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Investigation of heat treatment conditions on the structure of 12% chromium reduced activation steels

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Abstract

The properties of 12% chromium reduced activation steels of the martensitic and ferritic–martensitic classes are governed by alloying, microalloying and heat treatment. In this study, the influence of processing and heat treatment on the structure and mechanical properties of Fe–0.16C–12Cr–2W–V–Ta–B steels was analysed. The main metallurgical factors affecting the properties and irradiation resistance were found to be the ratio between martensite and δ-ferrite phases; a high homogeneity of the initial solid solution; an adequate level of martensitic block fragmentation and carbide phase dispersion. All of these are highly sensitive to the conditions of heat treatment and thermomechanical processing.

1. Introduction

High chromium martensitic and ferritic steels containing alloying elements that minimize radioactivation are being considered as candidate materials for fusion reactor structural applications. The properties of these steels are sensitive to the microstructure, which in turn is a function of composition, processing and heat treatment. In this study, we investigate the effects of processing and heat treatment on the microstructure and mechanical properties of a 12% chromium steel.

2. Experiment

The data below are results of an investigation of an industrial heats of a 12% chromium reduced activated steel Fe–0.16C–12Cr–2W–V–Ta–B. The chemical composition of the steel (in weight per cent) is given in Table 1.

The results of thermal analysis and austenite phase transformation under continuous cooling conditions show that the basic transformation in the steel is a martensitic one. As in other 12% Cr steels it proceeds upon cooling in air [1]. This steel was subject to microstructural analysis and mechanical property testing following processing and heat treatment.

3. Results and discussion

3.1. Matrix structure and precipitates

The steel structure after heat treatment in the temperature range of 1000–1100 °C was martensitic. The amount of δ-ferrite did not exceed 20%. With an increase in the normalization temperature, growth of austenite grains and an increase in martensite lath and lath packet size became noticeable. Microhardness was also found to rise, and the scatter was reduced. This suggests an increase in the degree of the solid solution and its homogeneity as the normalization temperature approached 1100 °C.

Optical metallographic and electron microscopy (TEM) studies were carried out on samples (Figs. 1–3).

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Table 1
Steel composition

C	Si	Mn	W	Cr	V	Ta	B	N	Ti	Zr	S	P	Cu	Co
0.14	0.37	0.94	1.1	11.2	0.29	0.17	0.004	0.044	0.02	0.06	0.007	0.007	<0.01	<0.01

The microstructure presented in Fig. 1(a) is for tempered martensite, which retains the orientation of the newly transformed martensite and carbide phase.

The temperature and duration of tempering (720 °C, 3 h) was chosen on the basis of joint consideration of the kinetics of phase change and the level of strength and ductility for short and long term mechanical tests. The structure after normalization from 1070 °C followed by tempering (720 °C, 3 h, Fig. 2(a)) is the optimal structure promoting a high level of thermomechanical performance with sufficient ductility. This structure features a dislocation substructure, which is partially fragmented by polygonization. Boundaries of martensite laths and lath packets are sites for precipitate formation. The precipitates are identified as carbides of the $M_{23}C_6$ type (fcc, lattice parameter $a = 0.106$ nm); VC carbides (fcc, $a = 0.422$ nm); and VN nitrides (fcc, $a = 0.406$ nm). The V(C,N) precipitates are shown in Fig. 3. The latter were more often observed within the matrix. The size of the $M_{23}C_6$ precipitates ranged from 100 to 500 nm. The sizes of the V(C,N) particles varied from 15 to 30 nm.

With an increase in normalization temperature, the quantity of dispersed particles increased and the range of precipitation was extended. The elemental analysis of carbide and carbonitride phases shows that they were complex compounds. The composition includes many alloying elements:

$$M_{23}C_6 - (\mathrm{Cr, W, V, Ta})_{23}C_6,$$
$$\mathrm{V(C, N)} - (\mathrm{V, Ti, Zr, Ta})(\mathrm{C, N}).$$

Previous assessment of the influence of metallurgical factors on radiation damage resistance has identified some of the common features that are needed [1–3]:

- an optimal ratio between martensite and δ-ferrite (not more than 20% of δ-ferrite);
- a high degree of solid solution homogeneity;

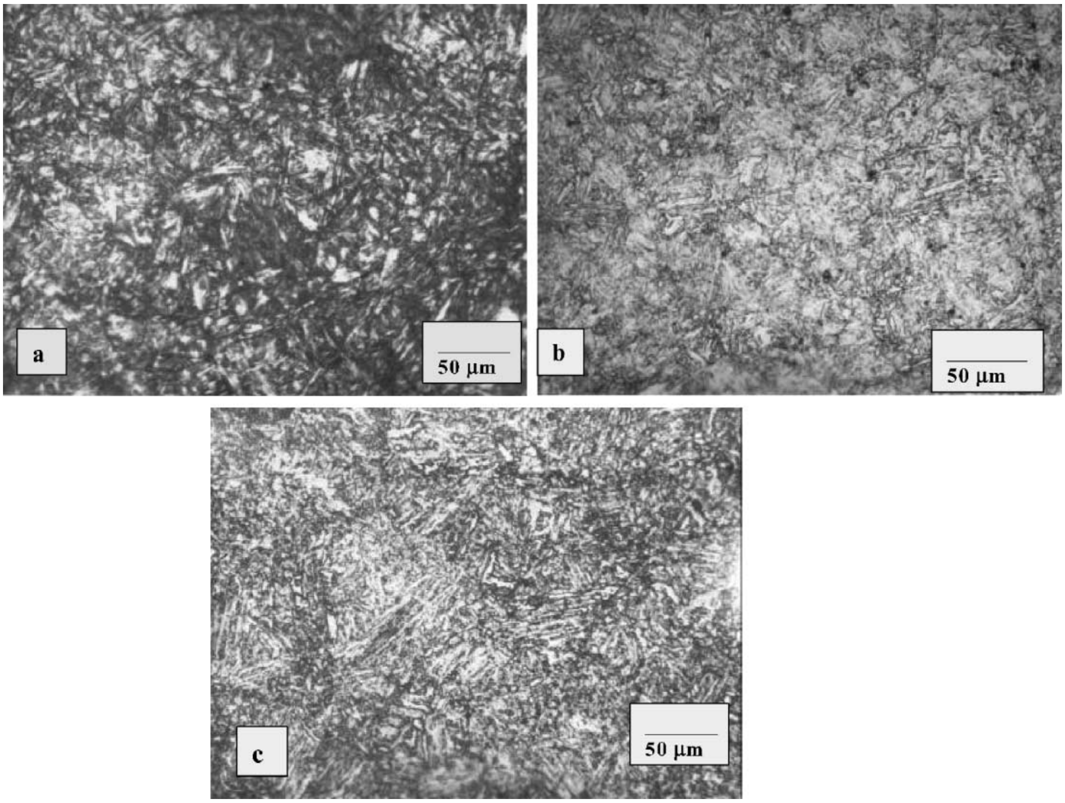

Fig. 1. Optical micrographs of the microstructure of 12Cr–2WVTaB steel after various modes of thermal processing: (a) normalization at 1070 + 720 °C, 3 h, $H\mu_{50}$ 263–270; (b) single cyclic processing, $H\mu_{50}$ 222–229; (c) multiple cyclic processing, $H\mu_{50}$ 197–203.

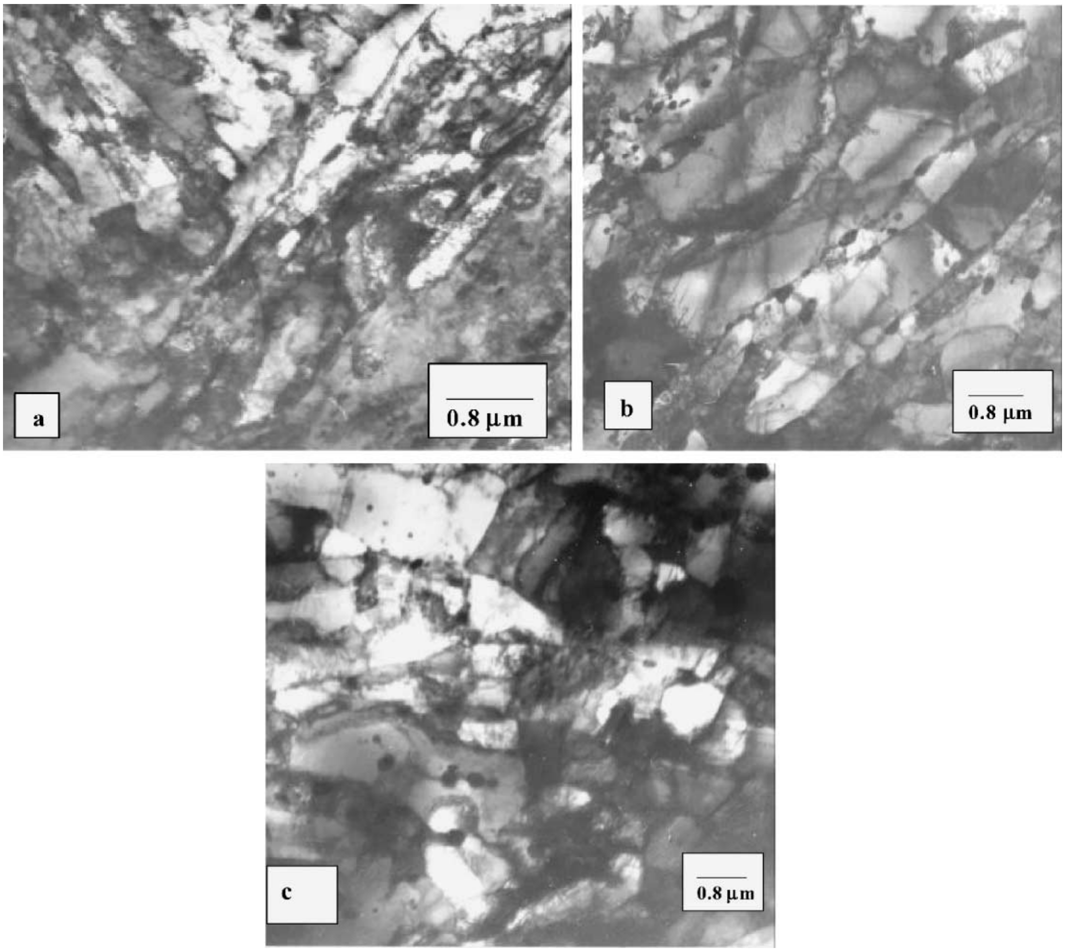

Fig. 2. TEM-micrograph of the substructure of the matrix phase and precipitates in 12Cr–2WVTaB steel after modes of thermal processing: (a) normalization at 1070 + 720 °C, 3 h; (b) single cyclic processing; (c) multiple cyclic processing.

- good dispersion of the dislocation substructure and carbide phases.

The above structural elements are sensitive to heat treatment conditions. It has been found that:

1. Twofold normalization after heating to different temperatures in the austenite range refines the martensite substructure, and increases the homogeneity of the matrix.
2. Normalization followed by single heating to a temperature close to the lower critical (A_{c1}) temperature and specified cooling (conventionally called single cyclic processing) forms a matrix substructure that increases the ductility of the steel.
3. Conditions equivalent to mode 2, but with multiple heating to temperatures close to A_{c1} are called multiple cyclic processing.

The final treatment for all conditions was tempering at 720 °C, 3 h. Examinations of the structural condition of the twofold normalized steel (mode 1) show that multiple heating to the austenite area followed by specified cooling result in refinement of the matrix substructure. The temperature range of heating provides the direct and reverse $\gamma \Longleftrightarrow \alpha$ transformation. The structure formed after single cyclic processing (mode 2) is distinguished by a larger martensite lath size and partial formation of a polygonal substructure in the matrix. This is shown in Figs. 1(b) and 2(b). The α-ferrite matrix phase, after multiple cyclic processing (mode 3), is distinguished by a block (polygonal) substructure, as shown in Figs. 1(c) and 2(c).

After all of these heat treatments, carbide and carbonitride phases $M_{23}C_6$ and V(C,N) were observed to form. The coarsest precipitates $M_{23}C_6$ ($\simeq$500 nm) were found after multiple cyclic processing. The V(C,N)

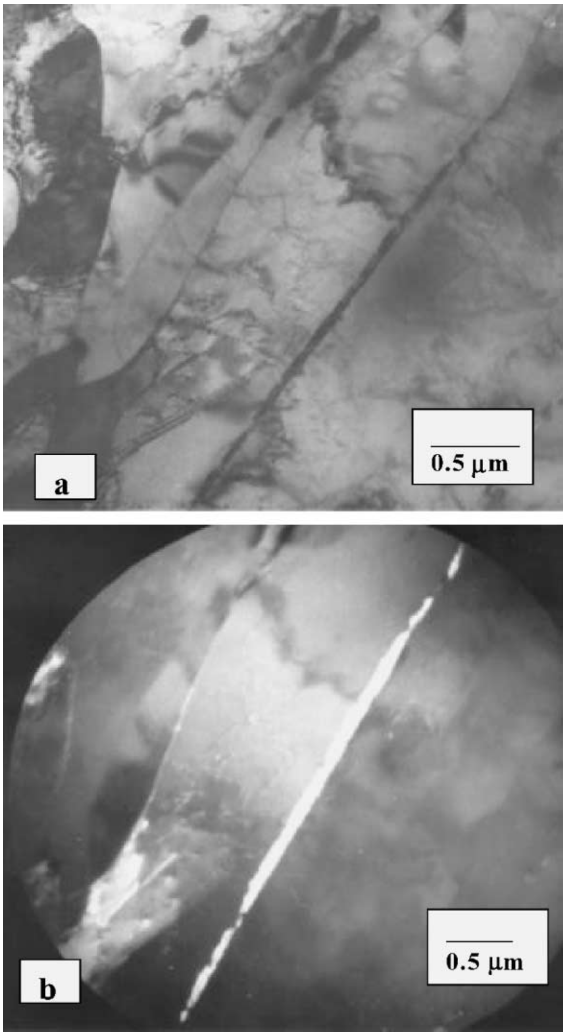

Fig. 3. TEM-micrograph of V(C,N) precipitates in 12Cr–2WVTaB steel: (a) bright field; (b) dark field.

phase dispersion, number density, and location were sensitive to heat treatment. Single cyclic processing results in precipitation of fine V(C,N) precipitates no more than 15 nm in size, basically along grain boundaries. The large particles ≃70 nm in size (Fig. 2(b)) are intergranular. As a result of multiple cyclic processing, precipitation of the V(C,N) phase was only observed within the matrix. The sizes of the precipitates were from 15 up to 80 nm. After the mode 3 heat treatment, the grain boundaries were basically free from precipitates of all types.

3.2. Mechanical properties, Young's modulus

The microhardness $H\mu_{50}$ was measured for each of the above structures, and values are given in the caption of Fig. 1. After multiple cyclic processing, the steel had the lowest microhardness. This is attributed to the formation of a polygonal structure, which provides high ductility. Multicyclic processing increases the impact strength of the steel in the unirradiated condition, an improvement which may be retained after irradiation.

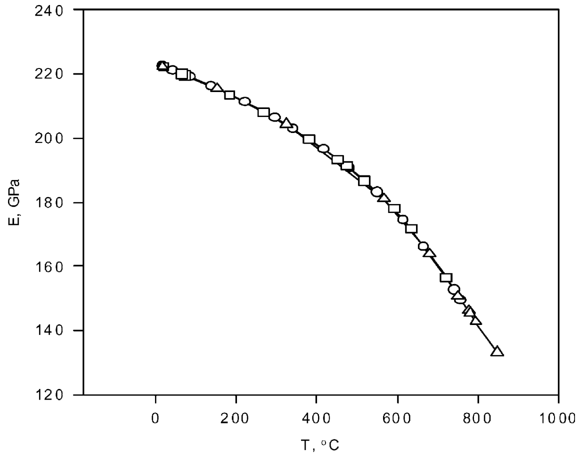

Fig. 4. Temperature dependence of Young's modulus *E* of 12Cr–2WVTaB steel upon heating and cooling: (○, □) test temperature up to 760 °C, (△) test temperature up to 850 °C.

The modulus of elasticity governing the hardness of a material is known to have a weak dependence on microstructure [4]. The temperature dependence of the Young modulus for the steel investigated here after annealing at 800 °C, 1 h, is shown in Fig. 4. The absence of hysteresis upon heating and cooling up to temperatures 760 and 850 °C shows that the investigated steel was thermally stable material.

4. Conclusion

Thermal analysis, austenite transformation during continuous cooling steel, and carbide precipitation were investigated for a 12% Cr martensitic steel. The effects of heat treatment on the matrix solid solution, microstructure and precipitate distribution were studied. The results indicate that the 12% chromium reduced activation steels in various metallurgical states are promising structural materials for a wide range of applications for fusion and fission power reactors.

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