

The Growth of Ferrite in Fe-C-X Alloys: The Role of Thermodynamics, Diffusion, and Interfacial Conditions

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The growth of allotriomorphic ferrite from austenite in Fe-C-X alloys is studied. Two systems have been selected: the Fe-C-Ni system, in which the substitutional alloying element is expected to have a weak interaction with both the C and the moving interface, and the Fe-C-Mo system, in which these interactions are expected to be non-negligible. The ferrite growth kinetics was measured using two types of experiments: classical isothermal heat treatments and decarburization experiments. All of the experimental observations can be quantitatively rationalized using a model that describes an evolution in interfacial conditions from paraequilibrium (PE) to local equilibrium with negligible partitioning (LENP) during growth.

I. INTRODUCTION

THE diffusional growth of ferrite from austenite in steels has been the focus of considerable research during the last 50 years. Since Zener's attempts at quantitatively rationalizing the ferrite growth kinetics,^[1,2] our theoretical understanding and quantitative treatments have constantly been renewed in the same tradition through the insight of Mats Hillert. Most of the questions investigated in the present contribution have been triggered by the continuous series of contributions he and his school have made to the field.

Of the variety of morphologies the ferrite can adopt when forming from the austenite, the allotriomorphic morphology is the simplest and its formation in the binary Fe-C system can be seen as the simplest example of an interface mediated phase transformation controlled by diffusion. The shape complexity associated with Widmanstätten ferrite is absent; the possible influence of displacive components, which makes bainite so controversial, can be discarded; and the elastic stresses, which play such an important role in martensitic transformations, are probably relaxed. The diffusion of carbon, C, into the austenite is clearly the limiting factor, and from this, we can write a very simple and efficient model for diffusional growth, assuming local thermodynamic equilibrium at the moving interface.

The situation immediately becomes more complex when a third element, X, is added to the system. The case of particular interest is when X is a substitutional atom that diffuses many orders of magnitude slower than the interstitial C. The transformation remains controlled by diffusion, but the rate of reaction can be widely different depending on whether the substitutional atoms are partitioned between the austenite and the growing ferrite. The general problem

of the possible thermodynamic conditions for growth, namely, local equilibrium with partitioning (LEP), local equilibrium with negligible partitioning (LENP), and paraequilibrium (PE), have all received considerable attention, both from the fundamental viewpoint^[3-7] and because of the practical implications for modeling phase transformations in industrial alloys. This contribution is concerned with two outstanding issues, both of which have received much interest in recent years. These are the selection of the appropriate interfacial conditions, and the possible role of solute drag in the ferrite growth kinetics in Fe-C-X alloys.

We first outline a simple classification of the different Fe-C-X systems according to the interaction between X atoms and C, and the binding between X atoms and the moving phase interface (Section II). We have used two different types of experiments to study the transformation: (1) classical isothermal treatments and (2) decarburization experiments. These experiments and the way in which they complement one another are discussed in Section III. In Section IV, we summarize the results for ferrite growth in the Fe-C-Ni system, which can be seen as a paradigm of the simplest situation where the substitutional element has only a weak interaction with both C and the moving interface. In spite of this simplicity, the need to take into account the evolution of the interfacial conditions while the reaction proceeds is stressed. We then consider the Fe-C-Mo system where the substitutional Mo atom interactions with both C and the moving phase interface are no longer expected to be negligible. The experimental evidence of a clear additional dissipation associated with the Mo addition is presented in Section V. This dissipation may be interpreted in terms of a solute drag effect on the moving interface.

II. A CLASSIFICATION OF Fe-C-X SYSTEMS

The addition of a slow diffusing substitutional element (X) to the Fe-C system introduces a large increase in complexity in the transformation behavior. Two aspects have to be considered in our attempts at understanding this behavior: the interaction between X and C, and the interaction between X and the moving interface. The interaction with C can be quantified by the dependence of the C activity on the solute content. This dependence is very weak for Ni additions but increases significantly with the strong carbide

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formers such as Nb. When carbides can form, further complexities such as additional carbon fluxes, modification of the interfacial conditions, or pinning of the interface by precipitates can appear (for an overview of this aspect, see Reference 8). In the present article, we will consider only alloying elements, which, in the range of heat treatments examined, do not lead to precipitation. The interaction of solute X with the moving interface is more difficult to measure or estimate. It is known that solute interaction energies with grain boundaries are well correlated to the size effect of the solute atom.^[9] This allows a ranking of the solute atoms in terms of increasing binding energies to the grain boundaries, and, in the absence of any more detailed information, it is expected that the same classification (though with different binding energies) should hold for interactions with the interphase boundary. Figure 1 illustrates schematically an estimated scale of the interactions between X and C and X and the interphase boundary. A similar scale was suggested earlier by Aaronson, Reynolds, and Purdy (Table 1 in Reference 10).

This very simple classification allows us to identify the model Fe-C-X systems that we are concerned with in this article. The Fe-C-Ni system will be the paradigm of a system where the solute atom is weakly interacting with carbon, and has barely any interaction with the interface. The solute drag effect, if any, is expected to be small. We will call such alloys, involving Ni and probably Co as solute elements, class I alloys. The Fe-C-Mo system, on the other hand, is expected to show a stronger interaction between the substitutional Mo atom and the moving interface, so that a solute drag effect may be more likely to be important in this system. We will call such alloys, involving Mo and Cr as solute elements, class II alloys. Systems such as Fe-C-Nb or Fe-C-V that exhibit a strong carbide forming tendency will form the third class of alloys. It must be stressed that this classification refers both to the alloying elements and to the heat-treatment conditions adopted. For instance,

the Fe-C-Mo system shifts from class II behavior to class III behavior as the transformation temperature is lowered and carbide precipitation occurs concurrently with the austenite-to-ferrite transformation.

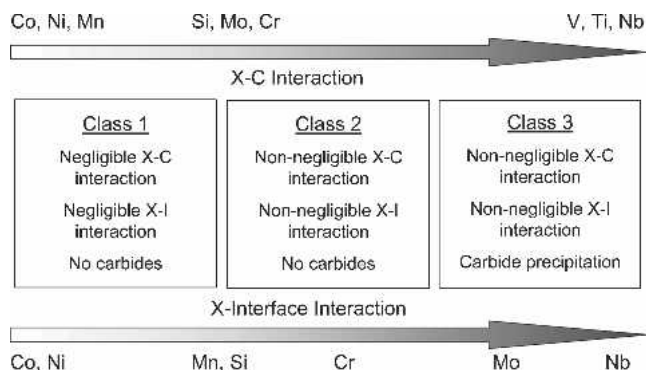


Fig. 1—Classification of the classical substitutional alloying elements (X) in steel: (i) according to their interaction with C and (ii) according to their estimated binding energies with the austenite/ferrite interface.

Table I. Comparison of Experimentally Measured α Volume Fractions in a Series of Fe-C-Ni Alloys and the Theoretically Expected Mole Fractions Assuming LENP and PE

Alloy Composition (Wt Pct)	Experimentally Measured α Volume Fraction	PE α Mol Fraction	LENP α Mol Fraction
Fe-0.0885C-2.02Ni	0.82	0.881	0.817
Fe-0.0780C-2.41Ni	0.77	0.884	0.761
Fe-0.0715C-2.76Ni	0.66	0.881	0.574
Fe-0.0625C-3.14Ni	<0.1	0.881	—
Fe-0.0550C-3.33Ni	<0.1	0.888	—

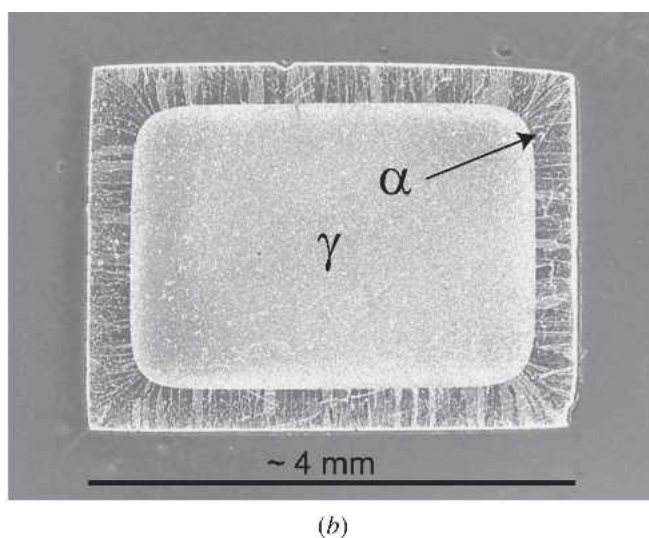
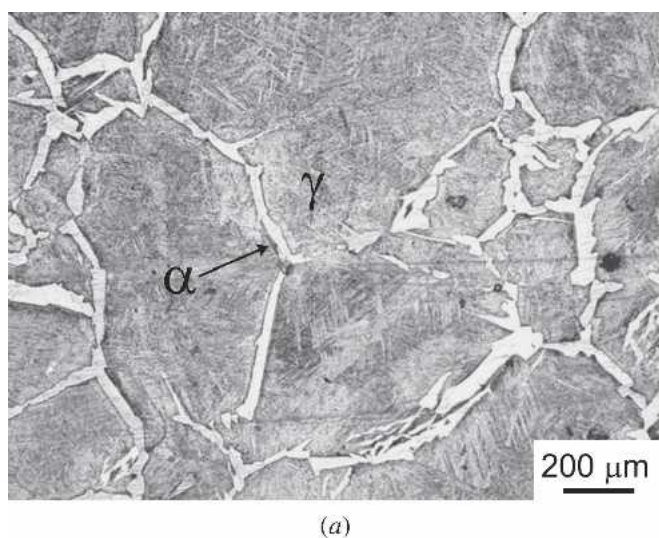


Fig. 2—Representative optical micrographs illustrating the difference in ferrite product arrangement in (a) traditional isothermal experiments and (b) in decarburization experiments. The sample shown in (a) is a Fe-0.1C-0.1Mo (wt pct) alloy transformed for 1 min at 800 °C. The sample shown in (b) is from a Fe-0.54C-0.51Mo (wt pct) alloy transformed for 128 min at 825 °C.

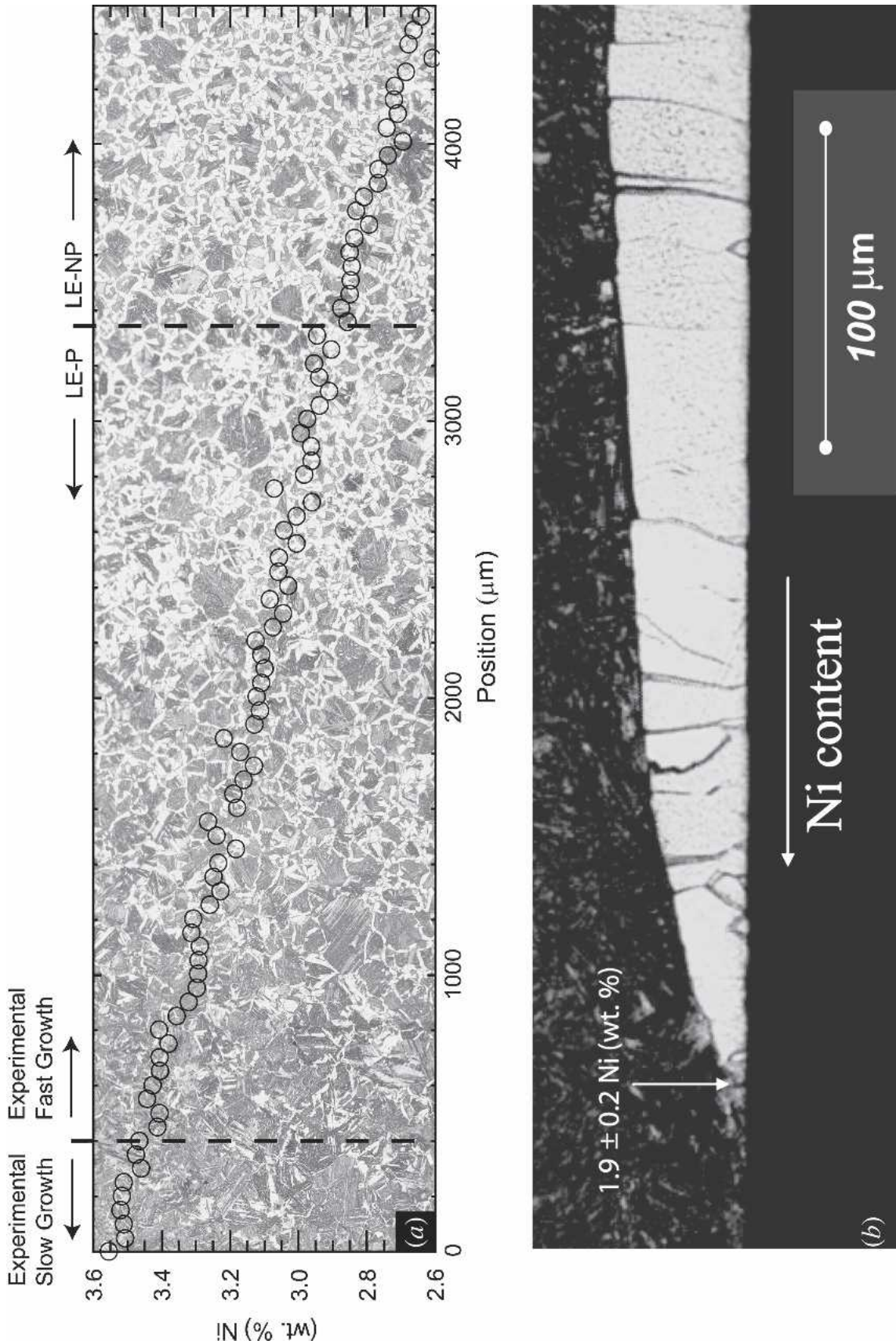


Fig. 3—Optical micrographs illustrating the formation of ferrite from austenite in samples containing a gradient in Ni during (a) traditional isothermal experiments and (b) decarburization experiments. The sample shown in (a) is an Fe-1Ni/5Ni-0.1C (wt pct) alloy transformed at 700 °C for 2 min.^[19] The sample shown in (b) is from a Fe-1Ni/5Ni-0.45C alloy transformed at 775 °C for 4 min.^[15]

III. EXPERIMENTAL INVESTIGATIONS OF THE AUSTENITE-TO-FERRITE TRANSFORMATION

Modeling phase transformation requires accurate experimental data for comparison. Although many different techniques have been used to measure the kinetics of the austenite-to-ferrite transformation, and in spite of the stereographical hypothesis necessary for the interpretation of two-dimensional observations of a three-dimensional phenomenon, conventional metallography still remains the most accurate source of growth kinetic data. The case of allotriomorphic ferrite, with its relatively simple shape, which can be characterized by the thickening aspect only, can be dealt with accurately using these methods.

The most straightforward and frequently used approach to measuring the transformation kinetics is to first austenitize a sample and then isothermally transform it for a defined time followed by quenching into water. The thickness of the allotriomorphic ferrite can be measured using standard metallographic techniques and the transformation kinetics can be obtained from a series of such treated samples (*e.g.*, References 11 through 13). An alternative technique, which dates back at least to the 1960s^[14] and which has recently been used to measure the transformation kinetics,^[15] is the decarburization of a sample initially in the fully austenitic state. The decrease in carbon content at the surface of the sample drives the transformation to ferrite, and one can measure the thickening rate of the ferrite layer growing into the sample from the surface. These two techniques allow measurements of the kinetics at a given temperature and the interpretation depends on the X and C compositions at the migrating ferrite/austenite interface (*i.e.*, the boundary conditions for the C diffusion problem). In the classical isothermal experiments, the C flux occurs in the austenite from

the migrating interface to the bulk. However, in the decarburization experiments, the carbon flux involves diffusion both from the ferrite to the outer surface of the sample and from the austenite to the ferrite. The growth velocity, at a given temperature, is much smaller for the decarburization experiments (typically by an order of magnitude) than for the isothermal transformation. Both techniques allow an analysis of the data in terms of interfacial X and C compositions, and their comparison allows a direct insight into the effect of interface velocity on the interfacial conditions. Representative micrographs illustrating the different arrangements of the product ferrite phase in the two types of experiments are shown in Figure 2.

A physically based model must be able to describe the kinetics of transformation for a range of temperatures and compositions. Varying the temperature is relatively easy. Changing the composition is more time and materials consuming, since alloys at various compositions must be prepared. An astute way to bypass this difficulty was proposed by Hillert^[16] and relies on the use of a diffusion couple to explore a range of compositions in the one sample. The technique is especially useful for identifying critical compositions corresponding to transitions in behavior.^[17,18] A practical difficulty in preparing diffusion couples with a gradient in X has been that, for reasonable heat-treatment times, the diffusion profile of the solute X element extends only a few hundred microns and this is far too narrow to perform the transformation and measure locally the plate thickening kinetics. This difficulty can be resolved by rolling the sample.^[19] The composition profile in X can then be spread to a few millimeters or more. The sample can then be carburized and the heat treatment or the decarburization treatment can be performed on a sample with a constant

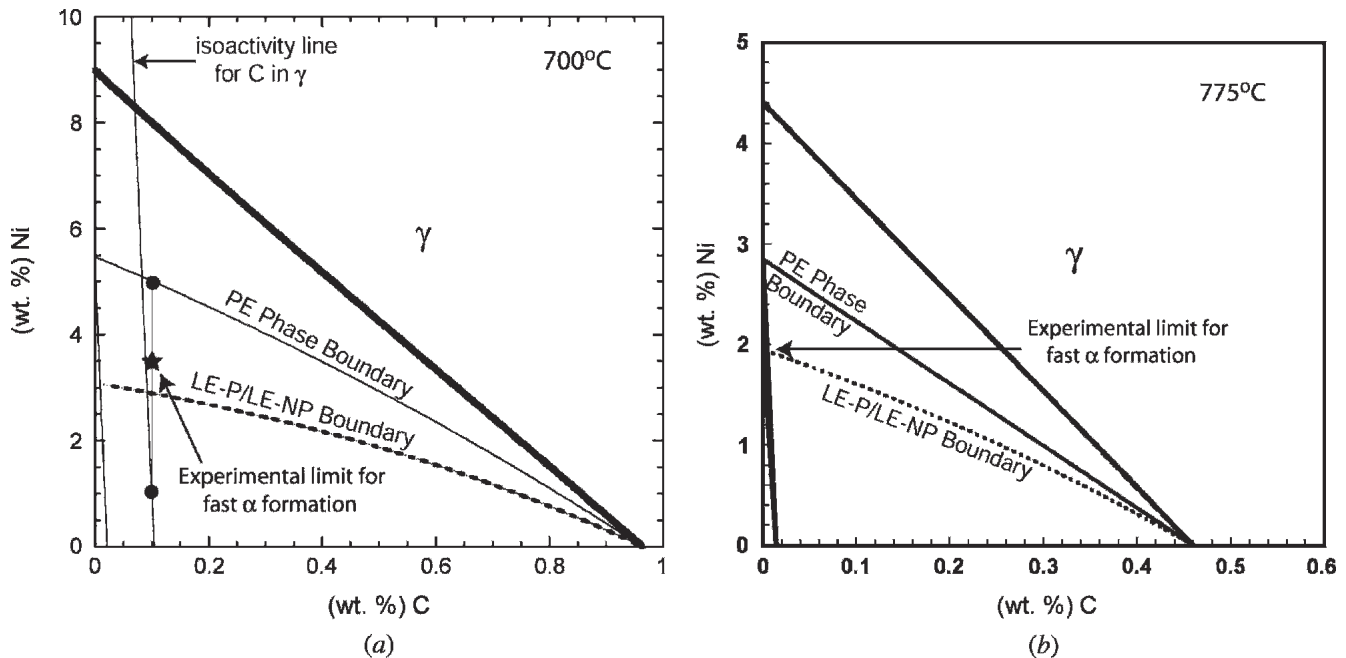


Fig. 4—Isothermal sections of the Fe-Ni-C phase diagram at (a) 700 °C and (b) 775 °C, illustrating the equilibrium phase boundaries, the LEPN boundary, and the PE phase boundaries. The full circles in (a) represent the limits of the Ni contents sampled in the diffusion couple. The critical composition above which the austenite-to-ferrite transformation becomes very slow is shown, both for (a) the classical isothermal heat treatment performed at 700 °C and (b) for the decarburization experiment performed at 775 °C.

carbon chemical potential and a gradient in the substitutional element. In a single experiment, one can identify the critical compositions above which the austenite-to-ferrite transformation becomes sluggish, due to the partitioning of the slow diffusing element between the two phases. Such experiments performed in the Fe-C-Ni system^[15,19] are illustrated in Figure 3 and allow an identification of the critical compositions, both for the traditional isothermal heat treatments (Figure 3(a)) and the decarburization experiments (Figure 3(b)).

This use of gradient samples allows for an identification of the compositions of interest for which the overall transformation kinetics can be studied more accurately in samples of homogenous composition. At this very preliminary stage of an experimental campaign, by comparing the critical compositions measured experimentally with the

limits of the phase diagram (Figure 4), we can see that the transition to very slow kinetics occurs exactly for the composition corresponding to the LENP boundary in the decarburization experiments (Figure 4(b)),^[15] whereas a fast ferrite reaction can still occur for compositions above the LENP boundary in the classical isothermal experiments (Figure 4(a)).^[19,20,21]

IV. THE Fe-C-Ni SYSTEM: THE PARADIGM FOR A NONINTERACTING SOLUTE SYSTEM

At first sight, the decarburization experiments seem simpler to interpret than the classical isothermal treatment experiments. Below the critical LENP composition, the ferrite inherits the solute X content of the austenite and

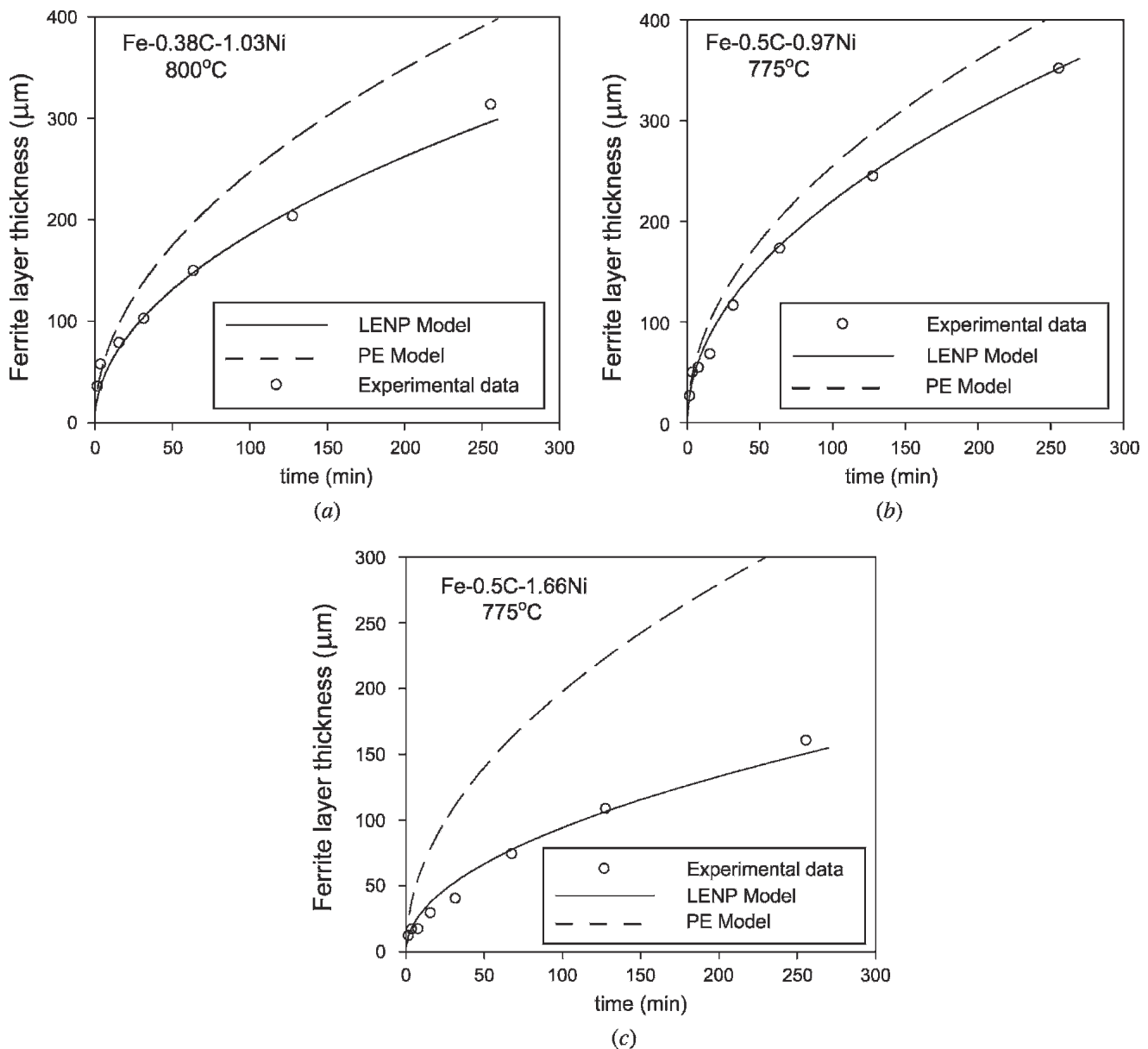
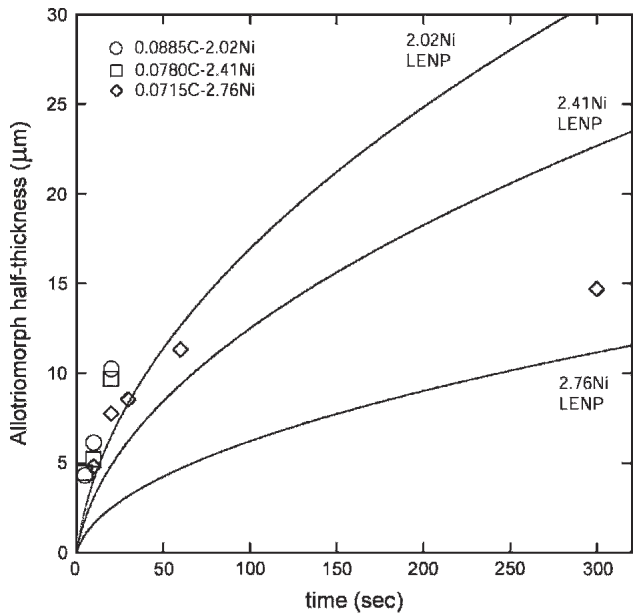
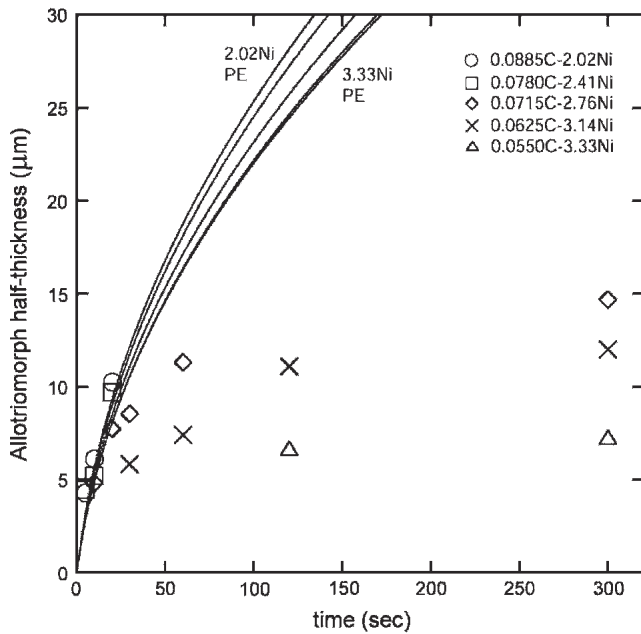


Fig. 5—Ferrite layer thickening kinetics observed in decarburization experiments. (a) Fe-0.38C-1.03Ni (wt pct) at 800 °C, (b) Fe-0.5C-0.97Ni (wt pct) at 775 °C, and (c) Fe-0.5C-1.66Ni (wt pct) at 775 °C.^[15] In each case, the kinetics predicted assuming LENP and PE have been included for comparison. The experimental data show excellent agreement with the LENP model.

growth occurs quickly, controlled by the diffusion of C. The model developed relies on the diffusion profile of C in the austenite and ferrite. Assuming for the interfacial conditions either PE or LENP (*i.e.*, assuming that a Ni concentration spike exists) allows us to compute the ferrite thickening kinetics, which vary as $t^{1/2}$. These have been compared with the experimentally observed kinetics in



(a)



(b)

Fig. 6—Ferrite growth kinetics measured in a series of traditional isothermal experiments for a range of Fe-C-Ni alloys transferred at 700 °C. (a) Comparisons with the LENP model predictions and (b) comparisons with the PE model predictions. The compositions containing 3.14 and 3.33Ni (wt pct) lie above the LENP boundary at 700 °C and therefore their kinetics are not shown in (a).

homogeneous samples,^[15] and the agreement between the model predictions* and the experimental results clearly

*Knowledge of the C diffusivity in ferrite and austenite is required for the PE and LENP calculations of ferrite growth in the decarburization experiments. We have used the expression developed by Agren^[22] for the diffusivity of C in austenite. For the case of C diffusion in ferrite, Agren^[23] reports that there is considerable scatter in the available experimental data in the vicinity of the ferromagnetic transition. This includes the range of temperatures that we have been investigating. For each temperature we examined, the C diffusivity in ferrite was calibrated by measuring the kinetics of ferrite growth in a binary Fe-C alloy under identical decarburization conditions assuming local equilibrium conditions at the interface. In each case, the C diffusivities in ferrite were very close to the values found from the expression proposed by Agren. The actual C diffusivities in ferrite used for the calculations, as a fraction of the values predicted by Agren's^[23] expression, are 0.95 at 775 °C, 0.8 at 800 °C and 806 °C, and 0.73 at 825 °C.

shows that the interfacial conditions are given by LENP (Figure 5). The kinetics predicted assuming PE conditions are much too fast.

The case of the classical isothermal treatments in comparison seems more difficult to interpret. A series of alloy compositions were chosen on the basis of the observations made in the gradient sample (Figure 3(a)); homogeneous samples were fabricated, and were then transformed at 700 °C for a range of times to determine the change in ferrite thickness with time.^[20] The results are compared with the LENP and PE model predictions in Figure 6.

The kinetics predicted by a diffusion model with LENP interfacial conditions is too slow at the beginning of the reaction, although it seems to describe reasonably the long-term behavior. The volume fraction of ferrite obtained at the end of the transformation coincides with those predicted by the LENP hypothesis (Table I). By contrast, the kinetics predicted assuming PE conditions describe correctly the initial

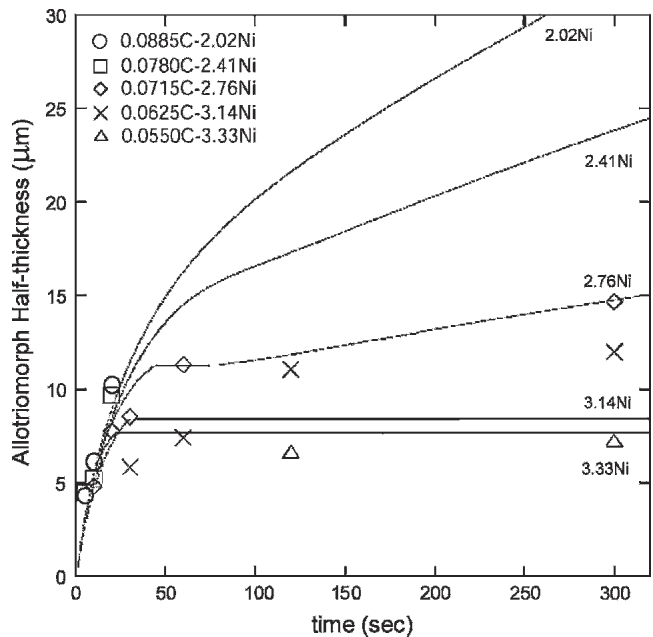


Fig. 7—Comparison of the ferrite growth kinetics measured in the traditional isothermal experiments in the series of Fe-C-Ni alloys (Figure 6) and the results of a model describing a change in interfacial compositions from PE to LENP during growth.^[20]

stages, but are much too rapid at the later stages of transformation. In addition, the experiments in the gradient sample shown in Figure 3(a) illustrate that a fast reaction is possible even above the LENP limit. This would not be possible if LENP conditions existed at the interface at all times.

This collection of experimental results suggests a transition in behavior from PE interfacial conditions at the beginning of the reaction toward LENP conditions at the end of the reaction. The concept of evolving interfacial conditions during transformation has also been suggested by Hillert and his co-workers.^[24,25]

A model, whose details can be found in Reference 20, has been developed in order to describe this PE \rightarrow LENP transition. It assumes PE interfacial conditions as the starting point, and during growth, a Ni spike progressively develops because of an unbalanced flux of Ni across the interface, driven by the differences in chemical potential of Ni and Fe. The additional ingredient is the efficiency of the cross-interface jumps, which depend on the interface velocity. When the interface is moving slowly, the spike will build up more rapidly since the cross-interfacial jumps will be more efficient. This model was solved numerically and provides an accurate description of both the decarburization kinetics (Figure 5) and the classical isothermal heat treatments (Figure 7).

Interestingly, the transinterface mobility of solute Ni required to adjust the model appears to be the geometric average of the mobilities of Ni in the austenite and in ferrite, a result to be compared with the recent model proposed by Martin and co-workers for the mobility of solute across diffuse interfaces.^[26,27] With these parameters, making similar calculations for the range of velocities obtained in the decarburization experiments (Figure 5), the transition from PE to LENP is calculated to occur within the first 1 min, and this explains the ability of the simplest diffusion model with constant LENP interfacial conditions to describe accurately the decarburization kinetics.

The results obtained from both the traditional isothermal experiments and the decarburization experiments for the Fe-C-Ni system can be quantitatively rationalized using the minimal ingredients included in the PE \rightarrow LENP transition model discussed in Reference 20. An important feature of this model is that it emphasizes only the variation in the boundary conditions for the C diffusion problem and assumes that the atomic rearrangements in the vicinity of the interface necessary to build the Ni spike and to maintain it during growth dissipate a negligible amount of free energy. This differentiates our approach from the recent approach of Odqvist *et al.*,^[25] which also considers a transition from PE to LENP during growth. Certainly, from the excellent agreement observed between the LENP model predictions and the growth kinetics observed in the decarburization experiments (which allow us to measure the kinetics very accurately and reproducibly), at least in some cases, the free energy dissipated by the rearrangements necessary to maintain the spike are negligible. In the Fe-C-Ni system, we have not needed to invoke any additional dissipations associated with the solute Ni interacting with the migrating boundary (solute drag) or jumping across it to obtain quantitative agreement with the experimental observations. Knowing the similarity between Fe and Ni atoms,

this may not be very surprising. However, another feature is worth highlighting: during a phase transformation, it is possible that the interface may slow purely because of a variation in the interfacial conditions from PE to LENP without any significant free energy dissipations associated

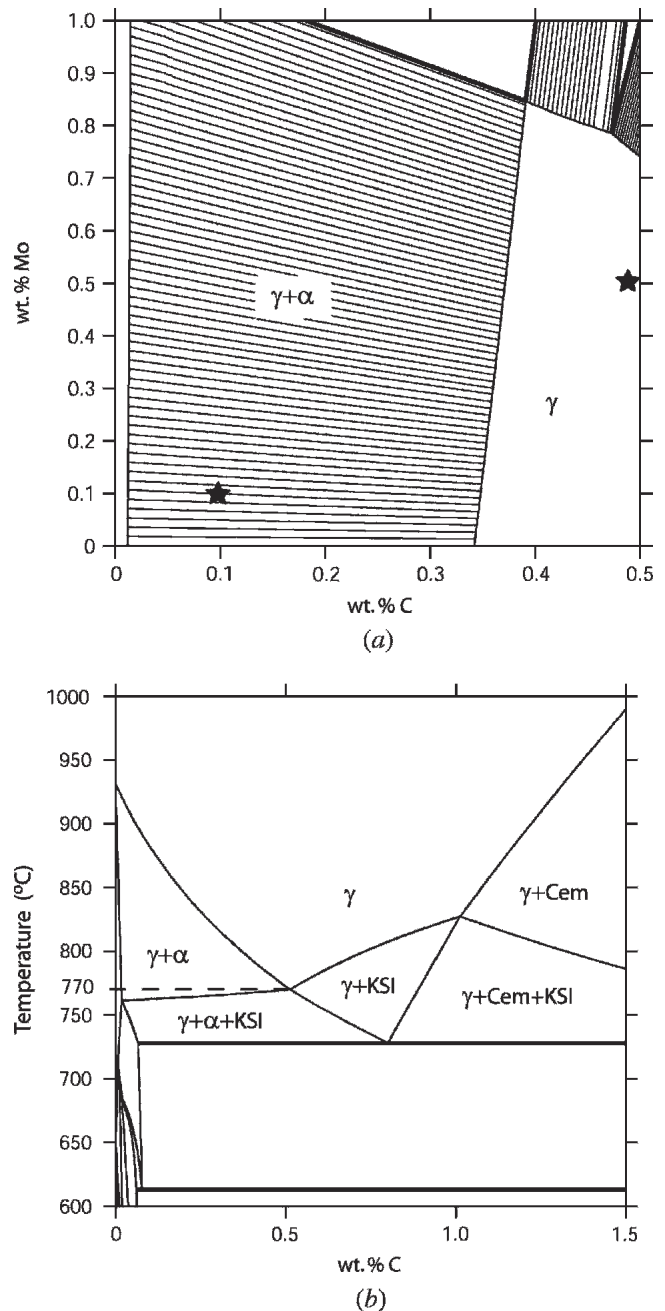


Fig. 8—(a) Isothermal section of the Fe-rich corner of the Fe-C-Mo phase diagram taken at 800 °C. For Mo concentrations less than ~ 1 (wt pct), the tie-lines in the two-phase field are relatively flat. The star located inside the two-phase field marks the alloy composition used for the traditional isothermal experiments. The star located in the austenite single-phase field represents the alloy used for the decarburization experiments. (b) An isopleth of the Fe-C-Mo system taken at 0.5 (wt pct) Mo illustrating that, at temperatures below ~ 770 °C, there is the possibility of the appearance of the KSI carbide. For experiments where the PE and LENP growth kinetics are near identical and there is no possibility of carbide precipitation, we are restricted to conditions of temperature above ~ 770 °C and Mo contents less than ~ 1 (wt pct).

with the interfacial processes. The difficulty then arises as to how we can unambiguously distinguish a solute drag effect (arising from the intrinsic interaction between the solute atom and the migrating interface) from the effect arising from a variation in the interfacial conditions. A solution is to identify experimental conditions where no PE to LENP transition can be observed, and it is this question that drives the design of our experiments on the Fe-C-Mo system.

V. THE Fe-C-Mo SYSTEM: THE EXPERIMENTAL EVIDENCE FOR A SOLUTE DRAG EFFECT

It is traditionally expected that in the Fe-C-Mo system, a non-negligible interaction between the alloying element

and the boundary will be observed and that this will manifest itself in the form of a solute drag effect.^[10,11,28-31] To investigate this effect, we have identified experimental conditions where the ferrite growth kinetics predicted from the PE model and the LENP model give identical results. This corresponds to conditions in the phase diagram where the tie-lines in the (austenite + ferrite) two-phase field coincide with lines at constant Mo U fraction (X_{Mo}/X_{Fe}). The isothermal section taken at 800 °C shown in Figure 8(a) illustrates that this condition is well satisfied for Mo concentrations less than ~1 wt pct. However, as an additional condition, we also want to avoid the complications associated with carbide precipitation, and the isopleth taken at 0.5 (wt pct) Mo shown in Figure 8(b) illustrates that at temperatures below ~770 °C, the possibility of

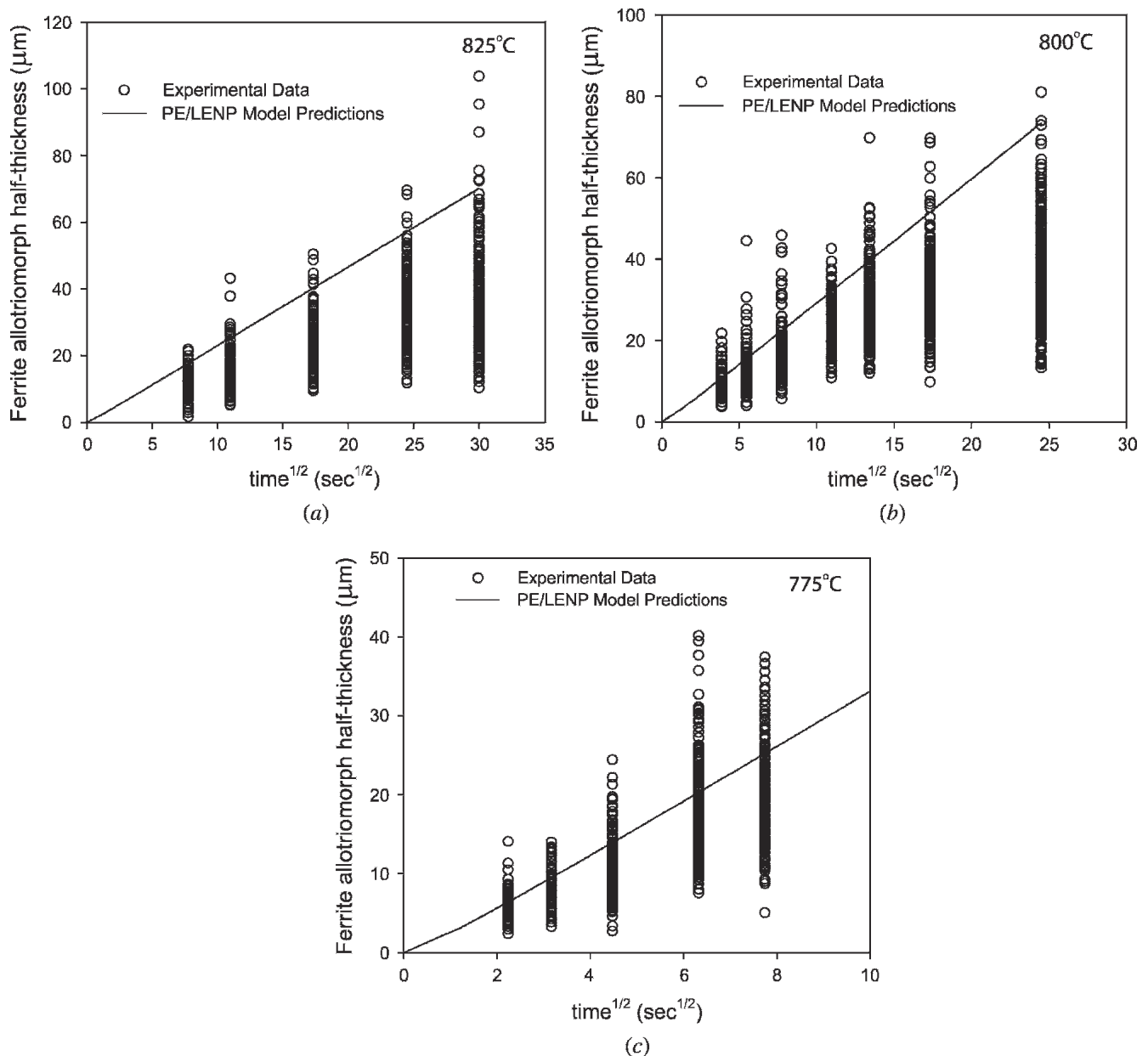


Fig. 9—Experimentally observed ferrite growth kinetics in an Fe-0.1C-0.1Mo (wt pct) alloy transformed at (a) 825 °C, (b) 800 °C, and (c) 775 °C. Each circle corresponds to an individual experimental thickness measurement. Between 100 and 300 measurements were made for each time at each temperature. The results are compared with the predictions of the PE/LENP model.

KSI-carbide formation arises. These considerations then allow us to identify experimental conditions where no carbides are expected and where the growth kinetics predicted by the LENP and PE models is near identical. In these cases, if a deviation is observed between the experimental ferrite growth kinetics and the PE/LENP model predictions, we can conclude (subject to the reliability of the kinetic and thermodynamic data) that an additional dissipation associated with the Mo interaction with the migrating interface exists.

We have measured the ferrite growth kinetics using both the traditional isothermal experiments and the decarburization experiments. An Fe-0.1C-0.1Mo (wt pct) alloy was used for the traditional isothermal experiments, and the

ferrite allotriomorph thicknesses as a function of time at 825 °C, 800 °C, and 775 °C are plotted in Figure 9. The experimental data are very scattered and the scatter increases with longer durations, suggesting an important role of the stochastic events associated with the nucleation step. For each temperature, the trend in the overall kinetics is described by the diffusion model (whose predictions lie within the experimental scatter), with a tendency of the model to overestimate the average of the experimental results as the temperature is increased. For these experiments, the scatter inherent in the experimental data precludes a critical test of the presence of solute drag.

The corresponding decarburization experiments, performed with a Fe-0.54C-0.51 (wt pct) alloy, allow the

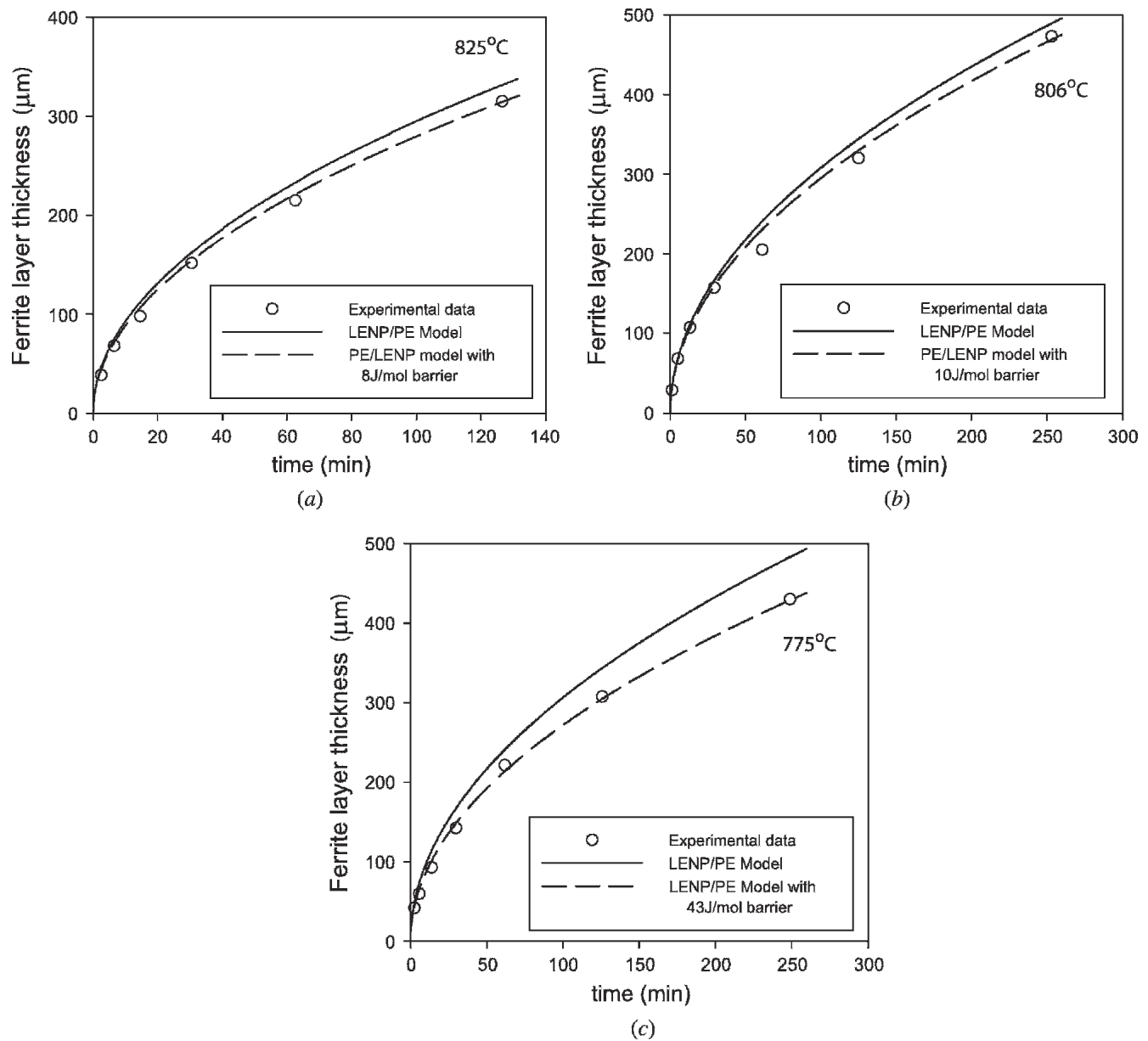


Fig. 10—Ferrite layer thickening kinetics observed in decarburization experiments of a Fe-0.54C-0.51Mo (wt pct) alloy at (a) 825 °C, (b) 806 °C, and (c) 775 °C. In each case, the thickening kinetics predicted assuming LENP/PE interfacial conditions are included for comparison. The experimental data are slightly slower than the model predictions. LENP/PE model predictions assuming additional barriers to growth of 8 J/mol at 825 °C, 10 J/mol at 806 °C, and 43 J/mol at 775 °C are also included and show excellent agreement with the experimental observations.

Table II. Estimation of the Additional Energy Dissipation Necessary to Bring the Experimental Observations into Agreement with the LENP/PE Model Predictions for an Fe-0.54-0.51Mo (Weight Percent) Alloy Transformed Under Decarburization Conditions

Treatment Temperature (°C)	Estimated Dissipation (J/mol)
825	8
806	10
775	43

determination of the ferrite growth kinetics much more precisely. The remarkable regularity of the ferrite layer is shown in Figure 2(b). The experimental ferrite growth kinetics in the decarburization experiments performed at 825 °C, 806 °C, and 775 °C are compared with the C diffusion model assuming LENP/PE conditions in Figure 10. The model predictions are slightly faster than the experimental observations. Remembering the accuracy of the predictions in the case of the Fe-C-Ni system (Figure 5), this discrepancy between the model and the experimental data are meaningful and reproducible. The difference between the results of the LENP/PE model and the experimental observations becomes larger as the temperature is lowered, as is expected from a solute drag effect. We can take these experimental results as unambiguous proof that there exists an extra dissipation mechanism involved in the austenite-to-ferrite transformation in this alloy. Instead of entering into the details of the different solute drag models, which require as inputs interfacial parameters that are not known with any certainty, we have instead estimated the magnitude of this dissipation by shifting the free energy curve of the ferrite up until agreement is obtained between the experimental and calculated growth kinetics. The magnitudes of the dissipations are listed in Table II, and they increase with decreasing temperature.

VI. CONCLUSIONS

A classification scheme for the effect of alloying elements on the growth of ferrite from austenite has been proposed based on the interactions between X, C, and the moving boundary. This classification resembles that listed by Aaronson *et al.*^[10] and used for their interpretation of the effect of X on ferrite growth in Fe-C-X systems. In the Fe-C-Ni system, in which the interactions are expected to be weak, all of the experimental observations can be quantitatively described using a C diffusion limited model provided the interfacial conditions are allowed to evolve from PE to LENP during growth. The kinetics of the Ni spike development associated with the LENP state depends on the interface velocity. No further dissipations associated with Ni interactions with the interface are required to explain the experimental data. In contrast, in the Fe-C-Mo system, where the interactions between Mo, C, and the interface are expected to be stronger, we have shown unambiguously an additional effect due to Mo interaction with the boundary. This can be interpreted as a solute drag effect.

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