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Phase Transformations in an Fe-8.3Al-28Mn-4.8Cr-1.2Si-1.2C Alloy

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Abstract

The Phase transformations in the Fe-8.3Al-28Mn-4.8Cr-1.2Si-1.2C alloy have been examined by means of transmission electron microscopy. When the as-quenched alloy was aged at temperatures ranging from 550°C to 1000°C, the phase transformation sequence as the aging temperature increased was found to be $((\text{Fe,Mn,Cr})_7\text{C}_3 + \text{D}_{03}) \rightarrow ((\text{Fe,Mn,Cr})_7\text{C}_3 + \text{B2}) \rightarrow ((\text{Fe,Mn,Cr})_7\text{C}_3 + \alpha) + \gamma \rightarrow ((\text{Fe,Mn,Cr})_7\text{C}_3 + \gamma) \rightarrow \gamma$, in which since the B2 phase present at 650°C contains more silicon than the ferrite phase(α) present at 750°C, the size of the D_{03} domains was much larger than that of the alloy aged at 750°C and then quenched.

Keywords: Fe-Al-Mn-Cr-Si-C, Phase transformations, Continuous ordering transition, TEM, D_{03} , B2 ordered phase

Introduction

In order to improve the high-temperature oxidation resistance and corrosion resistance, silicon and chromium have been added to the Fe-Al-Mn-C alloys[1-5]. Based on their studies, it can be generally concluded that the silicon and chromium additions could achieve these results. The effects of silicon on the microstructures of the Fe-Al-Mn-C alloys have also been examined principally by either optical microscopy or scanning electron microscopy[6,7]. It was proposed that the addition of silicon would enhance the formation of the ferrite phase in the Fe-Al-Mn-C alloys. Recently, we performed transmission electron microscopy observations on the phase transformations in Fe-7.8Al-29.5Mn-1.5Si-1.05C, Fe-9.8Al-28.6Mn-0.8Si-1.0C and Fe-8.3Al-28Mn-4.8Cr-1.2C alloys[9-11]. Consequently, we found that the addition of silicon would enhance the formation of D_{03} and B2 phase, rather than the ferrite phase. This result is quite different from that observed by other workers in the Fe-Al-Mn-Si-C alloys[6,7]. In addition, the phase transformation sequence of the Fe-8.3Al-28Mn-4.8Cr-1.2C alloy as the temperature increased was found to be $\gamma + \kappa'$ -carbide $\rightarrow \gamma + (\text{Fe,Mn,Cr})_{23}\text{C}_6 \rightarrow \gamma + (\text{Fe,Mn,Cr})_7\text{C}_3$.

However, information concerning the microstructural development of Fe-Al-Mn-C alloys with the additions of both silicon and chromium are deficient. Therefore, the purpose of this work is an attempt to examine the phase transformation of the Fe-28Mn-8.3Al-4.8Cr-1.2Si-1.2C.

Experimental Procedure

The present alloy was prepared in a vacuum induction furnace by using 99.5 pct iron, 99.7 pct aluminum, 99.9 pct manganese, 99.5 pct chromium, ferrosilicon and pure

carbon powder. After being homogenized at 1250°C for 24 hours under a controlled protective argon atmosphere, the ingot was hot-forged and then cold-rolled to a final thickness of 2.0mm. The sheet was subsequently solution heat-treated at 1150°C for 1 hour and rapidly quenched into room-temperature water. Aging processes were carefully performed at temperatures ranging from 550°C to 1000°C for various times in a muffle furnace under a controlled protective argon atmosphere and then quenched. The samples were examined by OM, TEM and EDS.

Results and Discussion

Figure 1(a) is an OM of the alloy after being solution heat-treated and then quenched into water. It reveals a single austenite phase with annealing twins. Figure 1(b) is a selected-area diffraction pattern (SADP) taken from the matrix. It reveals that in addition to the reflection spots corresponding to the disordered austenite phase (γ), no extra spots could be observed. Figure 2(a) is a BF of the alloy after being aged at 550°C for 6 hours, showing that fine precipitates were formed within the austenite matrix. It is clear seen in Figure 2(b) that the fine precipitates are $(\text{Fe,Mn})_3\text{AlC}$ carbides (κ' -carbide) with an $\text{L}'1_2$ type structure. On the basis of the above observations, it is concluded that the microstructure of the present alloy aged at 550°C for 6 hours was the austenite phase containing fine κ' carbides. However, with increasing aging time at 550°C, some plate-like precipitates could be observed on the grain boundaries, as shown in Figures 3(a). Figures 3(b) and (c) are two SADPs taken from the plate-like precipitate marked as "A" in Figure 3(a). It was determined that the plate-like precipitate is $(\text{Fe,Cr,Mn})_7\text{C}_3$ carbide having h.c.p. structure with lattice parameters $a=1.0735\text{nm}$ and $c=0.609\text{nm}$. For convenience, the $(\text{Fe,Cr,Mn})_7\text{C}_3$ is

designated as M_7C_3 thereafter. Figure 3(d), an SADP taken from the area marked as “B” in Figure 3(a), shows that the crystal structure is $D0_3$ phase[6-7, 9-11]. Figure 3(e), a (111) $D0_3$ DF electron micrograph of the same area as in Figure 3(a), indicates that the $D0_3$ phase with bright contrast was existent at the aging temperature. Therefore, the microstructure of the alloy present at 550°C was the mixture of M_7C_3 carbide and $D0_3$ phase, which could be preserved up to 625°C .

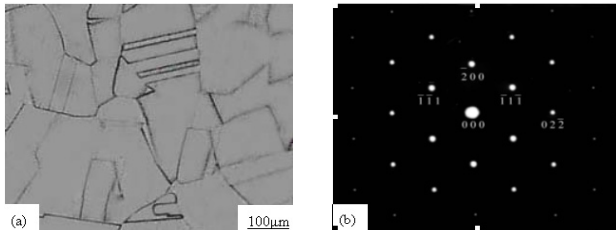


Fig.1 (a) An optical micrograph of the as-quenched alloy. (b) An SADP taken from the austenite matrix of the alloy. The foil normals is [011].

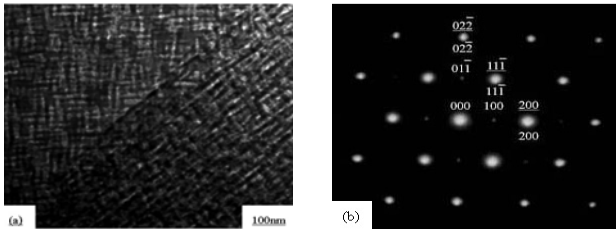


Fig.2 Electron micrographs of the alloy aged at 550°C for 6 h, (a) BF, (b) the SADP taken from a mixed region covering the fine precipitates and the austenite matrix. The zone axis is [011] (hkl = austenite matrix; hkl = κ' -carbide)

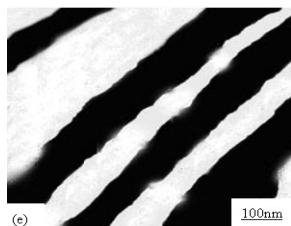
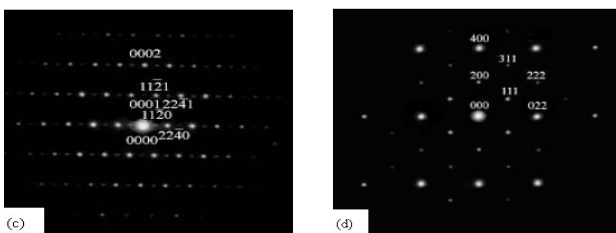
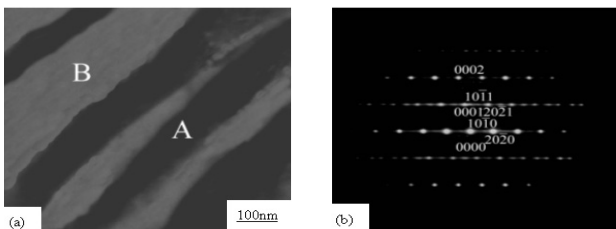


Fig.3 Electron micrographs of the alloy aged at 550°C for 72 hours, (a) BF, (b) and (c) two SADPs taken from the precipitate marked as A in (a). The foil normals are [1210] and [1100], respectively. (d) an SADP taken from the area marked as B in (a). The zone axis is [011]. (hkl = $D0_3$) (e) a (111) $D0_3$ DF.

When the alloy was aged at 650°C for 3 hours and then quenched, the microstructure is similar to the alloy aged at 550°C for short times. Figure 4(a) is a BF electron micrograph of the alloy aged at 650°C for 72 hours. Analyses by the SADP indicated that these two phases were M_7C_3 and $D0_3$, respectively. Therefore, it is likely to conclude that the stable microstructure of the alloy at 650°C was also the mixture of ($M_7C_3 + D0_3$) phases. However, the (111) $D0_3$ DF electron micrograph showed that only extremely fine $D0_3$ domains with $a/2\langle 100 \rangle$ APBs (Anti-Phase Boundaries) were present within the matrix, and the (200) $D0_3$ DF electron micrograph revealed that the whole plate were bright in contrast as illustrated in Figures 4(b) and (c). This indicates that the microstructure of the matrix present at 650°C should be B2 phase and the extremely fine $D0_3$ domains were formed during quenching process from 650°C to room temperature by a $B2 \rightarrow D0_3$ continuous ordering transition[12,13]. On the basis of the above observations, it is concluded that the stable microstructure of the alloy at 650°C should be the mixture of M_7C_3 and B2 phase. It is thus to suggest that a ($M_7C_3 + D0_3$) $\rightarrow$ (B2 + M_7C_3) transition has occurred between 625°C and 650°C .

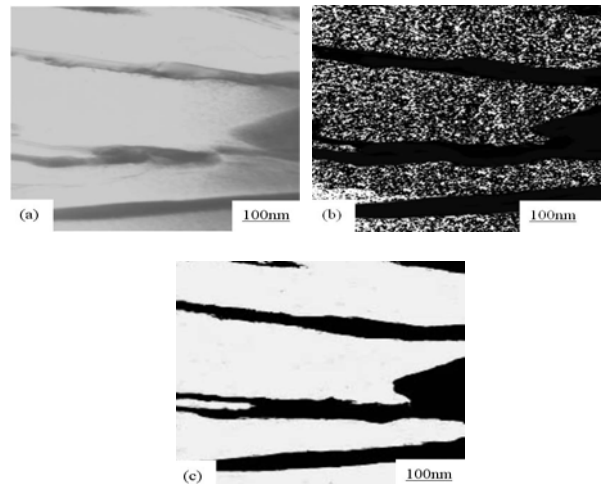


Fig.4 Electron micrographs of the alloy aged at 650°C for 72 hours, (a) BF. (b) and (c) (111) and (200) $D0_3$ DF, respectively.

Figure 5(a) is a BF electron micrograph of the alloy aged at 750°C for 24 hours and then quenched. Analyses of the particles marked as “C” and “D” showed that the microstructures of these two particles were also M_7C_3 and $D0_3$ phase, respectively. However, as shown in Figures 5(b) and 5(c), the (111) and (200) DF electron micrographs revealed that only extremely fine $D0_3$ domains with $a/2\langle 100 \rangle$ APBs and small B2 domains with $a/4\langle 111 \rangle$ APBs were present within the $D0_3$ phase. This indicates that a $\alpha \rightarrow B2 \rightarrow D0_3$ continuous ordering transition has occurred during quenching within the α phase[9-11]. Therefore, the stable microstructure of the present alloy at 750°C was a mixture of austenite(γ) phase containing the precipitations of both M_7C_3 carbide and ferrite(α) phase on the grain boundaries.

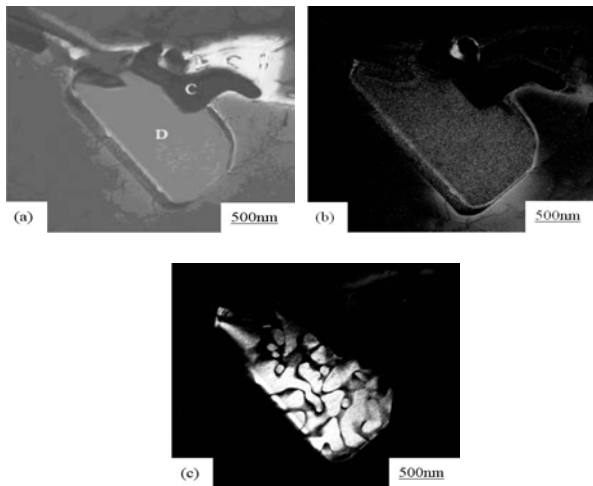


Fig.5 Electron micrographs of the alloy aged at 750°C for 24 hours, (a) BF. (b) and (c) (111) and (200) D0₃ DF, respectively.

When the aging temperature was increased to 850°C, only M₇C₃ carbides were formed on the austenite grain boundaries, as shown in Figure 6. Therefore, the stable microstructure of the present alloy aged at 850°C was a mixture of (γ + M₇C₃) phases. Transmission electron microscopy examinations revealed the stable microstructure of the (γ + M₇C₃) phases could be preserved up to a temperature of 950°C. However, when the alloy was aged at 1000°C for various times and then quenched, only austenite phase could be detected. This indicates that microstructure of present alloy aged at 1000°C or above should be the single austenite phase.

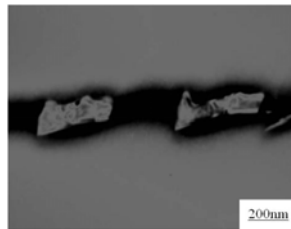


Fig.6 An BF electron micrograph of the alloy aged at 850°C for 4 hours

Based on the above experimental results, some discussions are appropriate. When the alloy was aged at temperatures ranging from 550°C to 1000°C, the phase transformation sequence was found to be (M₇C₃+D0₃) → (M₇C₃+B2) → (M₇C₃+ α + γ) → (M₇C₃+ γ) → γ . This result is quite different from that in our previous studies, in which neither α →B2→D0₃ nor B2→D0₃ continuous ordering transition could be found in Fe-8.8Al-30.0Mn-6.0Cr-1.0C alloy[14]. In order to clarify the apparent difference, an STEM-EDS study was undertaken. The average concentrations of the alloying elements were obtained by analyzing at least ten different EDS spectra of each phase. EDS with a thick-window detector is limited to detect the elements of atomic number of 11 or above; therefore, carbon cannot be examined by this method. The results are summarized in Table I. It is clearly seen in Table I that the concentrations of both the aluminum and silicon in the M₇C₃ carbides

are much less than that in the α , B2 or D0₃ phase, and the reverse result is obtained for the concentrations of the manganese and chromium. It is well-known that manganese and carbon are austenite former elements in the Fe-Mn-Al-C quaternary alloy systems[15, 16], and the carbon content in the M₇C₃ carbide is much higher than that in the present alloy. Therefore, it is reasonable to believe that along with the precipitation of the M₇C₃ carbides, the surrounding austenite phase would be lacked in manganese as well as carbon, and enriched in aluminum and silicon. The austenite phase in the vicinity of the M₇C₃ carbides would become unstable and readily transform into the ferrite phase. Subsequently, it is likely to expect that the α →B2→D0₃ and B2→D0₃ continuous ordering transition would occur during quenching from the aging temperature of 750°C and 650°C in the present alloy, respectively, as shown in Figures 5 and 4. However, in the Fe-Al binary alloys, a α →B2→D0₃ continuous ordering transition could be just occurred as containing 25 at.% aluminum or above[12, 13]. It is clear in Table I that the aluminum content of the ferrite phase at 750°C is only 11.05 wt.% or 20.00 at.%. The reason why a α →B2→D0₃ continuous ordering transition could be observed is silicon addition in the present alloy. From our previous studies in Fe-Al-Mn-Si-C alloys, we have shown that the addition of silicon would lead the α →B2→D0₃ continuous ordering transition to occur[9-11]. Therefore, it is reasonable to expect that owing to the enrichment of silicon in α and B2 phases, a α →B2→D0₃ and B2→D0₃ continuous ordering transition would occur during quenching from 750°C and 650°C in the present alloy, respectively.

Furthermore, when the present alloy was aged at 750°C or 650°C and then quenched, the D0₃ domains would be formed during quenching by a B2→D0₃ continuous ordering transition. However, as compared Figure 4(b) with 5(b), it is obvious that the size of D0₃ domains in Figure 4(b) is much larger than that in 5(b). Based on our previous studies[9-11], it was also found that the addition of silicon would expand the D0₃ phase region. This means that the B2→D0₃ transition temperature would be increased with increasing the addition of silicon. As shown in Table I, it is clearly seen that the silicon concentration of the B2 phase at 650°C is higher than that of the ferrite phase(α) at 750°C. Thus, when the present alloy was aged at 650°C and then quenched, the B2→D0₃ ordering transition would occur at higher temperature, which results in the larger size of D0₃ domains.

Finally, it is interesting to note that the precipitation of (Fe,Cr,Mn)₂₃C₆ carbides was absent in the present alloy. This is contrary to that found in the previous Fe-8.8Al-30.0Mn-6.0Cr-1.0C alloy[14]. Compared to our previous Fe-8.8Al-30.0Mn-6.0Cr-1.0C alloy, in addition to contain 1.0 wt.% silicon in the present alloy, both alloys were composed of similar compositions. Therefore, it is possible to suggest that the addition of silicon to the Fe-Al-Mn-Cr-C alloys would inhibit the precipitation of (Fe,Cr,Mn)₂₃C₆ carbides.

Table I. Chemical Compositions of the Phases Revealed By an Energy-Dispersive Spectrometer (EDS)

Heat Treatment	Phase	Chemical Composition (wt%)				
		Fe	Al	Mn	Cr	Si
As-Quenched	γ phase	Bal.	8.75	30.02	5.86	1.01
550°C Aging	D0 ₃ phase	Bal.	12.29	17.21	1.24	1.70
	(Fe,Cr,Mn) ₇ C ₃ carbide	Bal.	2.10	38.93	36.42	0.54
650°C Aging	B2 phase	Bal.	11.58	22.95	2.53	1.62
	(Fe,Cr,Mn) ₇ C ₃ carbide	Bal.	2.15	39.60	37.82	0.61
750°C Aging	α phase	Bal.	11.05	25.44	2.98	1.22
	(Fe,Cr,Mn) ₇ C ₃ carbide	Bal.	2.17	41.4	38.03	0.64
850°C Aging	(Fe,Cr,Mn) ₇ C ₃ carbide	Bal.	2.25	40.80	38.11	0.66

Conclusion

In the present study, the phase transformations in the Fe-8.8Al-30Mn-6Cr-1.0Si-1.0C alloy have been examined by transmission electron microscopy.

- (1). In the as-quenched condition, the microstructure of the present alloy was the single phase austenite.
- (2). When the as-quenched alloy was aged at temperature ranging from 550 to 1000°C, the phase transformation sequence as the aging temperature increased was found to be $(M_7C_3 + D0_3) \rightarrow (M_7C_3 + B2) \rightarrow (M_7C_3 + \alpha) + \gamma \rightarrow (M_7C_3 + \gamma) \rightarrow \gamma$
- (3). When the as-quenched alloy was aged at 650°C or 750°C and then quenched, the D0₃ domains would be formed during quenching by a B2→D0₃ continuous ordering transition. Since B2 phase present at 650°C contains much more silicon than the ferrite phase present at 750°C, then the size of the D0₃ domains was much larger than that of the alloy aged at 750°C and then quenched.
- (4). When the as-quenched alloy was aged at temperatures ranging from 550 to 1000°C, the (Fe,Cr,Mn)₂₃C₆ carbides were always observed in the Fe-Al-Mn-Cr-C alloys. However, no (Fe,Cr,Mn)₂₃C₆ carbides could be found in the present alloy. Therefore, it is suggested that the addition of silicon to the Fe-Al-Mn-Cr-C alloys would inhibit the precipitation of (Fe,Cr,Mn)₂₃C₆ carbides.

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