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

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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. The mean grain size at all measured locations increased with a lower 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 thermo-mechanically affected zone, and a deformed region of the base material, was clearly observed for all welds at the weld border. 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.

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 to 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

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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].

Highly homogenous structures, where the majority of grains have a low driving force for growth, can allow a few preferred grains, with higher driving force, to grow at the expense of the other grains leading to AGG, if there is enough energy available i.e. at high temperature [27]. Inhomogeneous distributions of pinning particles and solutes reduce grain boundary mobility and can contribute to the conditions that lead to AGG by inhibiting local normal grain growth, while also resisting AGG. This is why very high temperatures are often required to destabilize or coarsen those particles by Ostwald ripening [28], and consequently to initiate the AGG process. Preferred or abnormal grain growth would therefore occur locally where the pinning points for grain boundary mobility have been affected or removed, and the local grain structure destabilized. An analytical model proposed to describe the annealing phenomena of cellular microstructures, based on the migration of high- and low-angle grain boundaries, allows to predict the minimum radius of a given grain with respect to the average grain size to undergo AGG as a function of the second phase pinning particle characteristics, namely the volume fraction and average particle diameter [29]. Furthermore, experimental results on the annealing behaviour of undeformed and deformed samples of pure iron revealed that undeformed samples undergo a continuous, normal growth, whereas a stage of abnormal grain growth is observed in the deformed samples. In fact, the start of abnormal grain growth shortens as the applied strain increases [30].

AGG has been observed in friction stir welded aluminium alloys subjected to post-weld heat treatments [31–35] While the process is complicated and many factors can contribute; the origin of AGG in these circumstances has been attributed to the presence of small grains with a large fraction of mobile high angle boundaries as a result of primary recrystallization [32,36]. If second phase particles are present in the welded material these can also be influential in the AGG process by stabilizing grain boundaries. AGG in FSWed aluminium has been reported to occur at temperatures where the particles significantly coarsen or enter solid solution [37,38].

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 h at 1300 °C, which did not induce AGG and produced only a subtle grain growth effect. ODS steels can retain a significant proportion of their particle dispersions [21,22,39], which likely contributes to the high temperatures required to initiate AGG, particularly as the particles are stable up to very high temperatures.

2. Materials and method

2.1. Base material

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 Y_2O_3 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 h at 1320 °C and was finally air cooled to room temperature [17,40,41]. 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 characterized by primary recrystallized grains (~1–2 μm) that are slightly elongated along the extrusion direction.

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. No significant variations in welding tool geometry were observed from weld to weld. An image of the tool prior to the welding process is shown in Fig. 1. An argon shielding gas was used 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 $\sim 40 \times 4 \times 2\,mm^3$. Those specimens were heat treated for 1-hour at 1380 °C in an argon atmosphere, in order to induce abnormal grain growth, followed by air cooling to room temperature.

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-X 200 K 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°.

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

Cr	Al	Y_2O_3	P	Ti	O	C	Mn	Si	Mo	Ni	Co	N	Cu	S	Fe
19.97	4.44	0.53	0.53	0.33	0.21	0.15	0.11	0.05	< 0.05	0.04	0.03	0.022	0.009	0.004	bal.