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Effect of heat treatment on microstructure and hardness of Eurofer 97, Eurofer ODS and T92 steels

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ABSTRACT

Eurofer ODS steel is a potential candidate for fusion reactor application due to its excellent swelling resistance, low thermal expansion coefficient and high temperature properties. One of the main issues is that high fluence neutron irradiation induces a significant increase of ductile-to-brittle transition temperature (DBTT) at temperatures below 400 °C which restricts its application. The aim of this study is to explore the methods to lower the initial DBTT of Eurofer ODS steel by heat treatment optimization. Two heats of Eurofer ODS steels with different C contents are heat-treated at different normalizing temperatures, cooling rates and tempering conditions, and are compared with Eurofer 97 and T92 steels heat-treated with similar conditions. The microstructure is characterized by optical microscopy, FEG-TEM and OIM-EBSD techniques. The effect of normalization, cooling rate and temper on grain size, precipitation, grain boundary misorientation and hardness are investigated. The influences of these properties on DBTT are discussed.

1. Introduction

Reduced activation ferritic/martensitic (RAFM) steels with 9 wt% Cr are leading candidates for structural components, e.g. first wall/blanket, in fusion reactors due to their excellent thermal properties and swelling resistance [1,2]. Eurofer 97 and F82H steels are European and Japanese examples, respectively. Eurofer 97 steels exhibit a lower ductile-to-brittle transition temperature (DBTT) than F82H [3]. Oxide dispersion strengthened (ODS) Eurofer 97 steel is manufactured by mechanical alloying and hot isostatic pressing by mixing Eurofer 97 powders and nano-size yttria particles (Y_2O_3). This has improved mechanical properties at high temperatures due to the thermal stability of the oxide dispersion even after irradiation. But the impact properties remain low and the DBTT is high compared to Eurofer 97 steels [4,5].

In this study, we focus on the effects of heat treatments, with varying normalizing temperature, temper temperature and cooling rate, on the microstructure and mechanical properties, and interpret these findings in terms of DBTT.

2. Experimental

Eurofer 97 steel, two Eurofer ODS variants (HXX and FZK, the latter having a very low carbon concentration) and T92 (or NF616, F82H being its low activation version) are studied. Their chemical compositions are listed in Table 1.

To investigate the effect of normalizing temperature on grain size, hardness and grain boundary misorientation, Eurofer ODS steels were subjected to solution treatment at 980, 1040, 1100, 1150, 1300 and 1350 °C, followed by cooling and tempering at 700 °C; T92 and Eurofer 97 steels were subjected to solution treatment at 980, 1040, 1100 and 1150 °C, followed by water quenching and air cooling, respectively, and tempering at 700 °C.

Different cooling rates, water quench (WQ), air cool (AC) and furnace cool (FC) are applied to Eurofer ODS (FZK) steels to observe the effect on grain boundary misorientation.

Eurofer ODS (HXX and FZK) and T92 were subjected to solution treatment at 980 °C, followed by water quenching, and tempering at 550, 650, 750 and 850 °C.

The microstructure and hardness were checked by optical microscopy (Reichert MEF-3), LEO 1530VP FEG SEM, TexSEM Laboratories (TSL) EBSD system, Tecnai F20 Field Emission Gun Scanning Transmission Electron Microscopy (FEG STEM) equipped with EDS, and Vickers hardness testing.

3. Results and discussion

The dependence of prior austenite grain size on normalizing temperature is shown in Fig. 1. For T92 and Eurofer 97 steels, the grain size increases with the increase of solution temperature. The grain coarsening rate of T92 is faster than that of Eurofer 97. This may be because there is higher Ta concentration in Eurofer

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Table 1
Nominal chemical composition of Eurofer 97, ODS and T92, wt%.

	C	Cr	W	Mn	V	Ta	Si	Nb	Ni	Mo	
97	0.12	8.93	1.07	0.49	0.28	0.15					
ODX–HXX	0.11	9.0	1.5	0.40	0.20	0.12					$0.3Y_2O_3$
ODX–FZK	N/A*	9.0	1.5	0.40	0.20	0.12					$0.3Y_2O_3$
T92	0.12	8.86	1.69	0.43	0.20		0.32	0.057	0.25	0.41	

Fe is the balance. *C content in ODX–FZK is very low compared to ODS–HXX.

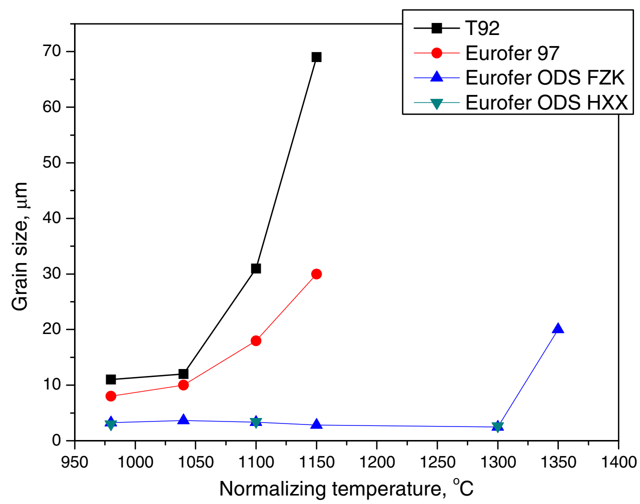

Fig. 1. Prior grain size vs. normalizing temperature.

97 than Nb in T92 (both Ta and Nb play a role of grain refinement). In Eurofer ODS steels (HXX and FZK), the grain size remains almost unchanged up to 1300 °C; After that, grain size increases with the increase of normalizing temperature. This indicates that the pinning of grain boundaries by yttria (Y_2O_3) particles is effective below 1300 °C. It should be pointed out that the grain sizes of T92 and Eurofer 97 steels are evaluated by optical microscopy; and the grain sizes of Eurofer ODS steels are measured by EBSD technique because their grains are too small to be distinguished in an optical microscope. It is known that the grain size obtained from EBSD is more precise than that from optical microscopy [6].

Fig. 2 shows the relationships between hardness and normalizing temperature. For T92 and Eurofer 97 steels, their hardness increases with the increase of normalizing temperature. From Fig. 1, we know that the grain size of T92 and Eurofer 97 increase with the increase of normalizing temperature. According to the Hall–Petch relationship, the hardness should decrease with the increase of grain size. Therefore, this indicates that there is another contribution to the hardness in T92 and Eurofer 97. The hardness of Eurofer ODS steels decreases with increased normalizing temperature. ODS FZK steel shows lower hardness than ODS HXX, possibly due to its very low carbon concentration.

TEM investigation shows that the microstructure of Eurofer 97 steel subjected to normalization, air cooling and tempering treatment is martensitic with $M_{23}C_6$, some Ta-rich carbides and few V-rich carbides. In Eurofer ODS steels subjected to normalization, water quench and tempering treatment, the martensitic structure is decorated with $M_{23}C_6$ and yttria (Y_2O_3) (see Fig. 3), but Ta-rich carbides are not formed. EDS analysis shows that the composition of the matrix and $M_{23}C_6$ particles are similar in Eurofer 97 and Eurofer ODS steels. However, in the ODS steel, Ta appears to enter the Y_2O_3, see Fig. 4. The microstructure of T92 is martensitic with $M_{23}C_6$ and Nb-rich carbides. The solubility of Ta and Nb in ferritic steels increases with increase of normalizing temperature from 980 to 1150 °C [7]. Thus more Ta/Nb-rich carbides can be formed in the ensuing temper treatment, and this contributes to the increase of hardness with temperature in the Eurofer 97 and T92 steels. In Eurofer ODS steels, Ta segregates to Y_2O_3 particles, resulting in the lack of Ta-rich carbides compared to Eurofer 97. Therefore there is no increase of hardness with increasing normalizing temperature in Eurofer ODS steels.

The influences of normalizing temperature and cooling rate on grain misorientation in Eurofer ODS steel (FZK) are shown in Figs. 5 and 6, respectively. With the increase of normalizing temperature from 980 to 1150 °C, the number of fraction of low angle

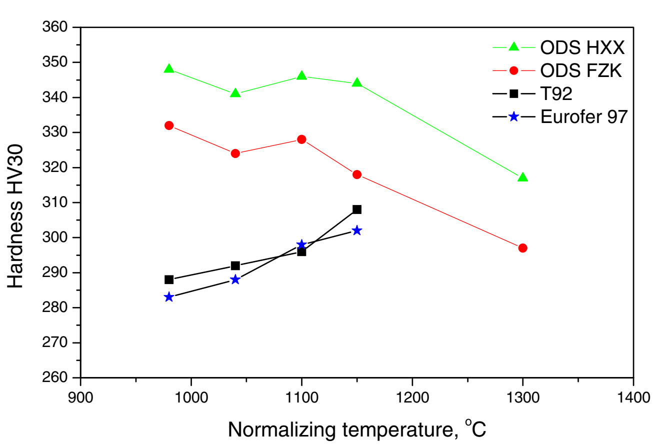

Fig. 2. Hardness vs. normalizing temperature.

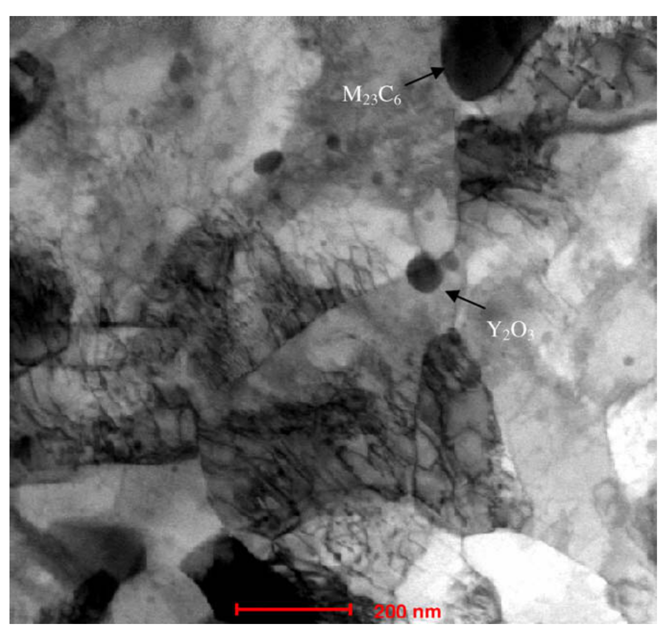

Fig. 3. The microstructure of Eurofer ODS steels.

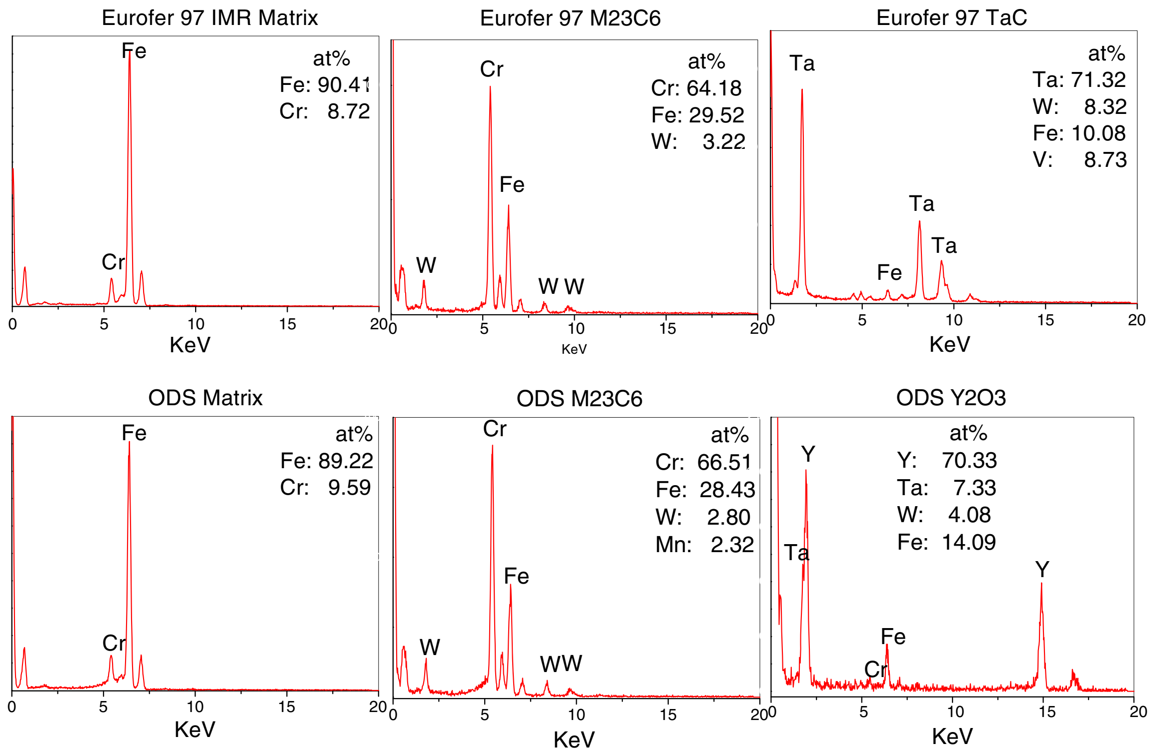

Fig. 4. The micro-chemical composition of matrix, $M_{23}C_6$ and Ta-rich carbides in Eurofer 97 steels and of matrix, $M_{23}C_6$ and Y_2O_3 in Eurofer ODS steels.

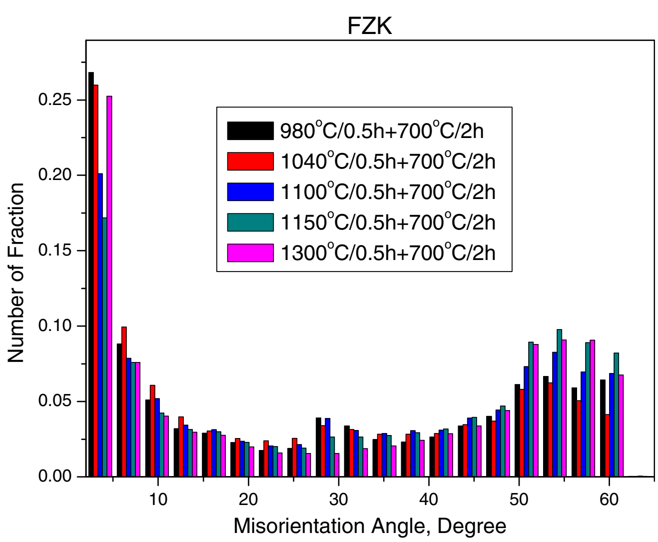

Fig. 5. The dependence of grain boundary misorientation angle on normalizing temperature.

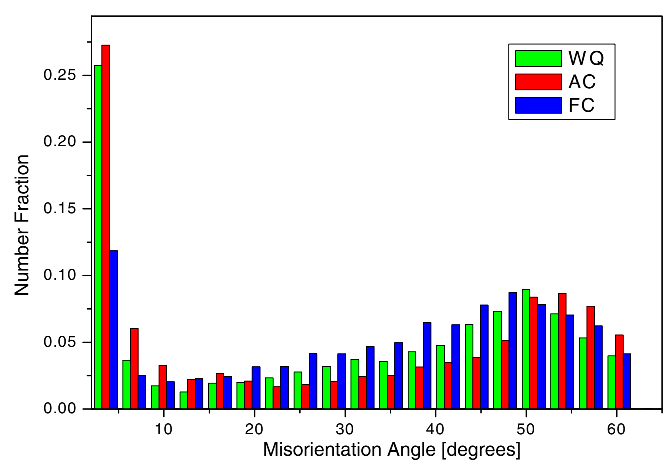

Fig. 6. The dependence of grain boundary misorientation angle on cooling rate.

boundaries decreases and the number of high angle boundaries increases. This trend is terminated at 1300 °C, as seen in Fig. 5. It is seen that fast cooling rates (water quench and air cooling) clearly increase the fraction of low angle boundaries compared to the slow cooling rate, i.e. furnace cooling, as shown in Fig. 6.

The relationships of hardness (HV30) to tempering temperatures in Eurofer ODS and T92 are seen in Fig. 7. For T92, hardness decreases with increasing tempering temperature. For Eurofer ODS steels, the hardness decreases with increase tempering temperature from 550 to 750 °C, then increases at 850 °C. This suggests that some reversion to martensite takes place above 750 °C in Eurofer ODS. The HXX steel shows higher hardness than FZK.

In general, fine grain size and high fraction of low angle grain boundary are beneficial to DBTT. Fine grain sizes provide greater barriers to cleavage cracks because of the large number of crack arrests that are made. Low angle boundaries have better atomic fitting on the GB planes and this results in a greater resistance to inter-granular crack propagation. Eurofer 97 would benefit on these grounds from a 980 °C solution treatment, followed by air cool or water quenching. The hardness of these grains is reflected in the hardness measurements. Increases in hardness promote a upward trend in DBTT shift. DBTT benefits for Eurofer 97 and T92 would be expected from the hardness viewpoint by solution treatment at a low temperature (980 °C) and tempering at temperatures around 750 °C. Eurofer ODS should have a lower DBTT with solution treatment at 1150 °C and above, followed by water quench and tempering at 750 °C. Charpy testing to confirm the effect of normalizing temperature on DBTT are currently in progress.

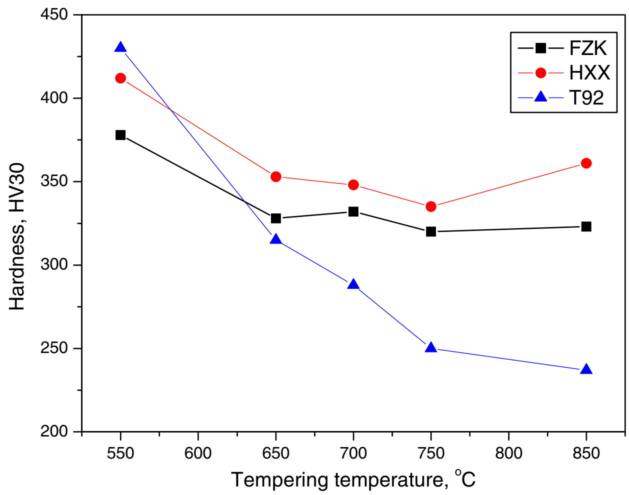

Fig. 7. Hardness vs. tempering temperature.

4. Summary

Different heat treatment conditions are explored to investigate methods to control grain size in Eurofer 97, Eurofer ODS and T92 steels. With the increase of normalizing temperature, the grain size increases in T92 and Eurofer 97 steels and remains almost unchanged in Eurofer ODS steels, the hardness increases in T92 and Eurofer 97 and decreases in Eurofer ODS steels. The formation of Nb-rich and Ta-rich carbides is related to the increase of hardness. Ta enrichment in yittra particles is observed, which may be the reason for the lack of Ta-rich carbides and resulting reduction in hardness with normalizing temperature in Eurofer ODS steels. With the increase of tempering temperature, the hardness decreases in T92 while in Eurofer ODS steels, the hardness decreases until tempering temperature up to 750 °C, then increases slightly.

These observations are determined in relating to DBTT trends. It is suggested that the ideal heat treatments for Eurofer ODS would be: heat treatment at 1150 °C followed by fast cooling and tempering at 750 °C.

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