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Solid state diffusion bonding of ODS Eurofer steel by spark plasma sintering

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Abstract

Oxide dispersion strengthened (ODS) steels are considered to be one of the candidate structural materials for advanced nuclear applications due to their high elevated-temperature strength, corrosion resistance and radiation tolerance. Joining of ODS steels by traditional fusion joining techniques is not applicable, because the melting process results in the coarsening of fine grains and agglomeration of nanosized oxide particles, and consequently a significant loss of strength. Spark plasma sintering (SPS) has recently been employed as a novel joining technique, which could be beneficial for joining ODS steels considering the solid state characteristic. A powder metallurgy prepared ODS Eurofer steel was successfully joined using SPS. The microstructure and mechanical properties of the joints were investigated. An almost defect-free joint was obtained at the selected processing condition. The tensile properties of the joints are comparable to the base material. Fracture analysis shows an intergranular fracture in the as-joined sample, while a ductile fracture with well-defined dimples is found in the tempered sample.

Keywords: Oxide dispersion strengthened alloy; Spark plasma sintering; Joining; Mechanical properties

1. Introduction

Oxide dispersion strengthened (ODS) steels are considered to be promising structural materials for advanced nuclear applications, because of their high elevated-temperature strength, creep strength and radiation damage resistance [1]. Favourable properties are mainly attributed to featured microstructures consisting of fine grains and homogenously dispersed nanosized oxide particles [2]. In order to apply ODS steels to large and complicated structures, reliable joining techniques are inevitable and essential. However, conventional fusion welding techniques, such as arc welding and electron beam welding, are problematic for joining ODS steels [3, 4]. These processes will cause a degradation in the mechanical performance of the joints due to grain growth and oxide particle agglomeration [5].

Diffusion bonding has the advantages of a low bonding temperature and minimum microstructure deterioration [6], which could be beneficial to join ODS steels. Diffusion bonding can be realised by spark plasma sintering (SPS), which is a novel sintering technique utilising uniaxial force and a pulsed direct electrical current under low atmospheric pressure [7]. During SPS, bonding occurs due to electrical resistance at the interface and Joule heating throughout the sintering process [8]. More importantly, the whole process occurs in the solid state, therefore prevents or minimises the degradation of featured microstructures including nanostructures and nanoparticles in the joint [9]. Recently, the SPS technique has been employed for joining similar and dissimilar materials. Yang *et al.* [10] joined 316L stainless steel samples using SPS. Parameters including heating power, heating time, pressure, pulse sequence, particle size and powder quantity were found to affect the joining process and joint behaviour. Miriyev *et al.* [11] successfully joined 4330 steel and Ti-6Al-4V alloy where a maximum tensile strength of about 250 MPa was obtained for joining at 1223 K for 1 h. TiAl intermetallics were successfully joined using SPS by Zhao *et al.* [6]. Examination of the effects of bonding conditions on the microstructure and mechanical properties of the joints showed that metallurgical bonding could be obtained at lower pressures and shorter times compared to traditional bonding techniques. So far, little information is available on using SPS to join ODS steels.

In this study, solid state diffusion bonding was achieved using spark plasma sintering to join ODS Eurofer, which is based on European reduced activation ferritic-martensitic (RAFM) reference steel Eurofer 97, reinforced with 0.3 wt% Y_2O_3 nanoparticles. The microstructure and mechanical properties of the joints were investigated by means of optical microscopy (OM), scanning electron microscopy (SEM) and tensile testing to evaluate the feasibility of SPS to join ODS Eurofer.

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2. Experimental details

The starting powders were mixed according to the nominal composition of ODS Eurofer (Fe-9Cr-1.1W-0.4Mn-0.2V-0.12Ta-0.3Y_2O_3 wt%). The blended powders were milled at 300 rpm for 30 h in a Retsch planetary ball mill in an Ar atmosphere. The mechanical alloyed powders were then consolidated by SPS at 1373 K with a pressure of 80 MPa under vacuum in a graphite mould. After a holding time of 30 min, disks with a diameter of 20 mm and a thickness of around 17 mm were produced. The processing parameters were selected based on our previous study [12].

Prior to joining, the bottom surface of the material was ground and polished to ensure a flat surface. Afterwards, two disks with a thickness of 15 mm were placed against each other with a layer of 4 g mechanical alloyed ODS Eurofer powder added in between (Fig. 1a). The specimens were sintered at 1373 K with a pressure of 80 MPa and a holding time of 40 min. In order to restore the ductility of the material, a post heat treatment was conducted by normalising at 1423 K for 1 h, air cooling to room temperature and then tempering at 973 K for 1 h, followed by air cooling to room temperature. Vickers microhardness was measured from the top surface to the bottom surface of the samples, with a load of 0.3 kg and a step size of 0.5 mm.

The joined samples were machined for microstructure examination and tensile properties investigation as illustrated in Fig. 1b. The microstructure of the joint was characterised using a Keyence Digital Microscope VHX-5000 and a JEOL 6500F scanning electron microscope (SEM), equipped with an energy dispersive spectrometer (EDS) system. Tensile testing was conducted using an Instron 5500R machine, with a nominal strain rate of 2.5×10^{-4} s^{-1} at room temperature and repeated for at least five times for the as-joined and heat-treated condition.

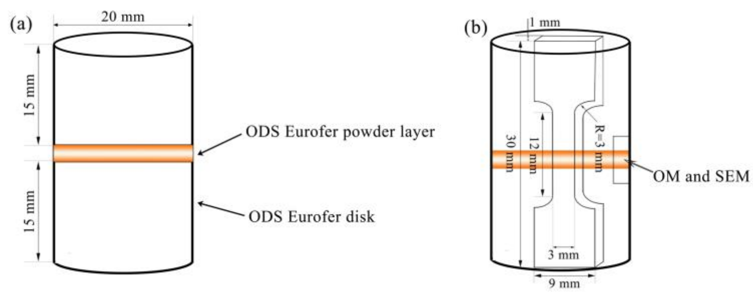

Fig. 1. (a) schematic of the preparation of ODS Eurofer joints and (b) locations and dimensions of the specimens for microstructure characterisation and tensile testing.

3. Results and discussion

Fig. 2 shows an optical micrograph of a cross-section of the as-produced joint. An almost defect-free joint is obtained at the selected condition. Cracks tend to initiate at the edges of the sample, probably due to the mismatch between the interfaces, and stop propagation until reaching approximately 0.4 mm. No heat-affected zone or evident interface is observed in the microstructure.

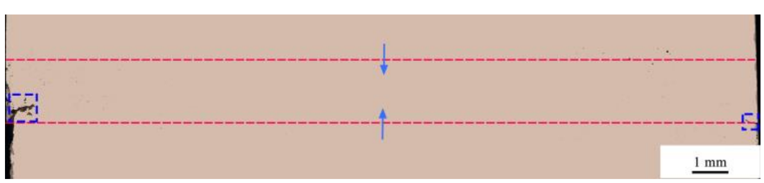

Fig. 2. Morphology of a cross-sectioned joint. The dashed red lines indicate the original interfaces; the dashed blue frames indicate the joining defects; the blue arrows indicate the locations of EDS measurement.

Fig. 3 shows the EDS line scanning results of the upper and lower interfaces of the as-produced joint (step size 2.5 μm), as illustrated in Fig. 2. It can be seen that the chemical composition of the material is very close to the intended composition. Moreover, the chemical composition of the interfaces does not show a significant change compared to the base material, indicating a homogenous and consistent composition across the joint. In addition, according to our previous study [13], the fine-grained features and nanoparticle distribution in the material show no significant change after the joining process, all of which are crucial for the mechanical properties of the joints.

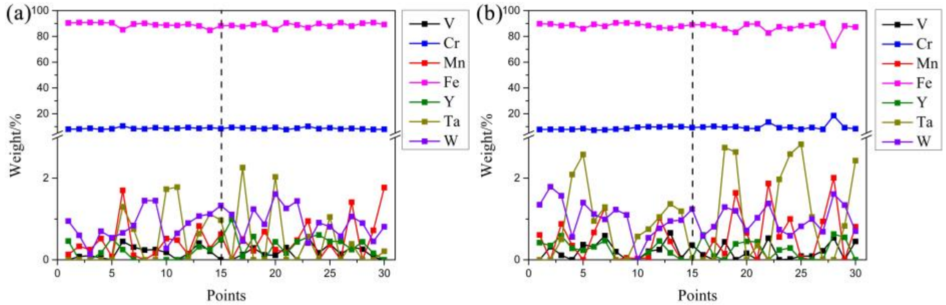

Fig. 3. EDS results of the (a) upper and (b) lower interfaces of the as-joined sample. (The dashed lines indicate the locations of the original interfaces.)

The microhardness of the cross-sections of the samples can be seen in Fig. 4. For the as-produced disk (thickness 17 mm), the hardness of the surface area is higher than the middle area due to a higher carbon content, resulting from the carbon diffusion from the graphite mould [12]. The hardness of the as-joined sample is higher than the as-produced disk at the same location, which could be ascribed to an additional thermal cycle and a rapid cooling during the joining procedure. In addition, the hardness of the interface region has a higher hardness than part of the base material, indicating that the joining interface is not the weakest point of the as-produced joint. After a heat treatment of normalising and tempering, the hardness of the joined material becomes more uniform, due to carbon diffusion and dislocation recovery.

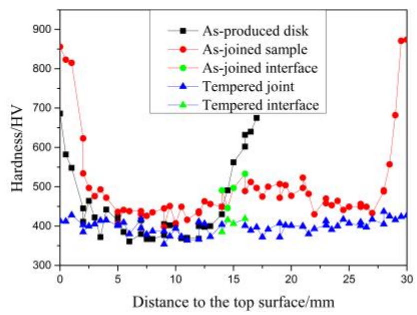

Fig. 4. Microhardness of the cross-sections of the as-produced disk and joined samples.

The tensile properties of the as-joined and tempered specimens are shown in Fig. 5. All of the tested specimens fractured in the base material region (Fig. 6). After a heat treatment of normalising and tempering, the strength of the material slightly decreases, while the elongation increases compared to the as-joined specimen. The tempering treatment favours the recovery of dislocations generated during the mechanical alloying and joining process, leading to enhanced ductility. The tensile properties of the base material obtained from our previous study are also listed in this figure [12], and it should be emphasized that the direction of the applied tensile force is perpendicular to the one in this study. It can be seen that the strength of the joined specimens is slightly higher, while the elongation is lower than that of the base material, which could be ascribed to a higher hardness of the joining material compared to the base material. It is known that the strengthening mechanisms of ODS steels are mainly based on grain boundary strengthening and dispersion strengthening [14], as neither the grain size nor the nanoparticle distribution is changed significantly after joining, the strength of the joint is therefore well retained.

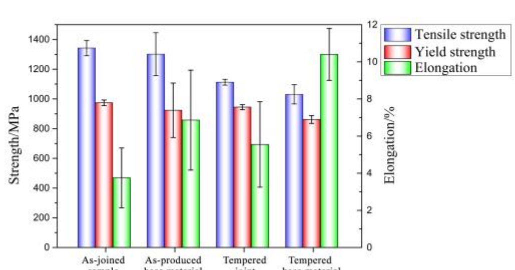

Fig. 5. Tensile testing results of the joined material and base material

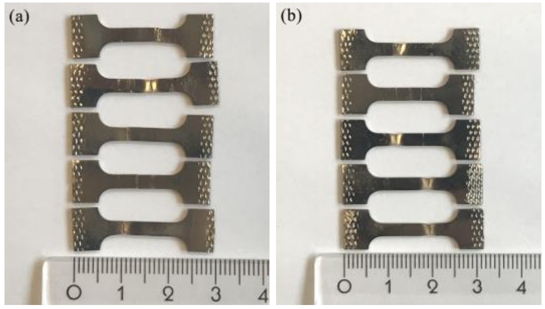

Fig. 6. Fractured specimens after tensile testing: (a) as-joined sample and (b) tempered joint

Fractographic examinations were performed on the fracture surfaces from tensile testing by SEM (Fig. 7). It can be seen that the fracture of the as-joined sample exhibits many small and smooth facets, indicating an intergranular fracture mode. In contrast, the tempered joint shows a typical ductile fracture mode with numerous dimple features, showing an enhanced ductility compared to the as-joined sample.

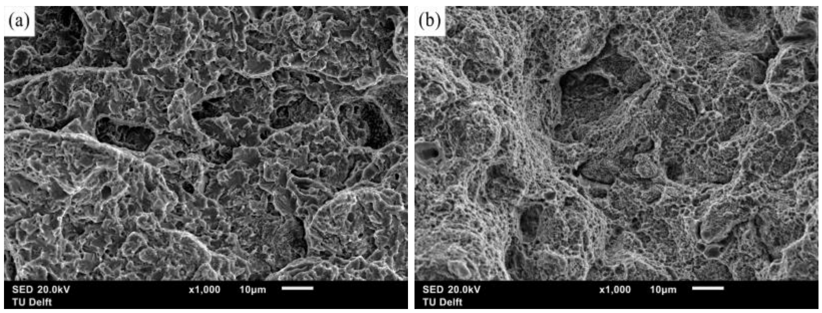

Fig. 7. SEM micrographs of the fractures for specimens after tensile testing: (a) as-joined sample and (b) tempered joint

Joining of ODS steels by fusion welding techniques such as electron beam welding and tungsten inert gas welding usually causes a significant loss of strength in the joint [3, 4]. By employing solid state joining methods such as hot isostatic pressing (HIP) and friction stir welding (FSW), the strength of the obtained joints can be as high as that of the base material [15, 16]. However, the cost of HIP is relatively high due to a longer processing time (1–5 h) compared to SPS [6]. The application of FSW is limited due to geometrical restrictions [4]. In addition, high residual stresses can be generated in the thermo-mechanically affected zone of the joining area, which will cause a reduction in the fatigue strength of the material [17]. Comparatively, SPS shows potential to be employed as a joining technique for ODS steels, considering the advantages of short processing time, ease of operation and good mechanical properties of the joints obtained.

4. Conclusions

An almost defect-free joint of ODS Eurofer was obtained via spark plasma sintering at the selected processing condition. The chemical composition of the interfaces shows no significant change compared to the base material. The microhardness of the material is slightly increased after joining, with the minimum hardness in the base material region. The tensile properties of the joints are comparable to the base material in both as-produced and heat-treated condition. It is concluded that spark plasma sintering is a promising technique for joining ODS steels owing to the good mechanical properties of the joints. This study can contribute to the application of ODS steels on complex structures in advanced nuclear reactors.

Acknowledgements

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