



Sigma phases in an 11%Cr ferritic/martensitic steel with the normalized and tempered condition



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ABSTRACT

At the present time 9–12% Cr ferritic/martensitic (F/M) steels with target operating temperatures up to 650 °C and higher are being developed in order to further increase thermal efficiency so as to reduce coal consumption and air pollution. An 11% Cr F/M steel was prepared by reference to the nominal chemical composition of SAVE12 steel with an expected maximum use temperature of 650 °C. The precipitate phases of the 11% Cr F/M steel normalized at 1050 °C for 0.5 h and tempered at 780 °C for 1.5 h were investigated by transmission electron microscopy. Except for Cr-/Cr-Fe-Co-rich $M_{23}C_6$, Nb-/V-/Ta-Nb-/Nd-rich MX, Fe-rich M_5C_2 , Co-rich M_3C and Fe-Co-rich M_6C phases previously identified in the steel, two types of sigma phases consisting of α -FeCr and α -FeCrW were found to be also present in the normalized and tempered steel. Identified α -FeCr and α -FeCrW phases have a simple tetragonal crystal structure with estimated lattice parameters $a/c = 0.8713/0.4986$ and $0.9119/0.5053$ nm, respectively. The compositions in atomic pct of the observed sigma phases were determined to be approximately 50Fe-50Cr for the α -FeCr, and 30Fe-55Cr-10W in addition to a small amount of Ta, Co and Mn for the α -FeCrW. The sigma phases in the steel exhibit various blocky morphologies, and appear to have a smaller amount compared with the dominant phases Cr-rich $M_{23}C_6$ and Nb-/V-/Ta-Nb-rich MX of the steel. The α -FeCr phase in the steel was found to precipitate at δ -ferrite/martensite boundaries, suggesting that δ -ferrite may rapidly induce the formation of sigma phase at δ -ferrite/martensite boundaries in high Cr F/M steels containing δ -ferrite. The formation mechanism of sigma phases in the steel is also discussed in terms of the presence of δ -ferrite, $M_{23}C_6$ precipitation, precipitation/dissolution of M_2X , and steel composition.

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1. Introduction

9% Cr ferritic/martensitic (F/M) steels have been used in ultra-supercritical power plants with operating steam temperatures up to 600–610 °C [1]. Since the creep rupture strength of 9% Cr F/M steels abruptly decreases during long term creep exposure at temperatures higher than 600 °C [2,3], 9–12% Cr F/M steels with target operating temperatures up to 650 °C and higher are now being developed in order to further increase thermal efficiency so as to reduce coal consumption and air pollution. High Cr F/M steel SAVE12 with an expected maximum use temperature of 650 °C has been laboratory developed in Japan [4–6]. Other developments of high Cr F/M steels for applications at 650 °C in conventional power plants have also been reported, such as 12% Cr F/M heat-resistant steels developed by Knežević et al. [7,8]. However, these developed steels are still in the testing phase. At the present time high Cr FM steels being suitable for actual application in fossil

fired power plants at service temperatures up to 650 °C are not available. Nevertheless, further efforts are needed to increase the upper operating temperature limit of high Cr F/M steels.

The 9–12% Cr steels are normally delivered after a final heat treatment consisting of normalizing at around 1050 °C and tempering in the range 650 °C to 780 °C. The normalizing and tempering leads to a microstructure of tempered martensite consisting of fine ferrite subgrains with carbides precipitated on subgrain boundaries and on prior austenite grain boundaries [9]. The 9–12% Cr steels with normalized and tempered condition usually contain Cr-rich $M_{23}C_6$ carbide and Nb-/V-rich MX carbide/carbonitride, and also contain Cr-rich M_2X carbide/nitride in some cases. The creep resistance of the 9–12% Cr steels can be enhanced by a dispersion of fine carbides/carbonitrides, which are resistant to coarsening. Klueh and Harries [10] have summarized the precipitate phases in normalized-and-tempered, aged, and creep-rupture tested high Cr F/M steels. These precipitate phases consist of $M_{23}C_6$, MX, M_2X , Z-phase ((CrVNb)N), η -carbide (M_6C), vanadium carbide (V_4C_3), Laves phase (Fe_2Mo , Fe_2W , $Fe_2(MoW)$), and Chi (χ) ($M_{18}C$).

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Sigma phase is a well-known intermetallic phase which occurs in transition-metal alloy systems. Sigma phase has been found to form in a number of binary and ternary alloy systems [11]. In 1927 Bain and Griffiths [12] discovered sigma phase in austenitic Fe-Cr-Ni ternary alloys. In 1950 Bergman and Shoemaker [13] determined that sigma phase (σ -FeCr single crystal with 46.5 Cr in atomic pct) has a primitive tetragonal structure containing 30 atoms per unit cell with the lattice parameters $a/c = 0.8799/0.4546$ nm. In engineering alloys, a typical composition for sigma phase is $(\text{Cr,Mo})_x(\text{Fe,Co})_y$ where x and y can vary from 1 to 7, but usually they are approximately equal [14]. In austenitic steels sigma phase usually precipitates after long-term aging at temperatures in the range 650 °C to 900 °C [11,15]. Most of Fe-Cr-Ni base austenitic steels containing more than 17 wt.% Cr are capable of developing sigma phase [14]. Sigma phase formed in the Fe-Cr system is well-known to be a hard and brittle intermetallic compound. Due to the existence of the sigma phase in the Fe-Cr system, this phase has been for long considered as a high danger for the high-alloyed steels [11]. Because generally sigma phase has negative effects on mechanical properties of steels, the investigation with regard to sigma phase formation in steels is of some technological importance.

Since sigma phase was discovered, a great many papers have dealt with the formation of this phase and its effect on the mechanical properties of austenitic steels. In our recent work we found the presence of sigma phase ($\text{Fe}_{0.45}\text{Cr}_{0.45}\text{W}_{0.1}$ having a simple tetragonal lattice) in a commercial 9% Cr F/M steel P92 normalized at 1050 °C for 0.5 h and tempered at 765 °C for 1 h [16]. This finding is important because sigma phase may affect the mechanical properties of the P92 steel. Except in the case of the P92, there appear to have no other previous reports of sigma phase identified in normalized-and-tempered, aged, and creep-rupture tested 9–12% Cr F/M steels. The purpose of this paper is to report two types of sigma phases identified in an 11%Cr F/M steel with the normalized and tempered condition.

2. Experimental Procedures

The material used in this investigation was an 11%Cr F/M steel prepared by reference to the nominal chemical composition of SAVE12 steel [4]. The chemical composition of the experimental steel along with SAVE12 steel is listed in Table 1. A 100 kg ingot of the experimental steel was prepared by vacuum induction melting method. The homogenization of the ingot was performed at 1180 °C for 3 h. The homogenization-treated ingot was then processed by hot-rolling at 940 °C to 1050 °C to a plate with 30 mm thick. The plate was subjected to a final

heat treatment consisting of a normalization at 1050 °C for 0.5 h followed by air cooling and subsequently, a tempering at 780 °C for 1.5 h followed by air cooling. Metallographic samples were chemically etched in a solution of 5 ml hydrochloric acid plus 1 g picric acid in 100 ml ethyl alcohol after mechanical grinding and polishing. Extraction carbon replicas and thin foils were used in this investigation to analyze precipitate phases in the steel. The carbon replicas were prepared by the method described in elsewhere [17]. Thin foil samples were twin-jet electropolished at 40–50 V in a 5% perchloric acid plus 95% ethyl alcohol solution cooled to temperatures in the range -20 °C to -30 °C. The replicas and thin foils were examined in JEM-2100 and JEM-2010HT transmission electron microscopes (TEM) operating at 200 kV. Energy dispersive X-ray (EDX) spectrometers equipped with the TEM were used to analyze the chemical composition of precipitate particles. Precipitate phases were identified by a combination of EDX analysis, selected area electron diffraction (SAED) along with the use of the Powder Diffraction File database of the Joint Committee on Powder Diffraction Standards (JCPDS). Thermodynamic equilibrium calculations of the steel were performed using JMatPro-8.0 computer program.

3. Results and Discussion

Fig. 1 shows the optical micrographs of the 11%Cr F/M steel with the normalized and tempered condition. The microstructure of the steel consists of tempered martensite, δ -ferrite, and precipitates. δ -ferrite grains with a bamboo-like shape formed along the rolling direction. There are obvious boundaries between δ -ferrite and martensite. The amount (area fraction) of δ -ferrite in the tempered 11%Cr F/M steel was determined to be about 15% [18]. The presence of δ -ferrite in the steel was attributed to steel composition having a high Cr equivalent Cr_{eq} , resulting in δ -ferrite formation during solidification process [18, 19]. Our previous TEM investigations of the tempered 11%Cr F/M steel showed that tempered martensite consists of laths, dislocations and precipitates distributed at martensite lath boundaries and in the matrix within laths, meanwhile precipitates with a relatively large size are also present at boundaries between martensite and δ -ferrite. We also have carried out a lot of research on the precipitate phases in the 11%Cr F/M steel with the normalized and tempered condition. There are a large number of precipitates with different sizes and morphologies in the steel. These precipitates have been identified to be mainly Cr-rich M_{23}C_6 carbide, Nb-rich/V-rich/Ta-Nb-rich MC carbides, Nb-rich M(C,N) carbonitride, Fe-rich M_5C_2 carbide [20,21], in addition to a small amount of Cr-Fe-Co-rich M_{23}C_6 carbide, Fe-Co-rich M_6C carbide,

Table 1
Chemical composition of 11%Cr FM steel along with SAVE12 steel (in wt.%).

Steels	C	Si	Mn	Cr	W	Co	V	Nb	Ta	Nd	B	N
11%Cr FM steel	0.10	0.24	0.24	11.2	3.16	2.94	0.22	0.068	0.07	0.03	0.006	0.032
SAVE12	0.10	0.3	0.20	11.0	3.0	3.0	0.20	0.07	0.07	0.04	—	0.04

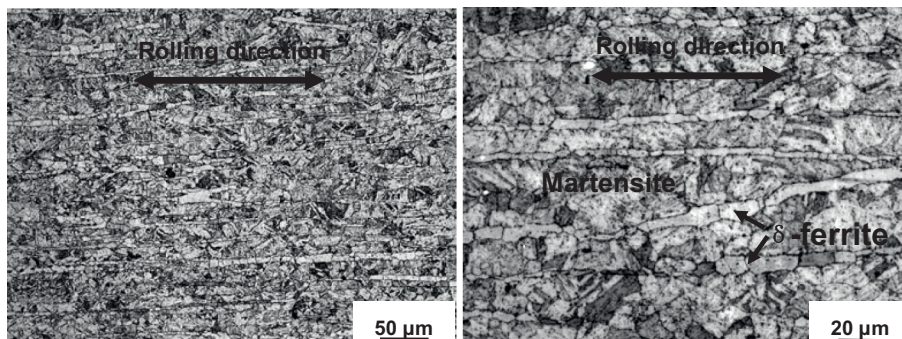


Fig. 1. Optical micrographs of 11%Cr F/M steel with the normalized and tempered condition, showing martensite and δ -ferrite.

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