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Formation of austenite from a ferrite–pearlite microstructure during intercritical annealing

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The formation of austenite from a coarse grained ferrite–pearlite microstructure during intercritical annealing was studied. According to the variation of the microhardness values in ferrite and austenite with intercritical annealing time, austenite formation was classified into four stages: (a) austenite growth into pearlite at a slower rate than the dissolution rate of pearlite, (b) subsequent growth of austenite into pearlite and formation of thin film type austenite at ferrite grain boundaries, (c) growth of austenite into ferrite, (d) equilibrium of ferrite and austenite. In particular, plate-like austenite was observed. The experimental results indicated that the growth process of this type austenite was controlled by carbon diffusion in austenite.

1. Introduction

Dual phase (ferrite–martensite) sheet steels are produced by annealing low carbon steels in the intercritical temperature range to produce ferrite–austenite mixtures, followed by accelerated cooling to transform the austenite phase into martensite [1]. Because the tensile strength and ductility of dual phase steels are primarily dependent on the amount, distribution, and carbon content of the martensite phase [2], an understanding of the formation of austenite during intercritical annealing of these steels is very important. However, current knowledge about this reaction is that it is a complex, sequential process, particularly in the case of initial microstructures composed of pearlite and ferrite [3–5].

Speich *et al.* [4] delineated the process of intercritical austenitization in such steels into three distinct stages: (1) very rapid growth of austenite into pearlite until pearlite dissolution is complete, (2) slower growth of austenite into ferrite, (3) very slow final equilibrium of ferrite and austenite. The nucleation of austenite seems to occur rather easily at carbide particles of ferrite–ferrite boundaries [3] and in the pearlite colonies [3, 4]. The growth of austenite into ferrite is diffusion controlled and either carbon [3] or substitutional partitioning [4] between ferrite and austenite has been proposed to be the critical step in the reaction.

However, very little quantitative information is available about the changes of the characteristics of ferrite and austenite (martensite) with intercritical condition, as well as the microstructural evolution of the early stage of austenite formation during intercritical annealing.

The purpose of the present work is to elucidate the growth process of austenite through the characteristic change of ferrite and austenite with the intercritical annealing condition.

2. Experimental procedure

The steel used in this study was prepared as a 22.6 kg air induction melt. The chemical composition (in wt %) of the steel is: 0.13C, 0.63Si, 1.42Mn, 0.16Mo, 0.005P, and 0.0068S. The ingot was hot forged, hot rolled, and cold rolled

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to 1.5 mm thickness sheet. In order to obtain a coarse-grained ferrite–pearlite starting structure, the sheet specimens were vacuum sealed in a quartz tube and annealed for 2 h at 1200° C, followed by furnace cooling to room temperature. These specimens were intercritically annealed under a flowing argon atmosphere in a vertical tube furnace. Intercritical annealing was carried out at 780 and 830° C for up to 2.5 h.

On water quenching the intercritically annealed specimens, martensite formed in the austenite grains available at the annealing temperature. The amount of martensite formed in each specimen was considered to be equal to that of austenite; in the present work, transformation strains and the existence of retained austenite which could not be detected by microscopy are ignored. Reported data [3] with similar steels indicated the volume fraction of retained austenite at the level of 3%. Such a figure would be irrelevant for the present work.

For optical microscopic study, prepared specimens were etched by using two types of etchants: nital 2% to reveal the ferritic boundaries, and Le Pera's etch [6] to disclose the martensite–ferrite boundaries and the non-martensitic quench product. The volume per cent of each phase was obtained by point counting. The change of the characteristics of ferrite and austenite with annealing time at the given intercritical temperatures was evaluated by measuring microhardness (Vickers) with a 50 g load on both phases.

Specimens for transmission electron microscopy were obtained from 10 mm × 10 mm sections of the intercritically annealed specimen and surface ground to 0.15 mm thickness using a flood cooling method. The ground pieces were chemically thinned to 0.02 mm in a solution containing 5% HF in H_2O_2 (30% concentration). Sample discs of 3 mm diameter were jet polished in a solution containing 65% butanol–30% methanol–5% perchloric acid maintained at − 15° C. Prepared specimens were examined by means of JEM 100C with an accelerating voltage of 100 kV.

3. Results and discussion

The starting microstructure which consisted of a coarse-grained ferrite–pearlite aggregate is shown in Fig. 1. The volume per cent pearlite was about 16%.

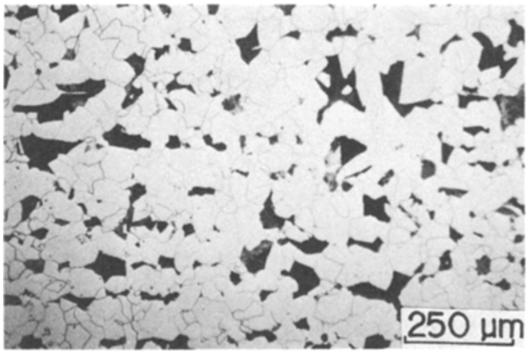


Figure 1 Optical micrograph showing a coarse-grained ferrite–pearlite microstructure (nital etch).

The variations of the microhardness values of ferrite and austenite and the volume per cent austenite with intercritical annealing time at 780° C are plotted in Fig. 2. It is interesting that the hardness value of ferrite has the maximum value at 270 sec. If hardness values of ferrite and austenite are assumed to be determined primarily by the carbon content in each phase, the process of austenite formation during intercritical annealing can be divided into four stages according to the trends of the measured hardness values of both phases.

Stage 1 is dissolution of the pearlite to form a high-carbon austenite phase. Austenite nucleates preferentially at pearlite colony boundaries [3–5] shown in the areas marked A in Fig. 3a and at the ferrite–pearlite interface [4] shown in the areas marked B in Fig. 3a. Fig. 3b shows that the dissolution of pearlite consists of gradual thinning of pearlitic cementite and spheroidization of the thinned pearlitic cementite.

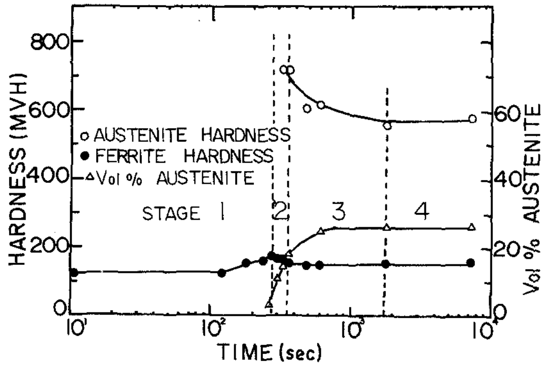
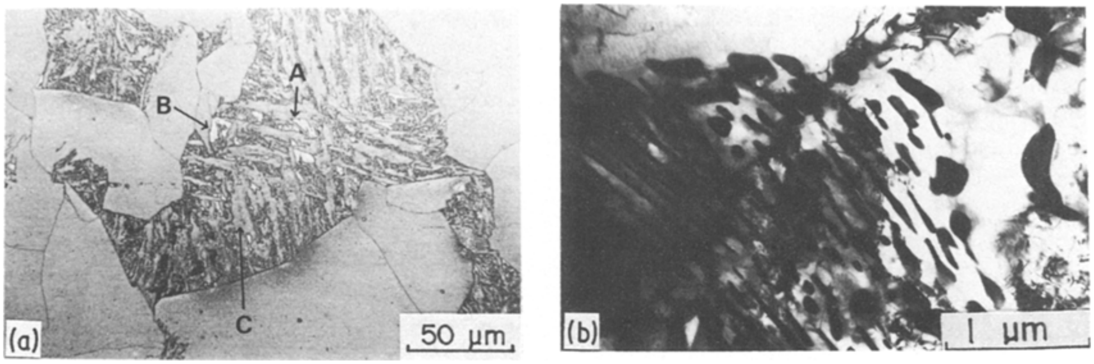


Figure 2 Plots of hardness values of ferrite and austenite and vol % austenite for specimens intercritically annealed at 780° C for different times. The three vertical broken lines indicate the boundaries of each stage of austenite formation.



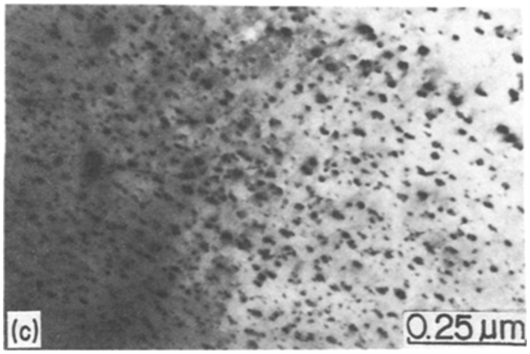


Figure 3 Micrographs representing microstructures intercritically annealed at 780° C for 270 sec: (a) optical micrograph showing pearlite dissolution and austenite formation in a pearlite grain (Le Pera's etchant; undissolved pearlite appears dark, austenite appears white, and ferrite grey); (b) transmission electron micrograph showing dissolution of pearlitic cementite and spheroidization; (c) transmission electron micrograph showing fine precipitates in ferrite of the periphery of pearlite grain.

At this stage, however, dissolved pearlite regions transformed only partially to austenite, consequently leading to the formation of ferrite in the regions of the dissolved pearlite remaining, without transforming to austenite, as shown in the areas marked C in Fig. 3d. This fact indicates that the dissolution rate of pearlite is faster than the growth rate of austenite. It is considered that this behaviour might result in the appearance of a peak value in ferrite hardness as a result of the movement of carbon from the dissolving pearlitic cementite to the ferrite region surrounding the pearlite grain. A number of fine carbide-like precipitates in the ferrite surrounding the pearlite shown in Fig. 3c may demonstrate this. On the contrary, these precipitates were observed in lesser amounts in ferrite with a low hardness value (Fig. 4). These fine precipitates appear to be precipitated on quenching from supersaturated ferrite in carbon.

Therefore, in Stage 1, the carbon from the dissolving pearlitic cementite seems to move simultaneously into the growing austenite, adjacent pearlitic ferrite, and ferrite surrounding pearlite. Of course, the growth rate of austenite in this stage is controlled primarily by the rate of carbon diffusion in austenite [4].

Stage 2 is characterized by the high hardness level of austenite and the gradual decrease in ferrite hardness. Microtwins in martensite substructure shown in Fig. 5a may ensure the high hardness level of austenite. Subsequent growth of this austenite into undissolved pearlite or supersaturated ferrite (originally pearlitic region) constitutes a second stage (Figs. 5b and c).

At the end of the second stage (Fig. 5c), all the pearlitic region was nearly displaced by austenite and the amount of austenite was approximately equal to that of pearlite which existed in the starting microstructure. The ferrite–austenite interface revealed planar boundaries similar to

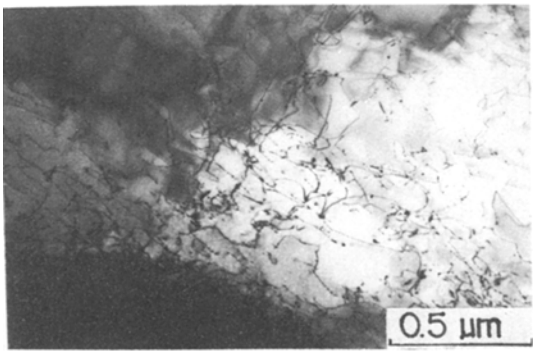


Figure 4 Transmission electron micrograph representing ferrite region of a specimen intercritically annealed at 780° C for 7200 sec.

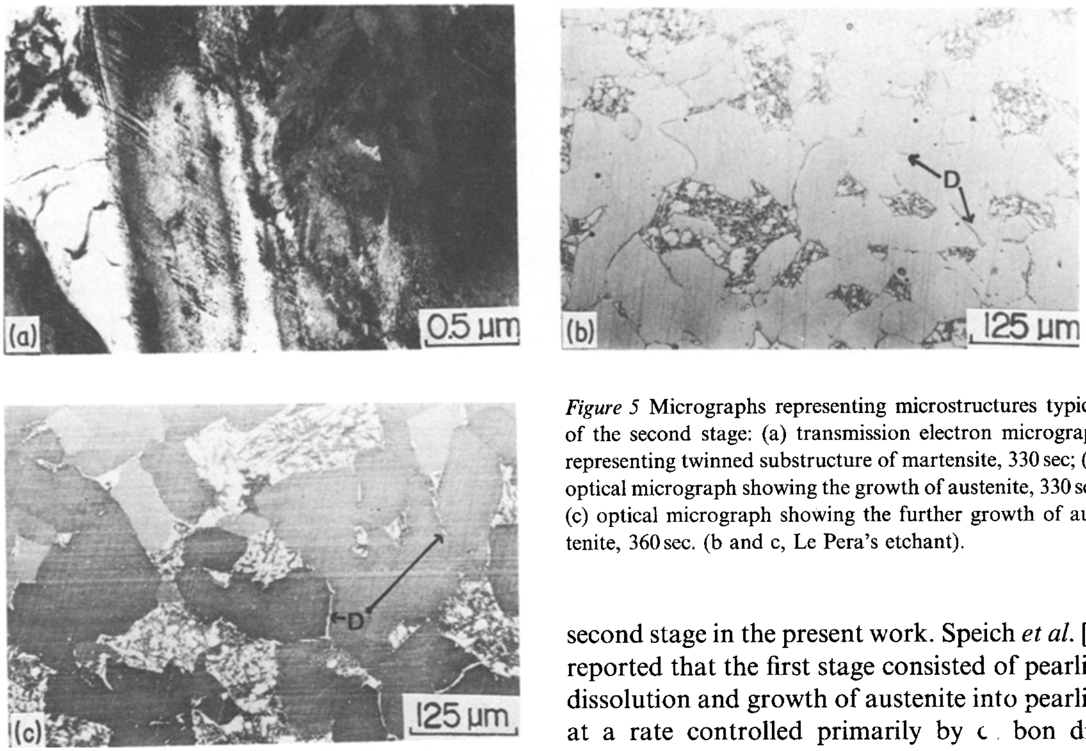


Figure 5 Micrographs representing microstructures typical of the second stage: (a) transmission electron micrograph representing twinned substructure of martensite, 330 sec; (b) optical micrograph showing the growth of austenite, 330 sec; (c) optical micrograph showing the further growth of austenite, 360 sec. (b and c, Le Pera's etchant).

the ferrite–pearlite interface of the starting microstructure.

The point of interest in this stage is the appearance of thin-film type austenite at the ferrite grain boundaries independent of austenite formed in the pearlitic region, as shown in the areas marked D in Fig. 5b and c. It is considered that the appearance of this thin-film type austenite may be related to the decrease in hardness value of ferrite.

Therefore, the growth process of austenite in Stage 2 can be divided into two types. The first type is the growth process of austenite in pearlitic region which is controlled by the diffusion rate in austenite of carbon moved from the adjacent dissolving pearlitic cementite. The second type is the growth process of thin-film type austenite formed at ferrite grain boundaries. The formation of thin-film type austenite is possible when carbon atoms of the supersaturated ferrite surrounding the pearlite grain diffuse towards ferrite grain boundaries, thus satisfying the carbon content for the formation of the austenite phase. Of course, the first type of growth process may affect the second type of growth to some degree, and vice versa.

The first stage reported by Speich *et al.* [4] corresponds approximately to the first stage plus second stage in the present work. Speich *et al.* [4] reported that the first stage consisted of pearlite dissolution and growth of austenite into pearlite at a rate controlled primarily by carbon diffusion in the austenite with the diffusion path lying along the pearlite–austenite interface.

However, there is a distinct difference between the growth mechanism reported by Speich *et al.* [4] and that of the present work in that the hardness peak value in ferrite appeared and could affect the growth process of the thin film type austenite.

At the end of the second stage, a high carbon austenite has been generated which, however, is not in equilibrium with the ferrite. Subsequent growth of this austenite into ferrite constitutes a third stage (Stage 3) (Fig. 6). In this stage, because the hardness value of austenite begins to

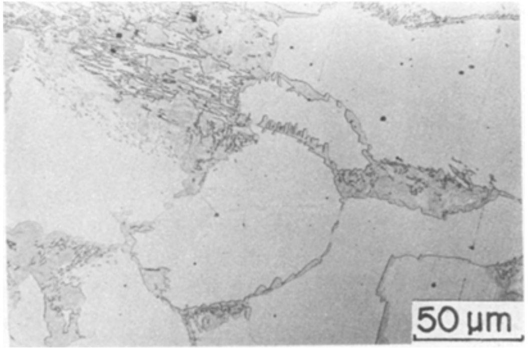
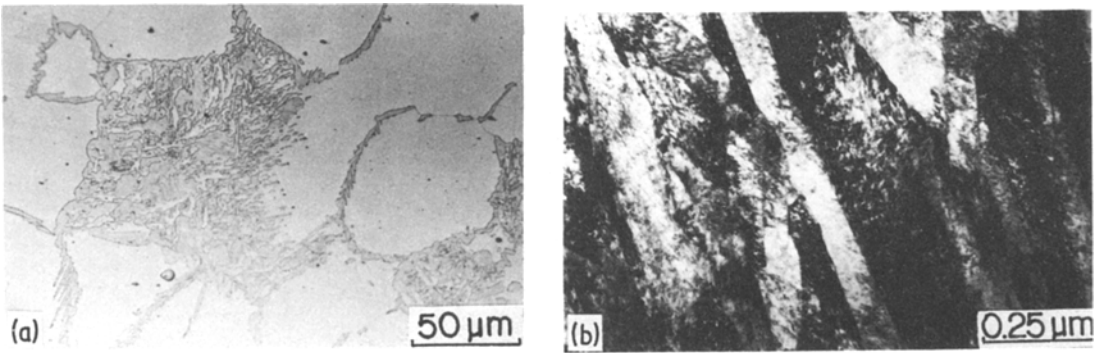


Figure 6 Optical micrograph representing microstructure intercritically annealed at 780° C for 1800 sec (nital etch).



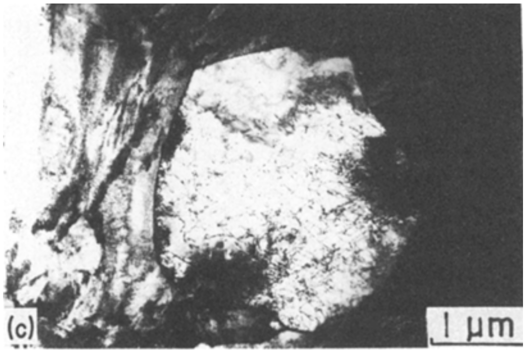


Figure 7 Micrographs representing microstructures intercritically annealed at 780° C for 7200 sec: (a) optical micrograph (nital etch), (b) transmission electron micrograph showing dislocated martensite, (c) transmission electron micrograph showing ferrite remaining in austenite even after annealing for 7200 sec.

decrease while that of ferrite already remains at a constant low value, it is clear that the growth of the austenite may be controlled by carbon diffusion from austenite to ferrite. This conclusion can apparently support the second stage reported by Speich *et al.* [4].

Another microstructural feature observed is that the ferrite–austenite interface changes to a curved boundary with small curvature. This change may result from the fact that the growth of austenite is favourable through the effective diffusion of carbon from austenite to ferrite when compared to the planar boundary.

The final stage (Stage 4) is characterized by the constant low hardness value of ferrite and austenite, and the constant volume per cent austenite. Therefore, the growth process of austenite in this stage may be related not to the diffusion of carbon but to that of a substitutional alloying element in austenite as reported previously [4, 5]. As a result of very slow diffusion of substitutional alloying elements, the increase in volume per cent austenite of the final stage was not observed even in the longest annealing time, 2 h, in the present work. The corresponding microstructure is shown in Fig. 7a. The substructure of martensite in Stage 4 consisted of dislocated laths, indicating low hardness value of austenite (Fig. 7b).

The point of interest is that pearlitic regions in the starting microstructure are not completely displaced by austenite even in the longest annealing time selected in this work, leading to them remaining locally in the form of ferrite as shown in Figs. 7a and c. This structural feature is believed to play an important role in the decomposition process of austenite on cooling after intercritical annealing [7].

Fig. 8 shows the variations of the microhardness values of ferrite and austenite, and the volume per cent austenite with intercritical annealing time at 830° C. The growth process of austenite at 830° C was also similar to that at 780° C and could be also divided into four stages. However, the first and second stage at 830° C are complete in about 30 and 20 sec, respectively. This behaviour indicates more rapid growth of austenite as a consequence of the more rapid diffusion of carbon at the higher temperature.

Figs. 9a to d show typical microstructures obtained in each stage of the austenite growth process at 830° C. Mean hardness values of ferrite and austenite and volume per cent austenite obtained in the final stage for the two temperatures are given in Table I. The results in Table I can be well illustrated by the fact that the higher annealing temperature, the lower is the carbon content of austenite and ferrite.

Another point of interest observed in the

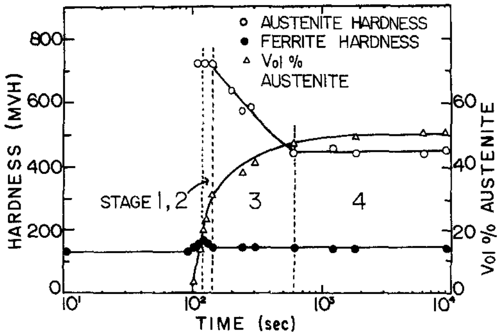


Figure 8 Plots of hardness values of ferrite and austenite and vol % austenite for specimens intercritically annealed at 830° C for different times. The three vertical broken lines indicate the boundaries of each stage of austenite formation.

TABLE I Vol % austenite and hardness values of ferrite and austenite obtained in the final stage

Annealing temperature (° C)	Vol % austenite	MVH (ferrite)	MVH (austenite)
780	26	152	570
830	50	140	440

present work is the appearance of plate-like austenite which grows along certain well-defined matrix planes from austenite formed in the pearlite region as well as from the thin-film type austenite formed at the ferrite grain boundaries. One characteristic feature observed for this type of austenite is that the higher annealing temperature and the longer annealing time lead to the more remarkable growth of plate-like austenite. The other feature is that growth of this plate-like austenite is most notable in the third stage at both temperatures. From these observed results, it may be concluded that the growth of plate-like austenite may be primarily controlled by carbon diffusion from austenite to ferrite and that carbon diffusion in austenite is the rate controlling process.

4. Conclusions

Austenite was formed by nucleation and growth processes under the experimental conditions used in this study. The growth process of austenite was characteristically divided into four stages according to the hardness variations of

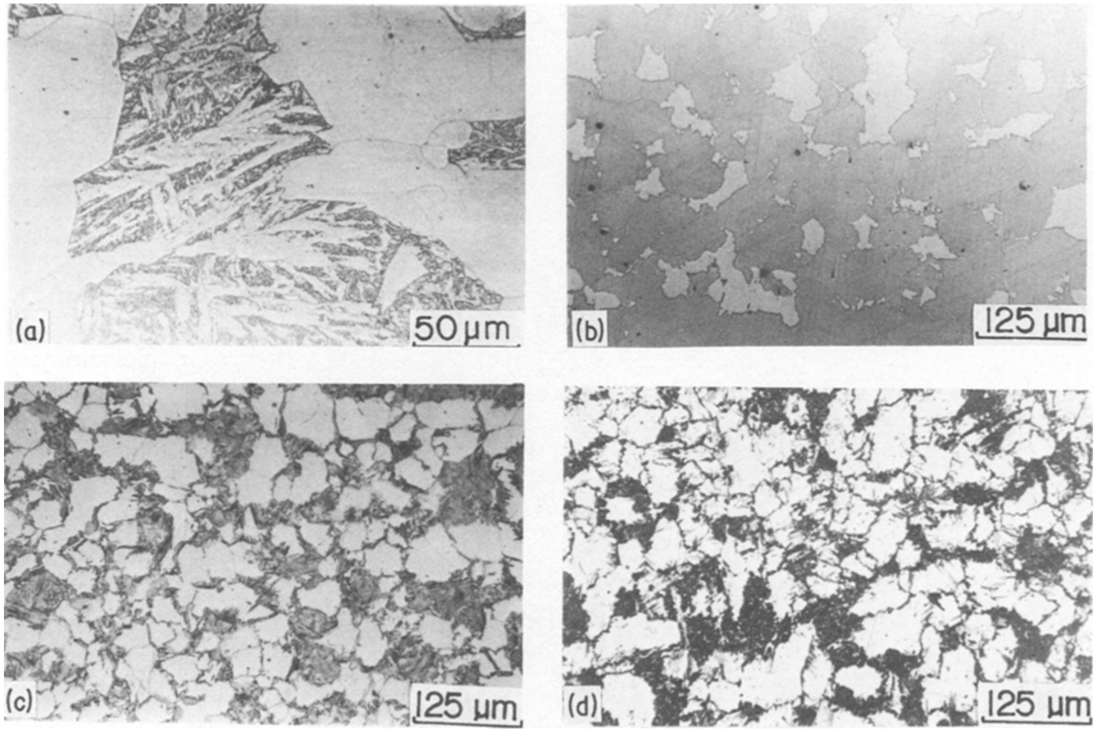


Figure 9 Optical micrographs showing typical microstructures obtained in each stage for austenite growth at 830° C: (a) 100 sec (Le Pera's etchant); (b) 130 sec (Le Pera's etchant); (c) 600 sec (nital etch); (d) 9000 sec (nital etch).

ferrite and austenite and volume per cent austenite with intercritical annealing time. In particular, the variation in ferrite hardness observed in the early stage of austenite formation affected the growth process of thin-film type austenite. Plate-like austenite was observed at both temperatures and the growth process of this type austenite also seemed to be controlled by carbon diffusion in austenite.

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