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Microstructure and texture evolution of cold-rolled ODS steel during annealing

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1. Introduction

Oxide dispersion strengthened (ODS) steels are being developed as a cladding material for sodium-cooled fast reactors (SFR). The thermally stable oxide particles dispersed in the matrix improve the irradiation and creep resistance at high temperature [1]. As a result, ODS steels have a great potential for high burn-up and high temperature applications. The general requirements for the cladding material are high resistances to irradiation-induced embrittlement, void swelling, and creep, as well as a good compatibility with the molten sodium. Creep resistance at high temperature is closely related to the thermal stability of the microstructure, and is also affected by the crystallographic texture developed during the thermo-mechanical processes. In general, a fine grain structure impairs the creep resistance, and a strong texture leads to mechanical anisotropy. The present work investigates the effects of oxide particles on the thermal stability of the microstructure and texture of ferritic ODS steel. For this purpose, Fe-15Cr base ferritic steel and its ODS counterpart were produced based on powder metallurgy, and their microstructures were compared.

2. Methods and Results

2.1 Experimental procedures

A 15.5Cr ferritic ODS steel and its yttria-free counterpart were produced based on a powder metallurgy method. Mixtures of elemental powders with nominal compositions given in Table 1 were mechanically alloyed with a Simoloyer CM20 horizontal mill.

Table I: Chemical compositions of experimental ODS steel and its yttria-free counterpart.

	Cr	Mo	Ti	Y_2O_3	Fe
ODS steel	15.5	2	0.1	0.35	Bal.
Yttria-free steel	15.5	2	0.1	-	Bal.

The mixtures of steel balls and elemental powders (steel ball to powder ratio is 15:1 in weight) were milled at 240 rpm for 48 h. The MA powders thus prepared were sealed in a mild steel container and degassed at 400°C for 1h under a vacuum of 10^{-4} torr. The steel containers filled with MA powders were hot-isostatically pressed (HIP) at 1150°C for 3h under 100 MPa and then hot rolled at 1150 °C to a reduction in thickness of 65%. The hot rolled plates were annealed at 1000 °C for 1h and then cold rolled to a reduction in thickness of 30%. The cold rolled plate were annealed at various temperatures between 800°C and 1400°C for 1h.

Microstructures of the annealed sample were analyzed using an electron backscatter diffraction (EBSD) technique. An EBSD analysis was undertaken using a FEI 3D Quanta field-emission-gun scanning electron microscope equipped with a TSL-OIM^{TM} data acquisition system. Samples for the EBSD analysis were mechanically ground and then further polished with an alumina suspension. All the analyses were conducted on the section normal to the transverse direction of the rolled plate. Variation of hardness with annealing temperature was determined using a Vickers micro-hardness measurement.

2.2 Results and Discussion

Variations of hardness in the experimental alloys with annealing temperature are shown in Fig. 1. For hot-rolled ODS steel and its yttria-free counterpart, there is no significant reduction in hardness with increasing annealing temperature up to 1100°C. A further increase of annealing temperature decreased the hardness of ODS steel abruptly, but that of yttria free steel is much less sensitive to annealing temperature. Cold-rolled ODS and yttria-free steels showed similar trends. However, for yttria-free steel, the amounts of decrease in hardness are much larger than those of hot-rolled samples.

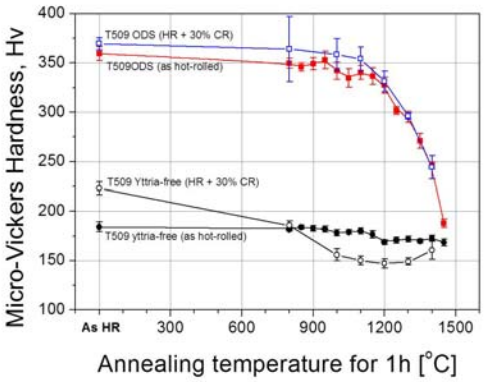

Fig. 1. Variations of Vickers micro-hardness of the hot-rolled and cold-rolled ODS and yttria-free steels with annealing temperature.

Inverse pole figure (IPF) maps for cold-rolled ODS steel and its yttria-free counterpart revealed that both steels have an elongated grain structure (Fig. 2). Coarse elongated grains and small equiaxed grains coexist in the ODS steel, while only elongated grains were observed in the yttria-free steel.

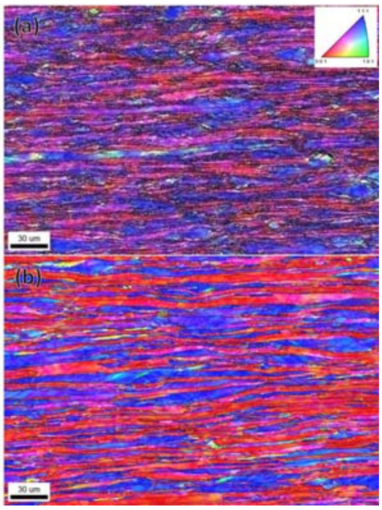

Fig. 2. EBSD ND-IPF maps of 30% cold-rolled (a) ODS steel and (b) yttria-free steel.

When the cold-rolled ODS steel was annealed at 800 °C for 1h, there was no significant change in microstructure. In the yttria-free steel, however, recrystallized grains that grew rapidly were observed (Fig. 3).

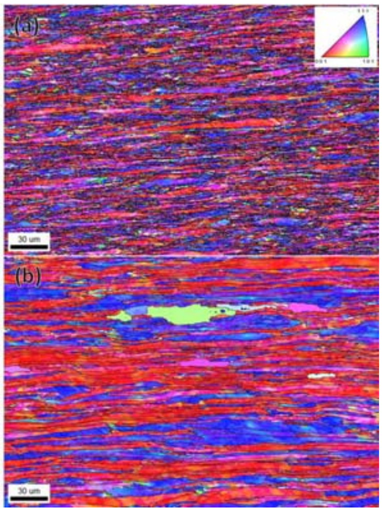

Fig. 3. EBSD ND-IPF maps of 30% cold-rolled and annealed (800°C/1h) (a) ODS steel and (b) yttria-free steel.

Annealing of the ODS steel at 1000°C for 1h had little influence on microstructure (Fig. 4(a)): only change identified by EBSD was very fine grains formed along the grain boundaries. The same heat treatment given to the yttria-free steel, however, resulted in a fully recrystallized microstructure (Fig. 4(b)). The width of recrystallized grains in the yttria-free steel ranges from 10 μm to 50 μm. It is noteworthy that recrystallized grains in yttria-free still have orientations different from those in the cold-rolled sample: differently colored grains in the IPF map suggest a change in orientation.

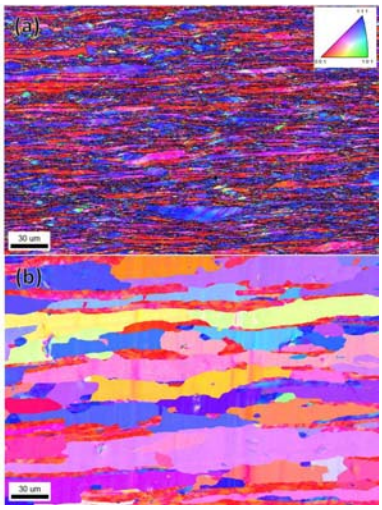

Fig. 4. EBSD ND-IPF maps of 30% cold-rolled and annealed (1000°C/1h) (a) ODS steel and (b) yttria-free steel.

Further increase of annealing temperature to 1300 °C did not change the microstructure of the ODS steel significantly and there was only a slight coarsening of deformed grains. The yttria-free steels annealed at temperatures above 1000 °C showed microstructures similar to that given in Fig. 4(b).

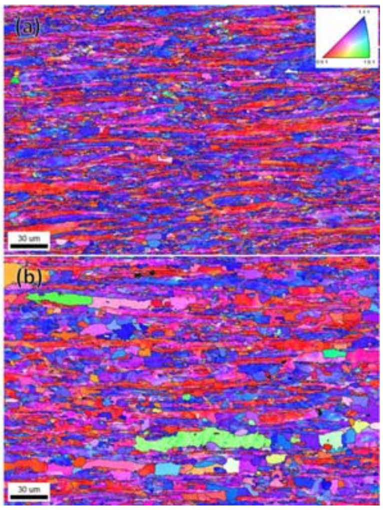

Fig. 5. EBSD ND-IPF maps of 30% cold-rolled and annealed ODS steel: (a) 1400°C/1h and (b) 1400°C/5h.

As the annealing temperature increased to 1400 °C, a remarkable change in microstructure was observed in the ODS steel: recrystallization took place in local regions of microstructure and the size of recrystallized grains ranges from 3 μm to 20 μm (Fig. 5(a)). When annealed at the temperature for 5h, a pronounced growth of recrystallized grains occurred, but there were still elongated grains in deformed state (Fig. 5(b)).

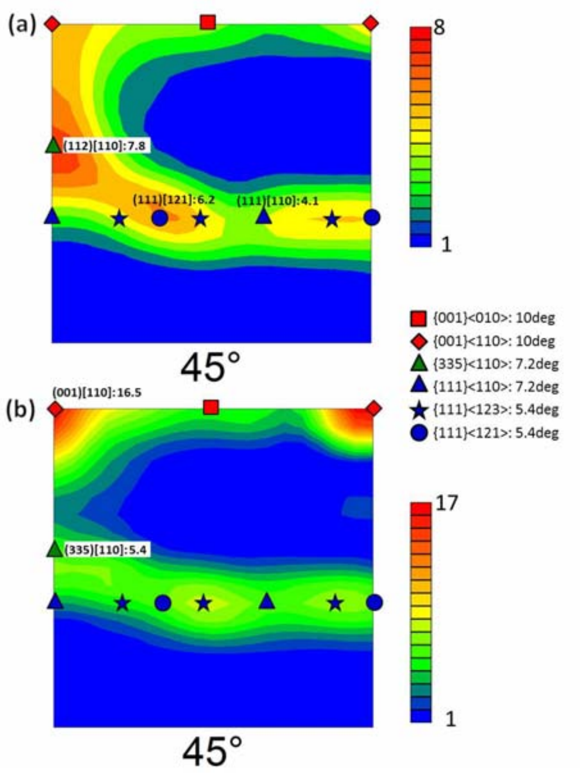

Fig. 6. φ_2=45° sections of ODF for the 30% cold-rolled (a) ODS steels and (b) yttria-free steel. The vertical and horizontal axes of ODF correspond to the Φ and φ_1 angles.

Orientation distribution functions (ODF) determined from EBSD measurement revealed that textures developed in both alloys are different. For the ODS steel, the major texture component is {112}<110> with the maximum ODF intensity of 7.8 and the γ-fiber texture (a series of texture components having {111} planes parallel to the rolling planes) was developed as a minor texture component (Fig. 6(a)). On the other hand, {001}<110> (rotated-cube) component and γ-fiber texture were developed in the yttria-free steel (Fig. 6(b)). The ODF intensity of {001}<110> component in the yttria-free steel is rightly two times higher than that of {112}<110> component in the ODS steel, which suggests that deformation by slip was more difficult for the ODS steel.

As the annealing temperature increases and recrystallization proceeded, there was a significant change of texture in the yttria-free steel: as can be seen from Fig. 4(b), grains with totally different orientations were developed and grew, resulting in a nearly randomized texture. On the other hand, there was no significant change of texture in the ODS steel. The ODS for the ODS steel annealed 1400 °C for 1h is presented in Fig. 7, which shows the major texture component of {112}<110> and γ-fiber texture as minor texture components.

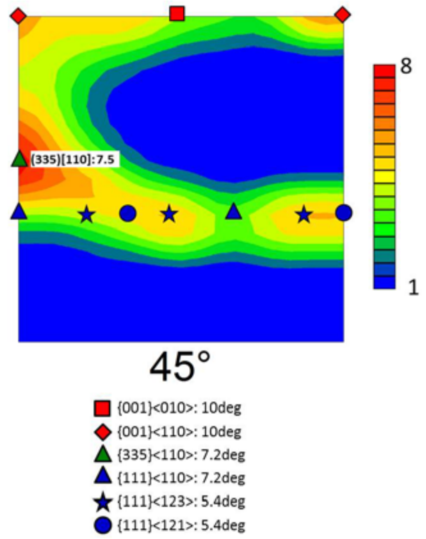

Fig. 7. φ_2=45° section of ODF for the 30% cold-rolled and annealed (1400°C/1h) ODS steel. The vertical and horizontal axes of ODF correspond to the Φ and φ_1 angles.

3. Conclusions

The microstructure and texture of the ODS steel is quite stable at temperatures up to 1300 °C, although significant reduction in hardness occurred at 1200 °C.

REFERENCES

[1] S. Ukai, Oxide Dispersion Strengthened Steels, Comprehensive Nuclear Materials, Vol. 4, p. 241, 2012.