ANNEALING BEHAVIOR OF RAFM ODS-EUROFER STEEL

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Oxide-dispersion-strengthened (ODS) ferritic-martensitic steels are candidates for applications in fusion power plants where microstructural long-term stability at temperatures of ~650°C to 700°C are required. The microstructural stability of 80% cold-rolled reduced-activation ferritic-martensitic 9% Cr ODS-Eurofer steel was investigated within a wide range of temperatures (300°C to 1350°C). Fine oxide dispersion is very effective to prevent recrystallization in the ferritic phase field. The low recrystallized volume fraction (<0.1) found in samples annealed at 800°C is associated with the nuclei found at prior grain boundaries and around coarse $M_{23}C_6$ particles. The combination of retarding effects such as Zener drag and concurrent recovery decrease the local stored energy and impede further growth of the recrystallization nuclei. Above 900°C, martensitic transformation takes place with consequent coarsening. Significant changes in crystallographic texture are also reported.

KEYWORDS: *ODS-Eurofer steel, recovery, recrystallization*

Note: Some figures in this paper are in color only in the electronic version.

I. INTRODUCTION

Oxide-dispersion-strengthened (ODS) ferritic-martensitic steels are potential candidates for several applications including the manufacture of structural parts for nuclear fusion technology. Because of their moderate creep properties, non-ODS reduced-activation ferritic-martensitic (RAFM) CrWVTa steels are currently limited to an upper operating temperature of ~550°C for blanket and divertor applications.[1,2] A replacement of conventionally produced RAFM steels by suitable ODS alloys would allow an increase in the operating temperature to 650°C or more.[3,4]

The addition of nanoscaled ODS particles to these RAFM steels is intended to increase high-temperature creep strength.[1,2] The combination of plastic deformation during the manufacture of these parts and subsequent long-term exposure at high temperatures may cause static recrystallization. In this scenario, the evaluation of the microstructural stability of ODS-Eurofer steel at conditions as close as possible to the expected ones during service is necessary.

Several microstructural changes take place during annealing of deformed metals including recovery of structural defects (excess vacancies and dislocations), recrystallization, grain growth, particle coarsening (Ostwald ripening), and other phase transformations such as solid-state precipitation. As a result, these microstructural changes lead to important changes in properties, in particular, the softening of the material. Besides their effect on the mechanical properties, recrystallization and grain growth, for instance, may also cause important changes in crystallographic texture. The microstructural changes can be followed by many characterization techniques including classical metallography, hardness testing, magnetic measurements, and electron backscatter diffraction (EBSD).

Earlier investigations on the microstructural stability of ODS-Eurofer steel under nonirradiation conditions have been reported in the literature.[5–7] In the present work, we

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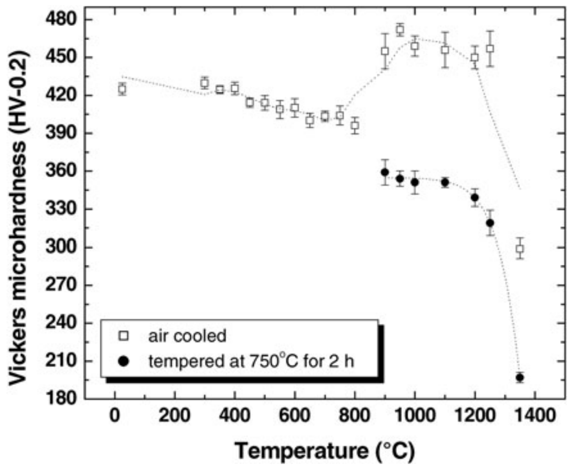

Fig. 1. Softening behavior of 80% cold-rolled RAFM ODS-Eurofer steel. Solid symbols refer to samples tempered at 750°C for 2 h (adapted from Ref. 7).

summarize these findings and present new results concerning the microstructural and textural evolution in 9% Cr RAFM ODS-Eurofer steel annealed within a wide range of temperatures (300°C to 1350°C). The microstructure of ODS-Eurofer steel in several metallurgical conditions was characterized by EBSD and Vickers hardness testing. The crystallographic texture was evaluated using X-ray diffraction in a texture goniometer. The results are useful to understanding the microstructural changes associated with the exposure of this steel to high temperatures.

II. EXPERIMENTAL

The RAFM 9% Cr ODS-Eurofer steel was processed by Plansee AG (Austria) using a suitable powder metallurgy route. The hot-isostatic pressed slabs were hot cross

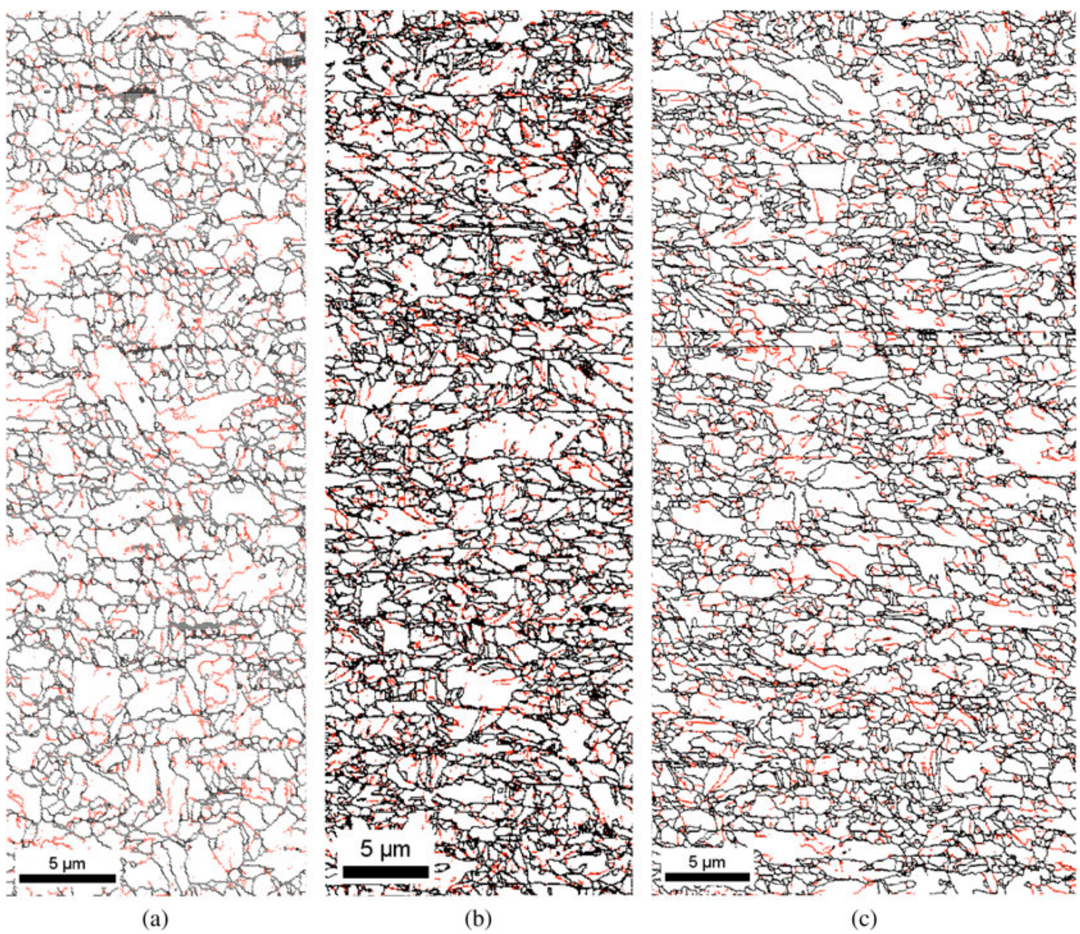

Fig. 2. EBSD scans of ODS-Eurofer steel in several metallurgical conditions: (a) as received, (b) 80% cold rolled and annealed at 1100°C for 1 h, and (c) same as (b), but followed by tempering at 750°C for 2 h. Black lines indicate high-angle boundaries (above 15 deg misorientation). Red lines (color online) indicate low-angle boundaries (2 to 15 deg misorientation). The rolling direction is parallel to the scale bar.

rolled in the austenitic field and then air cooled. This steel undergoes a martensitic transformation even under air cooling.[4] The hot-cross-rolled sheets were tempered at 750°C for 2 h for the present study (as-received condition). The nominal composition of this steel was 9Cr-1W-0.08Ta-0.2V-0.07C-0.4Mn-0.3Y_2O_3 (in weight percent). For further details, see Ref. 2. Cold rolling to 80% thickness reduction was carried out in multiple passes. Isothermal annealing in vacuum was performed from 300°C to 1350°C for 1 h followed by air cooling. Specimens annealed in the austenitic field (900°C to 1350°C) were tempered at 750°C for 2 h in vacuum in sealed glass. Textures were determined in five different conditions using an X-ray multipurpose diffractometer with a Cu-K α radiation source. The results were analyzed using the popLA software. The microstructure of the material was evaluated with a JEOL-6500F field emission gun scanning electron microscope operating at 15 kV. EBSD scans of the as-received and annealed samples were carried out in longitudinal sections.

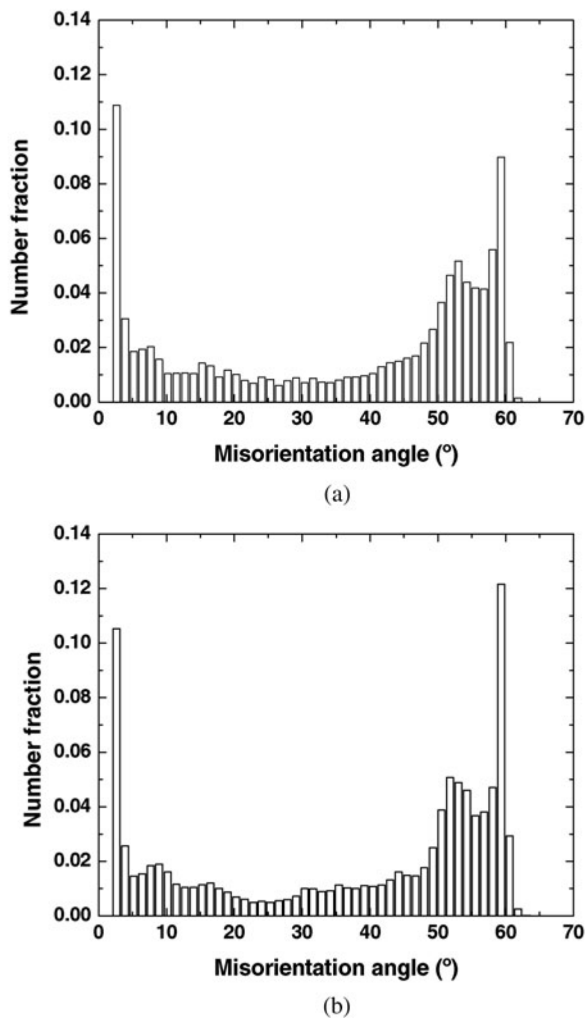

Fig. 3. Misorientation angle distribution of ODS-Eurofer steel: (a) as-received condition and (b) 80% cold rolled followed by annealing at 1100°C for 1 h (as-quenched state).

III. RESULTS AND DISCUSSION

The ODS-Eurofer steel shows an increase in hardness from 367 ± 7 in the as-received material to 425 ± 5 after 80% thickness reduction. The significant increase in the Vickers microhardness can be attributed to the presence of nanosized Y_2O_3 particles. These particles act as pinning obstacles to moving dislocations during plastic deformation increasing strain hardening.[6] The mean size of Y_2O_3 particles was reported as 12 nm (Ref. 5). Recovery and recrystallization are both driven by the energy stored in the dislocation structure found in deformed metals. The fine dispersion of oxide particles also affects the softening behavior of this steel in a significant manner as shown in Fig. 1.

Earlier studies on the softening behavior of 80% cold-rolled ODS-Eurofer steel during annealing in ferritic and austenitic phase fields have already been reported in Refs. 6 and 7. In the ferritic phase field, below A_{c1} (A_{c1} = 875°C for a heating rate of 30°C/s), static recovery was the predominant softening mechanism.[6] Recrystallization was only partial. The volume fraction of recrystallized grains after isothermal annealing at 800°C for 1 h was <0.1. The kinetics of static recrystallization in particle-containing materials depends on the balance between driving pressures (provided by stored dislocations) and retarding pressures (Zener drag) in the deformed microstructure. The fine dispersion of oxide particles is responsible for preventing recrystallization under these conditions. The low recrystallized volume is associated

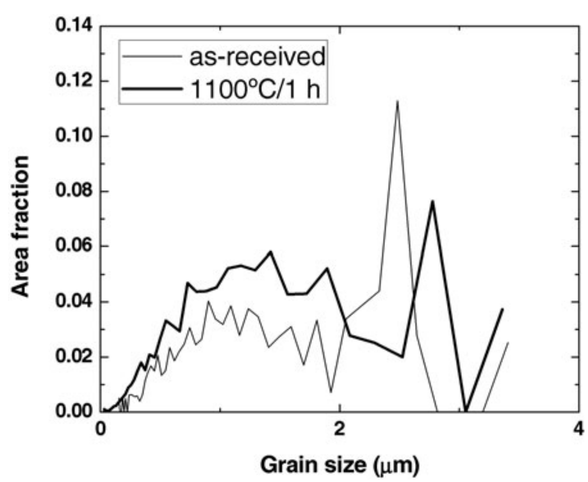

Fig. 4. Grain size distribution in ODS-Eurofer steel in the as-received condition and after 80% cold rolling followed by annealing at 1100°C for 1 h (as-quenched state).

with the nuclei found at prior grain boundaries where large curvatures are present and around coarse $M_{23}C_6$ particles due to particle-stimulated nucleation.[8] When annealing is performed in the austenitic phase field (above $\approx 900°C$), results show an increase in Vickers microhardness due to the martensitic transformation after cooling.[7] The martensitic transformation is accompanied by significant hardening due to the increase in dislocation density and the increase in the number of interfaces. Further softening is observed after tempering at 750°C for 2 h when martensite transforms into a mixture of ferrite and carbides. The pronounced softening observed for the sample annealed at 1350°C for 1 h compared to those annealed in the range 900°C to 1250°C for either tempered or untempered conditions can be explained by the occurrence of intensive austenite grain growth.

The EBSD technique was used to image the microstructure at the grain and subgrain scales. Maps with same size in area were performed in ODS-Eurofer steel with distinct metallurgical conditions. Results are shown in Fig. 2. Figure 2a shows the results of mesotexture determined in the longitudinal section in the as-received condition. The black lines shown in Fig. 2 correspond to high-angle boundaries (misorientation >15 deg), while red lines (color online) correspond to low-angle boundaries (2- to 15-deg misorientation range). Grains with irregular morphology can be noticed. Figure 2b shows the mesotexture of the sample cold rolled to 80% reduction followed by annealing at 1100°C for 1 h. The grain morphology is very similar to that shown in Fig. 2a. This structure consists of martensitic laths and blocks and is commonly observed in water-quenched low-carbon steels.[9,10] The mesotexture of the sample annealed at 1100°C and subsequently tempered at 750°C for 2 h is shown in Fig. 2c. The misorientation angle distribution for samples in both as-received condition and annealed-at-1100°C condition is shown in Figs. 3a and 3b, respectively. The amount of high-angle grain boundaries with misorientation angles between 40 and 60 deg is predominant in the as-received condition (57%). Low-angle grain boundaries, with misorientation angles between 2 and 15 deg, are also present in a lower fraction (27%). A quite similar result is observed in Fig. 3b. The major differences between these two structures show up when grain size distribution is evaluated (Fig. 4). The grain structure found in the sample annealed at 1100°C for 1 h is slightly coarser than the one found in the as-received condition, and their average grain sizes are, respectively, 1.4 and 1.0 μm.

Important changes in the texture of ODS-Eurofer steel are observed for different annealing conditions (Fig. 5). Sections of the orientation distribution function for $\varphi_2 = 0$ deg and $\varphi_2 = 45$ deg (Bunge notation) are shown for purposes of comparison. These sections contain the most important texture components in ferritic steels. The texture found in the as-received steel is rather weak as depicted in Fig. 5a. The main texture

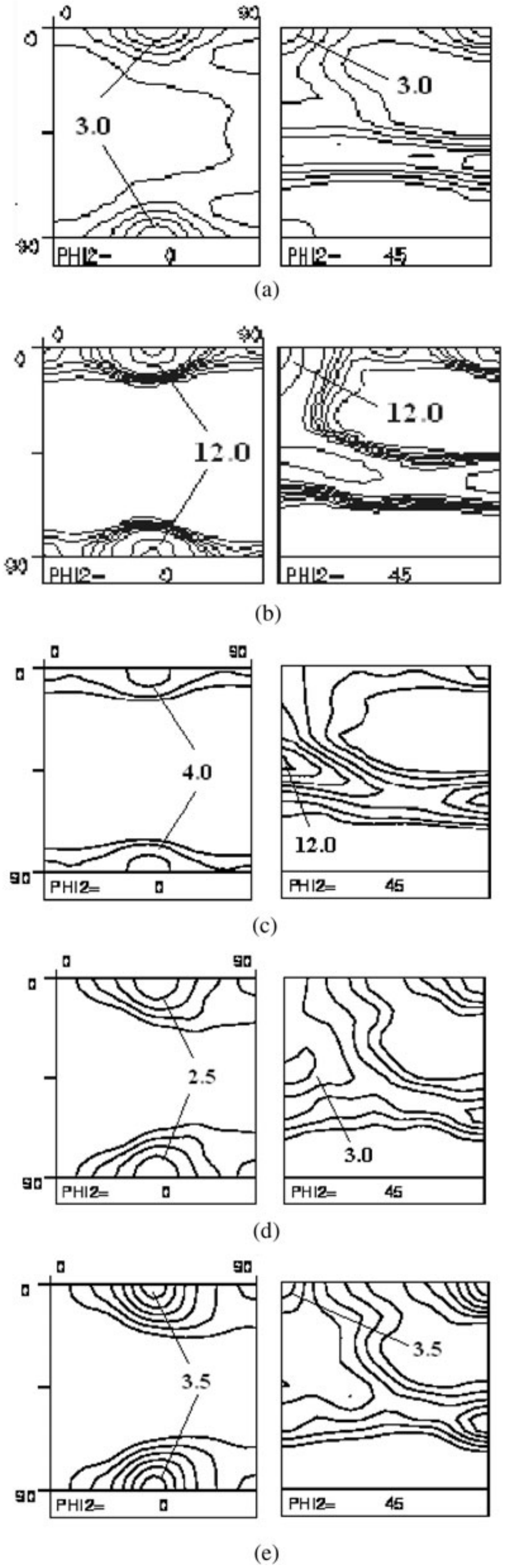

Fig. 5. Texture results of ODS-Eurofer steel in the following conditions: (a) as received, (b) 80% cold rolled, (c) 80% cold rolled and annealed at 800°C for 1 h, (d) 80% cold rolled and annealed at 1100°C for 1 h, and (e) 80% cold rolled and annealed at 1350°C for 1 h.

components belong to α- and γ-fiber components. The cold rolling textures of ferritic steels can be described by a set of texture fibers such as the α-fiber collecting grains with a $\langle 110 \rangle$ direction parallel to the rolling direction and the γ-fiber summarizing crystals with a $\{111\}$ plane parallel to the sheet surface.[11,12] The (100)[110] (rotated-cube) texture component is also present in the as-received condition. Texture is strengthened after 80% cold rolling as shown in Fig. 5b. The main texture components are the same as observed in the as-received condition but with higher intensities. Annealing at 800°C for 1 h promotes the weakening of the rotated-cube component and strengthens the γ-fiber component with a maximum at the $\{111\}\langle 110 \rangle$ component (Fig. 5c). For samples annealed in the austenitic phase field (Figs. 5d and 5e), texture is weakened considerably. This effect can be attributed to the martensitic transformation.

IV. SUMMARY AND CONCLUSIONS

The microstructure and texture evolution of 80% cold-rolled ODS-Eurofer steel during isothermal annealing from 300°C up to 1350°C under nonirradiation conditions was investigated. The small amount of softening experienced by this steel at temperatures below 800°C (ferritic phase field) is predominantly caused by recovery rather than recrystallization. Annealing above 900°C promotes pronounced hardening upon cooling due to the martensitic transformation. Tempering at 750°C for 2 h promotes a pronounced softening in the as-quenched samples. Important changes in texture were observed regarding both intensity and spread around ideal components. The main texture components for all investigated metallurgical conditions belong to α- and γ-fibers as well as the rotated-cube (100)[110] component.

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