

Communications

Grain Boundary Precipitation Reactions in a Wrought Fe-8Al-5Ni-2C Alloy Prepared by the Conventional Ingot Process

C.N. HWANG and T.F. LIU

Phase transformations in the Fe-8Al-5Ni-2C alloy, prepared by rapid solidification processing (RSP), have been studied by Choo and Kim.^[1] They showed that the microstructure of the alloy in the as-solidified condition was austenite phase (γ) containing fine ordered L'₂-type precipitates. The formation of these fine ordered L'₂-type precipitates, lying along the $\langle 100 \rangle$ directions in the matrix, was attributed to the spinodal decomposition. It was also shown that, on aging at 823 K, the fine ordered precipitates grew and, at the same time, α (ferrite) and κ -carbide formed by a heterogeneous reaction at the γ/γ grain boundaries. With increasing aging time, the heterogeneous reaction became predominant, leading to a two-phase microstructure of α and κ -carbide in the end.^[1] Recently, we performed transmission electron microscopy (TEM) observations of the phase transformations in the Fe-8Al-5Ni-2C alloy, prepared by conventional casting process. It was found that the microstructure of the alloy in the as-quenched condition was austenite phase containing fine ordered L'₂-type precipitates, similar to the previous results for the as-solidified alloy. However, when the as-quenched alloy was aged at 823 K for a long time, the equilibrium microstructure was observed to be a mixture of ($\alpha + \text{B2} + \kappa$ -carbide), rather than $\alpha + \kappa$ -carbide as reported by Choo and Kim.

The alloy, Fe-8Al-5Ni-2C, was prepared in an air induction furnace by using 99.5 pct iron, 99.7 pct aluminum, 99.5 pct nickel, and pure carbon powder. After being homogenized at 1523 K 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 1323 K for 2 hours and quenched into room-temperature water. The aging treatments were performed at 823 K for various times in a salt bath. The microstructures of the alloy were examined by means of optical microscopy and TEM. Thin foil specimens for TEM were prepared in a double jet electropolisher, using an electrolyte of 30 pct acetic acid, 60 pct ethanol, and 10 pct perchloric acid at 263 to 283 K. The current density was kept in the range from 1.5 to 2.0×10^4 A/m². Electron microscopy was performed on a JEOL* 2000FX scanning

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transmission electron microscope (STEM) operating at 200 kV. Elemental distributions were examined using a LINK-AN 10000 energy-dispersive X-ray spectrometer

(EDS). Quantitative analyses of elemental concentrations for Fe, Al, and Ni were made with the aid of a ZAF-corrected program on the LINK system (where Z = backscatter coefficient, A = absorption coefficient, and F = fluorescence coefficient).

The optical micrograph of the as-quenched alloy exhibited austenite grains with annealing twins, as shown in Figure 1(a). Figures 1(b) and (c) are bright-field (BF) and dark-field (DF) electron micrographs of the as-quenched alloy, revealing that a high density of fine ordered precipitates formed within the austenite matrix. Figure 1(d), a selected-area diffraction pattern (SADP), demonstrates that the fine precipitates have an ordered L'₂-type structure.^[1-9] Hence, obviously, these observations are essentially identical to those reported by Choo and Kim for their as-rapidly solidified alloy.^[1] When the as-quenched alloy was aged at 823 K for moderate times, the fine ordered L'₂-type precipitates grew in the matrix. At the same time, a heterogeneous reaction started to occur on the γ/γ grain boundaries. Electron diffraction analysis indicated that the two kinds of coarse precipitates were α and κ -carbide having an ordered L'₂-type structure,^[1-4] respectively. Figures 2(a) through (c) are BF, 110 α , and 100 κ -carbide DF electron micrographs of the same area, showing the presence of the α and κ -carbide. This reaction can be written as $\gamma \rightarrow \alpha + \kappa$ -carbide. With increasing aging time at the same temperature, the $\gamma \rightarrow \alpha + \kappa$ -carbide transition proceeded toward the inside of austenite grains. Apparently, the microstructure of the alloy in the equilibrium stage at 823 K seems to be a mixture of ($\alpha + \kappa$ -carbide), as reported earlier.^[1]

Figure 3 shows an optical micrograph of the alloy aged at 823 K for 20 minutes, in which grain boundary reactions have taken place. However, a careful TEM examination revealed that B2-type precipitates were formed within the α phase, as follows. Figure 4(a) shows a BF image of lamella product. Figure 4(b) is an SADP taken from an area covering a ($\alpha + \text{B2}$) particle and a κ -carbide, indicating the Nishiyama-Wassermann orientation relationship^[10] between the α (or B2) and κ -carbide: $[001]_{\alpha} // [0\bar{1}1]_{\kappa}$, $(\bar{1}10)_{\alpha} // (\bar{1}11)_{\kappa}$. Figures 4(c) and (d), 100 κ -carbide, and 100 B2 DF electron micrographs clearly exhibit the κ -carbides and B2 precipitates, respectively. Accordingly, the stable microstructure of the present alloy at 823 K should be a mixture of ($\alpha + \text{B2} + \kappa$ -carbide).

This result is quite different from that reported by Choo and Kim.^[1] The apparent discrepancy may be attributed to the following two possible reasons. (1) The preparations of the alloys were different. In Choo and Kim's studies, the Fe-8Al-5Ni-2C alloy was prepared by RSP, whereas the present alloy was prepared by conventional casting process. (2) In their studies, only low magnification BF electron micrographs were provided. No electron diffraction investigation was made. In order to get some insight into the origin of the precipitation of B2 phase, EDS analyses were undertaken. The average concentrations of the substitutional alloying elements were obtained from at least ten different EDS profiles of each phase. The results are summarized in Table 1. It is worthwhile to note that, since in the present study the EDS analyses were made in the STEM mode and the size of the B2 precipitates (about 70 nm) examined is slightly larger than that of the electron beam spot (40 nm), some errors are inevitable in determining the elemental concentrations in the B2 precipitates. Nonetheless,

C.N. HWANG, Graduate Student, and T.F. LIU, Professor and Chairman, are with the Institute of Materials Science and Engineering, National Chiao Tung University, Taiwan, 30049, Republic of China.

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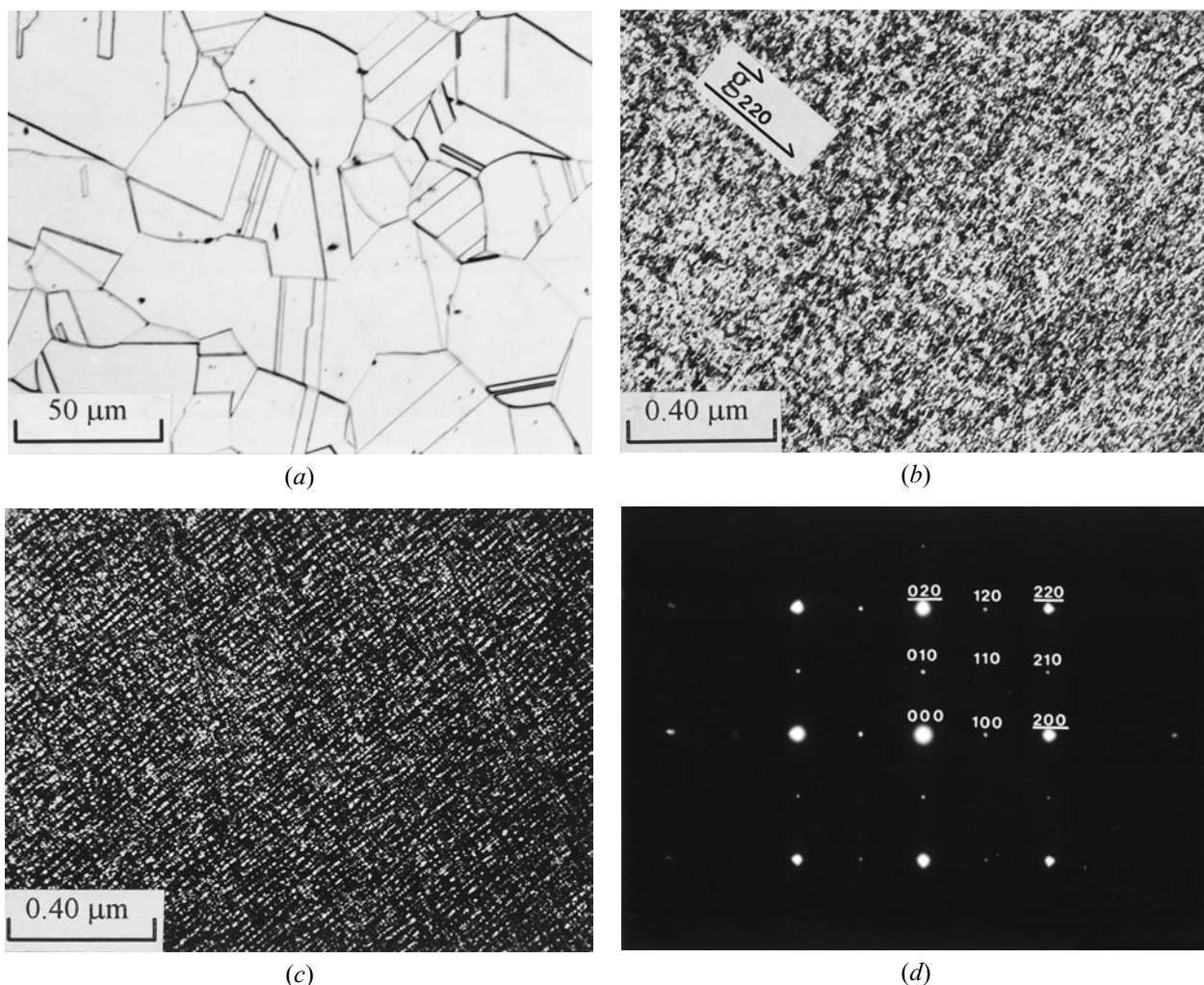


Fig. 1—(a) An optical micrograph. (b) through (d) Electron micrographs of the as-quenched alloy: (b) BF, (c) a 100 DF of the L'_{2} precipitates, and (d) an SADP taken from an area covering the austenite matrix and fine precipitates. The foil normal is $[001]$ (hkl = austenite matrix, and hkl = L'_{2} precipitate).

less, the results given in Table 1 indicated that the nickel concentration in both the α and B2 precipitates is distinctly higher than that in the κ -carbide.

Therefore, it is reasonable to conclude that both the α and B2 phases are enriched in nickel. In a previous study, we have shown that the addition of 4.1 at. pct nickel to an Fe-23.1Al alloy pronouncedly expanded the B2 phase field and caused the formation of a high density of B2 precipitates within the α matrix.^[11] Therefore, it is reasonable to believe that during the heterogeneous precipitation of ($\alpha + \kappa$ -carbide) on the γ/γ grain boundaries, the ferrite particle would be enriched in nickel. The enrichment of nickel then would lead to the formation of the B2 precipitates within the α phase. Since this process involves diffusion of nickel, it is thus anticipated that the formation of the B2 precipitates occurred only after prolonged aging at 823 K.

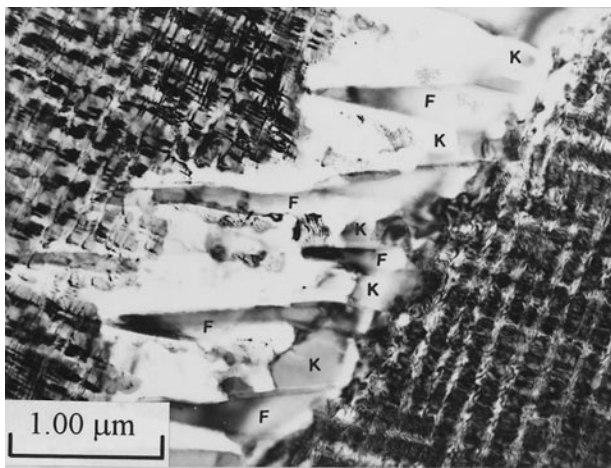
Finally, it is worth mentioning that the microstructures of the Fe-Al-C alloys have been studied by many workers.^[12-19] However, because of high temperature instability of the alloys, all of their studies were only focused on the as-quenched microstructures. To the authors' knowledge, no detailed information concerning the aged microstructure has been provided. In order to enhance the phase stability of austenite, some elements such as manganese and nickel

have been added to the alloys so as to form Fe-Al-Mn-C and Fe-Al-Ni-C alloy systems. In fact, the phase transitions in the Fe-Al-Mn-C austenitic alloys have been extensively investigated recently.^[2,3,5-9,20-26] In contrast, the experimental studies of the Fe-Al-Ni-C austenitic alloys are relatively deficient. The present preliminary study provided further evidence that, although both manganese and nickel are austenite-stabilizing elements, the later stage microstructural changes occurring during aging of Fe-Al-Ni-C alloys differ from those in the Fe-Al-Mn-C alloys. Obviously, in order to further understand the phase transitions in the Fe-Al-Ni-C alloys, much more work is needed.

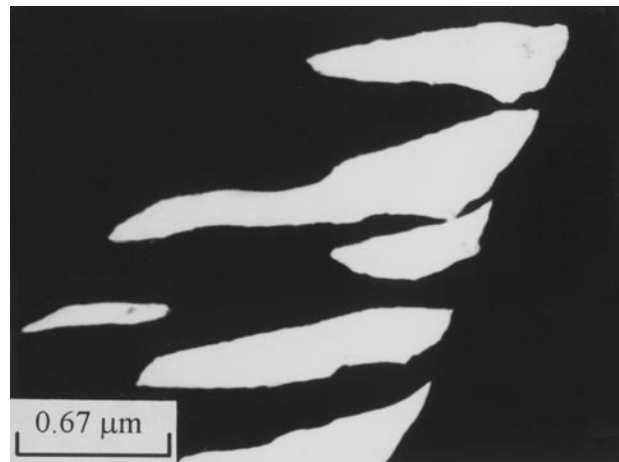
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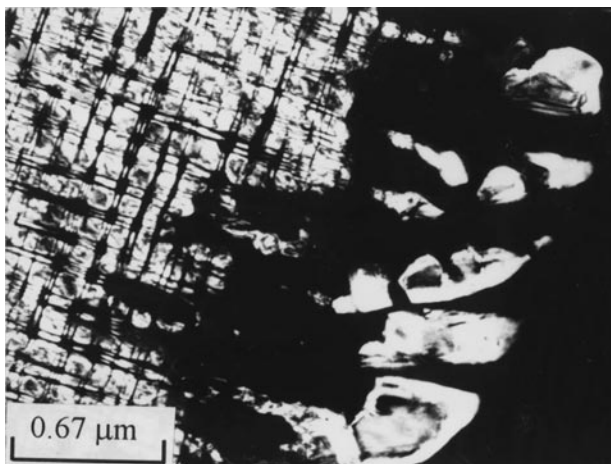
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(a)



(b)



(c)

Fig. 2—Electron micrographs of the alloy aged at 823 K for 3 min: (a) BF (F = ferrite, and K = κ -carbide), (b) 110 α DF, and (c) 100 κ DF images.

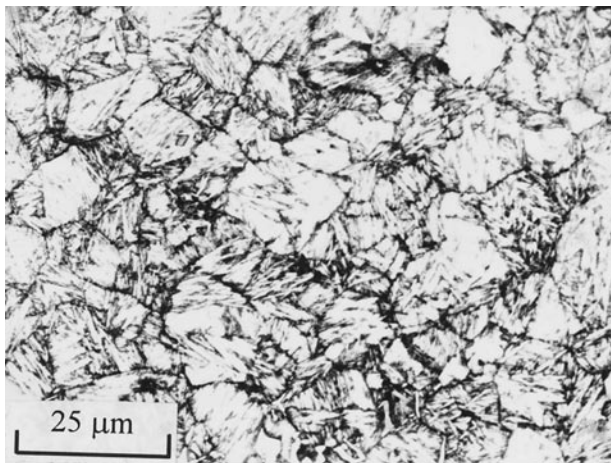


Fig. 3—An optical micrograph of the alloy aged at 823 K for 20 min.

Table I. Chemical Compositions of Substitutional Elements in the Phases, as Determined by EDS Ignoring the Carbon Content

Heat Treatment	Phase	Chemical Composition (Wt Pct)		
		Fe	Al	Ni
823 K aging 3 min	α	85.98	7.81	6.21
	κ	88.88	8.35	2.77
823 K aging 20 min	α	88.85	7.52	4.01
	B2	81.67	8.42	9.51

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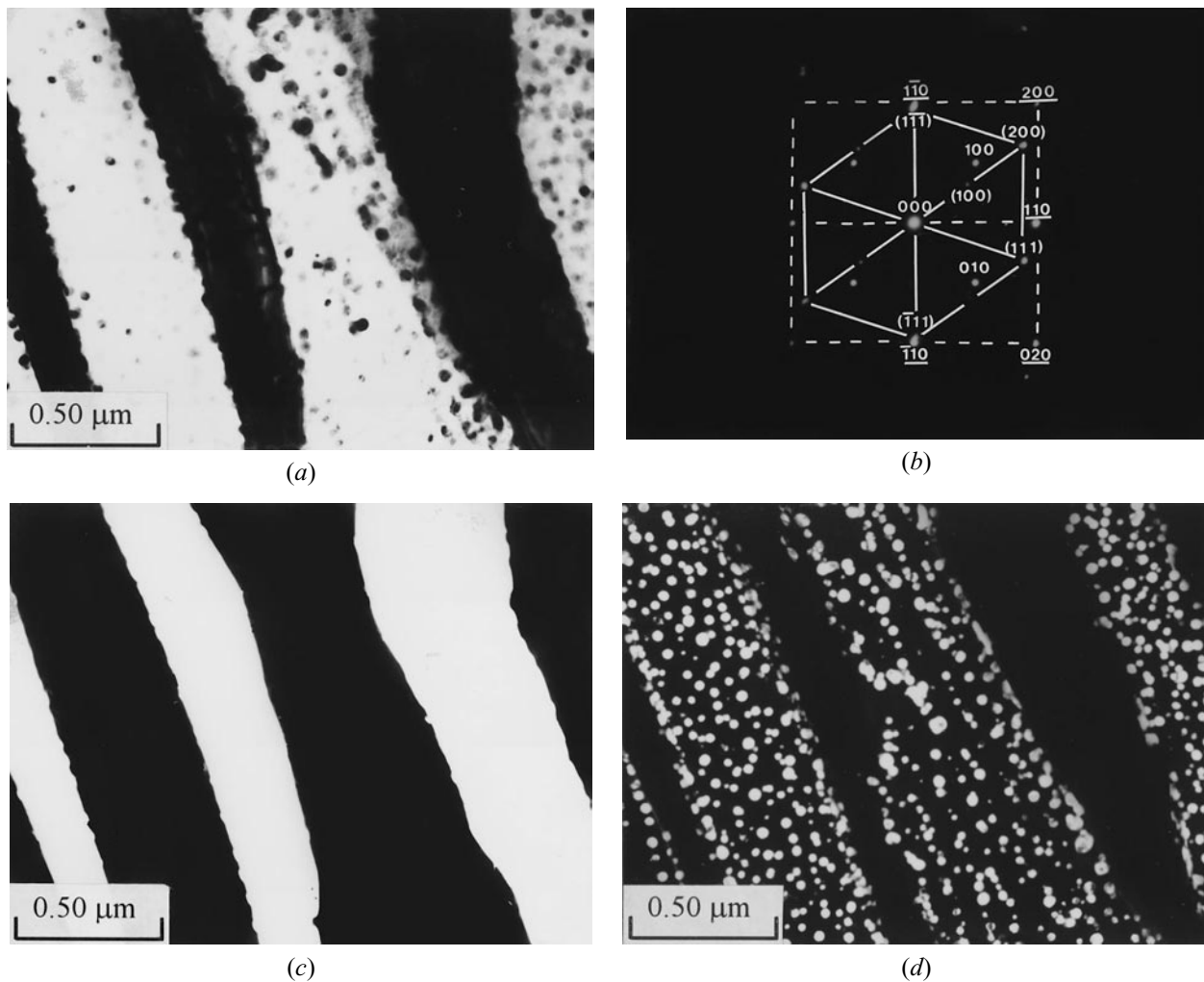


Fig. 4—Electron micrographs of the alloy aged at 823 K for 20 min: (a) BF (b) An SADP taken from an area covering (α + B2) particle and κ -carbide lamella. The foil normals of the α and κ -carbide are $[001]_{\alpha}$ and $[011]_{\kappa}$, respectively ($hkl = \alpha$, $hkl = B2$, and $(hkl) = \kappa$ carbide). (c) and (d) 100 κ -carbide and 100 B2 DF images, respectively.

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Sticking Mechanism during Hot Rolling of Two Stainless Steels

SUNGHAK LEE, DONGWOO SUH, SEUNGCHAN OH, and WON JIN

Sticking refers to the phenomenon occurring in the hot-rolling process in which fragments of a rolled material are detached and get stuck to a work roll surface, deteriorating the surfaces of both the roll and the rolled material.^[1] It

SUNGHAK LEE, Professor, Center for Advanced Aerospace Materials, and WON JIN, Senior Research Engineer, Stainless Steel Research Team, Technical Research Laboratories, are with the Pohang University of Science and Technology, Pohang, 790-784 Korea. DONGWOO SUH, formerly Research Assistant, Center for Advanced Aerospace Materials, Pohang University of Science and Technology, is Postdoctoral Research Associate, Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139-4307. SEUNGCHAN OH, formerly Research Assistant, Center for Advanced Aerospace Materials, Pohang University of Science and Technology, is Research Scientist, Materials Research Department, Kia Technical Center, Seoul, 152-030 Korea.

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