

Precipitation of Carbides in Low-Carbon Fe-Al-C Alloys

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High-purity Fe-C alloys containing up to 5.8 pct Al were quenched from 730°C, then aged at temperatures up to 400°C. Precipitation of a carbide transition phase (presumably ϵ) and of cementite were followed by transmission electron microscopy, coercive force measurements, and changes in yield strength. The presence of aluminum increases the temperature at which cementite begins to precipitate during aging. This effect is similar to that produced by silicon in ferrite, and opposite to that produced by manganese. It is suggested that the effects of Si, Mn, and Al on cementite precipitation from ferrite arise from the effects of these elements on the stability of cementite, as reflected in the equilibrium partition of carbon between cementite and ferrite.

THE effect of substitutional solutes on the precipitation of carbon from α -iron has long been a subject of considerable interest. Despite a number of previous studies¹⁻¹¹ on the effects of various alloying elements we can predict in only a very general way how each element will modify the rate of precipitation, the type of precipitate, and the nucleation sites. As discussed previously,² an alloying element having a higher solubility in the carbide than in ferrite is not likely to have a large effect on the rate of growth of the carbide, although it may govern the structure of the carbide precipitated, the rate of nucleation and the nucleation sites. On the other hand, if the alloying element is essentially insoluble in the carbide, it can have a very large effect on the rate of growth of the carbide. Beyond this, it is still necessary to determine experimentally the effects of each alloying element on the morphology, structure, and rate of growth of carbide particles. Herein we describe the results of a study of the effect of aluminum on the precipitation of carbon from α -iron.

Although diffusion of carbon and the rate of precipitation of carbon in Fe-Al-C alloys have been studied by internal friction techniques^{5-9,12} and by magnetic after-effects,¹³ it appears that there has been no systematic study of the effect of aluminum on the precipitates themselves. To remedy this lack, we used transmission electron microscopy, supplemented by measurements of coercive force and yield strength, to determine the type of carbide precipitated, its rate of growth, and how both type and growth rate are modified by aluminum content.

EXPERIMENTAL PROCEDURES

The compositions of the alloys are listed in Table I. The impurity content given for "Base" is that of the Plastiron A104 electrolytic iron base. The alloys were vacuum melted and cast, hot rolled, cold rolled, heated 4 h at 730°C to dissolve carbides, then quenched

into iced brine containing 10 pct NaCl and 1 pct NaOH. All specimens had a virtually identical grain size of about ASTM No. 6. There was no evidence of undissolved carbides or precipitation after the solution treatment except in Alloy 5, in which not all the carbides were dissolved. Specimens were stored in liquid nitrogen after quenching and after aging, which was done isothermally at 100 to 400°C.

Foils for transmission electron microscopy were made from the same specimens used for coercive force measurements, to ensure that identical structures were present in both instances. The coercive force measurements were made as before.¹⁰ They were used as one measure of the rate of aging and to relate our data to the internal friction study by Jaeniche, *et al.*,⁸ in which coercive force changes were measured only after aging at 250°C. The coercive force of each specimen was measured before and after aging at 100, 150, 200, or 250°C.

Tensile test specimens were cut from the cold rolled Alloy 1, 2, 3 and 7 strip, in the longitudinal direction. The gage sections measured 0.500 × 0.240 × 0.03 in. (12.7 × 6.35 × 0.76 mm). These specimens were heated, two at a time, to 730°C in vacuum, then quenched in iced brine and aged at 100 or 150°C.

RESULTS

Two types of precipitates were observed, one of which is shown in Fig. 1. This precipitate, formed at 150°C, has a $\{100\}_\alpha$ habit plane and grows on all three of the $\{100\}_\alpha$ planes from a common center, thereby assuming a dendritic form. Previous work^{9,14,15} has indicated that this is the "low-temperature" ϵ -carbide.

At higher aging temperatures, or after longer aging, cementite precipitates, at least in Fe-C alloys. The typical appearance of such cementite particles is shown in Fig. 2. Cementite forms on $\{110\}_\alpha$ growing in $\langle 111 \rangle_\alpha$ directions. Although its appearance in Fig. 2 is markedly different from that of the metastable carbide in Fig. 1, such is not always the case, and usually the habit plane must be used to distinguish the two.

At higher aging temperatures, or with lower supersaturation, the carbides precipitate only on dislocations, but at lower aging temperatures and high supersatura-

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Table I. Compositions of Alloys, Wt Pct

Alloy	Al	C	N	Mn	P	S	Si	Cu	Ni	Cr	Mo	V	Ti	B	Cb	Co	Zr
Base				<0.01	0.002	<0.002	0.004	<0.005	0.003	0.002	<0.005	<0.005	<0.005	<0.0005	<0.005	<0.005	<0.002
1		0.023	0.0008														
2	0.43	0.023	0.0009														
3	2.0	0.022															
4	5.7	0.008	0.0009														
5	5.7	0.046	0.001														
6	5.8												0.15				
7	5.6	0.018															

tion, precipitates form both on dislocations and in the matrix, as shown in Fig. 3.

During the transition from one carbide phase to another, both can coexist over a range of aging conditions, as illustrated in Fig. 4. *In situ* transformation does not occur; that is, the carbon contained in ϵ -carbide goes into solution before precipitating as cementite.

Figures 5, 6 and 7 present the results of a number of habit-plane determinations in three of the alloys. The points appearing at the shortest times are not intended to indicate the beginning of observable precipitation of the metastable carbide. The data presented in Fig. 5 agree qualitatively with the results of previous work on binary Fe-C alloys.^{4,9} For example, Langer⁹ shows that cementite appears in an Fe-0.02 pct C alloy after aging 3.5×10^4 min at 150°C. In our Fe-0.023 pct C alloy cementite appeared after about 1.7×10^4 min at 150°C. The precise time-temperature combination for cementite initiation depends primarily on the concentration of carbon in solid solution before aging; therefore, it was essential that the carbon con-

tent of our alloys be constant. Under these conditions, cementite begins to form in the Fe-C alloy after about 10^4 min at 150°C and is the only carbide present after an hour at 250°C.

Figure 6 shows a similar plot for the 0.43 pct Al alloy, in which traces of the metastable carbide persist to much longer times at 250°C. However, the general shift of cementite formation to longer times and higher temperature is not great.

Figure 7 shows that 2.0 pct Al has a larger effect; even after 11 days at 250°C no cementite was detected. A mixture of the two carbides was seen after 3 days at 300°C and cementite alone was found after 3 days at 375°C. The transition temperature of the carbide was thus raised about 100°C by the presence of 2 pct Al.

The results of the coercive force measurements are presented in Figs. 8 to 11. The increase in coercive force is not great at 100°C (Fig. 8) and there is

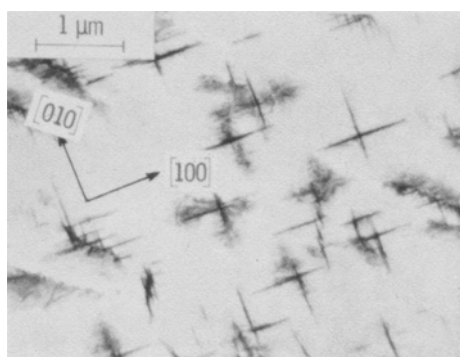


Fig. 1— ϵ -carbide in Fe-0.023 pct C, aged 256 min at 150°C.

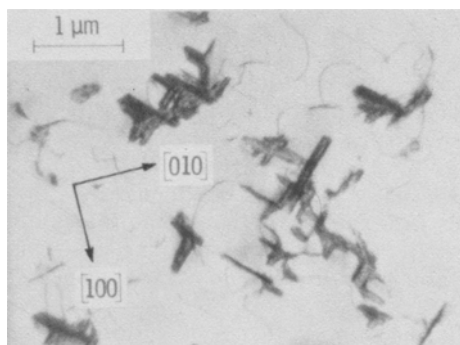


Fig. 2—Cementite in Fe-0.023 pct C, aged 64 min at 250°C.

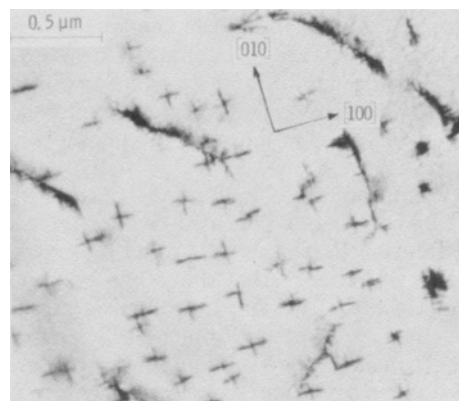


Fig. 3— ϵ -carbide in Fe-0.43 pct Al-0.023 pct C, aged 64 min at 100°C.

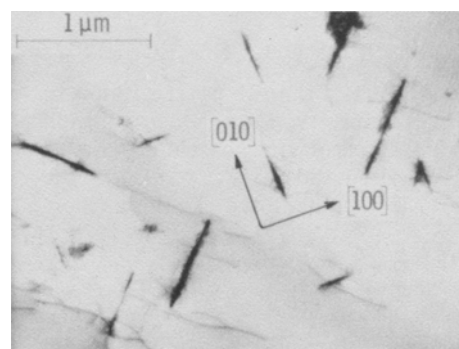


Fig. 4— ϵ -carbide and cementite in Fe-0.43 pct Al-0.023 pct C, aged 1024 min at 250°C.

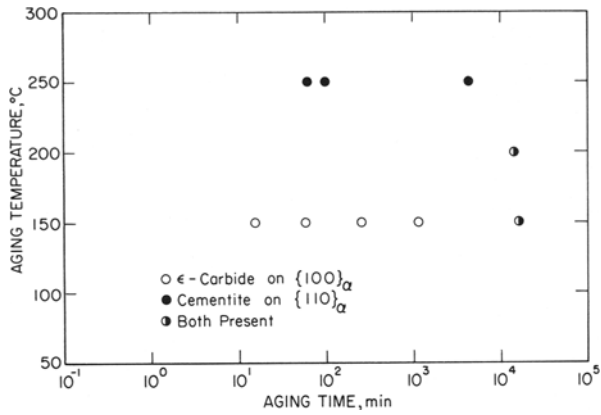


Fig. 5—Time-temperature relations for the transition from ε-carbide to Fe₃C. Alloy 1 (no Al).

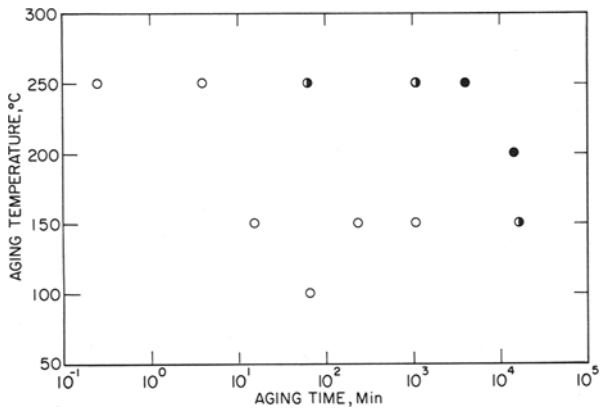


Fig. 6—Time-temperature relations for the transition from ε-carbide to Fe₃C. Alloy 2 (0.43 pct Al).

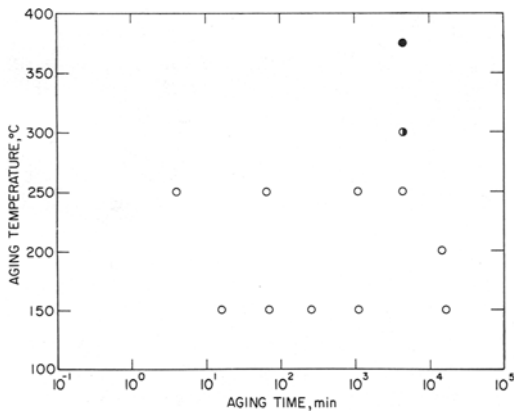


Fig. 7—Time-temperature relations for the transition from ε-carbide to Fe₃C. Alloy 3 (2.0 pct Al).

essentially no difference in the aging of the Fe-C and the Fe-0.43 pct Al-C alloy. Aging of the 2.0 pct Al alloy is delayed initially, but after about 4000 min the increase of coercive force is about the same as for the other two alloys. The first slight increase in coercive force correlates quite well with appearance of the first precipitate resolvable by electron microscopy. Alloys 4 through 7, which contain 5.6 to 5.8 pct Al, showed no change in coercive force during aging at 100°C.

The course of aging at 150°C is shown in Fig. 9. Again, the alloys containing 0 and 0.43 pct Al behave similarly, whereas 2 pct Al inhibits precipitation.

The increase in coercive force is marked, even before the appearance of cementite. With one exception, Alloys 4 to 7 showed no increase in coercive force during aging. The exception was Alloy 5 (5.7 pct Al, 0.046 pct C) which showed a large increase in coercive force after 16,400 min at 150°C.

At 200°C, Fig. 10, well-defined aging peaks appear for Alloys 1 and 2. The addition of 0.43 pct Al (Alloy 2) appears to accelerate aging slightly, but addition of 2 pct or more Al strongly inhibits aging. In the high-

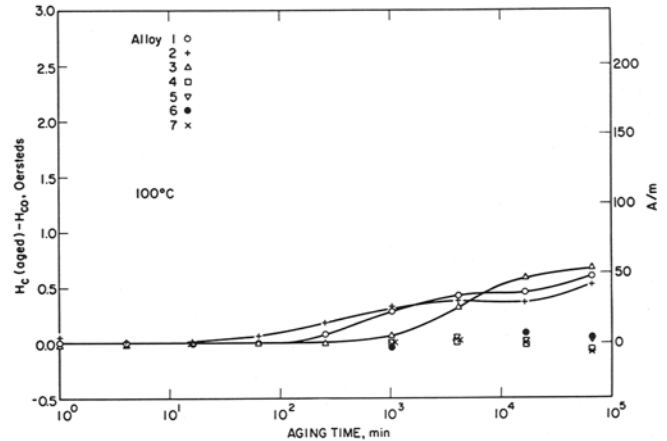


Fig. 8—Aging at 100°C, as measured by change of coercive force.

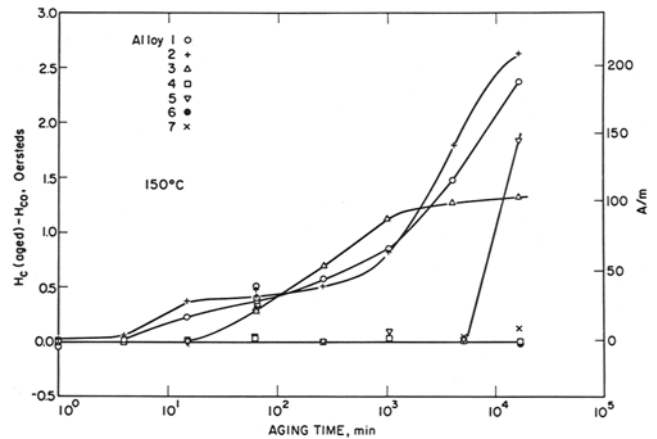


Fig. 9—Aging at 150°C, as measured by change of coercive force.

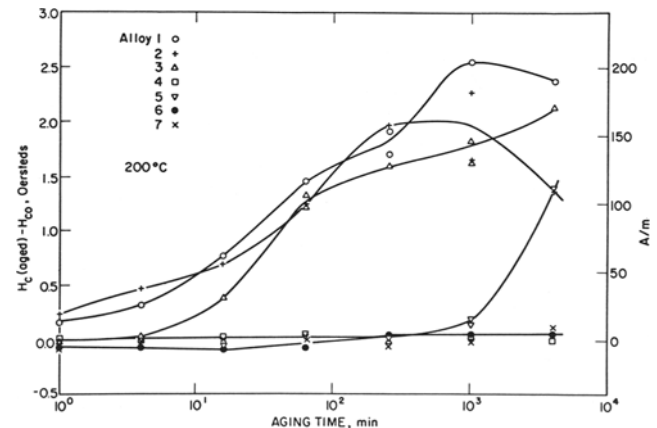


Fig. 10—Aging at 200°C, as measured by change of coercive force.

Al alloys only the one containing the most carbon shows any aging at all.

At 250°C the 0 and 0.43 pct Al alloys overage (Fig. 11) and again, the peak of aging is reached earlier in the 0.43 pct Al alloy than in the binary alloy. Aging is delayed in the 2.0 pct Al alloy, but it appears to have reached a peak even though no cementite has precipitated. Within the range of aging time employed, none of the higher Al alloys showed any aging.

We can make a direct comparison between aging of our Fe-C and Fe-2.0 pct Al-C alloys at 250°C, and the aging of two similar alloys at 250°C, reported by Jaeniche *et al.*⁸ Both Fe-C alloys show a maximum in coercive force after about 10 min at 250°C, but our peak is higher, an increase of about 2.25 Oe (180 A/m) as compared to an increase of about 1.7 Oe (140 A/m) reported by Jaeniche *et al.* The agreement on the aging of the 2 pct Al alloys is excellent, as shown by the dashed curve in Fig. 11, taken from Jaeniche *et al.*⁸

The absolute values of coercive force in these alloys may be of some interest; therefore, the coercive force data obtained immediately after quenching are listed in Table II. At the peaks of aging, the ratio $H_C(\text{aged})/H_{C0}$ had a value of 3 to 4 for Alloy 1 and about 2.5 for Alloys 2 and 3.

The results of the tensile tests are presented in Fig. 12 and 13. Aging peaks, as measured by yield strength, are reached much earlier than the aging peaks measured by coercivity, because the precipitate size leading to maximum restraint of dislocation motion² is much smaller than that exerting maximum restraint of magnetic domain wall motion.¹⁰ At 150°C a

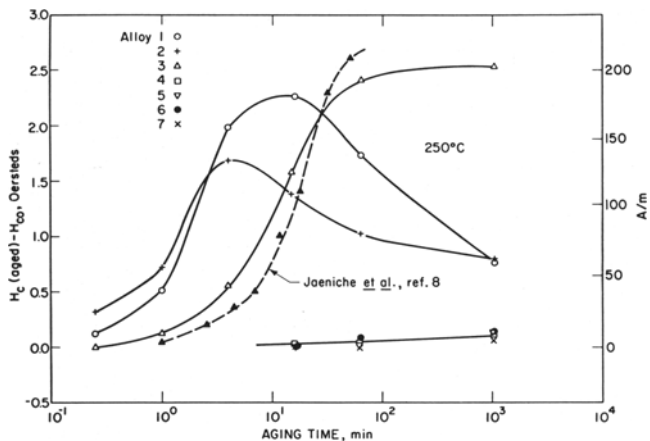


Fig. 11—Aging at 250°C, as measured by change of coercivity. The dashed line represents data obtained by Jaeniche *et al.*⁸ for a 2 pct Al alloy.

Table II. Range of Coercive Force In As-Quenched Specimens

Alloy	Coercive Force	
	Oersteds	A/m
1	0.87 to 1.49	69 to 119
2	1.16 to 1.60	92 to 127
3	0.94 to 1.86	75 to 148
4	1.46 to 2.80	116 to 223
5	2.35 to 4.78	187 to 380
6	1.66 to 3.20	132 to 255
7	1.09 to 1.86	87 to 148

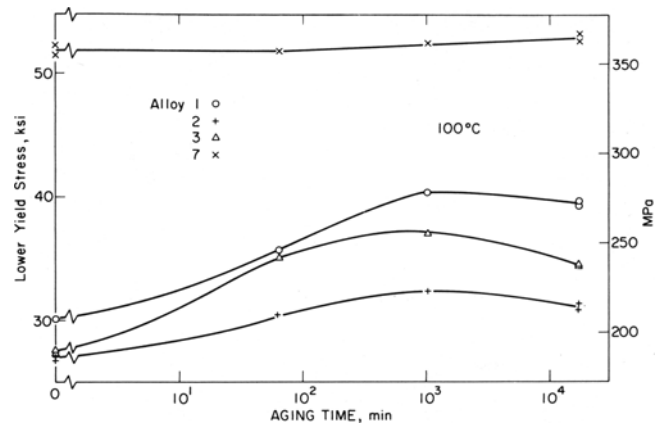


Fig. 12—Aging at 100°C, as measured by yield stress in tension.

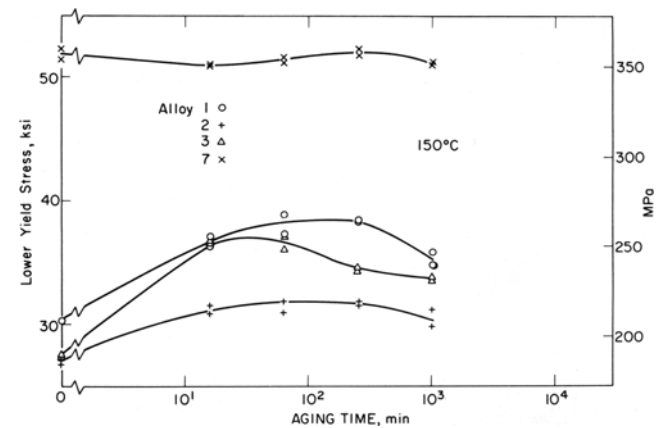


Fig. 13—Aging at 150°C, as measured by yield stress in tension.

peak of yield strength is reached for Alloy 1 after about 100 min aging and for Alloy 3 after about 30 min aging. When coercive force was the criterion, neither alloy had reached a maximum even after more than 10,000 min at 150°C. Alloy 7, containing 5.6 pct Al and 0.018 pct C, showed no aging at either 100 or 150°C.

DISCUSSION

At least three different substitutional solutes—Mn, Si, and Al—have now been shown to have a significant effect on the transition from ϵ -carbide to cementite during quenching and aging of iron-base alloys. Manganese lowers the cementite initiation temperature for a given aging time.⁴ Silicon raises this temperature markedly,^{3,4} though it has not been established that the metastable carbide is ϵ -carbide in this case. The present data show that aluminum also increases the cementite initiation temperature for a given aging time, and greatly increases the time required for cementite formation at a given temperature.

Although it has not been established that the precipitation process and sequence of transition carbides is the same in all three systems, it is desirable to examine possible mechanisms and the available data to determine whether the effects of Mn, Si and Al can be explained on a common basis. The ways in which the solute can act to modify carbide precipitation can

be considered in two categories, those affecting the relative thermodynamic stabilities of the various precipitated carbides, and those affecting kinetics. These two areas are not unrelated, since the thermodynamic driving force or free energy change will influence the critical nucleus size for precipitation.

After a relatively pure iron containing approximately 0.02 pct C is solution treated near the eutectoid temperature and then quenched to room temperature, the carbon in supersaturated solid solution is unstable with respect to graphite, cementite, and ϵ -carbide. In principle, any of the three could form from the unstable carbon in supersaturated solution. In fact, ϵ -carbide forms first, at low aging temperatures or short times. Cementite, Fe_3C , forms after longer times at a given temperature, the metastable ϵ -carbide dissolving in the process; at higher aging temperatures, cementite is the only carbide observed. Thus, in relatively pure Fe-C, cementite precipitation is controlled by kinetic factors, since at sufficiently long times cementite is ultimately formed over a range of aging temperatures. (Although graphite is more stable than cementite, its formation is also constrained by kinetic factors.)

If thermodynamic effects are to furnish an explanation of the influence of substitutional solutes, then the solute addition must modify the relative stabilities of cementite, ϵ -carbide (or other transition carbide), and carbon in supersaturated solid solution.

We will consider cementite first. Neither silicon^{16,17} nor aluminum¹⁶ have significant solubility in cementite. Manganese, on the other hand, substitutes freely for Fe in $(\text{Fe}, \text{Mn})_3\text{C}$,^{16,18,19} and the equilibrium distribution coefficient of manganese in cementite relative to that in ferrite rises with decreasing temperature.^{18,19} Because substitutional diffusion is very slow at the temperatures employed in quenching and aging studies, the initial cementite to form even in alloys with Si and Al probably has the same content of substitutional solute as the matrix. However, Kuo and Hultgren,¹⁶ whose data provide the chief support for this contention for silicon additions, did not specifically identify the cementite by X-ray diffraction, and other carbide phases are known to precipitate under their conditions.³ In any event, the free energy of $(\text{Fe}, M)_3\text{C}$ is higher than that of Fe_3C when $M = \text{Si}$ or Al , and is lower when $M = \text{Mn}$. Neglecting other effects, then, the presence of Si or Al decreases the relative stability of cementite, and Mn increases it.

Of course, other effects cannot be neglected. It is not known, for example, how substitutional solutes change the relative stability of the low-temperature ϵ -carbide or other transition carbide. There are, however, additional data to help assess possible changes of the relative stability of carbon in ferrite, as reflected in the activity coefficient of carbon in ferrite. A change in the activity coefficient of carbon in ferrite can act in various ways. In alloys containing carbon in excess of the solubility limit at the solution temperature, a decrease in the activity coefficient will increase the amount of carbon dissolved, unless there is a compensating change in the carbon activity of the precipitated phase. After quenching, there will thus be increased carbon in supersaturated solution. (Whether this increased carbon represents increased carbon activity at the aging temperature depends on the varia-

tion of activity coefficient with temperature.) An increased activity coefficient will have the opposite effect, decreasing the concentration of carbon in solution after quenching. On the other hand, in alloys containing less than the maximum amount of carbon soluble at the solution temperature, as in the present Fe-Al-C alloys, a change in activity coefficient changes the activity of the carbon present. Whether the driving force for precipitation is also changed depends on whether any compensating changes occur in the activity of carbon in available precipitate phases.

Complete data on activity coefficients of carbon in the systems Fe-Si-C, Fe-Mn-C, and Fe-Al-C do not exist. Available data are, however, quite instructive. Smith²⁰ determined the influence of silicon on activity coefficients of carbon in both ferrite and austenite, in equilibrium with graphite at 1000°C. He found that silicon significantly increases the activity coefficient of carbon in both ferrite and austenite; the addition of silicon accordingly decreases the solubility of carbon in ferrite with respect to cementite^{21,22} (although one study⁶ showed a slightly increased solubility).

The influence of manganese on the activity coefficient of carbon in ferrite has not been determined, but Smith²⁰ found that manganese caused a decrease of the activity coefficient of carbon in austenite with respect to graphite. The absolute magnitude of the change in activity coefficient was approximately the same for additions of silicon and of manganese, on an atomic basis. It is logical to assume that manganese similarly lowers the activity coefficient of carbon in ferrite, but this is not known. If manganese does lower the activity coefficient of carbon in ferrite, carbon solubility should be increased; however, both Lagerberg²³ and Borchers and Koenig⁶ report a slight decrease in solubility with respect to cementite in ferrite. Lagerberg²³ has suggested that a decrease in the carbon activity of cementite may be responsible for the decreased solubility; Lagerberg suggests that this mechanism is reasonable, because Fe_3C and Mn_3C are isomorphous and mutually soluble²⁴ and the carbon activity of Mn_3C is much lower than that of Fe_3C .²⁵ In this instance reduced carbon activity coefficient in ferrite does not result in greater carbon solubility because of the modified cementite composition and resulting decrease in carbon activity in cementite.

The influence of aluminum on the activity coefficient of carbon in ferrite has not been explicitly evaluated in any previous work known to the authors. There is disagreement concerning the influence of aluminum on the activity coefficient of carbon in austenite,²⁶ but the most recent work indicates that aluminum decreases the activity coefficient.²⁷ Measurements of the effect of aluminum additions on solubility of carbon in ferrite relative to cementite should provide an indication of the behavior of the activity coefficient, since aluminum is essentially insoluble in cementite at equilibrium.¹⁶ Such measurements are available from internal friction studies. Borchers and Koenig⁶ concluded that aluminum markedly increased the solubility; Jaeniche *et al*⁷ reported a more modest increase in solubility. Laxar *et al*⁵ concluded that aluminum decreased the solubility of carbon; however, the proportionality factor between damping and carbon content was not explicitly evaluated as a function of aluminum content in this study. When the variation of

proportionality factor with aluminum content⁶ is taken into account, the data of Laxar *et al* also indicate increased carbon solubility in the presence of aluminum. Solubility data thus indicate that aluminum decreases the activity coefficient of carbon in ferrite.

Further evidence that aluminum decreases the activity coefficient of carbon is provided by measurements of magnetic after-effects reported by Kozłowski *et al*,¹³ who estimated the binding energy of Al-C pairs in solid solution in ferrite (Fe-0.1 pct Al-C) to be about -5200 cal/mol (-22 kJ/mol) and that for C-C pairs (again in α -Fe-0.1 pct Al-C) to be about -2600 cal/mol (-11 kJ/mol). These energies should be compared with -1900 cal/mole (-8 kJ/mol) for C-C pairs in pure Fe-C alloys, and with the generally accepted value of about -0.6 eV/atom or -14,000 cal/mol (-58 kJ/mol) for the binding energy of carbon to a dislocation.²⁸

Available evidence on the influence of substitutional solutes on carbon activity thus leads to the tentative conclusions summarized in Table III. A more complete description would require knowledge of the influence of solute additions on the stability of the transition carbide, and of more quantitative data on the carbon activities in cementite. A rationalization of the effects of manganese, silicon, and aluminum on the carbide transition is presented in the following, based on Table III.

It appears that changes in the activity coefficient of carbon in ferrite cannot alone explain the observed influence of substitutional solutes on carbide transition temperature. For the three solutes studied thus far, a better correlation exists with the characteristics of the cementite itself. Both aluminum and silicon have negligible equilibrium solubility in cementite, and both retard Cementite precipitation. Manganese distributes preferentially to cementite and lowers the temperature at which cementite begins to form. The effect of silicon could be caused by a necessity to diffuse silicon away from growing cementite,^{4,29} but in the Fe-Al-C alloys cementite forms at temperatures too low to permit extensive diffusion of aluminum. Any cementite formed will thus be less stable than Fe₃C, and there will be less driving force for its formation. Quantitative microanalysis of carbide composition and simultaneous identification of carbide type would be required to more fully characterize the precipitation sequence. Since manganese is soluble in both ferrite and cementite, there is no necessity for manganese diffusion; the

presence of manganese does, however, make cementite more stable, and increases the driving force for precipitation.

In this view, the variation of carbon activity coefficient in ferrite with alloy addition works in the same direction as the alloy effect on carbon activity in cementite only for aluminum; both decreased carbon activity coefficient in ferrite and increased carbon activity in cementite can be expected to retard cementite precipitation. In the manganese case, it is presumed that the increased stability of cementite more than compensates for the decreased carbon activity coefficient in ferrite. For silicon, the increased carbon activity coefficient in ferrite similarly appears to work against the influence of silicon on the cementite stability, but in this case an additional mechanism has been proposed. Owen²⁹ has suggested that the increase of the carbon activity coefficient in ferrite itself inhibits precipitation, since silicon rejected from the growing cementite forces carbon to diffuse up an activity gradient to reach the cementite. Cementite growth thus stops after a time in silicon alloys,² while it continues in Fe-Al-C alloys. Aluminum is thus not as effective as silicon in preventing magnetic aging.

CONCLUSIONS

- 1) Aluminum additions to low-carbon iron inhibit the precipitation of cementite, increasing the time and temperature required for cementite to replace the transition carbide.
- 2) The effect of substitutional solute additions on cementite stability is the predominant influence on carbide precipitation during quenching and aging in the systems Fe-Mn-C, Fe-Si-C and Fe-Al-C.

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Table III. Influence of Substitutional Solute On Carbon Activity and Carbide Precipitation

Solute	Mn	Si	Al
Activity coefficient of carbon in ferrite	Decreased	Increased	Decreased
Carbon activity in cementite	Decreased	Increased	Increased
Carbon activity in transition carbide	Unknown	Unknown	Unknown
Carbide transition temperature	Decreased	Increased	Increased
Rate of carbide growth	Unchanged	Decreased	Decrease d

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