

## Microstructure and mechanical properties of friction stir welded 9Cr ODS steel

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### 1. Introduction

Oxide dispersion strengthened (ODS) ferritic-martensitic (FM) steel containing 9 wt%Cr is a promising candidate material for high temperature components operating in aggressive environments such as nuclear fusion and fission systems because of the excellent elevated temperature strength, corrosion and radiation resistance. These characteristics come from microstructures consisting of fine grains and nano-oxide particles dispersed in high number density [1-2]. However, for more applications of ODS steel in nuclear systems, its weldability is the one of the barrier to be solved. It is well known that the welding of ODS steel with a conventional melting–solidification process is not adequate to reserve nano-oxide particles in the matrix homogeneously [3-4]. To reserve nano-oxide particles in the matrix homogeneously, friction stir welding (FSW) is the most promising technique to join ODS alloys.

In this study, the effects of FSW on the microstructure and mechanical properties of a ODS steel were studied to apply the FSW process to 9Cr ODS steels. Microstructures were observed by means of optical microscopy, electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). A tensile test and hardness test were carried out to the investigate mechanical properties.

### 2. Methods and Results

#### 2.1 Experimental procedure

The material used in this study was FM ODS steel (Fe(bal.)-9Cr-2W-0.2Ti-0.1C-0.35Y<sub>2</sub>O<sub>3</sub> in wt.%) and FM steel (Fe(bal.)-9Cr-1Mo-0.1C in wt.%). The test specimens were cut into a 90 mm length x 18 mm width x 0.5 mm thickness, with the longitudinal direction parallel to the rolling direction. The material of the tool used in this study is a W-12wt.% Co alloy. The rotating tool is plunged into the plate and moved along the welding direction. The ODS steel was friction stir processed at a tool traveling speed of 90 mm/min and tool rotating speed of 1500 rpm. Consequently, the process was done in parallel with the rolling direction. Tempering was carried out at 750°C for 1h after welding.

The samples for optical microscopy were mechanically wet ground and etched in a 3% HF + 3% HNO<sub>3</sub> + 94% H<sub>2</sub>O solution for 60seconds. The EBSD specimens were electro-polished in a 5% HClO<sub>4</sub> + 95% methanol solution in vol. % at 20V for 25 seconds at room temperature to remove the work-hardened surface induced by mechanical buff-polishing. The thin foil specimens for TEM observation were prepared by jet-polishing using 5% HClO<sub>4</sub> + 95% CH<sub>3</sub>OH at 25V at -40°C. The carbon extraction replication technique was used to investigate the nano-oxide particle size distribution.

To investigate the mechanical property differences between the base material (BM) and stirred zone (SZ), hardness and tensile tests were carried out. The hardness profile was measured on the transverse cross section, using a Vickers micro-hardness tester with 100gf load for 15 s. The tensile properties were evaluated with miniaturized tensile specimens (Fig. 1) at room temperature.

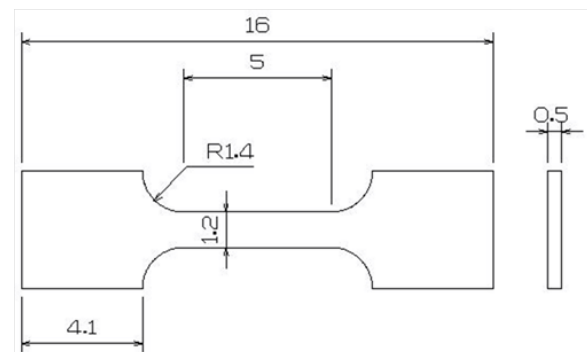


Fig. 1. Schematic illustration of tensile specimen.

#### 2.2 Microstructural evolution of friction stir processed ODS steel

Fig. 2 shows the friction stir welded ODS plate. A satisfactory weld bead was observed and an undercut sometimes occurring during the weld process was not observed.

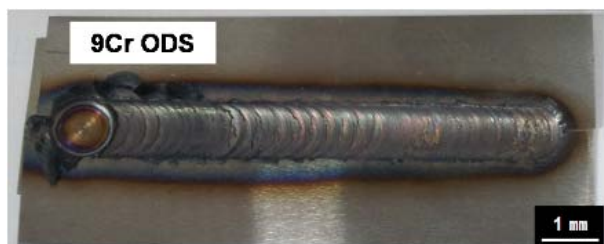


Fig. 2. Friction stir welded 9Cr ODS plate.

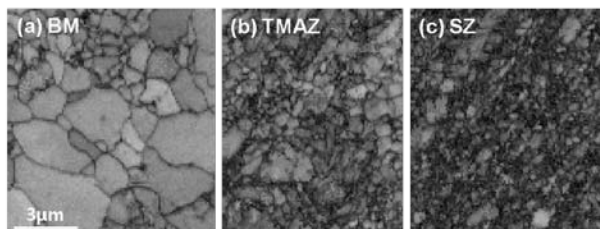


Fig. 3. EBSD grain boundary maps in BM (a), TMAZ (b) and SZ (c)

EBSD grain boundary maps from the BM to SZ were drawn in Fig. 3a-c. The grain size decreased towards the SZ.

### 2.3 Mechanical properties evolution of friction stir processed ODS steel

Fig. 4 shows the Vickers hardness profiles of the 9 Cr ODS weld and the FM steel weld. In the SZs before tempering, the hardness sharply decreased from SZ toward the BM. Under previous research of FM steel, the maximum temperature during FSW of steel lies around 1063 K and 1143 K of FM steel, so that austenite phases are partially or completely formed at maximum temperature, and then they transform into martensitic structure during cooling cycle of FSW [5].

The tensile test results of a FSWed ODS specimen deformed at room temperature are shown in Fig. 5. The 0.2% off-set yield strength (YS) of the weld was slightly higher than that of the base. On the other hand, the elongation of the base and weld had a similar value. The tensile strength (UTS) of the weld was 80% that of the BM.

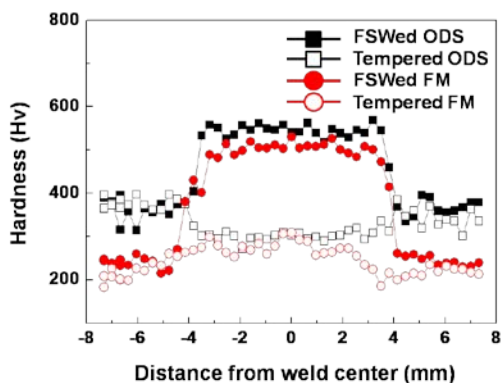


Fig. 4. Vickers hardness profiles of the welds for FSWed 9Cr ODS steel and 9Cr FM steel and after tempering of them at 0.2 mm depth from the weld top surface.

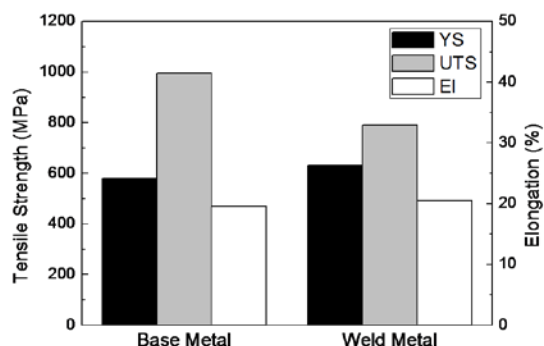


Fig. 5. Tensile properties of the base and FSWed ODS steel tested at room temperature.

### 3. Conclusions

FSW could successfully produce defect-free welds on ODS plates. FSW produced a fine grain structure consisting of ferrite and martensite. Tensile strengths and elongations of the SZs were excellent at 298 K, compared to those of the BM. This study suggests that FSW might be an appropriate welding method of ODS steels.

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