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Precipitation on growth ledges of planar, low energy interphase boundaries in Fe–C–X alloys

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This paper is concerned with a detailed investigation into the formation of particulate precipitates on the mobile growth ledges of low-energy austenite–ferrite boundaries in an Fe–10%Cr–0.2%C alloy. It is shown that, despite their high mobility, such ledges can act as nucleation sites for precipitation if the local conditions at the interface are satisfactory. The application of these results to other iron-based alloys is briefly discussed.

1. Introduction

Precipitation at advancing austenite/ferrite interphase boundaries in both steels and carbon free iron-based alloys is an important process both scientifically and commercially [1]. This form of precipitation has been termed "interphase precipitation" and its characteristics have been studied in a wide range of iron-based alloys [2–6]. One aspect of this form of precipitation, which has received detailed attention, is the often observed sheet-like morphology of the precipitate dispersion, the sheets of precipitates lying parallel to the advancing interphase boundary [4]. Since these precipitate sheets have been observed to be closely associated with stepped austenite/ferrite boundaries, it was concluded that the planar sheets of precipitates were formed on low energy facets which require a ledge mechanism [7] to migrate [8]. Campbell and Honeycombe [9] have studied this type of precipitation in a chromium steel and shown that the precipitates nucleated on the low energy facets, and not on the migrating ledge since the latter exhibits high mobility. Similarly Aaronson *et al.* [10] have shown that the high mobility of disordered boundaries in Fe–C alloys makes then unsuitable nucleation sites for interphase precipitation under most transformation conditions.

However, the results of a number of recent studies [6, 11] have shown that particulate precipitates can form on migrating disordered interphase boundaries. In this paper it is shown that precipitation at the ledges, necessary for low-energy boundary migration, is possible, providing the conditions at the interface during the phase transformation are suitable. The results presented relate to an Fe–10%Cr–0.2%C alloy (precipitate phase: $M_{23}C_6$).

2. Experimental details

The alloy used in this investigation had the following nominal composition (wt %) Fe–10/Cr–0.2%C. Specimen material in the form of a 3 mm diameter rod was solution treated at 1150° C for 0.5 h and isothermally transformed in the temperature range 700 to 650° C in a molten tin bath. The respective transformation temperatures (T) and isothermal times (t) are indicated in the figure captions. Thin foil specimens for electron microscopy were prepared in a commercial twin-jet electropolisher using an electrolyte consisting of 10% perchloric acid, 20% glycerol and 70% ethanol. All transmission electron microscopy was performed on a Phillips EM400 operating at 120 kV.

3. Results

A typical example of precipitation at non-planar, mobile interphase boundaries in this alloy is shown in Fig. 1. The exact details concerning the crystallography and mechanism of formation of this type of precipitate dispersion may be found elsewhere [11]. It is sufficient to state here that, in general, this "incoherent interphase precipitation" [6] obeys a unique orientation relationship variant

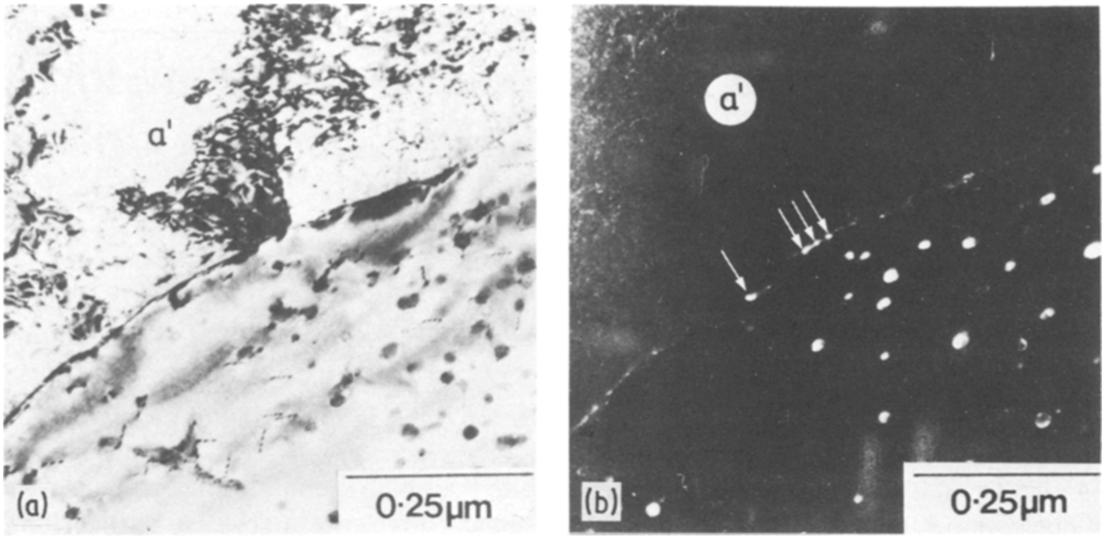

Figure 1 An example of precipitation at a non-planar austenite-ferrite interphase boundary in the Fe–10%Cr–0.2%C alloy: (a) bright field electron micrograph showing a dispersion of $M_{23}C_6$ precipitates in the ferrite (bottom of micrograph); (b) corresponding $M_{23}C_6$ centred dark field electron micrograph. Note the fine precipitation observed on the interface (arrowed). ($T = 655°$ C: $t = 30$ min).

with the ferrite and delineates the prior positions of the disordered interphase boundary in much the same way as planar sheets of precipitates which form on stepped low-energy boundaries.

Fig. 2 shows a bright field micrograph of a partially transformed specimen. The morphology of the austenite/ferrite interface is seen to be complex, containing a "double ended" ledge (A–B). Planar sheets of interphase precipitation can also be seen in the ferrite (arrowed) and also in association with the growth of the ledge AB. The corresponding $M_{23}C_6$ dark field electron micrograph of this area is shown in Fig. 3 (at a lower magnification), using the $006_{M_{23}C_6}$ reflection. An interlath film of retained austenite is also imaged in the martensitic region (arrowed) indicating that the 002 is almost coincident with the $006_{M_{23}C_6}$ reflection.* Full crystallographic analysis of this interfacial region yielded the following three phase crystallography

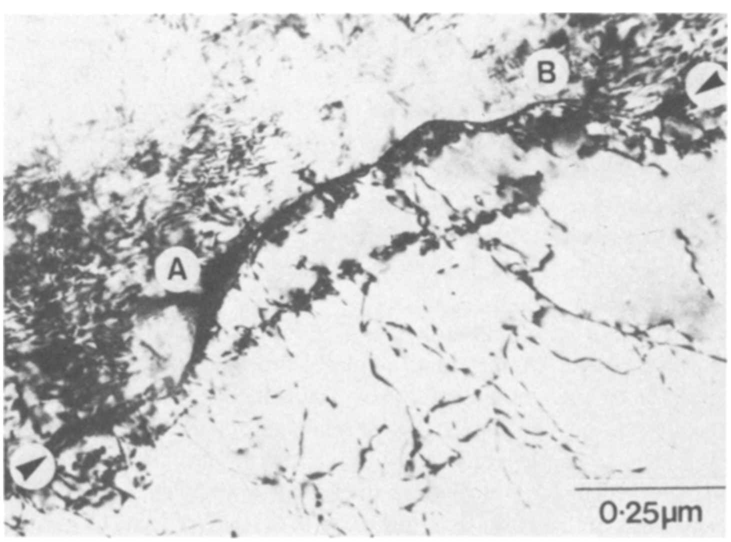

Figure 2 Bright field micrograph of an austenite–ferrite interface, the growth of which has produced planar sheets of $M_{23}C_6$ precipitates (arrowed). The interface itself contains a "double ended" growth ledge A–B ($T = 655°$ C: $t = 30$ min).

*The austenite and $M_{23}C_6$ lattice parameters differ by a factor which is very close to three.

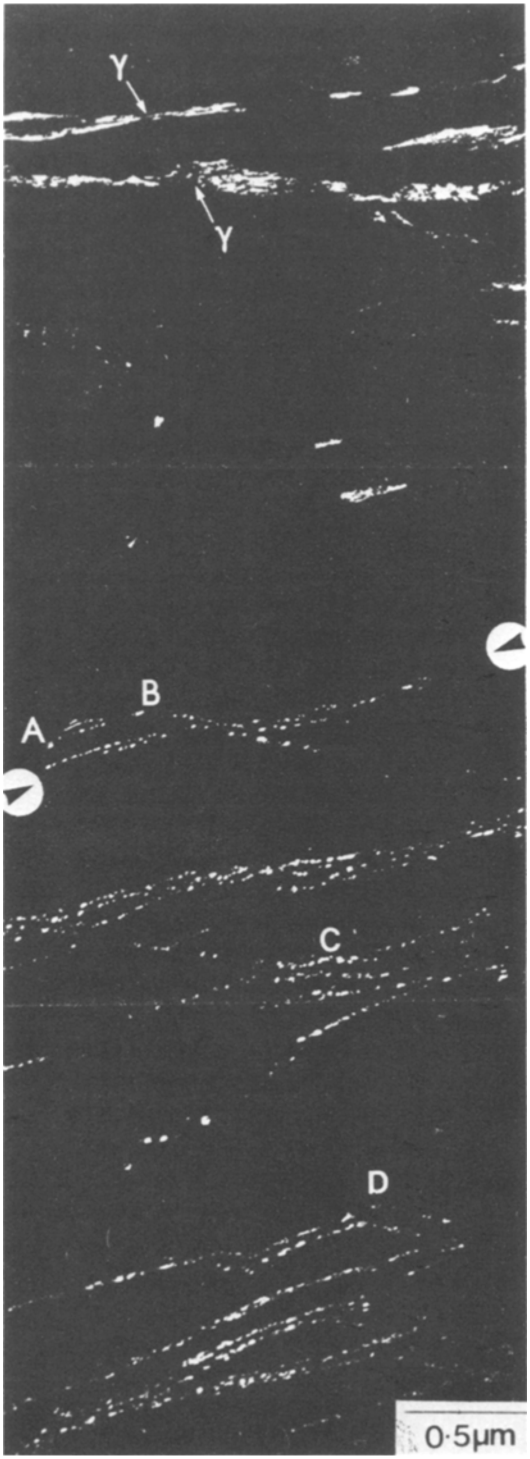

Figure 3 A combined $\langle 0\,0\,2\rangle_\gamma/\langle 0\,0\,6\rangle_{M_{23}C_6}$ centred dark-field micrograph (at a lower magnification) of the area shown in Fig. 2. The interphase boundary is shown arrowed, and evidence for precipication on both risers of the ledge is observed (A–B). Further evidence for the precipitation of $M_{23}C_6$ on the migrating ledges is shown for example at C and D.

$$(1\,1\,1)_\gamma \,\|\, (1\,1\,0)_\alpha \,\|\, (1\,1\,1)_{M_{23}C_6}$$
$$\{\bar{1}\,1\,0\}_\gamma \,\|\, \{\bar{1}\,1\,1\}_\alpha \,\|\, \{\bar{1}\,1\,0\}_{M_{23}C_6}$$

(and see Howell *et al.* [12]).

Close examination of the interface illustrated in Fig. 2 shows evidence for precipitation on the ledge A–B shown enlarged in Fig. 4. It should be noted that this particular precipitate is considerably larger than those situated in the interphase sheets. Furthermore the trace of the ledges shown in Fig. 2a are approximately parallel to the "kinks" which occur frequntly in the planar precipitate sheets, shown in the precipitate-centred dark field micrograph in Fig. 3 (e.g. at C and D). This suggests that precipitation on the migrating ledges has occurred frequently during the formation of this precipitate colony. That the traces of the "kinks" in the precipitate sheets, and the ledges on the interface are approximately parallel in this region could suggest that the ledges are crystallographically specific with the local interface normal conforming to a low energy orientation.

Examination of other interphase precipitate colonies provides further supportive evidence for precipitation occurring on the migrating ledges. Fig. 5 shows a bright field micrograph of a stepped austenite/ferrite interphase boundary (the steps being arrowed) which is producing an approximately planar series of sheets of precipitates. Examination of these precipitate sheets reveals regions such as at A and B where the abrupt change of sheet habit strongly supports the hypothesis that these precipitates were formed on the ledges.

4. Discussion

The results detailed in the previous section have confirmed the earlier investigations [6, 11] concerning the formation of discrete precipitates on disordered boundaries. In particular the results of this study strongly suggest that precipitation may occur on the disordered growth ledges of partially coherent interphase boundaries. These growth ledges are widely accepted [8, 13] to be necessary for partially coherent boundary migration and there is now considerable evidence for the formation of interphase precipitation on the immobile facets of such boundaries. Previous work [9] concluded that, although the disordered growth ledges were energetically the more favoured nucleation sites, there high mobility prohibited nucleation occurring. This hypothesis seems to be generally

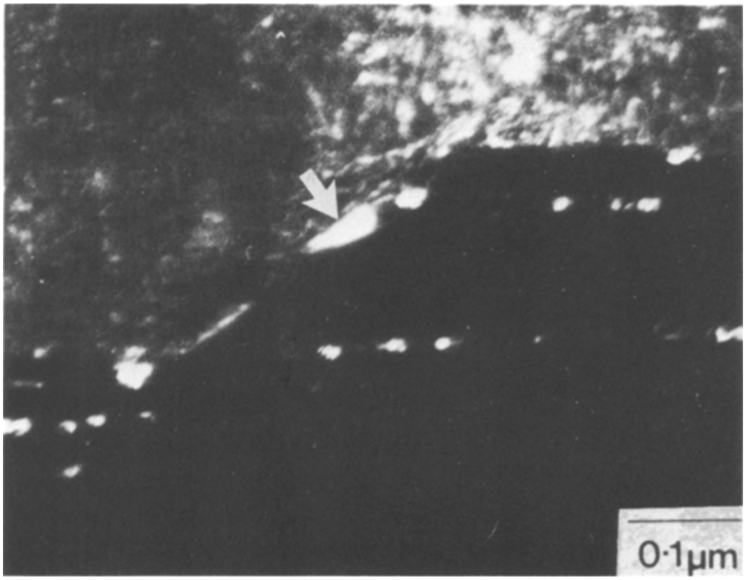

Figure 4 An enlargement of the austenite–ferrite interface shown in Figs. 2 and 3. A large $M_{23}C_6$ precipitate is observed on the growth ledge (arrowed).

obeyed, since precipitation on the immobile facets to produce regularly spaced sheets is the dominant reaction in the majority of alloy systems. However, the addition of large amounts of alloying elements, such as in the alloy used in this investigation, will both reduce the mobility of the growth ledges and increase the solute concentration at the interface. These conditions will favour the formation of precipitates on the ledges which will be effectively prevented from further movement. The sequence of events following this pinning would presumably require the passage of subsequent ledges to overtake and unpin the ledge containing the precipitate.

The direct microscopical observation of a ledge pinned by precipitation, shown in Fig. 2, clearly reveals that the actual pinning precipitate is larger than those contained in the interphase precipitation colony. This correlates well with the hypothesis of Ricks *et al.* [6] concerning the diffusion path of the substitutional solute atoms in the interphase boundary. Thus the ledge would

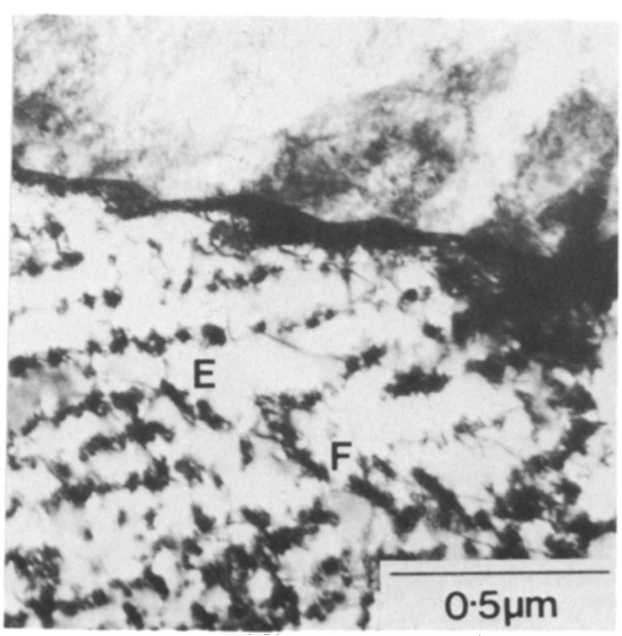

Figure 5 Bright field micrograph of a stepped austenite–ferrite interphase boundary, the growth of which has produced a series of planar sheets of $M_{23}C_6$ precipitates. Abrupt changes in the precipitate sheet habit (at E and F) are indicative of previous precipitation events on the growth ledges ($T = 700°$ C: $t = 10$ min).

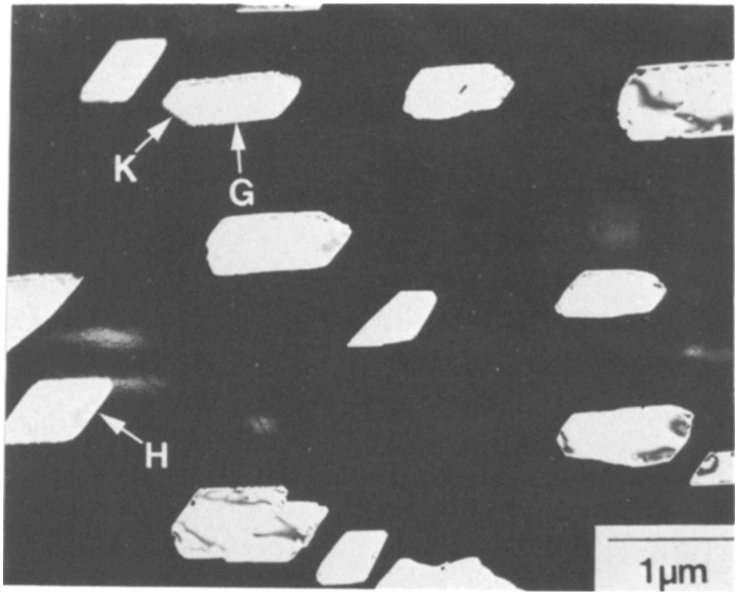

Figure 6 An austenite centred dark-field micrograph from an aged Fe–26%Cr–6%Ni alloy. In addition to the $(1\,1\,1)_\gamma/(1\,1\,0)_\alpha$ interfacial habits which are observed to develop (e.g. at G) other planar facets have also developed (e.g. at H and K).

acquire a large concentration of chromium atoms as it migrates through the crystal and any stable nucleus formed on the ledge would grow rapidly.

It was noted in Section 3 that the habit plane of the ledges on the interphase boundary were approximately parallel to the "kinks" in the precipitate sheets. It was suggested that these "kinks" were formed by previous precipitation events on ledges. If this were the case it implies that the growth ledges can adopt another boundary habit plane, presumably of relatively low energy, when the boundary is forced away from the $\{1\,1\,1\}_\gamma/\{1\,1\,0\}_\alpha$ plane. These observations imply the existence of at least one other low energy boundary configuration when there is a K–S orientation relationship between the austenite and ferrite.

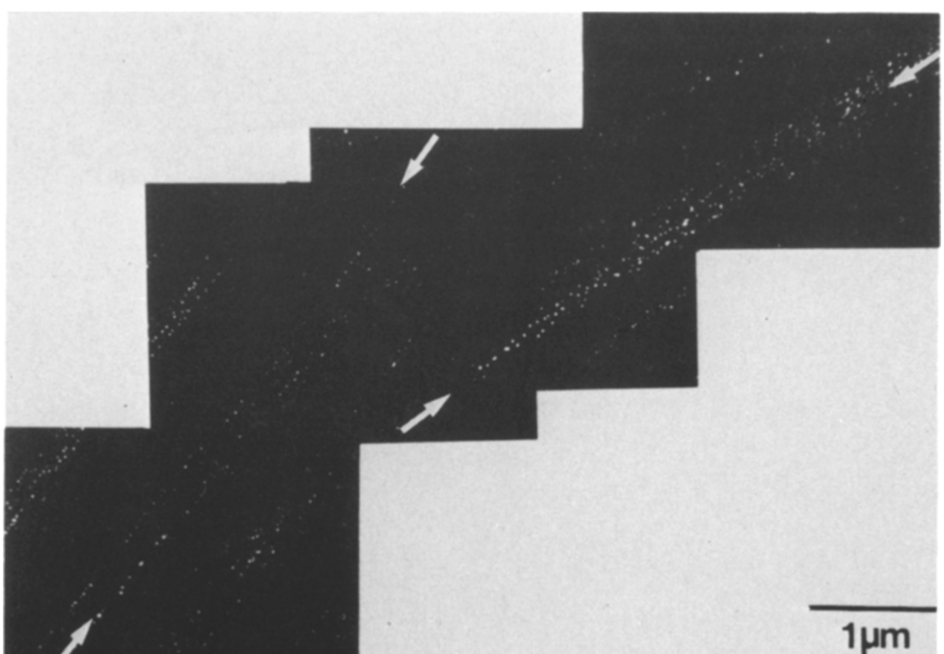

Figure 7 An ϵ-copper centred dark-field micrograph for an isothermally transformed Fe–2%Cu–5%Ni alloy. Two distinct planar colonies of interphase precipitates sheets are observed (arrowed) ($T = 610°$ C: $t = 20$ min).

This hypothesis that K–S related austenite and ferrite can form planar boundary facets on more than one habit plane is supported by the following observations. Fig. 6 shows a series of austenite precipitates in a duplex stainless steel, and in addition to the low-energy $(1\,1\,1)_\alpha/(1\,1\,0)_\alpha$ facets, other planar facets are clearly observed. Similarly the planar sheets of ϵ-Cu interphase precipitation shown in Fig. 7, which delineated the prior austenite/ferrite boundary positions, can be clearly seen to have two distinctly different habit planes.*

Finally, it is interesting to compare the behaviour of this particular alloy system, where the three-phase crystallography mentioned in Section 3 can be obeyed, with the precipitation of NaCl-type carbides in low alloy steels. These carbides generally adopt a Baker–Nutting [15] orientation relationship with the ferrite and therefore a three-phase crystallography is not possible. Despite this a unique orientation relationship is frequently observed within a precipitate colony between the carbides and the ferrite. This implies that the free energy of activation for nucleation is reduced by minimizing the angle between the $\{0\,0\,1\}_{\mathrm{NaCl\ carbide}}$ facets of the precipitates and the interphase boundary. Further work is in process to investigate the exact details of such interphase boundary precipitation.

5. Summary

This paper has shown that in Fe–C–X alloys precipitation can occur on mobile interphase boundaries. In particular, precipitation on the mobile sections of low-energy boundaries which migrate by a ledge mechanism, is possible if the overall ferrite reaction kinetics are sufficiently slow.

Acknowledgements

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*A recent theoretical study [14] concerning the energy of γ/α phase boundaries has demonstrated the existence of more than one low-energy interfacial orientation.