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Research on abnormal grain growth in reversely transformed austenite structure

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Research on abnormal grain growth in reversely transformed
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ABSTRACT

Control of austenite grain structure is of great importance in heat treatment process, because the prior austenite grain structure entirely affects the size and morphology of the final microstructure, and thus to largely determine the final mechanical properties of steels. The present study mainly focuses on the abnormal grain growth (AGG) of reversely transformed austenite in 0.2 mass% carbon steel and attempts to control it. The AGG of austenite which leads to extraordinary coarse austenite grains and bimodal grain size distributions degrades final mechanical properties of steels, because an initial variation in prior austenite grain size can result in a coarse and inhomogeneous final grain structures. Hence, AGG in austenite grain structure during the heat treatment processes is very harmful, which is quite worth to be investigated.

AGG has been extensively investigated because of both its technological relevance in the steel industry and scientific interest. In spite of the considerable research effort, there are still some important issues that remain to be resolved. For example, it is unclear whether the initial microstructure of steel, such as ferrite/pearlite (F/P) banded and non-banded structure affect the austenite abnormal grain coarsening or not. Since the banded structure with a great inhomogeneity in alloy elements distribution, it may have a great effect on the austenite abnormal grain coarsening behavior. On the other hand, it is unclear how to control the AGG of austenite during the heat treatment process. Therefore, the purpose of the present study is to investigate the abnormal grain coarsening behavior of reversely transformed austenite and attempts to control it.

The contents of this thesis are described as follows. In Chapter 1, the research background is illustrated. By describing some steel manufacturing processes, the importance of refinement of austenite grains during reheating is emphasized. Moreover, it is explained that the occurrence of AGG of austenite grains degrades the final mechanical properties of steel. The motivation and purpose of present study are also illustrated. Chapter 2 is the review of the literatures related to the AGG of reversely transformed austenite grain structures. The reported mechanisms for the occurrence of AGG and the effects of cold deformation on the austenite grain structure and precipitation are summarized.

In Chapter 3, the grain growth behavior of austenite reversely-transformed from F/P banded and non-banded steels has been studied systematically. It was found that the grain-coarsening temperature, T_c , (the temperature at which abnormal grain growth occurs) of the initially banded F/P steel is quite low compared with that of the non-banded steel. In the F/P banded steel, it was found that the abnormal grains originate from the former ferrite region. The occurrence of AGG is

essentially attributable not to the austenite nucleation process during heating but to the grain growth process after the completion of austenizing. It was proposed that the lowered T_c in the F/P banded steel is due to the non-uniform pinning-effect of AlN precipitates between former ferrite and pearlite regions. In chapter 4, the effect of cold deformation on AGG of austenite in F/P banded steel has been investigated. It was found that the low T_c in the F/P banded steel can be increased significantly by applying cold deformation prior to austenitizing. The severity of AGG above the T_c is also largely reduced by the cold deformation. This is attributed to the fine and uniformly distributed AlN precipitates caused by the cold deformation.

In Chapter 5, the effects of cold deformation on AGG of austenite in non-banded steel have been studied. It was found that the effect of cold deformation on AGG of austenite depends on the initial state and distribution of AlN precipitates. When the non-banded steel with aluminum and nitrogen in solution, cold deformation reduces the AGG temperature-range (a temperature range of occurrence of AGG) of austenite. While the non-banded steels are subjected to a long time subcritical annealing treatment making the AlN fully precipitated and growth, the occurrence of AGG can be completely inhibited by applying cold deformation. This can be explained by the size and spatial distributions of the precipitates affected by the cold deformation.

In Chapter 6, the local plastic strain distribution and its effect on the on the austenite grain growth has been studied. It was found that large macroscopic plastic strains with high strain rates lead to steep and narrow distribution profile of local plastic strain. The peak of the local plastic strain profile does not appear at the deformed surface but immediately beneath the surface. The deformation at high strain rates is favorable to localize the plastic strain in the vicinity of the surface. On the other hand, the deformation at lower strain rates makes plastically deformed zone wider. The local plastic strain distribution has a strong effect on the austenite grain growth. The austenite grain structure near the surface can be effectively controlled by the local plastic strain distribution. In Chapter 7, the misorientation manifestations in chemical etching contrast has been studied in cold deformed iron. The chemical etching is performed by using nital. It was found that the chemical etching contrast strongly reflects the crystallographic orientation. The gradual change in chemical etching contrast inside the individual deformed grains gives information of both the misorientation and local plastic strain within the grains. This method can provide an easy and alternative way to qualitatively understand the misorientation and local plastic strain distributions in the microstructures.

Finally, the important findings in this study are summarized in Chapter 8.

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Chapter 1

Introduction

Steels are defined as iron-based alloys, with small amounts of carbon and other chemical elements. In particular, carbon steels are defined as alloys with a maximum of 2.11 wt pct C, a maximum of 1.65 wt pct Mn, a maximum of 0.60 wt pct Si, and a maximum of 0.60 wt pct Cu. Maxima in S and P are also specified, but no minimum content of other elements is listed [1]. Carbon steels have been used as the most important structural materials because of their superior mechanical properties and competitive price. Over the years, numerous studies have been carried out to improve the mechanical properties of steels in order to meet the requirements of more and more stringent applications. The mechanical properties of steels can be tailored by changing the composition and/or the microstructure. Microstructure is the most important materials parameter which can bring about drastic change in mechanical properties of steels. It is well known that grain refinement is the only method to improve both strength and toughness simultaneously.

During heat treatment processes, the controlling of austenite grain structure is important because it entirely affects the size and morphology of the final microstructure, and thus to largely determine the final mechanical properties of steels. The present study mainly focuses on the abnormal grain growth (AGG) of reversely transformed austenite and the effect of cold deformation on it. The AGG of austenite which leads to extraordinary coarse austenite grains and bimodal grain distributions degrades final mechanical properties of steels, because an initial variation in prior austenite grain size can result in a coarse and inhomogeneous final grain structures.

In this chapter, the steel manufacturing processes are generally described and the methods used to refine the austenite grain structure are reviewed. Moreover, the grain growth modes of austenite structure are summarized. Especially, the grain growth mode of AGG which is the main focus of this study is emphasized. At the last part, the purpose of this study is declared.

1.1. Steel manufacturing process

1.1.1 Continuous casting

Continuous casting of steel, as an industrialized method of solidification processing, has a relatively history of only about 50 years. In fact, the continuous casting ratio for the world steel industry, now approaching 90% of crude steel output, attained mere 4% in 1970 (Fig. 1.1) [2].

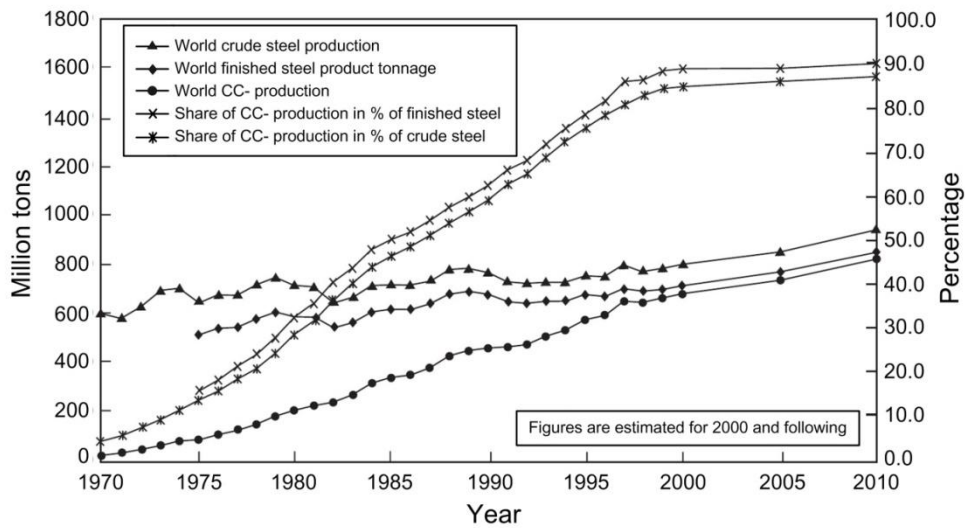


Fig. 1.1. Evolution of world steel production and share of continuous casting [2].

Figure 1.2 schematically presents the continuous casting process. Firstly, molten steel is poured from a ladle via a tundish into a copper mold, which is keeping oscillating and water cooled. The cast steel solidifies from the wall of the mold and forms a thin solidified shell near the wall. Then, it is continuously pulled out from the mold and is further cooled by the spraying water. Finally, the ingot was cut into slabs with fixed length.

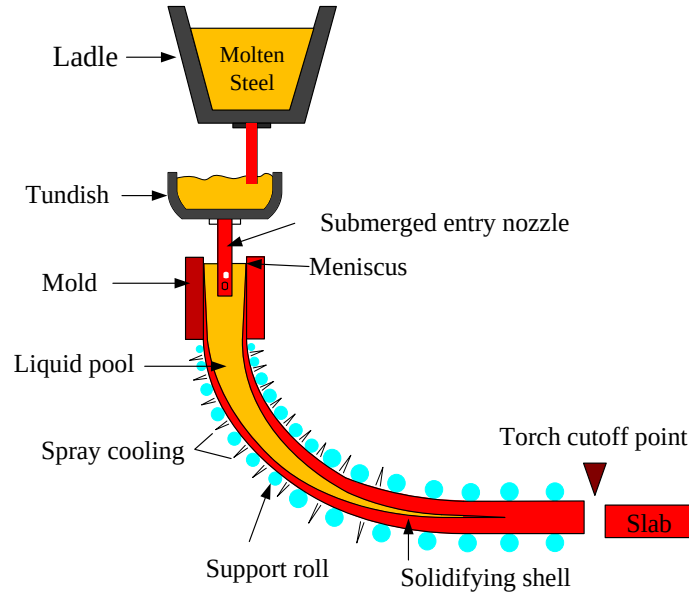


Fig. 1.2. Schematic illustrations of continuous casting process [2].

While liquid steel at the end of state-of-the-art continuous casting processes is essentially uniform in chemistry [3], solidification produces macroscopic and microscopic partitioning of chemical elements between parent liquid and growing solid crystals, producing nonuniformity in the distribution of chemical elements inherited in fully solidified as-cast products. This partitioning or segregation of chemical elements occurs on a macroscopic level at the centerline of continuously cast steel products, and at the tops and bottoms, as well as centerlines, of ingots. The effects of macroscopic segregation are outside of the scope of this study. On a microscopic scale, segregation occurs between dendrites throughout a solidified section, so called microsegregation.

Solute atom redistribution during dendritic solidification is driven by the equilibrium partitioning of chemical elements within the liquid-solid phase field. The two-phase field is defined as a function of temperature by liquidus and solidus curves of the equilibrium phase diagram. At any given temperature, the solute concentration of the solid may be designated as C_S and that of the liquid as C_L , as defined by a tie-line at that temperature. The redistribution or partitioning of solute is then defined as the equilibrium partition ratio, k , as [4]

$$k = C_S / C_L \quad (1.1)$$

With this parameter, solute redistribution in the solid C_S as a function of the weight fraction of solid in a volume element, f_s , is given by the Scheil equation as

$$C_S = kC_0(1 - f_s)^{k-1} \quad (1.2)$$

where C_0 is the initial alloy composition within the volume element [4]. Eq. (1.2) accurately demonstrates solute enrichment in the solid as solidification proceeds.

Table I lists k values, assumed to be independent of temperature, of some elements commonly found in steels [5]. Solute elements with low values of k have the greatest tendency of segregate. Therefore, phosphorus has a very strong tendency to segregate during solidification. However, the amount of the element present is also a factor. Therefore, Mn, generally present in much higher concentrations than P, plays a more important role in segregation than P despite its higher value of k . For a 1.0 pct Mn steel, according to Eq. (1.1), Mn would vary from 0.70 pct at the beginning of solidification to 1.60 pct at the end of solidification [5].

Table 1 Equilibrium Partition Ratios for Various Elements in Steel [5]

Element	K
P	0.14
Nb	0.23
Cr	0.33
Mn	0.71
Ni	0.83

1.1.2 Cold Charge Rolling

In the above described conventional continuous casting processes, semi-products with a thickness more than 100 mm are fabricated by a continuous caster and air-cooled to room temperatures where ferrite and pearlite structure forms [6]. Then, during the subsequent hot rolling process, the cast slabs are reheated to the single austenite phase temperature region in a furnace and hot rolled to obtain final products, i.e. steel sheet. Figure 1.3 schematically shows a typical cold charge rolling process. The rolling process is of great significance in the accomplishment of desired fine final

microstructure as well as the adjustment of thickness of steel ingot.

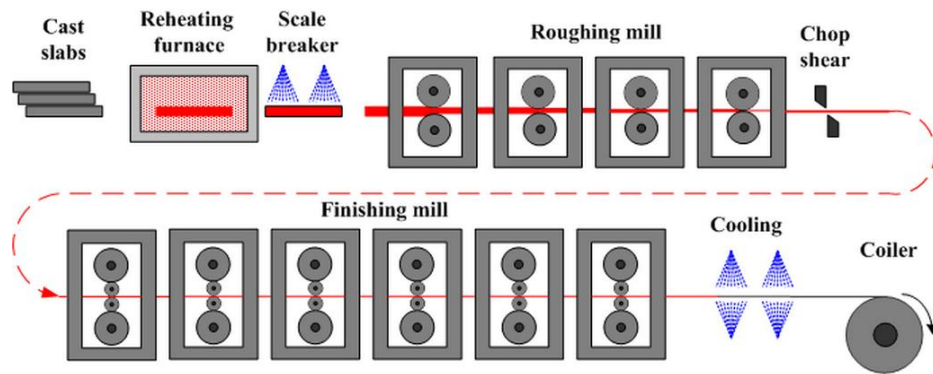


Fig. 1.3. Schematic illustrations of cold charge rolling process.

In the reheating process, the heated steel experiences the reverse transformation, i.e., ferrite-to-austenite transformation, as indicated by the red arrow in the Fe-C phase diagram as shown in Fig. 1.4. During the reversion process, the austenite phase nucleation and growth form the fine reversed austenite grain structure prior to the hot rolling.

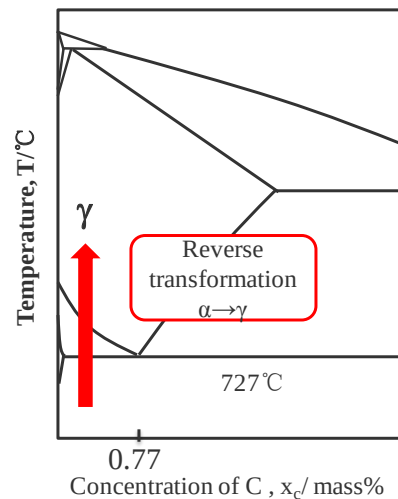


Fig. 1. 4. Fe-C phase diagram. Red arrow indicates the reverse transformation.

During the hot rolling, the occurrence of dynamic or static recrystallization (see Fig. 1.5 [7]) further refines the austenite grains. In the final cooling process, the

as-rolled austenite phase decomposes to low temperature structures, such as ferrite, pearlite or martensite depending on the cooling rate. It was reported that the above described interdendritic chemical segregation is modified, but not eliminated, by the typical industrial hot rolling processing [7]. The typical hot rolled microstructure of hypoeutectoid carbon steel is discussed in the next section.

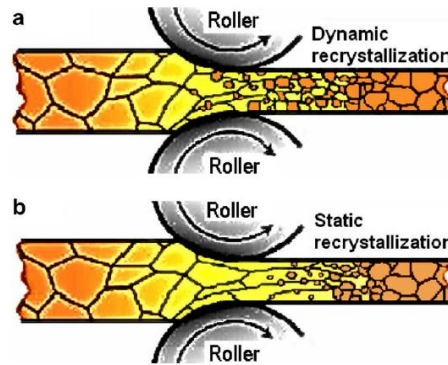


Fig. 1.5. Schematic illustrations of (a) dynamic and (b) static recrystallization during hot rolling process [7].

1.2. The microstructure of the hot rolled carbon steel

The ferrite/pearlite (F/P) banding is commonly observed in hot rolled low alloy steels [8-14], consisting of alternate layers of proeutectoid ferrite and pearlite. Fig. 1.6 shows an example of a typical F/P banded structure, with alternate layers of ferrite (the bright region as indicated by F) and pearlite (the dark region as indicated by P) banding running parallel to the rolling directions.

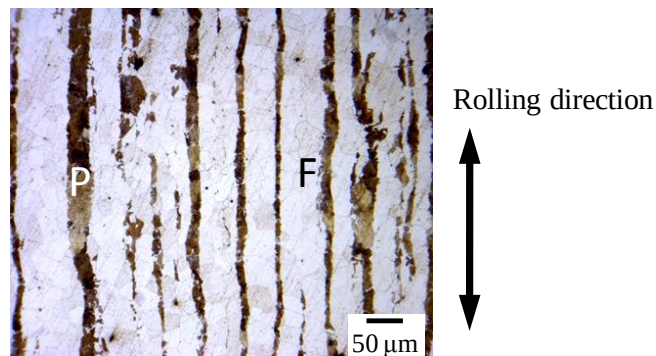


Fig. 1.6. Typical ferrite/ pearlite banded structure of the hot rolled low carbon steels found in the present study.

It was confirmed [15] that the banding in low-alloy steel is due to the interdendritic segregation of substitutional alloying elements. During solidification, alloy elements (e.g. silicon, manganese, phosphorous, sulfur, aluminum, and so on) are rejected from the primary crystal dendrites, which causes higher solute contents in the interdendritic regions and lower solute contents in the dendritic regions, as schematically shown in Fig. 1.7.

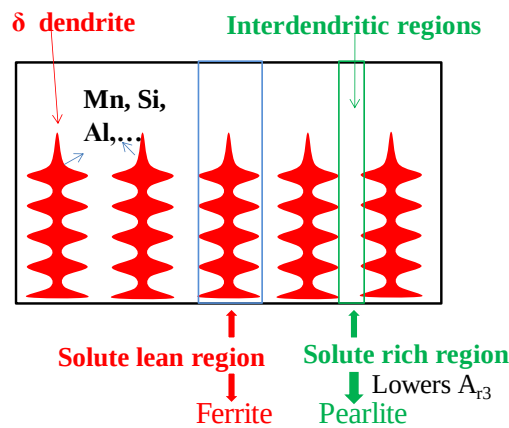


Fig. 1.7. Schematic illustrations of interdendritic segregation.

These regions of high and low solute contents are elongated into parallel bands during the subsequent hot rolling process. The decomposition behavior of austenite is different between the bands due to the difference in solute content between them. Because manganese, for example, lowers the austenite to ferrite transformation temperature, regions with lower manganese concentration transforms into ferrite at higher temperatures, while regions with higher manganese concentration remains as austenite at lower temperatures and finally transforms into pearlite, leading to the formation of banded microstructure with alternate layers of ferrite and pearlite [15]. Several investigations have shown manganese to be the alloy element most responsible for the development of microstructural banding in hypereutectoid steels [15-16].

As stated earlier, the primary cause of banding is substitutional element concentration gradients. However, cooling rate, austenite grain size, and austenitizing temperature also influence the severity of microstructural banding. Thompson and

Howell [15] investigated banding in 0.15 wt pct C, 1.49 wt pct Mn steel and concluded that the intensity of micro-structural banding increases as the cooling rate decreases during the austenite-to-ferrite transformation. The study showed that furnace cooling produces intense banding and air cooling yields poorly defined ferrite and pearlite bands. Increasing the cooling rate from the austenitic condition reduces the intensity of banding because it reduces the A_{r3} temperature differences of the segregated bands. They also demonstrated that ferrite-pearlite banding is completely eliminated at cooling rates greater than 5 /s [15]. Ferrite-pearlite banding also disappears when the austenite grain size exceeds the spacing of the segregated bands by a factor of 2 or 3 [15, 17]. In this case, the influence of the grain boundary as a preferred site for nucleation dominates the effect of the compositional gradients. However, Samuels [17] showed that banding eliminated by fast cooling or large austenite grains can reappear if the material is subsequently reaustenitized and slowly cooled.

Permanent elimination of microstructural banding can only be achieved by high temperature homogenization treatments that remove the underlying compositional gradients [18]. However, such treatments are often beyond the range of economic feasibility due to the sluggish diffusion rates of substitutional elements.

According to the above descriptions, the microstructural banding of hot rolled, low-alloy steels is associated with the chemical banding of substitutional alloying elements. In other words, microstructural banding is a manifestation of the segregation of substitutional alloying elements. This heterogeneity in substitutional alloying elements distribution may affect the reversed austenite grain growth during reheating.

1.3. Cold forging

Cold forging is widely utilized as a method of manufacturing in the industry since high volumes of near net shape components such as gears (as shown in Fig. 1.8 [20]), special fasteners, tools, shafts and bearing parts etc. can be produced with

sufficiently high dimensional accuracy to minimize or eliminate costly machining operations [19, 21]. Cold forged products are used in a variety of industries, including as moving parts and drive systems for the automotive, agri-machinery and power tool industries.



Fig. 1.8. Cold forged gears [20].

Although cold forged steel will satisfy the property requirements for many applications, some of the more highly stressed drive train components are carburized and hardened after cold forging to increase wear and fatigue resistance. Component processing in the latter case typically comprises an annealing to increase the cold shearability of the incoming bar stock, multiple cold forming and sizing operations with intermediate anneals, a single quench carburizing operation, and a low temperature temper.

This annealing process generally leads to the reverse transformation from ferrite to austenite and the subsequent austenite grain growth. It is well known that cold deformation has a strong effect on the kinetics of reverse transformation, thus the initial as-transformed austenite grain size. Cold deformation also has a great effect on the precipitation kinetics of second phase particles in steels.

1.4. Austenite grain structure during reheating

1.4.1 Refinement of reverted austenite grain structure

In the heat treatment process, the austenite grain size before cooling/quenching tremendously influences diffusive and diffusionless phase transformations,

precipitation, and mechanical properties such as strength, hardness, toughness, and ductility of the final structure. When austenite is transformed to a final structure such as ferrite, pearlite or martensite as shown in Fig. 1.9 [22], the initial austenitic condition, particularly the grain size, is important to develop the refined final microstructure and excellent mechanical properties of the steel. Therefore, it is quite important to control the austenite grain structure.

A plenty of researches have been made to get better understanding and controlling of the austenite grain size during austenitizing process of the steels over the past half a century [23–30].

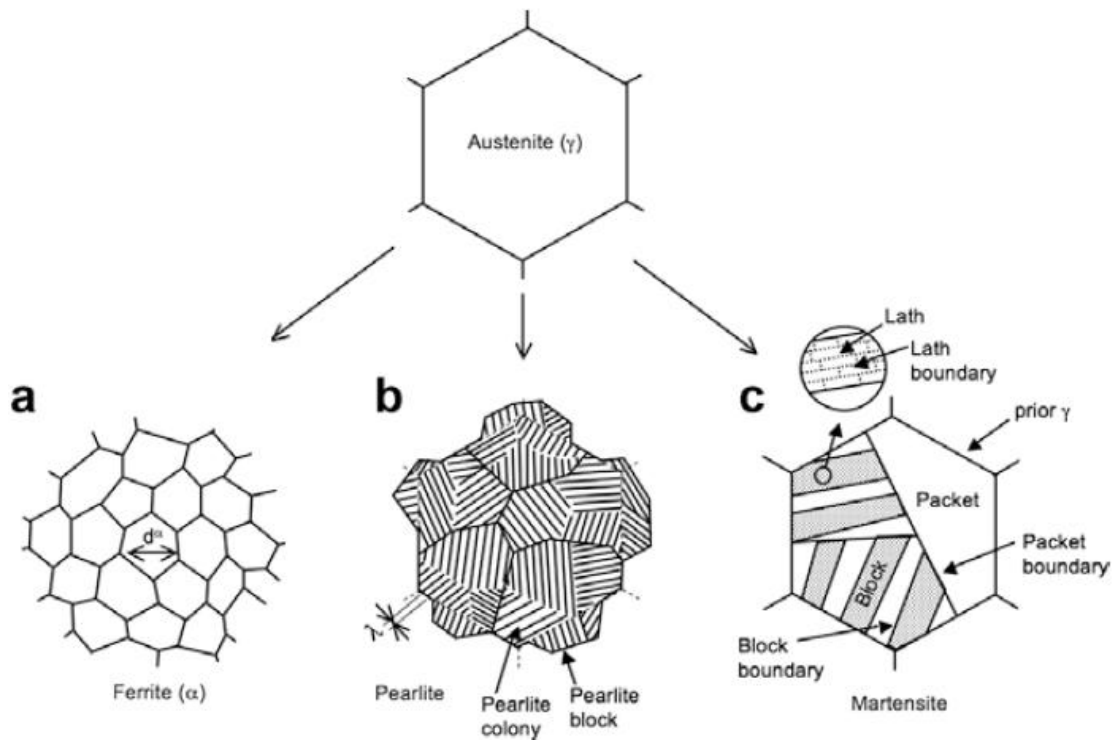


Fig. 1.9. Three typical microstructures obtained by phase transformation from austenite in steels. (a) Ferrite. (b) Pearlite. (c) Martensite [22].

In the conventional processes such as controlled rolling, recrystallization during hot deformation is used to refine austenite grain size, as described in Section 1.1.2. Refinement of austenite grain size was also studied by using other methods in low-carbon steels, such as repetition of martensite-to-austenite transformations through thermal cycling of rapid heating and quenching [31] and plastic deformation

of the mother phase prior to phase transformation [32-34].

Microalloying and appropriate thermo-mechanical processing are also used as an important austenite grain refinement technology of steels. Austenite grain growth can be inhibited by microalloying which causes the precipitation of second-phase particles in austenite [35]. Grain refinement was initially done with the addition of Al and N through the formation of AlN precipitates [36]. Later, other additives such as Ni, V, Ti, and Nb which also produces austenite grain refinement with the formation of carbides or nitrides [37-40].

1.4.2 Reverted austenite grain growth during reheating

The austenite grain size is fine just after the reverse transformation during heat-up to austenite region. However, the grain growth can easily occur especially in plain carbon steels to reduce the grain boundary free energy by thermally activated atomic processes. As alloying elements are added in the carbon steels, the grain growth rate usually decreases due to the solute dragging effect of the alloying elements segregated into austenite grain boundaries [41, 42]. If the alloying elements precipitate as carbides or nitrides in austenite, the precipitates also lower the grain growth rate by pinning the grain boundaries [43, 44].

Grain growth can occur in two modes: normal grain growth (NGG) and abnormal grain growth (AGG). The two modes have been studied since 1940s, employing theoretical and experimental approaches [45-55]. NGG is said to occur when the microstructure exhibits a uniform increase in grain size [56]. This type of growth is a steady state kinetic process characterized by time invariance of the normalized grain size distribution function $F(R/\bar{R})$. For NGG, the kinetic law is represented by [56]

$$\bar{R} = kt^n \quad (1.3)$$

with isothermal annealing, where $\bar{R}$ is the average grain size, t the time, and k is material and temperature related constant, and $n \leq 0.5$. Namely, NGG is characterized by a self-similar coarsening process of the microstructure.

The second type of AGG (also known as secondary recrystallization) is

characterized by the rapid increase of the largest grains at a rate much faster than the mean $\bar{R}$. During the AGG process a few large grains grow discontinuously and consume the neighboring matrix of smaller grains, leading to the development of extraordinarily large grains, perhaps up to tens of millimeters in size, mixed with fine residual matrix grains [57].

A typical example of a grain structures resulted from AGG is shown in Fig. 1.10 [58], which clearly abnormal coarse grains mixed with fine matrix grains. The mixed grain structures exhibits a bimodal grain size distributions, as shown in Fig. 1.11 [59].

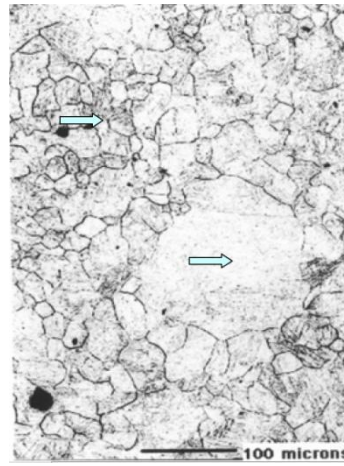


Fig. 1.10. Mixed austenite grain structure with abnormally large grains present along with fine austenite grains after 1100 °C reheating (for 1 hr.) of 0.1 C - 0.04 w % Nb microalloyed steel [58]. Arrows indicate both large and small grains.

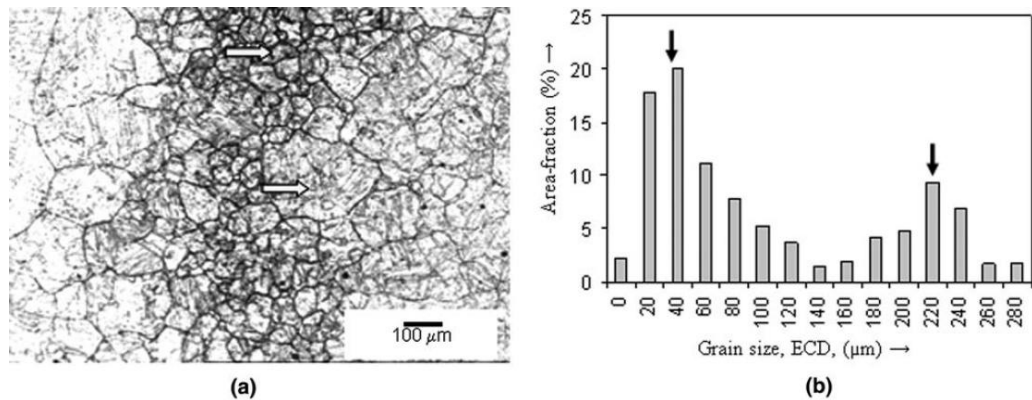


Fig. 1.11. Prior austenite grain structure in (a) slab 1-1150 showing coarse and fine grain regions (arrowed), and the corresponding grain size distribution; (b) showing bimodality (peaks indicated by arrows) [59].

For comparison, Fig. 1.12 schematically shows the grain growth process in both grain growth modes. As stated above, NGG leads to uniform grain structure, while AGG results in duplex grain structure.

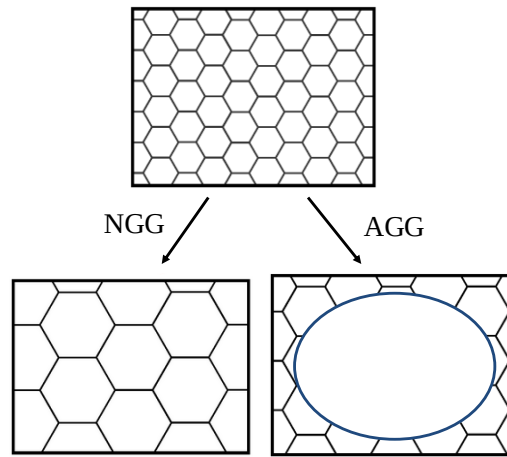


Fig. 1.12. Schematic illustrations of the grain growth process in both NGG and AGG grain growth modes

The inhomogeneous prior austenite grain size [60, 61], which affect the local recovery, recrystallization and phase transformations, hence, the final microstructure and mechanical properties of steels. An initial variation in prior austenite grain size can result in an inhomogeneous ferrite grain size distribution upon rolling [59]. In recent years, a variation in grain size, particularly a bimodal ferrite grain size distribution, has been observed in some Nb- and Nb-V-containing microalloyed steel plates, with abnormally large ferrite grains present in the matrix of smaller grains, in a typically-banded ferrite/pearlite microstructure [62, 63]. The bimodal grain size distribution is reported to cause a variation in mechanical properties, and is particularly significant for the cleavage fracture toughness (causing significant scatter) of the steels [64, 65]. Therefore, AGG in austenite grain structure during manufacturing processes is very harmful, which is quite worth to be investigated.

1.5. Purpose

As described above, to obtain steel products with excellent mechanical properties, the control of former austenite grain size during reheating is essential. It was widely reported that the mechanical properties of materials exhibiting a bimodal austenite

grain size distribution caused by AGG are degraded [66]. Therefore, it is quite important to investigate the AGG of reversely transformed austenite during the reheating process.

AGG has been extensively investigated because of both its technological relevance in the steel industry and scientific interest. In spite of the considerable research effort, there are still some issues that remain to be resolved. For example, it is not clear whether the initial microstructure of steel, such as F/P banded and non-banded structure affects the austenite abnormal grain coarsening or not. Since the banded structure with a great inhomogeneity in alloy elements distribution, it may have a great effect on the austenite abnormal grain coarsening behavior. On the other hand, since the occurrence of AGG of austenite grains degrades the final mechanical properties of the steel, it should be avoided during the steel manufacturing process. However, it is unclear how to control the AGG of austenite during the heat treatment process. Therefore, the purpose of the present study is to clarify the effects of initial microstructure on the abnormal grain coarsening behavior of reversely transformed austenite in 0.2 mass% carbon steels and attempts to control it.

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Chapter 2

Review of the abnormal grain growth of reversely transformed austenite in steels

2.1 Mechanisms of abnormal grain growth

Grain growth occurs by the growth of larger grains at the expense of smaller grains such that the material volume remains constant. The driving force for growth is the minimization of grain boundary energy and can be considered in terms of grain boundary curvature and grain angle. The grain boundary energy is reduced by the flattening of curved interfaces and the change in grain boundary and edge angles towards their equilibrium values [1]. Hence grains with convex curvature and less than equilibrium edge and corner angles tend to shrink, whereas concave and large-angled grains tend to grow.

As described in Chapter 1, abnormal grain growth (AGG) is the rapid growth of a few large grains, which is much faster than the matrix fine grains. This type of growth is a transient process which can be described in some cases by the kinetic model of Johnson and Mehl [2] and Avrami [3].

$$X = 1 - \exp[-g(t)] \quad (2.1)$$

where X is the volume fraction of abnormal grains, and $g(t)$ is a general function of time. The growth of individual abnormal grains into the matrix of stable normal grains has been characterized as exhibiting a linear increase in radius with time, with the growth velocity obeying an Arrhenius temperature dependence [4, 5].

The most complete theoretical treatment of AGG has been given by Hillert [6]. Hillert discusses AGG in the context of a mean field formalism, in which the driving force is derived solely from the decrease in total grain boundary energy. He distinguishes two ways in which AGG can occur. In the first process grain growth takes place in the absence of an inhibition mechanism, and primary recrystallization has resulted in a broad initial grain size distribution. Those recrystallized grains

having a size greater than $2\bar{R}$ ($\bar{R}$ is the mean grain size) in two dimensions or $9/4\bar{R}$ in three dimensions are predicted to exhibit abnormal growth. The second condition under which Hillert treats AGG is in the presence of particle dispersion. In this case he derives necessary (but not sufficient) requirements for initiation. He predicts that AGG can develop in a material if three conditions are simultaneously satisfied:

(1) Normal grain growth (NGG) cannot take place due to pinning by second phase particles.

(2) The microstructure has been pinned at a mean grain size such that the absolute value of the Zener back stress due to the particles is less than the absolute value of the average capillarity pressure.

(3) There is at least one grain with a size much larger than the mean such that the capillarity pressure associated with its boundary is less than the difference between the absolute value of the average capillary pressure and the absolute value of the Zener back stress.

Hillert suggests that these conditions might be established in the microstructure if NGG has proceeded to be pinned at a fixed number and size of particles, and then a time dependent decrease in the Zener back stress occurs due to a reduction in the number of particles (either by dissolution or ripening).

Hunderi and Ryum have also examined AGG by two separate procedures [7, 8]. In the first method they performed a computer experiment in which an array of spherical bubbles underwent grain growth in the presence of a Zener back stress [7]. They found complete pinning of the system for large values of the Zener drag, but indications of a direct transition to AGG for small values. They also considered the effect of Zener drag on grain growth by obtaining time dependent solutions to the Hillert mean field equations [8]. Starting with Hillert's normal grain size distribution, they numerically integrated the mean field equations to obtain the kinetics and the grain size distribution. In this case pinning was predicted for all values of the Zener drag and no transition to AGG occurred.

In summary, these treatments suggest that AGG can occur in one of three ways:

(1) If an abnormally large grain (greater than $2\bar{R}$ in two dimensions or $9/4\bar{R}$ in three dimensions) is introduced into the grain ensemble in a system free from grain growth restraint.

(2) If particle retardation of grain growth takes place but the Zener drag is small and the system has not been completely pinned.

(3) If the microstructure is completely pinned by particles but a time dependent decrease in Zener drag (through a reduction in the number of particles by dissolution or ripening) takes place.

Therefore, AGG requires NGG to be strongly impeded, with the exception of a few grains which act as nuclei for AGG [9, 10]. Inhibition of NGG is attributed to a number of mechanisms:

(1) Particle pinning of boundaries.

(2) Impurity inhibition.

(3) Texture inhibition in a material with strong preferred orientation.

(4) Grain boundary grooving in the case of thin films and sheet materials.

Specifically, as for bulk materials, the mechanisms of occurrence of AGG has been explained and discussed in terms of the particle-pinning effects on the grain boundary and texture [11, 12-18]. Recently, solute drag effect was found to be another mechanism for AGG [19-20]. These three mechanisms are described in detail in the following sections.

2.1.1 Pinning effect of precipitates leads to AGG

Austenite grain growth can be inhibited by microalloying which causes the precipitation of second-phase particles in austenite. The interaction of grain boundaries with second-phase particles has been a subject of great interest since Zener proposed a simple model of that interaction in an article written by Smith in 1948 [21]. The intersection of an isolated grain of radius, R , by a spherical second-phase particle of radius, r , is shown in Figure 2.1. In this model, Zener balanced the driving force, F_d ($F_d = \frac{2\sigma}{R}$) for motion acting on a rigid grain boundary with the pinning force, F_p

($F_p = \frac{3f\phi}{2r}$) exerted by spherical particles on the boundary.

At equilibrium, the following equation was obtained:

$$\frac{2\phi}{\bar{R}} = \frac{3f\phi}{2r} \quad (2.2)$$

Here, ϕ is grain boundary energy, $\bar{R}$ is the average radius of spherical grains, f is the volume fraction of the second phase particles and r is the average particle radius.

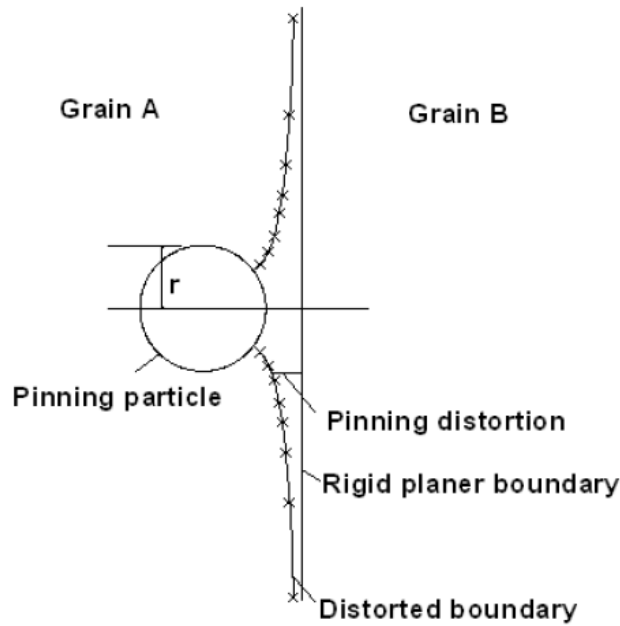


Fig. 2.1. Pinning of grain boundaries (between two grains, A and B) by a spherical second phase particle (particle radius, r) [22].

Effective grain boundary pinning is classically considered in Zener's balance between the pinning forces of the precipitate array and the driving force for grain growth [9]. For AGG of austenite to occur, this force balance must be upset either by a reduction in the pinning force or by a very high driving force for grain growth. On the pinning-force side of Zener's equation, a degradation of the pinning array can occur by partial dissolution or by ripening of the precipitate distribution.

For the analysis of AGG in presence of second phase particles, Gladman [22] has developed a well-known approach that takes into account the interplay between the parameters of the pinning particle size distribution and grain size distribution. In Gladman's equation, r_{crit} is the critical radius of the second phase particles, which

represents the maximum particle size that would effectively counteract the driving force for grain coarsening [22]

$$r_{crit} = 3 \frac{R_0 f}{\pi} (3 - 4/Z)^{-1} \quad (2.3)$$

where, R_0 is the initial matrix grain size, f is the volume fraction of the particles, and Z is the term used to account for the heterogeneity of the matrix grain size. It is clear from Eq. (2.3) that for some given uniform initial grain size, r_{crit} decreases as either the volume fraction of particles decreases or the grain heterogeneity Z increases.

According to Eq. (2.3) some particles can become ineffective in pinning the grain boundaries (particle size, $r > r_{crit}$) during reheating due to coarsening (increase in r) or even dissolution (decrease in f , and hence, decrease in r_{crit}). In such a situation, the growth of the large grains in the microstructure can offer the higher release in energy (due to higher Z value), and that can exceed the energy increase in unpinning. Therefore, the large grains become unpinned and grow in size. Small grains cannot grow as those are still pinned.

Therefore, selected grains with a suitable Z value will grow in regions with a major presence of coarse precipitates as a consequence of the low pinning ability of those precipitates; these growing grains will increase further their size advantage over their neighbors, which will in turn diminish the critical radius for pinning. This will create the situation of AGG. Hence, precipitate coarsening or partial dissolution of precipitates can lead to the occurrence of AGG. Upon complete dissolution of precipitates AGG will stop and NGG will resume (as in the absence of particles) for the larger grains, $Z > 4/3$, consuming smaller grains, till the large grains impinge upon each other.

The same with the above discussed mechanisms of austenite grain growth with presence of second phase particles, three stages grain growth modes in Al-killed low carbon steel has been observed by Gladman [23] which is displayed in Fig. 2.2. At low temperatures, where the precipitates are stable and the mobility of the grain boundaries is low, the austenite grain boundaries can be effectively pinned, the grain growth is suppressed. However, the second-phase particles become unstable at high

temperatures. They will either coarsen due to a difference in the chemical potential of the solute atoms resulting from a difference in particle size, or they will dissolve because of an increasing solubility of the solute atoms in the matrix at elevated temperatures. If grain growth inhibition fails, the grain growth may become abnormal, producing a few much larger grains. During the AGG a few large grains grow discontinuously and consume the neighboring matrix of smaller grains, leading to the development of extraordinarily large grains. And the critical temperature for normal-to-abnormal grain-growth transition has been called the grain-coarsening temperature, T_c . At temperatures much higher than T_c , NGG takes place again due to the nearly complete dissolution of pinning particles.

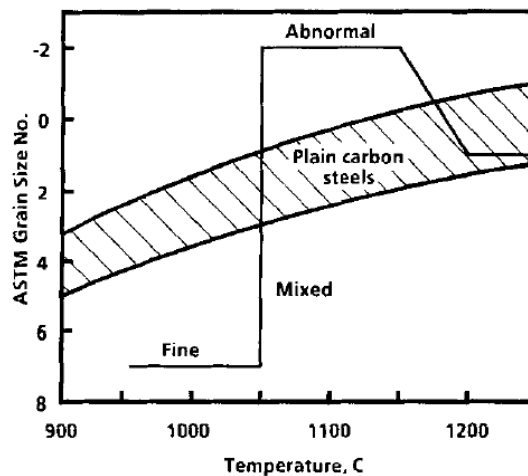


Fig. 2.2. Austenite grain growth characteristics of Al-treated low carbon steel [23].

2.1.2 Solute drag effect leads to AGG

Solute atoms often segregate at defects in crystalline structures. Even a small amount of the segregated impurity can fundamentally alter not only the microscopic defect dynamics, but also the macroscopic behavior of the materials. In alloys with impurities or alloying elements, the solutes are often enriched or depleted in grain boundary regions as compared with the concentration in the interior regions of the grains [24]. In an equilibrium state where a grain boundary is not moving, the grain boundary segregation gives rise to the absence of net force at the interface. When a

grain boundary is moving under a driving force, however, the concentration profile falls behind the grain boundary position. The resultant broken symmetry between the concentration profile and the grain boundary position brings about a net attractive force between them, which has been called the solute drag effect [25–28].

Therefore, solute drag is explained as a dynamic segregation effect. Solutes segregate to the grain boundaries and due to the migration of the grain boundary an asymmetric segregation profile develops. This profile leads to a net force on the grain boundary, opposing the migration, and slowing it down. The kinetics of grain growth when solute atoms segregate at grain boundaries is dependent on whether the migrating grain boundaries can break away from the segregation atmosphere.

Experimental results suggest that the main effect of solute atoms is on the growth process rather than recrystallisation nucleation [29-36]. When solute atoms are located on and attached to a grain boundary, the migration velocity of the boundary resulting from a given driving force is influenced by the solute atoms. Most of the work related to solute drag was conducted for recrystallisation experiments where the driving force is quite high. The theory for solute drag was developed in two classic papers by Cahn [37] and Hillert and Sundman [38]. Cahn assumes an ideal very diluted binary mixture in his model, whereas Hillert and Sundman consider the more general case of non-diluted nonideal binary mixtures. The solute drag force in the theory of Cahn is linearly proportional to the solute concentration, while Hillert and Sundman find a more complex concentration dependence, using a regular solution model. The theory of Cahn is applied in most of the present models. According to Cahn's theory [37], the rate of grain coarsening G can be given as

$$G^2 = (2\gamma V_M \cdot n / t) / (\lambda + \alpha C') \quad (2.4)$$

where, γ is the specific grain boundary energy per unit volume, V_M is the molar volume of austenite, n is the isothermal grain coarsening law exponent ($n=10$ in equation 2.4), t is the coarsening time, λ is the reciprocal of boundary mobility of pure austenite, α is the reciprocal of boundary mobility at unit solute concentration, and C' is the bulk solute concentration. G continues to increase with C' but with a

decreasing rate.

The grain boundary velocity as a function of the driving force is shown in Fig. 2.3 [26–28, 39–42]. With a small driving force, the grain boundary velocity is low, which allows solute atoms to diffuse to catch up to the grain boundary. In this regime, the migration kinetics is governed by the solute diffusion near the grain boundary region. As the driving force increases, the solute atoms gradually become a heavier burden for the grain boundary motion due to an increase in grain boundary velocity. When the driving force reaches a critical value, the grain boundary eventually breaks away from the solute atmosphere. At this critical condition, the grain boundary undergoes a transition from dragged migration in the low-velocity regime to free migration in the high-velocity regime. At this condition, the grain boundary velocity makes a discontinuous jump, as indicated by an arrow mark in Fig. 2.3. In the high-velocity regime, the migration kinetics is governed by the atomic jump across the grain boundary [42].

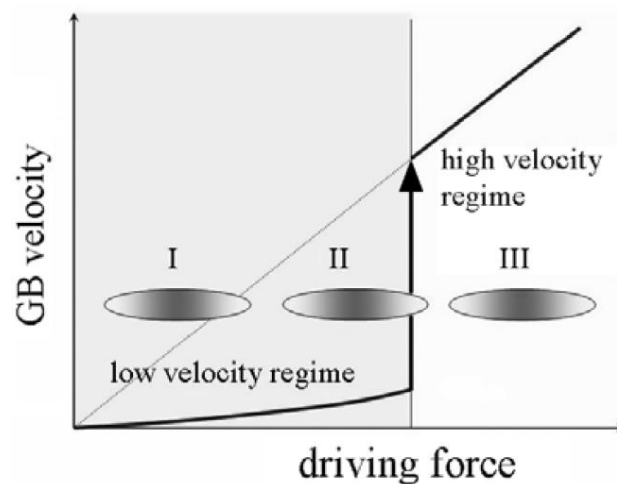


Fig. 2.3. Grain boundary velocity under the solute drag effect as a function of driving force. When the driving force reaches a critical value, the grain boundary breaks away from the solute atmosphere and undergoes a discontinuous jump from dragged migration in the low-velocity regime to free migration in the high-velocity regime [42].

In the case of a polycrystalline system with a range of grain sizes, it is undergoing a curvature-driven grain growth. When the average grain size is so large

that the average driving force for grain growth is small compared with the critical driving force (Envelope I of Fig. 2.3), all the grain boundaries migrate and slowly drag the solute atoms. NGG occurs in this case. When the average grain size is small and the average driving force for grain growth is large (Envelope III of Fig. 2.3), all the grain boundaries migrate rapidly free from the solute atmosphere. As a result, NGG takes place as in Case I, but at a much higher rate.

The most interesting situation is the case where the supercritical driving force acts upon only a minor portion of the grain boundaries in a system (Envelope II of Fig. 2.3). The driving force for growth of an i th grain with radius R_i is given approximately by $1/\bar{R} - 1/R_i$, where $\bar{R}$ is the average radius of the grains neighboring with the i th grain [6, 43–44]. Therefore, among the grains in Envelope II, the large grains surrounded by the small grains will be subjected to the supercritical driving force and grow rapidly free from the solute atmosphere. By contrast, the solute atoms will almost pin the remaining grains. Consider a grain which eroded small neighbor grains by a supercritical driving force. Depending on the local surroundings, the average value of the radii of its new neighbor grains can be either larger or smaller than that of previous neighbors. Therefore, the new driving force for the grain can be either supercritical or subcritical, which implies that all the grains that have eroded their neighbor grains do not grow any further. The grains that have the fortune to meet small grains again will be subjected to the supercritical driving force and rapidly grow further. A grain that undergoes this action repeatedly may grow abnormally.

The mechanism of AGG induced by the solute drag effect is quite similar to the AGG by the particle-pinning effect, because both are based on grain boundary pinning in the matrix grains.

2.1.3 Texture leads to AGG

Grain boundaries have a major influence on the properties and behavior of polycrystalline materials where numerous grains having different orientations form the grain boundary network. Low angle grain boundaries ($<15^\circ$ of misorientation) are

generally formed by arrays of dislocations. The grain boundaries which have misorientation between -20° and -45° generally have a random structure and thus higher energies compared to the low angle boundaries [45]. The driving force for grain growth is the reduction of grain boundary area, and thus the total energy of the system. Grain growth is achieved by the migration of grain boundaries. In general, the ability of the grain boundary to migrate is characterized by a property known as grain boundary mobility. Grain boundary mobility depends on grain boundary energy, grain boundary structure, solute interaction, etc., and is often linked to the misorientation between the neighboring grains [45]. During the grain growth process, differences in the grain boundary motilities determine the final texture and grain size distribution of a material.

On annealing after very heavy cold rolling, a strong recrystallization texture is usually found, which may involve the partial retention of the deformation texture but quite often a very different but very strong new texture forms. A classic example is the formation of a very strong cube texture in some (but not all) heavily rolled fcc metals [46]. The cube orientation is a finite but very small part of the deformation texture. Two major alternative models exist for the formation of a strong new texture—usually described as ‘oriented nucleation’ or ‘oriented growth’ [46-47]. The oriented growth factor, β , is determined by the relative sizes $\bar{R}_c / \bar{R}$ of the cube to the average grains [48]. That is, there is a strong oriented growth effect if:

$$\beta = \bar{R}_c / \bar{R} \gg 1 \quad (2.5)$$

One interesting new suggestion for understanding how different grain sizes might occur comes from Juul Jensen’s [49] hypothesis of ‘orientation pinning’, Fig. 2.4.

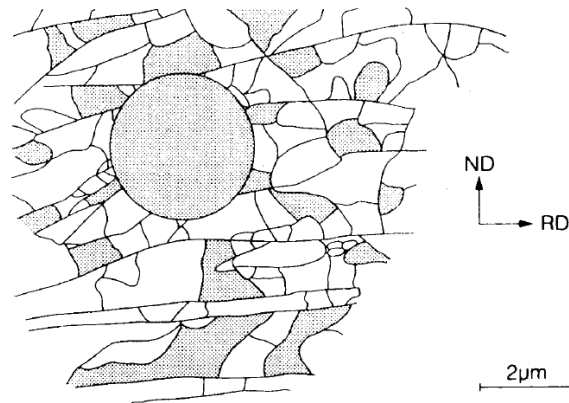


Fig. 2.4. Orientation pinning of a new grey circular grain when it meets regions of similar orientation (also grey) with which it has low angle, low mobility boundaries. After Juul Jensen [49].

This idea is that a recrystallizing grain growing in a very heavily deformed material can meet many regions of different orientation. A grain such as cube, in most cases, will meet very few regions of similar orientation with which it shares low misorientation low mobility boundaries. Other grains, especially with orientations within the deformation texture, will meet many regions with similar orientations, and thus, on average, will have lower mobility and grow more slowly. Evidence supporting this idea of orientation pinning was presented where, for several examples of deformed aluminum, a value of $\beta > 1$ for the cube grains was found [49].

A very similar idea to that of orientation pinning was put forward at the same meeting by Doherty et al. [50]. Their idea was called 'variant inhibition', Fig. 2.5. The only difference is the recognition based on experimental studies of warm plane strain extruded aluminum, is that the deformed bands of nearly constant orientation are stretched out in the extrusion direction, equivalent to the hot rolling direction, so that very strong inhibition of growth in the normal direction will occur. Grains from a deformation texture component will be inhibited from thickening by the low angle boundaries of the same variant of the deformation texture component. This analysis identified two important effects apparently rising from the low mobility of low angle boundaries formed between a recrystallizing grain and deformed bands elongated in the rolling direction after heavy plane strain deformation.

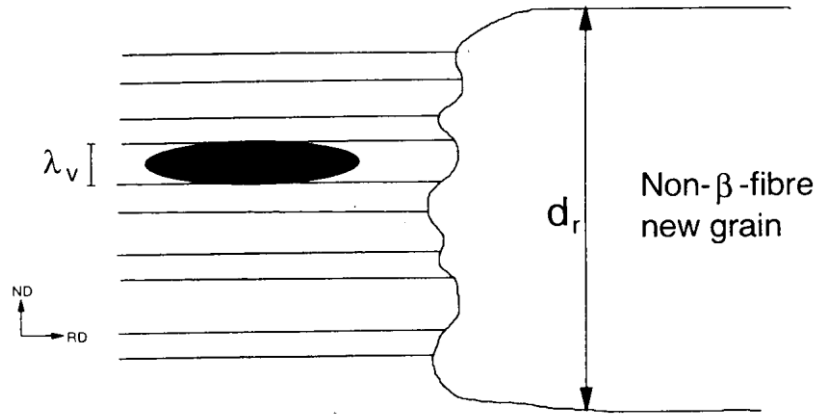


Fig. 2.5. Variant inhibition, the inhibited growth, in the normal direction, of a new grain belonging to one of the deformation texture components when it meets a thin deformed band of similarly oriented material. The spacing between the variants is λ_v . The idea is identical to that of Fig. 2.4 except for presence of the banded structure drawn out in the rolling direction of warm rolled alloys. Such a thin grain could be easily destroyed by grain coarsening by the thicker, uninhibited grain whose orientation is found more rarely in the deformed matrix. After Doherty et al. [50].

Therefore, the inhibition of NGG may occur by the existence of a very sharp primary texture. In this case the low angle grain boundaries between most of the grains prevent NGG and only those grains with strongly deviating orientations may grow abnormally. It should be mentioned that in the case of polycrystals which do not contain any second-phase particles and show a random texture large grains will always grow more slowly than small grains and will finally join the normal grain size distribution [6, 51]. Thus, in such ‘ideal’ polycrystals AGG will never occur.

2.2 Effect of cold deformation on reverted austenite grain structure

According to Eq. (2.3), the occurrence of AGG is strongly affected by the initial austenite grain size and the size and volume fraction of precipitates. Since the cold deformation has a strong effect on the kinetics of reverse transformation, thus the initial as-transformed austenite grain size. Cold deformation also has a great effect on the precipitation kinetics of second phase particles in steels. Therefore, as a candidate method to control the AGG of austenite, it is necessary to summarize the effect of cold deformation on the initial austenite grain size and the distribution and morphology of the precipitates.

2.2.1 Effect of deformation on reverse transformation

The behavior of austenite formation on heating carbon steels having various initial structures has received much attention from the point of view of grain refinement of austenite [52-56]. However, a limited amount of research has investigated the effects of cold work and the reverse transformation on the generation of austenite from cold deformed microstructures.

Beswick [57] investigated the effect of cold deformation on the ferrite-to-austenite transformation in SAE 52100 steel. He found that the temperatures for the start (A_{c1}) and end (A_{c3}) of the ferrite to austenite transformation are lowered due to cold work, as shown in Fig. 2. 6. The prior cold deformation results in a refinement of the austenite grain size.

	Cold rolled 	Hot rolled 
	Mean values: 95% conf. limits ± 1	
A_{c1}	744.1	755.9
A_{c3}	775	789.7

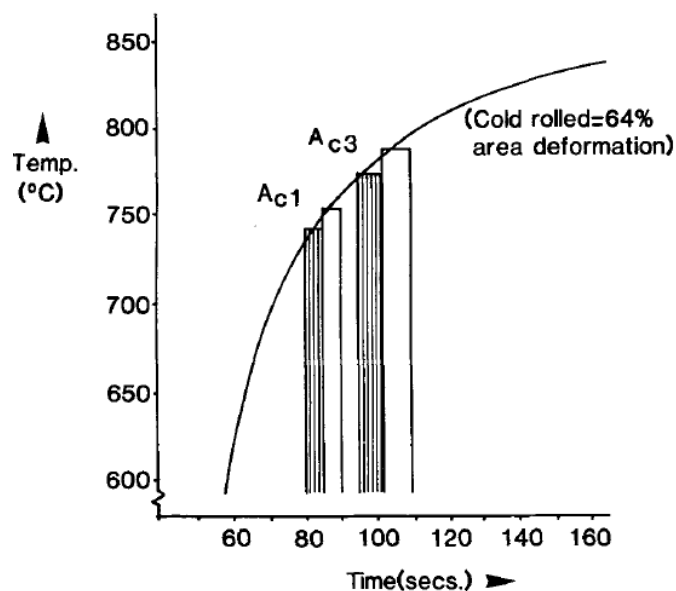


Fig. 2.6. Diagram showing the influence of cold deformation on the ferrite to austenite transformation temperatures [57]. The word “values” in the above Table should be “values”.

Tokizane et al [58] investigated the effect of prior deformation on the austenitizing of lath martensite. It was found that prior cold deformation results in a refinement of the austenite grain size, as shown in Figure 2.7. They schematically explained the formation process of austenite from non-deformed or deformed lath martensite as shown in Fig 2.8. In the case of non-deformed lath martensite, recrystallization does not occur and austenite forms directly from the tempered lath martensite. Austenite particles precipitate at first preferentially at prior austenite grain boundaries, and then form within the prior austenite grains mainly along the packet, block, and lath boundaries. Since the austenite particles (acicular type) formed along the parallel block or lath boundaries have almost the same orientation to each other, some of these austenite particles coalesce to somewhat larger massive grains [52-56, 59].

On the other hand, when lath martensite is deformed, the recrystallization occurs prior to or simultaneously with the austenite formation. Therefore, the behavior of austenite formation in deformed specimens becomes different from that in non deformed specimens. Although the recrystallization is slightly accelerated with increase in the amount of deformation, the formation rate of austenite from lath martensite is much more accelerated with increase in the amount of deformation. Consequently, the austenitizing behavior (and thus the austenite grain size) is controlled by the competition between the recrystallization of martensite and the austenite formation.

In the case of about 30 to 50 pct deformation, the recrystallization of deformed lath martensite occurs first and is almost completed prior to the beginning of austenite formation. Thus, a high density of austenite particles is nucleated at the boundaries of recrystallized fine ferrites. In this case, the austenite particles may have different orientations from each other, and the coalescence is hard to occur, and then the final austenite grain size becomes fairly fine.

When the lath martensite is much more heavily deformed such as 70 to 85 pct, the nucleation of austenite starts almost simultaneously with the recrystallization of deformed lath martensite at the very early stages of holding. Then, austenite grains are

precipitated densely in the deformed (unrecrystallized) regions as well as at the grain boundaries of recrystallized ferrites. Thus, the nucleation sites of austenite are markedly increased in the heavily deformed steel, and consequently the final austenite grain becomes very fine.

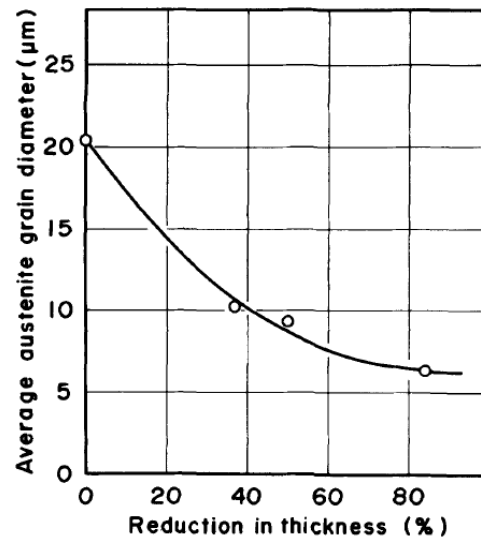


Fig. 2.7. Austenitic grain size at the completion of austenitizing as a function of the amount of prior deformation of lath martensite in 0.2 pct C steels [58].

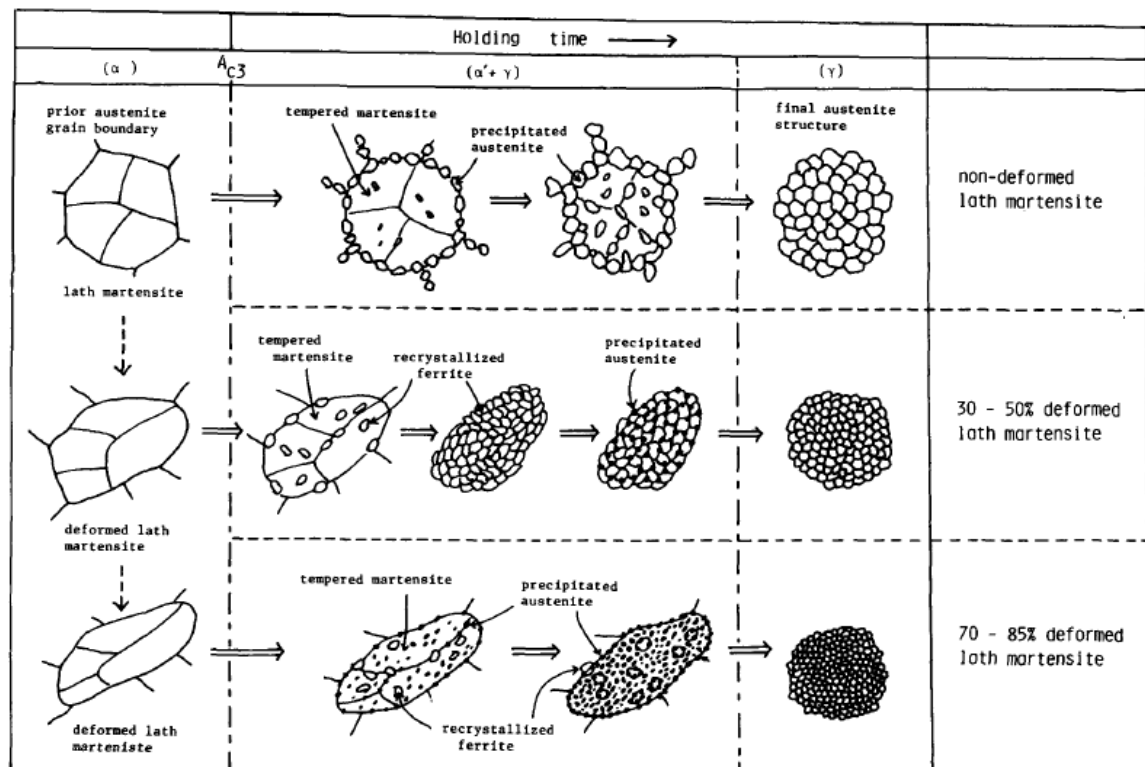


Fig. 2.8. Schematic drawing showing the formation process of austenite from non-deformed or deformed lath martensite in 0.2 pct C steels [58].

Therefore, prior cold deformation results in the refinement of as-transformed austenite grain structure. Thus, it should affect the kinetics of the subsequent austenite grain growth.

2.2.2 Effect of cold deformation on precipitates

Precipitation of nitride and/or carbide in steels has been extensively studied in the past [60-69]. Dislocations play a major role in these precipitation processes and precipitation in materials with high dislocation densities allows one to produce fine precipitates distributions which are of practical importance [70]. Nevertheless, limited works has investigated the precipitation in materials with high dislocation densities, due to the difficulty in observing extremely fine precipitates by transmission electron microscopy (TEM) in the presence of the dislocation strain-fields. Furubayashi et al [71] used a high voltage TEM and Kesternich [70] used Moire fringe imaging for observation of the fine precipitates with the presence of high dislocation densities.

Furubayashi found that small tensile deformation, e.g. 5% produced a striking increase in the precipitate density. And the resultant fine precipitates were uniformly distributed in the matrix, as shown in Fig. 2.9.

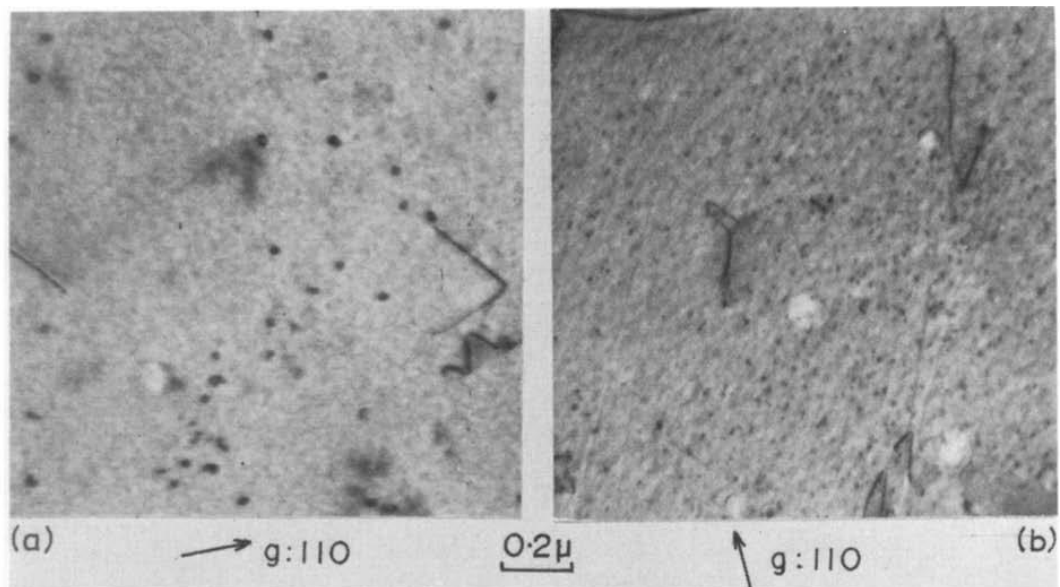


Fig. 2.9. Effect of prior deformation upon the distribution of AlN in specimens annealed up to 755 ° C; (a) ST specimen, (b) STD specimen deformed 5% [71].

Prior deformation tends to keep the high densities of the precipitates to higher temperatures, see Fig. 2.10 and Table 2.1. They attributed this to the deformation induced dislocation loops which act as nucleation sites of the precipitates. Both Furubayashi et al. [71] and Kesternich [70] found that the cold deformation made the precipitates resist to coarsening at lower temperatures (below 900°C), as shown in Fig. 2.10.

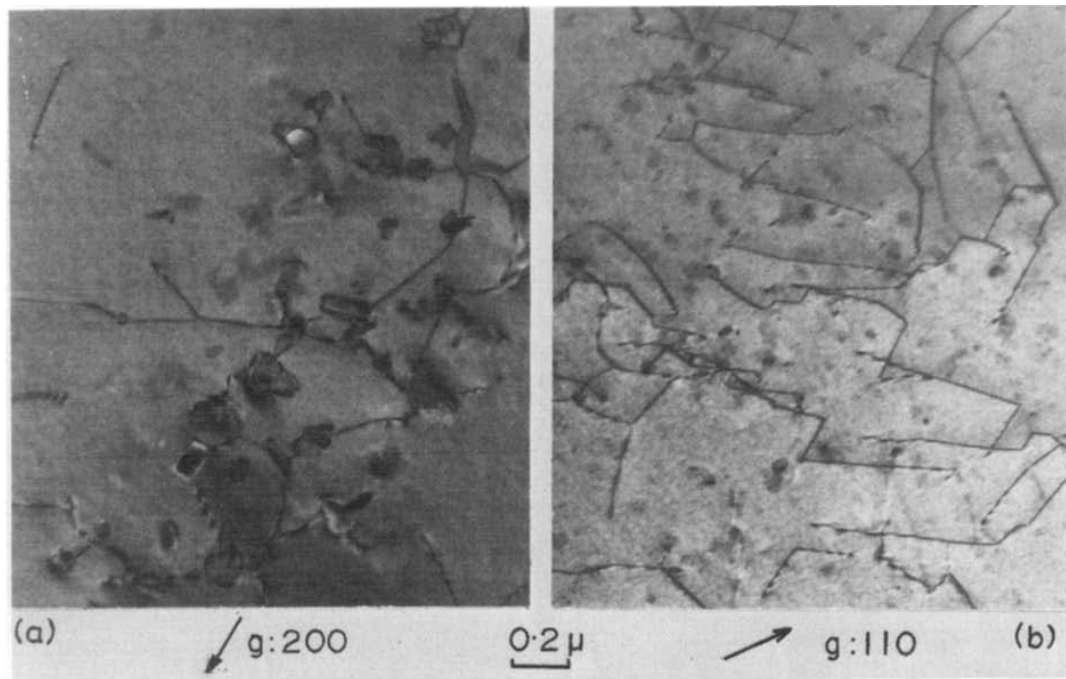


Fig. 2.10. Effect of prior deformation upon the distribution of AlN in specimens annealed up to 870 ° C; (a) ST specimen, (b) STD specimen deformed 5% [71].

Table 2.1 Density of precipitates of AlN [71]

Density of precipitates of AlN (10^{13} cm^{-3})			
<i>Specimen</i>	<i>Strain at prior deformation</i>	<i>755° C</i>	<i>870° C</i>
ST	0%	10	9
STD	5%	84	23

Hence, the initial austenite grain size, and the distribution and morphology of the precipitates are remarkably affected by cold deformation. This strongly suggests that the cold deformation should affect the subsequent austenite grain growth behavior.

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Chapter 3

Abnormal grain growth in austenite structure reversely transformed from ferrite/pearlite banded structure

3.1. Introduction

As described in Chapter 1, ferrite/pearlite (F/P) banding is commonly observed in hot rolled low alloy steels [1-7], consisting of alternate layers of proeutectoid ferrite and pearlite. It was reported [8] that the banding in low-alloy steel is due to the interdendritic segregation of substitutional alloying elements. The F/P banding has been widely investigated by many researchers regarding its formation mechanism [1-5, 8-11] and its effects on the mechanical properties [7, 12-18]. However, little has been studied for the effect of F/P banded structure on austenite grain growth after the reverse transformation during heating process.

Hot-rolled steels with the banded structure are usually reheated prior to further processing such as carburization, nitridation and ausforming etc. This reheating process generally leads to the reverse transformation from ferrite to austenite structure and the subsequent austenite grain growth. The grain growth can be classified into two types: Normal Grain Growth (NGG) and Abnormal Grain Growth (AGG). NGG is characterized by the self-similar coarsening process of the microstructure [19], while AGG takes place by the rapid growth of a small number of grains [20]. AGG in austenite grain structure during manufacturing processes should be avoided because the mechanical properties of materials exhibiting a bimodal austenite grain size distribution caused by AGG are degraded [21]. AGG has been extensively investigated because of both its technological relevance in the steel industry and scientific interest. The mechanism of occurrence of AGG has been explained and discussed in terms of the particle-pinning effects on the grain boundary, texture and surface effects [19, 22-28]. Recently, solute drag effect was found to be another mechanism for AGG [29-30]. Among several mechanisms for AGG, an

inhomogeneity in microstructures such as non-uniform distribution of the precipitated particles and alloying elements should be one of the important factors causing the occurrence of AGG. In this regard, it is quite important to investigate how the inhomogeneous banded F/P structure affects the grain growth of reversely transformed austenite structure. The present chapter will show that the non-uniform pinning effect of AlN particles in the F/P banded structure lowers the onset temperature for the AGG of austenite phase.

3.2. Experimental

A hot rolled bar of a hypoeutectoid steel was used in this study, and its chemical composition is shown in Table 3.1. To remove the residual stress in the as-received steel bar, the samples were normalized at 900 °C for 30 min in air and then furnace-cooled to room temperature. All specimens were taken from the same bar. To obtain non-banded F/P microstructures, some normalized samples were homogenized for 3 h at 1300 °C in an argon atmosphere, then air-cooled to room temperature. This homogenization temperature was determined based on the preliminary experiments of homogenization treatments at both 1200 and 1300 °C. It was found that the non-banded F/P microstructures could not be produced by homogenization treatment at 1200 °C, while 1300 °C was a suitable homogenization temperature to obtain the non-banded F/P microstructures.

Table 3.1 Chemical composition of the carbon steel used in this study (mass%)

C	Si	Mn	P	Al	N
0.2	0.2	0.8	0.015	0.04	0.006

The initial samples thus prepared were cut into a disk shape with 20 mm in diameter and 5 mm in thickness. The heating experiment was carried out by holding the disk-shaped samples in a salt bath held at temperatures between 900 °C and 1200 °C, followed by quenching with an iced-water bath. Longitudinal sections of the quenched samples were observed by optical microscopy after grinding and polishing

followed by chemical etching with 3% nital or supersaturated picric acid at 60 °C (to reveal the prior austenite grain boundaries). The grain size distributions were plotted in area percent as a function of grain size range. The grain size distributions were investigated to identify the unimodality or bimodality of the grain structure. An Electron Probe Micro-Analyzer (EPMA) was employed to investigate the concentration distribution of elements in the quenched sample. The microanalysis of AlN precipitates was conducted by using Transmission Electron Microscopy (TEM) examination on carbon extraction replicas taken from the samples. To prepare the replicas, the samples were first ground and then polished with 3 to 1 µm diamond paste. After slightly etching the sample in a 3% nital for 5 seconds, a thin film (about 30 nm) of carbon was evaporated onto the etched surface. The carbon film was then scored with a sharp-pointed tool into 3 mm squares followed by etching of the specimen in a 5% nital solution until the replicas lift off. The observations and analyses were performed at an operating voltage of 200 kV in a JEOL JEM-2010 TEM equipped with an Energy Dispersive Spectroscopy (EDS). The size of the particles were measured either directly on the screen at a suitable magnification or by measuring enlarged micrographs. A minimum of 1000 particles were measured for each case to give the mean particle size.

3.3. Results and discussion

3.3.1 Characterization of the initial metallurgical state

Figure 3.1(a) shows a micrograph of a sample normalized at 900 °C for 30 min and cooled in the furnace to room temperature, which displays a typical morphology of banded ferrite (the bright region)/pearlite (the dark region) structure. This is hereafter referred to as the banded sample. The manganese and silicon concentrations along the line marked with A and B (Fig. 3.1(a)) were examined by EPMA and the results are shown in Fig. 3.1(b). It can be seen from Fig. 3.1(b) that there are higher and lower manganese concentrations in pearlite and ferrite regions, respectively. The average compositional amplitudes of manganese are around 0.5%. On the other hand,

the silicon concentration is relatively uniform, with no apparent segregation pattern.

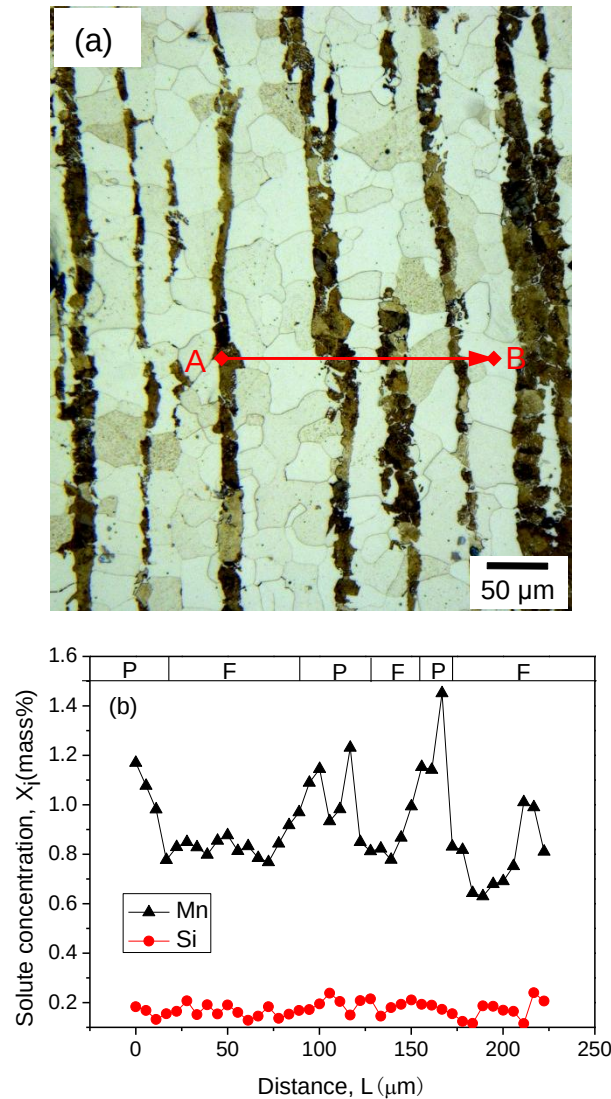


Fig. 3.1. Correlation between the microstructural banding and the segregation patterns of the alloying elements: (a) micrograph of the banded sample etched by 3% nital, where light and dark regions corresponds to ferrite and pearlite regions, respectively, (b) manganese and silicon concentrations along the line AB in (a). Regions of ferrite and pearlite are denoted by F and P, respectively.

It was reported that the redistribution of manganese during austenite to ferrite transformation hardly takes place [31]. Also, it was reported that microchemical banding of manganese is the cause of microstructural banding in low-alloy steels [3, 8]. Hence, the varying manganese concentrations between pearlite and ferrite regions indicate banded segregation of manganese in the F/P banded sample. This inhomogeneity of manganese concentration originates from the segregation during

solidification as described in the introduction section.

The F/P banded structure was completely removed due to the homogenization treatment at 1300 °C for 3 h, as shown in Fig. 3.2(a), in which ferrite and pearlite phases are randomly distributed. This is hereafter referred to as the non-banded sample.

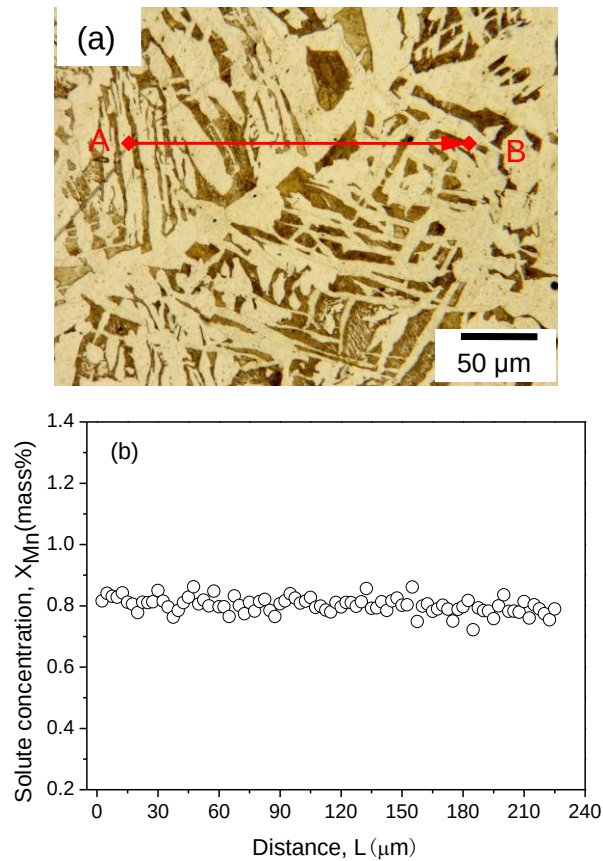


Fig. 3.2. Correlation between the ferrite-pearlite phases and the concentration of manganese in the non-banded sample: (a) micrograph of the non-banded sample etched by 3% nital, where light and dark regions corresponds to ferrite and pearlite regions, respectively, (b) manganese concentrations along the line AB in (a).

The manganese concentration along the line marked with A and B (Fig. 3.2(a)) was examined by EPMA and the result is shown in Fig. 3.2(b). The concentration difference from the average concentration is just within 0.1 % and the distribution of manganese is almost uniform. Therefore, the banded segregation of manganese is removed by this homogenization heat treatment.

3.3.2 Growth behavior of austenite grains

Figure 3.3 displays austenite grain structures and the corresponding grain size distributions of the reheated non-banded samples held at 1000 °C for 0 and 30 min. Fine and uniform grain structures are observed at both holding times and the both grain size distributions are typical lognormal distributions. This indicates NGG occurred in the non-banded sample when held at 1000 °C.

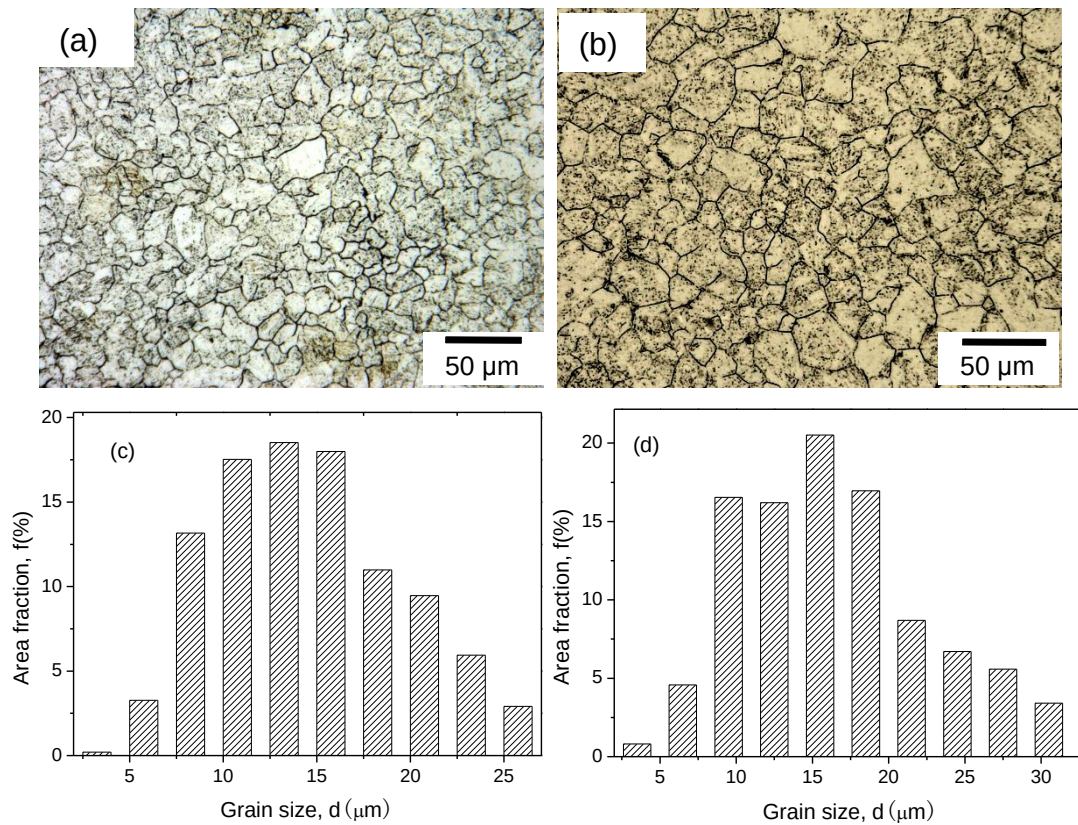


Fig. 3.3. Prior austenite grain structures of non-banded samples held at 1000 °C for: (a) 0 min, (b) 30 min (c) and (d) are the corresponding grain size distributions of (a) and (b), respectively.

The austenite microstructure evolution of the banded sample during holding at 1000 °C for 30 min is shown in Fig. 3.4 and the corresponding grain size distributions are displayed in Fig. 3.5. At 0 min, the structure consists of fine grains uniformly distributed over the observation area (Fig. 3.4(a)) and the corresponding grain size distribution is lognormal (Fig. 3.5(a)). However, as the holding time increases, grains much coarser than the surrounding grains appeared and grew, as indicated by arrows in Figs. 3.4(b) and (c). Correspondingly, the grain size distributions exhibit

bimodality (peaks indicated by arrows in Figs. 3.5(b) and (c)). In other words, the duplex grain structure forms in the beginning of the holding period, and the grains finally grew into extremely large ones accompanied by some of the retained fine matrix grains (Fig. 3.4(d) and Fig. 3.5(d)). Therefore, AGG occurred in the banded sample when held at 1000 °C, while NGG took place in the non-banded sample at this temperature. Similar grain growth behaviors took place at 900 °C. In the non-banded samples, slow and continuous grain growth occurred when held for 30 min at 900 °C. The austenite grain structures were similar to those shown in Figs. 3.3(a) and (b). In the banded samples, on the other hand, discontinuous grain growth occurred, although the large grains appeared at the later period of holding (after holding for 25 min). Fig. 3.6 shows the austenite grain size distributions of the banded samples held at 900 °C for 25 and 30 min. Lognormal grain size distribution (Fig. 3.6(a)) develops into bimodality (Fig. 3.6(b)) when the holding time increases. Therefore, at 900 °C AGG also occurred in the banded sample, while NGG took place in the non-banded sample at the same temperature.

To understand the austenite grain growth behavior above 1000 °C, both the banded and non-banded samples were heated up to 1100 and 1200 °C and the results are displayed in Figs. 3.7 and 3.8, respectively. In the banded sample, AGG also occurred at 1100 °C (Fig. 3.7(a)), but NGG took place on reheating at 1200 °C (Fig. 3.7(b)). Similarly, in the non-banded sample, AGG occurred at 1100 °C (Fig. 3.8(a)), while NGG occurred at 1200 °C (Fig. 3.8(b)). The grain growth modes of both the banded and non-banded samples at holding temperatures are summarized in Table 3.2. It can be seen that both samples experienced the normal grain growth mode above 1100 °C, although they have different grain growth modes below 1000 °C.

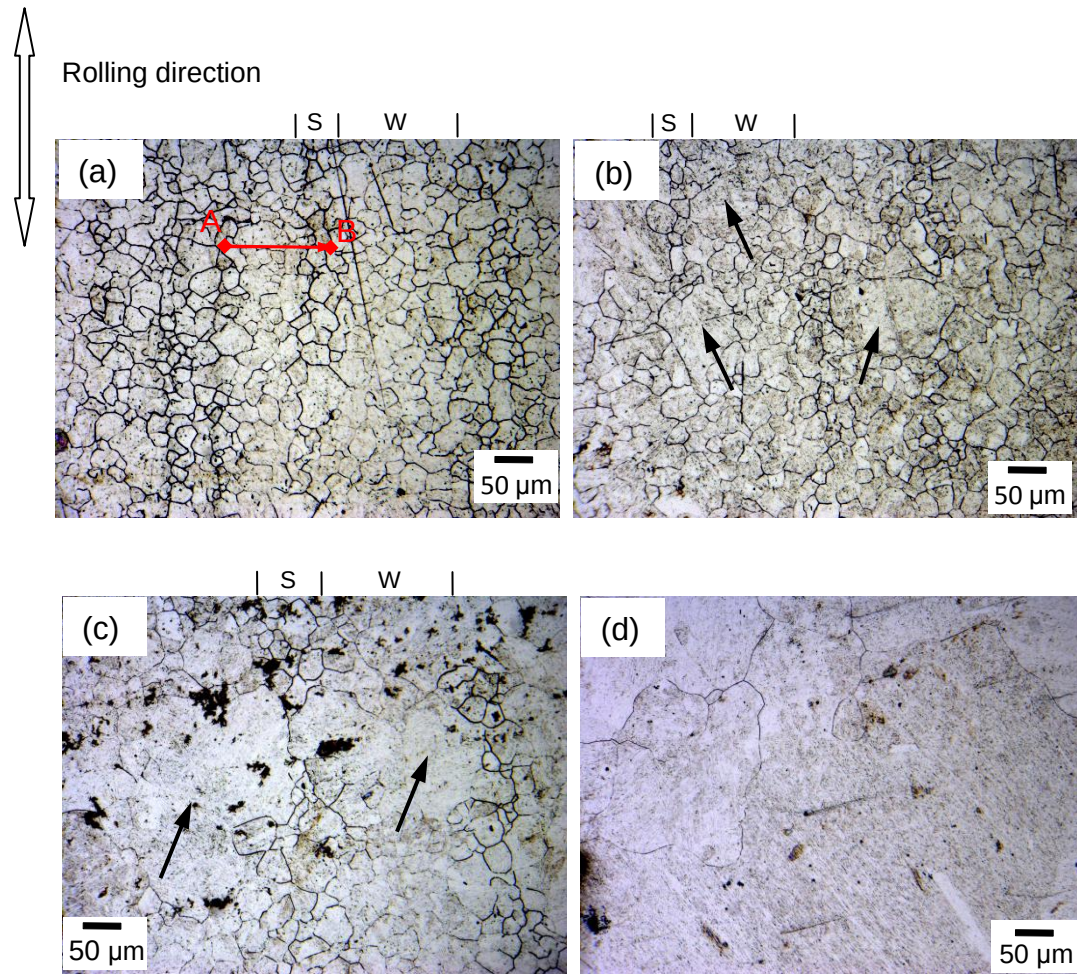


Fig. 3.4. Prior austenite grain structures of banded samples held at 1000 °C for: (a) 0 min, (b) 3 min, (c) 10 min, (d) 30 min. The strongly and weakly etched regions are denoted by S and W, respectively.

It was reported in numerous works that partial dissolution or Ostwald ripening of the second-phase particles upon reheating is one of the mechanisms that lead to AGG [22-23, 25-27]. Typical “normal-abnormal-normal” grain growth in micro alloying steels during heating process has also been widely reported [32-35]. Below a critical temperature which is called grain-coarsening temperature, all the grain boundaries are effectively pinned by the second phase particles and the increase in grain size is very little. However, above the grain-coarsening temperature, AGG takes place due to the coarsening or partial dissolution of the particles. When the temperature is higher than the temperature at which the precipitates completely dissolve into the matrix, the NGG accordingly takes place. The change of grain growth behavior from AGG to

NGG observed in both banded and non-banded samples above 1100 °C corresponds to this mechanism. However, both the samples exhibit the different grain-coarsening temperature as shown in Table 3.2: it is below 900 °C for the banded sample while it is between 1000 °C and 1100 °C for the non-banded sample.

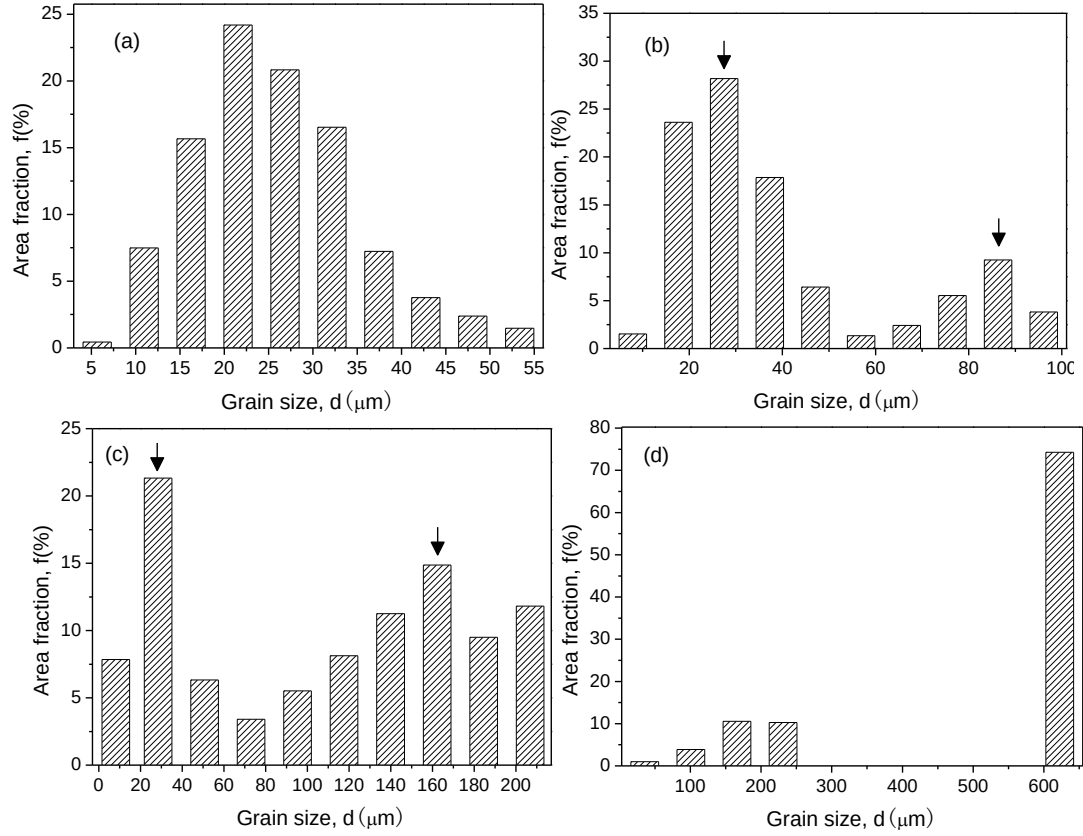


Fig. 3.5. Prior austenite grain size distributions corresponding to Fig. 3.4(a) to (d).

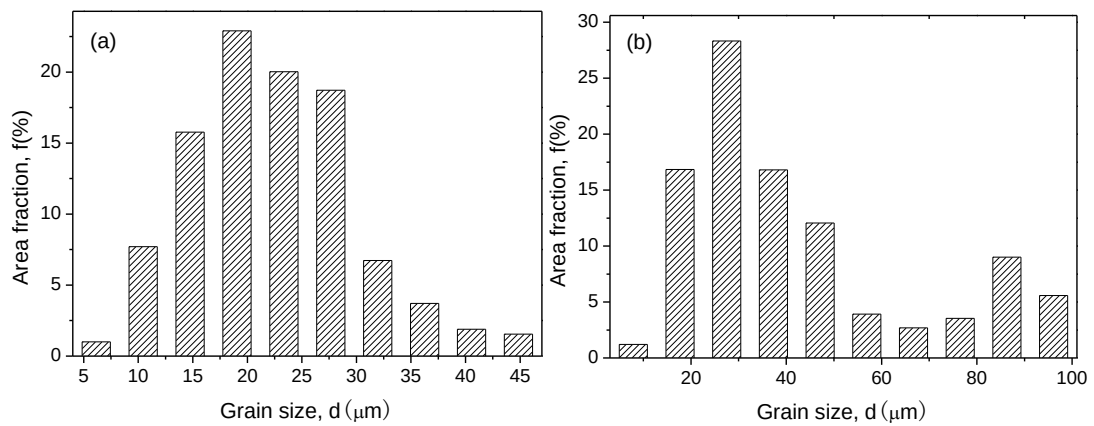


Fig. 3.6. Prior austenite grain size distributions of banded samples held at 900 °C for: (a) 25 min, (b) 30 min.

Table 3.2 Austenite grain growth modes at different holding temperatures

Temperature, °C	900	1000	1100	1200
Banded sample	AGG	AGG	AGG	NGG
Non-banded sample	NGG	NGG	AGG	NGG

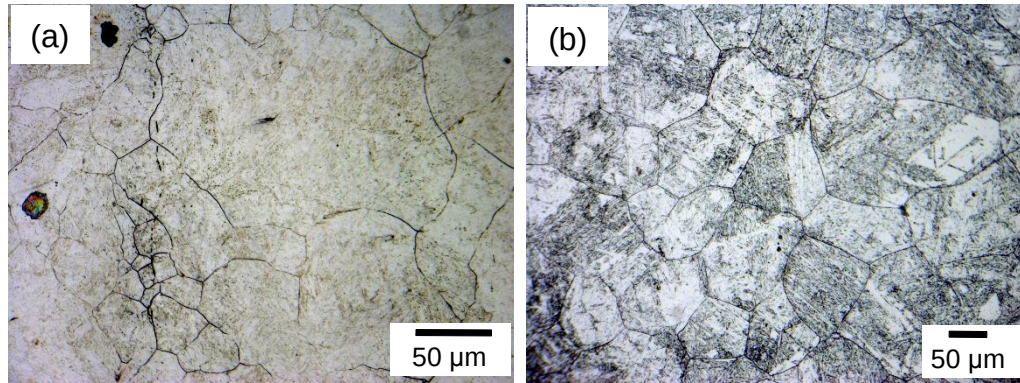


Fig. 3.7. Prior austenite grain structures of banded samples held at: (a) 1100 °C, 2 min, (b) 1200 °C, 2 min.

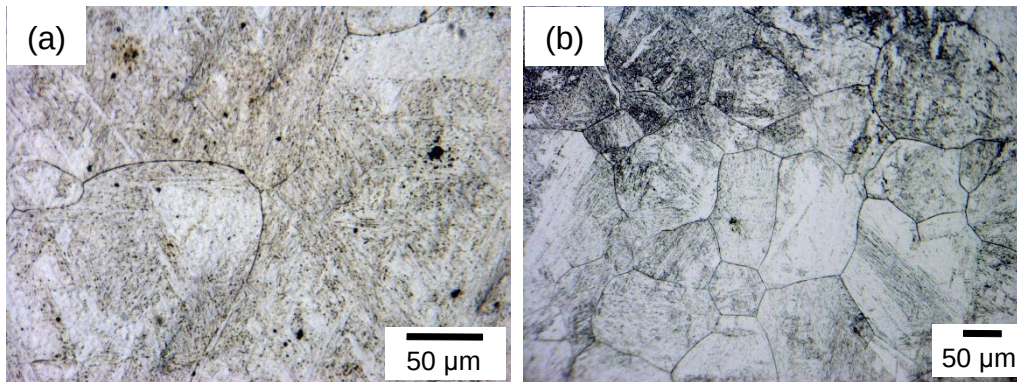


Fig. 3.8. Prior austenite grain structures of non-banded samples held at: (a) 1100 °C, 2 min, (b) 1200 °C, 2 min.

Precipitates were observed in the banded sample, as indicated by the arrows in Fig. 3.9, and EDS analysis revealed that they are AlN. It is known that the solubility of microalloyed metal carbide or nitride can be expressed by

$$\log [M][C] = F - K/T \quad (3.1)$$

where $[M]$ and $[C]$ are the concentrations of the metal and carbon/nitrogen in wt pct, K and F are constants determined from the solubility data, and T is the absolute

temperature. Thus, the temperature T_s at which these elements are completely in solution is given as

$$T_s = \frac{K}{F - \log(M \cdot C)} \quad (3.2)$$

Wilson and Gladman [36] critically evaluated the wide variety of AlN solubility products proposed in the literature. They concluded that for practical heat-treatment purposes, the solubility of AlN can be represented by [37]:

$$\log [Al][N] = 1.03 - 6770/T \quad (3.3)$$

By employing Eq. (3.3) the solubility temperature of AlN particles in this study, T_s , is calculated to be 1180 °C.

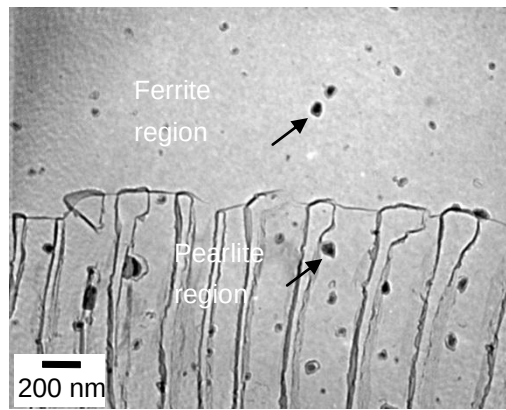


Fig. 3.9. TEM image of carbon extraction replica showing the precipitates distribution in both ferrite and pearlite phases.

A phenomenological relation between the steel composition and grain-coarsening temperature, T_c , (defined as the temperature above which abnormal grain growth commences) of microalloyed steels has been derived from the experimental data for the similar Al steels [33]. Under the conditions of these experiments undertaken between 950 °C and 1250 °C, a linear increase in the observed grain-coarsening temperature, T_c , with T_s was found, that is,

$$T_c = A + B \left(\frac{K}{F - \log(M \cdot C)} - 273 \right), ^\circ\text{C} \quad (3.4)$$

here A and B are the intercept and slope of the line segments for the curve of observed

grain-coarsening temperature and the temperature for complete solution of carbides or nitrides of the microalloyed elements. For AlN, A and B were given as 385 and 0.535 respectively. Thus for this study, T_c is 1020 °C, and the grain-coarsening temperature range is 1020-1180 °C.

According to Table 3.2, the AGG occurred in the non-banded sample at 1100 °C which falls between T_c and T_s estimated above and the grain growth behavior can be well explained by the typical “normal-abnormal-normal” grain growth mode. The grain-coarsening temperature of the non-banded sample was found to be 1050 °C by searching the minimum temperature at which AGG occurs (see Fig. 3.10). Thus, the measured grain-coarsening temperature is very close to $T_c = 1020$ °C estimated from Eq. (3.4). What is the most important here is that the grain-coarsening temperature of the banded sample is quite low (lower than 900 °C) compared with that in the non-banded sample. This point is discussed in more detail in the next section.

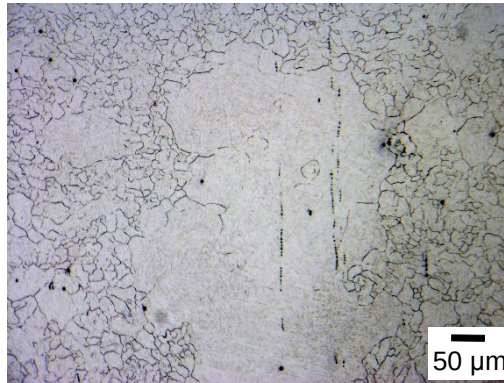


Fig. 3.10. Prior austenite grain structure of non-banded sample held at 1050 °C for 1 min.

3.3.3 Origin of abnormally grown grain from banded structure

In order to make a better understanding for the low grain-coarsening temperature in the banded sample, the origin of the abnormal grains has been first investigated. From careful examination of the microstructures shown in Fig. 3.4, it can be noticed that the etching contrast shown in Figs. 3.4(a)-(c) is not uniform. There are the strongly etched regions (dark region as denoted by S) and weakly etched regions

(bright region as denoted by W) and they consist of alternate layers in parallel to the rolling direction of the steel bar. This is similar to the microstructural banding in the mother F/P structure. It is important to note that the large grains as indicated by the black arrows in Figs. 3.4(b) (c) are always located in the weakly etched regions.

The concentration of manganese along the line marked with A and B in Fig. 3.4(a) was examined by EPMA, and the result is shown in Fig. 3.11.

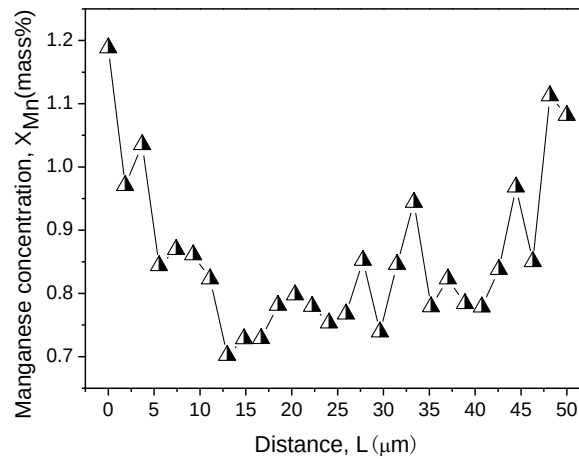


Fig. 3.11. Manganese concentrations along the line AB in Fig. 3.4(a).

From Fig. 3.11, it can be understood that the strongly and weakly etched regions observed in Fig. 3.4 corresponds to manganese rich and lean regions, respectively. It is very important to point out that the manganese profile in the reversely-transformed austenite agrees with the pattern of F/P banded mother structure. It is known that manganese is difficult to be homogenized by a low-temperature heat treatment due to its low diffusion coefficient. It takes around 57.2 h to reduce the compositional amplitudes of manganese from 0.6% to 0.4% between F/P bands at 1100 °C [38]. Therefore, the banded segregation of manganese is very hard to remove in the present experimental condition. Hence, it is clear that the strongly and weakly etched regions correspond to the former pearlite and ferrite bands, respectively. According to the above analysis, abnormal grains are found to originate from the former ferrite regions in the banded sample. This is a very important fact which helps us understand the occurrence of AGG at low temperatures in the banded sample. In the case of the non-banded sample, on the other hand, the nucleation sites of abnormal grains were

found to be randomly distributed by checking the microstructures of the sample annealed at 1050 °C for 1 min (same sample as that shown in Fig. 3.10).

Additionally, it is noted that the austenite grain structure initially consists of fine grains almost uniformly distributed over the observation area (Fig. 3.4(a)). This indicates that the occurrence of AGG is essentially attributable not to the austenite nucleation process during heating but to the grain growth process after the completion of the austenizing.

3.3.4 Possible mechanism analyses

The grain-coarsening temperature of the banded sample is much lower than that of the non-banded sample. Furthermore, it was found that the formation of abnormal grains is always associated with the former ferrite region in the banded structure, in contrast with that of the irregular distributed abnormal grains in the non-banded sample. As described in section 3.1, the segregation in F/P banded structure was completely removed by homogenization at 1300 °C. Hence, the AGG that occurred at lower temperatures in the F/P banded sample has a strong relationship with the inhomogeneity in its microstructures. In the austenite grain structure transformed from the banded F/P structure, there should exist two types of inhomogeneity affecting the grain growth of austenite phase: one is the inhomogeneity of manganese concentration and the other is the inhomogeneity of distribution of AlN particles.

3.3.4.1 Banded segregation of manganese

The segregation of manganese plays an important role in the development of microstructural banding. Enomoto et.al [39] have investigated the segregation of substitutional alloying elements at austenite grain boundaries in five different Fe-0.4 wt pct C-X alloys (where alloys X=Mn, Ni, Co Si, and Mo) and concluded that (1) Mo has the largest grain-boundary enrichment factor, (2) Mn and Si has the intermediate values, and (3) no grain boundary enrichment was observed in Ni and Co. It is known that the segregation of the alloying atoms at grain boundaries yields the

retardation of the grain boundary migration, which is called solute drag effect [40-44] and AGG can originate from this effect [29-30].

The level of segregation of solute atoms at grain-boundaries is generally proportional to the concentration of solute atoms in the bulk [30]. Therefore, if the solute drag effect exists in our samples, this effect might be strong in the former pearlite regions because of relatively high manganese concentration in this region. On the other hand, the solute drag effect should be weak in the former ferrite regions having low manganese concentration. Namely, there might be the non-uniformity in the grain boundary velocity on the scale of the banded structure. Then, this non-uniformity is likely to incur the occurrence of AGG from the former ferrite bands because the velocity of the grain boundary migration is higher in the former ferrite regions due to the low manganese concentration. Therefore, we have analyzed the solute drag effects of manganese segregation on the occurrence of AGG as follows.

According to the theory of solute drag, when the grain boundaries begin to move due to the thermal activation, the grain boundaries have to drag the segregated solute atoms to move together [41]. It was reported that this interaction between solute atoms and the boundary is affected by the difference in atomic size between the solvent and solute elements and the degree of the grain boundary segregation [40, 45]. Since Fe and Mn are neighboring elements in the periodic table, the difference in atomic size between them is only 0.001 nm and is very small compared with that between Fe and other alloy elements, like 0.013 nm between Fe and Mo, and 0.019 nm between Fe and Nb [46]. Furthermore, as reported by Enomoto et.al [39] that the tendency of Mn segregation to grain boundary is not very strong. These indicate that Mn atoms may not have a strong effect on the austenite grain boundary movement.

An empirical equation of the austenite grain boundary mobility considering the alloy elements effects was proposed [47] and can be also used to estimate the effect of manganese concentration on the austenite grain boundary mobility in this study. The mobility of grain boundary is defined as [48]:

$$M = M_0 \exp(-Q/RT) \quad (3.5)$$

where M_0 denotes a kinetic constant that describes the grain boundary mobility, and it was assumed to be $4 \times 10^{-3} \text{ (m}^2\text{s}^{-1}\text{)}$ [49], T is the absolute temperature, R is the gas constant, and Q is the apparent activation energy for grain growth, which can be evaluated by the following empirical relationship [47]:

$$Q = 167686 + 40562 \cdot c_p \quad (3.6)$$

where c_p is the equivalent carbon content, which can be calculated by using a simplified form of the formula proposed by Howe [50]:

$$c_p = \text{mass\%C} - 0.14 \cdot \text{mass\%Si} + 0.04 \cdot \text{mass\%Mn} \quad (3.7)$$

By employing Eqs. (3.5) to (3.7), the austenite grain boundary mobility was calculated and the results are summarized in Table 3.3. It can be seen that the difference in grain boundary mobility between manganese rich and lean regions is nearly negligible. This indicates that the austenite grain boundary has almost the same mobility regardless of manganese concentration difference between the former ferrite and pearlite regions in the banded sample. Therefore, the occurrence AGG in the banded sample at low temperatures should not be attributed to the banded segregation of manganese.

Table 3.3 Austenite grain boundary mobility ($\times 10^{-11}$) at different manganese concentrations

Manganese, mass%	900 °C	1000 °C
0.6	6.30	25.83
1.4	5.44	22.57

3.3.4.2 Non-uniform distribution of second-phase particles

In this study, the occurrence of AGG in the non-banded sample was well explained by the dissolution of AlN particles. In the case of the banded sample, it was observed that the grain-coarsening temperature is quite low (lower than 900 °C) compared with that in the non-banded sample. Additionally, the abnormal grain always originates from the former ferrite regions. This fact may indicate that the pinning effect is non-uniform between the former ferrite and pearlite regions. Thus,

some grains may grow preferentially in the weaker pinning region. It causes the formation of coarse grains and the occurrence of AGG. To confirm this, the size and volume fraction of AlN precipitates in the ferrite and pearlite regions were separately examined by analysis of TEM-micrographs of the carbon extraction replica taken from the banded and non-banded samples. In this study, the Ac_1 temperature (the temperature at which ferrite begins to transform to austenite) was measured to be 740 °C. In order to know the distribution of AlN precipitates in the ferrite and pearlite regions just before the ferrite-to-austenite transformation, both samples were reheated to 735 °C and held for 0 and 10 min, and then air cooled to room temperature. Very fine AlN particles (diameter, $d < 10$ nm) were observed on the replica of the non-banded sample annealed at 735 °C for 0 min. It was reported that the precipitates smaller than 5 nm could not be reliably extracted [51]. Therefore, the AlN precipitates in the sample annealed at 735 °C for 0 min may be too fine to be reliably detected. While, after held for 10 min, the AlN particles grew into relatively bigger ones ($d \geq 10$ nm) which can be reliably detected. Therefore, the samples held at 735 °C for 10 min were used to evaluate the size and volume fraction of AlN precipitates just before the transformation. Some examples of the AlN precipitates observed on the replicas of both the banded and non-banded samples after held at 735 °C for 10 min are shown in Fig.3.12.

The volume fraction of AlN precipitates from carbon extraction replica can be calculated based by the McCall-Boyed method [52] as follows:

$$f = \left(\frac{1.4\pi}{6} \right) \left(\frac{ND^3}{V} \right) \quad (3.8)$$

where N is the number of the precipitates, D is the mean diameter of the particles and V is the volume of the matrix from which the precipitates were extracted. The thickness of the matrix can be assumed as the mean diameter of the particles [53]. Thus, Eq. (3.8) can be written as

$$f = \left(\frac{1.4\pi}{6} \right) \left(\frac{ND^2}{S} \right) \quad (3.9)$$

where S is the area of the corresponding matrix.

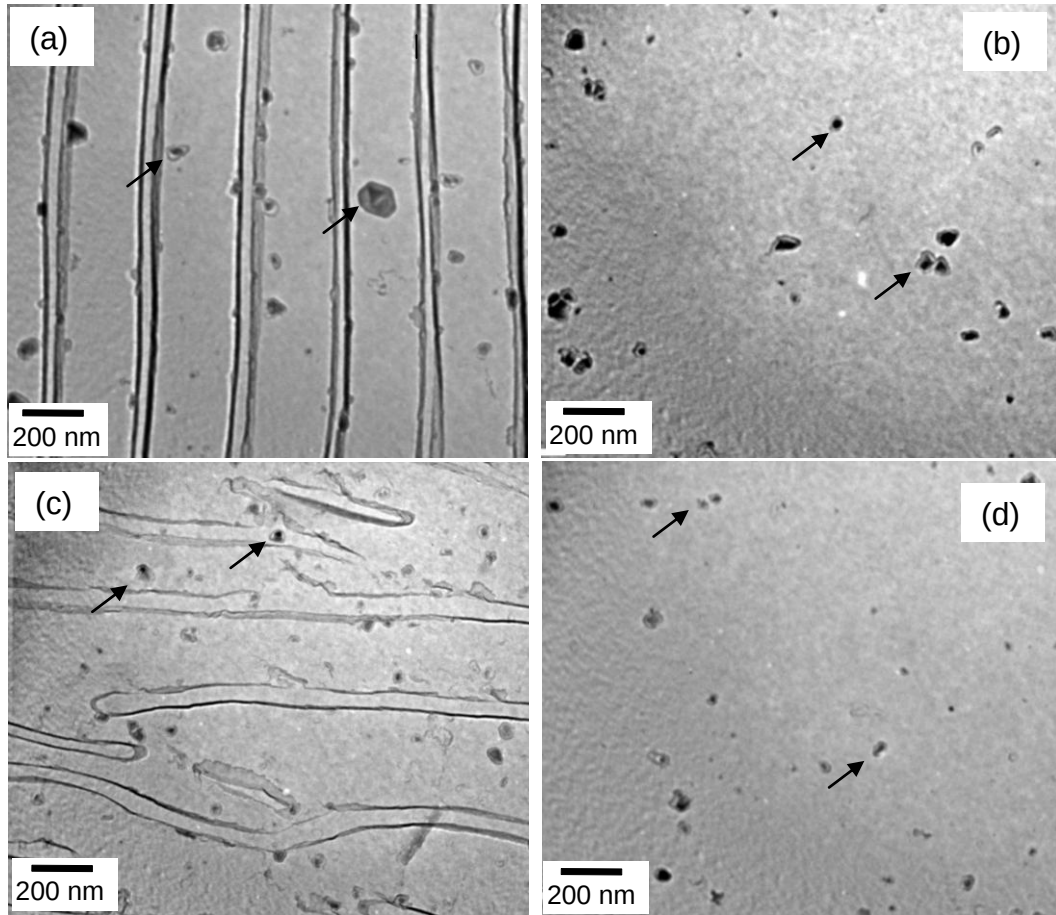


Fig. 3.12. TEM images of carbon extraction replicas showing the precipitates (indicated by arrows) distribution in: (a) pearlite (b) ferrite phase of banded sample and (c) pearlite (d) ferrite phase of non-banded sample after held at 735 °C for 10 min.

The measured precipitates size (r) and volume fraction (f) of AlN for the ferrite and pearlite regions in both the banded and non-banded samples are summarized in Table 3.4. According to the Zener pinning theory [54], the pinning force (F_p) exerted by a random particle distribution of mean radius (r) and volume fraction (f), is given by

$$F_p = \frac{3f\gamma}{2r} \quad (3.10)$$

where γ is the grain boundary interfacial energy. Thus, the pinning force is proportional to f/r . If we assume that the size and volume fraction of AlN precipitates do not substantially change during the reverse transformation, the pinning force in the former ferrite and pearlite regions can be evaluated by using the values shown in Table 3.4. In order to compare the pinning effect between ferrite and pearlite regions,

f/r in each region is displayed in Table 3.4. It can be seen that the value of f/r in the former pearlite region is about two times larger than that in the former ferrite region in the banded sample. This clearly indicates that in the banded sample the pinning effect is two times stronger in the former pearlite region than in the former ferrite region, which indicates that grain growth is two times easier in the former ferrite region. As for the non-banded sample which experienced homogenization at 1300 °C above the predicted dissolution temperature 1180 °C of AlN (at lower than this temperature non-banded structure cannot be produced), the AlN precipitates were completely dissolved and re-precipitated during the air cooling or reheating process.

Table 3.4 Precipitates sizes and volume fractions for the ferrite and pearlite regions

Sample	Average size, $r(\text{nm})$		Volume fraction, $f(\times 10^{-4})$		$f/r(\times 10^{-4} \text{ nm}^{-1})$	
	Ferrite	Pearlite	Ferrite	Pearlite	Ferrite	Pearlite
Banded	15.0	10.0	5.79	7.37	0.39	0.74
Non-banded	7.5	5.0	1.64	1.4	0.22	0.28

From Table 3.4 in can be seen that the values of f/r are almost the same in both regions, thus uniform pinning effect exists in the non-banded sample. Furthermore, according to the empirical relationships regarding the mean austenite grain size, volume faction and average radius of the pinning particles given by

$$R_{crit} = \phi \frac{r}{f} \quad (3.11)$$

where R_{crit} is the critical mean austenite grain size and ϕ is a constant. It can be seen that the non-uniform pinning-effect may lead to the average austenite grain size in former ferrite regions about two times larger than that in former pearlite regions, in the banded sample. Hence, it causes the heterogeneity in grain size distribution. It was reported that AGG takes place when the heterogeneity parameter Z , defined as the ratio of the size of a growing grain to the size of the neighboring grains, is larger than 2 [55]. Therefore, AGG will take place from the former ferrite region, which is consistent with what we observed in Fig. 3.4. As for the non-banded sample, on the other hand, the resultant average austenite grain size may be the same in both regions,

due to the uniform pinning effect. Therefore, the non-uniform pinning effect between former ferrite and pearlite regions is proposed as the reason for the onset of AGG at quite low temperatures in the banded sample.

3.4. Conclusions

The grain growth behavior of austenite reversely transformed from ferrite/pearlite (F/P) banded and non-banded steels has been studied and the important findings are summarized as follows:

- (1) Grain coarsening due to the abnormal grain growth was observed. The grain-coarsening temperature is quite low in the F/P banded sample compared with that in the non-banded sample.
- (2) In the F/P banded sample, the abnormal grains always originate from former ferrite regions. The occurrence of abnormal grain growth is essentially attributable not to the austenite nucleation process during heating but to the grain growth process after the completion of the austenizing.
- (3) Non-uniform pinning effect of AlN precipitates between ferrite and pearlite regions is proposed as the reason for the lowered grain-coarsening temperature in the banded sample rather than the banded segregation of manganese.

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Chapter 4

Increase of austenite grain coarsening temperature in banded ferrite/pearlite steel by cold deformation

4.1. Introduction

The occurrence of Abnormal Grain Growth (AGG) of austenite during reheating process is a serious problem in steels with carbides and/or nitrides. AGG in austenite grain structure during manufacturing processes should be avoided because the mechanical properties of steels exhibiting a bimodal austenite grain size distribution caused by AGG are degraded [1-5]. Grain coarsening temperature, T_c , is defined as the temperature above which abnormal grain coarsening commences [6]. The T_c of steels during reheating for thermal processing is an important factor in achieving fine-grained products. To increase the T_c is an effective way to increase the stability of the steels against abnormal grain coarsening during thermal processing [7].

There are numerous studies about the austenite grain growth behavior in many types of microalloyed steels. While AGG takes place in all the steels alloyed with Al, Ti, V, and Nb during the austenitizing treatment [6], T_c is quite different depending on the type and concentration of microalloying elements [6]. In the author's previous study, as discussed in the last chapter, it was found that the T_c of the initially ferrite/pearlite (F/P) banded steel is about 900 °C which is quite lower than that of the non-banded steel (1050 °C). Namely, the F/P banded steel is very susceptible to abnormal grain coarsening during reheating. It was proposed that the lowered T_c in the F/P banded steel is due to the non-uniform pinning-effect of AlN precipitates between former ferrite and pearlite regions [8].

Furubayashi et al. [9] investigated the effect of cold-deformation on the precipitation behavior of second phase particles. They concluded that the AlN precipitates are made very fine and distributed uniformly by cold deformation. This suggests that it may be possible to eliminate the nonuniformity of AlN in the F/P banded steel, thus to increase the T_c by applying cold deformation prior to

austenitizing. Hence, the purpose of this study is to clarify the effect of cold deformation on the T_c of the steel with F/P banded structure. In this study, it is shown that the low T_c of the F/P banded steel compared with non-banded steel is greatly increased by applying cold deformation prior to austenitizing. Furthermore, the severity of abnormal grain coarsening above the T_c is also largely reduced by the cold deformation.

4.2. Experimental

A hot rolled bar of a hypoeutectoid steel (0.2C-0.2Si-0.8Mn-0.015P-0.04Al-0.006N, mass %) was used in this study. To remove the residual stress in the as-received steel bar, the samples were normalized at 900 °C for 30 min in air and then furnace-cooled to room temperature. All specimens were taken from the same bar. Some of the samples were cold-rolled by 10 to 50% reductions before the austenitizing treatment. The samples were austenitized for 30 min in an argon atmosphere at temperatures between 900-1200 °C. The heating rate of the samples was approximately 5 °C/s. After the austenization, the samples were immediately quenched into an iced-water bath. Longitudinal sections of the quenched samples were observed by an optical microscope after grinding and polishing followed by chemical etching with supersaturated picric acid at 60 °C for 40 s (to reveal the prior austenite grain boundaries). A linear intercept method was employed to obtain the average grain size. The grain size distributions were plotted in area-percent as a function of grain size range. The grain size distributions were investigated to identify the severity of AGG.

The microanalysis of AlN precipitates was conducted by using transmission electron microscopy (TEM) examination on carbon extraction replicas taken from the samples. The method for carbon extraction replica preparation was described elsewhere [8]. The observations and analyses were performed at an operating voltage of 200 kV in a JEOL JEM-2010 TEM equipped with an energy dispersive

spectroscopy (EDS). The size of the particles were measured either directly on the screen at a suitable magnification or by measuring enlarged micrographs. A minimum of 1000 particles were measured for each case to give the mean particle size.

4.3. Results and discussion

Figure 1 shows the effect of the reduction ratio on T_c . It can be noticed that T_c increases with the increase in reduction ratio up to 30% and then it reaches a constant value of 1050 °C which equals that of the non-banded steel. This strongly indicates that the low T_c of the F/P banded steel can be increased significantly by the cold deformation.

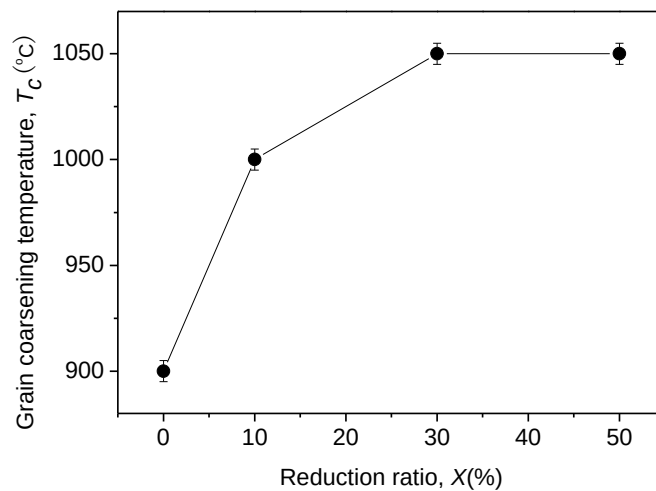


Fig. 4.1. Effect of reduction ratio on the austenite grain coarsening temperature.

Figure 4.2 shows the optical microstructures of the 30%-deformed and undeformed samples austenitized in the range of 1000-1100 °C for 30 min. The same as the previous finding in last chapter that at 1000 °C the undeformed sample (Fig. 4.2(a)) displays extremely coarse grain structure which is resulted from AGG. While the grains in the cold deformed sample (Fig. 4.2(b)) are fine and uniform. This clearly indicates that the AGG that occurred at lower temperatures in the F/P banded steel can be effectively inhibited by the cold-working prior to austenitizing.

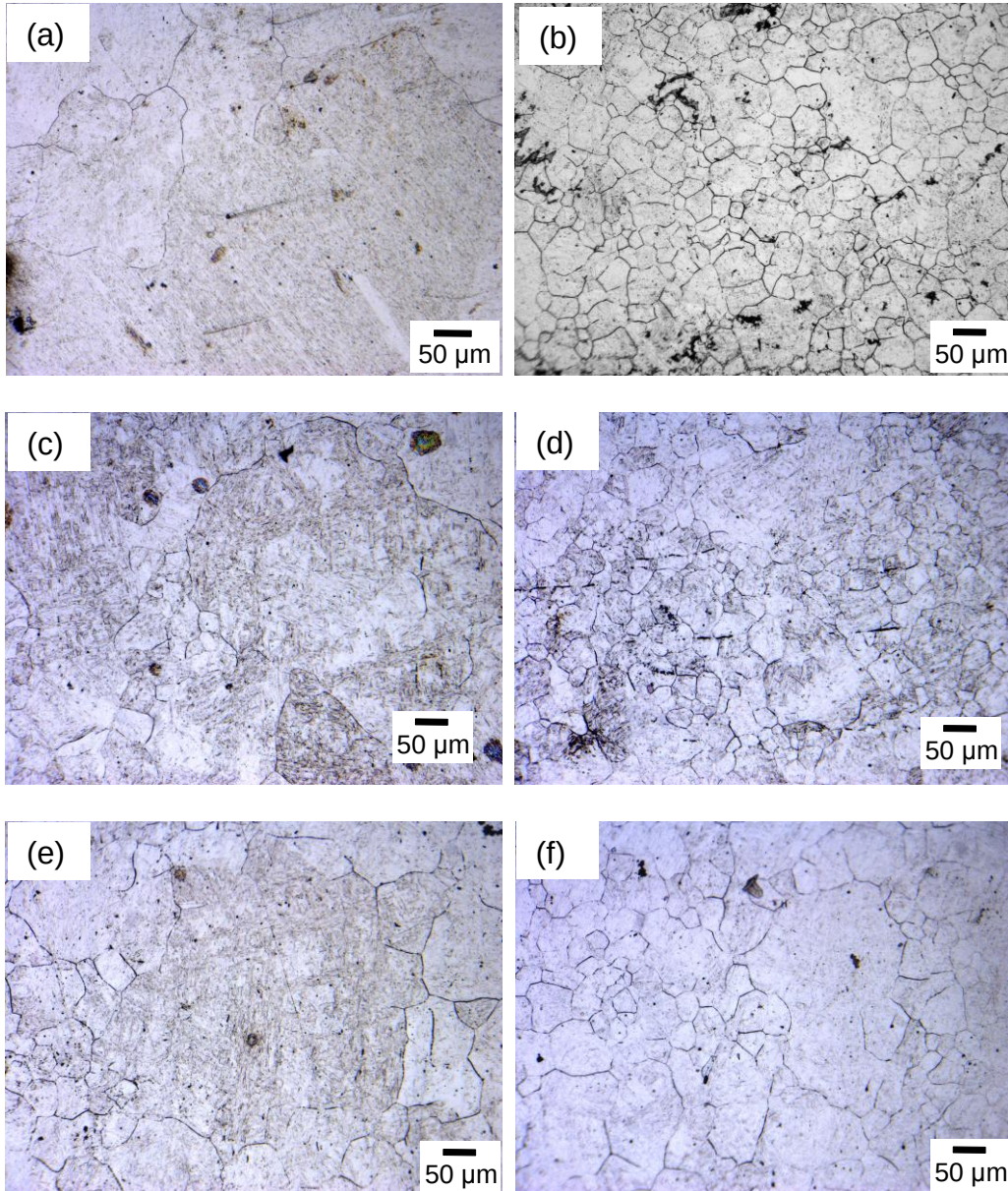


Fig. 4.2. Microstructures after austenitizing for 30 min at 1000 °C in (a) undeformed and (b) 30% deformed, 1050 °C in (c) undeformed and (d) 30% deformed and 1100 °C in (e) undeformed and (f) 30% deformed samples.

At 1050 °C, mixed grain structure are observed in both the samples, as shown in Figs. 4.2(c) and (d). The extent of AGG in 30%-deformed sample seems not as severe as that in the undeformed sample, according to Figs. 4.2(c) and (d). To compare the severity of AGG in the both samples quantitatively, the grain size distribution for both the samples were examined and the results are shown in Fig. 4.3. It can be seen that the extent of abnormal grain coarsening in 30% deformed sample is far less severe than that in undeformed sample. Same phenomenon is observed when held at 1100 °C

for 30 min, according to Figs. 2 (e) and (f).

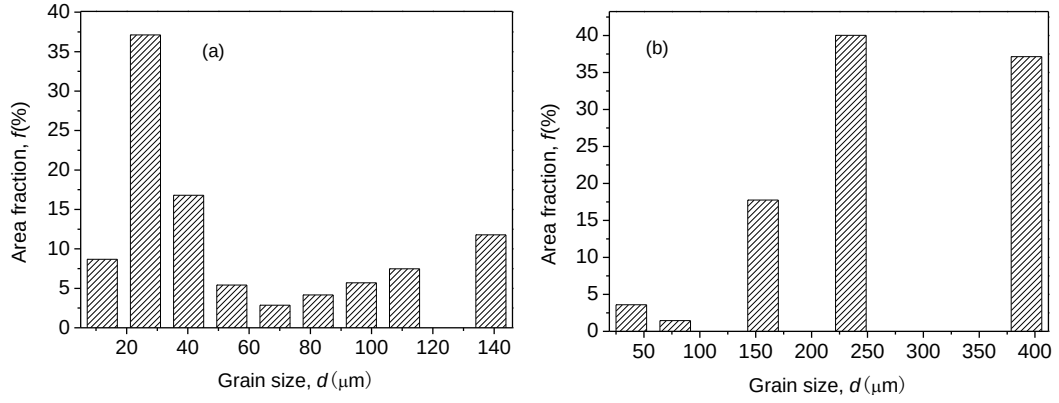


Fig. 4.3. Prior austenite grain size distributions of (a) 30% deformed and (b) undeformed samples held at 1050 °C 30 min. Large size grains accounted for higher area fractions indicates the severer extent of AGG.

Hence, the lower T_c (900 °C) in the F/P banded steel is greatly increased by the cold deformation. The AGG that occurred below 1050 °C can be completely inhibited by cold deformation. Furthermore, the severity of AGG at temperatures above T_c is also largely reduced by the cold deformation. These indicate that AGG in the F/P banded steel can be well inhibited by applying cold deformation prior to austenitizing.

In order to understand the mechanism of the effect of cold deformation on T_c of the F/P banded steel, the distribution and morphology of AlN precipitates in the pearlite and ferrite regions in the 30%-deformed sample after held at 735 °C for 10 min (the same condition with that of Chapter 3) were examined and the results are shown in Fig. 4.4. It visually shows that the AlN particles are fine and uniformly distributed between ferrite and pearlite regions. The volume fraction of AlN precipitates from carbon extraction replica can be calculated by the following equation [10-11]:

$$f = \left(\frac{1.4\pi}{6} \right) \left(\frac{ND^2}{S} \right) \quad (4.1)$$

where N is the number of the precipitates, D is the mean diameter of the particles and S is the area of the matrix from which the precipitates were extracted.

The measured precipitates radius (r) and volume fraction (f) of AlN in the ferrite

and pearlite regions are summarized in Table.4.1. It can be seen that the AlN particles are uniformly distributed between ferrite and pearlite regions in the 30% deformed sample. According to the Zener pinning theory [12], the pinning force is proportional to f/r . In order to compare the pinning effect between ferrite and pearlite regions, f/r in each region is displayed in Table 4.1. As for the undeformed sample, the pinning effect of AlN precipitates is quite different between the ferrite and pearlite regions, which was proposed to be the reason for the lowered T_c in the banded sample [8]. As for the 30%-deformed sample, on the other hand, the values of f/r are almost the same in both regions viz., the pinning effect is uniform thought the sample. Hence, the AGG that occurred at low temperatures (below 1050°C) in the F/P banded steel due to the non-uniform pinning effect can be completely inhibited by the cold deformation. And T_c can be increased up to the same value as that of the non-banded steel which should be determined by the stability of AlN precipitates at elevated temperatures.

As for the effect of cold deformation on the AGG of F/P banded steel above T_c , it is possible that some large grains might be formed below 1050 °C during the continuous heating process in the undeformed sample. These large grains with the size advantage may grow preferentially during holding at 1050°C. As for the deformed sample, on the other hand, the AGG is developed from uniform grain matrix during holding at 1050 °C. Thus, the AGG kinetics may be lower than that of the undeformed sample. Therefore, the extent of the AGG in the 30%-deformed sample is severe than that of the undeformed sample. There may be also other possibilities of the effect of AlN particle size distribution on the AGG kinetics, which needs to be further confirmed.

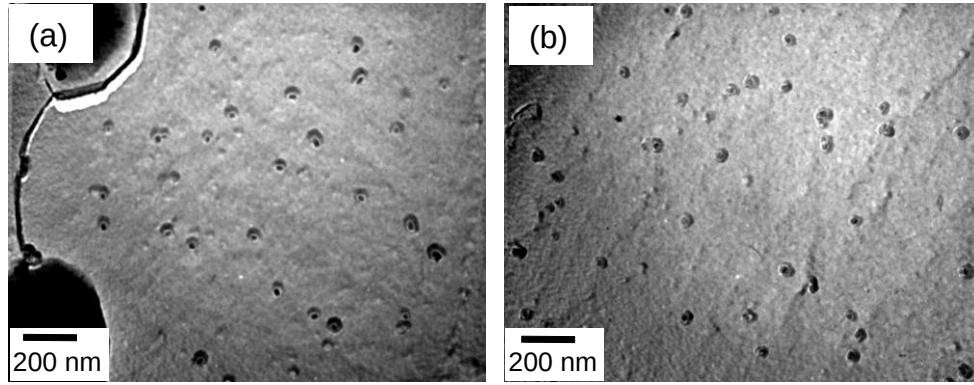


Fig. 4.4. AlN particles in the 30% deformed sample held at 735 °C for 10 min: (a) pearlitic ferrite (b) ferrite.

Table 4.1 Precipitates sizes and volume fractions for the ferrite and pearlite regions

Sample	Average size, $r(\text{nm})$		Volume fraction, $f(\times 10^{-4})$		$f/r(\times 10^{-4} \text{ nm}^{-1})$	
	Ferrite	Pearlite	Ferrite	Pearlite	Ferrite	Pearlite
Undeformed	15.0	10.0	5.79	7.37	0.39	0.74
30% deformed	8.5	8.5	7.34	7.51	0.86	0.88

According to the above analysis, cold deformation is effective in controlling the AGG in the F/P banded steel. It is well known that cold plastic deformation introduces high densities of dislocations into the steels. The dislocations may accelerate the diffusion rate of alloying elements during reheating making the Al distributed more uniformly. Moreover, the dislocation loops introduced by the cold deformation which act as nucleation sites of the precipitation made the AlN precipitates very fine and distributed uniformly [9].

4.4. Conclusions

In summary, this study of effect of cold deformation on the grain coarsening temperature, T_c , in ferrite/pearlite (F/P) banded steel has shown that T_c is greatly increased by applying cold deformation prior to austenitizing. The abnormal grain growth that occurred in the F/P banded steel at low temperatures compared with non-banded steel is completely inhibited by the cold deformation. The extent of abnormal grain coarsening at temperatures above T_c is also largely decreased by the cold deformation. This is attributed to the homogeneous distribution of AlN precipitates caused by the cold deformation.

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Chapter 5

Effect of cold deformation on the abnormal grain coarsening behavior of austenite in non-banded steel

5.1. Introduction

Inhibition of austenite grain growth above the eutectoid temperature in steels is of great importance in the steel production processes. The presence of second phase particles substantially changes the grain growth behavior of austenite. Early attempts of grain refinement have centered on the formation of AlN precipitates by the addition of Al and N in steels [1]. The other additives such as Ni, V, Ti, and Nb have also been shown to lead to the grain refinement due to the formation of carbides or nitrides [2-5]. At lower temperatures, the precipitates are stable and the austenite grain boundaries can be effectively pinned and the grain growth is thus suppressed. At elevated temperatures, however, the second phase particles are unstable and they readily coarsen or dissolve [6]. Then, unpinning might occur and the grain growth becomes abnormal. During the abnormal grain growth (hereafter, referred to as AGG) a few large grains grow discontinuously and consume the neighboring matrix of smaller grains, which results in the development of extraordinarily large grain [7]. There exists a critical temperature above which the grain growth kinetics changes from normal mode to abnormal mode. This temperature is called the grain-coarsening temperature, T_c . Furthermore, when the temperature is much higher than T_c , normal grain growth (hereafter referred to as NGG) again takes place due to the nearly complete dissolution of pinning particles [6]. There also exists a critical temperature above which the grain growth kinetics changes from abnormal mode to normal mode. This temperature is called the AGG finishing temperature, T_f . Therefore, AGG occurs in a finite range of temperature which is defined as AGG temperature-range. AGG temperature range of steels during reheating is an important factor in the design of thermo-mechanical processing to achieve fine-grained product, which is quite worth

to be investigated.

There are numerous studies about the austenite grain growth behavior in many types of microalloyed steels. While AGG takes place in all the steels alloyed with Al, Ti, V, and Nb during the austenitizing treatment [8], T_c is quite different depending on the type and concentration of microalloying elements [8]. Since the coarsening of austenite grains is dependent on the presence (or absence) of the precipitates, any factors which affect the presence of the precipitates might have some effects on the AGG temperature range of austenite. Plastic deformation is a well-known factor affecting the precipitation kinetics. Hot deformation was found to have great effects on the coarsening behavior of precipitates, since the presence of dislocations introduced by the hot deformation causes a significant acceleration in nucleation, growth and coarsening of the precipitates [9-11]. Furubayashi et al. [12] investigated the effect of cold deformation on the precipitation behavior of second phase particles. They concluded that the precipitates are made very fine and distributed uniformly by cold deformation. Both Furubayashi et al. [12] and Kesternich [13] found that the cold deformation made the precipitates resist to coarsening at lower temperatures (below 900 °C). Hence, the cold deformation should affect the AGG temperature-range of austenite. In the previous study as discussed in Chapter 3 and 4 it was found that the T_c of the initially ferrite/pearlite (F/P) banded steel is about 900 °C which is quite lower than that of the non-banded steel (1050 °C). And the low T_c of the F/P banded steel can be increased significantly by applying cold deformation prior to austenitizing. However, it is unclear whether or not the cold deformation has effects on the AGG of austenite in non-banded steel. Therefore, the aim of this study is to clarify the effect of cold deformation on the AGG of austenite in non-banded steel.

5.2. Experimental

A hot rolled bar of a carbon steel (0.2C-0.2Si-0.8Mn-0.015P-0.04Al-0.006N, mass %) was used in this study. The samples were homogenized at 1300 °C for 2 h in an argon atmosphere and then air-cooled to room temperature to obtain the

non-banded structure. All specimens were taken from the same bar. After the homogenization at 1300 °C for 2 h the AlN precipitates should be completely dissolved and there may be only a slight amount of AlN reprecipitated during the subsequent air-cooling process, due to the difficult nucleation of AlN in steel [14]. In the steel of our focus, AlN should precipitate at temperatures below 800 °C according to the precipitation-time-temperature (PTT) diagram, and the nose precipitation temperature is at around 750 °C [15]. To obtain complete precipitation of initial AlN precipitates, some of the homogenized and air-cooled samples were additionally annealed at 750 °C for 10 h.

Some of the samples were cold-rolled by 10 to 50% reductions before the austenitizing treatment. The disk-shaped samples (10 mm in diameter and 5 mm in thickness) were austenitized for 30 min in an argon atmosphere at temperatures between 900-1200 °C. The heating rate of the samples was approximately 5 °C/s. After the austenization, the samples were immediately quenched into the iced-water bath. Longitudinal sections of the quenched samples were observed by an optical microscope after grinding and polishing followed by chemical etching with supersaturated picric acid at 60 °C for 40 s (to reveal the prior austenite grain boundaries). A linear intercept method was employed to obtain the average grain size. The microanalysis of AlN precipitates was conducted by using transmission electron microscopy (TEM) examination on carbon extraction replicas taken from the samples. The observations and analyses were performed at an operating voltage of 200 kV in a JEOL JEM-2010 TEM equipped with an energy dispersive spectroscopy (EDS).

5.3. Results and discussion

5.3.1 Austenite grain coarsening behavior

The austenite grain sizes at each austenitizing temperature in the homogenized and deformed 50% (HD 50%) and undeformed (HU) samples are shown in Fig. 5.1. Figure 5.2 displays some examples of the austenite grain structures of the HD 50% samples held at different austenitizing temperatures.

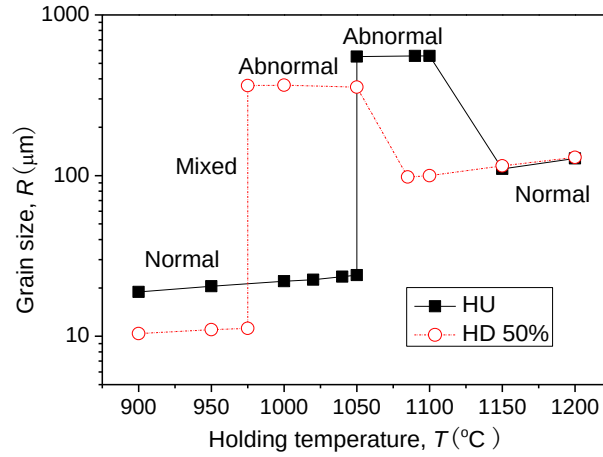


Fig. 5.1. Austenite grain size of the samples held at various austenitizing temperatures for 30 min. HU: Homogenized and Undeformed sample, HD 50%: Homogenized and 50%-Deformed sample.

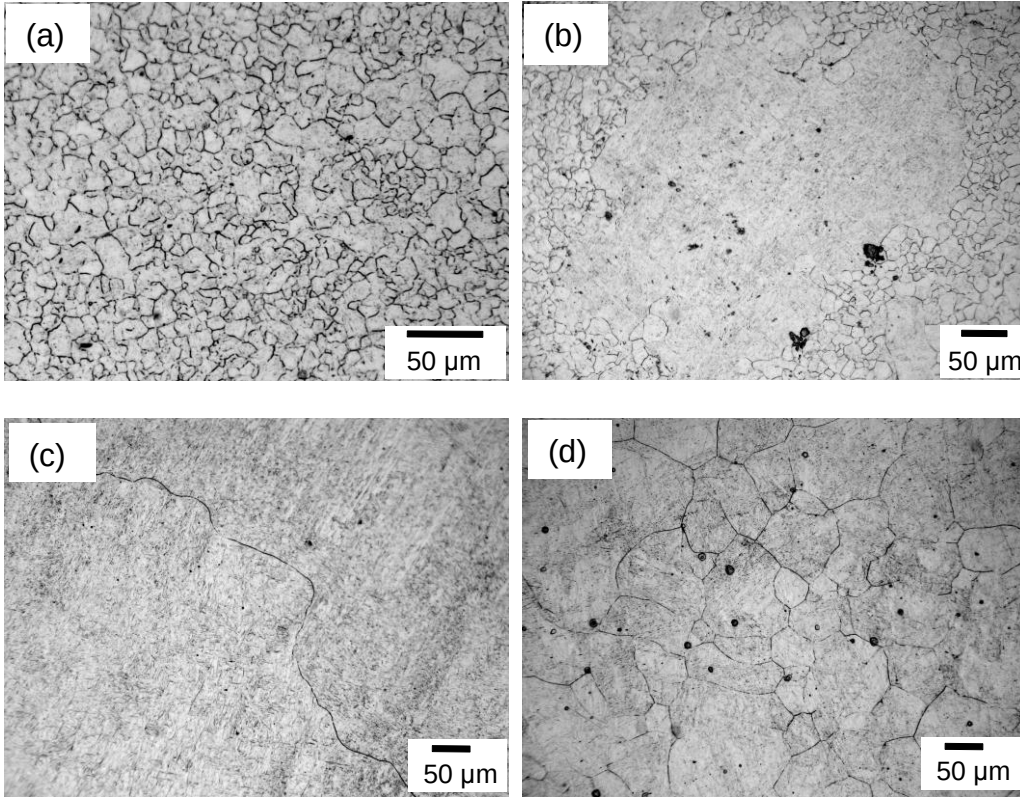


Fig. 5.2. Prior austenite grain structures of HD 50% samples held at (a) 900 °C for 30 min, (b) 975 °C for 15 min, (c) 975 °C for 30 min and (d) 1080 °C for 30 min.

Figure 5.1 clearly shows three distinct grain growth modes in both the samples: (1) at low temperatures, the NGG occurs and the grains are fine and uniform as shown in Fig. 5.2(a); (2) at the critical temperatures (1050 °C for HU and 975 °C for HD 50%) the grain size abruptly increases, as exemplified by extraordinary coarse grain

structure in Fig. 5.2(c); (3) at temperatures much higher than this critical temperature (1150 °C for HU and 1080 °C for HD 50%), a slightly coarse but uniform grain structures form (Fig. 5.2(d)). Hence, both the samples experienced the typical “normal-abnormal-normal” grain growth mode as indicated in Fig. 5.1. T_c in HU and HD 50% samples corresponds to 1050 and 975 °C, respectively. In Fig. 5.1, two plots were used at T_c to signify the occurrence of AGG. These lower and higher plots indicate the grain sizes at early and late stages of austenitizing treatment. The grain structure between them (i.e., during holding period) exhibits mixed grain structures, a typical structure of AGG (Fig. 5.2(b)). The AGG temperature ranges of HU and HD 50% samples correspond to 1050-1150 °C and 975-1080 °C, respectively. Therefore, it is found that the cold deformation yields the shift of the AGG temperature range to the low temperature region.

The effect of reduction ratio of the cold rolling on the AGG temperature-range of austenite has been also examined and the results are shown in Fig. 5.3. It can be seen that the AGG temperature range nearly linearly decreases with the increase of the reduction ratio from 10 to 50%.

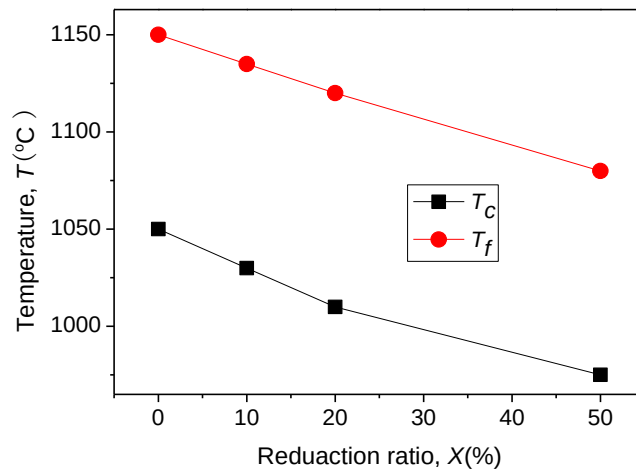


Fig. 5.3. Effect of reduction ratio on the AGG temperature-range of austenite.

To further clarify the effect of cold deformation on the austenite grain coarsening behavior of the samples with complete initial precipitation of AlN, the annealed and deformed 50% (AD 50%) and undeformed (AU) samples were reheated up through

1150 °C to observe the austenite grain structures. The grain growth modes at varying austenitizing temperatures are summarized in Table 5.1.

Table 5.1 Austenite grain growth modes at different austenitizing temperatures for samples additionally annealed at 750 °C for 10 h after homogenization

Temperature, °C	900	1000	1050	1100	1150
AU	NGG	NGG	AGG	AGG	NGG
AD 50%	NGG	NGG	NGG	NGG	NGG

As shown in Table 5.1, the AGG temperature range is not reduced but the AGG is completely inhibited by the cold deformation. This is quite important because it can provide a guideline to the industry process to avoid the occurrence of AGG.

5.3.2 The evolution of AlN particle size and volume fraction

As already mentioned the mechanism of AGG should be partial dissolution or coarsening of the second phase particles upon reheating. In order to understand the mechanism of the effect of cold deformation, the size and volume fraction of AlN precipitates were measured by means of TEM for both HD 50% and HU samples quenched from 800 to 1080 °C. The diameter of AlN particles was assumed to equal the average of the particle length and width, and the method used to evaluate the volume fraction of precipitates was described elsewhere [16]. For comparison, the equilibrium of the volume fraction of AlN precipitates has also been calculated by using Eq. (5.1)

$$\log [Al][N] = 1.03 - 6770/T \quad (5.1)$$

which is the solubility of AlN for practical heat treatment purposes[17]. The results are summarized in Fig. 5.4.

In Fig. 5.4, it can be seen that at 800 °C AlN precipitate is finer and its volume fraction is higher in HD 50% sample as compared with HU sample. The measured volume fraction in HD 50% sample is quite close to the predicted equilibrium value. This indicates that the AlN has been completely precipitated in the HD 50% sample. It was reported that the dislocation loops introduced by the cold deformation acts as the

nucleation sites for AlN [12]. In the steel of our focus, AlN should precipitate in the temperature below 800 °C according to the PTT diagram [15]. Hence, in HU sample, the AlN mainly precipitated in the ferrite region during the reheating process. On the other hand, it was observed in this study that in HD 50% sample the recrystallization of ferrite grains takes place and finishes at around 700 °C during the heating process. Therefore, the AlN precipitation induced by the cold deformation (dislocation loops) should be completed below 700 °C. Namely, the complete precipitation of AlN finished below 700 °C during the heating process.

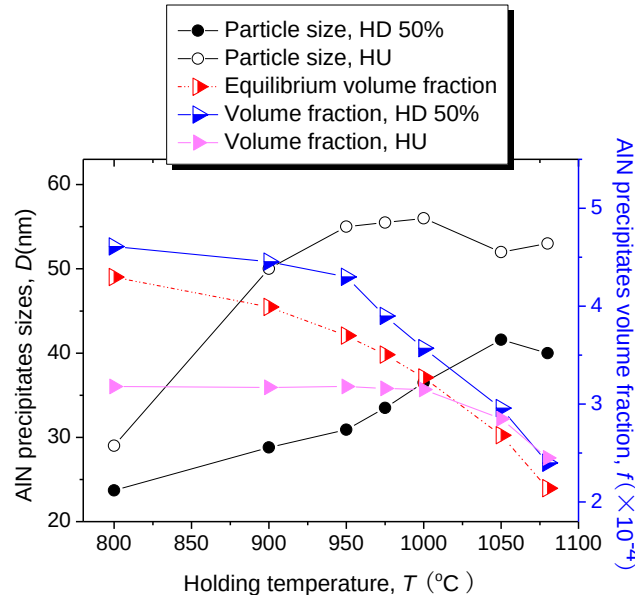


Fig. 5.4. Size and volume fraction of AlN precipitates in the samples held at various austenitizing temperatures for 30 min.

It is worth noting that when the holding temperature increases to 900 °C (just after the reverse transformation), the AlN precipitates in HD 50% sample are slightly coarsened comparing with that in the HU sample. The AlN precipitates in both the samples quenched from 900 °C are shown in Figure 5.5, which clearly shows that the AlN precipitates of HD 50% sample are much finer than that of HU sample. Hence, the precipitates in HD 50% sample exhibit a sluggish coarsening behavior below 900 °C. Similar phenomenon was found by Furubayashi et al. [12] and Kesternich [13]. Although the mechanism(s) for this are still not very clear now.

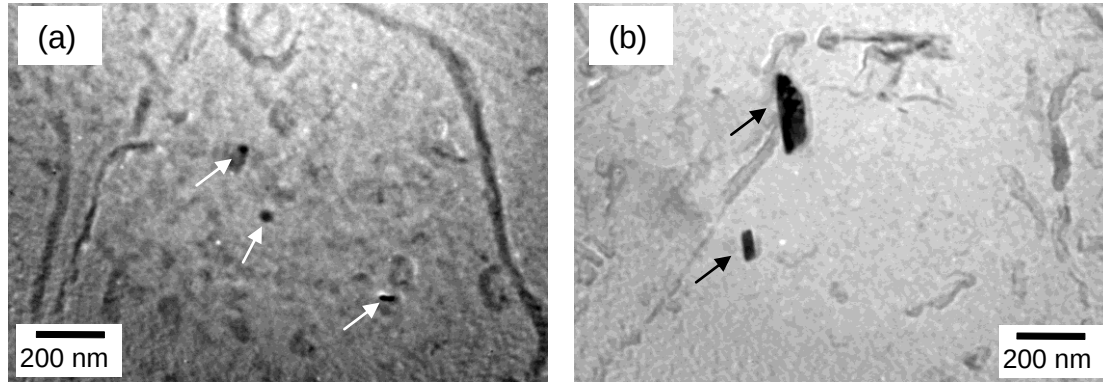


Fig. 5.5 TEM images of carbon extraction replicas showing the precipitates (indicated by arrows) distribution in: (a) HD 50% and (b) HU samples held at 900 °C for 30 min.

As shown in Fig. 5.4, up to 950 °C, the equilibrium volume fraction does not substantially changes. Since the diffusion rate of the alloying elements is low [14], and the holding time is limited, there is nearly no change in the volume fraction of AlN precipitates in the HD 50%. Here it can be noticed that the size of AlN precipitates increases very slightly with temperature below 950 °C. These indicate that the pinning force, which is proportional to f/r (where f and r are volume fraction and mean radius of the precipitates, respectively), keeps a high value. This is reflected in the nearly constant austenite grain size of HD 50% samples below 950 °C, as shown in Fig. 5.1. As for the HU sample, the volume fraction of AlN is lower than the equilibrium value, which is due to the difficulty in AlN precipitation usually found especially below 1000 °C [14]. However, the volume fraction of AlN in HU sample does not change with temperature below 950 °C. Hence, the pinning force keeps a relatively high value also in HU sampl, while due to the lower volume fraction and coarser AlN precipitates the pinning forces should be lower than those in HD 50% sample, which is reflected in the difference in the grain size in Fig. 5.1.

However, it is more important to note that as the holding temperature increases to 975 °C, the volume fraction of the AlN precipitates in HD 50% sample tends to approach the equilibrium and the particles become coarser. At higher holding temperatures, the volume fraction follows the same changing tendency as that of the equilibrium. This can be attributed to the accelerated diffusion rate of the alloying elements at elevated temperature and the readily dissolved fine AlN precipitates in

HD 50% sample. Even though we did not measure the volume fraction of AlN precipitates in HD 50% held at 975 °C for 0 min, it should be close to the data shown at 950 °C in Fig. 5.4, due to the short heating-time interval. Therefore, obvious decrease in volume fraction and accelerated ripening of the AlN precipitates occurred during holding at 975 °C. As for the pinning force, there is a critical radius of the second phase particles r_{crit} for grain unpinning, which linearly decreases with the decreasing volume fraction of the precipitates [7]. Hence, the volume fraction of AlN particles available to retardation of the grain growth may decrease drastically. This may lead to the unpinning and the occurrence of AGG. Furthermore, the fine initial austenite grains in the HD 50% sample have a strong driving force for grain growth and it therefore facilitates the occurrence of AGG.

It is well known that the dislocation densities are proportional to the degrees of deformation. Thus, the dislocation loops which act as the nucleation sites for AlN [12] should decrease with the decrease of reduction ratio. Hence, the precipitation kinetics and thus the volume fraction of AlN should be decreased. This may lead to the linear decreasing T_c with the decrease of reduction ratio as shown in Fig. 5.3.

On the other hand, in HU sample, there is no obvious change in the volume fraction of AlN below 1000 °C as shown in Fig. 5.4. Hence, the pinning force and r_{crit} may have no big change and the austenite grain boundaries can still be effectively pinned. However, when the HU sample is held at 1050 °C, the volume fraction of the precipitates starts decreasing and r_{crit} accordingly decreases. At the same time, the grain boundary mobility becomes high at the elevated temperature. These may cause the onset of the AGG in HU sample at this temperature.

As shown in Fig. 5.4, the volume fractions of AlN precipitates in both samples become very low at 1080 °C. It was found that the AGG onsets at 1 min during holding at 1080 °C in HU sample. The size of AlN precipitates in HU and HD 50% samples at 1080 °C for 0 min were measured to be 57 and 29 nm, respectively. It is known that the time dependence of size of precipitate during dissolution process follows a parabolic law [18]. According to such a parabolic law, the dissolution of AlN precipitates in HU samples should require about four times longer time than that

in HD 50% sample. Hence, it is possible that during the holding process, the fine particles in the HD 50% sample dissolved very quickly which then avoids the occurrence of AGG. However, slow dissolution rate of the coarse precipitates at this temperature may induce the occurrence of AGG in the HU sample. Since lower reduction ratio leads to coarser AlN precipitates [13], this may lead to the increase of T_f with the decreasing reduction ratio as displayed in Fig. 5.3.

Therefore, the cold deformation produced fine and high volume fraction of AlN precipitates. The AlN precipitates in the HD 50% sample exhibit a sluggish coarsening behavior below 900 °C. The volume fraction and size of AlN precipitates have no obvious change below 950 °C, and the austenite grain boundaries can be effectively pinned. However, obvious decrease in volume fraction occurred during holding at 975 °C due to the accelerated diffusion rate of alloying elements and the readily dissolved fine AlN precipitates in the HD 50% sample. This should lead to the unpinning and the occurrence of AGG. Furthermore, the cold deformation produced fine austenite grains which have large driving force for grain growth and thus facilitates the occurrence of AGG. On the other hand, it may be because the quick dissolution rate of the fine precipitates in HD 50% sample avoid the occurrence of AGG during holding at 1080 °C.

In order to understand the mechanism of the effect cold deformation on AGG in the annealed sample, the evolution of the AlN particles at different austenitizing temperatures needs to be further clarified. But, here, based on the results of Fig. 5.4 and the observation of AlN at low temperatures, a primary analysis is carried out. In the HD samples, the AlN precipitates are distributed quite randomly throughout the matrix due to the both the intragranular and intergranular precipitation occurred simultaneously induced by the dislocations. While as for the annealed samples, ferrite grain boundaries are the main nucleation sites for AlN during the intercritical annealing process. After cold deformation, the AlN precipitates distributed along ferrite grain boundaries in the annealed samples are lined up in rows, as shown in Fig. 5.6. Therefore, AlN concentrated in linear arrays on pin the austenite grain boundaries, which are more effective in pinning [6]. Hence, the susceptibility of the annealed and

cold deformed sample to AGG should be decreased. Furthermore, similar to that of HD 50% sample, the cold deformation made the AlN precipitates in the annealed sample resistant to coarsening and uniform in size distribution. Thus, it is possible that they may dissolve into the matrix simultaneously at elevated temperatures. Hence, the AGG due to the coarsening or partial dissolution of the pinning particles can be completely inhibited.

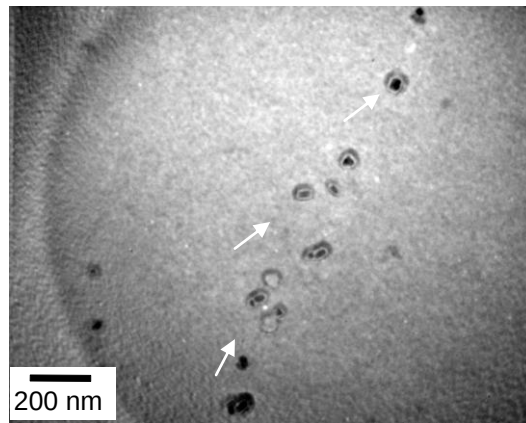


Fig. 5.6. TEM images of carbon extraction replicas showing the AlN precipitates (indicated by arrows) distribution in: AD 50% sample held at 900 °C for 30 min.

According to the above results and discussions, the effect of cold deformation on the abnormal grain coarsening behavior of non-banded steels depends on the initial state of the precipitates. When the non-banded steel with aluminum and nitrogen in solution, cold deformation reduces the AGG temperature range. While the non-banded steels are subjected to a long time subcritical annealing treatment making the AlN completely precipitated and well growth, then the cold deformation can inhibit the AGG.

5.4. Conclusions

In summary, this study of austenite grain coarsening in non-banded steel has shown that the effect of cold deformation on the abnormal grain growth (AGG) of austenite depends on the initial state of precipitates. When the non-banded steel with aluminum and nitrogen in solution, cold deformation reduces the AGG

temperature-range of austenite. While the non-banded steels are subjected to a long time subcritical annealing treatment making the AlN fully precipitated and growth, the occurrence of AGG can be completely inhibited by the cold deformation. This can be explained by the size and spatial distributions of the precipitates affected by the cold deformation.

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Chapter 6

Quantification of local plastic strain distribution and its effect on the austenite grain growth

6.1. Introduction

Several deformation techniques such as forging, ball milling, rolling, shot peening, or hammering have been widely used in industrial processes. Recently, surface severe plastic deformation (S^2PD) techniques have been developed to realize the surface nanocrystallization of bulk engineering components, as exemplified by surface mechanical attrition treatment (SMAT) [1-4], ball-drop [5-6], high energy shot peening [7], surface nanocrystallization and hardening [8-9], etc. All of the above-mentioned manufacturing techniques are characterized with wide ranges of strain and strain rate, and belong to surface local plastic deformation (SLPD). The degree of plastic deformation near surface in the SLPD process is crucial in determining the mechanical and microstructural features of the work-piece surface, because the degree of plastic deformation strongly affects development of grain structure in recrystallization, phase transformation kinetics and the reversely transformed austenite grain growth (as studied in Chapters 4 and 5) during heat treatment processes. In addition, most of material failures originate from the exterior layers of the work piece (fatigue, fretting damage, corrosion, wear, etc.). These phenomena are all extremely sensitive to the structure and properties of the surface of the material. Therefore, it is quite important to investigate the local plastic strain distribution beneath the deformed surface, thus to control the microstructure near the surface.

Although significant effort has been devoted to investigations about the plastic deformation process [10-12], little has been studied for the local plastic strain distribution beneath the deformed surface. Dai et al. [9, 13] studied the effects of kinetic energy of balls on the surface nanocrystallization, the depth of the work

hardened layer and effective plastic strain during surface nanocrystallization process by finite element modeling method. Lee et al. [14] measured the shear strain distribution through thickness of the accumulative roll-bonding processed sheets from the inclination of the pin which was embedded in the sheet before rolling. However, little is known of the extent to which the macroscopic plastic strain and strain rate affect the local plastic strain distribution in the depth direction of the SLPD-processed work piece. Especially, the effects of strain rate on the profile of local plastic strain and the penetration depth of the plastic deformation in the depth direction have not been well clarified yet. In this chapter, a series of experiments were conducted to investigate these issues. Importantly, the local plastic strain distribution on the reversely transformed austenite grain growth is studied.

The investigation of local plastic strain distribution in SLPD is of scientific interest as well as technological importance because it renders understanding of the effects of the macroscopic plastic strain and strain rate on the local plastic strain distribution, local plastic strain gradient and plastic deformation penetration depth (PD) beneath the surface-deformed work piece. Moreover, understanding of these effects will contribute to development of guidelines in controlling deformation parameters (macroscopic plastic strain and strain rate)—during surface plastic deformation processes thus to control the microstructure beneath the surface. Since the indentation depth (which corresponds to macroscopic plastic strain) and strain rates are controllable parameters during SLPD treatment. The present study focuses on the effects of indentation depth and strain rate on local plastic strain distribution in the depth direction of SLPD-processed work piece and the depth of plastically deformed zone. In this study, electron backscatter diffraction (EBSD) analysis is performed to provide quantitative description of the local plastic strain. Pure iron was selected as the test material for the plastic strain measurement due to its simple structure. And 0.2 mass% carbon steel was selected as the test material for austenitizing treatment. It is demonstrated that the profile and the gradient of local plastic strain strongly depend on the indentation depth and strain rate. Deep indentation depth with high strain rates leads to steep and narrow profiles of local

plastic strain distribution. The PD produced by high strain rate is much smaller than that deformed by low strain rate under the same degree of indentation depth. Additionally, the deformation at high strain rates is favorable to impose local plastic deformation on the selected area near the surface. The local plastic strain distribution has a great effect on the reversely transformed austenite grain growth.

6.2. Experimental

The material used for the local plastic strain study is commercial pure iron (Si < 0.01, Mn < 0.01, P < 0.002, S < 0.002 and Al < 0.001 mass%.) with a purity of 99.95 mass%. The as-received pure iron bars were annealed at 1000 °C for 60 min and then slowly cooled to room temperature to obtain homogeneous ferrite grains. All the specimens were taken from the same lot in each case. It was cut into disk-shaped samples with 10 mm height and 30 mm diameter. Before deformation experiments detailed below, the surface of the sample was polished with silicon carbide papers.

In this study, ball-dropping (BD) and ball-pressing (BP) tests were performed to realize the SLPD with high and low strain rates, respectively. The high-strain-rate deformation was carried out by BD test, schematic drawing of which is shown in Fig. 6.1(a). A spherical steel ball attached on the bottom of a cylindrical steel weight was dropped from a height of 2 or 0.5 m onto a flat surface of pure iron sample. The ball had a diameter of 8 mm and the weights of 1.5 to 5 kg. All the tests were carried out at room temperature in air atmosphere. To make the experimental parameters less complicated, single dropping test was performed for each case. The BP test for deformation at low strain rates was carried out by using a computer controlled servohydraulic machine, and its schematic illustration test is shown in Fig.6.1 (b). The speeds of deformation were set to 8.33×10^{-5} (5 mm/min) and 1.67×10^{-6} m/s (0.1 mm/min).

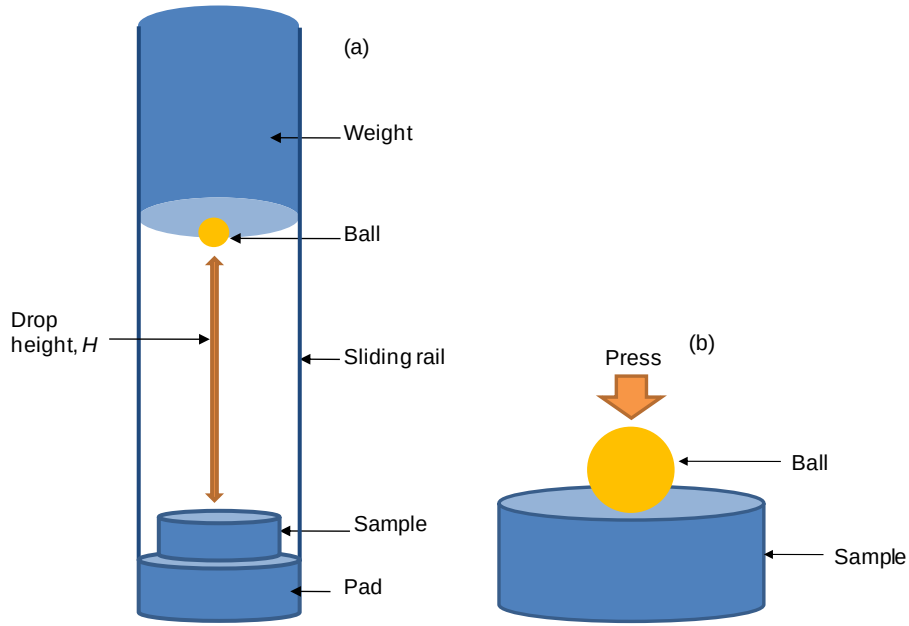


Fig. 6.1. Schematic illustrations of the ball dropping (a) and ball pressing (b) tests.

Both the BD and BP tests produce the concavity of crater-shape on the surface of the sample as schematically shown in Fig. 6.2. The crater depth denoted as d was measured by optical microscopy for each case and the measurement error is about 0.9%. The BD and BP tests were performed so as to produce the craters of $d=1.1, 0.9, 0.57, 0.41$, and 0 mm. It is noted that our focus is the distribution of local plastic strain along the depth direction from the bottom of the crater. As mentioned in the introduction, the strain rate should be one of the factors affecting the local plastic strain distribution. Since it is very difficult to estimate the macroscopic strain rate with high accuracy especially for the BD test, we estimate the value in the following manner, which is sufficient for our purpose. The velocity of the ball just before hitting the specimen was calculated by Eq. (6.1).

$$v_0 = \sqrt{2gH} \quad (6.1)$$

where g is the gravity acceleration and H is the initial height of the ball. The time required for total deformation t can be estimated as

$$t = d/v \quad (6.2)$$

where d is the depth of crater in the deformed sample as shown in Fig. 6.2 and v is the average velocity of the ball during collision, and it was assumed in this study that

$v=0.5v_0$. For example, in the test for $H=2$ m and the resulting depth of $d=1.1$ mm, t is calculated to be 3.5×10^{-4} s. We defined the average plastic strain as follows

$$\varepsilon_{ave} = d / (d + d_p) \quad (6.3)$$

where d_p corresponds to the depth below which the plastic deformation does not occur, as schematically shown in Fig. 6.2. Then, the average strain rate is given by

$$\dot{\varepsilon} = \varepsilon_{ave} / t \quad (6.4)$$

The estimation method for d_p in Eq. (6.3) will be discussed later. The strain rates thus estimated are summarized in Table 6.1. For convenience, the strain rates of 6.8×10^2 and $3.1 \times 10^2 \text{ s}^{-1}$ are called high strain rates, while 1.3×10^{-2} and $2.4 \times 10^{-4} \text{ s}^{-1}$ are called low strain rates in this report.

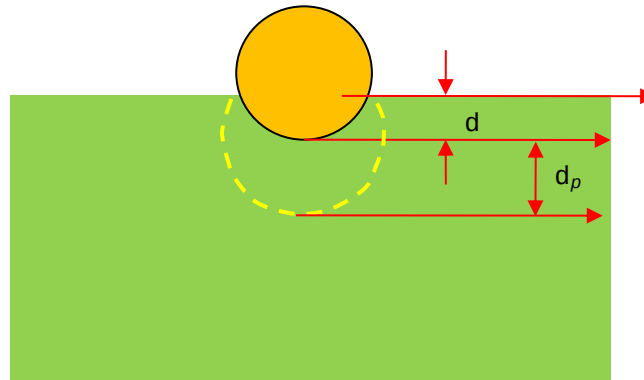


Fig. 6.2. Crater depth measurement.

Table 6.1 Strain rate used in this study

Deformation form	Ball velocity, m/s	Strain rate , s^{-1}
BD, Case I , $H=2$ m	6.26	6.8×10^2
BD, Case II , $H=0.5$ m	3.13	3.1×10^2
BP, Case I	8.33×10^{-5}	1.3×10^{-2}
BP, Case II	1.67×10^{-6}	2.4×10^{-4}

During BP process, the deformation is performed by the contact between the ball and the component. The problem of stress distribution between two bodies in contact has been successfully solved by the previous article [15]. According to the analysis, the pure iron plate being BP-processed can be treated as a semi-infinite body (Fig. 6.3) and the stress distribution between the ball and component can be solved by using the

same theory.

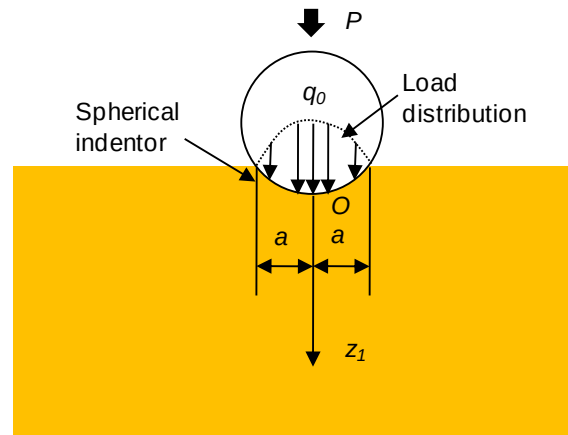


Fig.6.3. The illustration of deformation on a semi-infinite body being compressed by a steel ball.

According to Ref. [16] which calculated the stress distribution between a ball and a spherical seat, the local stress for points along the axis OZ_1 (the thickness direction, as shown in Fig. 6.3) is schematically illustrated in Fig. 6.4. The maximum pressure q_0 at the center of the contact surface is taken as a unit of stress, while the radius of the contact surface a is taken as a unit of distance as shown in Fig. 6.3. Note that the larger d value corresponds to the greater applied pressure in the calculation.

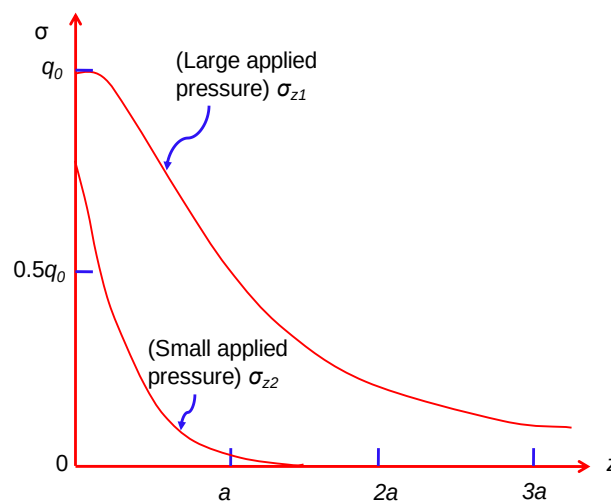


Fig. 6.4. The stress distribution in the depth direction along axis OZ_1 in Fig. 6.3.

After the deformation, longitudinal mid-plane sections were prepared for EBSD

measurement. The surface was polished by using 100 nm alumina polishing slurry followed by colloidal silica in order to achieve flat surfaces without any damage associated with the sample preparation for the EBSD analysis. It was an important step to obtain good diffraction patterns of the surface. The SEM micrograph of iron processed by SLPD is shown in Fig.6.5, in which the region represented by white rectangle is a part of the local area beneath the deformed surface subjected to the EBSD analysis. The EBSD measurements were done with a JEOL JSM-6500F field-emission scanning electron microscope (FE-SEM) equipped with a TSL software. The step size was set to 3 μm in all the cases. The grain boundary is defined as a boundary involving misorientation larger than 5°.

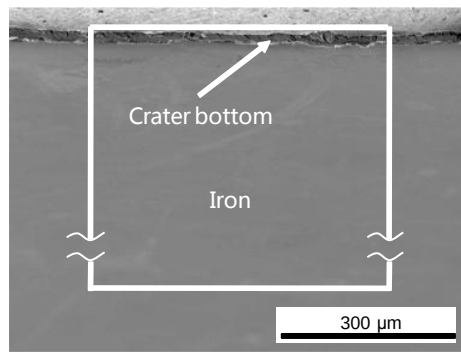


Fig. 6.5 SEM micrograph of iron processed by BD-case I.

The same ferrite/pearlite (F/P) banded steel as Chapter 4 was used for the austenitizing treatment in this study. The chemical composition of the steel is given in Table 6.2. All the samples are the cylindrical shape and the same size (25 mm in diameter and 10 mm in thickness). The samples were plastically deformed by the BD test with a crater depth of 0.61, 0.52, and 0.39 mm.

Table 6.2 Chemical composition of the carbon steel used in this study (mass%)

C	Si	Mn	P	Al	N
0.2	0.2	0.8	0.015	0.04	0.006

The plastically deformed samples with a crater depth of 0.61 mm were austenitized for 30 min in an argon atmosphere at 1000 °C. The heating rate of the

samples was approximately 5 °C/s. After the austenization, the samples were immediately quenched into an iced-water bath. Longitudinal sections of the quenched samples were observed by an optical microscope after grinding and polishing followed by chemical etching with supersaturated picric acid at 60 °C for 40 s (to reveal the prior austenite grain boundaries).

6.3. Results and discussion

6.3.1 Relation between misorientation and plastic strain

Kernel Average Misorientation (KAM) can be used to assess a quantitative rotation angle of the crystalline [17-20]. Fig. 6.6 shows the KAM maps of the samples with $d=0.0$, 0.41 and 1.1 mm produced by BP tests (case I) at a strain rate of $1.3 \times 10^{-2} \text{ s}^{-1}$. These are the structures observed about 1.0 mm beneath the surface. Note the sample with $d=0.0$ mm corresponds to undeformed sample. The top part of each figure corresponds to the surface of the deformed sample. The grain sizes are roughly the same in all the cases. Importantly, the rotation of crystal orientation inside a grain is observed in the deformed samples ((b) and (c)). It becomes more notable as the crater depth increases. The local change in the crystal orientation can also be observed in the inverse pole figures shown in Fig. 6.7.

The number of grains used for the analysis and the number of the total data points are almost the same in all the cases. The fact that the orientation spread in each grain is increased according with increase of deformation is shown as changes of inverse pole figures (Fig.6.7 (a) (b) (c)). Spots (cluster of points) in the pole Fig. 6.7(a) correspond to each grains of undeformed sample. And these spots are spreading as increase of deformation which makes more uniformly scattering of points like Figs. 6.7 (b) and (c). Clearly the plastic deformation caused the points in the inverse pole figure to spread from the concentrated groups of points. Thus, the retained plastic strain is manifest as local variations in lattice orientation. This result is quite consistent with the finding in the earlier study [21]. In the present study, the local plastic strain is evaluated based on the misorientation within individual grains as explained below.

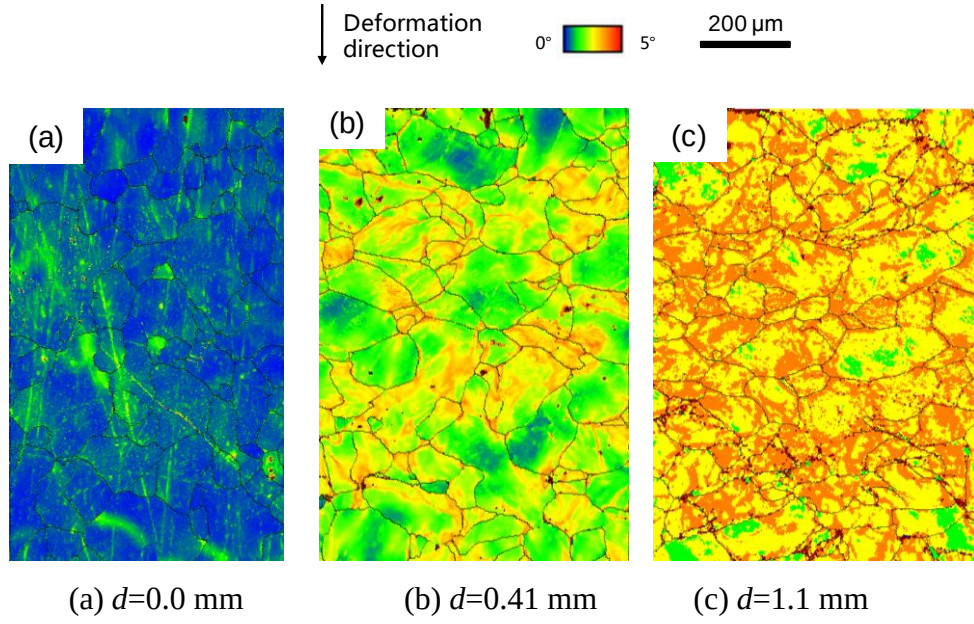


Fig. 6.6. KAM maps of pure iron with 10th nearest-neighbor kernels deformed in BP test at $\dot{\epsilon}=1.3 \times 10^{-2} \text{ s}^{-1}$.

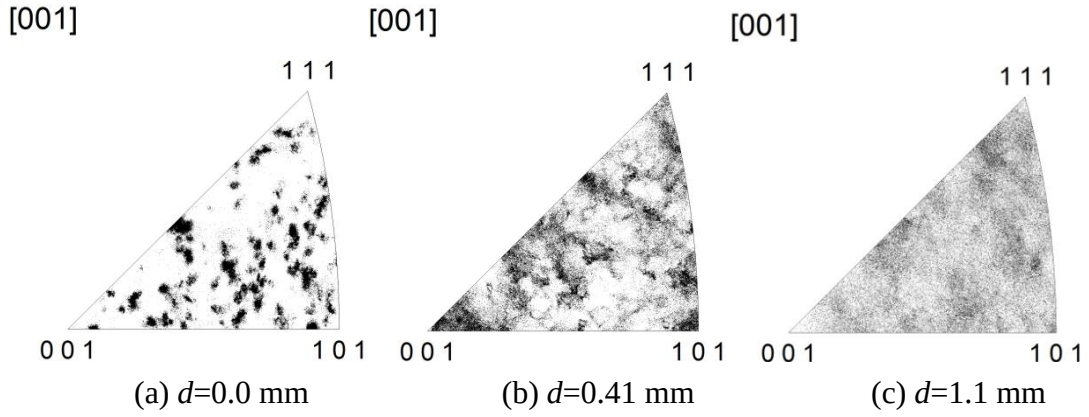


Fig. 6.7. Inverse pole figures of crystal orientation of pure iron deformed in BP test at $\dot{\epsilon}=1.3 \times 10^{-2} \text{ s}^{-1}$.

The local plastic strain is evaluated based on a local misorientation method. First, the variation in crystal orientation inside a grain is quantified in reference to an average orientation measured for each grain. The data for misorientation angle thus obtained are averaged by the following equation:

$$M_{\text{ave}} = \sum_{k=1}^{n_g} \left(\sum_{i=1}^{n_k} f_{i,k} / n_k \right) \quad (6.5)$$

where $f_{i,k}$ denotes the misorientation angle at the point i inside the grain k , n_g is the number of grains and n_k is the number of points in the grain k .

The misorientation is not zero even if the sample is undeformed. A typical example is shown in Fig. 6.8. The stress due to the machining process may have caused this variation shown in Fig. 6.8. M_{ave} calculated by Eq. (6.5) was about 1.16° for the undeformed sample (orientation measurement has an error of $0.5\text{-}1^\circ$). This value serves as a reference in the determination of the depth of the deformed zone in the deformed sample, d_p , in Fig. 6.2. For instance, from the spatial profile of M_{ave} (which will be seen as the spatial profile of the local plastic strain in Fig. 6.12), d_p was estimated to be $d_p=3.5\text{ mm}$ for the case of $d=1.1\text{ mm}$ at the strain rate of $6.8\times 10^2\text{ s}^{-1}$ and $d_p=6.0\text{ mm}$ for $d=1.1\text{ mm}$ and $2.4\times 10^{-4}\text{ s}^{-1}$.

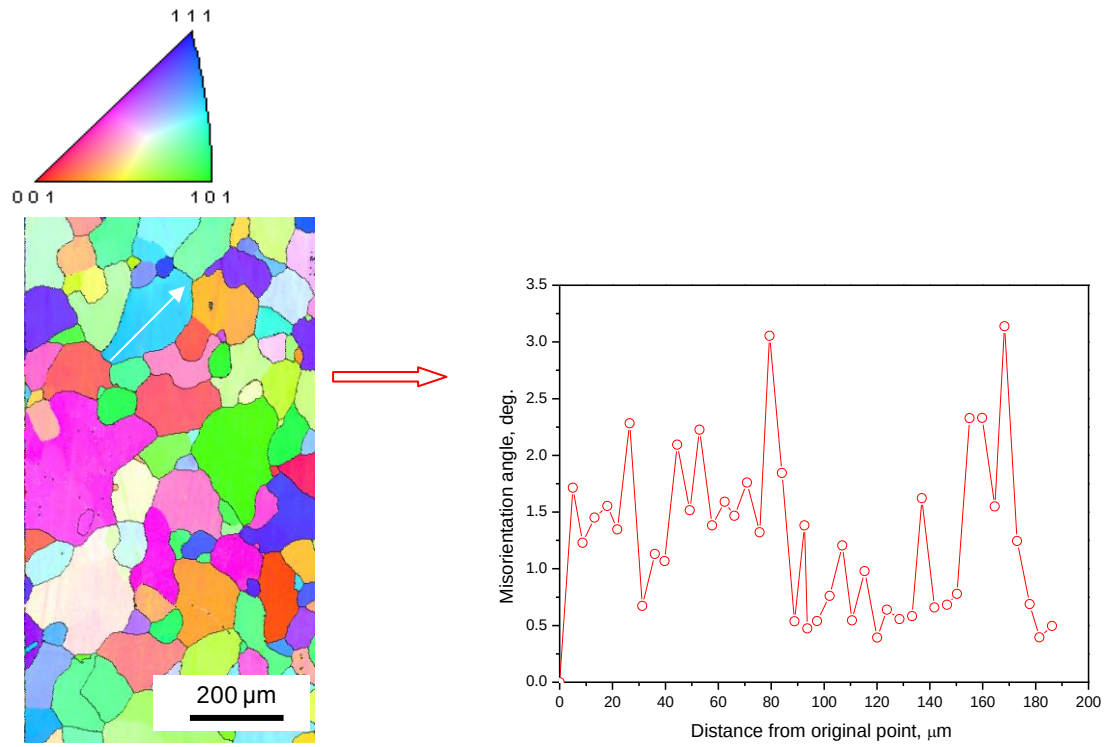


Fig. 6.8. Misorientation angles along a line of pixels: point-to-origin in undeformed pure iron.

M_{ave} obtained from samples deformed at the strain rate of $2.4\times 10^{-4}\text{ s}^{-1}$ was calculated by Eq. (6.5), and the results were plotted on a M_{ave} versus ε_{ave} diagram as shown in Fig. 6.9. A straight line obtained by linear regression provides a very good fit to the data. The relationship between M_{ave} and the average plastic strain can be expressed by

$$M_{ave} = 1.08 + 0.2\varepsilon_{ave} \quad (6.6)$$

where ε_{ave} is given in percent. This linear relationship was always observed in all the samples in this study. This implies that the local plastic strain can be estimated from local value of M_{ave} given by Eq. (6.5). Therefore, the local plastic strain $\varepsilon_{L(ave)}$ is estimated from Eq. (6.6) by substituting the local value of M_{ave} , $M_{L(ave)}$, into it as follows,

$$\varepsilon_{L(ave)} = \frac{M_{L(ave)} - 1.08}{0.2} \quad (6.7)$$

$\varepsilon_{L(ave)}$ was estimated from a square shape with a 500 μm side.

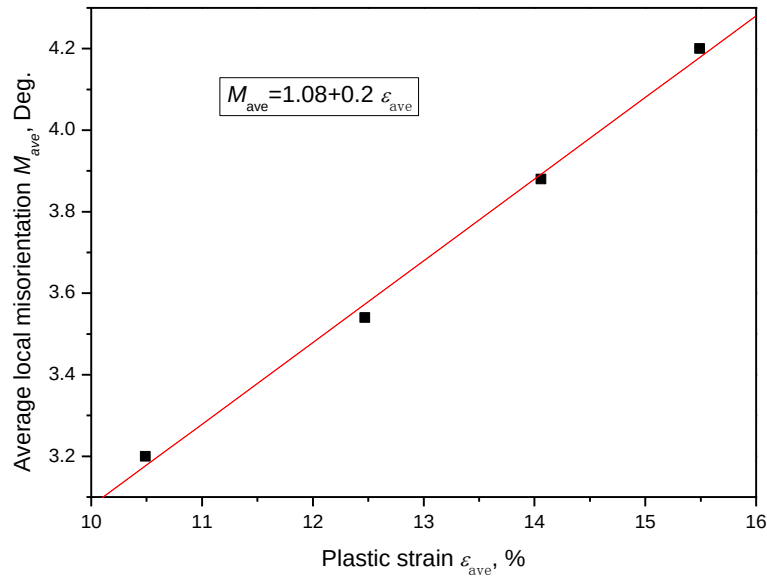


Fig. 6.9. Relationship between average local misorientation (M_{ave}) and average plastic strain for pure iron.

6.3.2 Average plastic strain and penetration depth

The average plastic strain ε_{ave} versus strain rate for $d=1.1, 0.9, 0.57$, and 0.41 mm is given in Fig. 6.10. ε_{ave} values are quite various depending on the strain rate at a fixed crater depth. The high strain rate results in large ε_{ave} value. It is also noteworthy that ε_{ave} value increases with increase in the crater depth. This indicates that the crater depth corresponds to a measure of macroscopic plastic strain in the deformed sample at a given strain rate.

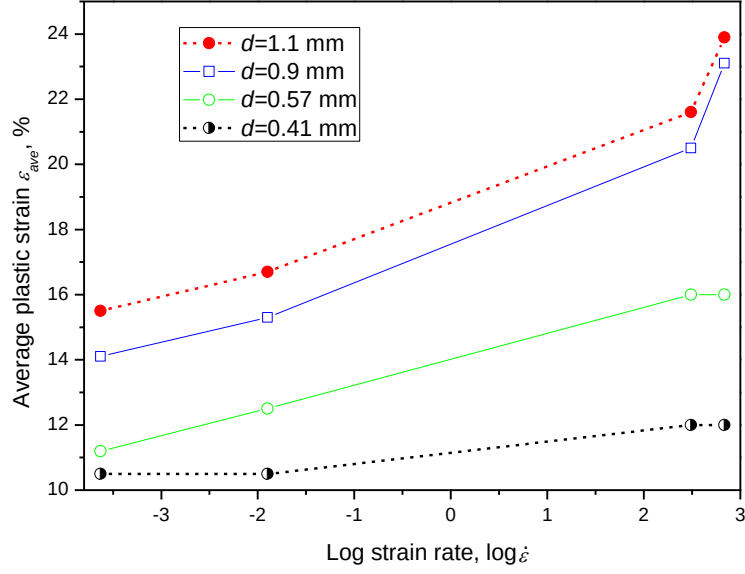


Fig.6.10. The influence of strain rate on the average plastic strain.

Shown in Fig. 6.11 is the penetration depth of the plastic deformation, d_p , as the function of crater depth. It can be seen that d_p is largely dependent on the crater depth and the strain rate. When the strain rate is low, d_p gradually increases with the crater depth. This behavior is quite similar to the variation of the stress profile shown in Fig. 6.4, in which larger applied pressure results in larger depth of the stress zone in OZ_1 . Importantly, d_p obtained by high strain rates is substantially smaller than that by the low rates, which indicates that the plastic deformation penetrates much shallower at high strain rates than that at low strain rates even though the crater depth is same. This also suggests that the deformation at high strain rates should be useful to impose localized plastic deformation on the selected area near the surface without introducing any effect on the materials beneath the deformed area.

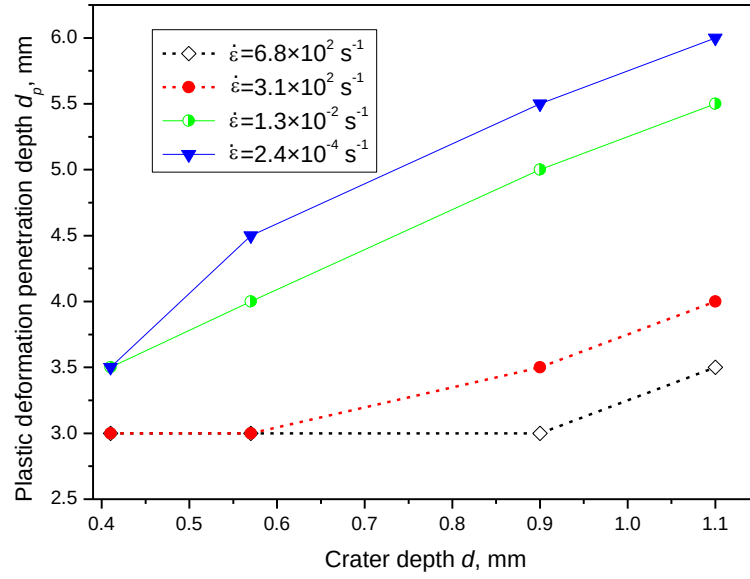


Fig.6.11. The influence of crater depth on the plastic deformation penetration depth at various level of strain rate.

6.3.3 Local plastic strain profile

Fig.6.12 shows the local plastic strain as a function of the position measured from the bottom of the crater. These are the results of the specimen for $d=1.1$ mm deformed by BD and BP tests.

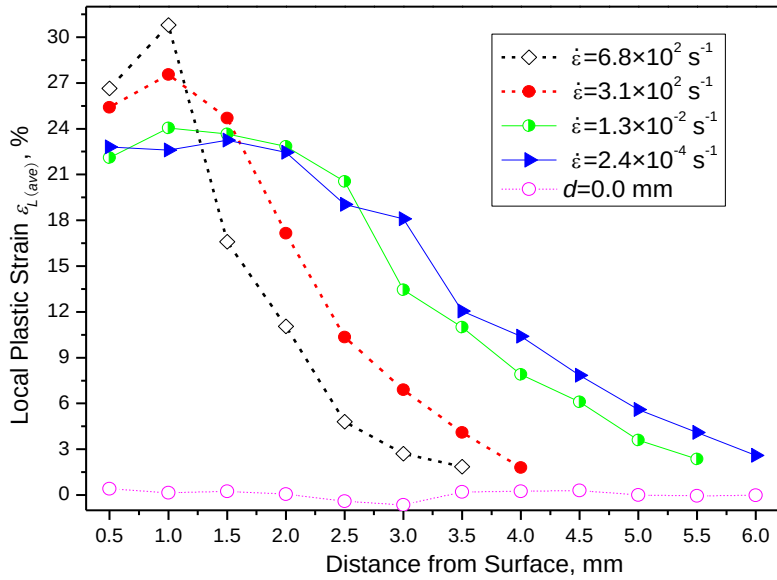


Fig. 6.12. Local plastic strain versus distance from the treated surface.

It can be seen that the profiles of local plastic strain are different, depending on the strain rate. The curve of local plastic strain versus position (hereafter, referred to as local plastic strain profile) produced by $6.8 \times 10^2 \text{ s}^{-1}$ strain rate decreases much

quicker than other cases, and it tends to be more flat with the decrease in strain rate. The local plastic strain of the undeformed sample ($d=0.0$ mm) is around zero.

The local plastic strain profiles observed for $d=0.9$, 0.57 , and 0.41 mm are shown in Fig. 6.13. As expected, the local plastic strain distributions are strongly affected by the crater depth as well. With the decrease in the crater depth, all the local plastic strain profile tend to coincide with each other and the slope of the curve tends to be small, suggesting that the local plastic strain profiles beneath the treated surface tend to uniform regardless of the strain rate when the crater depth is small. From the comparison between Fig. 6.12 and Fig. 6.13, it is easy to find that the local plastic strain increases with the increasing crater depth. Also, the local plastic strain near the surface (within 1.0 mm beneath the treated surface) obtained at high strain rate is always larger than that at low strain rates even though the crater depth is same. This fact, in good accordance with the previous studies in [10-12], indicates that high strain rates lead to highly localized plastic strain near the surface. It is noteworthy that the maximum local plastic strain is not right at the treated surface but around 1.0 mm (thus in the range of 0.5 - 1.0 mm) beneath the surface, as for strain rates higher than $2.4 \times 10^{-4} \text{ s}^{-1}$, according to Figs. 6.12 and 6.13. This result is consistent with the famous Hertz contact analysis, which predicts that the maximum shear stress and thus the plastic strain is at a small depth equal to about a half of the radius of the contact surface[21]. The radii of 1.1 , 0.9 , 0.57 , and 0.41 mm depth craters in this study are 2.76 , 2.58 , 2.06 , 1.89 mm respectively, and half of them are all around 1.0 mm. Hence, the peaks of the local plastic strain obtained from the experiment show good agreement with the theoretical prediction. A similar result was also reported by Dai et al [13].

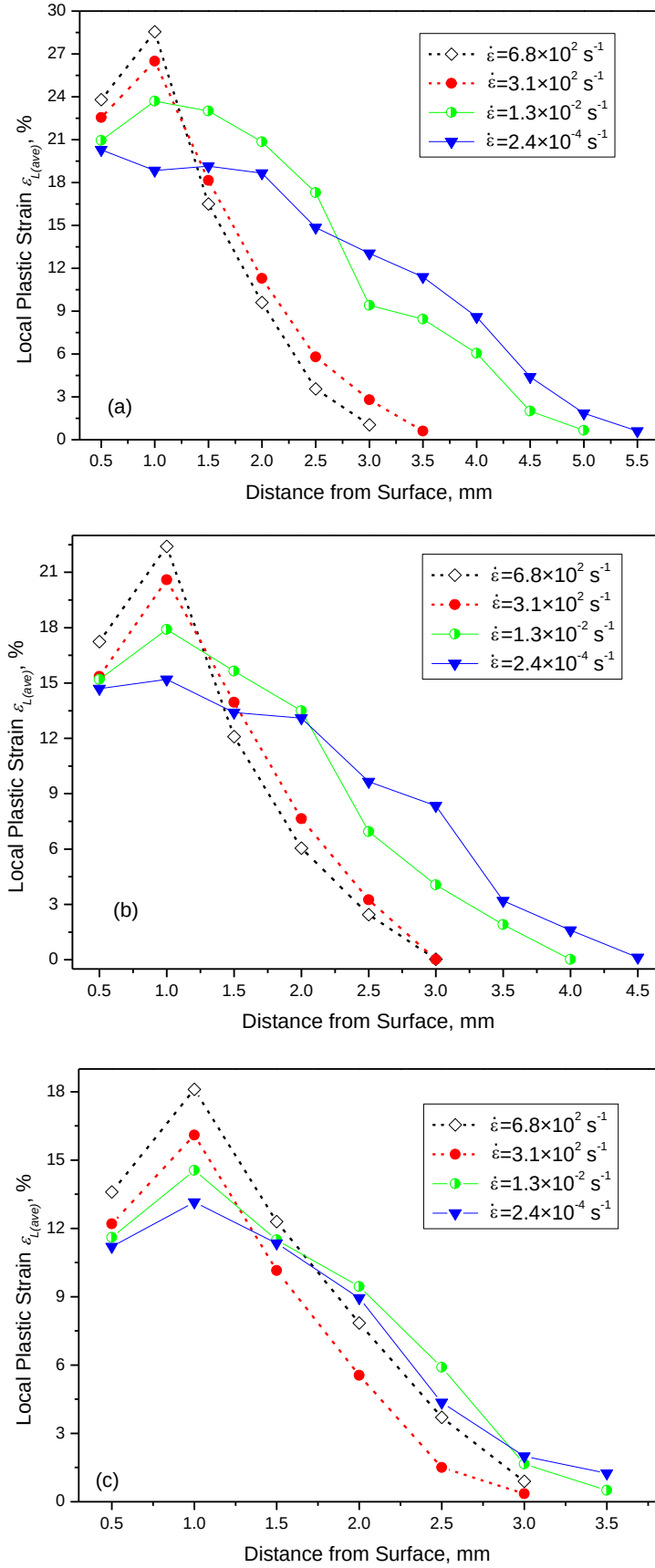


Fig. 6.13. Local plastic strain versus distance from the treated surface: (a) $d=0.9 \text{ mm}$, (b) $d=0.57 \text{ mm}$, (c) $d=0.41 \text{ mm}$.

6.3.4 Strain gradient

In this study, in order to characterize the local plastic strain profile, the strain gradient K is defined as the slope between the maximum and minimum local plastic strain. The strain gradient is plotted against the crater depth in Fig. 6.14.

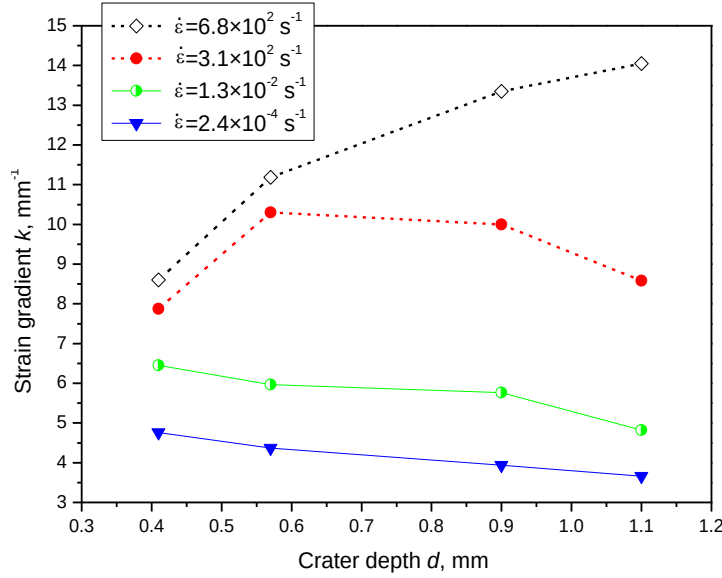


Fig. 6.14. The influence of crater depth on the strain gradient at various level of strain rate.

The strain gradient significantly depends on the strain rate and the crater depth. High strain rate produces large strain gradient, especially when the crater depth is large. The strain gradient decreases with increase in the crater depth at strain rates of $1.3 \times 10^2 \text{ s}^{-1}$ and $2.4 \times 10^4 \text{ s}^{-1}$. This shows good agreement with the stress distribution in Fig. 6.4, in which the stress gradient is smaller when the applied pressure is larger. This is also consistent with the widely known fact that greater reduction results in more uniform plastic deformation in the sheet in rolling process. However, for the strain rate of $6.8 \times 10^2 \text{ s}^{-1}$ the strain gradient monotonically increases with increase in the crater depth. To well understand this phenomenon, the KAM maps of the samples with $d=1.1$ and 0.41 mm at strain rate of $6.8 \times 10^2 \text{ s}^{-1}$ (as shown in Fig. 6.15) are analyzed qualitatively.

The top part of Figs. 6.15(a), (b) correspond to the surface of the deformed sample and these are the structures observed about 3.0 mm beneath the surface.

Comparing Figs. 6.15(a) with (b), it can be seen that crystals in the upper part were much more severely deformed for $d=1.1$ mm than $d=0.4$ mm. On the other hand, there is no considerable difference of the local plastic strain in the lower parts. Therefore, with the increase in crater depth, the upper part of the sample was more severely deformed, while the lower part was almost unchanged. This can be also seen in Fig. 6.11 where the penetration depth changes little when d changes from 1.1 to 0.4 mm. Hence the increase in crater depth resulted in the increase in strain gradient at strain rate of $6.8 \times 10^2 \text{ s}^{-1}$. Additionally, Fig. 6.15 also qualitatively shows that the position of the maximum local plastic strain does not exactly correspond to the treated surface but the region of 0.5-1.0 mm beneath the surface.

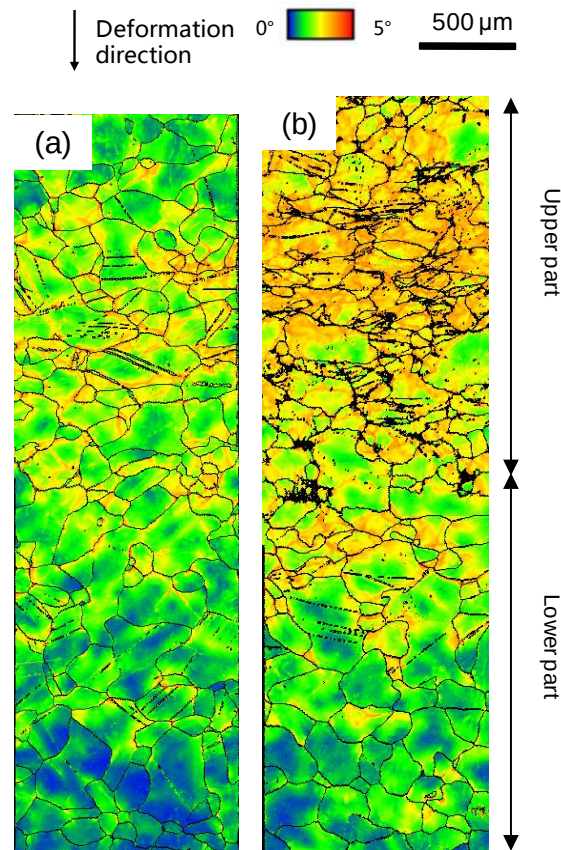


Fig.6.15. KAM map of pure iron with 10th nearest-neighbor kernels deformed in BP test at $\dot{\epsilon}=6.8 \times 10^2 \text{ s}^{-1}$: (a) $d=1.1$ mm, (b) $d=0.41$ mm.

According to the above analysis, level of the local plastic strain, local plastic strain gradient, and the PD are strongly dependent on the strain rate and macroscopic

plastic strain, i.e. they all can be effectively controlled. High strain rate with large macroscopic plastic strain leads to steep and shallow distribution profile of local plastic strain, which is considered to be useful to impose severe and localized plastic deformation on the selected area near the surface. On the other hand, the deformation at low strain rates with large macroscopic plastic strains yields uniform and deep local plastic strain distributions. The peak of the local plastic strain profile does not appear at the deformed surface but immediately beneath the surface. The local plastic strain gradient decreases with the increase in macroscopic plastic strain when the strain rate is low, while it increases when the strain rate is high.

6.3.5 Austenitizing treatment

Similar with Eq. (6.6), a linear relationship between misorientation (M_{ave}) and plastic strain (ε_{ave}) has been also found in 0.2 mass% carbon steel in this study. The linear relationship between M_{ave} and ε_{ave} was fitted to be:

$$M_{ave} = 0.94 + 0.3\varepsilon_{ave} \quad (6.8)$$

therefore, the local plastic strain $\varepsilon_{L(ave)}$ can be estimated from Eq. (6.8) by substituting the local value of M_{ave} , which is described as $M_{L(ave)}$, into it as follows,

$$\varepsilon_{L(ave)} = \frac{M_{L(ave)} - 0.94}{0.3} \quad (6.9)$$

By using Eq. (6.9), the calculated local plastic strain versus the distance from the deformed surface of the sample with crater depth of 0.61 mm is shown in Fig. 6.16. From Fig. 6.16 it can be seen that the local plastic strain distribution in the 0.2 mass% carbon steel deformed by BD is quite similar with that in the pure iron deformed with similar strain rate. Namely, the high strain rate deformation leads to steep and narrow profile of local plastic strain near the surface.

Fig. 6.17 schematically shows a F/P banded sample being plastically deformed by BD. Fig. 6.18 displays the prior austenite grain structures observed at different positions of a , b , and c shown in Fig. 6.17 after held the sample at 1000 °C for 30 min. According to Fig. 6.18, it is very interesting to note that the austenite grain structures of the different local regions are totally different, although they are observed in the

same sample. At the position *a* (Fig. 6.18(a)) just beneath the deformed surface, very fine and uniform grain structures is observed. While at the position *c* (Fig. 6.18(c)) which is 5 mm beneath the deformed surface, extremely coarse grain structures is exhibited. What most interesting here is that at position *b* (Fig. 6.18(b)) where fine grain structure is observed at its upper part, while extremely coarse grain structure is observed at its lower part. And a very clear boundary is existed between them as indicated by the red line in Fig. 6.18(b). This strongly indicates that the austenite grain growth behavior of the local areas in the cold deformed carbon steel is strongly depended on the local plastic strain. Hence, the austenite grain structure, thus the microstructure near the surface can be effectively controlled by the local plastic strain distribution.

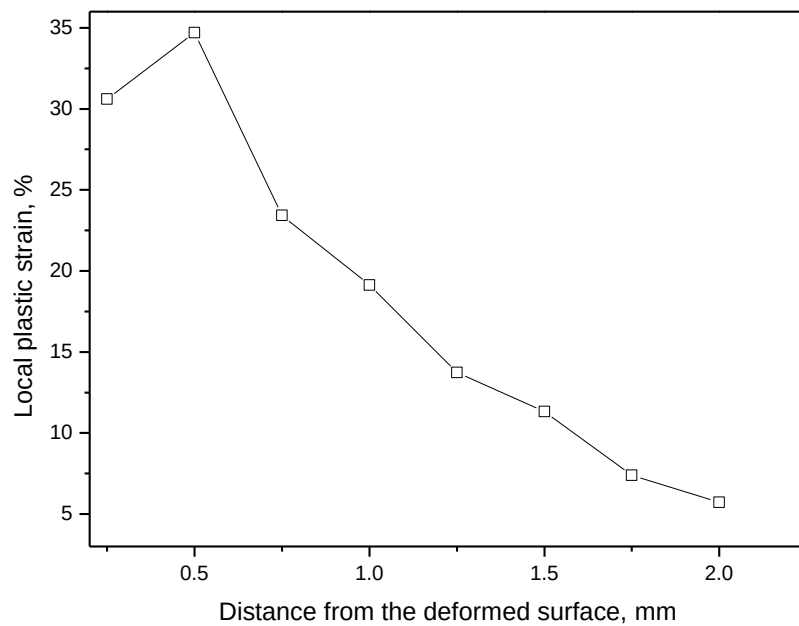


Fig. 6.16. Local plastic strain profile versus distance from the deformed surface

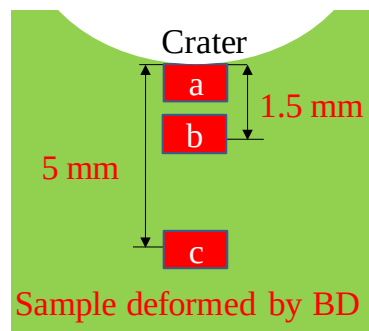


Fig. 6.17. The illustration of deformation on a F/P banded sample being deformed by BD.

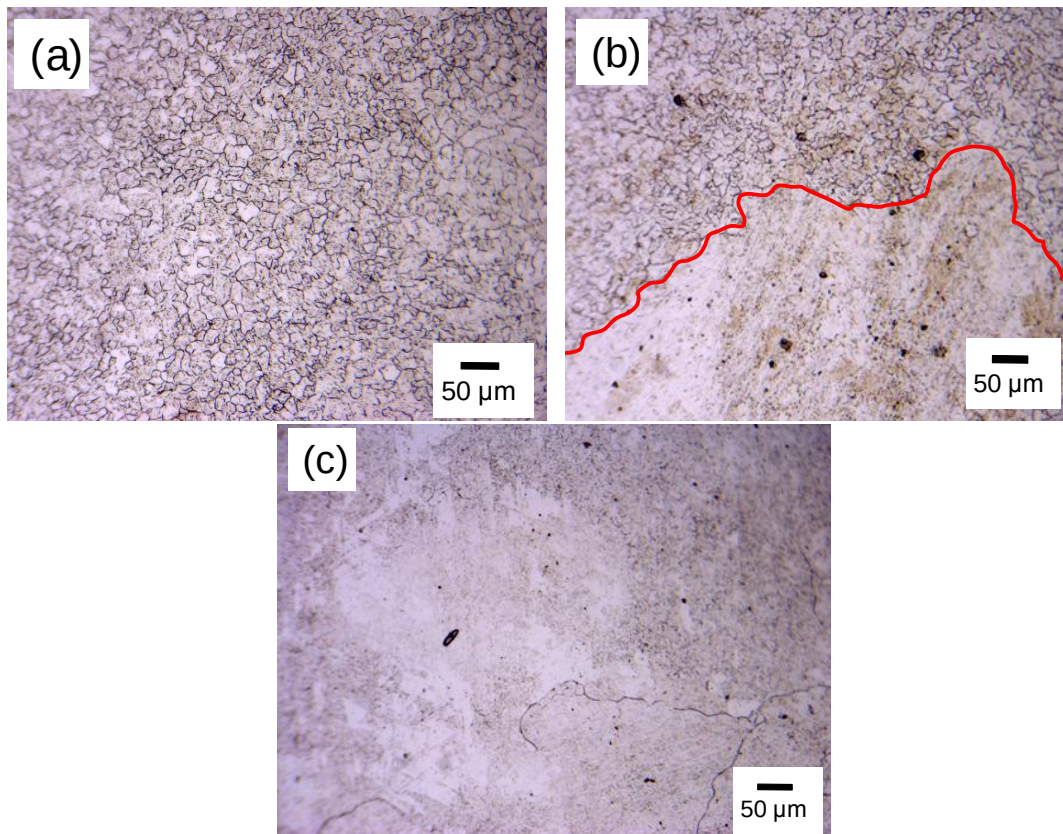


Fig. 6.18. Prior austenite grain structures observed at the different positions of *a*, *b*, and *c* shown in Fig. 6.17.

6.4. Conclusions

Ball dropping and pressing experiments, which correspond to a model experiment for surface plastic deformation process, were conducted to investigate local plastic strain distribution in the vicinity of the deformed surface of pure iron. The local plastic strain was estimated from the misorientation angle measured by the electron backscatter diffraction (EBSD) analysis. The effects of crater depth and the strain rate on spatial profile of the local plastic strain were detailed in this study. Furthermore, the effect of local plastic strain distribution on austenite grain growth has also been studied. The important results are summarized as follows.

- (1) The local plastic strain beneath the surface is large near the surface and decreases with the increase in depth, in general. However, in case of deformation at higher strain rates a peak of local plastic strain appears near the surface. The position of the peak is not at the deformed surface but immediately beneath the surface.

- (2) Both the larger strains and higher strain rates lead to higher peaks and shallower deformation depths of the local strain distribution beneath the deformed surface, although the effects of strain rate are much stronger than those of strain.
- (3) The local strain gradient beneath the peak increases with the level of the macroscopic plastic strain when the strain rate is high, while it decreases when the strain rate is low.
- (4) The local plastic strain distribution has a great effect on the austenite grain growth. The microstructure near the surface can be effectively controlled by the local plastic strain distribution.

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Chapter 7

Misorientation/local plastic strain manifestations in chemical etching color

7.1. Introduction

Microstructures including crystal grains and constituent phases are made visible by etching during a metallographic investigation. Two main types of etching are applied: the classical chemical etching and color etching. The chemical etching using an etchant is a very simple technique and is most widely used in the metallographic investigation, which preferentially attack the crystal grain boundaries to reveal the microstructures. On the other hand, the so called color etching is carried out by deposition of a thin surface film on the metal, causing light interference between the different reflections from the metal surface and the film surface [1], which is relatively complicated compared with the chemical etching.

It is widely known that plastically deformed metal materials maintain the geometric continuity of its structure by creating dislocations, which leads to variations in lattice orientation (misorientation) in the crystal grains. The degree of local plastic strain can be evaluated by the local average misorientation due to the proportional relationship between them [2-3].

The correlation between chemical etching and crystallographic orientation has been investigated in many works. The dependence of the etching rates of single-crystal silicon on crystallographic orientation was reported by Sato et al. [4]. Szabó and Bonyár [5] studied the effect of grain orientation on the effectiveness of chemical etching. Just as mentioned above, the chemical etching is used to reveal the microstructure of the metals or alloys through preferential etches at the grain boundaries. Different morphology of microstructures can be observed by chemical etching depending on the degrees of etching: (1) with light etching only the grain boundaries can be revealed; (2) while with further etching both grain boundaries and

grain shading can be manifested [6]. Since the rate of etching is affected by the grain orientation, the etching creates contrast in the different oriented grains through differences in reflectivity [6]. Zhu et al. [7] observed the grain etching contrast in α -titanium. Orientation-dependent topography formation was observed in FIB cutting [8] and mechanical polishing [9]. The dependence of the shade of color-etching on crystal orientation was investigated by previous researchers [10]. However, it remained unclear whether or not the variation in the crystal orientation (misorientation) inside the individual grains can be revealed by the etching color. Since the chemical etching is an easy way to reveal microstructures, if such misorientation can be revealed by the chemical etching contrast, it would provide a novel and simple way to understand the misorientation and local plastic strain distributions in the microstructures. On the other hand, the dependence of chemical etching contrast on crystallographic orientation itself actually has not been seriously studied yet.

In this study, we aim at manifesting the misorientation caused by the local plastic strain using the chemical etching contrast. For this purpose, the chemical etching contrast dependence of crystallographic orientation is first examined in the undeformed iron, and then the distribution of the misorientation inside the grains of the deformed iron is studied. The results clearly show that the chemical etching contrast strongly reflects the crystallographic orientation and the gradual change in chemical etching contrast within the individual deformed grains give information of both the misorientation and local plastic strain inside the grains.

7.2. Material and methods

The material used in the present study is commercially pure iron with a purity of 99.95 mass% (Si < 0.01, Mn < 0.01, P < 0.002, S < 0.002 and Al < 0.001 mass%). The as-received pure iron bars were annealed at 1000 °C for 60 min and then slowly cooled to room temperature to obtain ferrite grains structure without residual strain. The deformation was carried out by a ball-pressing (BP) test at room temperature; the

set-up and procedure of which can be found in Chapter 6. After the deformation, longitudinal mid-plane sections were prepared for both electron backscatter diffraction (EBSD) measurement and optical microscopy (OM) observation. The surface was mechanically polished using silicon carbide paper to grade 2000 and then it was further polished using a polishing cloth with a liquid suspension of 40 nm alumina in order to achieve flat surfaces without any damage associated with the sample preparation. It is an important step to produce high quality images. The EBSD measurements were done with a JEOL JSM-6500F field-emission scanning electron microscope (FE-SEM) equipped with a TSL software. The step size was set to 3 μm in all the cases. The grain boundary was defined as a boundary involving misorientation larger than 5° . After the EBSD measurement, the samples were chemically etched by 3% nital at room temperature for 5-8 sec. followed by OM observation (KEYENCE VHX-1000). The etching time should be carefully controlled to get a clear etching contrast, and to avoid overetching. The difference in height of each grain on the etched surface was measured using an atomic force microscope (AFM; KEYENCE VN-8000) working in the tapping mode.

7.3. Results and discussion

Figs. 7.1(a) and (b) show an optical image of the undeformed sample after chemical etching and the corresponding crystal orientation map of the same area, respectively. The individual grains are numbered in both figures. It can be seen in Fig. 7.1(a) that the etching colors of the grains are varying over the observation area. By comparing Figs. 7.1(a) with (b), it is very interestingly found that the different etching colors of the grains in Fig. 7.1(a) correspond to different grain orientations in Fig. 7.1(b), which clearly indicates that the variety of the etching contrasts of the grains, is a manifestation of the variety of the grain orientations. This shows good agreement with the previous report [6].

In order to determine the dependence of chemical etching color on the grain orientation, the correlation between the luminance of each grain in the chemically

etched image and the orientation angles between surface normal and directions of [001], [101] and [111] was investigated. The luminance (Y) can be calculated by the following equation [11]:

$$Y = 0.299 \cdot R + 0.587 \cdot G + 0.114 \cdot B \quad (7.1)$$

where R , G and B are color components of red, green and blue and their values can be measured for each grain in Fig. 7.1(a). Fig. 7.2 shows the relationship between the luminance and the angle between surface normal and directions of [001], [101] and [111]. It can be clearly seen in Fig. 7.2 that when the grain orientation is getting close to [001] direction, the luminance tends to increase, which means that bright colored grains in the etched structure has crystal orientation close to [001] direction. While the grain orientation is getting close to [101] and [111] directions, the corresponding etching color tends to be darker. Hence, the chemical etching contrast clearly depends on the crystallographic orientation, and the grain orientations can be roughly identified from their chemically etched color. Importantly, the variation in crystal orientation, viz. misorientation inside the grains caused by plastic deformation can be revealed by the chemically etched microstructures, as described in detail in the following part.

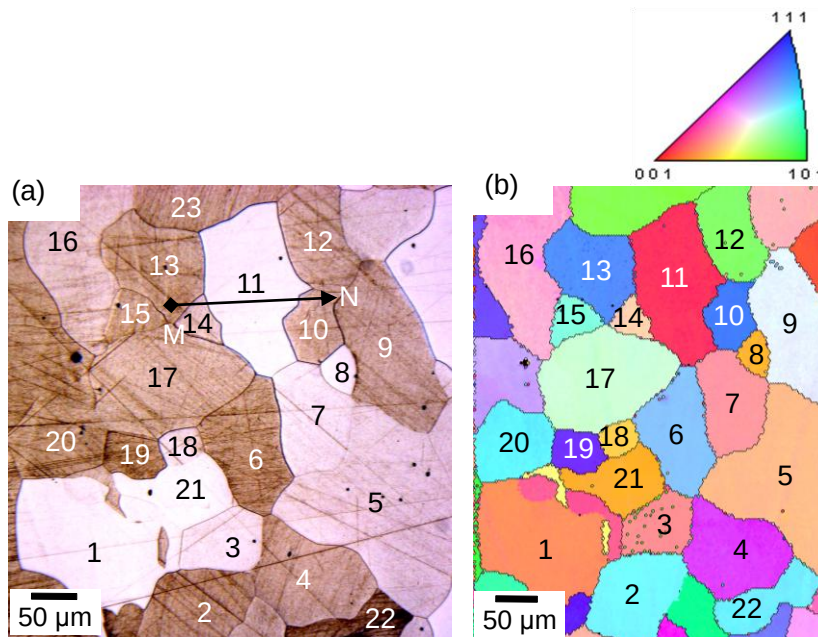


Fig. 7.1. (a) Optical image of chemically etched undeformed sample and (b) orientation map of the same sample area.

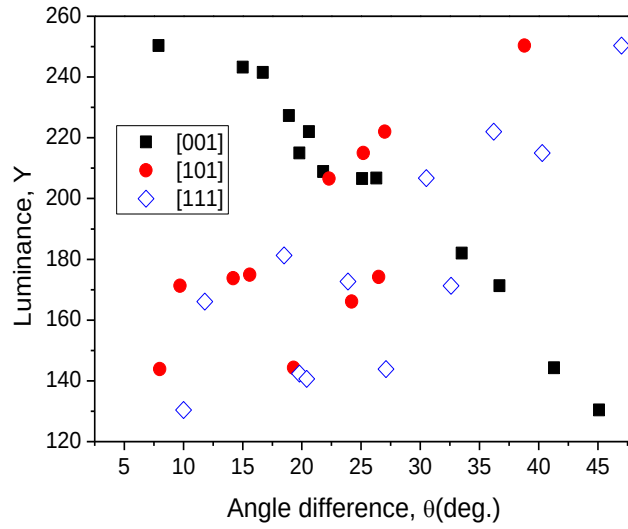


Fig. 7.2. Correlation between luminance and the angle between surface normal and [001], [101] and [111] directions.

Fig. 7.3(a) shows the chemically etched image of the deformed sample and Fig. 7.3(b) is the corresponding crystal orientation map. From Fig. 7.3(b) it can be seen that the individual grains show varying shades of colors, indicating local rotation of crystal orientation, viz. misorientation inside the grains caused by plastic deformation. Importantly, gradual change in etching color inside the individual grains is also observed in Fig. 7.3(a). It is very interesting to note based on the comparison between Figs. 7.3(a) and (b) that this gradual change in etching color well corresponds to the gradual change in crystallographic orientation, thus misorientation inside the grain. For example, red and green colors in grain-1 in Fig. 7.3(b) indicating variation in crystallographic orientation, viz. the existence of plastic strain inside the grain well correspond to the light and dark etching colors in the same grain in Fig. 7.3(a). The striking observation is that the subtle color contrast displayed in grain-16 in Fig. 7.3(b) can even be finely revealed by the varying shades of chemical etching contrast in the same grain in Fig. 7.3(a). Hence, in the individual grains, when the local lattice orientation was changed by plastic deformation, the corresponding etching contrast is also changed. The misorientation, thus the local plastic strain inside the grain can be clearly revealed by the etching contrast. This is a very important fact to help understand the distribution of misorientation and local plastic strain in the

microstructures in a direct and simple way.

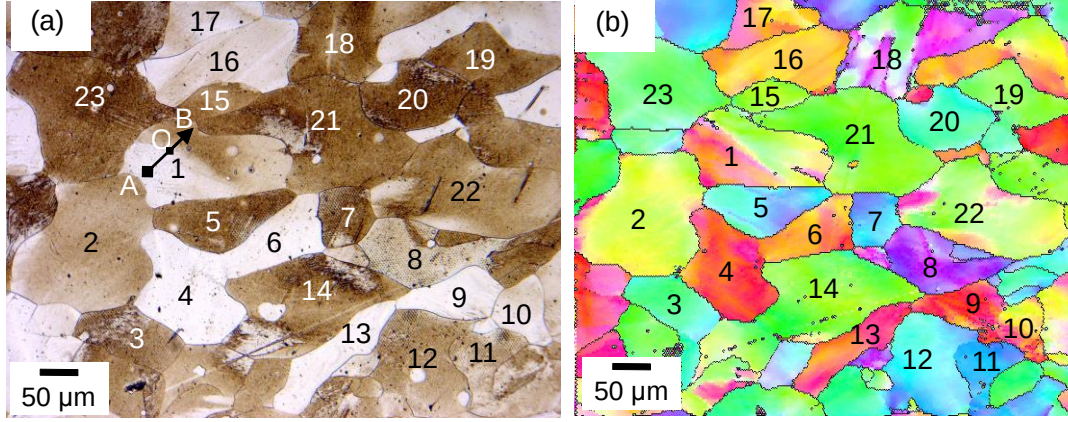


Fig. 7.3. (a) Optical image of chemically etched as-deformed sample and (b) orientation map of the same sample area.

From careful examination of the microstructures shown in Fig. 7.3(a), it can be noted that the etching contrasts of the grains are varying inside the grains. This should indicate different degrees of plastic strain inside the grains. For example, in Fig. 7.3(a) the etching contrast of grain-1 is stronger than that of grain-22. The average misorientation of grains -1 and 22 were calculated to be 10.0° and 5.8° , respectively, by using the following equation:

$$M_{k(ave)} = \sum_{i=1}^{n_k} f_{i,k} / n_k \quad (7.2)$$

where $M_{k(ave)}$ is the average misorientation of grain k , $f_{i,k}$ denotes the misorientation angle at the point i inside the grain k , which is quantified in reference to the average orientation of each grain, and n_k is the number of points in the grain k , which indicates varying degrees of plastic strain inside the grains. Hence, the different levels of etching contrast of the grains correspond to the varying degrees of plastic strain inside the grains. Thus, the distribution of local plastic strain over the observation area can be qualitatively understood from the chemically etched image.

Additionally, the relative local plastic strains between the different local regions or different samples can be qualitatively determined by the chemical etching contrast. Figs. 7.4 (a) and (b) show the chemically etched microstructures of different regions

of the same sample. By comparing these two figures, it can be noted that Fig. 7.4(a) exhibits a relatively stronger overall etching contrast than that of Fig. 7.4(b). It is known that Kernel Average Misorientation (KAM) mapping approach can be used for visualizing of plastic deformation. In order to compare the plastic deformation in these two regions visually, the KAM maps of the same areas correspond to Figs. 7.4 (a) and (b) were measured by EBSD, and the results are shown in Figs. 7.4 (c) and (d), respectively. It is very interesting that the magnitude of the KAM shown in Fig. 7.4(c) is more notable than that of Fig. 7.4(d). This clearly indicates that the stronger etching contrast of the local regions correspond to the higher magnitude of KAM, thus the higher degrees of local plastic strain.

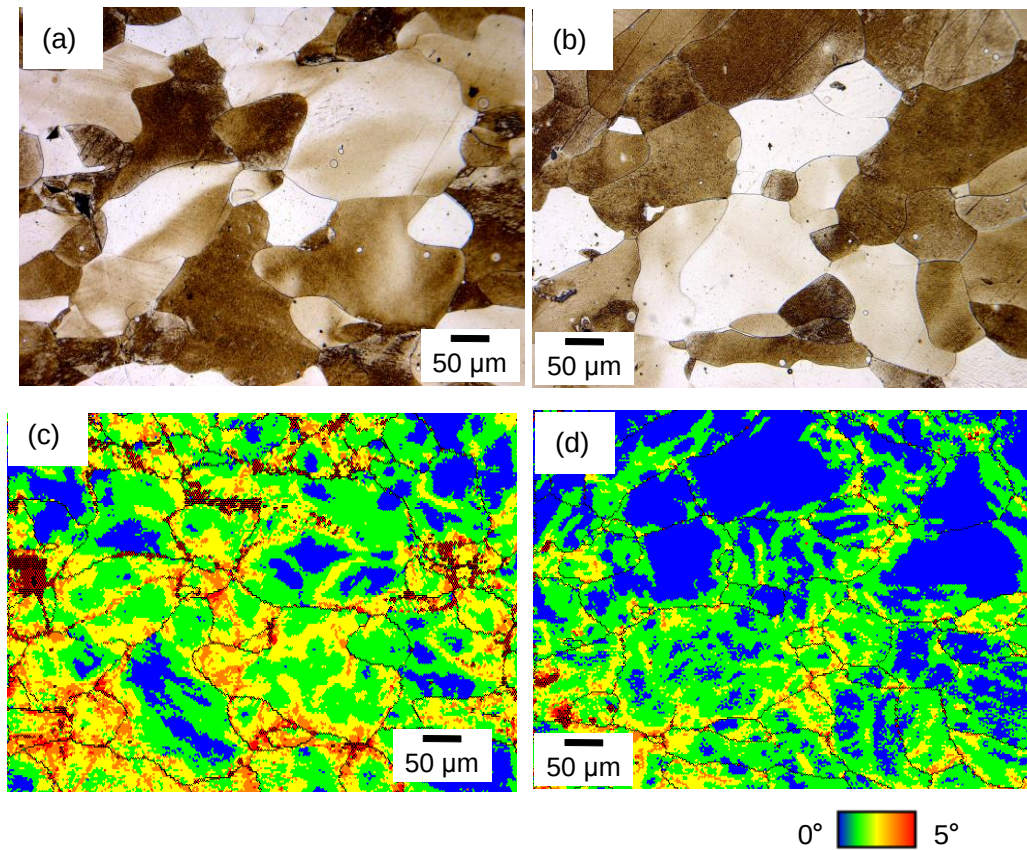


Fig. 7.4. (a) (b) Optical images of different regions of the same chemically etched sample and (c) (d) KAM maps of the same sample area in (a) (b) correspondingly with 3rd nearest-neighbor kernels.

The above results show that both the grain orientation and the variations in lattice orientation (misorientation) inside the grains can be clearly revealed by the

chemical etching contrast. The crystals having similar orientations are etched with similar colors in both undeformed (Fig. 7.1(a)) and deformed samples (Fig. 7.3(a)). Possible reasons for this behavior are explained as follows. Fig. 7.5 displays the heights of the positions on the sample surface along the lines marked with MN in Fig. 7.1(a) and AOB in Fig. 7.3(a). It can be observed from line (h) in Fig. 7.5 that the surface height is different between grains. The grain-11 as indicated by G11 in Fig. 7.5 with orientations near [001] exhibited higher surface height than the grains with orientations close to the other two directions. This indicates that the grain with orientation near [001] have a lower etching speed than that of the grains close to [101] and [111]. Furthermore, it is important to point out that the etched grain surface is roughened and become jagged surface as shown in Fig. 7.5, which might be caused by an imperfect etching with nital. It is interesting that the degrees of the jagged surface, viz. the roughness, seems to correlate with the etching speed (the grain height). When the etching speed is higher (lower grains), the roughness is severer, and vice versa. Grain surfaces with different roughness may have different reflectivity due to the diffuse reflection. As a result, the amount of reflected light captured by the objective lens of a microscope varies between the grains.

From Fig. 7.5 it can be also noticed that the etched grain surfaces are not flat, they have angles with the sample surface (hereafter, angle for short), as indicated by θ in Fig. 7.5. From careful examination of the data shown in Fig. 7.5, it can be noticed that the angle θ (or the amount of corrosion of the grains) are correlated with the etching speed (the grain height) as well. It exhibited the same changing tendency with the etching speed as that of the roughness. Grains with different angles may also have different reflectivity. Therefore, there are two possibilities which may affect the reflectivity of the grain. For example, as shown in Fig. 7.5, the grain-10 with big angle θ_2 may have a low reflectivity, thus appearing to be dark; while grain-11 with small angle θ_1 may have a high reflectivity, thus shinier. This is consistent with what we observed in Fig. 7.1. It is possible that the two possibilities coexist and affect each other. Here, it is worth to point out that no matter which factors, both of them are resulted from the varying etching speed between the different orientated grains. Or we

can set the surface roughness and angle of the grain as one concept of topography.

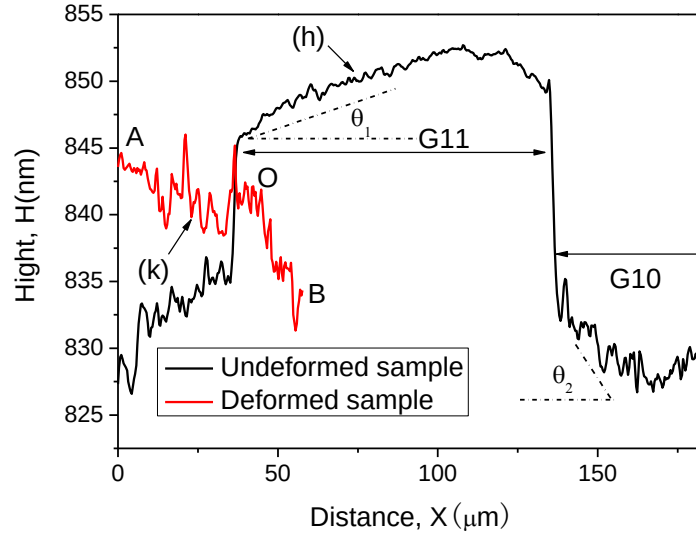


Fig. 7.5. The change in (h) grain height and (k) surface height along the lines marked with MN presented in Fig. 7.1(a) and AOB in Fig. 7.3(a). G10, 11 stands for gain-10,-11, and θ is the angle of the etched grain with the sample surface.

As the same with the undeformed samples, the surface of the individual deformed grains also becomes irregular and angled after chemical etching, as shown by line (k) in Fig. 7.5. However, the change of the surface height is not as smooth as that of the undeformed grains, but quite various between the local regions. In the chemically etched light-color region (orientations near [001]), the change is relatively slight as shown by the curve AO in Fig. 7.5; while a very rapid decrease in the surface height in the dark-color region (orientations near [101]) as shown by the curve OB in Fig. 7.5. Hence, the angles of local regions inside the grain are quite different, which is strongly depending on the local orientations. It is possible that both the surface roughness and angled local regions, i.e. the topography result in different reflectivity between the local regions. Finally, different etching shades can be observed. Hence, the local-orientation dependent topography of the local regions leads to the gradual change of the etching contrast. Therefore, it can give the information of misorientation.

According to the above analysis, a clear correlation can be made between the

chemical etching contrast and crystallographic orientation of the grain. And the most striking fact here is the locally varying lattice orientations inside the individual grains can also be finely revealed by the etching contrast. Therefore, the chemical etching can provide another possible way to intuitively understand the misorientation and local plastic strain distributions in the microstructures. Since the chemical etching and OM observation are simple and most widely used in the metallographic investigations, one can get the qualitative information of misorientation and local plastic strain distributions in the microstructures conveniently just by doing careful sample preparation and chemical etching. Since this method is direct and timesaving, it can be also used tentatively before EBSD analysis to check the presence or semi-quantitative data of misorientation or local plastic strain in the grain and will be useful for determining the necessity of the detailed subsequent EBSD analysis. Here, it should be pointed out that this method which is based on OM and is somewhat qualitative may have some limitations in the usage. For example, when the grain size is too small that the regular OM is not suitable or more accurate examination is needed, for example. In general, this method provides an easy and alternative way to qualitatively understand the misorientation and local plastic strain distributions in the microstructures.

7.4. Conclusions

Both cold-deformed and undeformed pure iron samples were chemically etched by using 3% nital etchant, while their crystal orientation was determined by electron backscatter diffraction (EBSD). It was found that the chemical etching contrast clearly depends on the crystallographic orientation of the ferrite grain in iron, and a clear correlation can be made between them. The gradual change in chemical etching contrast within the individual deformed grains was found to give information of both the misorientation and local plastic strain inside the grains. The chemical etching can provide an alternative, direct, and simple way to qualitatively understand the misorientation and local plastic strain distributions in the microstructures.

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Chapter 8

Conclusions

In this thesis, studies have been carried out in three major areas:

(a) Grain growth behavior of austenite reversely transformed from ferrite/pearlite (F/P) banded and non-banded steels.

(b) Effects of cold deformation on the abnormal grain coarsening behavior of austenite in both F/P banded and non-banded steels.

(c) Local plastic strain distribution and its effect on the austenite grain growth.

The important findings are summarized as follows:

In Chapter 3, the grain growth behavior of austenite reversely transformed from F/P banded and non-banded steels has been studied and the important findings are summarized as follows: Grain coarsening due to the abnormal grain growth (AGG) was observed. The grain-coarsening temperature, T_c , is quite low in the F/P banded sample compared with that in the non-banded sample. In the F/P banded sample, the abnormal grains originate from former ferrite regions. The occurrence of AGG is essentially attributable not to the austenite nucleation process during heating but to the grain growth process after the completion of the austenizing. Non-uniform pinning effect of AlN precipitates between former ferrite and pearlite regions is proposed as the reason for the lowered T_c in the banded sample rather than the banded segregation of manganese.

In Chapter 4, the effect of cold deformation on AGG of austenite in F/P banded steel has been studied. It was found that T_c can be greatly increased by applying cold deformation prior to austenitizing. The AGG that occurred in the F/P banded steel at lower temperatures is completely inhibited by the cold deformation. The extent of abnormal grain coarsening at temperatures above T_c is also largely decreased by the cold deformation. This is attributed to the fine and uniformly distributed AlN precipitates caused by the cold deformation.

In Chapter 5, the effect of cold deformation on AGG of austenite in non-banded

steel has been studied. It was found that the effect of cold deformation on AGG of austenite depends on the initial state of precipitates. When the non-banded steel with aluminum and nitrogen in solution, the cold deformation reduces the AGG temperature-range of austenite. While the non-banded steels are subjected to a long time subcritical annealing treatment making the AlN fully precipitated and growth, the occurrence of AGG can be completely inhibited by the cold deformation. This can be explained by the size and spatial distributions of the precipitates affected by the cold deformation.

In Chapter 6, the local plastic strain distribution and its effect on the austenite grain growth has been studied. It was found that the local plastic strain is large near the surface and decreases with the increase in depth, in general. However, in case of deformation at higher strain rates a peak of local plastic strain appears near the surface and the position of the peak is not at the deformed surface but immediately beneath the surface. Both the larger strains and higher strain rates lead to higher peaks and shallower deformation depths of the local strain distribution profile beneath the deformed surface, although the effects of strain rate are much stronger than those of strain. The local plastic strain distribution has a great effect on the austenite grain growth. The microstructure near the surface can be effectively controlled by the local plastic strain distribution.

In Chapter 7, the misorientation manifestations in chemical etching contrast has been investigated. Both cold-deformed and undeformed pure iron samples were chemically etched by using 3% nital etchant, while their crystal orientation was determined by electron backscatter diffraction (EBSD). It was found that the chemical etching contrast clearly depends on the crystallographic orientation of the ferrite grain in iron, and a clear correlation can be made between them. The gradual change in chemical etching contrast within the individual deformed grains was found to give information of both the misorientation and local plastic strain inside the grains. The chemical etching can provide an alternative, direct, and simple way to qualitatively understand the misorientation and local plastic strain distributions in the microstructures.