

Morphological evolution of α/γ interface in Fe-Mn-Al alloy

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Series of multiphase diffusion couples were assembled with various terminal α and γ alloys in a Fe-Mn-Al system. These couple series were employed to investigate the development of diffusion structures with planar and non-planar interfaces. Morphology in the diffusion interface was observed by scanning electron microscopy (SEM) and the concentration profile for the constituent element was obtained by electron probe X-ray microanalysis (EPMA) with the aid of quantitative analysis. Transitions from planar interfaces in the α/γ two-phase diffusion couples were indexed by varying the compositions of the terminal alloys. Couples with planar α/γ interfaces exhibited path crossings of the (α/γ) two-phase region parallel to tie-lines. However, the two-phase path crossings for couples developing non-planar interfaces were inclined to tie-lines. The α/γ phase boundary can be determined by the interfacial compositions of the diffusion couple in which the interface is planar.

1. Introduction

A great deal of research interest has focused on the development of potential Fe-Mn-Al-based alloys as a substitute for conventional Fe-Ni-Cr stainless steel in the past year [1-4]. A series of work has been carried out continuously in our research group on the elemental distribution and formation morphology in the oxidation layer [5-9], the effect of gaseous atmospheres and pre-treatment [10], the development of the oxidation-induced ferrite layers [11], the proposed diffusion-related mechanism in the oxidation-induced phase transformation [12], and the associated nitriding kinetics [13, 14]. Most of the previous work on Fe-Mn-Al-based alloy is related to the single γ phase. The purpose of this study is to further investigate the two phase α/γ region, in particular, the morphological evolution associated with the interface movement. For a two-phase equilibrium in a ternary system at constant temperature and pressure, the phase rule predicts the availability of one degree of freedom. This permits the formation of non-planar interfaces or two-phase layers in ternary diffusion couples.

The first systematic investigation of multiphase diffusion with ternary alloys was carried out by Clark and Rhines [15] in an Al-Mg-Zn system at 335 °C. They described the formation of one-phase and two-phase layers in the diffusion zone in terms of schematic diffusion paths drawn between the terminal compositions on the ternary isotherm. Kirkaldy and Fedak [16] investigated diffusion between the α and γ phase in a Cu-Zn-Sn system at 775 °C and observed the formation of a planar and non-planar α/γ interface, as well as isolated and non-isolated precipitates of γ in the matrix. Multiphase diffusion in a Fe-Ni-Cr system was studied by Pilliar and Kirkaldy with α and γ

couples at 1000 °C. Cheng and Dayananda [17] investigated multiphase diffusion in a Fe-Ni-Al system at 1000 °C with selected β and γ alloys. An α/γ two-phase diffusion in Fe/Ni/Cr was further carried out by Duh and Dayananda [18] at 1100 °C. The α/γ interface was observed to change from a planar to a non-planar morphology as one of the terminal compositions was varied. In this study, multiphase diffusion couples were fabricated in a Fe-Al-Mn alloy system. The morphology of the diffusion interface was obtained by SEM and the elemental concentration along the diffusion path was detected with an electron microprobe through quantitative analysis.

2. Experimental procedure

The alloys were prepared from electrolytic iron (99%), electrolytic manganese (99.96%), and high-purity aluminium (99.99%) in a L-HIS 8/III vacuum melting furnace. Iron and aluminium were first loaded at about 1 Pa. Manganese was then charged at the end of the melting process by introducing argon gas into the chamber to reduce the loss of manganese due to

TABLE I Chemical compositions (wt %) of the alloys employed in two-phase diffusion

Sample	Mn	Al	Fe	Phase
B	9.91	-	Balance	γ
C	20.97	-	Balance	γ
D	29.68	-	Balance	γ
E	42.36	-	Balance	γ
F	52.54	-	Balance	γ
H	9.41	9.06	Balance	α
I	21.46	15.18	Balance	α
J	29.69	15.16	Balance	α

TABLE II Couples employed to investigate the α/γ two-phase diffusion structures at 1000 °C

Series	Couple designation	Remarks
I	H/C, H/D, H/E, H/F	Couples with the α terminal alloy H bonded to γ terminal alloys with a gradual change in composition
II	I/B, I/C, I/D, I/E, I/F	Couples with the α terminal alloy I bonded to γ terminal alloys with a gradual change in composition
III	J/B, J/C, J/D, J/E	Couples with the α terminal alloy J bonded to γ terminal alloys with a gradual change in composition

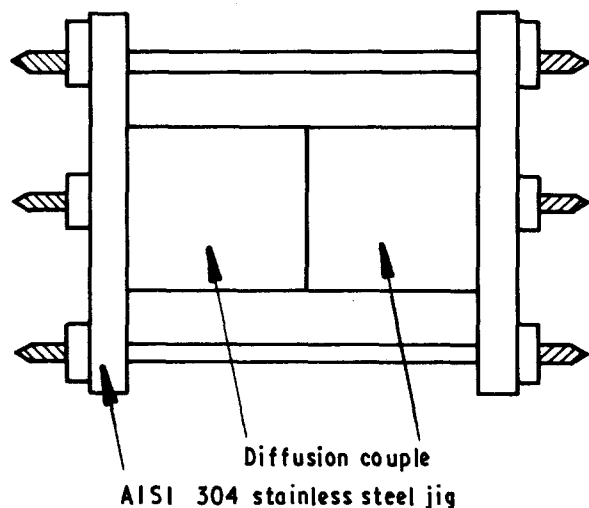


Figure 1 Schematic of the diffusion-couple assembly.

evaporation. Then, after melting, it was cast in a steel mould at about 1600 °C. The compositions of the alloys were obtained by wet chemical analysis and are listed in Table I. The as-fabricated ingots were forged at 1200 °C with 75% reduction and then homogenized at 1200 °C for 4 h. After surface finishing, the alloys

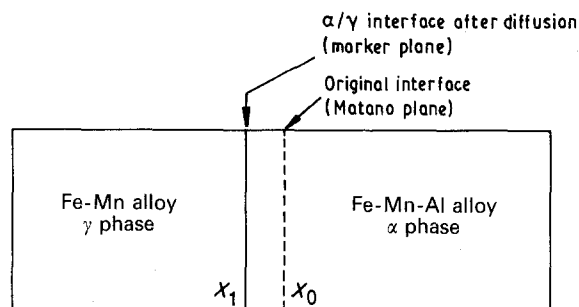


Figure 2 Schematic of a α/γ two-phase diffusion zone.

were cut into pieces with dimensions 9 × 9 × 5 mm. The specimen was then abraded up to 1200 grit SiC paper and polished up to 0.05 μm aluminium-oxide powder.

The sample holder for the diffusion assembly was made of AISI 304 stainless steel. Fig. 1 shows a schematic diagram of the sample holder. The discs at either end were abraded up to 1200 grit SiC paper to have a smooth surface. The supporting screw was made from a 3 mm wire and mechanically threaded. Prior to bonding, a thin layer of 0.05 μm aluminium-

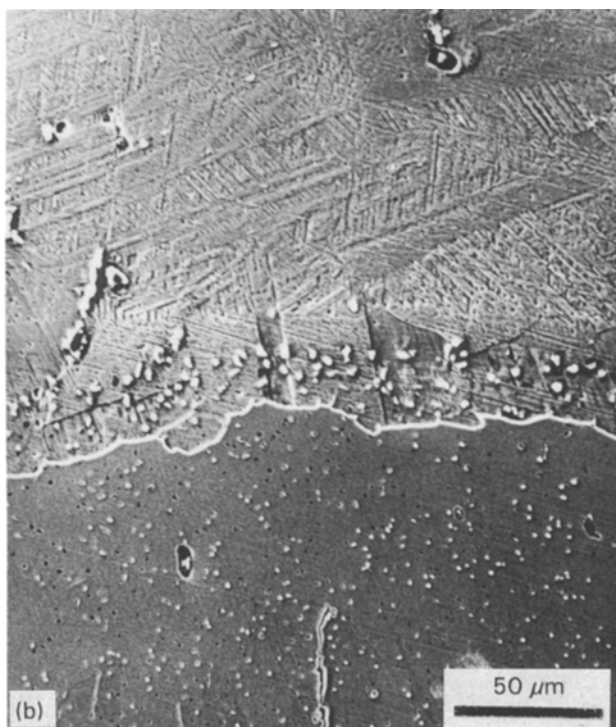


Figure 3 Interface morphologies for the couples in series I, diffused at 1000 °C for 158 h: (a) H/C, (b) H/D, (c) H/E, and (d) H/F.

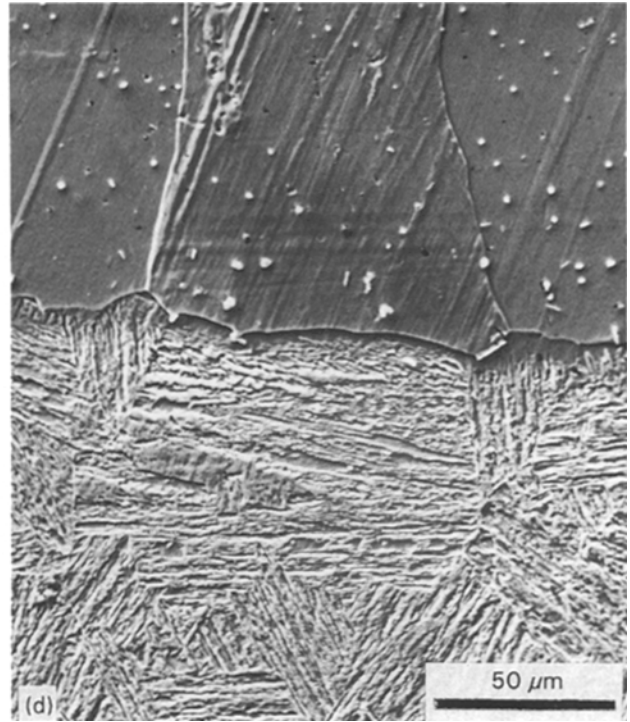
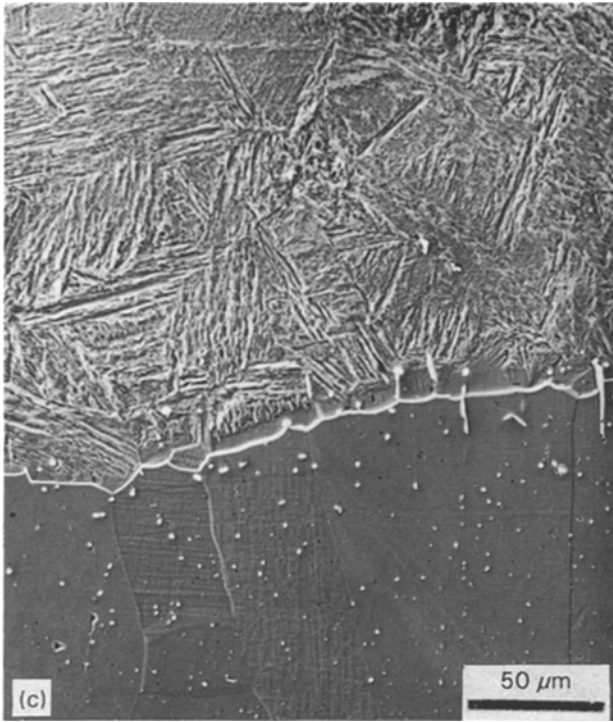


Fig. 3. Continued

oxide powder was deposited onto the interface by dipping as the marker materials. The diffusion couple was put into a specially designed stainless steel (AISI 304) tube, which was purged with argon and out-gassed consecutively three times. The diffusion experiment was carried out at 1000 °C for 157 h in a 250 kPa argon atmosphere. The constant temperature zone with a variation ± 1 °C in the furnace is about 8 cm.

After this diffusion experiment, the couple was water quenched to freeze the high-temperature state. The couple was cold mounted, cut with a low-speed diamond saw and polished through 0.05 μm aluminium-oxide powder.

The morphology of the diffusion interface was examined by scanning electron microscopy (SEM). Quantitative analysis of elemental redistribution in

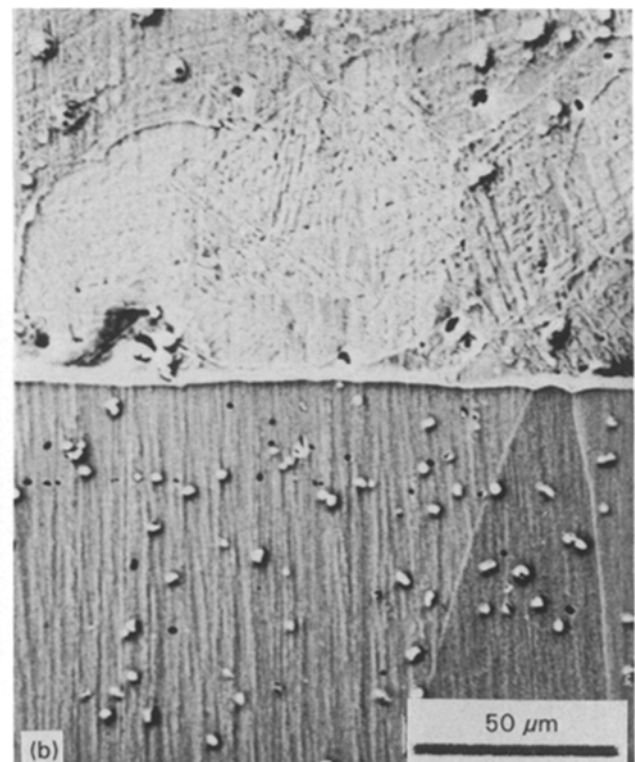
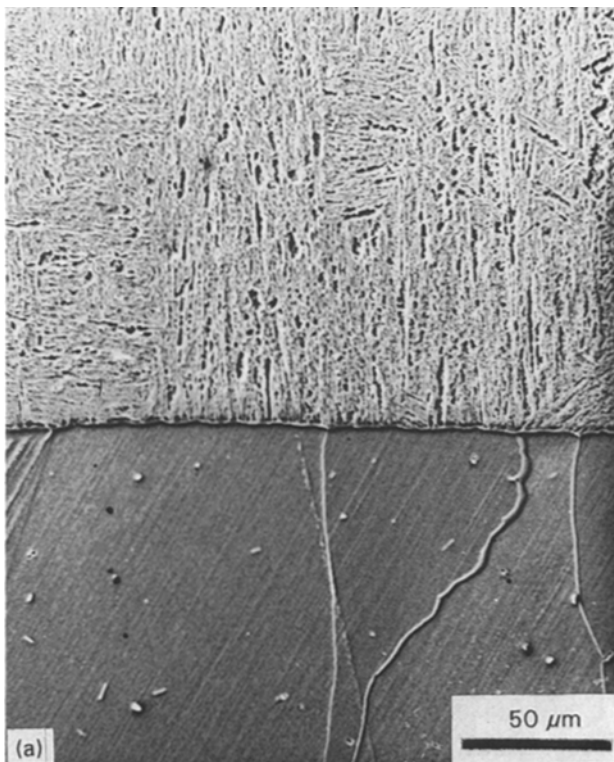


Figure 4 Interface morphologies for the couples in series II, diffused at 1000 °C for 158 h: (a) I/B, (b) I/C, (c) I/D, (d) I/E, and (e) I/F.

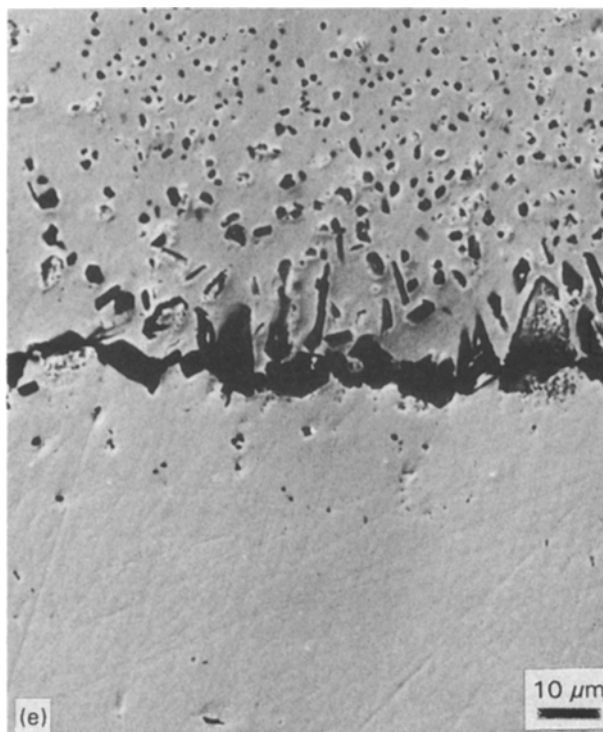
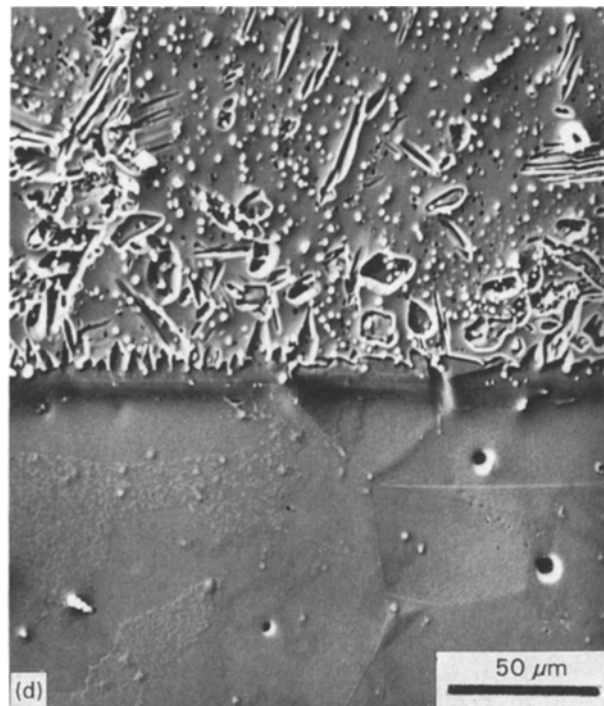
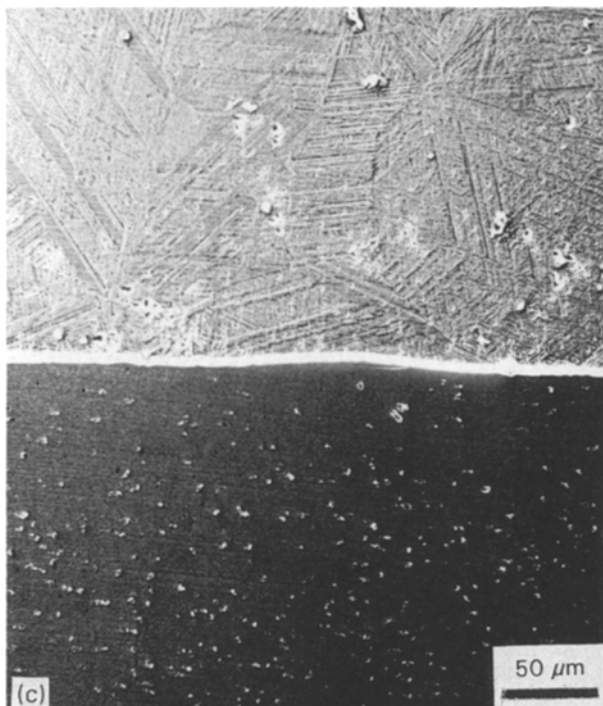


Fig. 4. Continued

the diffusion couple was detected with the aid of an electron microprobe (JEOL JCXA-733).

3. Results

Three series of diffusion couples were assembled with various terminal α and γ alloys and are presented in Table II. These couple series were used to investigate the development of diffusion structures with planar and non-planar interfaces. Each series of experiment was characterized by couples with one common α terminal alloy bonded to several γ alloys with a gradual change in the compositions. Such series of

couples would help index transitions from planar to non-planar interface development that might occur with changes in one of the terminal alloy compositions.

Fig. 2 shows a schematic of the two-phase diffusion zone. The original interface moves toward the γ phase when the couple is annealed at 1000 °C for 157 h. This implies that alloy elements in the α phase diffuse faster than those in the γ phase. Fig. 3 shows SEM micrographs of diffusion structures for the couple series I characterized by four couples with the common terminal alloy H. The couple H/C exhibited a planar α/γ interface, while the couples H/D, H/E and H/F developed non-planar α/γ interfaces. Diffusion structures for the couple series II characterized by couples with the common terminal alloy I are shown in Fig. 4. The couples I/B, I/C and I/D showed planar α/γ interfaces. However, the couples I/E and I/F showed non-planar α/γ interfaces. Diffusion structures for the couple series III consisting of couples with the common terminal alloy J are presented in Fig. 5. Only the couple J/B exhibited a non-planar interface, while the others, J/C, J/D and J/E developed planar interfaces. A summary of the interface morphologies of these three couple series and composition of the phases at the α/γ interface are listed in Table III. The interfacial compositions are only reported for those couples exhibiting a planar interface, since the interfacial compositions associated with a non-planar interface are not unique and depend on the choice of the trace line [17, 18].

A typical concentration profile of the couple I/C with planar interfaces is shown in Fig. 6. The diffusion paths for all the couples in series I, II and III are presented on the Fe–Mn–Al isotherm in Figs 7–9, respectively. The thick dashed lines within the ($\alpha + \gamma$)

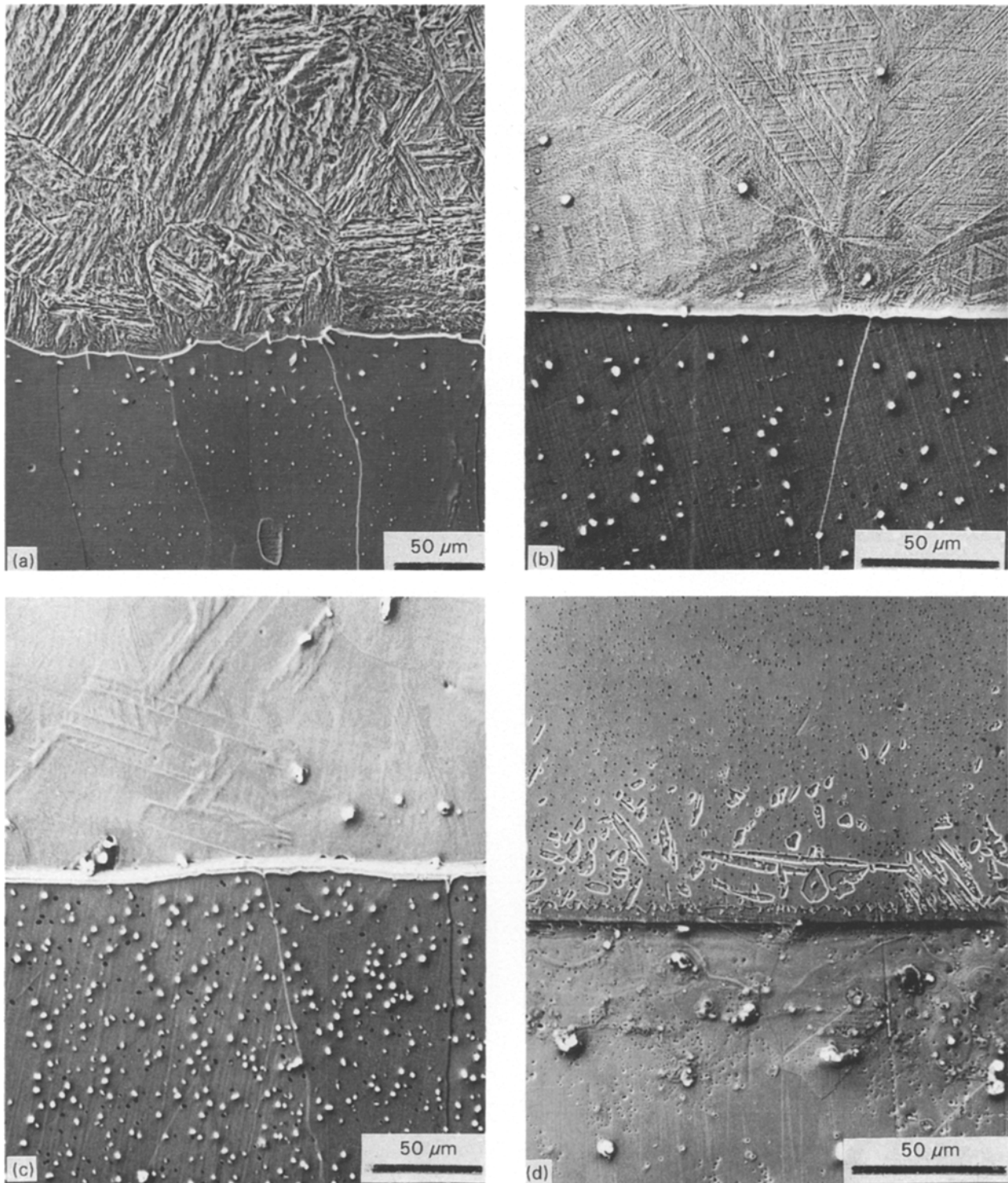


Figure 5 Interface morphologies for the couples in series III, diffused at 1000 °C for 158 h: (a) J/B, (b) J/C, (c) J/D, and (d) J/E.

region of the isotherm correspond to the equilibrium tie-lines.

4. Discussion

4.1. The movement of the α/γ interface

The schematic diagram of the diffusion zone shown in Fig. 2 indicates that the α/γ interface of the diffusion couple moves toward the γ phase after annealing. This implies that the Matano plane moves towards the α phase after diffusion. The concentration profile of couple I/C in Fig. 6 shows that the α/γ interface is located at $X_1 = 102 \mu\text{m}$ after diffusion, while the Matano plane is at $X_0 = 125 \mu\text{m}$. This demonstrates

that the diffusivity of the element in the α phase is larger than that in γ phase. Several studies on the diffusivity of Mn in various Fe–Mn–Si-based alloys [19] were reported. Only one study concerning the diffusivity of Mn in Fe–8.9 Al–3 Cr–31 Mn–0.87 C is available in the literature, in which the value is $1.5\text{--}1.7 \times 10^{-13} \text{ m}^2 \text{ s}^{-1}$ at 1000 °C in the transformed α region [12]. The diffusivity of manganese calculated by Nohara [20, 21] were 3.49×10^{-16} and $3.39 \times 10^{-14} \text{ m}^2 \text{ s}^{-1}$ for $\gamma\text{-Fe}$ and $\alpha\text{-Fe}$, respectively, at 1000 °C. It appears that the diffusivity of manganese in the ferritic iron is approximately two orders of magnitude larger than that in the austenitic iron. Thus, the α

TABLE III Interface morphologies and compositions at the interface for two-phase α/γ diffusion couples at 1000 °C

Couple series	Couple designation	Interface morphologies	Compositions at α/γ interfaces (at %)					
			α Fe	Mn	Al	γ Fe	Mn	Al
I	H/C	Planar	79.72	14.97	5.31	79.87	17.08	3.05
	H/D	Non-planar	—	—	—	—	—	—
	H/E	Non-planar	—	—	—	—	—	—
	H/F	Non-planar	—	—	—	—	—	—
II	I/B	Planar	84.67	11.19	4.13	84.68	13.04	2.28
	I/C	Planar	73.92	19.21	6.87	73.87	22.34	3.79
	I/D	Planar	68.84	22.65	8.51	68.50	25.79	5.71
	I/E	Non-planar	—	—	—	—	—	—
	I/F	Non-planar	—	—	—	—	—	—
III	J/B	Non-planar	—	—	—	—	—	—
	J/C	Planar	66.00	25.97	8.03	66.22	28.91	4.87
	J/D	Planar	60.92	28.75	10.33	60.70	32.81	6.49
	J/E	Planar	56.61	32.02	11.37	55.02	37.58	7.40

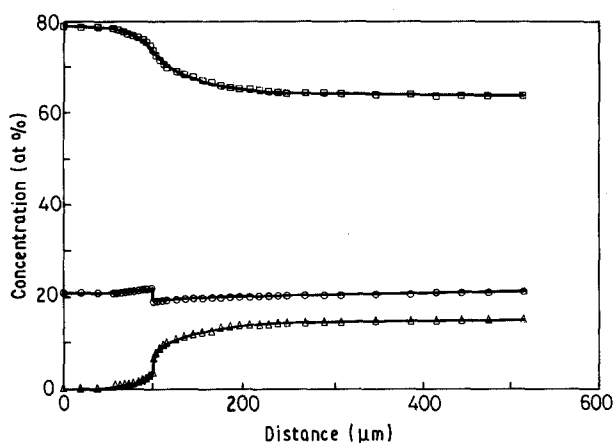


Figure 6 Concentration profiles for the couple I/C diffused at 1000 °C for 157 h: (□) Fe, (○) Mn, and (△) Al.

phase grows at the expense of the γ phase and the α/γ interface moves toward the γ side after diffusional annealing.

It should be pointed out that when the difference of terminal alloy composition of a diffusion couple is

large, voids can be produced near the diffusion zone due to the Kirkendall effect [22]. Fig. 4e shows a great deal of pores exist around the α/γ interface. This kind of couple is discarded in the quantitative analysis since it is difficult to draw a trace line without porosity in the microprobe measurement.

4.2. α/γ two-phase couples

A diffusion path that passes through a two-phase region on the isotherm across tie-lines is considered to represent the formation of a two-phase layer in the diffusion zone, while a planar interface indicates a diffusion path is parallel to a tie-line within the two-phase region [15–18].

The α/γ two-phase couples listed in Table III clearly indicated that the α/γ interfaces can be either planar or non-planar dependent on the terminal alloy composition. Couples in the series I and II show a transition from planar to non-planar morphology, as the compositions of the γ terminal alloy are varied toward the Mn-rich side of isotherm. Couples in the series III, however, show a transition from non-planar to planar

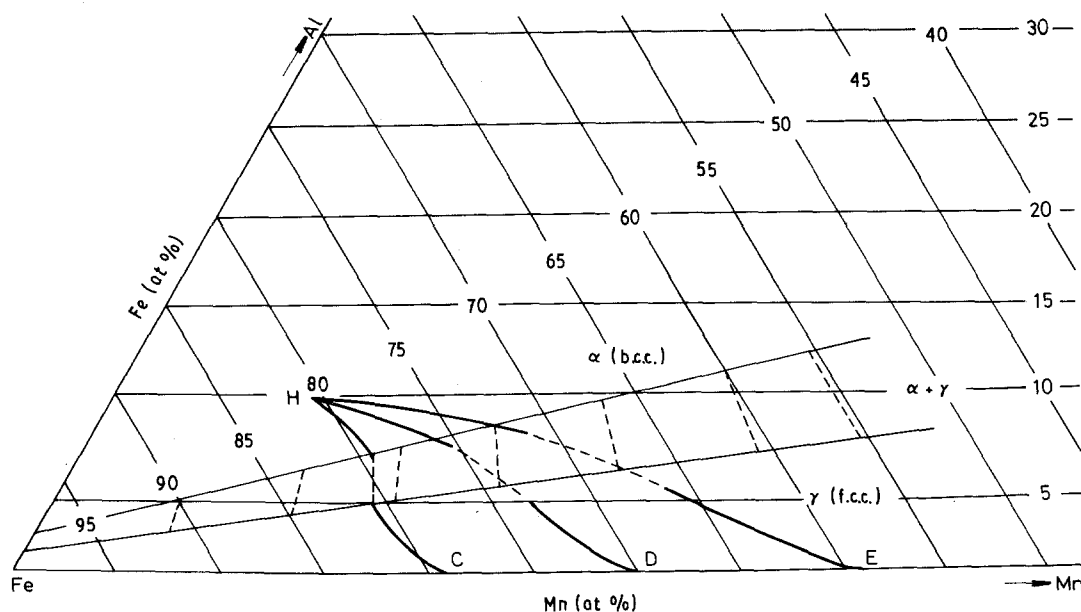


Figure 7 Experimental diffusion paths for the α/γ couples in series I at 1000 °C.

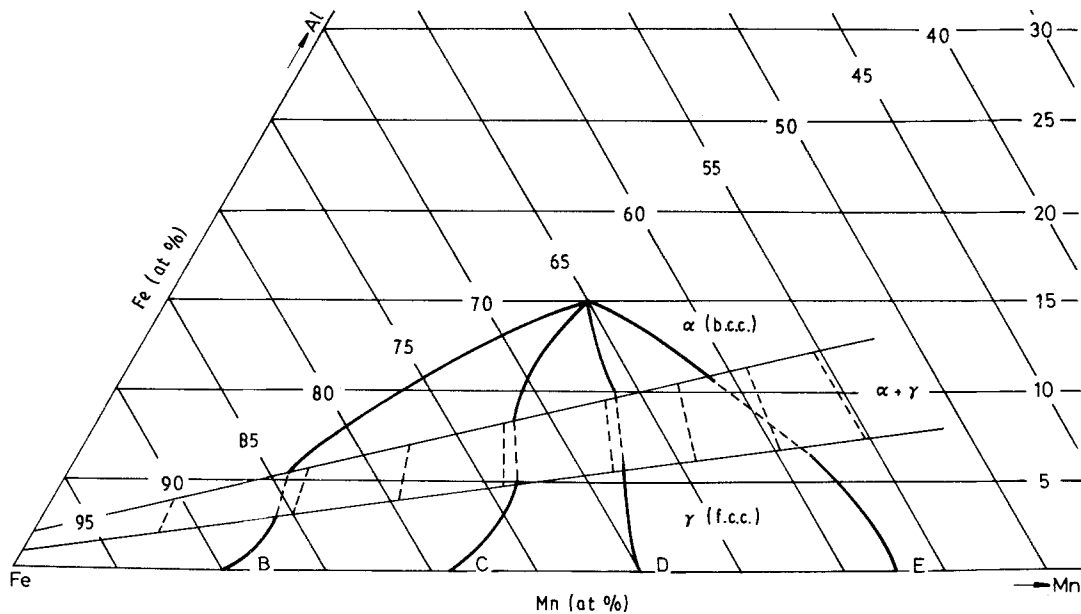


Figure 8 Experimental diffusion paths for the α/γ couples in series II at 1000°C.

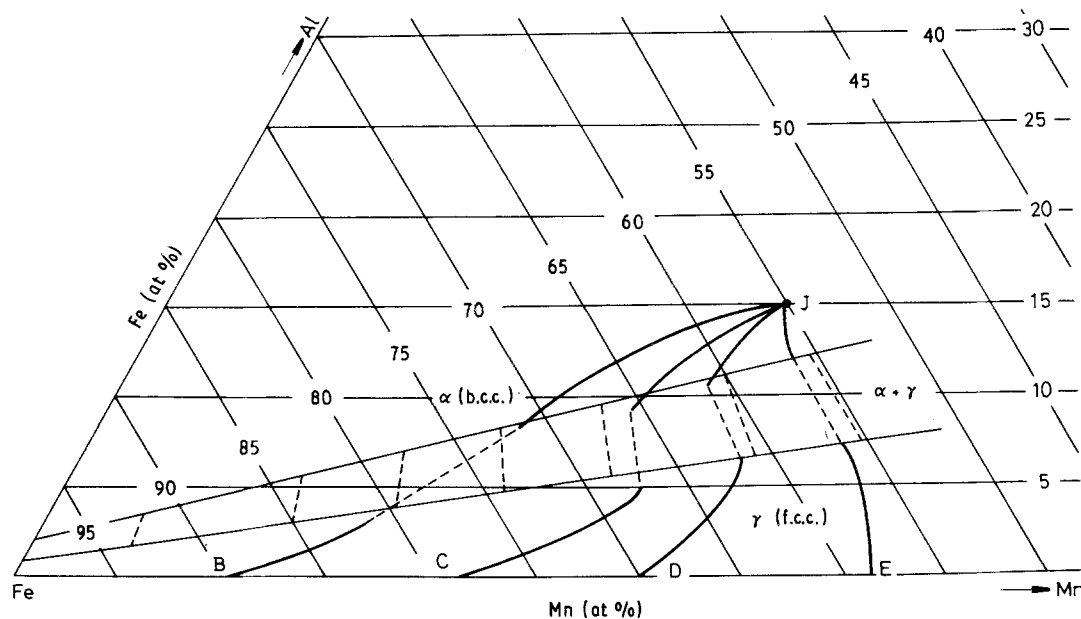


Figure 9 Experimental diffusion paths for the α/γ couples in series III at 1000°C.

morphology, as the compositions of the γ terminal alloy are varied toward the Mn-rich side of isotherm. This implies that the development of both planar and non-planar α/γ interfaces is strongly dependent on the terminal alloy of the multiphase assembly. Such series of couples, characterized by couples with a common α terminal alloy and a gradual change in the composition of the other γ terminal alloy, would help index the development of transitions from planar to non-planar interfaces. As shown in Figs 7–9, the diffusion paths for the couples developing planar interfaces pass through the α/γ two-phase region coincident with the tie-lines, and local equilibrium is specified by those tie-lines. For the non-planar morphology, deviation from equilibrium composition can be expected on either side of the unstable interface, and the corresponding path crossing is inclined to the tie-lines.

Compositions of the phase at the planar α/γ interface listed in Table III are represented on the ternary

isotherm in Fig. 10. The composition of these planar interfaces can be utilized to construct the phase boundary line and provide useful information concerning the α/γ phase boundary in the Fe–Mn–Al system at 1000°C.

5. Conclusions

1. Three series of diffusion couples in a Fe–Mn–Al system were assembled with various α and γ alloys. Each series of experiment was characterized by couples with one common α terminal alloy bonded to several γ alloys with a gradual change in the composition.

2. Couples in the series I and II show a transition from planar to non-planar morphology, as the compositions of the γ terminal alloys are varied toward the Mn-rich side of the isotherm. However, couples in the series III show a transition from non-planar to planar

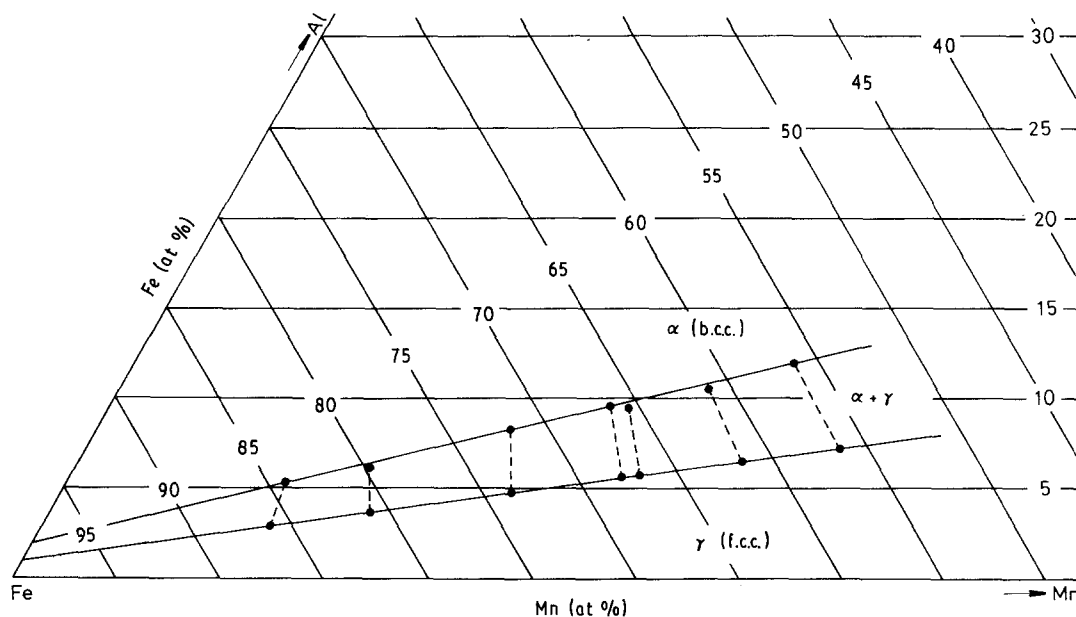


Figure 10 Determination of the α/γ phase boundary in a Fe-Mn-Al system at 1000°C.

morphology, if the compositions of the γ terminal alloy change toward the Mn-rich side of the isotherm.

3. Series of couples characterized by couples with a common α terminal alloy and a gradual change in the composition of the other γ terminal alloy would help index transition from planar to nonplanar interface development.

4. The α/γ phase boundary can be determined by the interfacial composition for a diffusion couple in which the interface is planar.

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