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Effects of Aging Treatment on Microstructure of High-Al-content Fe-15Mn-10Al-1.0C Alloy

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The purpose of this study is to examine the effects of aging treatment on the microstructures of the dual-phase Fe-15Mn-10Al-1.0C alloy by optical microscopy (OM) and transmission electron microscopy (TEM). On the basis of the results of our experiments, we reached the following conclusions. Under the as-quenched condition, the microstructure of the Fe-15Mn-10Al-1.0C alloy was a mixture of ferrite and austenite phases. TEM examination revealed that (Fe,Mn)3AlC $_X$ carbides precipitated within the austenite matrix, and (B $_2$ + D0 $_3$) ordered phases could be found within the ferrite region owing to the mechanism of the $\alpha \rightarrow \alpha + B_2 \rightarrow \alpha + D0_3$ ordering transition. This result indicated that an increase in the amount of Al added to the Fe-Mn-Al-C alloy would enhance both the formation of ordered phases within the ferrite matrix and the precipitation of κ -carbides within the austenite matrix. When the alloy was aged at 650 °C for 24 h, phase decomposition of $\gamma \rightarrow \kappa' + B_2$ was observed on the austenite grain boundary.

1. Introduction

Recently, Fe-Mn-Al-C alloys have been successfully developed for some specialized applications, such as high-strength alloy plates, cold-rolled plates, alloy bars, metal inert gas (MIG) welding wires, high-temperature furnace strips, golf clubs, and precision industrial castings. Several patents concerning Fe-Mn-Al-C alloys have been granted, indicating that the Fe-Mn-Al-C alloys have great potential for specific applications. (1-4)

A considerable amount of interest has focused on the effects of various alloying elements and of aging conditions on the microstructure changes and mechanical properties of Fe-Mn-Al-C alloys. To date, most examinations have been focused on the effects of Si, Ti, Mo, Cu, Nb, Ni, and Cr additions as well as the different aging processes in the Fe-Mn-Al-C alloy. However, there has been little investigation into the effects of aging treatment on high-Al-content Fe-Mn-Al-C alloy. Therefore, the purpose of this study is to examine the effects of aging treatment on the microstructure and mechanical properties of the Fe-15Mn-10Al-1.0C alloy by transmission electron microscopy (TEM).⁽⁵⁻⁸⁾

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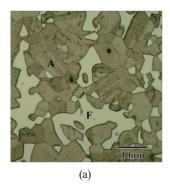
2. Experimental Procedures

Fe-15Mn-10Al-1.0C alloy ingots were prepared in an air induction furnace. After being homogenized at 1200 °C for 24 h, the ingots were hot-forged and then cold-rolled to a final thickness of 30 mm. The sheets were subsequently solution-heat-treated at 1050 °C for 1 h and rapidly quenched in room-temperature water. The aging processes were carefully performed at temperatures ranging from 450 to 750 °C for various durations in salt baths and then the ingots were quenched in water.

Specimens for TEM were prepared by means of a double-jet electrolyte of 10% perchloric acid, 30% acetic acid, and 60% ethanol. The polishing temperature was kept in the range from -10 to 0 °C, and the current was kept in the range of 30 to 40 mA. Electron microscopy was performed using a Philips FEG-20 scanning transmission electron microscope (STEM) operating at 200 kV.

3. Results and Discussion

Under the as-quenched condition, the microstructure of the Fe-15Mn-10Al-1.0C alloy was a mixture of ferrite and austenite phases, as shown in Fig. 1(a). When comparing Fe-15Mn-8Al-1.0C alloy and Fe-15Mn-10Al-1.0C alloy under the as-quenched condition, it was found that the Fe-15Mn-8Al-1.0C alloy was a mixture of only austenite phases, as shown in Fig. 1(b), because an increase in the amount of Al in the Fe-Mn-Al-C alloy causes ferrite phase stabilization. The microstructure was further observed by TEM. Figures 2 and 3 are electron micrographs of the Fe-15Mn-10Al-1.0C alloy under the as-quenched condition. Figure 2(a) is the selected area diffraction pattern (SADP) of the austenite matrix, where the foil normal is [001], and it shows the existence of austenite matrix diffraction spots and the precipitate of κ -carbide diffraction spots. Figure 2(b) is the dark-field (DF) electron micrograph of the (100) κ -carbide precipitate spot in Fig. 2(a), revealing the precipitation of (Fe,Mn)³AlC χ carbides having a L'12 crystal structure, which could be observed within the austenite matrix. Figures 3(a) and 3(b) are the SADPs of the ferrite region, where the foil normals are [001] and [011], respectively, showing



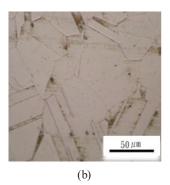


Fig. 1. (Color online) Optical micrograph of Fe-Mn-Al-C alloys after solution heat treatment at 1050 °C for 1 h. (a) Fe-15Mn-10Al-1.0C and (b) Fe-15Mn-8Al-1.0C.

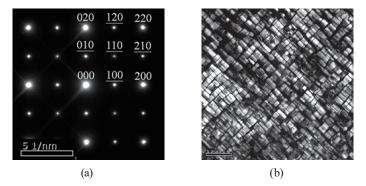


Fig. 2. TEM images of Fe-15Mn-10Al-1.0C alloy under the as-quenched condition. (a) SADP of the austenite matrix. The foil normal is [001]. (b) (100) κ -carbide DF electron micrograph.

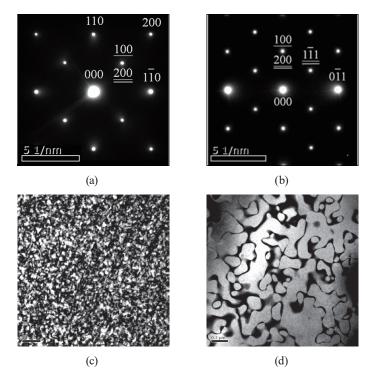


Fig. 3. TEM images of Fe-15Mn-10Al-1.0C alloy under the as-quenched condition. SADPs of the ferrite matrix where (a) the foil normal is [001] and (b) the foil normal is [011]. DF electron micrographs of (c) the (111) D0₃ phase and (d) the (200) D0₃ and (100) B₂ phases.

the existence of ferrite matrix diffraction spots and the precipitate of B_2 and $D0_3$ diffraction spots. Figure 3(c) is the DF electron micrograph of the (111) $D0_3$ spot in Fig. 3(a), showing the existence of a tiny $D0_3$ ordered phase. Figure 3(d) is the DF electron micrograph of the (200) $D0_3$ and (100) B_2 spots in Fig. 3(b), showing the existence of $D0_3$ and B_2 phases. When we compare Figs. 3(c) and 3(d), we find that the B_2 phase is larger than the $D0_3$ phase because the B_2 phase is more stable at high temperature. Additionally, the $D0_3$ phase precipitates during quenching. The experimental results indicated that the B_2 and $D0_3$ ordered phases are found within the ferrite owing to the $\alpha \to \alpha + B_2 \to \alpha + D0_3$ ordering transition mechanism. This

result indicated that an increase in the amount of Al in the Fe-Mn-Al-C alloy would enhance both the formation of ordered phases with the ferrite matrix and the precipitation of κ -carbides within the austenite matrix. (7,8)

Figure 4 and 5 are TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 450 °C for 24 h. Figure 4(a) is the SADP of the austenite matrix and the foil normal is [011]. It shows the existence of austenite matrix diffraction spots and the precipitate of κ -carbide diffraction spots. Figure 4(b) is the DF electron micrograph of the (100) κ -carbide spots in Fig. 4(a), showing the well-grown and newly precipitated (Fe,Mn)³AlC $_X$ carbides with L'12 crystal structures and

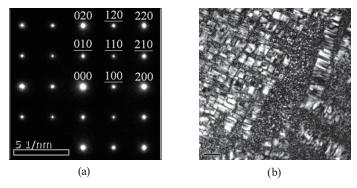


Fig. 4. TEM images of Fe-15Mn-10Al-1.0C alloy aged at 450 °C for 24 h. (a) SADP of the austenite matrix. The foil normal is [001]. (b) $(100) \kappa$ -carbide DF electron micrograph.

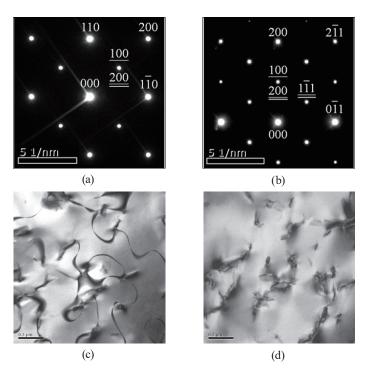
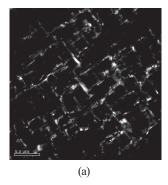


Fig. 5. TEM images of Fe-15Mn-10Al-1.0C alloy aged at 450 °C for 24 h. SADPs of the ferrite matrix where (a) the foil normal is [001] and (b) the foil normal is [011]. DF electron micrographs of (c) the (111) $D0_3$ phase and (d) the (200) $D0_3$ and (100) B_2 phases.

different morphologies within the austenite matrix. Figures 5(a) and 5(b) are SADPs of the ferrite region and the foil normals are [001] and [011], respectively. They show the existence of ferrite matrix diffraction spots and the B_2 and $D0_3$ precipitate diffraction spots. Figures 5(c) and 5(d) are DF electron micrographs of the (111) $D0_3$ and (100) B_2 + (200) $D0_3$ spots in Figs. 5(a) and 5(b), respectively. These show that the growth of the $D0_3$ ordered phase within the ferrite region is stable, with different morphologies of ($\alpha + D0_3$) phases within the ferrite region.

Figures 6(a) and 6(b) are TEM images of the Fe-15Mn-10Al-1.0C alloy that was aged at 550 °C for 24 h. Figure 6(a) is the DF electron micrograph of the (100) κ -carbide spot within the austenite matrix, revealing the precipitation of κ -carbides with an L'12 crystal structure within the austenite matrix. Figure 6(b) is a DF electron micrograph of the (111) D0₃ spot within the ferrite matrix, showing the existence of only the D0₃ phase. No B₂ phase within the ferrite matrix could be observed. It can be seen that the stable temperature of the D0₃ phase is below approximately 550 °C.

Figure 7(a) is an optical micrograph of the Fe-15Mn-10Al-1.0C alloy aged at 650 °C for 24 h, which shows the austenite phase decomposition. Figure 7(b) is a bright-field (BF) electron micrograph of the austenite grain boundary, showing rod-like κ' -carbides within both the austenite grain boundary and the ferrite mixture. Figure 7(c) is the SADP electron micrograph of the region marked P near the carbides, showing the existence of ferrite matrix diffraction spots and the precipitate of B_2 and $D0_3$ diffraction spots. Figure 7(d) is the DF electron micrograph of the (111) D0₃ spot in Fig. 7(c). It shows that in the Fe-15Mn-10Al-1.0C alloy aged at 650 °C for 24 h, phase decomposition of $\gamma \to \kappa' + B_2$ could be observed on the austenite grain boundary. It is worth noting that the $\gamma \to \kappa' + B_2$ transformation was quite different from the γ $\rightarrow \alpha + \kappa', \gamma \rightarrow \kappa' + D0_3$, and $\gamma \rightarrow \kappa' + \beta$ -Mn transformations observed by others. Figures 8(a) and 8(b) are TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 600 °C for 24 h. Figures 8(a) and 8(b) are the DF electron micrographs of the D0₃ and $B_2 + D0_3$ spots within the ferrite mixture, showing the growth of the B₂ phase within the ferrite mixture but the beginning of D0₃ phase decomposition. Figures 9(a) and 9(b) are DF electron micrographs of the Fe-15Mn-10Al-1.0C alloy aged at 650 °C for 24 h, which show tiny D0₃ phases and B₂ phases within the ferrite mixture. It can be seen that the transition temperature of the $B_2 \rightarrow \alpha + D0_3$ ordered transformation was found to be within the narrow range of 600-650 °C, which was quite



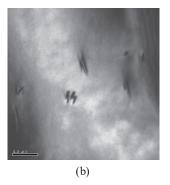


Fig. 6. TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 550 °C for 24 h. DF electron micrographs of (a) the (100) κ -carbide and (b) the (111) D0₃ phase.

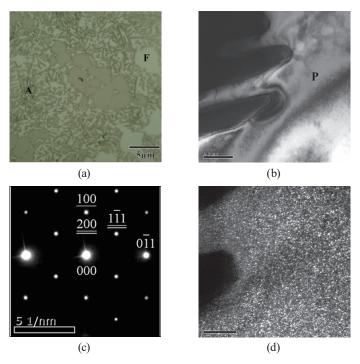


Fig. 7. (Color online) Fe-15Mn-10Al-1.0C alloy aged at 650 °C for 24 h. (a) Optical micrograph. (b) BF electron micrograph. (c) SADP of the region marked as P. The foil normal is [001]. (d) (100) κ -carbide DF electron micrograph.

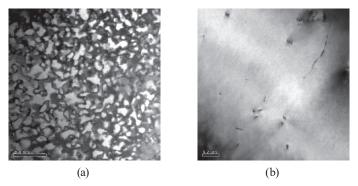


Fig. 8. TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 600 °C for 24 h. DF electron micrographs of (a) the (111) D0₃ phase and (b) the (200) D0₃ and (100) B₂ phases.

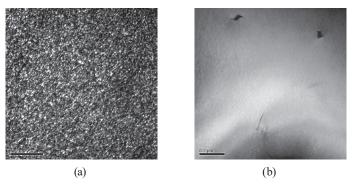


Fig. 9. TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 650 $^{\circ}$ C for 24 h. DF electron micrographs of (a) the (111) D0₃ phase and (b) the (200) D0₃ and (100) B₂ phases.

different from the $\alpha \to B_2 \to \alpha + D0_3$ transition previously observed in the Fe-Mn-Al-C alloy by other researchers.

Figure 10 is an optical micrograph of the Fe-15Mn-10Al-1.0C alloy aged at 750 °C for 24 h, showing the austenite phase decomposition. The microstructure was further observed using a TEM. Figures 11(a) and 11(b) are TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 750 °C for 24 h. Figure 11(a) is a BF electron micrograph of the area mixed austenite and ferrite. Figure 11(b) is the SADP of the austenite matrix and the foil normal is [011]. It shows the existence of only austenite matrix diffraction spots; no precipitate of κ -carbide diffraction spots could be observed. It can be seen that the κ -carbide stability growth temperature range is 450–750 °C, but under the as-quenched condition, the existence of κ -carbide within the austenite matrix could be observed. Figures 12(a) and 12(b) show energy dispersive X-ray spectroscopy (EDS) spectra obtained from the austenite matrix of the Fe-15Mn-10Al-1.0C alloy aged at 750 °C for 24 h and from the austenite matrix of the Fe-15Mn-10Al-1.0C alloy under the as-quenched condition, respectively. The difference between these two EDS spectra could be caused by the presence of manganese. It is clear that austenite matrix of the alloy under the asquenched condition is manganese-rich and has a manganese-to-aluminum ratio of 3:1, which is confirmed by electron diffraction analysis. According to Ref. 5, this result is caused by the κ -carbide precipitation by spinodal decomposition during quenching.

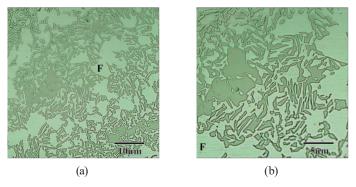


Fig. 10. (Color online) Optical micrograph of the Fe-15Mn-10Al-1.0C alloy aged at 750 $^{\circ}$ C for 24 h. (a) 100^{\times} and (b) 500^{\times} .

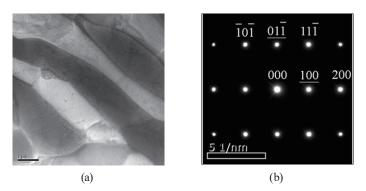
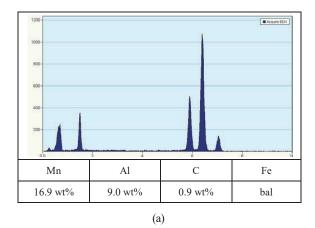


Fig. 11. TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 750 °C for 24 h. (a) BF electron micrograph. (b) SADP of austenite matrix. The foil normal is [011].



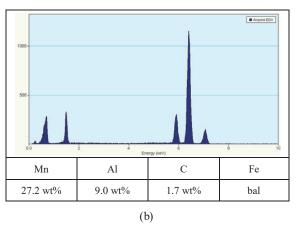
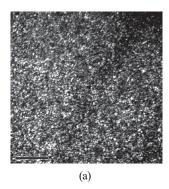


Fig. 12. (Color online) EDS spectra obtained from the austenite matrix of Fe-15Mn-10Al-1.0C alloy. (a) EDS spectrum of austenite matrix under the as-quenched condition and (b) EDS spectrum of the austenite matrix aged at 750 °C for 24 h.



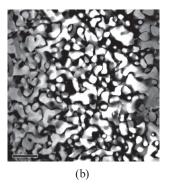


Fig. 13. TEM images of the Fe-15Mn-10Al-1.0C alloy aged at 750 °C for 24 h. DF electron micrographs of (a) the (111) $D0_3$ phase and (b) the (200) $D0_3$ and (100) B_2 phases.

Figure 13(a) is the DF electron micrograph of the D0₃ phase within the ferrite mixture, showing the existence of a tiny D0₃ ordered phase. Figure 13(b) is the DF electron micrograph of the D0₃ and B₂ phases, showing the existence of the B₂ phase. This microstructure is similar to that under the as-quenched condition. Experimental results indicated that when the Fe-15Mn-10Al-1.0C alloy was aged at 450 to 750 °C, the ordering phase transformation of $\alpha \to B_2 \to \alpha + D0_3$ occurred within the ferrite region. The transition temperature of the B₂ $\to \alpha + D0_3$ ordered transformation was found to be within the narrow range of 550–600 °C, which was quite different from the $\alpha \to B_2 \to \alpha + D0_3$ transition observed in the Fe-Mn-Al-C alloy by other researchers.

4. Conclusions

1. When the Fe-15Mn-10Al-1.0C alloy was aged at 450 °C for 24 to 72 h, the growth of the D0₃ ordered phase within the ferrite region revealed stable ($\alpha + D0_3$) phases with different morphologies within the ferrite region. Additionally, well-grown and newly precipitated

- $(Fe,Mn)^3AlC_X$ carbides with L'12 crystal structures with different morphologies could be observed within the austenite matrix.
- 2. When the Fe-15Mn-10Al-1.0C alloy was aged at 650 °C for 24 h, the phase decomposition of $\gamma \to \kappa' + B_2$ could be observed on the austenite grain boundary. It is worth noting that the $\gamma \to \kappa' + B_2$ transformation was quite different from the $\gamma \to \alpha + \kappa'$, $\gamma \to \kappa' + D0_3$, and $\gamma \to \kappa' + \beta$ -Mn transformations observed by other researchers.
- 3. When the Fe-15Mn-10Al-1.0C alloy was aged at 450 to 750 °C, the ordering phase transformation of $\alpha \to B_2 \to \alpha + D0_3$ occurred within the ferrite region. The transition temperature of the $B_2 \to \alpha + D0_3$ ordered transformation was found to be within the narrow range of 550–600 °C, which was quite different from the $\alpha \to B_2 \to \alpha + D0_3$ transition observed in the Fe-Mn-Al-C alloy by other researchers.

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