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PHASE TRANSFORMATIONS IN AN Fe-8Al-10Ni-2C ALLOY

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Introduction

Recently, we have performed transmission electron microscopy examinations of the phase transformations in an Fe-8Al-5Ni-2C alloy, prepared by conventional casting process [1]. In the as-quenched condition, the microstructure of the alloy was austenite (γ) phase containing fine (Fe,Ni)₃AlC carbides. This is similar to that observed by Choo and Kim [2] in an as-solidified Fe-8Al-5Ni-2C alloy, prepared by rapid solidification process. However, when the as-quenched alloy was aged at 550°C for moderate times, the fine (Fe,Ni)₃AlC carbides grew within the austenite matrix and a $\gamma \rightarrow$ coarse (Fe,Ni)₃AlC carbide $+\alpha$ (ferrite) reaction occurred on the austenite grain boundaries. For convenience, the κ' -carbide and *k*-carbide were used to represent the (Fe,Ni)₃AlC carbide formed within the austenite matrix and on the austenite grain boundaries, respectively [2]. With increasing aging time, B2 particles started to precipitate within the ferrite phase. Consequently, the final microstructure of the Fe-8Al-5Ni-2C alloy at 550°C was a mixture of κ -carbide, α and B2 phases. This result is quite different from the mixture of κ -carbide and α phases found by Choo and Kim in the rapidly solidified Fe-8Al-5Ni-2C alloy aged at 550°C for a long time [2]. Extending this work, it is interesting to study successively the effects of the higher nickel content on the phase transformations of the Fe-Al-Ni-C alloy. Therefore, the purpose of this work is an attempt to examine the phase transformations in an Fe-8Al-10Ni-2C alloy. Based on the present study, it was found that when the present alloy was aged at temperatures ranging from 550°C to 1100°C for a long time and then quenched, the phase transformation sequence, as the aging temperature increased, was κ -carbide + B2 $\rightarrow \gamma$ + B2 $\rightarrow \gamma$ + $\alpha \rightarrow \gamma$. This phase transformation has never been observed in the Fe-Al-Ni-C alloys before.

Experimental Procedure

The alloy, Fe-8wt%Al-10wt%Ni-2wt%C, was prepared in a vacuum induction furnace by using 99.5% iron, 99.7% aluminum, 99.9% nickel, and pure carbon powder. After being homogenized at 1250°C for 12 hours under a protective argon atmosphere, the ingot was hot-forged and then cold-rolled to a final thickness of 2.0 mm. The sheet was subsequently solution heat-treated at 1200°C for 2 hours and quenched into room-temperature water. Aging processes were carefully performed at temperatures ranging from 550°C to 1100°C for various times in a muffle furnace under a protective argon atmosphere and then quenched.

Electron microscopy specimens were prepared by means of a double-jet electropolisher with an electrolyte of 30% acetic acid, 60% ethanol, and 10% perchloric acid. Electron microscopy was



Figure 1. Electron micrographs of the alloy aged at 550°C for 10 min. (a) BF (K = κ -carbide, B = B2), (b) and SADP taken from an area covering two precipitates marked as K and B in (a). The foil normals of the κ -carbide and B2 phases are $[0\overline{1}1]$ and [001], respectively. (hkl = κ -carbide, *hkl* = B2 phase).

performed on a JEOL-2000FX scanning transmission electron microscope operating at 200 kV. This microscope was equipped with a Link ISIS 300 energy-dispersive X-ray spectrometer (EDS) for chemical analysis. Quantitative analyses of elemental concentrations for Fe, Al, and Ni were made with the aid of a Cliff-Lorimer Ratio Thin Section method.

Results and Discussion

Transmission electron microscopy examinations indicated that in the as-quenched condition, the microstructure of the alloy was austenite containing fine κ' -carbides. This is similar to that found in the as-quenched Fe-8Al-5Ni-2C alloy [1], or in the as-solidified Fe-(7-8)Al-(5-10)Ni-(1.6-2.0)C alloys [2]. When the as-quenched alloy was aged at 550°C for moderate times, the fine κ' -carbides grew and a precipitation started to occur on the austenite grain boundaries. A typical microstructure is shown in Figure 1(a). Electron diffraction analyses indicated that the two kinds of grain boundary precipitates were κ -carbide and B2 phase, respectively. Figure 1(b), an SADP taken from an area covering two precipitates marked as K and B in Figure 1(a), indicates that the orientation relationship between the κ -carbide and the B2 phase is $[0\bar{1}1]_{\kappa}/[001]_{B2}$ and $(1\bar{1}\bar{1})_{\kappa}/((1\bar{1}0)_{B2})$. This result indicates that a $\gamma \rightarrow 1$ κ -carbide + B2 reaction occurred on the austenite grain boundaries. With increasing aging time at the same temperature, the $\gamma \rightarrow \kappa$ -carbide + B2 reaction proceeded toward the inside of austenite grains, as shown in Figure 2. Figure 2(a) is a BF image of the alloy aged at 550°C for 1 hour, showing that the precipitation was made up of alternating κ -carbide and B2 plates to form a lamellar structure. Figures 2(b) and (c) are $(100)_{\kappa\text{-carbide}}$ and $(100)_{B2}$ DF images, exhibiting that the whole $\kappa\text{-carbide}$ and B2 plates are bright in contrast. Apparently, the microstructure of the alloy in the equilibrium stage at 550°C was a mixture of κ -carbide and B2 phases.

Figure 3(a) shows a BF image of the alloy aged at 750°C for 1 hour and then quenched, revealing the presence of the lamellar product. However, it is clearly seen in this figure that a mottled structure could be observed within the κ -carbide plates. The $(100)_{\kappa'\text{-carbide}}$ DF image showed that only fine κ' -carbides were present, as illustrated in Figure 3(b). Since the size of the fine κ' -carbides formed within the austenite phase is comparable to that observed in the as-quenched alloy, it is suggested that these fine κ' -carbides were formed during quenching from the aging temperature. Shown in Figure 3(c) is a $(100)_{B2}$ DF image, indicating that the whole B2 plates are bright in contrast. Therefore, the final microstructure of the alloy at 750°C was a mixture of γ and B2 phases. Transmission electron microscopy examinations of thin foils revealed that the mixture of γ and B2 phases could be preserved up to 800°C.



Figure 2. Electron micrographs of the alloy aged at 550°C for 1 hour. (a) BF, (b) $(100)_{\kappa\text{-carbide}}$ DF, and (c) $(100)_{B2}$ DF.

When the alloy was aged at 850°C, the morphology of the precipitates changed from plate-like to irregular shape as shown in Figure 4. Figures 4(a) and (b) are $(100)_{\kappa'\text{-carbide}}$ and $(100)_{B2}$ DF images, clearly showing that only fine κ' -carbides and small B2 domains with $a/2\langle 111 \rangle$ anti-phase boundaries could be observed [3,4]. This indicates that the final microstructure of the alloy present at 850°C should be a mixture of γ and α phases, and the small B2 domains were formed by a $\alpha \rightarrow B_2$ continuous



Figure 3. Electron micrographs of the alloy aged at 750°C for 1 hour. (a) BF, (b) $(100)_{\kappa'-carbide}$ DF, and (c) $(100)_{B2}$ DF.



Figure 4. Electron micrographs of the alloy aged at 850°C for 1 hour. (a) $(100)_{\kappa'-\text{carbide}}$ DF, and (b) $(100)_{B2}$ DF.

ordering transition during quenching from the aging temperature [3,4]. Progressively higher temperature aging and quenching experiments indicated that the mixture of γ and α phases could exist up to 1050°C. However, the amount of the ferrite phase decreased with increasing the aging temperature, as shown in Figure 5. As the aging temperature was increased to 1100°C, only fine κ' -carbides were formed within the austenite matrix and no evidence of the ferrite phase could be detected. This feature is similar to that observed in the as-quenched alloy. This indicates that the microstructure of the alloy present at 1100°C or above should be an austenite phase.

On the basis of the above observations, some discussion is appropriate. When the present alloy was aged at 550°C, the final microstructure was a mixture of κ -carbide and B2 phases. This result is quite different from the mixture of κ -carbide, α and B2 phases found by the present workers in the Fe-8Al-5Ni-2C alloy [1]. In the previous study, we have shown that during the early stage of isothermal aging at 550°C, a $\gamma \rightarrow \kappa$ -carbide + α reaction occurred on the austenite grain boundaries. With increasing aging time at the same temperature, the B2 particles started to precipitate by nucleation and growth mechanism within the ferrite phase. Whereas, on aging at 550°C for various times, a $\gamma \rightarrow$ κ -carbide + B2 reaction occurred on the austenite grain boundaries and no evidence of the ferrite phase could be detected in the present alloy. In addition, when the present alloy was aged at a higher temperature (i.e. 850°C) and then quenched, a $\alpha \rightarrow B2$ continuous ordering transition occurred within the ferrite phase. This transformation has never been observed in the Fe-Al-Ni-C alloys before. In order to clarify this feature, a STEM-EDS study was undertaken. The average concentrations of the substitutional alloying elements were obtained from at least ten different EDS profiles. The results are summarized in Table 1. It is clearly seen in Table 1 that the nickel concentration in the κ -carbide is only 2.82 wt%, which is similar to that of the κ -carbide in the Fe-8Al-5Ni-2C alloy [1]; while the nickel concentration in the α or B2 phase is much greater than that in the α or B2 phase of the previous alloy. This result seems to imply that the higher nickel addition in the Fe-Al-Ni-C alloy may not increase the nickel concentration of the κ -carbide, but the nickel concentration of the α or B2 phase. Therefore, it



Figure 5. Electron micrographs of the alloy aged at 1050° C for 1 hour. (a) $(100)_{K'-carbide}$ DF, and (b) $(100)_{B2}$ DF.

Heat Treatment	phase	Chemical Composition (wt.%)		
		Fe	Al	Ni
As-quenched	$\gamma + \kappa'$ -carbides	81.93	7.95	10.12
550°C aging	κ-carbode	89.91	7.27	2.82
	B2 phase	76.84	8.40	14.76
750°C aging	$\gamma + \kappa'$ -carbides	87.32	7.36	5.32
	B2 phase	76.90	8.72	14.38
850°C aging	$\gamma + \kappa'$ -carbides	85.20	7.48	7.32
	B2 phase ($\alpha \rightarrow B2$)	77.33	9.02	13.65
1050°C aging	$\gamma + \kappa'$ -carbides	83.40	7.65	8.95
	B2 phase ($\alpha \rightarrow$ B2)	78.22	9.26	12.52

TABLE 1 Chemical Compositions of the Phases Revealed by an Energy-Dispersive Spectrometer (EDS)

is proposed that the higher nickel content not only pronouncedly expanded the B2 phase field, as reported in the previous studies [1,5], but also changed the B2 formation mechanism from the nucleation and growth mechanism to the continuous ordering transition.

A second important feature of the present study is that when the alloy was aged at 750°C or above and then quenched, the fine κ' -carbides were always formed within the austenite phase. This feature has also never been observed in the Fe-Al-Ni-C alloys before. In the present study, although no direct evidence shows the carbon concentration in the phases, it is well-known that the solubility of carbon in the α or B2 phase is very low [6]. Furthermore, in the previous studies of the Fe-(8–11)Al-(28– 35)Mn-(0.8–2.0)C alloys [7–18], it was reported that the as-quenched microstructure of the alloy containing C \leq 1.3 wt% was single-phase austenite; however, the fine κ' -carbides could be formed during quenching within the austenite phase in the alloy containing C \geq 1.62 wt%. Therefore, it is reasonable to believe that along with the precipitation of the α or B2 phase, the carbon concentration in the austenite phase would be increased to more than 2.0 wt% of the as-quenched alloy, which resulted in the formation of the fine κ' -carbides within the austenite phase during quenching.

Finally, it is interesting to note that in order to enhance the phase stability of austenite and fcc-based κ -carbide, the austenite-stabilizing elements such as manganese and nickel have been added to the Fe-Al-C alloys so as to form Fe-Al-Mn-C or Fe-Al-Ni-C alloy system [1–2,7–18]. In our previous studies [15–18], we have shown that when the Fe-Al-Mn-C alloys were aged at various temperatures, the manganese content in both the austenite and κ -carbide was much greater than that in the α phase. In contrast, the present study provided an evidence that the nickel concentration in both the α and B2 phases is significantly higher than that in the austenite and κ -carbide.

Conclusions

The as-quenched microstructure of the Fe-8Al-10Ni-2C alloy was austenite containing fine κ' -carbides. During the early stage of isothermal aging at 550°C, the fine κ' -carbides grew and a $\gamma \rightarrow \kappa$ -carbide + B2 reaction started to occur on the austenite grain boundaries. With increasing aging time at the same temperature, the $\gamma \rightarrow \kappa$ -carbide + B2 reaction proceeded toward the inside of austenite grains. Therefore, the final microstructure of the alloy at 550°C was a mixture of κ -carbide and B2 phases. With increasing the aging temperature from 550°C to 1100°C, the final microstructure of the alloy was found to be (κ -carbide + B2) $\rightarrow (\gamma + B2) \rightarrow (\gamma + \alpha) \rightarrow \gamma$. In addition, during quenching from the aging temperature, the fine κ' -carbides were formed within the austenite phase and a $\alpha \rightarrow B2$ continuous ordering transition occurred within the ferrite phase.

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