, in a TEM image, there is no such alignment seen. The large scale alignment’s effect on the grain size during recrystallization will be extremely limited when considering such small grains.

Fig. 8. Transmission electron micrograph showing the dispersoids in MA956 aligned parallel to the extrusion direction [29].
ODS alloys are often given this further heat treatment to induce AGG to create the large columnar grains and to reduce the hardness of the alloys, which are initially very hard [29] due to the large number of dislocations created during the plastic deformation of the MA process. However, the heat treatment must be done at a very high temperature as ODS alloys typically have a recrystallization temperature of 0.9 of their melting temperature, $T_m$, whereas standard alloys have a recrystallization temperature typically of 0.6 $T_m$, despite the large stored energy in ODS steels from the MA process [29]. A typical annealing temperature of ODS steels is 1300-1400 °C.

Work is still ongoing into fully understanding the mechanisms of recrystallization and AGG in mechanically alloyed ODS steels. However, the main causes of the extremely high temperature required to induce AGG is the strong pinning force, Zener drag, from the fine particle dispersion, along with a very homogeneous and highly textured microstructure [45-47].

The presence of a fine particle dispersion with small interparticle spacing and the presence of a strong (100)<110> texture which ferritic ODS steels tend to have following rolling and/or extrusion, have been shown to produce highly uniform distributions of dislocations following cold rolling [48]. This lack of heterogeneity suppresses the presence of nucleation sites [49].

The limited number of nucleation sites when above the recrystallization temperature also helps produce the particularly coarse microstructure (> mm grains) often seen in ODS alloys since only a small number of grains will begin to grow at the expense of others. The high grain aspect ratio (GAR) of the coarse grains is due to the pinning forces of the particles that are aligned along the extrusion axis. This results in a grain boundary migration velocity that is significantly greater along the extrusion axis, causing growth that creates long columnar structures along the extrusion direction, rather than grain growth being isotropic [50].

The MA956 plate of this thesis, after its high temperature annealing and hot rolling, is expected to have “large pancake” shaped grains [33]; large round grains in the Longitudinal-Transverse (LT) plane and long columnar grains on the Transverse-Normal (TN) and Longitudinal-Normal (LN) planes. The columnar grains are roughly between 200-500 µm thick and very long. [27, 29, 43, 51]. Krishnardula [27] describes them as typically 20 mm but Capdevila and Bhadeshia [43] reported that they can be as long as the sample. This particularly coarse microstructure may be particularly advantageous to the material’s
performance during long exposures at elevated temperature since the low grain boundary density, along with a high GAR, will reduce the creep rate.

The dispersoids of ODS alloys are added as $Y_2O_3$ particles during mechanical alloying. Some of the particles remain unchanged after extrusion. However with the heat and time of the extrusion and annealing processes, the dispersoids tend to react with elements such as Al and Ti in the alloy to produce more complex ceramics; Y-Al-O and Y-Ti-O [36].

<table>
<thead>
<tr>
<th>$3Y_2O_3.5Al_2O_3$</th>
<th>YAG</th>
<th>yttrium aluminium garnet</th>
</tr>
</thead>
<tbody>
<tr>
<td>$Y_2O_3.Al_2O_3$</td>
<td>YAH</td>
<td>yttrium aluminium hexagonal</td>
</tr>
<tr>
<td>$2Y_2O_3.Al_2O_3$</td>
<td>YAM</td>
<td>yttrium aluminium monoclinic</td>
</tr>
<tr>
<td>$Y_2O_3.Al_2O_3$</td>
<td>YAP</td>
<td>yttrium aluminium perovskite</td>
</tr>
<tr>
<td>$3Y_2O_3.Al_2O_3$</td>
<td>YAP'</td>
<td>yttrium aluminium pseudo-perovskite</td>
</tr>
<tr>
<td>$3Y_2O_3.5Al_2O_3$</td>
<td>YAT</td>
<td>yttrium aluminate tetragonal</td>
</tr>
</tbody>
</table>

Table 4. Y-Al-O compounds found in iron and nickel ODS alloys [36].

The types of Y-Al-O that are present in the matrix are dependent on the time and temperature exposure of the ODS alloy along with composition of the alloy. In MA956, the aluminium tends to react with the yttrium preferentially and the titanium tends to form titanium nitrides or carbonitrides Ti(CN) [27, 52, 53]. The three types of particle generally seen in ODS alloys are Y-Al-O, Al-O and Ti(CN), the latter two being much larger, typically several hundreds of nanometres. The yttrium oxides, including those containing aluminium, are much smaller and have a much greater number density. For MA956 these mostly range from 2-100 nm with a mean diameter of around 20 nm and a mode of around 12-15 nm. Particle size distributions for MA956 can be seen in [54-56]. These were created by using TEM imaging either on MA956 discs or carbon extraction replicas. In these cases both methods were able to determine very similar distributions, however using TEM directly may lead to bias when selecting areas for analysis.

Although all the compositions in Table 4 have been observed in ODS alloys, YAM, YAP, and YAG are the most thermodynamically stable [57]. In the annealed coarse grained condition, MA956 appears to be dominated by YAP and YAG type particles [56]. There is not much corroborating evidence since much of the related work on MA956 is on the as-extruded material condition. However, it agrees well with work by Chen et al. [58] on PM2000, which is a very similar ODS steel to MA956. Chen used high resolution STEM on
extraction replicas to determine that PM2000 after a 1 hour 1380 °C was dominated by YAP particles (~80%) with the remainder comprised of YAG and unreacted Y₂O₃.

The dispersions in ODS alloys have been found to be very stable up to very high temperatures and for prolonged periods of time. Krautwasser et al. [59] found that for PM2010, no substantial coarsening of the dispersoids was seen up to 1127 °C and Okasu and Fujiwara [60] also found the yttria particles were stable up to 1150 °C. However some coarsening will occur at high temperatures compared to the starting condition caused by uptake of aluminium, particularly by the Y₂O₃ particles. Most reported coarsening of the particles tends to be this initial uptake of Al, generally for alloys that have not received the initial high temperature anneal. Once in the Y-Al-O state the particles become even more stable. Backhouse et al. [61] observed little effect on the particles in coarse grained MA956 even when held at 1400 °C for 72 hours. It should be noted however that a small change in the dispersoids’ distribution may be able to cause a significant change in mechanical properties. At very high temperatures coarsening via Ostwald ripening can begin to occur. This can be particularly deleterious since the larger particles grow at the expense of the smaller particles, increasing the interparticle spacing.

Throughout the project, the MA956 will be subject to a significant range of temperatures during FSW, heat treatments and elevated temperature tensile testing. In some alloys, exposure to certain temperature ranges can induce phase changes. Fig. 9, shows a binary Fe-Cr phase diagram. With the ~20% Cr present in MA956 we do not expect a phase change at any temperature up to the solidus. The binary phase diagram is a simplification; however the other significant alloying element present in MA956 is Al which, like Cr, is a ferritic stabilizer. Therefore we do not expect any phase changes during testing or welding of MA956 and no phase changes have been reported in the literature.
1.3.4. ODS Strengthening mechanisms

The equation below (Eq. 8) shows the yield strength, $\sigma_{YS}$, of a crystalline material given as the sum of all of the individual strengthening mechanisms that act within the material. Although this equation is commonly used, it is far from perfect since the individual strengthening mechanisms are not truly independent. Eq. 8 is therefore prone to overestimating the true strength [63].

$$\sigma_{YS} = \sigma_0 + \sigma_{SS} + \sigma_P + \sigma_{GB} + \sigma_D$$ (8)

$\sigma_0$, commonly referred to as the Peierls–Nabarro stress, represents representing the intrinsic stress due to resistance of dislocation motion in the matrix.

$\sigma_{SS}$ is the solid solution strengthening contribution that originates from the presence of solute atoms in the solid solution and is therefore applicable to all alloys. Alloying elements that are similar in size to those of the solvent metal take their place in the lattice, in what is known as substitutional solid solution. If the solute atoms are considerably smaller than the solvent atoms, elements such as nitrogen and carbon, then the atoms will occupy the gaps in the lattice between the solvent atoms. This is known as interstitial solid solution. In both cases, the solute atom creates a local strain field in the surrounding lattice, see Fig. 10, which interacts with dislocations impeding there motion through the lattice [64].

Fig. 9. Binary phase diagram for Fe-Cr, calculated using Thermo-Calc [62].
\[ \sigma_P \] represents the strengthening contribution from the particles. Of the several strengthening mechanisms involved in ODS steels, perhaps the most of interest is the direct strengthening from the particles in the matrix, particularly those of the nanoparticle dispersion. At relatively low temperatures most of the strengthening in coarse grained ODS alloys is attributed to Orowan looping. Apart from the very smallest particles (< 10 nm) [53], the Y-Al-O particles tend to be incoherent with the matrix and unshearable. Dislocations must therefore bend around the oxide particles. Fig. 11 illustrates the simple Orowan bowing mechanism. When a dislocation line meets two particles in its glide plane it must bend and elongate between the two particles. The bowing line will reach a critical radius at which it can continue to move forward without further curvature. The segments of the dislocation line that meet on the other side of the particles are of opposite sign and so annihilate. This will leave a residual dislocation loop about the particles and a dislocation line that is again free to move. The bowing of the dislocation line requires additional stress and hence the particles act to strengthen the material.

**Fig. 10.** Left, diagram of a lattice with a substitutional solute atom, in red. Right, lattice with interstitial solute atoms, in black [65].

**Fig. 11.** Diagram showing a dislocation line bowing around an unshearable particle [66].
The strengthening contribution from Orowan looping, $\sigma_{Or}$, by particles is given by:

$$\sigma_{Or} = \frac{0.4}{\pi} \frac{M G b}{(1-\nu)^{0.5}} \frac{\ln(\frac{r}{b})}{\lambda} \quad (9)$$

$M$ is an orientation factor known as the Taylor factor, $G$ shear is the shear modulus, $b$ is the Burger’s vector. $\lambda$ is the mean interparticle spacing, $\nu$ is Poisson’s ration and $r$ is mean radius of the particles and $b$ is the Burger’s vector. Eq. 9 assumes that the particles are fully incoherent. The dislocation line may in reality be able to cut through the smaller particles that are coherent or semi-coherent with the surrounding matrix.

$$\lambda = \left[ \left( \frac{3\pi}{4f} \right)^{0.5} - 1.64 \right] r \quad (10)$$

The interparticle spacing is given by Eq. 10. For a fixed volume fraction of nanoparticles, it is far more effective to strengthen using a large number of small particles than a smaller number of larger particles since this yields a smaller interparticle spacing.

The Orowan strengthening was denoted as $\sigma_{Or}$ rather than $\sigma_P$. This is because the strengthening mechanism of particles significantly changes with temperature. At higher temperatures, when diffusion is easier, these particles may be overcome by dislocation climb. Orowan looping will therefore not be the dominant mechanism in these conditions. It has been observed in ODS steels that dislocation lines at high temperatures are straighter than at room temperature [67]. The details of the mechanism are still not fully understood, but it is believed that a large contribution of the strength at high temperature in ODS alloys arises from an attraction between the nano-particles and dislocations. In a model proposed by Artz et al. [68], originally based on the work by Slorovitz et al. [69], the dislocations undergo climb at the particle interface but then require additional stress to further advance due to the attraction between them. Evidence of this has been found in PM2000 [70], a similar ODS steel, and in MA956 [71] where dislocations were seen to be pinned on the departing side of the particles.

The strengthening due to grain boundaries is denoted as $\sigma_{GB}$. At relatively low temperatures, below approximately 500 °C [72], grain boundaries strengthen the material again by inhibiting the motion of dislocations through the matrix. Grain boundaries are the region between adjacent grains with differing crystallographic orientation. They are planar defects in which there is a discontinuity of the perfect crystallographic structure across a
plane in the material. This means a greater critical shear stress is required to allow slip across a grain boundary than through the centre of a grain. Therefore the dislocations tend to pile up at the grain boundaries. The relation between the grain size, d, and the strengthening due to the presence of grain boundaries, $\sigma_{\text{GB}}$, is given by the Hall-Petch relation,

$$\sigma_{\text{GB}} = k_{\text{HP}} \cdot d^{-0.5}$$  \hspace{1cm} (11)

where $k_{\text{HP}}$ is a constant often known as the locking constant. From the Hall-Petch equation we can see that having smaller grains will cause a significant increase in strength. However, at temperatures around 500 °C and above, the effect of strengthening from grain boundaries drops significantly. At high temperatures small grains may in fact be adverse for the strength of the material, since the high total length of the grain boundaries act as high diffusivity paths for dislocations [73].

$\sigma_D$ is the strengthening contribution from dislocation forest hardening. Dislocations require a larger stress to move through the matrix near to other dislocations since their presence induces local strains in the lattice and the dislocations may become entangled. So dislocation motion requires more energy in a material with higher dislocation density [74]. The strengthening contribution from dislocation forest hardening, $\sigma_D$, is given by:

$$\sigma_D = M \alpha G b \rho^{0.5}$$  \hspace{1cm} (12)

where $\alpha$ is a material dependent constant and $\rho$ is the dislocation density. All other symbols denote the same parameters as in Eq. 9. Mechanically alloyed materials, such as ODS steels, tend to have very large dislocation densities. This is caused by the severe amount of plastic deformation during the MA process which causes the multiplication of dislocations [67]. This creates a significant strengthening contribution from dislocation forest hardening. Annealing can greatly reduce the dislocation density but it can still remain relatively high compared to other alloys [63]. However, it is often decided that ODS steels should undergo a very high temperature heat treatment to induce AGG, such as the alloy at the centre of this thesis, in order to create a coarse grain structure that may be beneficial for high temperature applications. The coarse grains following AGG are essentially free of dislocations [75].

1.3.5. Radiation damage resistance

The nanoparticles that give ODS alloys much of their high temperature strength and creep resistance also provide the alloys’ excellent resistance to radiation damage. The particle-matrix interface acts as an effective recombination site for the self-interstitials and
vacancies, greatly reducing the number of defects in the lattice caused by radiation damage [76-78], such as dislocation loops and voids. The interfaces also act as nucleation sites for helium bubbles [79-81] which then agglomerate around the cluster, shown in Fig. 12. Having sites for trapping helium is an effective method of reducing bubble growth in radiation damaged materials [82, 83]. There have been several studies that show the swelling and creep in irradiated ODS steels is lower than in equivalent steels without dispersoids [77].

![Fig. 12. Illustration of how helium bubbles nucleate and agglomerate around nano-clusters [80].](image)

The nano-oxides are expected to be very stable under all types of irradiation [27, 84-93]. It is also thought that high energy neutrons or ions may even “refine” the nanoparticles [94-96] helping to provide extra strength and creep resistance.

1.4. Friction stir welding

1.4.1. Introduction

Since it is not possible to build a net shape nuclear reactor there is a critical need to develop a joining technique that conserves the structure and properties of ODS alloys enough to safely withstand the harsh environment to which they will be subjected. Most joining processes involve melting the material. However due to ODS alloys achieving their excellent properties largely from the dispersion of nano-particles, these methods are not viable since melting would cause severe agglomeration and/or dissolution of the nanoparticles. This would cause dispersoid free zones in the structure with inferior properties and therefore would not be fit for purpose.

Therefore for ODS steels, such as MA956, to ever be used in nuclear reactors, an adequate joining process must be found and developed that maintains the uniform dispersion of stable nano-oxides across the joint and in the parent material. It must also maintain the
alloy’s specific composition and the process must not form brittle phases or create porosity in the material which would act as points of weakness.

The potential solution to joining ODS steels with limited disruption to the nano-oxide dispersion is hoped to lie with solid-state welding. This is essentially any method of joining metals together below their melting temperatures (solidus). Krishnandum’s thesis [97] and Wei’s thesis [98] both contain thorough overviews of many of the considered joining techniques for ODS alloys. Despite the many possible solid-state joining techniques available, it is friction stir welding that currently holds the most exciting prospects for use with ODS alloys.

1.4.2. Friction stir welding (FSW) process

Friction stir welding (FSW) was invented by TWI in 1991 [99] for the purpose of welding high strength aluminium alloys. Since then the process has been further developed for many other metals, and some non-metals, which can now be friction stir welded at a range of thicknesses and is able to produce many different weld geometries from many different positions, which many other solid-state processes are unable to achieve. Although this thesis is looking at using ODS steel as a structural material in reactor plate components, the friction stir welding process could also potentially be applied to welding tubes for cladding which is where another possible use of ODS alloys lies.

The FSW process involves a rotating tool, consisting of a pin and shoulder, see Fig. 13. The toolhead, while rotating, is plunged into the material at the joint line. The friction from the rotating shoulder causes heat that plasticises the material. When sufficiently plasticised, the tool will traverse forward and the toolhead mechanically mixes the material across the joint line. Along with creating frictional heat, the shoulder also forces the displaced material downwards. This is a solid-state process, since the material is softened but not melted. Hence the potential for joining ODS alloys.
Fig. 13. Diagram showing the microstructural features of a FSW joint [100].

After materials have been friction stir welded they present several different microstructural zones, dependent on the distance away from the joint line:

a) **SZ**: The stir zone (SZ) also known as the weld nugget or dynamically recrystallized zone, is the central region of the weld, loosely located about the path of the tool’s pin. It is the region where the material has incurred the heaviest deformation and is usually recognisable by its roughly equiaxed grains that are often greatly refined in comparison to the base material [101].

b) **TMAZ**: The thermo-mechanically affected zone (TMAZ) technically refers to the entire deformed region but is commonly used to represent the deformed outside of the stir zone since the deformation here may be significantly less.

c) **HAZ**: The heat affected zone (HAZ) is unaffected by the mechanical processes of the weld but, even though the heat input is lower than in the TMAZ, the microstructure may have been affected by the heat input alone.

FSW is still a relatively new process that has a relatively long list of significant advantages. Here I shall briefly discuss the main potential advantages that FSW has over other processes specifically regarding ODS alloys for use as a structural material in nuclear reactors. Many other advantages can be found from other sources including the TWI website [102]. Some of these are advantages of solid state processes and others specific to FSW.
a) FSW is a solid-state welding process so the nanoparticles may avoid coarsening, agglomeration and/or dissolution.

b) There is little or no change to the composition of the material with FSW and there is likely to be limited segregation of the elements.

c) FSW generally produces welded structures with very low porosity and shrinkage.

d) The relatively low heat input of FSW can significantly reduce the distortion along the welded structure [103, 104], compared to arc welding.

e) A low level of gas entrapment together with no requirement for shield gases means that the chance of hydrogen embrittlement in the material is greatly reduced.

f) The near isotropic microstructure of the welded region may be desirable over a strong texture of the base material. In ODS steels, particularly problematic for tubes, the anisotropic microstructures and high grain aspect ratio can cause severely anisotropic mechanical properties and increased grain boundary sliding at elevated temperatures. However this is far less problematic in the fully recrystallized coarse grained state than the fine grained state [105-107].

g) FSW is also an automated process and therefore the quality of the weld is consistently reliable and has little reliance on the ability of an individual welder.

1.4.3. FSW of steel

Despite the joining of these steels being a hugely important step in their technological application, there is still relatively little literature available on the solid-state joining of ODS steels. This is in part due to the area of interest still being in its relative infancy and partly due to the limited availability of ODS steels, such as MA956, and of friction stir welding facilities of scientific standards. The friction stir welding of steels is also a costly process. Most friction stir welding is performed on light alloys such as aluminium or magnesium which have relatively low melting temperatures. The temperature required to plasticise steel is much greater. Therefore, toolheads for welding steel have to be made of ceramics or alloys containing significant levels of refractory metals which are vastly more expensive than the standard steel tools used on light alloys. The greater temperature and forces required during welding steel also mean that wear of the tool is much greater and distance the tool can weld is also considerably shorter.

Ceramic toolheads, such as used for the welds of this thesis, are also very brittle. This means that features or threads on the probe of the tool, designed to enhance flow around the material, can break easily and are very difficult to machine in the first place. Therefore
ceramic tools tend to have featureless pins or limited threading, which can mean that the efficiency of mixing in FSW of steel can be reduced, potentially causing an increased chance of defects.

1.4.4. Recrystallization due to FSW

The FSW process imparts heavy deformation on the welded material along with significant heating, typically producing temperatures 70-90% of the material’s melting temperature [108]. Therefore FSW can be considered a hot deformation process. These conditions can cause the accumulation of sufficiently high local differences in dislocation density that new grains can nucleate during deformation. This is known as dynamic recrystallization [109]. There are 3 mechanisms of dynamic recrystallization by which the microstructure is likely to evolve during FSW [110]:

i. Discontinuous dynamic recrystallization (DDRX): DDRX occurs by the nucleation of growth of new grains. This process generally occurs in metals and alloys with low to moderate stacking fault energy (SFE) during hot deformation [111]. The process is illustrated, below, in Fig. 14. DDRX normally occurs at the old grain boundaries when a critical strain or deformation is reached, which causes new grains to be nucleated Fig. 14(a)-(b). New grains then nucleate at the boundaries of the new, growing grains Fig. 14(c)-(d). The critical strain required decreases with an increase in temperature or decrease in strain rate or grain size [112].

Fig. 14. Schematic diagram representing the microstructural evolution occurring during DDRX. The dotted lines represent the prior grain boundaries [111].
ii. Continuous dynamic recrystallization (CDRX): In CDRX the dislocation density is reduced by continuous evolution of the size and misorientation of the sub-grain structure [113]. The strain from progressive rotation of the subgrains leads to the development of new high-angle grain boundaries, illustrated in Fig. 15. This process generally occurs in metals and alloys with a high SFE and in which dislocation motion is hindered by lack of slip systems or by solute drag since these conditions lead to increased inhomogeneous plastic deformation and hence increased dynamic recovery at the initial grain boundaries [110, 114].

![Figure 15](image)

**Fig. 15.** Schematic diagram representing recrystallization by progressive lattice rotation [111].

iii. Geometric dynamic recrystallization (GDRX): GDRX occurs when hot deformation causes the grains of a metal or alloy to elongate [115-117], see Fig. 16(b). The prior grain boundaries become closer together as the grains flatten and also become increasingly serrated as they respond locally to the interfacial tensions due to the sub-grain boundaries. The serrations of the prior grain boundaries eventually have a wavelength of the order of the sub-grain boundaries and can begin to impinge on other prior grain boundaries, creating new homogenously distributed, fine and equiaxed grains [118], see Fig. 16(c).
All of these mechanisms have been reported in the literature for friction stir welded alloys: CDRX [120-124], DDRX [125-127] and GDRX [117-118]. The alloy material and stacking fault energies are generally good indicators of the type of recrystallization to be expected during FSW, but by no means a safe bet. Microstructure, temperature, strain and strain rate may all affect the recrystallization mechanism.

All 3 types have been observed in the friction stir welding of Al alloys. Sabooni et al. [128], welding AISI 304L austenitic stainless steel, observed CDRX in the initially ultra-fine grained condition and DDRX in the initially coarse grained condition. Etter et al. [129] observed CDRX in a pre-strained 5251 aluminium sheet, but GDRX for the same alloy in the annealed state. Some authors have reported that more than one recrystallization mechanism can be in competition in the SZ of the same weld [130-132].

For literature on the FSW of ferritic ODS alloys, the mechanism is most commonly referred to a simply “dynamic recrystallization”. What little work that has been carried out investigating the particular mechanism, by Fonda et al. [133-134] and Han et al. [135-136], has found that CDRX is the dominant mechanism, as would be expected due to the high stacking fault energy of bcc ferritic alloys. The dispersed particles also promote CDRX as they can pin the grain boundaries and continuously absorb dislocations, aiding low-angle boundaries to transform into high-angle boundaries and sub-grains to transform into grains without growing. However, Han et al. [136] also reported serrated grain boundaries implying GDRX was also a competing mechanism.

1.5. Abnormal grain growth

Chapter 5 investigates the microstructure ODS steel friction stir welds following AGG induced by PWHT. AGG can occur directly due to the friction stir welding process without the need for PWHT, though this generally occurs in lighter alloys and it is usually considered undesirable. AGG is also an important part of the processing route for many ODS alloys.
including MA956, as discussed briefly earlier in Section 1.3.3. Here I shall provide an introduction to grain growth and the AGG phenomena, to supplement the aforementioned Chapter.

1.5.1 Types of grain growth

There are two types of grain growths that can occur: normal and abnormal grain growth, otherwise referred to as primary and secondary recrystallization, respectively. The driving force for both is the reduction in grain boundary energy. Normal grain growth is a continuous process in which all the grains grow at approximately the same rate. After any given time of normal grain growth in a material, there will remain a relatively narrow range of grains sizes and shapes.

AGG is a discontinuous process, in which a small number of grains grow excessively in the matrix at a much greater rate compared to the rest of the in matrix, which may not be growing at all. The growth of the small number of grains leads to a bimodal or heterogeneous grain size distribution until the large grains impinge on each other, at which point normal grain growth resumes [111]. Since a small number of grains consume the surrounding grains in the matrix, AGG is able to produce much coarser grain structures than normal grain growth. Fig. 17 shows the comparative grain growth evolutions due to the two processes.

![Fig. 17. Representative grain structure evolutions with time, along with the associated grain size distributions for top: normal grain growth and bottom: abnormal grain growth [137].](image-url)
1.5.2 Factors affecting grain growth

There are four main factors which affect grain growth [111]:

i. Temperature: the kinetics of high angle boundary migration is strongly dependent on temperature. The driving force for grain growth is usually relatively small and growth usually only occurs at elevated temperatures.

ii. Solutes and particles: solutes and particles can pin grain boundaries increasing the required driving force necessary for grain boundary migration.

iii. Texture: a strongly textured material will contain many low angle boundaries. The low angle boundaries have a low energy and therefore there is a reduced driving force for grain growth.

iv. Specimen size: When the grain size of a material becomes greater than the thickness of the sheet specimen, the grains become curved in only one direction instead of two. This reduces the force and hence the grain growth rate.

1.5.2 Factors leading to AGG

It can be shown that in an ‘ideal grain assembly’, one which has no impurities and a constant grain boundary energy, that a particularly large grain will grow more slowly than the average grain and therefore it will eventually become part of the normal grain size distribution again [138-139]. Therefore AGG only occurs when normal grain growth is inhibited and nucleation is limited.

The conditions for AGG are therefore more likely to be prevalent in thin sheets where normal grain growth is inhibited by the free surfaces. AGG also occurs more prevalently in materials with a single strong texture due to the high number of low angle grain boundaries inhibiting normal grain growth. In these materials there will be a small number of grains with orientations significantly different to the majority of grains. These grains will have a higher energy and mobility [111] and therefore may be able to grow at the expense of the other matrix grains via AGG, usually during high temperature annealing.

The pinning force of second phase particles is an important parameter as to whether AGG, or any growth, will occur in a material and is particularly relevant for ODS alloys. Fig. 18, is a graph illustrating what type of growth is to be expected in a material given the mean grain diameter and the particle dispersion level. This graph assumes an ideal grain assembly [140]. $F_v$ is the particle volume fraction and $d$ is the mean particle diameter.
Fig. 18. Expected grain growth regimes in a material with an ideal grain assembly. Mean grain diameter against particle dispersion level [140].

If we consider an arbitrary constant grain diameter within the given range, we can see at low particle dispersion levels, normal grain growth occurs since the grain boundary mobility will be relatively high with limited pinning of the grain boundaries. With an increase in particle dispersion level, normal grain growth becomes inhibited. A small number of grain boundaries may be able to overcome the particle pinning and AGG occurs. With a further increase in the particle dispersion level, the pinning force on the grain boundaries is further increased. Here, normal grain growth is suppressed, but AGG can also not initiate due to the high pinning forces. A more detailed version of the previous graph is given in Fig. 19. $\bar{R}$ is the mean starting grain size and $R$ is the mean final grain size. Here the expected growth regimes are:

- $\Psi = 0$: Normal grain growth possible
- $0 < \Psi < 0.1$: Broadening of grain size distribution
- $0.1 < \Psi < 0.25$: Abnormal growth and normal grain growth
- $0.25 < \Psi < 1$: Abnormal growth but no normal grain growth
- $\Psi > 1$: No growth possible
Fig. 19. Grain growth regimes in a material with an ideal grain assembly. Mean grain diameter against particle dispersion level [111].

1.6. Residual stresses

1.6.1. The significance of residual stresses

Residual stresses can arise from all manufacturing processes. Often these stresses are not life-limiting and so do not need to be considered. However, it is well known that residual stresses can significantly impact the strength, fatigue behaviour and corrosion resistance along with introducing distortion to a component [141-143]. Residual stresses are often introduced deliberately to produce improved material properties, as in the case of shot peening, but residual stresses can be extremely detrimental to a component’s performance and can often be the cause of premature failure. Therefore it is vitally important to consider the residual stresses remaining after manufacturing and processing before a component goes into service.

The heat and plastic deformation resulting from FSW can certainly leave significant residual stresses into ODS steels [144-145] and any resulting decrease in strength, fatigue or corrosion resistance may potentially produce catastrophic consequences. Chapter 4 of this thesis will focus on determining the magnitude and distributions of the stresses that result from FSW.
1.6.2. Origins of residual stress

Residual stresses are self-equilibrating stresses that reside within a stationary solid body when no external forces are applied. They arise when different parts, regions or phases of a component have a permanent shape misfit, known as an eigenstrain, caused by inhomogeneous inelastic deformation. These eigenstrains can be caused by temperature gradients, plastic deformation, different phases or phase changes, or magnetorestriction.

It is often useful to categorize residual stresses by the length scale over which the self-equilibrate, although there are other possible methods to categorize the stresses. Residual stresses must balance out to 0 within a material, since they are self-equilibrating. They therefore obey the following relation,

\[ \int_V \sigma_{ij} dV = 0 \]  

(13)

where \( V \) is the sample volume and \( \sigma \) is the stress \( (i,j = x,y,z) \). If the length scale of the volume needed for the stresses to integrate 0 is large, then the residual stresses are known as macrostresses (Type I). Large here represents a significant fraction of the total sample length and the stresses are considered to be continuous over grain and phase boundaries. Macrostresses would typically introduced by thermal gradients and plastic deformation.

When the stresses equilibrate over a small length, so that the stresses cannot be thought of as continuous over grain or phase boundaries, then they are known as microstresses. Microstresses are usually divided into Type II and Type III. Type II stresses equilibrate over length scales equivalent to grain size. They are caused by ill-fitting grains generally caused by a phase change or by different thermal expansion of grains of different phases, though they can be present in single phase materials due to anisotropic behaviour of grains. Type III stresses act over an even smaller, sub-grain, length scale. Type III stresses are caused by crystalline defects such as dislocations, vacancies and solute atoms [142, 146].

1.6.3. Measuring residual stresses by neutron diffraction

There are many established techniques for measuring residual stresses which all have their various advantages and disadvantages. For this study, it was chosen to use neutron diffraction to measure the residual stresses left over from FSW. We were granted beamtime at the Institut Laue–Langevin (ILL) to use the SALSA strain imager. Although the method used is discussed in further detail in Chapter 4, here I shall discuss the basic concepts of neutron diffraction. As with X-rays in XRD, neutrons are also able to diffract when passing
through a crystalline material since they travel as both a wave and a particle. This diffraction obeys Bragg’s law, see Eq. 14, which states that constructive interference will be observed at certain scattering angles, $\theta$, in a lattice with atomic spacing length $d$, using wavelength $\lambda$, where $n$ is an integer.

$$n\lambda = 2d\sin\theta$$  \hspace{1cm} (14)

Fig. 20 helps show how Eq. 14 arises. The path length difference of coherent beams scattered by two adjacent atoms in adjacent lattice planes is equal to $2d\sin\theta$ (or $n\cdot2d\sin\theta$ for an atom $n$ lattice planes away). When this path length difference is equal to a whole number, $n$, of wavelengths then the two waves will be in phase and will interfere constructively to create a greatly amplified signal. Therefore at certain angles diffraction creates waves with greatly increased amplitude by what is known as coherent scattering; while at all intermediary angles only relatively smaller amplitude signals made up from all of the incoherent scattering events that so happen to be scattered at that angle will be detected.

Since Bragg’s law is dependent on the lattice spacing of the crystalline material, diffraction can be used to determine residual stresses by using the lattice itself as a strain gauge. The increased or decreased lattice spacing in a stressed lattice means that constructive interference will occur at a different angle compared to an unstressed lattice, see Fig. 21 which shows the diffraction beam position of the unstrained lattice, solid blue line, and strained lattice, dotted blue line. The peak shift in this simple example represents a uniform strain; a non-uniform strain would be accompanied by a broadening of the peak.
\[ \varepsilon = \frac{d-d_0}{d_0} \]

\[ \sigma = E\varepsilon \]

Once the scattering angles, and hence lattice parameters have been determined, it is then possible to determine the strain in the stressed lattice using Eq. 15 which in turn can be converted to stress using Hooke’s Law, Eq. 16. Where \( d_0 \) is the lattice spacing of the unstressed lattice and \( d \) is the lattice spacing of the stressed lattice in Eq. 15 and \( E \) is the Young’s modulus of the material in Eq. 16. However, this only considers the strain in one sample direction. For a true residual stress, the strains must be measured in all 3 directions. It is possible to derive a generalised Hooke’s law that accounts for the strains in all 3 directions.

The generalised Hooke’s law, which can be used to determine the residual stress in a material, is given by Eq. 17, where \( \nu \) is the material’s Poisson’s ratio. \( i \) represents the chosen direction of interest: \( x, y \) or \( z \), where \( x, y \) and \( z \) represent the longitudinal, transverse and normal directions, respectively. The derivation of Eq. 17 can be found in [142] and [149].

\[ \sigma_i = \frac{E}{1+\nu} \left[ \varepsilon_i + \frac{\nu}{1-2\nu} (\varepsilon_x + \varepsilon_y + \varepsilon_z) \right] \]

1.6.4. Advantages of using neutrons

The neutron diffraction technique has a number of very significant advantages, compared to other methods, which have led to increasing use of this method for characterising residual stresses:
• Large penetration depth. Neutrons have a larger penetration depth (> cm’s) and so it is possible to measure the bulk properties of large components. X-rays have a much smaller penetration depth. Neutrons have no charge and a small magnetic moment which means that nuclear scattering of neutrons by nuclei is rare, although there is significant magnetic scattering of neutrons in magnetically ordered materials below the Curie temperature. Photons regularly interact with electrons via Compton scattering and the photoelectric effect. Therefore the rate of attenuation is much faster for X-rays than neutrons in metals. The rate of attenuation increases for the metals with increasing proton number since the density of electrons scales with the proton number. For iron, Z = 26, and steels the penetration depth is typically < 100 µm. Therefore it is only possible to measure stresses close to the surface.

• Non-destructive technique. Some methods for measuring residual stresses destroy the specimen upon measurement. A non-destructive technique is far more suitable if the component is to be put into service following the measurement, or if the specimen is very expensive or in short supply, such as with ODS steels.

• Adjustable spatial resolution.

• Can measure bulk macroscopic stresses and microstresses.

• Indifferent to surface finish.

• Triaxial measurement. Other methods of measuring residual stress, e.g. the contour method, are only to perform biaxial strain measurements. Though this can sometimes provide an acceptable estimation of the residual stress, often all three directions are of interest and it requires all three axes to measure the true residual stress.

• It is possible to produce residual stress distributions in 2D and 3D.

However, there are downsides to using neutrons. Perhaps most importantly is that using neutrons requires specialist facilities and it is very expensive. Therefore it is often not justifiable to use neutrons if time or money is an issue, which they often are. Neutron diffraction also requires relatively long measurement times and the accuracy can be affected by the grain size and texture.

1.6.5. Residual stresses in friction stir welds

FSW can leave residing stresses in components due to the severe heat and plastic deformation imparted by the process. However, the frictional heat from the tool shoulder has been shown to be the dominant source of the residual stresses in steels and other alloys.
As with fusion welding, the heat introduced by the welding causes the lattice to expand. As the heat input is concentrated along and close to the joint line, strong thermal gradients arise in the surrounding material. Material just ahead of the weld expands due to the intense heating. This region of expanding material is constrained by the surrounding cold material and compressive stresses evolve in the hot material. As the weld tool moves forward, the material behind the tool cools and begins to contract which, since it is also restrained by the surrounding cold material, causes a region of tensile stresses behind the tool. The material surrounding this tensile region must compensate so that the total stresses in the welded sample equilibrate to zero, following Eq. 13, and a region of compressive stresses, generally much smaller in magnitude to the tensile stresses is created either side of the weld. The cooling rates experienced during welding are usually sufficiently fast that the stress gradients do not even out and the stresses become locked in, residing in the material after the welding process has been completed [104, 142].

![Graph of residual stress distribution measured in an MA956 ODS steel plate as measured by X-ray diffraction](image)

**Fig. 22.** Graph of residual stress distribution measured in an MA956 ODS steel plate as measured by X-ray diffraction [145].

Fig. 22, above, shows a fairly typical distribution of longitudinal residual stresses of a plate following FSW, notably with the larger tensile stresses close to the weld line and smaller compressive stresses away from the TMAZ, on either side.

Relatively little work has been done on the residual stresses of friction stir welded steel. This is mainly because FSW is predominantly used on light alloys. However the process is essentially the same and a similar distribution of stresses are seen in steels as in light alloys. However due to the higher temperatures required to stir steel and the lower thermal
conductivity of steel compared to typical light alloys, the magnitudes of residual stress in steel can often be much greater. Very little literature appears to be available on this topic for ODS steels but some work has been done on PM2000 [144], and MA956 [145].

Much more work needs to be done investigating the residual stresses of FSW ODS steel. The residual stresses can be significantly changed by many factors such as plate thickness, welding parameters, cooling rate etc. Brewer et al. [145] did observe a trend with the magnitude of residual stress with heat input but still much more investigation is required. The effects of welding parameters will be discussed at greater length in the discussion of Chapter 4. All of the variables need to be soundly understood before the nuclear industry can have confidence of welding reactor components with acceptable levels of residual stress.

References


[95] (Serrano, M., personal communication, April, 2013)


Preface to Chapter 2

Chapter 2, entitled “Impact of friction stir welding on the microstructure of ODS steel”, has been published as a research paper in the Journal of Nuclear Materials, see bottom of page. The Chapter focuses on assessing the microstructure of MA956 ODS steel before and after friction stir welding and the effect of changing the welding parameters. The microstructural analysis utilised a combination of optical and scanning electron microscopy techniques to quantify the grain structures and grain size distributions of the base material and of the welds. Transmission electron microscopy and Small Angle Neutron Scattering (SANS) was used to observe and quantify changes to the nano-particle distributions.

As in all of the Chapters throughout the thesis, the ODS steel plates were provided by CIEMAT and the friction stir welding was carried out by H. Dawson and S. Cater at TWI Technology Centre, Yorkshire. TWI also provided the tool head used for all welds. In this Chapter the welds were carried out in the form of bead-on-plate welds i.e. the process was carried out on a single plate rather than joining two plates together.

All optical and electron microscopy and micro-hardness was carried out by H. Dawson, either at the University of Manchester or the affiliated Dalton Cumbrian Facility (DCF). The SANS beamline experiment was carried out at the Budapest Neutron Centre (BNC) in Hungary by H. Dawson, Q. Tian and L. Almásy. Q. Tian and L. Almásy also performed the data reduction on the SANS data.

Reference:


DOI: https://doi.org/10.1016/j.jnucmat.2016.12.033
Chapter 2

2. Impact of friction stir welding on the microstructure of ODS steel

H. Dawson\textsuperscript{a,}\textsuperscript{*}, M. Serrano\textsuperscript{b}, S. Cater\textsuperscript{c}, N. Iqbal\textsuperscript{c}, L. Almásy\textsuperscript{d,}\textsuperscript{e}, Q. Tian\textsuperscript{e}, E. Jimenez-Melero\textsuperscript{a,}\textsuperscript{f}

\textsuperscript{a}School of Materials, The University of Manchester, Manchester M13 9PL, UK
\textsuperscript{b}Structural Materials Division, Technology Department, CIEMAT, Avda de la Complutense 40, 28040 Madrid, Spain
\textsuperscript{c}Friction and Forge Processes Department, Joining Technologies Group, TWI Technology Centre, Advanced Manufacturing Park, Wallis Way, Catcliffe, Rotherham S60 5TZ, UK
\textsuperscript{d}State Key Laboratory Cultivation Base for Nonmetal Composites and Functional Materials, Southwest University of Science and Technology, Mianyang 621010, China
\textsuperscript{e}Wigner Research Centre for Physics, Institute for Solid State Physics and Optics, P.O. Box 49, H-1525 Budapest, Hungary
\textsuperscript{f}Dalton Cumbrian Facility, The University of Manchester, Moor Row CA24 3HA, UK
Abstract

We have assessed the impact of the welding parameters on the nano-sized oxide dispersion and the grain size in the matrix of an ODS steel after friction stir welding. Our results, based on combined small angle neutron scattering and electron microscopy, reveal a decrease in the volume fraction of the particles smaller than 80 nm in the welds, mainly due to particle agglomeration. The increase in tool rotation speed or decrease in transverse speed leads to a higher reduction in nano-sized particle fraction, and additionally to the occurrence of particle melting. The dependence of the average grain size in the matrix on the particle volume fraction follows a Zener pinning-type relationship. This result points to the principal role that the particles have in pinning grain boundary movement, and consequently in controlling the grain size during welding.
2.1. Introduction

Oxide Dispersion-Strengthened (ODS) steels are currently considered as leading candidate materials for the production of thin-wall cladding tubes for Gen IV fast nuclear reactors, and also for first-wall components in magnetically-confined fusion reactors [1-3]. Their attractiveness as structural materials for future nuclear energy systems lies in their enhanced high-temperature strength and creep resistance, which allows to extend the high-temperature safe limit of reactor operation beyond the value of ~600 °C characteristic of ferritic/martensitic steels [4], coupled to their high resistance to the expected neutron doses potentially beyond 100 dpa [5,6]. ODS steels are Fe-Cr based materials that contain a high density of yttrium-based oxide particles, of only a few nm in size, embedded in the ferritic matrix [7,8]. These particles act as effective barriers for the movement of dislocations, whereas the particle/matrix interface constitutes an effective sink for interstitial atoms (e.g. H, He) and lattice defects induced by radiation [9,10].

These materials are produced by mechanically alloying (MA) a mixture of high-purity metal powders along with Y₂O₃ fine particles. MA is able to produce a near homogenous distribution of alloying elements and dispersion of nanoparticles [11-13]. The powders are then consolidated by either hot isostatic pressing or hot extrusion, followed by a series of cold and/or hot rolling [14-16]. However, full reactor components cannot be directly produced from MA material, therefore a successful method for ODS steel joining is required. Fusion joining techniques are generally unsuccessful since melting the alloy severely disrupts the nanoparticle dispersion to the extent that they are unlikely to remain fit for purpose in nuclear reactor. Therefore there has been a large interest in developing a solid-state technique to join ODS steels.

Friction Stir Welding (FSW) has in recent years has stood out as a promising solid-state welding solution for ODS steels. Several authors have been able to successfully join ODS steels by FSW while largely preserving the dispersion of nanoparticles [17]. During FSW a rotating tool is plunged in the workpiece and advances along a pre-defined direction while welding the two metallic plates together. This is a thermo-mechanical process that produces local temperatures between 70-90% of the melting point of the material [18,19]. The dispersed nano-scale particles present a high thermal stability during annealing up to ~1150 °C [20,21], with only a relatively small coarsening at prolonged times that is attributed to the uptake of Al by the yttrium oxide particles [22-24]. However, the severe plastic
deformation and complex material flow caused by the FSW rotating tool as it advances along the weld line, in combination with the relatively high heat input, can potentially cause significant changes in the local microstructure of the welded zone.

The heat input and local plasticity introduced during FSW of high-Cr ODS steels triggers a process of dynamic recrystallization within the stir zone, leaving a roughly homogeneous distribution of roughly equiaxed grains. This normally leads to a decrease in hardness and strength of the weld with respect to the base material, and to a potential strain localization phenomenon at the boundary between the stir zone and the heat-affected zone on the advancing side of the weld [25,26]. Additionally, a bcc torsional texture has been reported in the stir zone, with a predominant <111> fiber component that is characteristic for torsional deformation processes in ferritic steels [26-28]. However, the recrystallization taking place during FSW can be used to form equiaxed grains, and therefore suppress the material anisotropy introduced during prior rolling and enhance the creep resistance of the material [29].

The influence of the FSW process on the oxide particle distribution is not yet well established and even less work has been done to investigate the effect of changing welding parameters. The aim of this paper is to investigate the impact of selected weld tool traverse speeds and rotation speeds on the particle size distribution of ODS friction stir bead-on-plate welds, while using the same tool geometry, plunge force and starting ODS steel microstructure. We also aim to assess the effect of the particle evolution on the grain size, and therefore on the recrystallization phenomenon during FSW, in the ferritic matrix. The results of this study will hopefully provide guidelines for optimising the FSW process of ODS steels, minimising the detrimental effect on the particle size distribution and optimising grain size distribution in the matrix.

2.2. Experimental

2.2.1. Base Material

The chemical composition of the steel used in this study, namely MA956 ODS steel, is given in Table 1. This material is produced by Special Metals Corporation, UK, via mechanical alloying of the mixture of the corresponding metallic powders with Y₂O₃ particles, followed by a hot extrusion step at ~1000 °C. The consolidated material is then hot rolled in both the transverse and longitudinal directions using a 3-high reversing mill,
followed by a recrystallization annealing treatment at 1320 °C for 1h, and finally air cooling to room temperature [30-32].

<table>
<thead>
<tr>
<th>Cr</th>
<th>Al</th>
<th>Y₂O₃</th>
<th>P</th>
<th>Ti</th>
<th>O</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
<th>Ni</th>
<th>Co</th>
<th>N</th>
<th>Cu</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.97</td>
<td>4.44</td>
<td>0.53</td>
<td>0.33</td>
<td>0.21</td>
<td>0.15</td>
<td>0.11</td>
<td>0.05</td>
<td>&lt;0.05</td>
<td>0.04</td>
<td>0.03</td>
<td>0.022</td>
<td>0.009</td>
<td>0.004</td>
<td>bal.</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Chemical composition of the studied MA956 ODS steel (wt%).

2.2.2. Friction Stir Welding

Bead-on-plate welds were performed on nominally 5 mm thick plates, which were produced by slicing the original plate in half through its thickness. We used a polycrystalline cubic boron nitride tool with a 25 mm diameter shoulder and a 3 mm diameter pin. Five bead-on-plate welds were performed using a 25 kN plunge force in the presence of an argon shielding gas atmosphere. Each weld corresponded to a selected combination of tool rotation and transverse speeds, see Table 2. In order to evaluate the variations in microstructure across the welds, and therefore identify representative locations for further structural characterization, we performed Vickers micro-hardness line scans on the weld cross sections, using a Struers DuraScan automatic hardness tester and a 1 kgf (HV₁₀). The indentations were made perpendicular to the welding direction, at approximately 2 mm from the top surface of the workpiece.

<table>
<thead>
<tr>
<th>Weld No.</th>
<th>Rotations per Minute (RPM)</th>
<th>Traverse Speed (mm/min)</th>
<th>Mean Grain Diameter (µm)</th>
<th>Relative Volume Fraction</th>
<th>Mean Diameter (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1</td>
<td>10.8(1)</td>
</tr>
<tr>
<td>1</td>
<td>200</td>
<td>140</td>
<td>2.49(5)</td>
<td>0.64</td>
<td>16.0(1)</td>
</tr>
<tr>
<td>2</td>
<td>200</td>
<td>120</td>
<td>2.58(6)</td>
<td>0.53</td>
<td>18.8(1)</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
<td>160</td>
<td>2.05(3)</td>
<td>0.77</td>
<td>10.8(1)</td>
</tr>
<tr>
<td>4</td>
<td>220</td>
<td>140</td>
<td>2.56(5)</td>
<td>0.58</td>
<td>17.2(1)</td>
</tr>
<tr>
<td>5</td>
<td>180</td>
<td>140</td>
<td>1.93(4)</td>
<td>0.83</td>
<td>11.4(1)</td>
</tr>
</tbody>
</table>

Table 2. Welding parameters and characteristic parameters of the welded microstructures: mean grain diameter of the ferritic matrix, and relative volume fraction and mean diameter of the oxide nano-particles. ‘BM’ denotes the base material.

2.2.3. Structural characterization

2.2.3.1. Optical and electron microscopy

The specimens were mechanically ground and polished down to 1 µm with diamond suspension, and further polished with 0.025 µm silica (OP-S) suspension. The specimens prepared for optical microscopy were finally etched with a solution containing 15 vol. % HCl and 3 vol. % HNO₃. Optical micrographs of the weld cross sections were taken using a
Keyence VK-X200K 3D laser scanning microscope, whereas micrographs of the particles present at the weld border were taken on a Struers DuraScan automatic hardness tester. The grain size distribution in the ferritic matrix and the few micrometre-sized oxide particles were probed by combining (1) Electron Backscatter Diffraction (EBSD) maps collected on a FEI Quanta 250 FEG-SEM, using an accelerating voltage of 20 kV and a scanning step size of 0.8 µm, (2) and Backscattered Electron (BSE) images taken on an FEI Magellan High Resolution FEG-SEM. Furthermore, the characteristics of the nanometre-sized particles were studied by Transmission Electron Microscopy (TEM) using an FEI Tecnai 20 200 kV Analytical Electron Microscope. For this purpose, 3 mm-thin foil samples were taken from both the base material and the centre of the transverse cross section of the weld. The electropolishing was performed using an electrolyte consisting of 95 vol. % methanol and 5 vol. % perchloric acid at a temperature of −40 ºC. Particle size distributions were derived from the TEM data using the ImageJ software package [33].

2.2.3.2 Small-angle neutron scattering

The nanometre-sized particle distribution in each of the welds and the base material was derived from the Small Angle Neutron Scattering (SANS) data collected on the Yellow Submarine instrument, which is installed at the cold neutron beamline of the 10 MW nuclear reactor of the Budapest Neutron Centre (BNC), Hungary. A monochromatic neutron beam with a mean wavelength of 8.1 Å was produced using a multidisk mechanical velocity selector. The SANS measurements were performed at room temperature, and under a magnetic field of 1.5 T in order to magnetically saturate the ferritic matrix in the ODS steel samples. The neutron scattering signal was recorded using a BF3 gas-filled multi-wire detector with a 64 × 64 cm² active area. This detector was placed at two distances behind the sample, namely 1.1 and 5.2 m, in order to cover a total q-range of 0.008-0.3 Å⁻¹.

2.3. Results

2.3.1. Base material

Fig. 1 shows the microstructure of the base material in the representative TD/LD and TD/ND planes, where TD, LD and ND denote the Transverse, Longitudinal and Normal Directions of the plate. At the surface, the optical micrographs reveal the presence of relatively large “pancake” shaped grains in the TD/LD plane, with their shortest dimension lying along the normal direction. These grains are created by abnormal grain growth during the annealing treatment at 1320 ºC after rolling. However, close to the centre line of the plate
the microstructure is characterised by predominantly fine grains, 1-2 µm slightly elongated in LD (Fig. 1c). The transition between the near-surface coarse grained structure and the fine grained structure inside the plate is characterised by a boundary region where both fine and coarse grains are present. This implies that the abnormal grain growth in the material is not complete, and the large central section of the plate retains its fine grained structure. The micrographs in Fig. 1 also reveal the presence of a number of particles distributed through the plate, typically 100 nm to 2 µm in diameter, together with a high density of nano-sized particles.

Fig. 1. (a)-(b) Optical image along the LD/TD and ND/TD planes respectively; (c)-(e) BSE images taken at the representative locations of the plate thickness denoted in (b); and (f) TEM image of the as-received plate material of MA956 ODS steel.
2.3.2. Friction stir welding

The friction stir welding process was performed with the tool being plunged with the fine grain structure at the top of the workpiece, after having sliced the original 10 mm plate in half through its thickness. Fig. 2 shows the optical micrograph of the cross section of one of the welds, together with the variation in hardness across the weld at approximately 2 mm from the upper surface of the workpiece. The darker central region in the optical micrograph of Fig. 2a corresponds to the zone where the tool has stirred the material, namely the stir zone (SZ), which presents a ~10-15% decrease in hardness as compared to the base material (BM). The region of ~4 mm in width on either side of the SZ corresponds to the heat-affected zone (HAZ). There is a noticeable asymmetry in the hardness profile across the weld. The transition between the SZ and the HAZ on the advancing side of the weld is relatively sharp, while the variation in hardness is more gradual in the retreating side. Moreover, we have also observed a build-up of micrometre-sized particles in a 30-50 μm thick region located at the boundary between the SZ and the HAZ. Defects, such as cavities and tunnelling voids, are visible towards the base of the SZ. Similar defects were observed in all of the different welds. Generally they were of similar size and location, though they were occasionally also observed on the retreating edge of the weld, close to the TMAZ-HAZ boundary.

![Optical micrograph of the cross section of a friction stir weld](image)

**Fig. 2.** (a) Optical image of the cross section of a representative bead-on-plate ODS steel weld; (b) micro-hardness measurements taken at approx. 2 mm from the surface of the weld, as denoted by the dashed line in (a).
2.3.3. Grain size distribution

Fig. 3 shows a representative example of an EBSD map of the stir zone, together with the grain size distribution and mean grain diameter in the matrix as a function of welding parameters. There is a weak texture at the centre of the weld for all the studied combinations of welding parameters. The grains in the matrix are elongated in the direction of the material’s flow around the rotating tool, and they have an aspect ratio of ~2. All of the welds present a similar grain size distribution. However, we have observed systematic changes in the mean grain diameter with the welding parameters: the mean diameter increased with increasing rotation speed and/or decreasing transverse speed, i.e. with increasing heat input during welding.

![EBSD map](image)

![Grain size distribution](image)

![Mean grain diameter](image)

**Fig. 3.** (a) EBSD map of the SZ of Weld 4; (b) grain size distribution of the matrix of selected welds; (c) variation of the mean grain diameter with the tool rotation speed and transverse speed.
2.3.4. Nano-particle size distribution

The changes in particle size distribution due to FSW were detected by measuring the SANS signal of samples taken from the base material and the welds, supported by TEM observations. The studied ODS steel can be considered as a dispersion of non-magnetic spherical (oxide) particles in a ferromagnetic (ferrite) matrix. For non-magnetic scatterers the magnetic contrast is equal to the square of the magnetic scattering length density of the ferromagnetic matrix. The scattering intensity of samples in a magnetic field is in general composed of two contributions:

\[ I = I_{\text{nuc}} + I_{\text{mag}} \cdot (\sin(\alpha))^2 \]  

(1)

where \( I_{\text{nuc}} \) and \( I_{\text{mag}} \) are the nuclear and magnetic scattering intensities respectively, and \( \alpha \) is the angle between the magnetic field direction and scattering vector \( q \). The magnetic scattering intensity can then be extracted from the intensity collected when the scattering vector is perpendicular (\( \alpha = 90^\circ \)) and parallel (\( \alpha = 0^\circ \)) to the applied magnetic field, see Fig. 4a, according to:

\[ I_{\text{mag}} = I(\alpha=90^\circ) - I(\alpha=0^\circ) \]  

(2)

The \( q \)-dependence of \( I_{\text{mag}} \) in a system of polydisperse particles can be expressed as [34,35]:

\[ I(q) = \Delta \rho^2 \int_0^\infty N(R)V(R)^2 F(qR)dR \]  

(3)

where \( \Delta \rho \) is the magnetic scattering length density of the matrix, \( R \) is the particle radius, \( V(R) \) is the particle volume with radius of \( R \), and \( F(qR) \) is the form factor of spherical particle that takes the form of:

\[ F(qR) = \frac{3[\sin(qR) - qR\cos(qR)]}{(qR)^3} \]  

(4)

We have analysed the intensity as a function of the scattering vector (Fig. 4b) using a lognormal particle size distribution:

\[ N(R) = \frac{N}{\sigma R \sqrt{2\pi}} \exp\left[-\frac{\ln(R/\mu)^2}{2\sigma^2}\right] \]  

(5)

where \( \mu \) is the median radius, \( N \) is the total number of particles and \( \sigma \) is the polydispersity parameter related to the width of the particle size distribution. The mean radius of nano-oxide particles is evaluated as \( \mu \exp(\sigma^2/2) \).
Fig. 4c shows the particle size distribution derived from the SANS data for the welds and the base material. There is a significant decrease in the volume fraction of the particles in the welds, in the size range probed by the SANS experiment. Fig. 4d shows the relationship between the welding parameters and the relative volume fraction of the nano-particles after welding. There is a clear decrease in the volume fraction of the particles with increasing rotation speed or decreasing traverse speed. Fig. 5 shows illustrative examples of the behaviour of the particles in the welds, together with the size distribution obtained from TEM. The size distributions obtained by SANS (Fig. 4c) and TEM (Fig. 5d) are in good agreement. All the welds show an increase in mean particle diameter with respect to the base material, see Table 2. The micrographs obtained by TEM show a clear evidence of particle agglomeration in the welds. In some cases, the particle clusters may even be of a few hundreds of nanometres in size (Fig. 5a), although they are normally below 100 nm diameter. Evidence of melting of the nanoparticles from TEM micrographs was relatively rare (Fig. 5c), and only observed in welds produced with the higher heat inputs.
Fig. 4. (a) SANS data collected from an ODS steel weld sample placed under an external magnetic field of 1.5 T; (b) intensity as a function of the scattering vector for the base material and selected welds; (c) oxide particle size distributions derived from the SANS data; (d) variation of the relative volume fraction of particles with the tool rotation speed and transverse speed.

2.4. Discussion

Our SANS results reveal a significant decrease in the measured volume fraction of the nano-particle dispersion in the welds as compared to the base material. There are three potential causes of this decrease: particle dissolution, agglomeration, or displacement away from the centre of the welds where the neutron beam was incident. As seen in Fig. 2a, there was a build-up of particles at the boarder of the TMAZ. At this scale only the larger sized particles, mostly titanium carbonitrides and aluminium oxides, were directly observed but it may be assumed that the nanoparticles would be similarly displaced away from the centre of the welds, as previously observed by Charit et al. [36].
Fig. 5. TEM images showing examples of (a)-(b) particle agglomeration and (c) melting; (d) particle size distribution obtained from the TEM images.

Fig. 5c shows that there has been at least some dissolution of the particles during FSW. As previously mentioned, it was however only observed for Welds 2 and 4 by TEM which were the two welds with the highest heat inputs due to having the lowest traverse speed and greatest rotational speed, respectively. The SANS data, Fig. 4c, shows that for Welds 3 and 5, with the lowest heat inputs, the smallest particles are largely unaffected and the mean diameter remains similar to that of the base material suggesting the temperature created by these parameters is low enough that the amount of dissolution is low or negligible. For the other welds there is a very low number of particles with diameters below 3 nm detected by SANS, suggesting that dissolution of the smallest particles has occurred. The $q$-range covered in the SANS experiment allowed the detection of particles or agglomerates with diameters up
to \( \sim 80 \) nm. Our TEM data showed that agglomerates could grow to at least this size limit, and occasionally reached several hundreds of nanometres. Particle agglomeration during FSW is likely to be the dominant cause for the reduced volume fraction of particles measured by SANS.

We have also detected a modest increase in the average particle size in all welds with respect to the base material, as have other studies on FSW of ODS steel [37-39]. In our study, the base material had been annealed at 1320 °C for 1h prior to FSW. During this annealing stage, most of the \( \text{Y}_2\text{O}_3 \) particles would have already transformed into yttrium aluminium perovskite (YAP), which is stable at temperatures lower than \( \sim 1150 \) °C. Thermocouple data on subsequent similar welds shows that the temperatures produced by these parameters did not exceed 1000 °C. Coarsening by pick-up of Al by the \( \text{Y}_2\text{O}_3 \) particles is unlikely to be a significant contributor to the increase in mean particle diameter. Consequently, both the reduction in relative fraction and the increase in average size of the particles, as measured by SANS and complementarily by TEM, are mainly due to particle dissolution and/or agglomeration during FSW. The measured increase in particle size was more pronounced for increased rotation speed and decreased traverse speed. Higher heat inputs will cause the dissolution of a greater number of the smallest nanoparticles and will produce a greater amount of stirring which in turn would likely increase the number of particles that agglomerate. Since levels of dissolution were relatively low, and negligible in the lower heat input welds, most of the nanoparticles in the stir zone of the welds will have been retained after FSW. However, many of the nanoparticles exist as part of an agglomerate, hence the number density of effective sites for pinning dislocations, trapping helium or acting as recombination sites for radiation-induced lattice defects will still be reduced.
Fig. 6. (a) Mean grain diameter of the matrix as a function of the relative volume fraction of the oxide particles. The red line corresponds to the estimated Zener pinning limiting grain size (see text); (b) experimental value of the mean grain diameter for each of the studied welds, together with the estimate from Eq. (6).

The average grain size in the matrix of the welds was found to increase with increasing tool rotation speed and/or decreasing traverse speed. One potential cause of the increased grain size is the direct effect of the heat input during welding. Strain and strain rate also have a role in determining grain size in the SZ, but this is usually not as significant as the temperature of the weld. The SZ grain structure coarsens from its initial sub-micron grain size, and becomes more equiaxed in the thermal wake of the tool in a static annealing process [40]. Another study on FSW of MA956 [38] showed that the grain size of friction stir welded MA956 is stable during post weld heat treatment at 1300 °C for one hour. Given that the temperatures achieved in this work during FSW remained lower than 1000 °C and that the exposure time during welding was below 1 min, the direct effect of the heat input on the
matrix is unlikely to be the sole determining factor for the final grain size in MA956 after FSW. The alternative is the effect of FSW on the oxide particle size distribution, since the finely dispersed nano-particles play an effective pinning role for the movement of grain boundaries in the matrix, including following FSW [41,42]. Fig. 6 shows the average grain size in the matrix as a function of the volume fraction of oxide particles. We have compared these experimental data with the Zener pinning limiting grain size, \( D_{\text{Zener}} \), given by [43]:

\[
D_{\text{Zener}} = \frac{4\bar{r}}{3f_v}
\]  

(6)

where \( \bar{r} \) is the average particle radius and \( f_v \) is the volume fraction of particles in the welds (Table 2). The volume fraction of particles in the base material is reported to be 0.62 ± 0.10% [34]. The data in Fig. 6 shows a relatively good correlation between the measured average grain size and the values derived using Eq. (6). This implies that the oxide dispersion strongly influences the grain size in the matrix, by acting as pinning particles for grain boundaries. The increase in particle size and decrease in volume fraction of particles with sizes below 80 nm, caused by the higher tool rotation speed and/or lower transverse speed, manifests in a higher interparticle spacing and a reduced role in retarding grain growth in the matrix during welding. Therefore, the observed decrease in hardness in the stir zone, as compared to the base material, is mainly due to (1) the increased average grain size with respect to the surrounding fine grained material, (2) the decrease in the number of particles in the size range below 80 nm, due to either particle agglomeration or, in the case of higher heat input during welding, due to particle melting or displacement to the boundary between the stir zone and the heat affected zone, and (3) a low dislocation density in the stir zone following dynamic recrystallization.

The presence of weld defects in the welds suggests that there was insufficient material flow during FSW to create fully consolidated welds. The relative motion of the tool means that the level of mixing is more inadequate on the retreating side of the weld, which is why weld defects were observed along the retreating side TMAZ boundary. It is very likely that all of the welds were too cold to sufficiently plasticise the material.

The insufficient material flow is likely to be the cause of the high levels of particle agglomeration observed. The low viscosity would cause the material to be subject to intense plastic deformation and large strain gradients to which agglomeration of dispersoids has been attributed previously by other authors in FSW [44]. Other studies on FSW of ODS steels,
using higher heat inputs have been able to produce defect free welds while keeping the near homogeneous distribution of particles in the SZ [29,41]. The results suggest that a compromise of welding parameters may have to be met to maximise the effectiveness of the nanoparticles distribution of ODS steels after FSW. Welding with too low a heat input leaves the weld vulnerable to defects and agglomeration of the particles. However, using a higher heat input can cause dissolution of the particles, which has been shown in some cases to severely alter the mean diameter and volume fraction of particles deleteriously.

2.5. Conclusions

We have performed a systematic study of the impact of the FSW parameters on both the nano-oxide particle distribution and the average grain size in the ferritic matrix of MA956 ODS steel. The main conclusions of this work are: (1) the relative volume fraction of nano-particles in the range of 2-80 nm, as measured by SANS, decreases in the stir zone with respect to the base material. This effect is more pronounced in the case of higher tool rotation speed and/or lower transverse speed, i.e. higher heat input during welding; (2) in all studied welds, the particles form agglomerates that can in some cases reach sizes above 100 nm. When the heat input during welding is relatively high, we have also observed particle dissolution; (3) the stir zone of all the welds consists of a largely homogenous grain structure with a nearly random texture. The average grain size in the matrix increases with decreasing volume fraction of particles with sizes below 80 nm; (4) the relationship between both structural parameters follows a Zener pinning-type relationship. This implies an effective pinning effect of the particles on the movement of grain boundaries in the matrix during welding. The majority of the oxide particles are retained after FSW, but their grain boundary pinning effect is reduced when they are forming relatively large agglomerates.

Acknowledgements

We gratefully acknowledge the financial support of the Engineering and Physical Sciences Research Council UK (EPSRC) through the Centre for Doctoral Training in Advanced Metallic Systems under Grant Agreement EP/L016273/1. We would like to thank CIEMAT in Spain for providing the base material, MA956 ODS steel, and to The Welding Institute for providing the welding tools and assistance during friction stir welding. We also thank the Budapest Neutron Centre for the granted beam time and the European Commission under the 7th Framework Programme through the Key Action: Strengthening the European
Research Area, Research Infrastructures under Grant Agreement no. 283883-NMI3-II, for support to perform the SANS experiment.

References
Preface to Chapter 3

Chapter 3, entitled “Mechanical properties and fracture behaviour of ODS steel friction stir welds at variable temperatures”, has been published as a research paper in the Journal of Material Science and Engineering A, see bottom of page. The Chapter focuses on assessing the mechanical properties, namely tensile strength and micro-hardness, of the base material and the welds at a range of test temperatures. The observed mechanical properties are interpreted with the information on the microstructures, assessed in the previous Chapter, and with micrographs of the fracture surfaces of the tensile specimens.

The ODS steel plates were provided by CIEMAT. The bead-on-plate friction stir welds were carried out at by H. Dawson and S. Cater at TWI Technology Centre, Yorkshire.

Micro-hardness testing was carried out by H. Dawson at the Dalton Cumbrian Facility (DCF). Tensile testing was carried out at CIEMAT, Spain, by the technical staff with the assistance of H. Dawson and M. Serrano.

Scanning electron micrographs of the tensile fracture surfaces were obtained at CIEMAT by R. Hernandez. The optical macrographs of the fractured tensile specimen were taken by H. Dawson. All other optical and electron microscopy of the welds was captured by H. Dawson at the University of Manchester.

Reference:


DOI: https://doi.org/10.1016/j.msea.2017.03.090
Chapter 3

3. Mechanical properties and fracture behaviour of ODS steel friction stir welds at variable temperatures

H. Dawson\textsuperscript{a,}, M. Serrano\textsuperscript{b}, R. Hernandez\textsuperscript{b}, S. Cater\textsuperscript{c}, E. Jimenez-Melero\textsuperscript{a}

\textsuperscript{a}School of Materials, University of Manchester, Manchester M13 9PL, United Kingdom

\textsuperscript{b}Structural Materials Division, Technology Department, CIEMAT, Avda de la Complutense

40, 28040 Madrid, Spain

\textsuperscript{c}Friction and Forge Processes Department, Joining Technologies Group, TWI Technology

Centre (Yorkshire), Advanced Manufacturing Park, Wallis Way, Catcliffe,

Rotherham S60 5TZ, United Kingdom
Abstract

We have assessed the microstructure and the temperature-dependent mechanical behaviour of five bead-on-plate friction stir welds of Oxide Dispersion-Strengthened (ODS) steel, produced using systematic changes to the tool rotation and traverse speed. Friction stir welding can potentially retain the fine dispersion of nanoparticles, and therefore also the high-temperature strength and radiation damage resistance of these materials. Tensile testing was carried out on the MA956 base material at a range of temperatures, from room temperature up to 750 °C. The mechanical properties of the welds were investigated via tensile testing at room temperature and at 500 °C, together with micro-hardness testing. The welds exhibited similar strength and ductility to the base material at both testing temperatures as welding caused a partial loss of particle strengthening, alongside an increase in grain boundary strengthening due to a greatly refined grain size in the stir zones. The microhardness data revealed a trend of increasing hardness with increasing tool traverse speed or decreasing rotation speed. This was attributed to the smaller grain size and lower nanoparticle number density in the welds created with these parameters. At 500 °C, the yield stress and ultimate tensile stress of the base material and the welds decreased, due to a progressive reduction in both the Orowan-type particle strengthening and the grain boundary strengthening.
3.1. Introduction

Friction Stir Welding (FSW) is currently being investigated as a suitable method of joining the difficult-to-weld Oxide Dispersion-Strengthened (ODS) steels, for use as structural materials in future generation nuclear fission and fusion reactors [1–3]. The ODS steel microstructure comprises in general of a fine homogeneous dispersion of Y(Al,Ti) oxide nano-particles embedded in an Fe-Cr ferritic matrix [4,5]. The nanoparticles strengthen the material to high temperatures by hindering dislocation and grain boundary motion through the matrix. Moreover, the nano-particle/matrix interfaces act as trapping sites for transmutant helium, and also as recombination sites for radiation-induced vacancies and self-interstitials, and therefore help to improve the damage resistance of these materials to high radiation doses [6,7]. As a solid-state technique, FSW can potentially join ODS steels with a minimal level of disruption to the nanoparticle dispersion in the matrix since no melting should occur. During FSW, the peak temperatures do not normally exceed 70–90% of the melting point of the material to be welded [8].

Recently several authors have reported successfully joined ODS steels via FSW [9–12]. However, the particle dispersions in the welded region of the specimen still tend to have deleterious agglomeration, dissolution or coarsening. This is usually accompanied by other significant changes to the microstructure such as grain coarsening, development of bcc torsional texture or changes to the dislocation density, due to the dynamic recrystallization process induced by FSW. These microstructural changes normally soften the welded areas with respect to the base material and, in the harsh nuclear environments to which ODS steels will be subject, even subtle microstructural changes may have critical implications for the weld's structural integrity and therefore for the safe operation of the nuclear power plant.

In order for Friction Stir Welded ODS steel to be approved for use in nuclear reactor environments, it is of paramount importance that the mechanical properties of the welded regions, along with the base material, be comprehensively examined, including the material's behaviour at elevated temperatures. This should be linked to the impact of key welding parameters and the microstructural properties of the welds, so that the grain structure and mechanical performance of the friction stir welds can be as close as possible, if not preferable, to the base material. Although some limited research has in recent years been conducted on the mechanical properties of Friction Stir Welded ODS steels [9–11], including the MA956 steel which is the subject of this study, the data is still highly fragmented. This is partly due to the fact that research into FSW of steels is still in its relative infancy [13,14],
and partly to the fact that many ODS steel grades proposed for use in nuclear reactors [15] are currently in limited supply. It is therefore difficult to obtain systematic experimental data on a relatively large range of welding parameters and testing temperatures, so that the process parameter windows can be selected for optimal heat input and flow of the plasticised material during welding. This paper analyses the mechanical properties of five welds produced using systematic variations in tool rotation and traverse speeds, combining tensile testing at variable temperatures, micro-hardness mapping and electron microscopy, and aims to link these to the microstructural characteristics of the welds.

3.2. Experimental

3.2.1. Friction stir welding

<table>
<thead>
<tr>
<th>Cr</th>
<th>Al</th>
<th>Y₂O₃</th>
<th>P</th>
<th>Ti</th>
<th>O</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
<th>Ni</th>
<th>Co</th>
<th>N</th>
<th>Cu</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.97</td>
<td>4.44</td>
<td>0.53</td>
<td>0.33</td>
<td>0.21</td>
<td>0.15</td>
<td>0.11</td>
<td>0.05</td>
<td>&lt;0.05</td>
<td>0.04</td>
<td>0.03</td>
<td>0.022</td>
<td>0.009</td>
<td>0.004</td>
<td>bal.</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Chemical composition of the studied MA956 ODS steel (wt%).

The chemical composition of the fully ferritic MA956 ODS steel used in this study is shown in Table 1. The MA956 plate was manufactured by Special Metals Corporation, UK, via mechanical alloying and subsequent consolidation by extrusion. The material was then hot rolled in both the longitudinal and transverse directions [16–18] at temperatures between 900 and 1100 °C, followed by a one hour annealing treatment at 1320 °C and a final air cooling step to room temperature. Five bead-on-plate friction stir welds were carried out on two nominally 4 mm-thick plates, which were produced by slicing the original plate in half through its thickness. A polycrystalline cubic boron nitride tool, with a 25 mm shoulder and 3 mm pin, and a downforce of 25 kN was used for all of the welds. Systematic variations of the tool rotation and traverse speeds were selected in this study, see Table 2. Micro-hardness maps of the weld cross sections were obtained using a Struers Durascan automatic indenter with a 0.5 kgf (HV₀.₅). The maps were derived from lines of indentations with 0.5 mm spacings.
<table>
<thead>
<tr>
<th>Weld No. #</th>
<th>Rotations per Minute (rpm)</th>
<th>Traverse Speed (mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>200</td>
<td>140</td>
</tr>
<tr>
<td>2</td>
<td>200</td>
<td>120</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
<td>160</td>
</tr>
<tr>
<td>4</td>
<td>220</td>
<td>140</td>
</tr>
<tr>
<td>5</td>
<td>180</td>
<td>140</td>
</tr>
</tbody>
</table>

Table 2. Welding parameters, i.e. tool traverse and rotation speed, used for the associated weld number.

3.2.2. Optical and electron microscopy

The macroscopic structure of the weld cross sections was revealed by etching with a solution of 15 vol% HCl and 3 vol% HNO₃, and afterwards collecting optical micrographs using a Keyence VK-X200K 3D Laser Scanning Confocal Microscope. The microstructure of the welded regions was studied by acquiring backscattered electron (BSE) images on an FEI Magellan High Resolution FEG-SEM using an accelerating voltage of 2 kV, and transmission electron micrographs taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope. 3 mm-diameter TEM discs were prepared by electropolishing at a temperature of −40 °C, using a Tenupol 5 Jet electropolisher and an electrolyte of 90 vol% methanol and 10 vol% perchloric acid. Scanning electron micrographs of the fracture surfaces after tensile testing were taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

3.2.3. Tensile testing

Dog-bone tensile specimens with a gauge length of 15 mm and a cross section of 3×2 mm² were machined by electrical discharge machining from the transverse-longitudinal plane of the welds. The weld tensile specimens were taken from the top 2 mm of the weld, with the longest axis of the specimens lying perpendicular to the weld line; such specimens after testing can be seen in the inserts of Fig. 8. Specimens of the base material were machined so that they were comprised of only the coarse grained microstructure, as discussed in Section 3.1. Tensile tests were performed under displacement control with a displacement rate of 0.1 mm/min, using a MTS servo-hydraulic testing machine. Tests on the base material were carried out at a range of temperatures from room temperature to 750 °C, whereas tests on the weld specimens were conducted at room temperature and 500 °C.
3.3. Results

3.3.1. Structural characterisation

Fig. 1. (a)-(b) Friction stir welds of ODS steel produced in this study. The welding parameters used for each of the welds are collected in Table 2. (c) Optical micrograph of the cross section of #2. Advancing side on the right of the cross section.

Fig. 1 shows the five bead-on-plate friction stir welds produced, together with an optical micrograph of a representative weld cross section after chemical etching. The darker central region corresponds to the thermo-mechanically affected zone (TMAZ), where the tool has stirred the material leaving a significantly altered microstructure. The surrounding base material presents two different grain structures: a fine grained section, with a grain diameter in the matrix of $\sim 1-2 \mu m$, located towards the top of the plate, and beneath that is a section dominated by coarse grains elongated along the axis of extrusion, hundreds of microns thick and several millimetres long. The origin of this dual microstructure is the incomplete abnormal grain growth that only occurred in the near-surface regions of the original plate, during the high-temperature annealing at 1320 °C prior to the welding process. The plates were oriented with the fine grain structure on the topside for all of the welds.
Fig. 2. (a)-(b) BSE images of the TMAZ of #1 and #2, respectively. (c) Transmission electron micrograph of the TMAZ of #3.
The microstructure in the TMAZ can be seen in Fig. 2. Fig. 2(a) and (b) correspond to BSE images taken at the centre of the weld, approx. 1.5 mm from the top surface of the weld. The TMAZs are comprised of a roughly equiaxed grain structure with a nearly random grain orientation distribution. The mean grain diameter in the ferritic matrix of the welds measured at this location was in the range of 1.9–2.6 μm, see Table 3, as determined by Electron BackScatter Diffraction (EBSD) [19]. Furthermore, the welding process induces significant agglomeration of the nano-oxide particle dispersion in all the welds, as evidenced by transmission electron microscopy (Fig. 2c).

### 3.3.2. Hardness measurements

![Hardness maps of the cross sections of the welds.](image)

Fig. 3. Hardness maps of the cross sections of the welds. The weld number is indicated in the bottom left corner of each map.
Fig. 3 shows the hardness maps of the weld cross sections. The hardest areas, to the top right and top left of all the maps, are due to the fine grained microstructure of the base material present at these locations. Below these regions there is the coarse grained material which has a significantly lower hardness. The TMAZ in all the studied welds presents hardness values that are lower than the surrounding fine grained microstructure, but are either similar or somewhat lower than the hardness values in the coarse grained region of the base material. The lowest hardness is located close to the surface of the welds, particularly on the advancing side. The welds show a heat affected zone (HAZ) of the order of 1 mm, in which there is an intermediate hardness between the TMAZ and fine grained base material.

![Graph a](image1.png)

**Fig. 4.** Mean hardness in the TMAZ of the welds as a function of (a) the rotation and traverse speed of the welding tool, and (b) the mean grain diameter of the ferritic matrix.

Fig. 4 shows the variation of the mean hardness in the TMAZ of the welds with the welding parameters used, Fig. 4(a), namely the tool rotation and traverse speed, and the mean grain size of the welds, Fig. 4(b). The hardness values were calculated by taking the mean value of the data within ~1.5 mm of the centre of the weld at ~1.5 mm depth, which corresponds to the same location used for the EBSD maps from which the values of the
grains size were determined and from where BSE images in Fig. 2(a) and (b) were taken. The mean hardness data reveal a strong correlation between the hardness and the studied welding parameters, i.e. higher hardness values for increasing traverse speed and decreasing with rotation speed. Fig. 4(b) also points to an inverse correlation between the grain size and the mean hardness. The lowest hardness values are present in Weld #2 (200 rpm and 120 mm/ min) and Weld #4 (220 rpm and 140 mm/min). These two welds have the highest mean grain size, and also the widest TMAZs of the studied welds.

3.3.3. Tensile testing

The temperature dependence of the mechanical properties of the base material is shown in Fig. 5. At room temperature the coarse grained material has a 0.2% offset yield stress (YS) of 535(24) MPa and an ultimate tensile stress (UTS) of 610(42) MPa. In the temperature regime of 150–400 °C, both the YS and UTS remain relatively constant with values of ~430 and 545 MPa, respectively. However, at 500 °C the strength of the material starts to decrease progressively with temperature. This reduction in strength is accompanied by a drastic change in ductility, Fig. 5(b). Above 400 °C the uniform elongation decreases while the total elongation significantly increases with temperature. At 750 °C the total elongation is more than twice its value at room temperature, whereas the uniform elongation at that temperature is below 3%.

![Graph](image)

**Fig. 5.** Mechanical properties of the coarse grained base material as a function of temperature. (a) Tensile strength, where YS and UTS represent yield stress and ultimate tensile stress, respectively. (b) Total and uniform elongation.

The mechanical properties of the welds at room temperature, together with the corresponding values of the base material as reference, are shown in Fig. 6. At room temperature the YS of the base material is similar to that of the different welds. Weld #1 had
the highest YS with a value of 554 MPa, but this is only slightly greater than the mean value for the base material of 535(24) MPa. The ultimate tensile strength of the welds at room temperature is noticeably increased by \(\sim 8-16\%\) as compared to the base material. The elongation after welding was similar to the base material and no trend in elongation with the traverse speed was observed at room temperature; see Fig. 6(b).

![Mechanical properties of the welds and base material tested at room temperature as a function of tool traverse speed.](image)

**Fig. 6.** Mechanical properties of the welds and base material tested at room temperature as a function of tool traverse speed, using a value of zero for the traverse speed in the case of the base material. (a) Tensile strength and (b) elongation.

The tensile behaviour of the welds in the 500 °C regime can be seen in Fig. 7. The increase in temperature to 500 °C causes a significant decrease in both the YS and UTS of all the welds. The YS of all the welds tested lie within 5% of the corresponding value of the base material at that temperature. The UTS values for the welds were all between \(\sim 5\%\) and 15% greater than the corresponding value of the base material.
Fig. 7. Mechanical properties of the welds and base material tested at 500 °C, as a function of welding parameters. (a) Tensile strength vs. traverse speed. (b) Tensile strength vs. rotation speed. (c) Elongation vs. traverse speed and (d) elongation vs. rotation speed.

Fig. 8. Scanning electron micrographs of the fracture surface of the base material (BM) and two representative welds (#2 and #3) at variable temperatures. The base material specimens shown have been chosen to be representative of the three main fracture modes.
3.3.4. Fractography

Representative fracture surfaces of the base material at variable temperatures, and also of selected welds at room temperature and at 500 °C, are displayed in Fig. 8. There seems to be three main fracture modes for the base material, depending on the test temperature: (i) at room temperature, the material fails by an almost purely brittle fracture mode, although some areas of the fracture surface present dimples (Fig. 8 BM-RT). Delamination of the coarse grain structure was also observed at room temperature; there was splitting of the specimen parallel to the rolling direction and long specimen axis. (ii) At 150–400 °C, we observed a mixed ductile-brittle fracture surface with small dimples. At these intermediate temperatures there was a tendency for shearing fracture, where the cleavage surface runs at 45° to the direction of the applied load (Fig. 8 BM-150 °C and BM-300 °C). (iii) Above 500 °C, a ductile failure mechanism dominates. The fracture surface no longer runs at 45°, and instead pronounced necking and microvoid coalescence occurs prior to fracture (Fig. 8 BM-600 °C). In the case of the test specimens from the welds, necking and a dimpled fracture surface was observed both at room temperature and 500 °C, resembling those fracture surfaces of the base material at intermediate temperatures.

3.4. Discussion

The width of the weld cross section increased with both increasing rotation speed and decreasing traverse speed of the welding tool. This effect can be attributed to the higher peak temperatures reached during welding with faster rotation speed, as a consequence of the greater heating and adiabatic shear. Lower traverse speeds also mean that the heat is retained locally for a longer period of time. More energy is thus available, and for longer, to propagate by conduction into the surrounding metal and hence there is also a larger HAZ. These parameters also lead to lower hardness values for their respective welds. We measured a peak temperature of 943 °C for a rotation speed of 200 rpm and the lowest traverse speed of 120 mm/min in the steady-state regime during the welding of a very similar butt weld [20].

Based on the data in Table 3, as the rotation speed was increased or the traverse speed decreased, the volume density of nano-oxide particles decreased and the grain size in the ferritic matrix of the TMAZ consequently increased [19]. It should be noted that the particle volume fractions in Table 3, as probed by small angle neutron scattering (SANS), only apply to particles less than 80 nm in diameter. Particles and agglomerates larger than this value fell beyond the size range probed by our previous SANS experiment. With fewer
particles and larger grains, there are fewer obstacles for dislocations to overcome and the hardness, which is a measure of the material's resistance to plastic flow, therefore reduces.

The correlation between peak temperatures during welding and hardness also explains the heterogeneity in hardness across the TMAZ, which generally presents lower hardness values towards the top of the welds, particularly on the advancing side of the weld. The hottest locations during welding will be just beneath the rotating shoulder of the welding tool, particularly on the advancing side where the relative velocity of the tool is greater. We could expect in this location for there to be fewer oxide particles and a larger grain size, as a result of the greater temperatures and strains at these locations. Dawson et al. [21] recently found an increased mean particle size close to the surface of friction stir weld in PM2000 steel. The room-temperature yield stress of a material can in general be expressed as a sum of its main strengthening components, assuming that they operate independently [7,9,22]:

\[
\sigma_Y = \sigma_0 + \sigma_{SS} + \sigma_D + \sigma_P + \sigma_{GB}
\]

where \( \sigma_0 \) represents the Peierls–Nabarro stress or the intrinsic stress due to resistance of dislocation motion in the matrix, \( \sigma_{SS} \) the solid solution strengthening caused mainly by the Cr atoms present in the ferritic matrix and amounts to 8.5 MPa/wt% [11], \( \sigma_D \) the strengthening due to dislocations, \( \sigma_P \) the particle strengthening and \( \sigma_{GB} \) the grain boundary strengthening. The reported value of \( \sigma_0 \) for pure annealed Fe is 50 MPa [11]. We can assume that \( \sigma_0 \) and \( \sigma_{SS} \) are constant for both the base material and the welds. The base material underwent recrystallization during processing and the TMAZ during welding, consequently leaving both microstructures with a relatively low dislocation density. The change in strengthening due to dislocations has therefore not been further considered. The strengthening contribution due to Orowan looping of dislocations around the non-shearable oxide particles, \( \sigma_P \), is given by:

\[
\sigma_P = \frac{0.4}{\pi} \frac{M G b}{(1-\nu)^{0.5}} \frac{\ln(\frac{2r}{b})}{\lambda}
\]

where \( M \) is the Taylor factor (2.7), \( G \) is the shear modulus (60 GPa), \( b \) is the Burger's vector (0.25 nm) and \( \nu \) is the Poisson's ratio (0.3). The values in parenthesis are taken from [11]. \( r \) is the mean radius of the nanoparticles, and \( \lambda \) is the mean interparticle spacing given by:

\[
\lambda = \left[ \left( \frac{3\pi}{4f} \right)^{0.5} - 1.64 \right] r
\]
The values of $\sigma_p$ and $\lambda$ for the base material and the different welds are collected in Table 3. The grain boundary strengthening can be expressed as a function of the grain size $d$ via the Hall-Petch type expression:

$$\sigma_{GB} = k_{HP} \cdot d^{-0.5}$$  \hspace{1cm} (4)

where $k_{HP}$ is the Hall–Petch constant which other authors on ODS steels have used or determined to be $\sim$0.2–0.6 MPa m$^{0.5}$ [9,23,24]. Welding induces a significant grain refinement in the matrix, as compared to the coarse grain structure of the tested base material. The associated increase in grain boundary strengthening seems to compensate for the particle agglomeration and dissolution observed in the different welds, so that the yield strength after welding remains close to the value for the base material in all studied welds, see data in Fig. 6(a) and Fig. 7(a) and (b). Assuming a Zener pinning effect of the particles on the ferrite grain size, the maximum achievable ratio between the grain boundary and particle strengthening contributions can be expressed as [23,25]:

$$\frac{\sigma_{GB}}{\sigma_p} = \frac{k_{HP}}{MGb} \left(\frac{\pi d_p}{3}\right)^{1/2}$$  \hspace{1cm} (5)

where $d_p$ corresponds to the mean particle diameter. Based on the previously mentioned values for the parameters included in Eq. (5), the grain boundary strengthening contribution would be comparable to or even greater than the particle strengthening at particle sizes $d_p >$ 4–40 nm, depending on the value used for the $k_{HP}$ constant.

<table>
<thead>
<tr>
<th>Weld No. #</th>
<th>Rotations per Minute (rpm)</th>
<th>Traverse Speed (mm/min)</th>
<th>Mean Grain Diameter ($\mu$m)</th>
<th>Relative Volume Fraction</th>
<th>Mean Particle Diameter (nm)</th>
<th>Interpart. Spacing $\lambda$ (nm)</th>
<th>Orowan Strengthening $\sigma_p$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1</td>
<td>10.8(1)</td>
<td>96</td>
<td>241</td>
</tr>
<tr>
<td>1</td>
<td>200</td>
<td>140</td>
<td>2.49(5)</td>
<td>0.64</td>
<td>16.0(1)</td>
<td>182</td>
<td>141</td>
</tr>
<tr>
<td>2</td>
<td>200</td>
<td>120</td>
<td>2.58(6)</td>
<td>0.53</td>
<td>18.8(1)</td>
<td>236</td>
<td>113</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
<td>160</td>
<td>2.05(3)</td>
<td>0.77</td>
<td>10.8(1)</td>
<td>111</td>
<td>209</td>
</tr>
<tr>
<td>4</td>
<td>220</td>
<td>140</td>
<td>2.56(5)</td>
<td>0.58</td>
<td>17.2(1)</td>
<td>206</td>
<td>127</td>
</tr>
<tr>
<td>5</td>
<td>180</td>
<td>140</td>
<td>1.93(4)</td>
<td>0.83</td>
<td>11.4(1)</td>
<td>113</td>
<td>209</td>
</tr>
</tbody>
</table>

Table 3. Welding parameters and characteristic parameters of the welded microstructures: mean grain diameter of the ferritic matrix, and relative volume fraction and mean diameter of the oxide nano-particles. ‘BM’ denotes the base material. Data taken from ref. [19]. The table also contains the values of the interparticle spacing ($\lambda$) and particle strengthening due to Orowan looping ($\sigma_p$), estimated using Eq. (2) and (3), see text.

Using Eqs. (2) and (3), and with the values for the volume fraction and the mean nanoparticle size for the base material given in Table 3, we estimated the strengthening contribution from the fine (< 80 nm) particles to be 241 MPa. Following FSW, we calculated that the reduction in particle strengthening to be $\sim$13–53% (Table 3) depending on the weld
microstructure. These calculations suggest that there should be a relatively large amount of variation in the strengthening for the different welds, and a correlation with the oxide particle size and density, especially since the welds with lower particle density also have larger grain sizes. However, this expected variation was not observed in the experimental values of the yield stress as a function of welding parameters. A significant number of oxide particles tend to form agglomerates larger than > 80 nm, rather than dissolving into the matrix. Although larger particles are in general less effective at strengthening, for a given volume fraction, it seems as though these agglomerates are still contributing significantly to the strength of the welds. Due to the relatively high number density of agglomerates, they are likely to provide a somewhat greater strengthening contribution than the other large particle populations present in the microstructure from the ODS steel production, namely Al-O-based particles and Ti(CN) in MA956 steel. The number of agglomerates over 80 nm was likely to be greatest for the welds with increased rotation speed or decreased traverse speed of the welding tool, due to greater levels of mixing, and these were the welds with greatest measured reduction in nanoparticle volume fraction.

A sharp decrease in both the YS and the UTS of the base material and the welds has been observed in the vicinity of 500 °C. Below that temperature, the variation of the Orowan stress with temperature arises mainly from changes in the matrix shear modulus, G [22]. However, at higher temperatures, the dispersed particles can be overcome by dislocation climb and the strengthening contribution from Orowan looping, which is dominant at lower temperatures, becomes diminished. The particles do still offer a non-negligible strengthening contribution at high temperatures, via an attractive particle-dislocation interaction after the dislocation has climbed over the particle [26–29]. The particle-dislocation interaction is however not able to produce stress exponents as high as Orowan looping, and we observe a sharp drop in strength as thermal effects overcome the activation energy for dislocation detachment. In addition to the temperature dependence of the Orowan particle strengthening mechanism, the grain boundary strengthening contribution falls quickly at high homologous temperatures, e.g. T/Tm > 0.4, even though the grain size does not change significantly at temperatures close to 500 °C. The transition temperature decreases with the grain size of the matrix [23,30]. This temperature effect on the grain boundary strengthening has been successfully modelled based on a steady-state deformation mechanism involving the thermally-activated absorption of dislocations at grain boundaries [23]. Due to the lack of grain boundary strengthening at elevated temperatures, it
is of even greater importance that the particle dispersion of the weld is as close as possible to that of the base material, so that the welds are still able to offer as much strength as possible at these elevated temperatures, whether or not it is decided to keep the refined microstructure or induce abnormal grain growth post weld [12]. The relatively close values for the YS and UTS observed for the welds and the base material at 500 °C seem promising in terms of ODS steels' prospective implementation in future nuclear reactors.

The uniform and total elongation in the base material present relatively close values to one another, and both remain close to constant with temperature up to 400 °C (Fig. 5(b)). At higher temperatures the total elongation increases progressively, whereas the uniform elongation reduces simultaneously. The welds retain a comparable strength to the BM at both room temperature and 500 °C, (Figs. 6(a) and 7(a) and (b)). This is the case for both YS and UTS, except at room temperature where the UTS of the welds was superior to the BM. The inspection of the fracture surfaces in Fig. 8(top row) revealed a change in fracture mode with temperature; from brittle at room temperature, to a mixed brittle-ductile at 150–400 °C and further to pure ductile fracture above 400 °C. For the MA956 base material, the ductile-to-brittle transition temperature is reported to take a value of 40–70 °C [31]. At room temperature, the work hardening of the base material is low, and fracture occurs relatively quickly after the UTS is reached and generates a predominantly brittle-like fracture surface. On the contrary, at temperatures higher than 400 °C, the fracture surface is characterised by a number of relatively large equiaxed dimples. This fact may imply that void nucleation at high temperatures takes place at local strain concentrations close to second phase micron-sized particles for relatively low strain levels, probably induced by dislocation pile-ups and decohesion of the particle/matrix interface, and therefore leads to relatively low values of uniform elongation. However, significant plastic deformation is still needed to induce fracture, as revealed by the increased necking and total elongation. In contrast, the welded microstructures are characterised by a fine grained structure and a significant number of particle agglomerates that can be larger than 100 nm diameter, see Fig. 2. This means that there are a higher number of void nucleation sites in the welded region as compared to the coarse grained base material, i.e. grain boundaries and particle agglomerates [32]. Consequently, the ductile fracture surface of the welds, see Fig. 8(bottom row), is characterised by a large number of roughly equiaxed dimples resulting from the random distribution of coalesced voids.
3.5. Conclusions

We have investigated the temperature-dependent mechanical behaviour of MA956 ODS steel before and after friction stir welding. The coarse grained base material retains a relatively large proportion of its room-temperature strength up to 500 °C. At higher temperatures both the yield stress and the ultimate tensile stress decrease with temperature, whereas the total elongation increases. The strength and ductility of the welds are similar to those of the base material, both at room temperature and at 500 °C. The decrease in particle strengthening due to agglomeration in the welds is compensated by the grain boundary strengthening due to the finer grain structure of the welds as compared to the base material. Furthermore, the additional presence of submicron- sized particle agglomerates and a higher grain boundary density in the welded regions seems to increase the number of void nucleation sites. Consequently the fracture surfaces of the welds comprise of a much larger number of smaller equiaxed dimples. No trend in mechanical properties of the welds was observed from the tensile data, but an increase in hardness was observed with increasing traverse speed or decreasing rotation speed, due to the finer grain size and larger number of strengthening nano-particles present in these welds.

Acknowledgements

We gratefully acknowledge the financial support of the Engineering and Physical Sciences Research Council UK (EPSRC) through the Centre for Doctoral Training in Advanced Metallic Systems (EP/L016273/1). We would like to thank The Welding Institute (TWI) for providing the welding tools and assistance during friction stir welding. We also acknowledge CIEMAT for providing the MA956 base material and performing the tensile tests and SEM imaging of the fracture surfaces. This work contributes to the Joint Programme on Nuclear Materials (JPNM) of the European Energy Research Alliance (EERA).

References


Preface to Chapter 4

Chapter 4, entitled “Residual stress distribution in Friction Stir Welded ODS steel measured by neutron diffraction”, has been published as a research paper in the Journal of Materials Processing Technology, see bottom of page. The Chapter focuses on measuring the size and distribution of residual stresses in butt welded plates by the use of neutron diffraction. The measurements were carried out on 3 welds, which each used a different traverse speed. With this we could observe how the traverse speed affects the magnitude and distribution of the residual stresses and to determine if changing the welding parameters may be an effective method of producing acceptable levels of residual stress. During the welding process, real-time torque and temperature profiles were collected in order to help understand the origins of the differences in residual stress distributions observed between the different welds.

The ODS steel plates were provided by CIEMAT. In this Chapter, and the following Chapter, the plates were butt welded together by friction stir welding. This was once again conducted at TWI, Yorkshire, by H. Dawson and S. Cater.

Optical micrographs and XRD texture measurements were obtained by H. Dawson at the University of Manchester. The neutron diffraction experiment was conducted at Institut Laue-Langevin (ILL), France, on the SALSA beamline by H. Dawson, P. Wady, T. Pirling and E. Jimenez-Melero.

Reference:


DOI: http://dx.doi.org/10.1016/j.jmatprotec.2017.03.013
Chapter 4

4. Residual stress distribution in friction stir welded ODS steel measured by neutron diffraction

H. Dawson\textsuperscript{a},*, M. Serrano\textsuperscript{b}, S. Cater\textsuperscript{c}, P. Wady\textsuperscript{d}, P. Thirling\textsuperscript{e}, E Jimenez-Melero\textsuperscript{a}

\textsuperscript{a}School of Materials, University of Manchester, Manchester M13 9PL, United Kingdom
\textsuperscript{b}Structural Materials Division, Technology Department, CIEMAT, Avda de la Complutense 40, 28040 Madrid, Spain
\textsuperscript{c}Friction and Forge Processes Department, Joining Technologies Group, TWI Technology Centre (Yorkshire), Advanced Manufacturing Park, Wallis Way, Catcliffe, Rotherham S60 5TZ, United Kingdom
\textsuperscript{d}Dalton Cumbrian Facility, University of Manchester, Westlakes Science & Technology Park, Moor Row, Cumbria, CA24 3HA, United Kingdom
\textsuperscript{e}Institut Laue-Langevin, BP 156, rue de Horowitz, 38042 Grenoble Cedex 9, France
Abstract

We have mapped the residual stress distribution in Oxide-Dispersion Strengthened (ODS) steel plates, joined by friction stir welding, using neutron diffraction. The measured stress maps were interpreted in terms of the temperature and torque profiles measured using three different tool traverse speeds. Friction stir welding constitutes a promising solid-state process for joining ODS steels that largely preserves the strengthening nano-oxide particles in the matrix. The particle dispersion is critical for future high-temperature and nuclear applications of ODS steels. We found that the largest peak tensile stresses, ~1200 MPa, are present in the weld produced using the fastest tool traverse speed, and corresponds to relatively high cooling rates. A reduction in tool traverse speed yields a significant decrease in tensile residual stresses in the thermo-mechanically affected zone (TMAZ) of the welds, but also causes higher peak temperatures during welding. Furthermore, the torque is mainly related to the amount of materials being stirred, and affects the width of the TMAZ and its residual stress distribution. Our results open the door to optimising the friction stir process of ODS steel by modifying the tool traverse speed, so that lower peak temperatures and residual stresses are generated in the welds, and therefore lead to enhanced performance in the expected service environments of next generation nuclear reactors.
4.1. Introduction

Oxide Dispersion-Strengthened (ODS) steels are one of the most promising structural material candidates for the cladding of Gen IV fission reactors and for first-wall components in magnetically-confined fusion reactors, discussed in a review by Zinkle and Snead (2014). ODS steels offer an outstanding combination of high-temperature strength and creep resistance. These properties are largely dependent on the presence of a fine homogeneous dispersion of Y(Al,Ti) oxide nano-particles throughout the matrix. ODS steels also present a high resistance to void and helium bubble formation induced by radiation. These properties stem largely from the high density of particle/matrix interfaces that act as preferential trapping sites for trapping radiation-induced lattice defects and helium atoms, see Brodrick et al. (2014) and Odette et al. (2008), and also as effective obstacles for dislocation movement, providing excellent high temperature strength and creep resistance.

Despite these beneficial properties for the realization of next generation nuclear reactors, the joining of ODS steel components still remains a technological challenge limiting their potential use in the nuclear industry, since the use of standard joining techniques will cause the ODS steel to melt. As a consequence, the oxide nano-dispersoids would either agglomerate or dissolve in the matrix, creating a welded region with inferior strength and radiation resistance as compared to the base material. Friction Stir Welding (FSW) is a solid-state technique that is potentially able to join ODS steel plates without melting the material, and therefore causing minimal disruption to the nano-oxide dispersion. FSW relies on the local deformation and localized heating caused by the welding tool, as it rotates and advances along the weld line between the two metallic plates to be joined together, see Fig. 2a from Su et al. (2013).

Unfortunately, this welding process can cause significant residual stresses that may detrimentally affect the properties of the material, such as fatigue strength, toughness and corrosion resistance of the material and could consequently lead to catastrophic failure of ODS reactor components close to the welding zone during service operations. The impact of residual stresses on the properties of materials is discussed at greater length in Totten (2002) and in Williams and Steuwer (2009). Current experimental data and knowledge about the generation of residual stresses resulting from FSW is very limited in ODS steels, and steels in general according to Kumar et al. (2014), and does not allow to link the welding parameters to the residual stresses. This hinders the application of FSW as joining technique in ODS steel components for future nuclear reactors. This paper aims to assess the residual stresses
created in ODS steel butt welds, and the effect of changing the traverse speed of the welding tool, by the use of neutron diffraction.

4.2. Experimental

4.2.1. Starting ODS steel material

MA956 is a fully ferritic ODS steel that contains high levels of Cr and Al for corrosion and oxidation resistance, see Table 1. The material was produced by Special Metals, UK, and was provided in the form of a 10 mm thick plate with a final recrystallization annealing treatment at 1320 °C for 1 h, to induce abnormal grain growth in the plate. Despite this high temperature anneal, the material mostly retained its fine microstructure in the centre line of the plate, with typically 1–2 µm ferritic grains elongated along the extrusion axis, while the plate surface did experience abnormal grain growth and therefore contains very coarse (> mm) grains, see Fig. 1a and b. More detail about the microstructure of the MA956 plates can be seen in Dawson et al. (2017). Texture measurements were made on a D8 Bruker X-ray Diffractometer with a collimated cobalt source, and the data analysis was carried out using the MTEX software, Bachmann et al. (2010). The plate showed a strong (100)<110> rolling texture, Fig. 1c. These plates were cut in half through thickness by electrical discharge machining to produce nominally 4 mm thick plates.

<table>
<thead>
<tr>
<th>Cr</th>
<th>Al</th>
<th>Y₂O₃</th>
<th>P</th>
<th>Ti</th>
<th>O</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
<th>Ni</th>
<th>Co</th>
<th>N</th>
<th>Cu</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.97</td>
<td>4.44</td>
<td>0.53</td>
<td>0.53</td>
<td>0.21</td>
<td>0.15</td>
<td>0.11</td>
<td>0.05</td>
<td>&lt;0.05</td>
<td>0.04</td>
<td>0.03</td>
<td>0.022</td>
<td>0.009</td>
<td>0.004</td>
<td>bal.</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Chemical composition of the studied MA956 ODS steel (wt%).
4.2.2. Friction stir welding of ODS steel

Butt welds were made on these plates using an MTI RM-2 Precision Spindle FSW machine in the presence of an argon shielding gas, using depth control and a steel backing plate. Due to the non-uniform distribution in ferrite grain size through the thickness of the plates, the butt welds were carried out with the coarse grains at the top of the workpiece. The tool used for all the welds was a Q70 grade Megastir polycrystalline cubic boron nitride (PCBN) tool with a shoulder diameter of 25 mm and pin length of 3 mm. The tool had a dwell time of 35 s after being plunged and before traversing along the welding line, and 25 s before exiting the welded plates. A downforce of 25 kN and a tool rotation speed of 200 rotations per minute were used. Three welds with varying tool traverse speed were produced: 70, 95 and 120 mm/min. Internal temperatures during welding were measured by embedding type k thermocouples with Alloy 600 sheath material into the plates, by boring 1 mm diameter holes by electric discharge machining from the edge of the plate. Fig. 2 shows the position of all of the thermocouples in the three welds. For Welds 2 and 3 the thermocouples holes were bored right up to the joint line, 2 mm from the top surface of the plate. For Weld 1 the holes were also 2 mm from the top surface of the plate but the position of the
thermocouples was gradually staggered back from the centre of the joint line in 1 mm steps, starting from the exit-hole end. The thermocouples were not positioned closer than 30 mm to each other. Ultrasound testing was used to check the position and survival of the thermocouples of Weld 1, see Fig. 2b. The macroscopic structure of the weld cross sections was revealed by etching with a solution containing 15 vol.% HCl and 3 vol.% HNO₃. The images were taken using a Keyence VK-X200 K 3D Laser Scanning Microscope.

Fig. 2. (a) Schematic diagram of the friction stir welding process, adapted from Su et al. (2013); (b) ultrasound image showing the bored thermocouple holes and diagram of thermocouple positions for Weld 1; (c)-(e) temperature profiles during FSW for the three welds; (f) torque measured during welding. Lengths are in units of mm.
4.2.3. Residual stress determination by neutron diffraction

Neutron diffraction is a suitable technique to probe residual stresses distributions in engineering alloys by measuring the relative change in the scattering angle, $2\theta$, as compared to the scattering angle from an unstressed state in the sample, $2\theta_0$. The scattering angles depend on the lattice spacing of the material, according to Bragg’s Law, and therefore the lattice itself can act as an effective strain gauge. Residual stress measurements were performed using the SALSA diffractometer at the Institut Laue-Langevin (ILL) (Grenoble, France), using a monochromatic neutron beam with a wavelength of $\lambda = 1.64$ Å. The gauge volume used was a cuboid with dimensions $0.6 \times 0.6 \times 2$ mm$^3$. The perpendicular bisectors of the square faces, lengths 0.85 mm, were aligned parallel to the transverse (y) and normal (z) directions, while the depth of the cuboid ran parallel to the welding direction (x). The main components of the SALSA diffractometer are shown in Fig. 3.

![Fig. 3. Main components of the SALSA diffractometer at ILL, used during the neutron diffraction experiment to obtain the residual stress maps of ODS steel welds.](image)

The welds were placed on the hexapod sample manipulator, which is able to move with high precision in six axes, and therefore maintain accurate alignment for all measured sample positions, see Pirling et al. (2006). The measurements were taken at five selected depths through the thickness of the welds, ranging from 0.8 mm to 3.8 mm in steps of 0.6 mm, in order to avoid potential surface effects on the residual stress determination. For Welds 1 and 3, the scanned position was at 70 and 87 mm from the edge of the plate on the tool entry side, respectively. Weld 2 was measured at three different locations along the weld; 35, 87 and 135
mm from the edge of the plate on the tool entry side. The measured scattering angle at the
selected positions in the welds was used to calculate the lattice strains:

\[
\varepsilon_{ii} = \frac{d_{1(110)} - d_{0,(110)}}{d_{0,(110)}} = \frac{\sin \theta_{0,(110)}}{\sin \theta_{(110)}} - 1
\]

(1)

where \(\varepsilon_{ii}\) denotes the lattice strain, and \(d\) represents the lattice spacing at a given weld position and \(d_0\) the reference unstressed lattice spacing. The unstressed position in the sample was characterized to be the average value of the scattering angle of several points, 70 or 80 mm away from weld centre on the advancing side of Weld 1. The strains of the plates were measured in the longitudinal, transverse and normal directions, represented by \(i = x, y\) and \(z\) respectively. Due to the relatively strong texture in the base material, only the strains in the \{110\} lattice planes were measured. The stresses along the three principal directions in the material were calculated using the measured strains according to:

\[
\sigma_i = \frac{E_{(110)}}{1 + \nu_{(110)}} \left[ \varepsilon_i + \frac{\nu_{(110)}}{1 - 2\nu_{(110)}} (\varepsilon_x + \varepsilon_y + \varepsilon_z) \right]
\]

(2)

where the Young’s Modulus \(E_{110} = 224.7\) GPa, and the Poisson’s ratio \(\nu_{110} = 0.28\). The values were used by Mathon et al. (2009) on a similar ODS steel, PM2000, and were originally based on the model developed by Kröner (1961) for non-textured bcc iron. The strong texture also meant that no \{110\} diffracted intensity could be detected when probing the normal direction in the base material. However, the microstructure in the thermo-mechanically affected zone (TMAZ) has a near random texture, see Fig. 1d, so it was possible to detect \{110\} diffracted intensity along the normal direction in this region. The thickness of the welded plates was relatively small, and therefore the normal stresses can be approximated as zero, \(\sigma_z \approx 0\), described further in Hutchings et al. (2005). The normal strains were therefore determined using the equation:

\[
\varepsilon_z = (\varepsilon_x + \varepsilon_y)\nu_{(110)}/(\nu_{(110)} - 1)
\]

(3)
4.3. Results and discussion

4.3.1. Friction stir welding parameters

Table 2 contains the characteristic values of the main friction stir welding parameters in the steady state regime. Fig. 3 displays the measured temperature and torque profiles for the three values of the tool traverse speed, together with a schematic diagram of the friction stir welding process. The thermocouple readings in the three welds revealed a relatively uniform temperature during the steady state regime during welding. The peak temperatures during welding increased with decreasing traverse speed. This is to be expected as the heat input (HI) is taken as the ratio of the rotational speed (ω) to the traverse speed (v), HI = ω/v.

Thermocouples 1 and 5 in Weld 1 were placed close to the entry and exit positions of the tool, respectively. Due to the dwell times at these positions, the temperature reached is significantly higher than at the intermediate thermocouple positions.

<table>
<thead>
<tr>
<th>Weld No.</th>
<th>Traverse Speed (mm/min)</th>
<th>Peak Temp. (°C)</th>
<th>Average cooling rate (°C/s)</th>
<th>Time above 400°C (s)</th>
<th>Average Torque (Nm)</th>
<th>Peak Longitudinal Tensile Stress (MPa)</th>
<th>Peak Transverse Tensile Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>120</td>
<td>943</td>
<td>36</td>
<td>15</td>
<td>83</td>
<td>1207</td>
<td>728</td>
</tr>
<tr>
<td>2</td>
<td>95</td>
<td>1074</td>
<td>58</td>
<td>18</td>
<td>88</td>
<td>1189</td>
<td>809</td>
</tr>
<tr>
<td>3</td>
<td>70</td>
<td>1220</td>
<td>30</td>
<td>41</td>
<td>159</td>
<td>901</td>
<td>770</td>
</tr>
</tbody>
</table>

Table 2. Friction stir welding parameters in the steady state regime, together with the peak tensile residual stress along the longitudinal and traverse directions for the three ODS steel welds produced in this study. The average cooling rate has been determined between the peak temperature and 400 °C. Weld 1 used a mean value of only thermocouples 2–4 in the calculation. Weld 2 and 3 used a mean value of both thermocouples.

The cooling rates were expected to increase with the traverse speed since the rotating tool, which is essentially the heat source, is further away from the temperature measurement position along the weld line at any specific time during the process. The cooling rates were calculated as the mean value of the rate from peak temperature to 400 °C. A mean value was calculated from thermocouples considered in the steady state section of each weld, i.e. thermocouples 1 and 5 for Weld 1 were not included in the calculation. Weld 3, with the slowest traverse speed of 70 mm/min, has indeed the slowest cooling rate (30 °C /s) of the three welds, and corresponds to the highest peak temperature (1220 °C). However, the cooling rate of Weld 1 (36 °C /s) is slower than that of Weld 2 (58 °C /s), despite Weld 1 corresponding to the highest traverse speed in this work. This fact may be partly ascribed to the lower peak temperature in Weld 1 causing a smaller temperature gradient. However, one would expect the distance of the welding tool from the measured position to be the dominant parameter, since the rotating tool is the main source of heat in this process. The time that the
weld was above 400 °C, arbitrarily, was approximately 15 s for Weld 1 and 18 s for Weld 2. The anomaly in the cooling rate for the highest traverse speed of 120 mm/min will have originated from the non-negligible difference in the distance between the bottom of the TMAZ and the steel backing plate. For Weld 1, see Fig. 4a, i.e. there is nearly 1 mm between the base of the TMAZ and the bottom of the workpiece which was in contact with the backing plate also acting as an efficient heat sink. Both Welds 2 and 3 presented a full penetration of the welding tool in the ODS steel plates, see Fig. 4b and c. Therefore the TMAZ was in contact with the backing bar in those two welds, causing the heat to be dissipated at a higher rate through the backing plate than for Weld 1. The TMAZ of Weld 1 has a similar depth to the other two welds; however a workpiece of slightly greater thickness was used for Weld 1.

![Fig. 4. Optical micrographs of the top view and cross section of the three welds: (a) Weld 1, (b) Weld 2, (c) Weld 3. Advancing side is on right hand side of the cross sections.](image)

The measured torque profile throughout the welding of the three butt welds can be seen in Fig. 2f. The welds have a relatively constant torque throughout the steady state section of the welding process. Furthermore, Welds 1 and 2 have a very similar torque throughout the weld. Several investigations have shown that torque generated during FSW turns out to vary weakly with the traverse speed, as stated in a review on FSW on Al alloys by Çam and Mistikoglu (2014). Su et al. (2013) and Yan et al. (2005) both showed an extremely gradual
increase in torque with increasing traverse speed. Arora et al. (2009) developed a model that closely matched the findings of Yan et al. (2005). For steel, Lienert et al. (2003) also observed no observable trend in torque with traverse speed.

The torque of Weld 3 was measured to be significantly greater than for the other two welds, despite the material becoming more plasticised due to the significantly greater peak temperature achieved during welding. The heat generated during the production of this weld was sufficiently high as to plasticise significantly more material, which means that a larger volume of material was stirred than for Weld 1 or 2. Moreover, Fig. 4 shows the wider TMAZ of Weld 3 compared to the other two welds. The increased volume of material stirred in Weld 3 will have increased the necessary torque to maintain the rotation speed of the welding tool. The TMAZs of Weld 1 and 2 seems to be much more comparable in size, and hence the torque measured during welding was similar in both welds. Work by Mayfield and Sorensen (2010) showed that the torque was not significantly altered by changes to the temperature in the SZ, agreeing with the findings of this study in which Weld 1 and 2 had similar torques despite significantly different internal temperatures.

### 4.3.2. Residual stress distributions

![Residual stress maps of the three ODS steel welds. Stress in units of MPa.](image)

**Fig. 5.** Residual stress maps of the three ODS steel welds. Stress in units of MPa.
The residual stress distributions for the three different welds are shown in Fig. 5 along the transverse and longitudinal directions. The measured normal stresses in the welded regions turned out to be relatively small, rarely exceeding a stress of 200 MPa. We can expect that the residual stresses outside of the weld will be significantly lower than this; therefore the assumption stated in Section 2.3 that the normal stresses are approximately equal to zero seems reasonable. The welds contain relatively large tensile stresses in the TMAZ where the most significant heating occurred during welding, although smaller tensile stresses were observed outside of the TMAZ, particularly on the advancing side towards the top of the workpiece. The distribution of these tensile stresses is approximately symmetric with respect to the welding line, with the highest residual tensile stresses measured close the edge of the TMAZ. Compensating compressive stresses were observed surrounding the TMAZ. The compressive stresses were generally greater on the retreating side and increased towards the bottom of the workpiece. Notably the transverse stresses, though also tensile in nature, are significantly lower than the longitudinal stresses, rarely exceeding the yield stress of the material. Kumar et al. (2014) describes this as being a common trend in friction stir welded alloys. Similar findings have been observed in other friction stir welds for steels, by Steuwer et al. (2012) and Reynolds et al. (2003), in ODS steels by Mathon et al. (2009), and in Al alloys by Peel et al. (2003) and Woo et al. (2006).

Fig. 6. Line scan residual stress data at a depth of 1.4 mm from the top surface of the welds, in the (a) longitudinal and (b) transverse orientation; (c) peak tensile residual stress and (d) peak temperature and cooling rate as a function of traverse speed during welding. The peak values are the mean of the 5 greatest values at any 5 measurement locations in each weld.
Fig. 6 shows the line scan residual stress data at a depth of 1.4 mm from the top surface of the three welds along with the peak stresses and the measured cooling rates of the welds as a function of traverse speed. The peak tensile stresses in the transverse do not seem to be affected significantly by the change in tool traverse speed. The longitudinal residual stresses, however, increase with increasing traverse speed (Fig. 6c), in agreement with previous reports on friction stir welded engineering alloys, see for instance Peel et al. (2003) or Brewer et al. (2015). It should be stressed that no phase change has been observed following FSW, and therefore this is not considered as a contributor to the residual stress.

The dominant factors in determining the magnitude of the residual stresses are likely to be the peak temperatures during welding and the subsequent cooling rates. The stresses are expected to increase with traversing speed, since there would be a faster associated cooling rate. Welds 1 and 2 were held at elevated temperatures for comparable times, see Table 2, and consequently the stresses would have had a similar amount of time to relax, resulting in comparable residual stresses. The cooling rate of Weld 3, with the reduced traverse speed of 70 mm/min, was significantly lower, and therefore the stresses had more time to relax. The resultant residual stresses are generally lower and spread more evenly through the weld, rarely exceeding the yield stress. Welds 1 and 2 had faster cooling rates and significantly less time for the stresses to relax, and therefore had much larger peak stresses and sharper stress gradients. The residual stresses measured at three selected locations along Weld 2 are reasonably consistent, see Fig. 7. The longitudinal stresses at the extreme locations appear to be slightly lower than at the intermediate position. This is likely to be due to their being closer to the entry or exit hole where the tool dwells, which would cause the cooling rate at these locations to be slightly lower than at the intermediate position. The tool, heat input and plate thickness were very stable throughout the weld, as can be seen by the stable torque and temperature profiles in Fig. 3. Therefore the residual stresses are near constant along the weld direction. Positions 1 and 2 also show similar transverse stresses, while somewhat different close to the exit position of the tool.
These results reveal that FSW can leave significant residual stresses in ODS steels, that should be considered before their implementation in next generation reactors. It is clearly possible to reduce the residual stresses in the TMAZ of the weld by decreasing the traversing speed of the tool; however this generates a higher peak temperature that, if excessively high, may affect the oxide particle stability in the welded zones as shown in Dawson et al. (2017). A post-weld heat treatment may be considered to reduce the residual stresses in the TMAZ of the weld.

**4.4. Conclusions**

The residual stresses resulting from the friction stir welding of ODS steel plates have been assessed along with the impact of varying the traverse speed. All of the welds produced in this study retained significant residual stresses after welding, with greater values in the longitudinal orientation than in the transverse. The magnitude of the tensile residual stresses in the TMAZ of the welds depends predominantly on the cooling rate of the welds after the
passage of the welding tool, and can therefore be reduced by decreasing the tool traverse speed. However, such a reduction in traverse speed generates higher peak temperatures that, if excessive, may pose a risk for the thermal stability of the nano-oxide particles in the welds. The torque during welding was found to depend predominantly on the amount of material stirred, and is therefore related the width of the TMAZ.

Acknowledgements

We gratefully acknowledge the financial support of the Engineering and Physical Sciences Research Council UK (EPSRC) (grant number EP/L016273/1) through the Centre for Doctoral Training in Advanced Metallic Systems. We would like to thank CIEMAT in Spain for providing the base material of MA956 ODS steel, and to The Welding Institute (TWI) for providing the welding tools and assistance during friction stir welding. We also acknowledge the Institut Laue-Langevin for the granted beam time for use of the SALSA instrument.

References


Preface to Chapter 5

Chapter 5, entitled “Characterization of MA956 ODS steel friction stir welds and their abnormal grain growth behaviour”, is the final research Chapter of this thesis. At the time of submission of the thesis, the work of this Chapter has not been published elsewhere. This Chapter once again assesses the impact of FSW on the microstructure of the ODS steel. The grain structures are assessed as they were in Chapter 2 to observe if the same relationship with altering the traverse speed occurs in the butt welds. This Chapter extends the investigation of the welds’ microstructures by also assessing how the grain size varies across the weld cross-section. This Chapter also includes pole figures illustrating the textures of the 3 welds, however no observations of the nano-particle distributions are included.

This Chapter also investigates the very high-temperature stability of the weld microstructures i.e. the welds were subjected to a 1-hour heat treatment at very high temperature to deliberately try and induce abnormal grain growth in the welded structure. This is because a coarse grain structure may be considered preferential, to improve creep resistance. The resultant changes to the macro-structure of the weld cross sections following the heat treatment were then investigated by a combination of optical microscopy and micro-hardness testing.

The ODS steel plates were provided by CIEMAT. The butt welds were friction stir welded at TWI Technology Centre, Yorkshire, by H. Dawson and S. Cater.

This Chapter includes optical and scanning electron microscopy, micro-hardness measurements and XRD texture measurements. These were all conducted by H. Dawson at the University of Manchester or the affiliated Dalton Cumbrian Facility (DCF). The high-temperature heat treatment was also carried out at DCF.
Chapter 5

5. Characterization of MA956 ODS steel friction stir welds and their abnormal grain growth behaviour

H. Dawson\textsuperscript{a}, M. Serrano\textsuperscript{b}, S. Cater\textsuperscript{c}, E. Jimenez-Melero\textsuperscript{a}

\textsuperscript{a}School of Materials, University of Manchester, Manchester M13 9PL, United Kingdom
\textsuperscript{b}Structural Materials Division, Technology Department, CIEMAT, Avda de la Complutense 40, 28040 Madrid, Spain
\textsuperscript{c}Friction and Forge Processes Department, Joining Technologies Group, TWI Technology Centre (Yorkshire), Advanced Manufacturing Park, Wallis Way, Catcliffe, Rotherham S60 5TZ, United Kingdom
Abstract

We have characterized three friction stir butt welds of MA956 ODS steel produced using different traverse speeds of the welding tool, by a combination of micro-hardness testing and optical and electron microscopy. The welds were also given a high temperature heat treatment at 1380 °C for one hour to induce abnormal grain growth, aimed to optimise the grain size for optimal high-temperature thermal creep resistance of the welds. The mean grain size at all measured locations increased with a decreased welding speed, due to the increased thermal energy into the weld. The grain size changed gradually across the stir zone of the weld, with larger grains present towards the top of the weld and on the advancing side. This was accompanied by lower hardness values at those locations. Shear banding, in the thermomechanically affected zone, and a deformed region of the base material, was clearly observed for all welds at the weld boarder. The post-weld heat treatment was able to induce abnormal grain growth in all the welds, creating a very coarse microstructure with grain sizes in the order of hundreds of microns or millimetres. The coarse grained structure seemed to develop from the top of the stir zone, close to the surface fine-grained layer, and progressed downwards until it generally covered the entirety of the welds’ stir zone. Abnormal grain growth did not occur in the border region of the welds, most likely due to the observed local particle pile-up in that region.
5.1. Introduction

Friction stir welding (FSW), invented at The Welding Institute (TWI) in 1991 [1], is a solid-state technique currently under the spotlight as a promising method for joining Oxide Dispersion-Strengthened (ODS) steel components for next generation nuclear reactors. The outstanding ODS steel performance in high-temperature radiation environments originates largely from the very fine dispersion of yttrium-based oxide particles present in the high-Cr body-centred cubic ferritic matrix [2-4]. Those nano-sized impenetrable particles act as effective pinning obstacles for glissile dislocations and also for grain boundary migration [5-7]. Moreover, the particle/matrix interface constitutes a relatively high sink strength for radiation-induced point defects and helium atoms, therefore minimising void swelling and helium embrittlement [8-11]. Consequently ODS steels are able to withstand high radiation damage levels, 100 dpa or higher, at the high service temperatures expected in high fuel burn-up claddings in fission reactors and also in the first wall of magnetically-confined fusion reactors [10, 12, 13]. Unfortunately, fusion joining techniques induce severe particle agglomeration and non-homogeneous distributions in the ODS steel matrix [14]. In contrast, during FSW local temperatures do not exceed 70-90% of the melting point of the material [15, 16], and therefore the fine dispersion of yttrium-based oxide particles can potentially be preserved in the matrix.

In this work we assess the impact of (i) changing systematically the traverse speed of the welding tool and (ii) a post-weld heat treatment designed to induce abnormal grain growth (AGG) in the welds, on the MA956 ODS steel microstructure produced by friction stir welding. The MA956 base material can be produced with either a fine-grained or a coarse-grained microstructure [17]. Friction stir welding typically produces microstructures in ODS steels with grain sizes in the order of micrometres to tens of micrometres due to a phenomenon of dynamic recrystallization [18-22]. However, enhanced thermal creep resistance is attained by minimizing the amount of grain boundary area perpendicular to the applied mechanical load, either via grain coarsening and/or a high aspect ratio [23, 24]. Consequently cavity growth and coalescence in the direction perpendicular to the applied load would be hindered [25]. This is why many ODS steels are manufactured to have an extremely coarse columnar grain structure.

The significant grain refinement resulting from FSW may therefore lead to insufficient creep resistance in the weld. A coarser microstructure may be attained by inducing AGG with a very high temperature post-weld heat treatment, enhancing the creep performance and
potentially increasing the maximum temperature for safe operation of these materials and their welds [26].

Currently, investigations into the abnormal grain growth of ODS steels following FSW remain limited. Chen et al [23] and Dawson et al. [20] were able to induce AGG in one friction stir weld of PM2000 ODS steel via a 1 hour heat treatment at 1380 °C. West et al. [19] heat treated an MA956 friction stir weld for 5 hours at 1300 °C, which did not induce AGG and produced only a subtle grain growth effect.

5.2. Materials and Method

5.2.1. Base material

<table>
<thead>
<tr>
<th>Cr</th>
<th>Al</th>
<th>Y2O3</th>
<th>P</th>
<th>Ti</th>
<th>O</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
<th>Ni</th>
<th>Co</th>
<th>N</th>
<th>Cu</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.97</td>
<td>4.44</td>
<td>0.53</td>
<td>0.33</td>
<td>0.21</td>
<td>0.15</td>
<td>0.11</td>
<td>0.05</td>
<td>&lt;0.05</td>
<td>0.04</td>
<td>0.03</td>
<td>0.022</td>
<td>0.009</td>
<td>0.004</td>
<td>bal.</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Table 1 Chemical composition of the studied MA956 ODS steel (wt%).

The chemical composition of the fully ferritic MA956 ODS steel is shown in Table 1. The MA956 base material was manufactured by Special Metals, UK, and provided in the form of a 10 mm thick plate. The production route entailed mechanical alloying of the constituent fine metallic powders with Y2O3 particles in a high energy ball mill. The mixed powders were then consolidated by hot extrusion at ~1000 °C, followed by hot rolling in both the transverse and longitudinal directions using a 3-high reversing mill. Afterwards, the plate received a recrystallization annealing treatment for 1 hour at 1320 °C and was finally air cooled to room temperature [17, 27, 28]. AGG during material production was incomplete, leaving a bimodal grain distribution across the plate microstructure [22]. The top and bottom parts of the plate contain the intended mm-sized columnar grains, whereas the centre of the plate was characterised by primary recrystallized grains (~1–2 µm) that are slightly elongated along the extrusion direction.

5.2.2. Welding and post-weld heat treatment

The plates for FSW were machined by cutting the original 10 mm-thick plates through thickness using Electrical Discharge Machining (EDM) to create ~4–5 mm thick plates. Three butt welds were carried out on the MA956 plates with an MTI RM-2 Precision Spindle FSW machine using a Q70 grade Megastir polycrystalline cubic boron nitride (PCBN) tool and running in depth control. The welding tool had a shoulder diameter of 25 mm and a pin length of 3 mm. The welds were carried out with the coarse grain microstructure at the top of the workpiece facing the welding tool as it plunged into the workpiece. We used an argon
shielding gas during welding and a steel backing plate. The traverse speed of the welding tool was varied: 120, 95 and 70 mm/min for Welds 1, 2 and 3, respectively. A downforce of 25 kN and a tool rotation speed of 200 rotations per minute were used in the three cases. Cross-sectional specimens from the three welds were subsequently machined with dimensions of \(~40 \times 4 \times 2\) mm\(^3\). Those specimens were heat treated for 1 hour at 1380 °C in Argon atmosphere in order to induce abnormal grain growth, followed by air cooling to room temperature.

5.2.3. Characterization of weld microstructures

Micro-hardness maps were carried out on the weld cross sections using a Struers Durascan automatic indenter with a 0.5 kgf (HV\(_{0.5}\)). The maps were derived from lines of indentations with 0.5 mm spacings, using OriginPro software. The measurements were carried out on the weld cross sections of both the as-welded and the post-weld heat treated material. Samples for optical microscopy were mechanically polished down to 4000 grit SiC paper followed by polishing with 1 μm diamond paste and finally 0.025 μm silica (OP-S) suspension. The microstructures were revealed by chemical etching in a solution containing 15 vol.% HCl and 3 vol.% HNO\(_3\). Optical micrographs of the weld cross sections were taken using a Keyence VK-X200K 3D laser scanning confocal microscope or a Zeiss Axio Imager 2 microscope. Backscattered Electron (BSE) images and Electron BackScatter Diffraction (EBSD) maps were acquired using an FEI Quanta FEG scanning electron microscope with an accelerating voltage of 20 kV. The EBSD maps used to obtain the grain size distribution of the welds were collected using a step size of 0.8 μm. Two neighbouring grains were considered as distinct grains when misorientation angle between them was larger than 10°.
5.3. Results

5.3.1. As-welded structure characterization

Fig. 1. Optical micrographs (left) and hardness maps (right) of the as-welded cross sections. The weld number is indicated in the bottom left corner of each optical micrograph.

Fig. 1 (left) shows the cross section of the welds in the as-welded condition, and (right) the associated hardness maps of the cross sections. The base material presents two regions of distinct hardness due to the bimodal grain size distribution present through thickness in the as-received base material. The plates used for Weld 1 contained a larger proportion of the base material in the coarse grained state, and therefore presents a larger soft region at the top of the plate, than Welds 2 or 3. The hardness of the coarse grained base material is similar to the values measured in the stir zone for Welds 1 and 2. Softening was observed in all the welds when compared to the fine grained microstructure of the base material. The softening was more severe for the lower traverse speed used, and also towards the top and on the advancing side of the weld. The fine grained material immediately surrounding the stir zone presents a region of intermediate hardness between that of the stir zone and the base material, typically 1–2 mm thick.
**Fig. 2.** a: EBSD map of the cross section of Weld 2. The colour scale represents the crystallographic orientation relative to the welding direction. b: Grain size distribution for the three butt welds.

<table>
<thead>
<tr>
<th>Weld no. #</th>
<th>Traverse speed (mm/min)</th>
<th>Peak welding temperature (°C)</th>
<th>Mean grain diameter (µm)</th>
<th>No. grains analysed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>120</td>
<td>943</td>
<td>2.55(2)</td>
<td>6657</td>
</tr>
<tr>
<td>2</td>
<td>95</td>
<td>1074</td>
<td>2.65(2)</td>
<td>5957</td>
</tr>
<tr>
<td>3</td>
<td>70</td>
<td>1220</td>
<td>3.86(3)</td>
<td>7048</td>
</tr>
</tbody>
</table>

**Table 2.** Mean grain size of the three studied friction stir welds with varying tool traverse speed, as determined from EBSD maps, together with the measured peak temperature during welding [31].
Fig. 2 shows a representative EBSD map of Weld 2 together with the grain size distribution obtained for the three welds, measured at depth 1.5 mm from the top of the plates at the joint, i.e. the centre of the weld. The results reveal that the welds all have a continuous distribution of grain diameters between ~1–10 µm with the majority of grains having a diameter ~1–4 µm. Weld 3 contains a significantly higher proportion of grains larger than 4 µm. The mean grain diameter increases with decreasing traverse speed, see Table 2. The associated pole figures are displayed in Fig. 3. The three welds present weak bcc torsional textures [29, 30] with a <111> component along the welding direction. This is in contrast to the (100)<110> rolling texture of the fine-grained base material [17, 31]. The EBSD maps and pole figures were created using the HKL Channel 5 Tango and Mambo data processing software [32]. The pole figures used a half width of 10° and a cluster size of 5°.

Fig. 3. Pole figures of the 3 butt welds with units in multiples times random. Weld number is indicated on the left. WD and TD are the Weld direction and Transverse direction, respectively.
The variation in grain size across the welds was investigated via optical microscopy at five regions across the weld cross section, see Fig. 4 and Table 3. The values of the mean grain size were relatively similar for all welds at the centre middle and centre bottom regions. However significant differences were observed in the top locations of the welds. There was a clear trend for the grain sizes to increase with decreasing traverse speed. The largest grain sizes consistently occurred at the top advancing side region for the three welds. Furthermore, the microstructure at the border presents an abrupt change relative to the stir zone (Fig. 5). Although the recrystallized weld border region is also comprised of quasi-equiaxed grains, there are a number of shear bands in which the typical grain size is significantly different from the neighbouring bands and from the stir zone itself. This banded region between the stir zone and base material corresponds to the thermo-mechanically affected zone (TMAZ). In addition, there is also a region adjacent to the TMAZ border that comprises of the base material, which has not been recrystallized by the welding process, however still shows definite signs of deformation. The different regions have been labelled in Fig. 5b. Fig. 5c shows a similar region, but with the deformed coarse grained base material adjacent to the TMAZ.
Table 3. Average grain size obtained from optical micrographs at the selected locations of the weld cross section indicated in Figure 4. \(d\) is the mean grain diameter with the standard error provided in the parentheses. \(\sigma\) is the standard deviation of the grain diameter distribution. Units are in micrometres.

### Weld #1

<table>
<thead>
<tr>
<th>Position</th>
<th>Retreating</th>
<th>Centre</th>
<th>Advancing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(d) (\sigma)</td>
<td>(D) (\sigma)</td>
<td>(d) (\sigma)</td>
</tr>
<tr>
<td>Top</td>
<td>5.5(3) 1.3</td>
<td>6.1(4) 1.7</td>
<td>6.4(3) 1.4</td>
</tr>
<tr>
<td>Middle</td>
<td>4.2(2) 1.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bottom</td>
<td>4.5(2) 1.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Weld #2

<table>
<thead>
<tr>
<th>Position</th>
<th>Retreating</th>
<th>Centre</th>
<th>Advancing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(d) (\sigma)</td>
<td>(D) (\sigma)</td>
<td>(d) (\sigma)</td>
</tr>
<tr>
<td>Top</td>
<td>5.6(3) 1.4</td>
<td>6.2(3) 1.3</td>
<td>7.3(4) 1.7</td>
</tr>
<tr>
<td>Middle</td>
<td>5.8(4) 1.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bottom</td>
<td>4.2(2) 1.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Weld #3

<table>
<thead>
<tr>
<th>Position</th>
<th>Retreating</th>
<th>Centre</th>
<th>Advancing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(d) (\sigma)</td>
<td>(D) (\sigma)</td>
<td>(d) (\sigma)</td>
</tr>
<tr>
<td>Top</td>
<td>11.9(8) 3.4</td>
<td>8.8(5) 1.9</td>
<td>18(1) 6</td>
</tr>
<tr>
<td>Middle</td>
<td>6.0(2) 0.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bottom</td>
<td>4.1(3) 1.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Fig. 4.** **Top:** optical cross section image of Weld 1. **Bottom:** representative optical micrographs of microstructure from the regions denoted by the inserted boxes in the top optical image.
Fig. 5. Microstructure at the weld borders. a: optical micrograph of bead-on-plate weld, b and c: back-scattered electron image of Welds 1 and 3, respectively. The top of the weld is towards the top and the advancing side is to the right of all of the micrographs. The red labels in b denote i: stir zone, ii: thermo-mechanically affected zone, iii: deformed base material and iv: base material.
5.3.2. Post-weld heat treated structure

Fig. 6. Left: Optical micrographs and right: hardness maps of the heat-treated weld cross sections. The weld number is indicated in the bottom left corner of each optical micrograph.

Fig. 6 shows the cross sections and corresponding hardness maps of the welds after the post-weld heat treatment for 1 hour at 1380 °C. The optical micrographs on the left of the figure confirm that abnormal grain growth has been induced in the three welds. The stir zone is comprised of very coarse grains in the order of millimetres or several hundreds of micrometres; the exception to this being the bottom of the stir zone for Weld 3, particularly on the retreating side. The coarse grains are in general larger in the heat treated welds that used a lower traverse speed, although the total number of coarse grains is not high enough for statistically relevant data. Moreover, the process has caused a significant reduction in hardness in both the base material and the welded regions. Welds 1 and 2 are characterized by highly homogenous hardness values across their entire cross section, whereas the hardness in the stir zone of Weld 3 remains significantly lower than in the surrounding base material. The exception to the homogenous hardness distribution across the cross sections of Welds 1 and 2, is a region with higher hardness values located along the weld borders, also present in Weld 3. This region is significantly narrower in Weld 1 than for the other two welds. This border of relatively greater hardness is comprised of a microstructure that is reminiscent of the TMAZ before heat treatment in all the welds, see Fig. 7, and therefore seems to have resisted abnormal grain growth during the post-weld heat treatment. The remainder of the region presenting greater hardness corresponds to the base material that was plastically deformed by the FSW process, as depicted by band iii in Fig. 5.
Fig. 7. Micrographs of the weld boarders after heat treatment for 1 hour at 1380 °C. a: Back scattered electron image of Weld 2 boarder on the advancing side, showing coarse grains at the top and below the thermo-mechanical affected zone that resisted abnormal grain growth. Optical micrographs of the weld boarders: b: Weld 2 advancing side, c: Weld 3 retreating side, close to top of weld, d: Weld 3 advancing side.

5.4. Discussion

5.4.1. As-welded structure

A lower traverse speed of the welding tool leads to a higher peak temperature during the FSW process, see Table 1. The welds exhibited a coarser grain structure and lower micro-hardness values when a lower traverse speed was used. The additional thermal energy will assist in the growth of the grains in the wake of the tool, following dynamic recrystallization. Besides that, higher local temperatures either dissolve or agglomerate a greater proportion of the nano-sized pinning particles, as evidenced by our previous Small Angle Neutron Scattering data [22]. The Zener-pinning limiting grain size ($D_{\text{Zener}}$) is given by [33]:

$$D_{\text{Zener}} = \frac{4}{3} \frac{\bar{r}}{f_v} \tag{1}$$

where \(\bar{r}\) denotes the mean particle radius and \(f_v\) the volume fraction of particles. A lower tool traverse speed is able to retain a lower number of pinning particles and the Zener-pinning
limiting grain size in the stir zone is therefore increased. Consequently, both a coarser grain size and larger reduction in the number of effective pinning particles with nanometre dimensions leads to lower hardness values in the case of a lower tool traverse speed. The increased heat will also be the reason why Weld 3, with the slowest traverse speed, produced a larger TMAZ and total weld width, since a greater volume of material was plasticised and hence could be stirred. The volume of deformed base material surrounding the welded structures was also greater for the two welds with lower traverse speed, namely Weld 2 and 3.

The temperature distribution in the weld is also the main cause for the observed variation in microstructure and hardness across the individual welds. The dominant source of heat during FSW is the friction of the plate with the shoulder of the tool. The fastest cooling rate will occur between the bottom of the plate and the steel backing plate, which would act as an effective heat sink [34, 35]. Therefore, the highest temperatures during welding will occur towards the top of the plate, particularly on the advancing side of the weld where the relative velocity of the tool shoulder is greatest [15, 36, 37]. The experimental data for the average grain size across the stir zone agrees with the expected temperature profile in the weld: in all investigated welds, the highest mean size was detected at the top advancing side of the stir, whereas the lowest corresponded to the bottom of the weld.

Furthermore, shear banding occurs in the border region of the three welds produced. This phenomenon has also been observed in FSW in analogous regions in other steel grades [38, 39] and also in aluminium using the “stop action technique” [40]. Each band is indicative of the material being sheared by each rotation of the tool as it traverses along the weld line. Shear bands are often not observed in these regions or are less stark than those observed in the ODS steel welds of this study. The nucleation of deformation bands may be easier in this ODS steel due to the presence of micron-sized Ti(C,N) inclusions that can act as nucleation sites [41]. Additionally, the retention of the banded structure can be achieved by the nano-sized particles present in this region of the weld, and consequently thermally-assisted grain migration is hindered.

5.4.2. Abnormal grain growth behaviour

Abnormal grain growth was observed in the three heat treated welds. The AGG was complete throughout the entirety of the stir zone, except for Weld 3 where the bottom lower section of the stir zone resisted AGG. FSW generates large gradients in plastic deformation and temperature inside the weld, together with a relatively complex material flow that
depends on the geometry of the welding tool, and consequently an inhomogeneous weld microstructure [15]. The surface layer of the workpiece is dragged by the shoulder of the welding tool, and is deposited behind the shoulder edge. This high deformation of the surface layer of the workpiece at the highest temperature would trigger the formation of a fine-grained surface microstructure [42, 43]. We have observed such a fine-grained surface layer in the ODS steel welds of this study, see Fig. 8, despite the remainder of the upper section containing the coarsest grains. Abnormal grain growth is expected to start at the near-surface region of the workpiece, especially close to the boundary between the previously mentioned surface layer and the stir zone of the weld [43]. The upper part of the stir zone is subject to an additional deformation, due to the specific material flow on the surface layer close to the welding tool. This additional deformation has been reported to locally increase the fraction of low-angle grain boundaries [42]. The heterogeneous grain boundary structure and size distribution in the workpiece close to the top surface seems to induce the AGG initiation in the upper region of the stir zone during the heat treatment, assisted by relatively large strain gradients and other local microstructural heterogeneities [43-46]. This agrees well with the AGG grain structure and morphology observed in Fig. 6. The preferred grains with lower strain energy are expected to grow downwards into the stir zone by consuming grains of higher strain energy.

Fig. 8. Optical micrograph of the top part of the cross section of Weld #2.
Weld 3 corresponds to the lowest traverse speed of the welding tool, and the conceivably greater displacement of particles towards the bottom of the stir zone, due to a greater amount of stirring, could have significantly increased the pinning forces on the grain boundaries and hence why this region resisted AGG. This weld was also at an elevated temperature during the welding process for a significantly longer period of time than Welds 1 and 2. This gave the stresses created during the weld more time to relax. Hence Weld 3 would have smaller strain gradients across the weld, and this was indeed evidenced by our previous neutron diffraction measurements [31]. Therefore there would have been a lower driving force for AGG [43-45].

Lower down the weld, AGG also appeared to initiate at the weld borders, due to heterogeneities in the microstructure at these locations and high stored energies at the shear bands of the TMAZ. However, the TMAZ itself predominantly resisted AGG. This may be ascribed to the dense particle build-ups that were commonly observed in these regions, see Fig. 9. Those particle build-ups would locally impose an increase in pinning force on the grain boundaries [43, 47].

Fig. 9. Optical micrographs showing dense particle build ups close to weld boarders.
The microstructure in the deformed region of the base material appears to have rotated to align along the material flow direction from the FSW process. Rather than simply rotating about the tool, this material is forced downward on the retreating side and upward on the advancing side [15], evidence of which can be observed in Fig. 5. However, the highly homogeneous grain structure and presumably also the particle distribution of the base material have been retained [17, 48]. This will mean that grain growth in this region will still be highly impeded. Explosive columnar grain growth occurs in MA956 ODS steel, since particle alignment along the extrusion direction created a favoured growth direction [49]. It seems as though the rotation of the base material adjacent to the weld alters the prior particle alignment, and therefore the favoured growth direction disappears and AGG is hindered.

5.4. Conclusions

We have characterised the microstructure of three MA956 ODS steel friction stir welds produced using a different traverse speed of the welding tool, and afterwards we have investigated their abnormal grain growth behaviour following a short high temperature heat treatment. The main findings of this study are:

(i) The mean grain size in the stir zone increased with a decreasing tool traverse speed, and was also larger close to the top of the weld and on the advancing side, due to higher local temperatures and increased particle dissolution or coarsening. This is manifested in the hardness maps that present lower values in regions with larger grain sizes.

(ii) The post-weld heat treatment for 1 hour at 1380 °C was able to induce abnormal grain growth. As a result, a highly coarse grain structure was observed in both the base material and the stir zone of the three studied welds. The grain structure and morphology suggest that abnormal grain growth initiated at the top of the stir zone, close to the fine-grained surface layer, and the preferred grains grew downwards into the stir zone at the expense of grains with higher strain energy.

(iii) The border region between the stir zone and the base material is composed of shear bands with a distinct microstructure. Those bands can be assigned to the thermo-mechanical affected zone. The fine microstructure was retained after the heat treatment around the weld boarder in the TMAZ and directly adjacent base material. The fine grain structure was also retained at the bottom of the stir zone for the weld of the slowest traverse speed. This resistance to abnormal grain growth was attributed to particle pile-ups in those regions.
Acknowledgements

We gratefully acknowledge the financial support of the Engineering and Physical Sciences Research Council UK (EPSRC) through the Centre for Doctoral Training in Advanced Metallic Systems under the Grant Agreement EP/L016273/1. This work contributes to the Joint Programme on Nuclear Materials (JPNM) of the European Energy Research Alliance.

References
[32] Channel 5 Tango (Mapping) & Mambo (Pole Figures), in, HKL.


Chapter 6

6. Final conclusions and future work

6.1. Conclusions

The microstructure of the ODS steel is significantly altered following friction stir welding. The grain size is different to the base material following the induced dynamic recrystallization, particularly compared to the coarse grained material. The welded region contains a weakly textured grain structure of quasi-equiaxed grains. This was observed in all welds. Additionally, a variance in the mean grain size was observed across the welds; with an increased grain size towards the top of the weld, more acutely on the advancing side of the welds. This is attributed to the greater temperatures at these locations creating greater grain boundary mobility from additional thermal energy and increased particle dissolution. However, the very top section, within very approximately 50 µm of the surface, contains relatively fine grains created by very high levels of strains in the material that “sticks” to the tool shoulder during FSW.

Measurable trends in the microstructure with the welding parameters have been identified. Mean grain size, mean particle size fraction increase for a lower traverse speed and for a faster rotation speed used. The volume fraction of particles in the welds followed the opposite trend. These trends were observed in the bead-on-plate welds. The butt welds, which extended the traverse speed range, also continued these correlations. Slower traverse speeds and greater rotation speed would create higher temperatures and mixing which would generate greater levels of dissolution and agglomeration. In the coldest welds, negligible dissolution of the particles was observed but significant agglomeration still occurred.

The micro-hardness exhibited by the welds was lower towards the top of the welds, especially on the advancing side, agreeing with the larger grain size observed in the regions and presumably a significantly lower volume fraction of particles. A clear relation in the micro-hardness of the stir zone with the welding parameters was observed; a lower hardness
present for a lower traverse speed or rotation speed used, due to a reduction in grain boundary and particle strengthening.

Tensile strengths of the welds were very similar to the tensile strength of the coarse grained base material. This arises from a greater grain boundary strengthening in the welds but decreased particle strengthening effect. The tensile testing was unable to reveal similar trend with relation to the welding parameters as the micro-hardness did.

Significant residual stresses were found in the butt welds investigated by neutron diffraction. The stresses were greater when a faster traverse speed was used. This is due to the fact that a faster traverse speed is associated with faster cooling rates, for all other factors held constant. The longer period over which the weld cools leads to lower thermal gradients and therefore creates relatively smaller residual stresses.

All of the welds, exposed to a subsequent very high heat treatment, experienced abnormal grain growth to produce very coarse grain structures, generally coarser for the welds using a slower traverse speed. The thermo-mechanically affected zones of the welds resisted abnormal grain growth, due largely to the presence of a relatively high particle density. The thermo-mechanically affected zone and consequently the size of the region that retained the relatively fine grain structure were larger for the reduced traverse.

6.2. Future work

Despite keen interest in the use of Friction stir welding for joining ODS steels we are still in the early stages of developing a comprehensive understanding of the process. A much greater level of investigation is required before implementation of ODS Friction stir welds is possible in future generation nuclear reactors. Indeed, this is also still the case for certain aspects of the proposed ODS steel base materials, particularly in predicting the effects of the unprecedentedly high levels of radiation on the components in fusion reactors.

Here I shall briefly discuss possible avenues of interest for research that extend from the results chapters included in this thesis:

With regards to the microstructural analysis of Chapter 2 and continued in Chapter 4, I believe an important future experiment would be mapping the distribution and volume fraction of nano-particles across the weld, ideally measuring the composition of particles with location too. It is important to fully assess what happens to the particles after welding. Understanding to what extent the particles have been dissolved, coarsened, agglomerated for
particular parameters will greatly assist in optimising the parameter window used for FSW; particularly ensuring that they are likely to be no areas of weakness across the weld – more susceptible to radiation damage or creep deformation etc. due to an insufficient volume of nano-particles or a low distribution homogeneity.

Mapping of the nano-particles would provide invaluable insights into the complex material and heat flow patterns experienced during FSW which is important for the understanding of all FSW projects, not just ODS steel, and may provide useful data to develop more accurate models for FSW.

To gain greater confidence and understanding in the tensile properties of the welds, a larger number of tested specimens are required. This may produce data from which one could observe any trends in mechanical strength that likely exist with the welding parameters. However, as was explained in Chapter 3, the MA956 alloy is in relatively short supply and destructive testing is undesirable. Performing similar tests with a small punch testing rig may alleviate this problem. Small punch testing is a mechanical testing technique that uses a relatively small amount of material to be tested. The mechanical properties can be derived from the load while a punch moves through a thin clamped disk of material being tested. This technique, if properly developed for ODS steels, would allow for a much greater number of tests for the same volume of welded ODS material.

A greater range of testing temperatures should also be studied, particularly in the range > 600 °C, which would be reminiscent of probable future nuclear environments. These temperatures would also see a greatly diminished effect of grain boundary strengthening, allowing one to more greatly isolate the effect of the post weld dispersion on the strengthening of the material.

An obvious continuation of the work from Chapter 4, on the residual stress distributions would be to investigate a larger set of welding parameters, particularly including a systematic change in rotation speed of the tool. However, with the limited parameters used so far, it seems that, in general, to reduce the residual stress, a hotter weld is required which maybe undesirable in terms of the microstructure post-weld. Therefore it might be of significant value to investigate alternative methods of mitigating significant residual stresses in the ODS steel welds. This may be by their removal, via means of post weld heat treatments, or it may be worthwhile investigating the possibility of reducing the residual stresses caused by FSW without altering the parameters. This may be possible by pre-heating the plates to be welded
or by changing the thermal conductivity of the backing plate. Or by more novel methods such as ultrasonic vibration enhanced friction stir welding (UVeFSW) [1]. UVeFSW may also be able to improve the material flow during welding by lowering the viscosity of the material and therefore greatly reduce the chance of tunnel defects [2][3]. This would allow for a broadening of the parameter window for defect free joints, in which the other microstructural properties can be optimized for high temperature nuclear applications. Due to the limited ODS material available, these investigations would best be initially further developed on other, less specialised alloys.

To further investigate the abnormal grain growth behaviour of the welds it would be useful to conduct a similar investigation but using several heat treatment temperatures and a range of heating times at which to observe the microstructures. Some of these times should be relatively short as to capture the initiation and intermediate stages of abnormal grain growth across the weld. With this one could observe, when and where nucleation occurs and how the following grain boundary migration proceeds. Coupling with the parameters and starting microstructures would allow for a deeper understanding of the process and potentially lead to great control over the coarse grain structures producible in the welds.

Two critically important properties of the ODS steel welds that were not investigated in this thesis are the creep and radiation damage resistance. These have been and are currently being investigated for the base ODS alloys; however, data for welds in these areas is particularly lacking in the literature. Both of these properties are heavily reliant on changes to the particle dispersion and grain structures which are significantly altered by FSW, so future research must be conducted here.

At the time of submission of this thesis, there are still a small number of in progress investigations being carried out by the author, on ODS steels and friction stir welding. While not included in this thesis, it is hoped, time permitting, that the work will be concluded and published in the relatively near future. This work includes investigations into the long term thermal stability of ODS steel Friction stir welds, into the magnetic behaviour of ODS steels depending on their microstructural condition, and how the microstructural condition affects the thermal helium desorption behaviour.
6.3. Publication of research


6.4. Contributions of authors

The main author, H. Dawson, wrote all sections of this thesis, with input from Enrique Jimenez-Melero. Named authors may have provided feedback on their respective research chapters in which they are included.

All experimentation and data analysis was carried out by the main author, with the following exceptions:

The neutron scattering experiments were all performed as part of a group. The SANS experiment performed at BNC, Chapter 2, was carried out by H. Dawson, Q. Tian and L. Almásy. The data handling of the SANS data was performed by Q. Tian and L. Almásy. In Chapter 3, the scanning electron micrographs of the fracture surfaces were captured by R. Hernandez at CIEMAT, Spain. And the residual stress measurements performed at ILL, Chapter 4, were conducted by H. Dawson, P. Wady, E. Jimenez-Melero and T. Pirling.
References


Chapter 7

7. Appendix

Due to the alternative submission format used in this thesis, some potentially useful information was omitted from the papers sent for publication due to criteria and limits set by the journals. This Chapter includes further pertinent data and figures on the subject of FSW of ODS steels.

Figs. 1 to 5 pertain to the FSW process. This includes images of the FSW machine and tool used for all the welds, carried out at TWI.

Figs. 6 to 19 and Table 1 provide further information on the various ODS microstructures presented in this thesis. For these, a range of microscopy techniques were used: optical microscopy, transmission electron microscopy and scanning electron microscopy, including EBSD. All micrographs were taken by H. Dawson who also produced and analysed the data for Table 1 and Figs. 19 and 20. Figs. 6 to 9, along with Table 1, are specifically related to Chapter 5; investigating a changing mean grain size across the weld cross section and investigating the microstructures at the TMAZ-HAZ interface, in both the as-welded and heat-treated conditions. It should be noted that Fig. 6, 7, and 8c are of bead-on-plate welds, as opposed to the butt welds used in Chapter 5. Fig. 19 contains inverse pole figures (IPFs) for the ODS steel base material and the 3 butt welds. The base material IPF (a) is the corollary, i.e. calculated using the same data, of the base material pole figure provided in Chapter 4, Fig. 1. The butt weld IPFs (b-d) are the corollary of the butt weld pole figures provided in Chapter 5, Fig. 3.

Figs. 20 to 26 compliment Chapter 3 of this thesis, on the mechanical behaviour of the ODS steel and the welds. Figs. 21 to 25 are further micrographs of fracture surfaces of the tensile specimens taken using a scanning electron microscope. The micrographs were taken by R. Hernandez at CIEMAT, Spain. The optical macrographs of the fractured specimens, shown in Fig. 26 and inserted in Fig. 25, were taken by H. Dawson, also at CIEMAT.
The final figure, Fig. 27, contains two further images of the experimental setup of the measuring of residual stresses in the welds, described in Chapter 4 and carried out at ILL, France. The beamline experiment was conducted by H. Dawson, P. Wady, T. Pirling and E. Jimenez-Melero.

![Image](image1.png)

**Fig. 1.** Image of TWI’s MTI RM-2 Precision Spindle FSW machine used for all welds. Located at TWI Technology Centre – Yorkshire. It is a gantry style FSW machine manufactured by Transformation Technologies Inc. (now MTI) of Elkhart, Indiana, USA.
Fig. 2. Image of the type of ceramic tool used for all welds. The tool used was a MegaStir PCBN tool, which uses particles of boron nitride embedded in a tungsten rhenium based matrix. We used a Q70 tool which is 70% boron nitride.

Fig. 3. (a) Setup of FSW and (b) FSW in progress, for butt Weld 3.
Fig. 4. Measured welding parameters during FSW of butt Weld 2 as a function of distance along the weld.

Fig. 5. Image of surface finish following FSW for butt Weld 2.
Fig 6. Optical micrographs of bead-on-plate Weld 2. (a)-(c) at the TMAZ/BM interface on the advancing side, (d) in the SZ.
Fig. 7. Top: optical cross section image of bead-on-plate Weld 2. Below: representative optical micrographs of the microstructure from regions across the cross sections, indicated by the inserted boxes in the cross section in their respective positions.

<table>
<thead>
<tr>
<th>Position</th>
<th>Retreating</th>
<th>Centre</th>
<th>Advancing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>d</td>
<td>σ</td>
<td>d</td>
</tr>
<tr>
<td>Top</td>
<td>2.96(8)</td>
<td>0.37</td>
<td>5.0(2)</td>
</tr>
<tr>
<td>Middle</td>
<td>3.4(2)</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Bottom</td>
<td>2.78(9)</td>
<td>0.43</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Measured grain sizes at various locations across the LN plane of the bead-on-plate Weld 2, indicated in Fig. 7. d is the mean grain diameter with the standard error provided in the parentheses. σ is the standard deviation of the grain diameter. Units are in micrometres.
Fig. 8. Micrographs of the weld boarders. The top of the weld is towards the top and the advancing side is to the right of all of the images. (a) BSE image of butt Weld 1 taken with an accelerating voltage of 10 kV at a 6.4 mm working distance. This image is the same image as Fig. 5, in Chapter 5, however, without the annotations. (b) BSE image of butt Weld 3 taken with an accelerating voltage of 8 kV at a 6.8 mm working distance. (c) BSE image of bead-on-plate Weld 3 taken with an accelerating voltage of 20 kV at a 10.7 mm working distance.

Fig. 9. Optical micrographs of the butt welds following 1380 °C heat treatment for 1 hour. (a)-(b) Weld 2 on the advancing side. (c) Weld 3 on the retreating side.
Fig. 10. Microstructures of the MA956 base material. (a) BSE image of the fine grained base material taken on an FEI Magellan High Resolution FEG-SEM at 2 kV accelerating voltage. (a)-(b) EBSD maps acquired using an FEI Quanta 650 scanning electron microscope at 20 kV. Maps of (a) fine grained material using a 0.6 µm step size and (b) coarse grained material in the longitudinal-normal plane using a 9 µm step size. Specimen was fully recrystallized by an additional 90 minute heat treatment at 1380 °C using a Lenton tube furnace with an argon atmosphere. Both IPF scales represent the crystallographic orientation with respect to the normal direction (out of plane).
Fig. 11. BSE micrographs from the bead-on-plate SZ’s. Micrographs were taken at the centre of the stir zone approximately 1.5 mm from the weld surface, using an FEI Magellan High Resolution FEG-SEM. The associated Weld #’s are inserted in the figure.
Fig. 12. EBSD maps of the stir zones of Welds 4 and 5 in the transverse-longitudinal plane, from between 0.5–1.5 mm depth from the top surface at the joint line i.e. the centre of the weld. Maps taken using an FEI Quanta 650 scanning electron microscope on 3 mm-diameter TEM discs were prepared by electropolishing at a temperature of \(-40\) °C, using a Tenupol 5 Jet electropolisher and an electrolyte comprising 90 vol.% methanol and 10 vol.% perchloric acid. The IPF colour scale is with respect to the normal direction, which is the only known direction for these specimens (out of plane).

Fig. 13. Etched microstructure of the stir zone of bead-on-plate #5 in the Transverse-Longitudinal plane. The image was taken close to the centre of the weld line on a sample face between 0.5–1.5 mm depth from the original top surface of the weld.
**Fig. 14.** Transmission electron micrographs of the fine grained base material. Micrographs taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.
**Fig. 15.** Various transmission electron micrographs of the SZ of the bead-on-plate Weld 1, taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.
Fig. 16. Various transmission electron micrographs of the SZ of the bead-on-plate Weld 3, taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.
Fig. 17. Various transmission electron micrographs of the SZ of the bead-on-plate Weld 4, taken on an FEI Tecnai 20 200 kV Analytical Electron Microscope.
Fig. 18. Grain size distribution of the stir zones of the bead-on-plate welds; provided in addition to Fig. 3b in Chapter 2.

Fig. 19. Inverse pole figures of (a) the fine grained BM from XRD data, b) Butt Weld 1, (c) Butt Weld 2, (d) Butt Weld 3. (b)-(d) Data from EBSD data collected using the same method and location on the sample as was used for Fig. 3(b) in Chapter 2. WD, TD and ND denote Welding Direction, Transverse Direction and Normal Direction, respectively.
Fig. 20. Stress-strain graphs of the tensile specimens tested at room temperature.

Fig. 21. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at RT, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.
Fig. 22. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at 150 °C, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

Fig. 23. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at 300 °C, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.
Fig. 24. Scanning electron micrographs of the fracture surfaces of the BM tensile specimens tested at 600 °C, taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.

Fig. 25. Scanning electron micrographs of the fracture surface of the bead-on-plate welds at 500 °C. (a) Weld 1, (b) Weld 4 and (c) Weld 5. Inserted are macroscopic fracture surfaces (top) and optical micrographs of fractured dog bone specimens (bottom). Images taken on a Carl Zeiss Auriga Compact field effect SEM using an accelerating voltage of 25 kV.
Fig. 26. Fractured tensile specimens: (a) Weld 1 at RT, (b) Weld 1 at 500 °C, (c) Weld 3 at RT and (d) Weld 3 at 500 °C. Welds are bead-on-plate welds as featured in Chapter 3.
Fig. 27. Images of the setup at the SALSA beamline at ILL, France, for measuring the residual stresses of the welded ODS steel plates by neutron diffraction.