

Dynamic Ferrite Transformation Behavior in 10Ni-0.1C Steel during Thermo-Mechanically Controlled Process

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Chapter 1: Background and purpose

1.1 Advantages of ultrafine grained ferritic steels

Steels are basic and important materials in human civilization. Improving mechanical properties of steels, e.g., to achieve excellent balance of strength and ductility (or toughness), has always been one of the important topics in metallurgy and materials engineering. Among the strengthening mechanisms in metallic materials, such as solid solution strengthening, strain hardening and precipitation strengthening, etc., grain refinement is the only method to improve strength and toughness simultaneously. The improvement in strength through grain refinement could be described by Hall-Petch relationship, which is expressed as [1-3]:

$$\sigma_y = \sigma_0 + k_y \cdot d^{-1/2} \quad (1.1)$$

where σ_y is the yield stress, σ_0 is the friction stress, k_y is a constant called Hall-Petch slope, and d is the mean grain size. According to the equation (1.1), the yield strength of steels is inversely proportional to the square root of the grain diameter. As mentioned above, the advantage of grain refinement is that it increases not only the strength but also the toughness of the materials. That is, it has been believed that grain refinement strengthening does not harm ductility of the material. For the ferritic steels having BCC structure which show ductile-to-brittle transition, grain refinement can decrease the transition temperature.

The ductile-brittle transition temperature (DBTT) could be described by the Cottrell-Petch type equation, of which simplified form is given as [4, 5]:

$$\text{DBTT} = A - k' \cdot d^{-1/2} \quad (1.2)$$

where A and k' are constants, and d is the mean grain size.

Equations (1.1) and (1.2) indicate that the strength and toughness can be improved at the same time simply by refining the grains in steels. If ultrafine grained (UFG) ferritic steels having mean grain sizes smaller than a few microns were achieved in relatively simple chemical compositions, they would have large potential and great benefits to replace some kinds of steels employing additional alloying elements and/or complicated heat treatments. Therefore, considerable attention has been concentrated on developing novel methods to obtain steels with fine, or ultrafine grained ferrite structures in the past decades around the world [6-29].

1.2 Methods for producing ultrafine grained ferritic steels

Traditionally, high-strength steels were produced through heat treatments, such as, normalizing, or quenching and tempering. More recently, improvements in strength, toughness and weldability, have been achieved in low-C plate steels through thermo-mechanically controlled process (TMCP) [6-9]. The TMCP techniques are capable of producing fine ferrite grains in the final products and have become very important methods

to produce, high-strength steels in a low cost. So-called *controlled rolling* [8, 9] is one of these TMCP techniques in which the deformation schedule, deformation temperature and cooling rates in hot-rolling processes, as well as the chemical compositions of the steels, are well controlled.

Figure 1.1 schematically illustrates the *controlled rolling* processes at different stages depending on temperatures and the change in microstructures with deformation. After austenitization, the specimen with austenite phase is cooled to a specific deformation temperature (**Fig. 1.1(a)**). The deformation (**Fig. 1.1(b)**) in the recrystallization region of austenite results in fine recrystallized austenite grains [6], leading to an increase in the fraction of austenite grain boundaries which are the preferential nucleation sites for ferrite transformation. The ferrite grains size could be refined to 15-20 μm by this route. When finishing deformation of austenite is carried out at relatively low temperatures (**Fig. 1.1(c)**), recrystallization of austenite cannot happen. Small amount of carbide and nitride forming elements, such as Ti or/and Nb, are added in the steels for the *controlled rolling*. Fine carbides and nitrides precipitate during hot-rolling and inhibit recrystallization of austenite. The unrecrystallized microstructures of austenite having deformed substructures [9] provide high density of nucleation sites for ferrite transformation in subsequent cooling. Accelerated cooling, that is sometimes used in the *controlled rolling*, increases the driving force for ferrite transformation by decreasing the transformation temperature (by increasing

supercooling). The increase in the nucleation sites and the driving force for ferrite transformation greatly decreases the resultant ferrite grain sizes. However, the smallest mean grain size of ferrite obtained in conventional *controlled rolling* processes has been around 5 μm [8, 9].

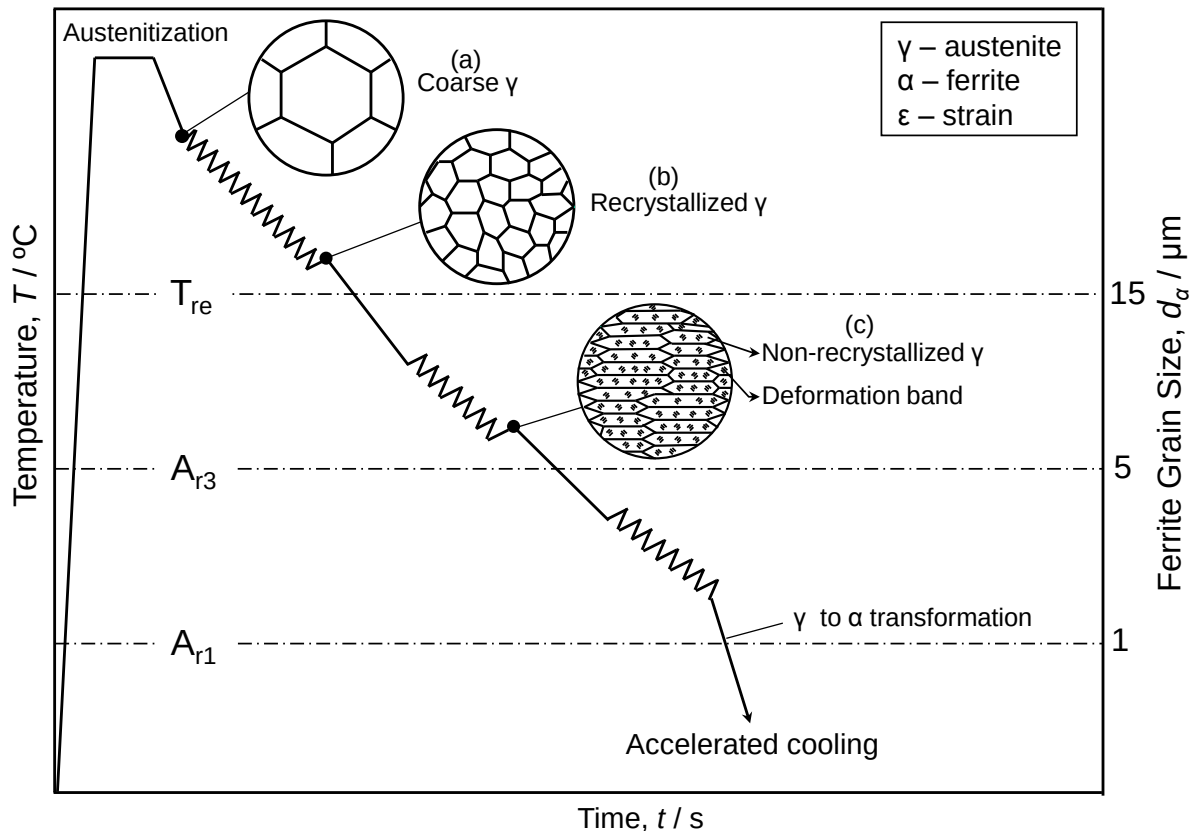


Fig. 1.1 Schematic illustration of controlled rolling and the change of microstructures.

Recently, various attempts have been tried in laboratory scales to break the limitation of 5 μm grain size of ferrite and to realize ultrafine grained steels. There are mainly two types of routes to produce UFG steels in laboratory scales: severe plastic deformation (SPD) techniques or TMCP techniques under extreme conditions. SPD techniques, such as, high

pressure torsion (HPT) [10-12], equal-channel angular pressing (ECAP) [13-18] or accumulative roll-bonding (ARB) [19-21], can apply huge amount of plastic strains to bulky metallic materials at relatively low temperatures. Submicron-sized grains could be achieved usually requiring a logarithmic equivalent strain above 4, through *grain subdivision* mechanism [21]. The SPD techniques are applicable to most metallic materials and very effective for grain refinement, but they encounter large difficulties in scaling up to large commercial dimensions of materials and mass production.

In contrast to the SPD techniques in which large plastic strain is the main and only factor for grain refinement, some newly developed TMCP techniques [22-28] are able to realize grain refinement (around 1 μm) in steels by employing relatively low accumulated strains in the range of 1-3.6. Especially, in some research projects carried out around 1990, the principle of *controlled rolling* was developed under more-severe conditions. That is, finishing rolling was carried out at much lower temperatures (around 500 $^{\circ}\text{C}$ -700 $^{\circ}\text{C}$) in a higher one-pass reduction of over 50%. Low finishing (deformation) temperature increases the driving force for ferrite transformation, and large one-pass deformation increases the density of nucleation site. Both lowering the finishing deformation temperature and increasing the last-deformation strain may lead to the occurrence of dynamic transformation (DT), that is, transformation occurs during deformation. As is schematically illustrated in **Fig. 1.2**, blue and red lines indicate static transformation (ST) and DT, respectively. The

transformation kinetics is higher at lower temperature (above the nose temperature of the C-curve), and the kinetics is accelerated by deformation of austenite, so that ferrite could be formed during the deformation, i.e., the occurrence of DT.

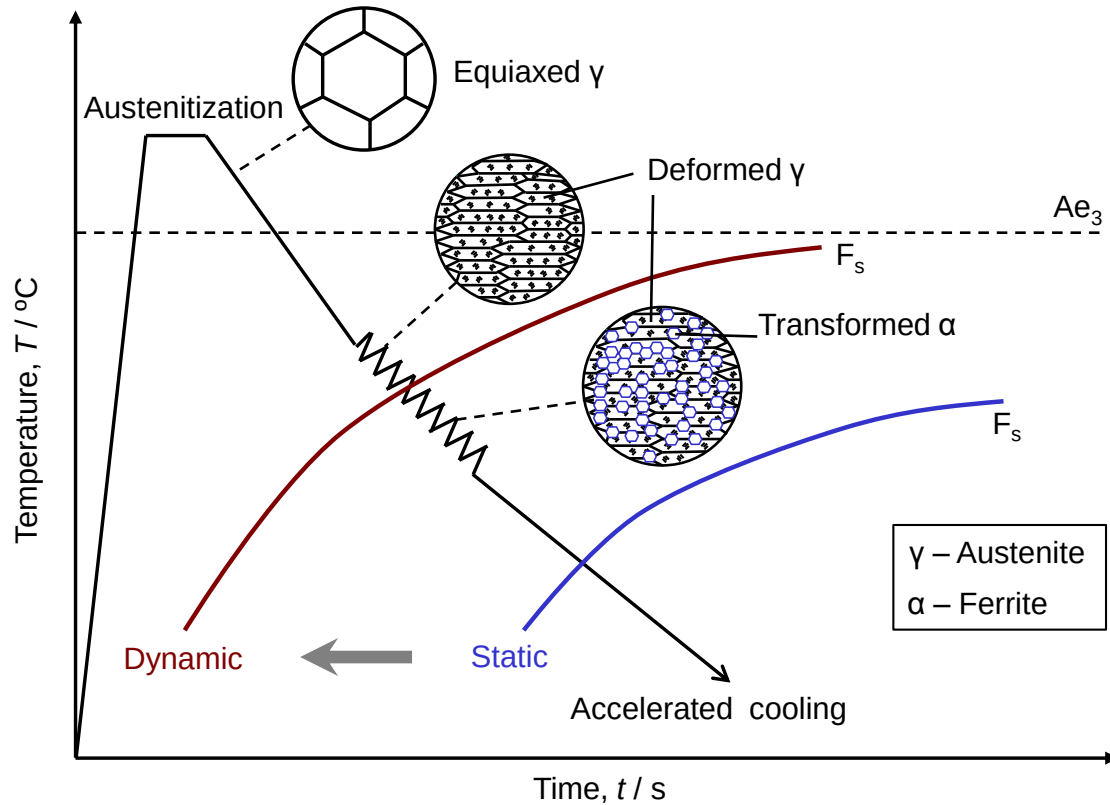


Fig. 1.2 Schematic illustration showing the possibility of the occurrence of DT.

1.3 Motivation for investigating dynamic ferrite transformation

The TMCP route using lower finishing temperature and higher last-pass strain is a good route for UFG ferrite formation. And during the process, DT could occur, which is considered to be effective for ferrite grain refinement. DT was firstly reported in the pioneering work by Yada and his colleague [27, 29, 30] in the mid 1980s. The ferrite grains

could be refined to around 1~4 μm after rapid cooling in TMCP route including DT. Many studies about DT in commercial alloys have been reported ever since [31-54]. However, up to now, the detailed features of DT have not yet been well clarified.

There are several significant issues which are still unclear or under debate. Some of these issues about DT are shown below:

Issue 1: The occurrence of DT has not been clearly confirmed. Even if ferrite is observed after quenching the deformed specimens [35, 46, 47], it is difficult to conclude whether the ferrite is dynamically formed or not. This is especially because it is difficult to confirm the occurrence of ferrite transformation during the process (especially during deformation). Several approaches have been proposed to determine the onset of DT metallographically [55-57], dilatometrically [58, 59] and by the analysis of stress-strain curves [58, 60, 61]. However, the metallographic and dilatometric methods are mainly employed on the specimens water-quenched or during isothermal holding (after deformation). They cannot make a direct confirmation of the occurrence of DT during the deformation. Stress-strain curve analysis can provide a direct view of dynamic softening process during the deformation. For instance, **Fig. 1.3** shows true stress-true strain curves of a plain low-carbon (0.15C) steel during compression under a strain rate of 0.01 s^{-1} at various deformation temperatures from 725 $^{\circ}\text{C}$ to 825 $^{\circ}\text{C}$. The flow stress increases with increasing strain until it reaches the maximum stress. After reaching the maximum stress, the flow stress decreases with

increasing strain, and finally keeps a constant value. However, this kind of softening can be resulted from not only DT but also dynamic recrystallization of austenite [62, 63] or adiabatic heating. Therefore, it is important and still necessary to develop an effective way to determine the occurrence of DT.

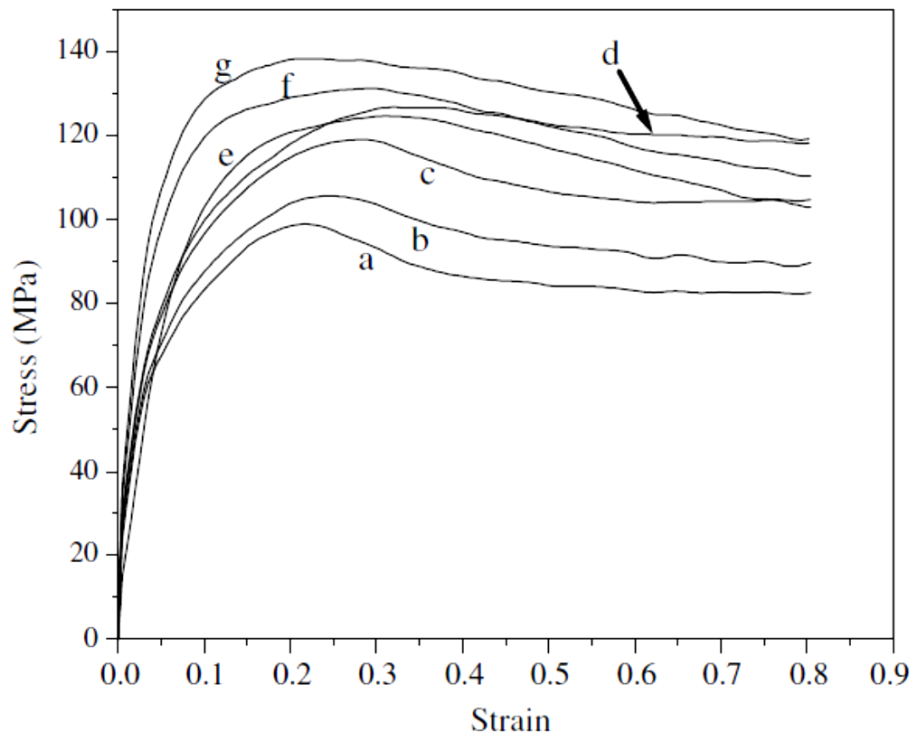


Fig. 1.3 True stress-true strain curves of a plain low-carbon (0.15C) steel during compression under a strain rate of 0.01 s^{-1} at various deformation temperatures: (a) 875 °C; (b) 850 °C; (c) 825 °C; (d) 800 °C; (e) 770 °C; (f) 750 °C; (g) 725 °C.

Issue 2: The effects of thermomechanical parameters on the kinetics of DT are still unclear.

Some works have reported that the formation of ferrite in DT is insensitive to strain rate [64, 65]. They claim that strain rate has little effect on changing the kinetics of DT. On the contrary, other reports have indicated that the volume fraction of ferrite increases with

decreasing the strain rate and strain rate is an important parameter to affect the kinetics of DT [66]. Another noteworthy parameter is the isothermal holding time. As is shown in **Fig. 1.4**, F_s and F'_s indicate the initiation of ferrite transformation with deformation (ϵ_1) and without deformation (ϵ_0), respectively. It shows that the ferrite transformation from austenite (γ) can be accelerated by deformation (ϵ_1). However, even without deformation (ϵ_0), ferrite transformation eventually occurs at temperatures below the equilibrium transformation temperature (Ae_3) during isothermal holding. This means that not only the strain rate but also the holding time before deformation affects the initiation of DT. Considering the controversial results previously reported, not only the effects of strain rate or isothermal holding time, but also the effects of other thermomechanical parameters (e.g., prior austenite grain size, strain and deformation temperature, etc.) on the kinetics of DT should be systematically studied for clearer understanding.

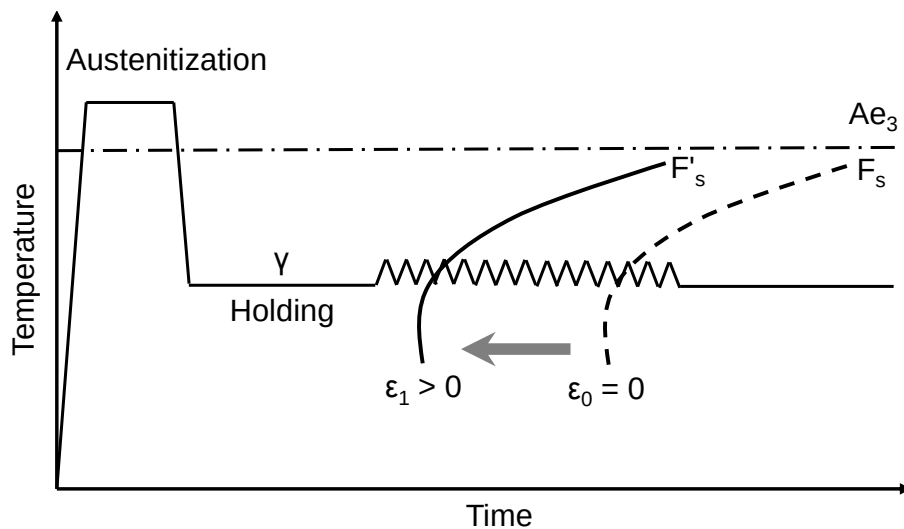


Fig. 1.4 Schematic illustration of the effect deformation and isothermal holding time.

Issue 3: The mechanism of ultrafine ferrite (UFF) formation is under debate. In most of the previous works, dynamically transformed ferrite exhibits equiaxed ultrafine morphology in the final structure [27, 32, 33, 67]. An example is shown in **Fig. 1.5** [67]. This is somewhat strange, since dynamically transformed ferrite should be continuously deformed after nucleation and growth, and if so they (at least some of them) should show more or less elongated morphologies. Up to now, it is under debate how these equiaxed UFF grains are formed. Two kinds of opinions have been mainly proposed. One is that deformation supplies sufficient nucleation sites for ferrite, leading to either two-dimensional or three-dimensional impingement between ferrite grains formed in high density, so that the fine and equiaxed ferrite grains are maintained [64, 68]. This opinion raises an important question: if UFF is formed due to large density of nucleation sites introduced into austenite, then what is the difference between DT and static transformation (ST) from deformed austenite? The other opinion claims the role of dynamic recrystallization (DRX) on the formation of equiaxed UFF [27, 69]. It is expected that the applied strain tends to be concentrate on ferrite, because ferrite is softer than (work-hardened) austenite especially at elevated temperatures. The deformation of ferrite at high temperatures may lead to the initiation of DRX, resulting in the formation of equiaxed UFF. This opinion raises another question: if DRX is involved in the refinement of ferrite, what is exactly the behavior of ferrite after it is initially transformed from austenite? The mechanism of the formation of

equiaxed UFF grains has not been clarified and thus will be particularly investigated in the present dissertation.

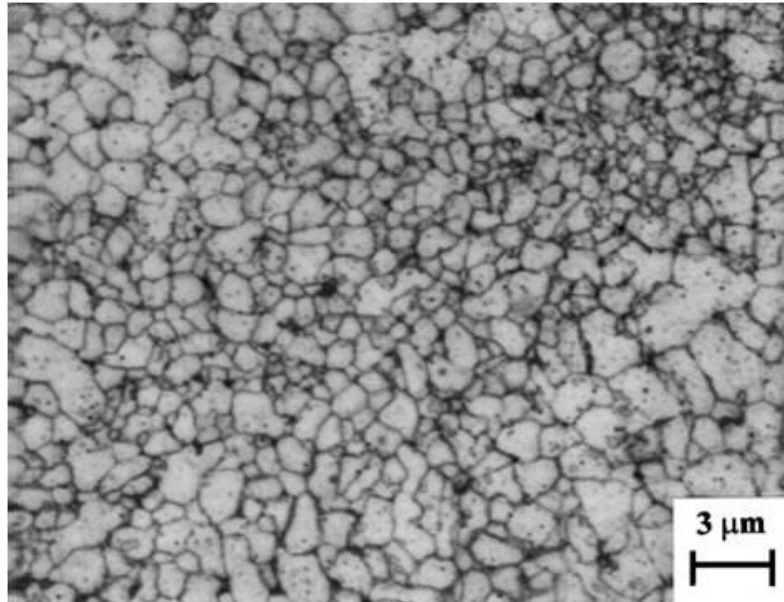


Fig. 1.5 An optical micrograph showing an example of ultrafine ferrite (UFF) structure formed through DT during a single pass rolling in a plain low-carbon steel [67].

Issue 4: There is still a big obstacle for industrial application of TMCP routes including DT.

The TMCP routes employing DT are thought to be potentially exploitable to industries due to the simplicity and efficiency in ferrite grain refinement. However, to fabricate UFF structures, there are still some academic and/or industrial barriers to overcome. With regard to the TMCP methods including DT, UFF grains have been only achieved on the surface layers of a strip [55]. To obtain a relatively homogeneous UFF structure throughout the thickness of strip, it usually requires large deformation (usually the accumulated strain is over 2) [70, 71], which could only be achieved at laboratory scale. The high required strain for

UFF formation is the main obstacle to the industrial application of the TMCP routes including DT. Hence, considerable fundamental research is still required to overcome this difficulty.

Not only the four issues mentioned above, but also many other unclear issues still exist in DT, which become barriers to our understanding and obstacles to industrial implementation. All of these issues inspire us to conduct systematic investigations of DT in the present study.

1.4 Outline of the dissertation

The present dissertation consists of six chapters. In **Chapter 1**, a full view of the present study such as background and purpose are introduced.

In **Chapter 2**, systematic and fundamental approaches are carried out to investigate the characteristics of DT under various thermomechanical conditions. Firstly, the occurrence of DT will be confirmed by stress-strain curves analysis in association with microstructural observation. And then the effects of various thermomechanical parameters (like strain rate, deformation temperature and prior austenite grain size, etc.) on the kinetics of DT and the ferrite microstructure are particularly investigated.

In **Chapter 3**, systematic experiments are designed to clarify the difference between DT and ST from deformed austenite. For this purpose, four experimental routes are designed and the differences in transformation kinetics and ferrite microstructures between the four routes are compared and discussed.

Chapter 4 aims to clarify the mechanism of UFF formation during the TMCP routes where DT occurs. For this purpose, microstructure evolution of ferrite grains is carefully observed during their first formation through DT as well as during subsequent deformation. Special attention is paid to examine the role of DRX in the formation of equiaxed UFF. Considering this motivation, investigation on DRX behavior of statically transformed ferrite is conducted. The similarity and difference between the DRX of dynamically transformed ferrite and the DRX of statically transformed ferrite is discussed. After the occurrence of DRX of dynamically transformed ferrite is confirmed, further investigation on the DRX behavior of dynamically transformed ferrite is conducted under different thermomechanical conditions.

In **Chapter 5**, on the basis of the obtained results in **Chapter 4**, potential strategies for fabricating UFF structures by controlling DT and DRX are explored. The aim of this chapter is to design simple TMCP routes to achieve ferrite grain refinement without large deformation.

Chapter 6 concludes the whole research findings in the present research work.

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Chapter 2: Characteristics of dynamic ferrite transformation (DT)

2.1 Introduction

Dynamic transformation (DT) is determined as the phase transformation occurring during deformation. DT, especially DT from austenite (γ ; FCC phase) to ferrite (α ; BCC phase) in steels has been studied since 1980s [1-3]. The thermomechanical processing routes including DT are considered potentially exploitable to achieve ferrite grain refinement (1-3 μm) utilizing the current industrial infrastructures. However, there are still a number of significant issues that are unclear or under debate in DT. One of the issues is that it is difficult to conclude whether the ferrite is dynamically transformed or not. This is especially because it is difficult to experimentally confirm the occurrence of ferrite transformation during the process (especially during the deformation). Several approaches have been proposed to determine the onset of DT metallographically [4-6], dilatometrically [7-8] and by the analysis of stress-strain curves [7, 9, 10]. However, the metallographic and dilatometric methods are mainly employed on the specimens water-quenched or during isothermal holding after deformation. They cannot make a direct confirmation of the occurrence of DT during the deformation. Stress-strain curve analysis can provide a direct view of the dynamic softening process during the deformation. The softening in true stress-true strain curves is considered to be caused by the occurrence of DT. However,

softening can be resulted from not only DT but also dynamic recrystallization of austenite or adiabatic heating. Therefore, it is still important and necessary to develop an effective way to confirm the occurrence of DT.

Most of the works up to date suggest that the kinetics of ferrite transformation and the ferrite grain refinement are significantly enhanced by DT [11-20]. However, the kinetics of DT and corresponding microstructure evolution of ferrite during DT have not yet been clarified. There still exist controversies about the effects of thermomechanical processing parameters on the kinetics of DT and ferrite microstructure. In some works, it is reported that the formation of dynamically transformed ferrite is independent of strain rate [21, 22], and the ferrite exhibits a fully equiaxed morphology. While in other works, it is said that the volume fraction of dynamically transformed ferrite increases with decreasing strain rate [23], and the ferrite is composed of both fine equiaxed grains and coarse elongated grains [24-26]. Not only the effect of strain rate, but also the effects of other thermomechanical parameters (such as, strain, deformation temperature and prior austenite grain size) on the characteristics of DT (e.g., kinetics and microstructure) should be systematically studied still.

In the present chapter, firstly a method for determining the occurrence of DT is proposed by analyzing stress-strain curves in association with microstructure observation. Then, the effects of thermomechanical parameters (e.g., strain rate, deformation temperature and

austenite grain size, etc.) on the kinetics of DT, and the ferrite microstructures are systematically investigated.

2.2 Experimental procedure

2.2.1 Material

The materials used in most of previous studies on DT were mainly commercial low-carbon steels with a small amount of alloying elements [27-30]. The deformation temperatures (usually between A_{r3} and A_{r1}) were relatively high. Therefore, DRX of austenite was possible to occur together with dynamic transformation to ferrite [30], which has made the research on DT very complicated. For example, both DRX of austenite and DT can cause softening in stress-strain curves, which increases the difficulty in confirming the occurrence of DT by analyzing stress-strain curves. Furthermore, for most of the commercial low-carbon steels, the incubation time for ferrite transformation is sometimes short, which would limit the condition of TMCP experiments.

Considering the issues mentioned above, a model alloy Fe-10Ni-0.1C is designed in the present study. Adding 10 wt.% nickel is to decrease the austenite-ferrite transformation temperature for enlarging the temperature range where austenite is stable and to prolong the incubation period for the onset of ferrite transformation. Ortho-equilibrium temperature (A_{e3}) and para-equilibrium temperature (A_{p3}) of the Fe-10Ni-0.1C alloy, calculated by

Thermo-Calc software, are 686 °C and 583 °C, respectively. The detailed chemical composition of this alloy is shown in **Table 2.1**. The as-received hot-rolled plate 15 mm thick was cold-rolled to a thickness of 11.2 mm with a reduction of 25%, then homogenized at 1100 °C for 172.8 ks in vacuum followed by water quenching. Cylindrical specimens with a height of 12 mm and a diameter of 8 mm were machined from the homogenized plate and installed in a thermomechanical processing simulator (Thermecmator-Z, Fuji Electronic Industrial Co. Ltd.) for conducting the experiments physically simulating thermomechanical processes.

Table 2.1 Chemical composition of the alloy studied (mass%).

C	Ni	Mn	P	Si	Al	S	Fe
0.111	10.08	0.01	0.001	0.006	0.33	0.0017	Bal.

2.2.2 Time-temperature-transformation (TTT) diagram

The TTT diagram of the Fe-10Ni-0.1C alloy was measured by a dilatometer (Formator-F, Fuji Electronic Industrial Co., Ltd.). Cylindrical specimens with 10 mm in height and 3 mm in diameter were machined from the homogenized plate of the Fe-10Ni-0.1C alloy and austenitized at 1000 °C for 300 s in the dilatometer, resulting in an average austenite grain size of 125 µm. After austenitization, the specimens were cooled at a rate of 30 °C s⁻¹ to the temperatures ranging from 390 °C to 480 °C using helium gas, and then isothermally held at the temperatures until a volume change (expansion) due to ferrite transformation was

detected. Because the incubation time for the onset of ferrite transformation at the temperatures above 500 °C was fairly long in the present alloy, the TTT diagram above 500 °C was constructed from microstructure observations.

2.2.3 Thermomechanical process

Figure 2.1 shows the experimental conditions simulating thermomechanical processes including deformation of austenite. The specimens were austenitized at 1200 °C for 300 s, at 1000 °C for 300 s or at 700 °C for 900 s to obtain different austenite grain sizes (d_γ) of around 450 μm , 125 μm or 10 μm , respectively. The austenitized specimens were cooled to the deformation temperatures ranging from 600 °C to 480 °C at a rate of 30 °C s⁻¹ by nitrogen gas, held for 60 s to homogenize the temperature of the cylindrical specimens, and then uniaxially compressed to various strains ranging from 0.05 to 0.92 (5% to 60% reduction in height) at strain rates ranging from 10⁰ s⁻¹ to 10⁻³ s⁻¹ followed by water-quenching for minimizing static change of microstructure during cooling. The temperature of the specimen during the physical simulation of TMCP was controlled by an induction heating system and monitored through a thermocouple welded on the side surface of the specimen. Mica and glass powder were used as the insulator and lubricant to minimize the temperature decrease and the friction between the contact surfaces of the anvils and the specimen during straining. The water injection was precisely controlled to minimize the time interval until

the initiation of cooling. Information of the applied load and stroke during hot-compression could be converted into flow stress and compressive strain so that true stress-true strain curve of each specimen was obtained.

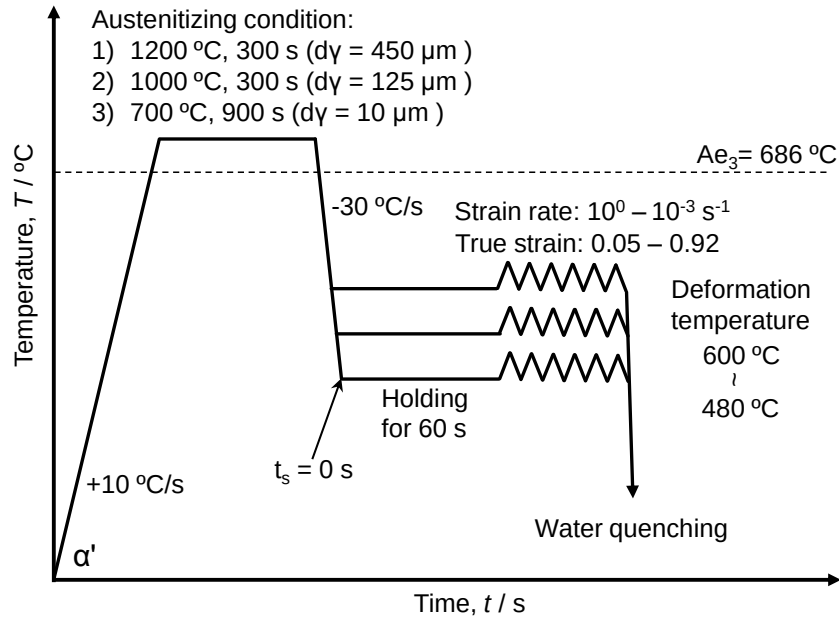


Fig. 2.1 Schematic illustration showing the experimental conditions for simulating thermomechanical processes including deformation of austenite.

2.2.4 Microstructure observation

Microstructures at the center of the cylindrical specimens on the section parallel to the compression axis were characterized by optical microscopy (OM), a field-emission type scanning electron microscope (FE-SEM, FEI XL30S FEG), electron back-scattering diffraction (EBSD) analysis performed in the FE-SEM and a transmission electron microscope (TEM, Philips CM200FEG). The specimens were mechanically polished and then etched using a 3% nital solution for the OM and SEM observations. For the EBSD

measurement, the specimens were mechanically polished and then electrically polished in a solution of 10% HClO_4 and 90% CH_3COOH at 20 °C. The EBSD measurement was performed using TSL OIM Data Collection system, and the obtained EBSD data were analyzed using TSL OIM Analysis software. For the TEM observations, thin-foil specimens were prepared by twin-jet electropolishing using the same solution as that used for EBSD.

For investigating the transformation kinetics and microstructure evolution of ferrite, the volume fraction and grain size of ferrite are two important parameters. **Figure 2.2** shows examples for measuring (a) the volume fraction and (b) the grain size of dynamically transformed ferrite. **Figure 2.2 (a)** is an OM image of a microstructure composed of ferrite and martensite, and (b) is an EBSD grain boundary map of a microstructure of ferrite. “F” and “M” indicates ferrite and martensite, respectively. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively, in the EBSD boundary map (b). The volume fraction of ferrite (**Fig. 2.2(a)**) was measured by point-counting method on OM images with low magnification. More than 1900 grids were drawn on each OM image. The grain sizes of ferrite (**Fig. 2.2(b)**) along the compression axis were measured by linear interception method on the EBSD maps counting all boundaries with misorientation larger than 2 ° (defined as d^{All}) or only counting boundaries with misorientation larger than 15 ° (defined as d^{HAGB}). More than 20 lines were drawn along the compression axis on each EBSD map.

Along each line (green color in the figure), there are at least 30 ferrite grains used for measuring the grain size. If the number of grains in one line is less than 30, additional EBSD maps taken from other observed areas of the specimen will be used for calculating an average grain size. Therefore, the total number of measured grains for one microstructure is more than 600. According to the methods introduced above, the volume fraction and grain size of ferrite in **Fig. 2.2(a)** and **Fig. 2.2(b)** are 13.4% and 0.93 μm , respectively.

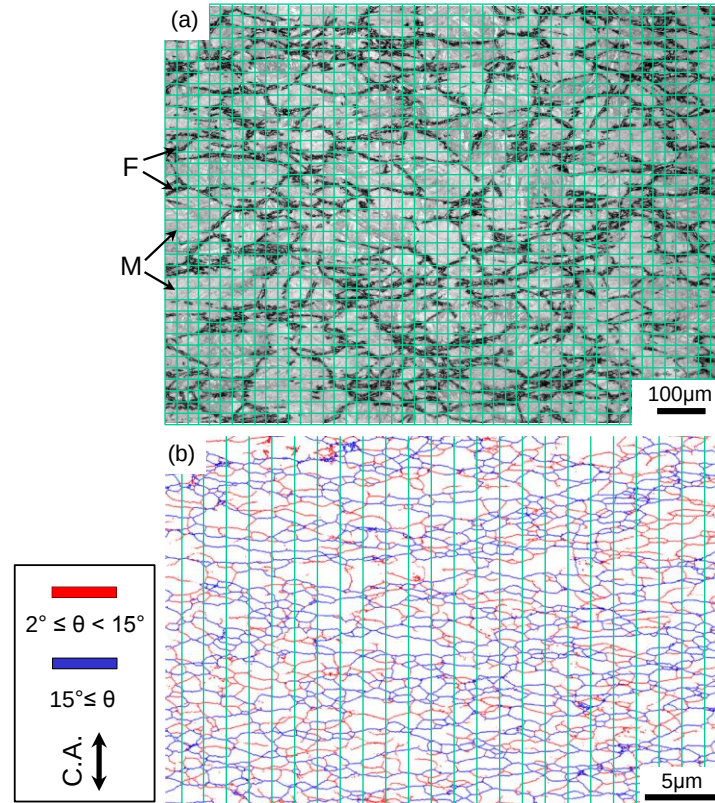


Fig. 2.2 (a) OM image and (b) EBSD grain boundary map for illustrating the methods to measure volume fraction and grains size of dynamically transformed ferrite. (a) is a microstructure composed of ferrite and martensite. “F” and “M” indicates ferrite and martensite, respectively. (b) is a microstructure composed of ferrite. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines in (b), respectively. Compression axis is along the vertical direction of the images.

2.3 Results and discussion

2.3.1 Kinetics and microstructure in static ferrite transformation (ST)

Figure 2.3 is a TTT diagram of static transformations in the Fe-10Ni-0.1C alloy. There is no transformation occurring within 60 s at temperatures between 480 °C and 600 °C in this alloy. This indicates that the microstructures of the specimens are fully austenite before applying the deformation in the TMCP experiments (**Fig. 2.1**). The incubation time for the onset of ferrite transformation at the temperatures above 500 °C is very long. For example, it requires 12,600 s for 0.7% formation of ferrite at 520 °C. In the present study, most of the TMCP experiments were carried out at 520 °C, because the kinetics of static transformation is sufficiently slow, thus it provides a possibility to investigate the characteristics of DT in detail through changing the TMCP parameters.

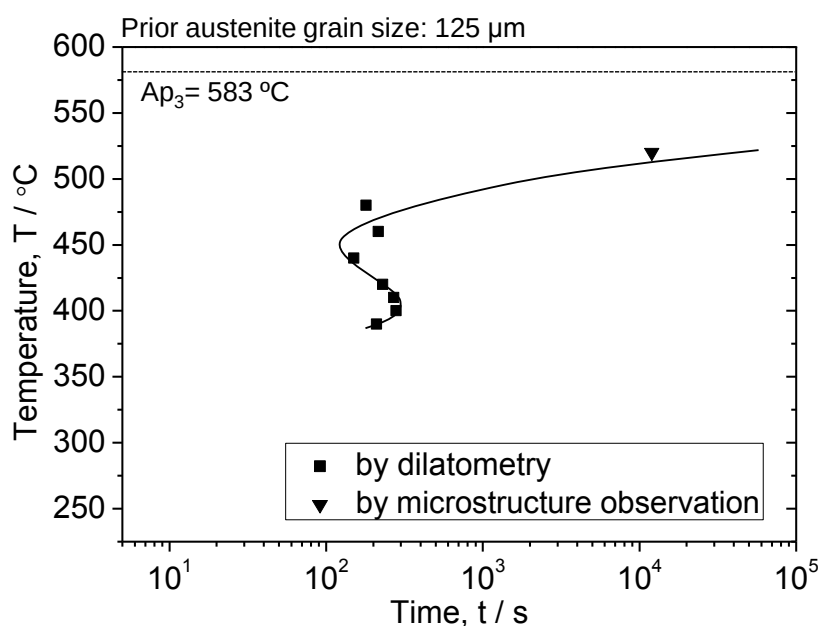


Fig. 2.3 Time-temperature-transformation (TTT) diagram of the Fe-10Ni-0.1C alloy.

Figure 2.4 shows (a) an OM image, (b) an image quality (IQ) map and (c) an grain average misorientation (GAM) map of the specimen austenitized at 1000 °C and then isothermally held at 520 °C for 88,200 s (without deformation) followed by water-quenching. The IQ map and GAM map were obtained from EBSD measurement and analysis for the identical area. The IQ map constructed from EBSD data provides a useful visualization of ferrite and martensite microstructure and can be used to distinguish the two phases by the contrast in the map. The GAM map, which shows the average misorientation angle among all pairs of adjacent points in one grain, can be used to evaluate the degree of misorientation inside the grain. The range of the GAM value in (c, d) is from 0 to 2°, a higher value indicates higher misorientation. “F” and “M” represent ferrite and martensite, respectively, which is the case for all the OM images shown in the chapter hereafter. In the EBSD maps (c, d), low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. In OM observations, it is rather easy to distinguish ferrite and martensite. For example in **Fig. 2.4(a)**, ferrite reveals as light areas while martensite reveals as dark areas. In the present study, EBSD measurements were frequently used for obtaining crystallographic information. Principally phases having different crystalline structures can be easily distinguished in EBSD. In fact, BCC and FCC phases were detected in the present EBSD analysis since Kikuchi-line patterns are different depending on crystal structures, and austenite phase having FCC

structure could be easily distinguished. However, both ferrite and martensite that are major phases in this study have BCC structure, so that it is difficult to distinguish ferrite from martensite. Although strictly speaking martensite in carbon steels have tetragonal structure (BCT), it is still difficult to distinguish ferrite and martensite in the present study because of small tetragonality in the low-C martensite. Therefore, in the EBSD analysis of the present study, ferrite and martensite were distinguished by the use of IQ (**Fig. 2.4(b)**) and GAM maps (**Fig. 2.4(c)**). In the IQ map, the contrast of ferrite and martensite is different, like OM structures (**Fig. 2.4(a)**). White area and dark gray area indicate ferrite and martensite, respectively. In the GAM map, martensite exhibits higher GAM values (dark red color) than ferrite, because martensite phase has high density of dislocations and fine substructures which can increase its interior misorientation. Therefore, by combining the information from IQ map (**Fig. 2.4(b)**) and GAM map (**Fig. 2.4(c)**), ferrite can be distinguished from martensite. The method for distinguishing ferrite from martensite as well as austenite applies to all the EBSD maps in the dissertation.

In order to particularly investigate ferrite microstructure, non-ferrite phases (i.e., martensite and/or austenite) in the EBSD maps (e.g., **Fig. 2.4(c)**) are painted in black, as is shown in **Fig. 2.4(d)**. From this GAM map, it is clearly shown that the statically transformed ferrite exhibits polygonal coarse grains surrounded by HABs and contains a small amount of LABs. The average GAM value of the ferrite grains is around 0.31 %

indicating that the average misorientation within the statically transformed ferrite is low [31].

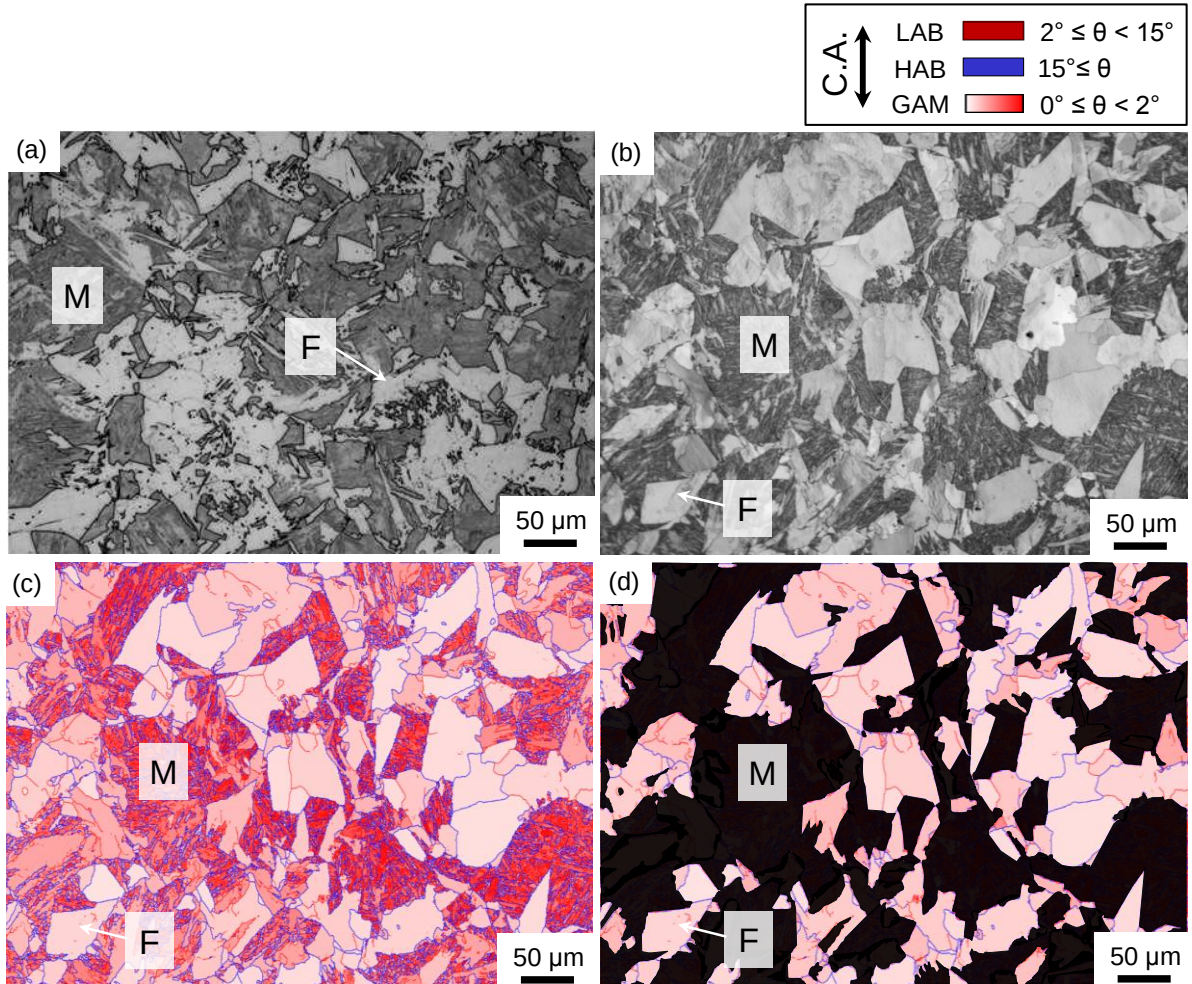


Fig. 2.4 (a) Optical microscope (OM) image, (b) image quality (IQ) map and (c, d) grain average misorientation (GAM) maps obtained by EBSD measurement of the specimen isothermally held at 520 °C for 88,200 s without deformation, followed by water-quenching. “F” and “M” represent ferrite and martensite, respectively. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively, in (c) and (d). In (c), the area with dark red color (high GAM value) represents martensite. The area with lighter red color represents ferrite. Martensite is painted in black in (d).

Figure 2.5 shows changes in grain size and volume fraction of statically transformed ferrite in isothermal holding at 520 °C for different periods of time. The static ferrite

transformation starts at about 12,000 s, and with increasing the holding time from 12,600 s to 88,200 s, the volume fraction of ferrite increases from 0.7% to 39.1%, and the grain size increases from 11.9 μm to 18.4 μm .

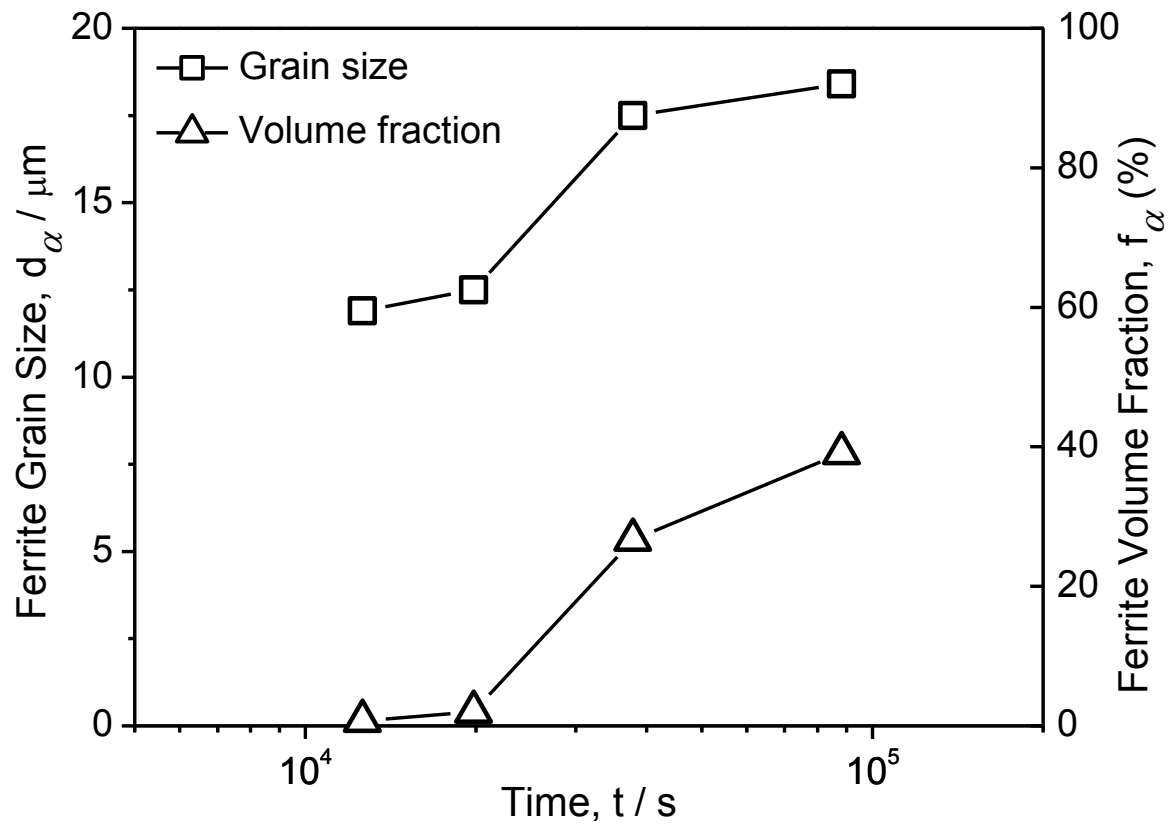


Fig. 2.5 Changes in grain size and volume fraction of statically transformed ferrite in the specimens isothermally held at 520 $^{\circ}\text{C}$ for different periods of time after austenitization at 1000 $^{\circ}\text{C}$ for 300 s.

2.3.2 Occurrence of DT

The occurrence of DT is investigated under various thermomechanical conditions, i.e., different strain rates, temperatures and austenite grain sizes. As mentioned before, the kinetics of static transformation (ST) is sufficiently slow at 520 °C, which provides a possibility to investigate the features of DT in detail. Therefore, most of the TMCP experiments were carried out at 520 °C in the present study.

Figure 2.6 shows true stress-true strain curves of the specimens deformed (a) at 520 °C at different strain rates ranging from 10^{-1} s^{-1} to 10^{-3} s^{-1} , (b) at a strain rate of 10^{-2} s^{-1} at different temperatures from 600 °C to 480 °C and (c) at a strain rate of 10^{-2} s^{-1} at 520 °C with different austenite grain sizes from 10 μm to 450 μm (after different austenitization conditions). In general, the flow stress increases with increasing the strain rate (**Fig. 2.6(a)**) or decreasing the deformation temperature (**Fig. 2.6(b)**), which is a typical tendency in high temperature deformation of metals. The stress-strain curves in **Fig. 2.6(a)** and **(c)** exhibit a slight peak followed by a decrease in flow stress, indicating softening during deformation. As discussed in the TTT diagram (**Fig. 2.3**), the specimens had fully austenite microstructures till the onset of deformation. Therefore, the softening is considered to be mainly due to the occurrence of DT, as has also been reported in literatures [9, 32, 33]. However, the occurrence of DT cannot be confirmed only by the softening in stress-strain curves.

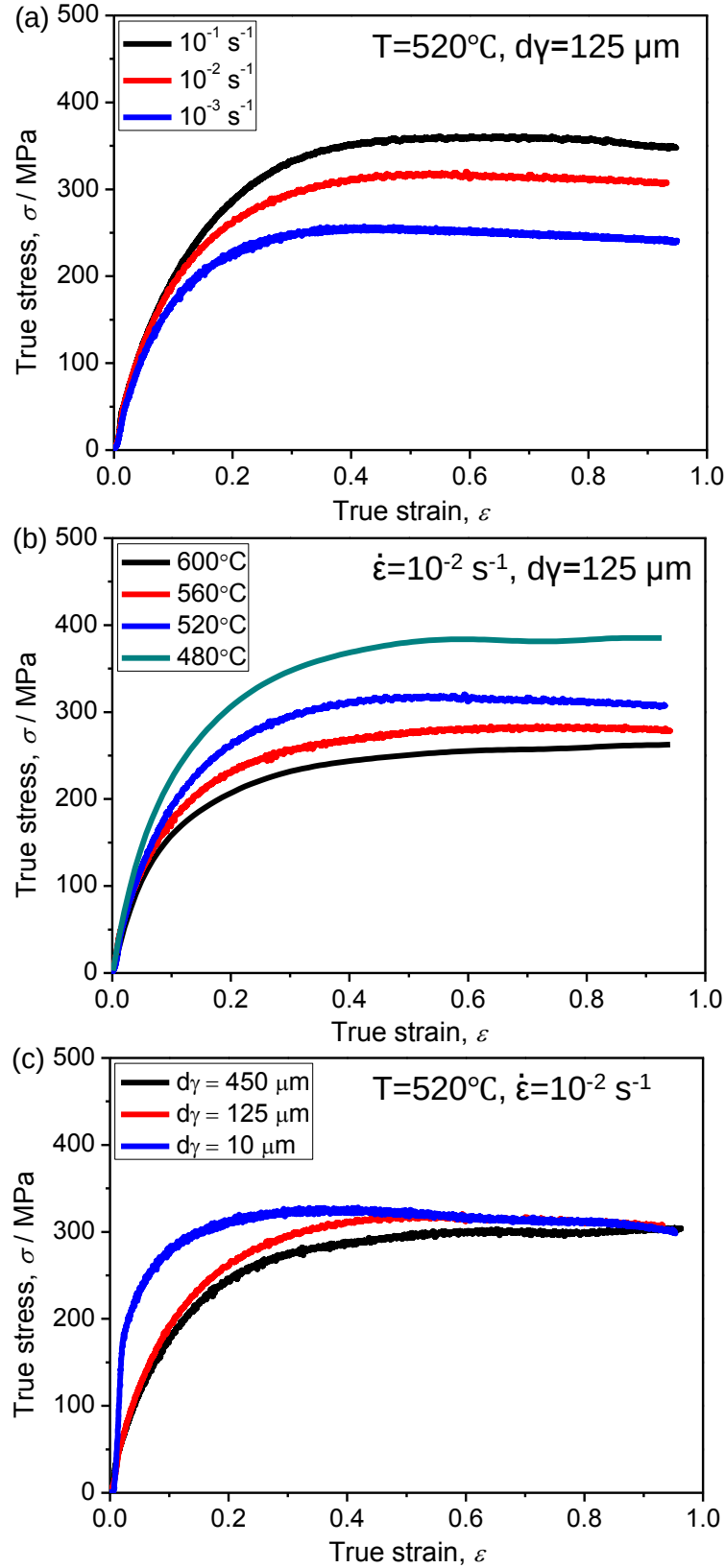


Fig. 2.6 True stress-true strain curves of the specimens deformed (a) at 520°C at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} , (b) at a strain rate of 10^{-2} s^{-1} at different temperatures from 600°C to 480°C and (c) at a strain rate of 10^{-2} s^{-1} at 520°C with different austenite grain sizes from $10\text{ }\mu\text{m}$ to $450\text{ }\mu\text{m}$ (after different austenitization conditions).

The microstructures of the specimens corresponding to the true stress-true strain curves of **Fig. 2.6** are shown in **Fig. 2.7**. Ferrite is observed as light etched phase in OMs, and “F” and “M” represent ferrite and martensite, respectively. **Figure 2.7(a)-(c)** shows OM images of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at 520 $^{\circ}\text{C}$ at different strain rates of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} . Ferrite is observed at all strain rates. The volume fraction of ferrite increases with decreasing the strain rate. **Figure 2.7(b)-(d)** shows OM images of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (d) 600 $^{\circ}\text{C}$, (e) 560 $^{\circ}\text{C}$ and (f) 480 $^{\circ}\text{C}$. Ferrite is formed at all the deformation temperatures. The volume fraction of ferrite increases with decreasing the deformation temperature. **Figure 2.7(g)-(i)** show OM images of the specimens with different austenite grain sizes of (g) 450 μm , (h) 125 μm and (i) 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. Ferrite is formed in all the specimens with different grain sizes. The volume fraction of ferrite increases with decreasing the austenite grain size. According to the OM images of the water-quenched specimens, it could be confirmed that ferrite is surely formed under various deformation conditions.

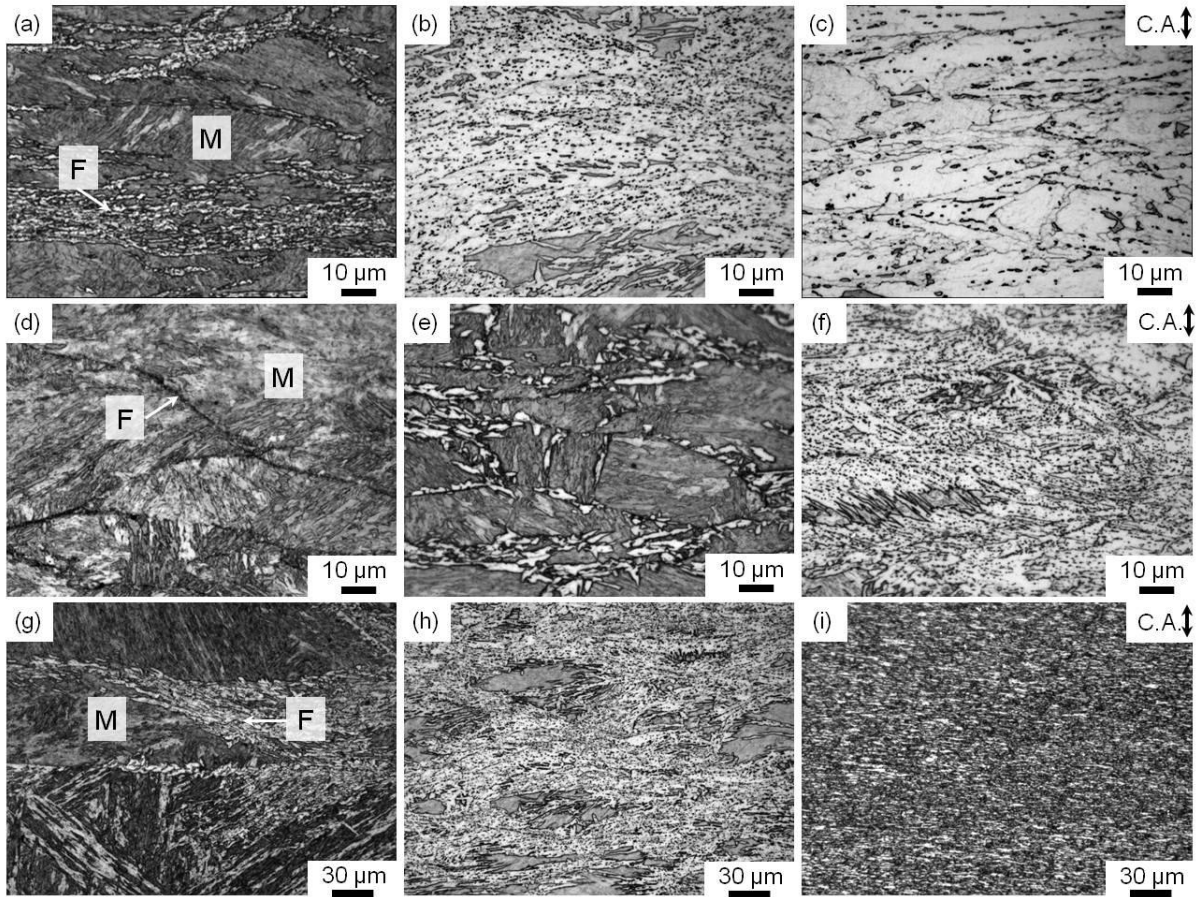


Fig. 2.7 (a)-(c): OM images of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at 520 $^{\circ}\text{C}$ at different strain rates of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} . (b)-(d): OM images of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (d) 600 $^{\circ}\text{C}$, (e) 560 $^{\circ}\text{C}$ and (f) 480 $^{\circ}\text{C}$. (g)-(i): OM images of the specimens with different austenite grain sizes of (g) 450 μm , (h) 125 μm and (i) 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. “F” and “M” represent ferrite and martensite, respectively. Compression axis is along the vertical direction of the image.

Figure 2.8 is a TEM image of ferrite in the specimen deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$ (corresponding to the OM image in **Fig. 2.7(b)**). The ferrite contains a number of dislocations and dislocation substructures, indicating a deformed microstructure. The ferrite dynamically transformed at a certain stage of compression is further deformed during the subsequent compression, resulting in a development of deformed

structure [31]. Therefore, it could be confirmed that DT occurs in the TMCP experiments in the present study.

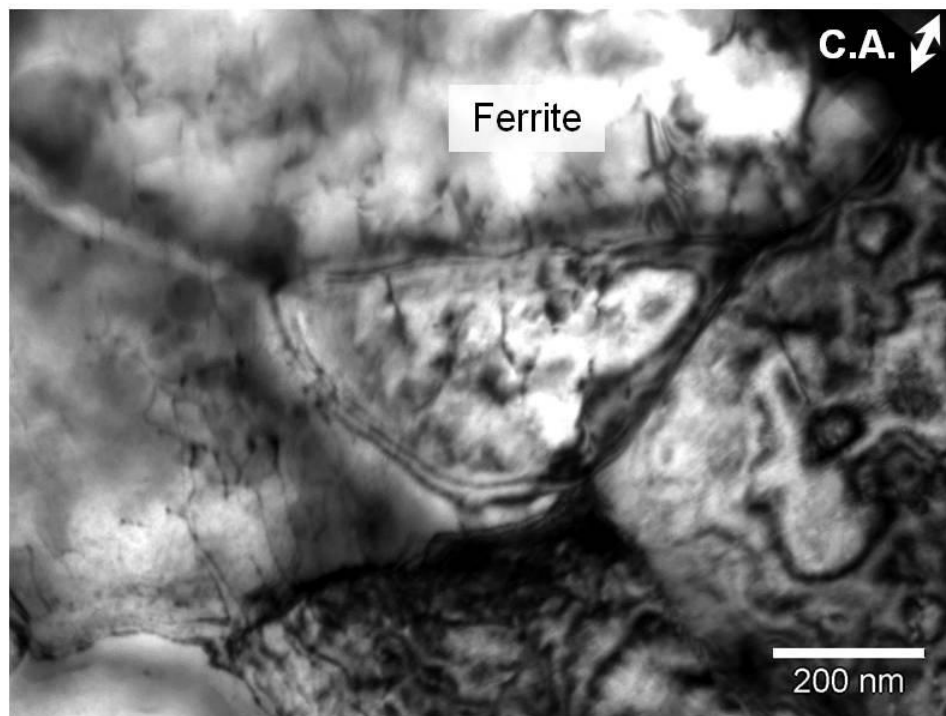


Fig. 2.8 A TEM image of ferrite in the specimen with an austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 °C (corresponding to the OM image in **Fig. 2.7(b)**). Compression direction is marked in the image.

The OM images in **Fig. 2.7** and the TEM image in **Fig. 2.8** confirmed DT (ferrite formation) in most of the deformation conditions. But they do not give us at what strain the transformation starts. In the next, the critical strain for the onset of DT is evaluated by analyzing softening in the true stress-true strain curves.

The critical strain for the onset of DT is determined by the double differentiation method, which is widely used for analyzing the initiation of dynamic softening process (such as DT and DRX) during deformation of austenite [7-10, 34, 35]. According to the double differentiation method, the initiation of DT corresponds to the following condition:

$$\frac{\partial}{\partial \sigma} \left(-\frac{\partial \theta}{\partial \sigma} \right) = 0 \quad (2.1)$$

where σ is the true stress, θ is the work-hardening rate ($\partial \sigma / \partial \varepsilon$). Equation (2.1) means that the onset of DT corresponds to the inflection point in the $\theta - \sigma$ curve, i.e., the minimum point in the $-(\partial \theta / \partial \sigma)$ versus σ curve. As an example, a true stress-true strain curve of the specimen deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 °C is shown in **Fig. 2.9(a)**. The stress-strain curve is fitted with a ninth order polynomial. The $\theta - \sigma$ curve and the $-(\partial \theta / \partial \sigma)$ versus σ curve can be obtained from the fitted stress-strain curve as shown in **Fig. 2.9(b)** and **(c)**, respectively. From the local minimum in **Fig. 2.9(c)**, the critical stress value (292 MPa) is determined and correspondingly the critical strain for the onset of DT under this deformation condition is obtained as 0.284 from the stress-strain curve (**Fig. 2.9(a)**). **Figure 2.9(d)** shows an OM image of the specimen deformed to a strain of 0.288 (very close to the calculated critical strain) at a strain rate of 10^{-2} s^{-1} at 520 °C. A small amount of ferrite is mainly formed along prior austenite grain boundaries. The fraction of dynamically transformed ferrite is around 6%, indicating a very early stage of

transformation. It can be concluded, therefore, that the double differentiation method can be used for determining the onset of DT.

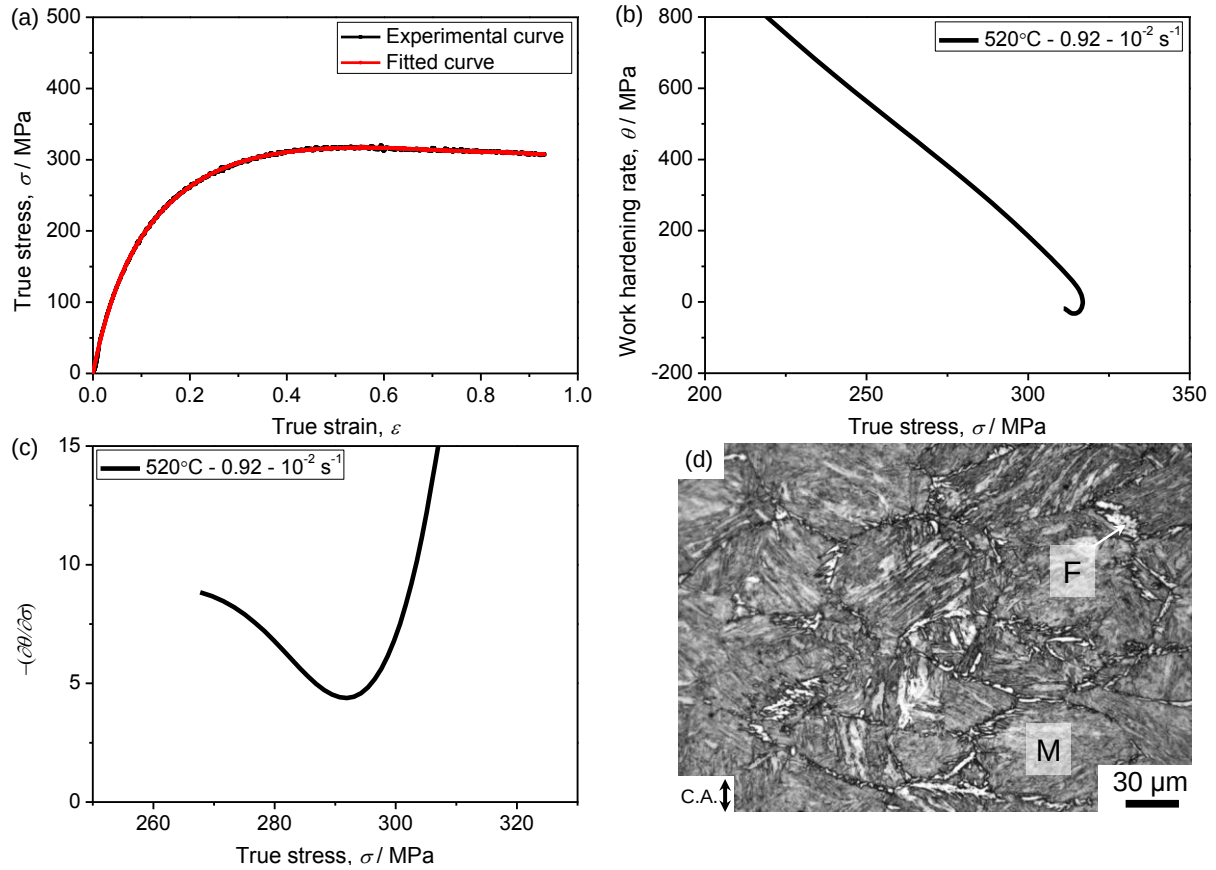


Fig. 2.9 (a) Experimental and fitted true stress-true strain curves, (b) $\theta - \sigma$ curve, and (c) $-(\partial\theta/\partial\sigma)$ versus σ curve of the specimen deformed at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. (d) An OM image of the water-quenched specimen deformed to a strain of 0.288 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. “F” and “M” represent ferrite and martensite, respectively. Compression axis is along the vertical direction of the image.

Figure 2.10 shows $-(\partial\theta/\partial\sigma)$ versus σ curves of the specimens deformed to a strain of 0.92 (a) at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C , (b) at a strain rate of 10^{-2} s^{-1} at different temperatures from 560°C to 480°C , and (c) at a strain rate of 10^{-2} s^{-1} at 520°C with different austenite grain sizes. The austenite grain size of the specimens in (a) and (b) was $125 \mu\text{m}$. It was hard to apply the double differentiation method to the curves of the specimens in which the transformation kinetics was either too fast or too slow. For example, the dynamic softening of the stress-strain curves of the specimen with an austenite grain size of $10 \mu\text{m}$ could not be detected, because the ferrite was formed at very early stages of transformation (**Fig. 2.6(c)**). In the case of the deformation at a strain rate of 10^{-2} s^{-1} at 600°C , there was very little amount of ferrite (0.3%) formed after a strain of 0.92 (**Fig. 2.7(d)**), which was probably not sufficient to cause softening in the stress-strain curve (**Fig. 2.6(b)**).

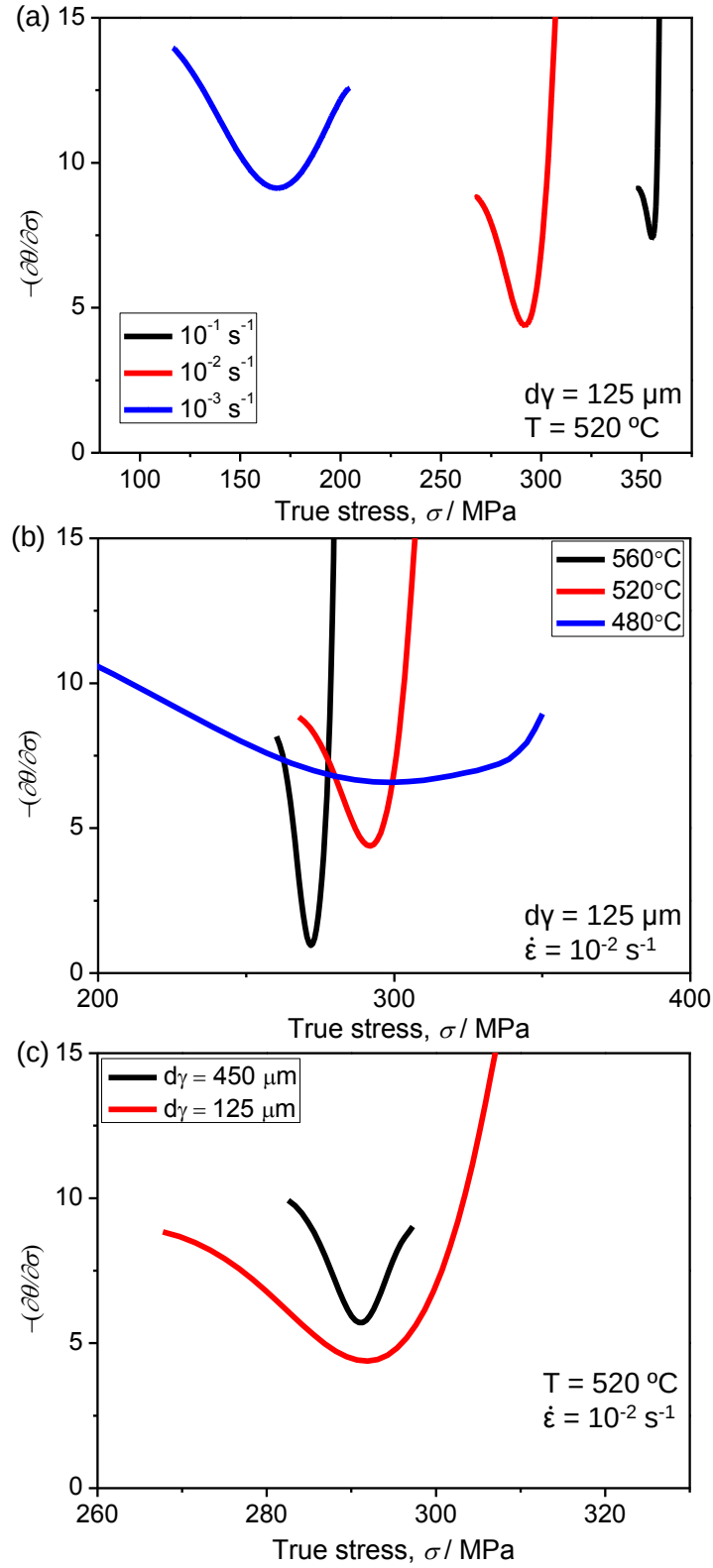


Fig. 2.10 $-(\partial\theta/\partial\sigma)$ versus σ curves of the specimens deformed to a strain of 0.92 (a) at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C , (b) at a strain rate of 10^{-2} s^{-1} at different temperatures from 560°C to 480°C , and (c) at a strain rate of 10^{-2} s^{-1} at 520°C with different austenite grain sizes from $125 \mu\text{m}$ to $450 \mu\text{m}$. The austenite grain size of the specimens in (a) and (b) was $125 \mu\text{m}$.

Figure 2.11 shows the critical strains obtained from the minima in the $-(\partial\theta/\partial\sigma)$ versus σ curves shown in **Fig. 2.10**. The lower the strain rate (**Fig. 2.11(a)**), lower the deformation temperature (**Fig. 2.11(b)**) and finer the austenite grain size (**Fig. 2.11(c)**), the critical strain for the onset of DT becomes smaller, i.e., onset of DT is enhanced.

The occurrence of DT was confirmed by a systematic analysis of stress-strain curves in association with microstructure observations. The critical strains for the onset of DT were determined by the double differentiation method. Microstructure obtained at the critical strain indicates an early stage of transformation. Therefore, it can be concluded that the double differentiation method is applicable to predict the occurrence of DT. The critical strain for the onset of DT is greatly affected by the thermomechanical processing parameters, that is, the onset strain is smaller under the deformation conditions of lower strain rates, lower deformation temperatures and finer austenite grains.

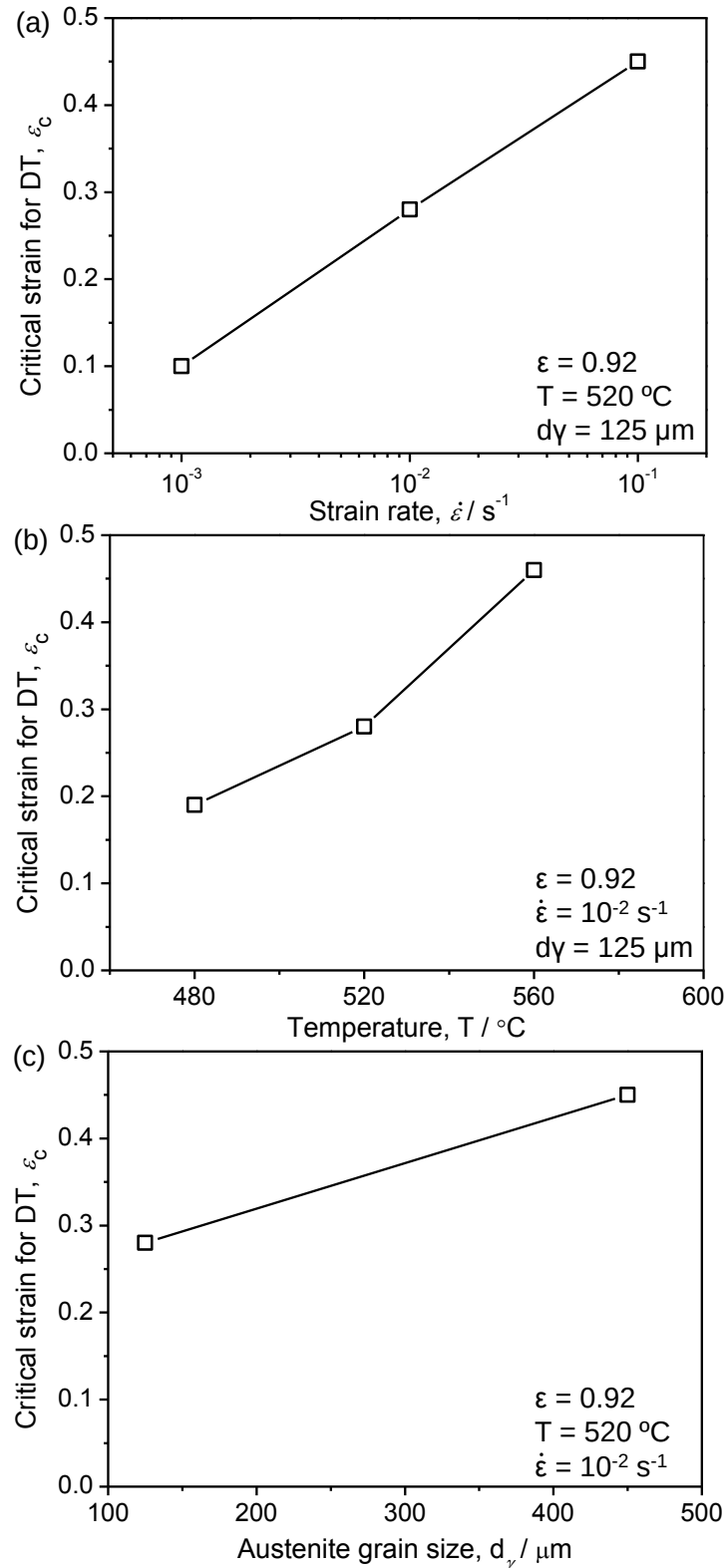


Fig. 2.11 Changes in the critical strains for the onset of DT obtained by the double differentiation method in the specimens deformed to a strain of 0.92 (a) at different strain rates from 10^{-1}s^{-1} to 10^{-3}s^{-1} at 520°C , (b) at a strain rate of 10^{-2}s^{-1} at different temperatures from 560°C to 480°C , and (c) at a strain rate of 10^{-2}s^{-1} at 520°C with different austenite grain sizes from $125 \mu\text{m}$ to $450 \mu\text{m}$. Austenite grain size in (a) and (b) is $125 \mu\text{m}$.

2.3.3 Kinetics of DT

In the last section, the occurrence of DT is confirmed by the analysis of stress-strain curves and the microstructure observation under various thermomechanical processing parameters (i.e., strain rate, temperature and prior austenite grain size). In this section, the effects of these parameters on the kinetics of DT are systematically investigated.

Figure 2.12 shows OM images of the specimens with an austenite grain size of 125 μm deformed to different strains from 0.16 to 0.92 at strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$. Black broken lines in **Fig. 2.12(b), (e) and (i)** indicate prior austenite grain boundaries (PAGBs). The volume fractions of dynamically transformed ferrite are shown on the top right corners of the micrographs, which is the case for all OM images in the chapter hereafter. At a strain rate of 10^{-1} s^{-1} (**Fig. 2.12 (a)-(d)**), DT did not occur till a strain of 0.16, and only martensite could be observed. In the specimen deformed to a strain of 0.36, ferrite initially formed along PAGBs. And then ferrite formed at the interior of austenite grains with increasing the strain to 0.60 and 0.92. At a lower strain rates (i.e., at 10^{-2} s^{-1} in **Fig. 2.12(e)-(h)** or at 10^{-3} s^{-1} in **Fig. 2.12(i)-(l)**), ferrite initially formed along PAGBs, then grew into the interior of the austenite grains at higher strains. The effects of stain and strain rate on the formation of ferrite by DT could be qualitatively described as follows: at a constant strain rate (along each row of the figure), the volume fraction of dynamically transformed ferrite increases with increasing the strain. At a constant strain (along each column of the

figure), the volume fraction of ferrite increases with decreasing the strain rate, indicating that DT proceeds much more at lower strain rate.

The effect of temperature on the formation of ferrite by DT is shown in OM images in **Fig. 2.13**, which reveals microstructures in the specimens with an austenite grain size of 125 μm deformed to different strains from 0.16 to 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures from 560 $^{\circ}\text{C}$ to 480 $^{\circ}\text{C}$. The microstructures of the specimens deformed at 600 $^{\circ}\text{C}$ are not shown, because the volume fraction of ferrite is very small even after a strain of 0.92 (around 0.3%, as shown in **Fig. 2.7(d)**). At 560 $^{\circ}\text{C}$, ferrite was not observed until the applied strain of 0.60. The occurrence of ferrite transformation becomes earlier with decreasing the temperature to 520 $^{\circ}\text{C}$ (**Fig. 2.13(e)**) and 480 $^{\circ}\text{C}$ (**Fig. 2.13(i)**). At a constant temperature (along each row of the figure), the volume fraction of dynamically transformed ferrite increased with increasing the strain. At a constant strain (along each column of the figure), the volume fraction of ferrite increases with decreasing temperature, indicating that DT proceeds much more at lower deformation temperature.

The effect of austenite grain size on the formation of ferrite by DT is shown in OM images in **Fig. 2.14**, which reveals microstructures in the specimens with different austenite grain sizes from 450 μm to 10 μm deformed to different strains from 0.16 to 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. In the case of a coarse austenite grain size (450 μm , **Fig. 2.14(a)-(d)**), the progress of DT is relatively slow. A very small amount of ferrite was

observed along PAGBs at a strain of 0.36. At the same strain of 0.36, the volume fraction of dynamically transformed ferrite increased markedly with decreasing the austenite grain size. This tendency could also be seen in the specimens deformed to other strains. Therefore, it could be concluded that the progress of DT is accelerated by decreasing austenite grain size.

$d\gamma = 125 \mu\text{m}$, $T = 520 \text{ }^{\circ}\text{C}$

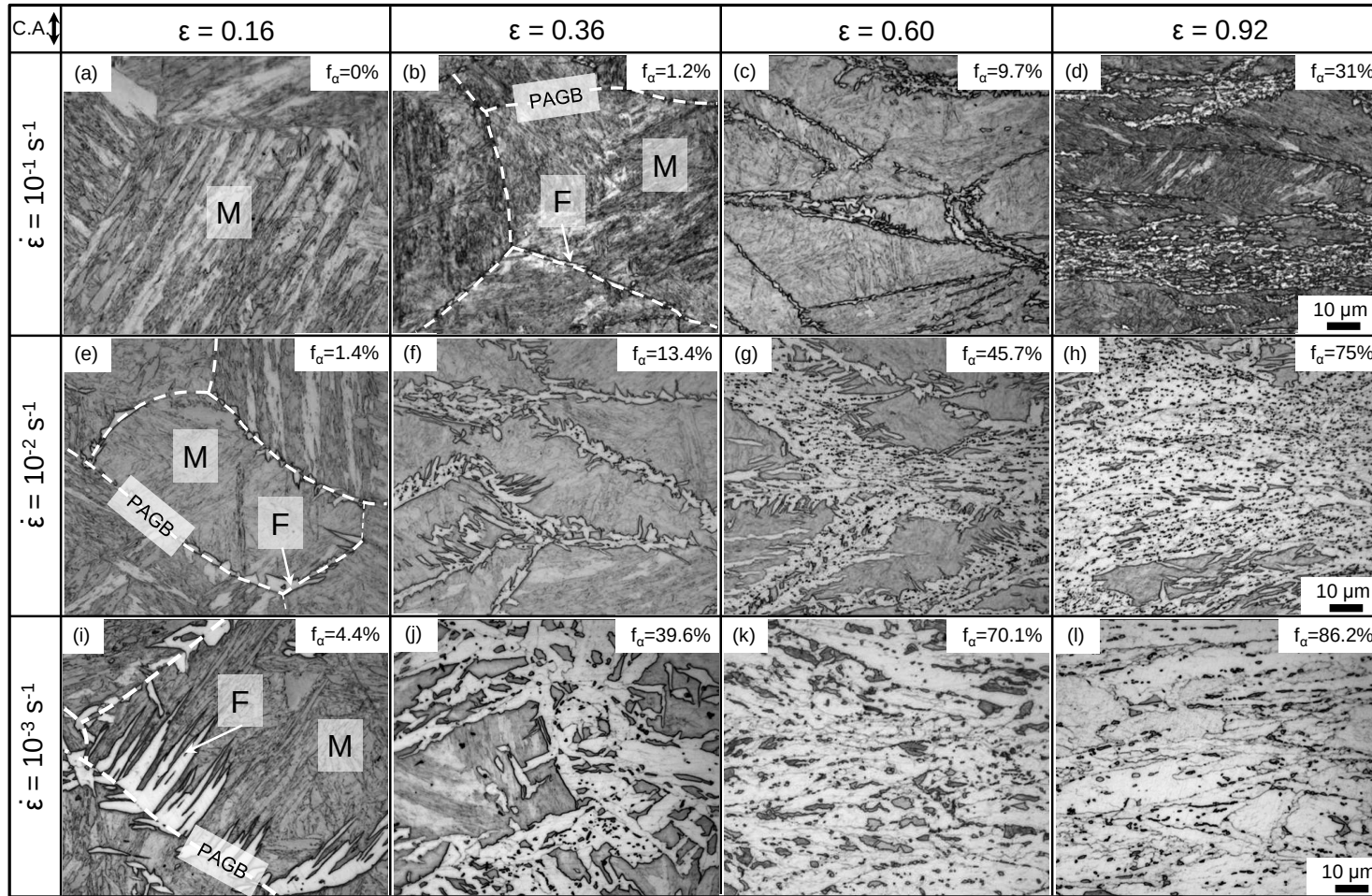


Fig. 2.12 OM images of the specimens with an austenite grain size of $125 \mu\text{m}$ deformed to different strains from 0.16 to 0.92 at strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at $520 \text{ }^{\circ}\text{C}$. “F” and “M” indicate ferrite and martensite, respectively. The volume fractions of ferrite are shown on the top right corners of the micrographs, respectively. White broken lines indicate prior austenite grain boundaries (PAGBs). Compression axis is along the vertical direction of the images.

$d\gamma = 125 \mu\text{m}$, $\dot{\epsilon} = 10^{-2} \text{s}^{-1}$

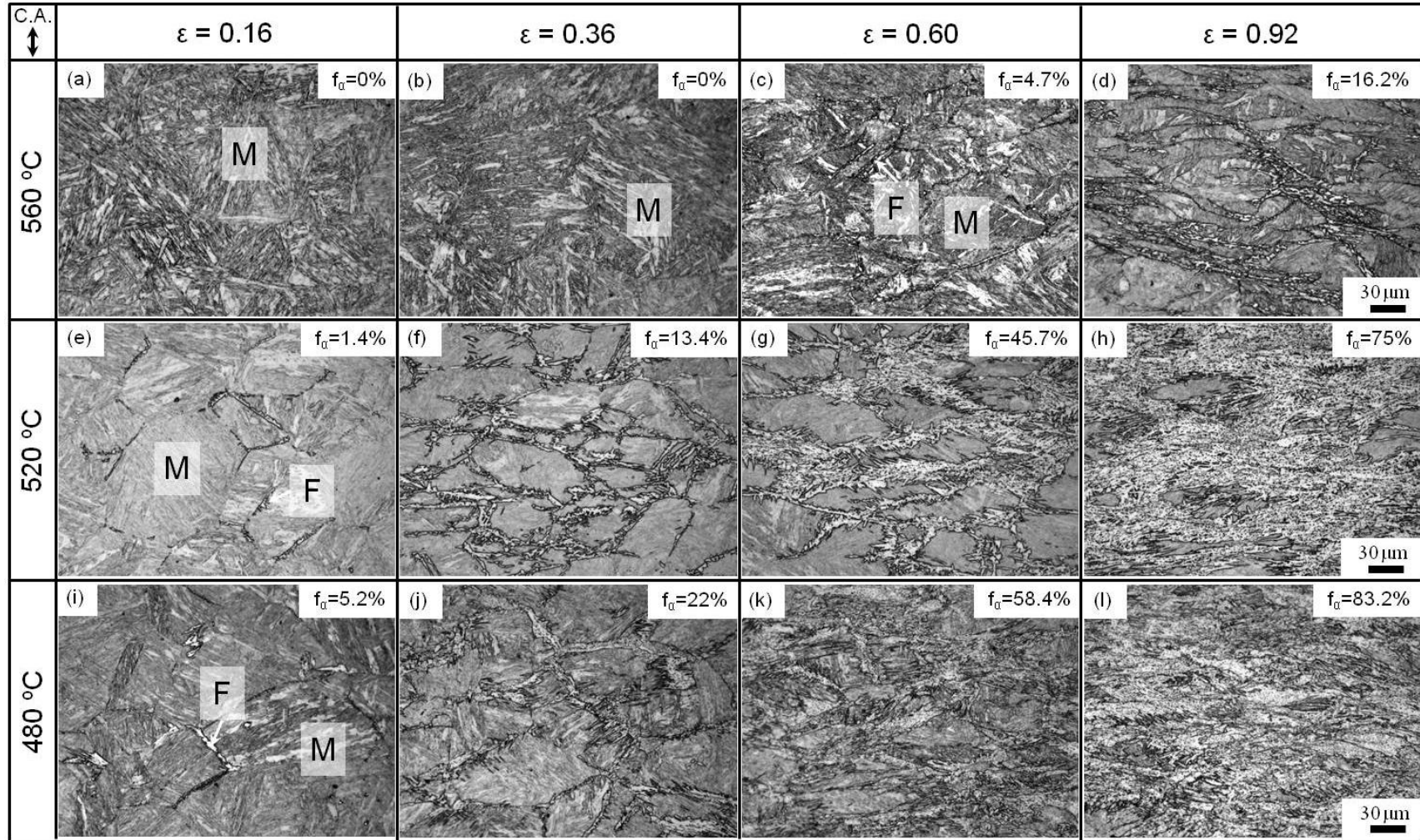


Fig. 2.13 OM images of the specimens with an austenite grain size of $125 \mu\text{m}$ deformed to different strains from 0.16 to 0.92 at a strain rate of 10^{-2}s^{-1} at different temperatures from 560 °C to 480 °C. “F” and “M” indicate ferrite and martensite, respectively. The volume fractions of ferrite are shown on the top right corners of the micrographs, respectively. Compression axis is along the vertical direction of the images.

$T = 520\text{ }^{\circ}\text{C}$, $\dot{\epsilon} = 10^{-2}\text{ s}^{-1}$

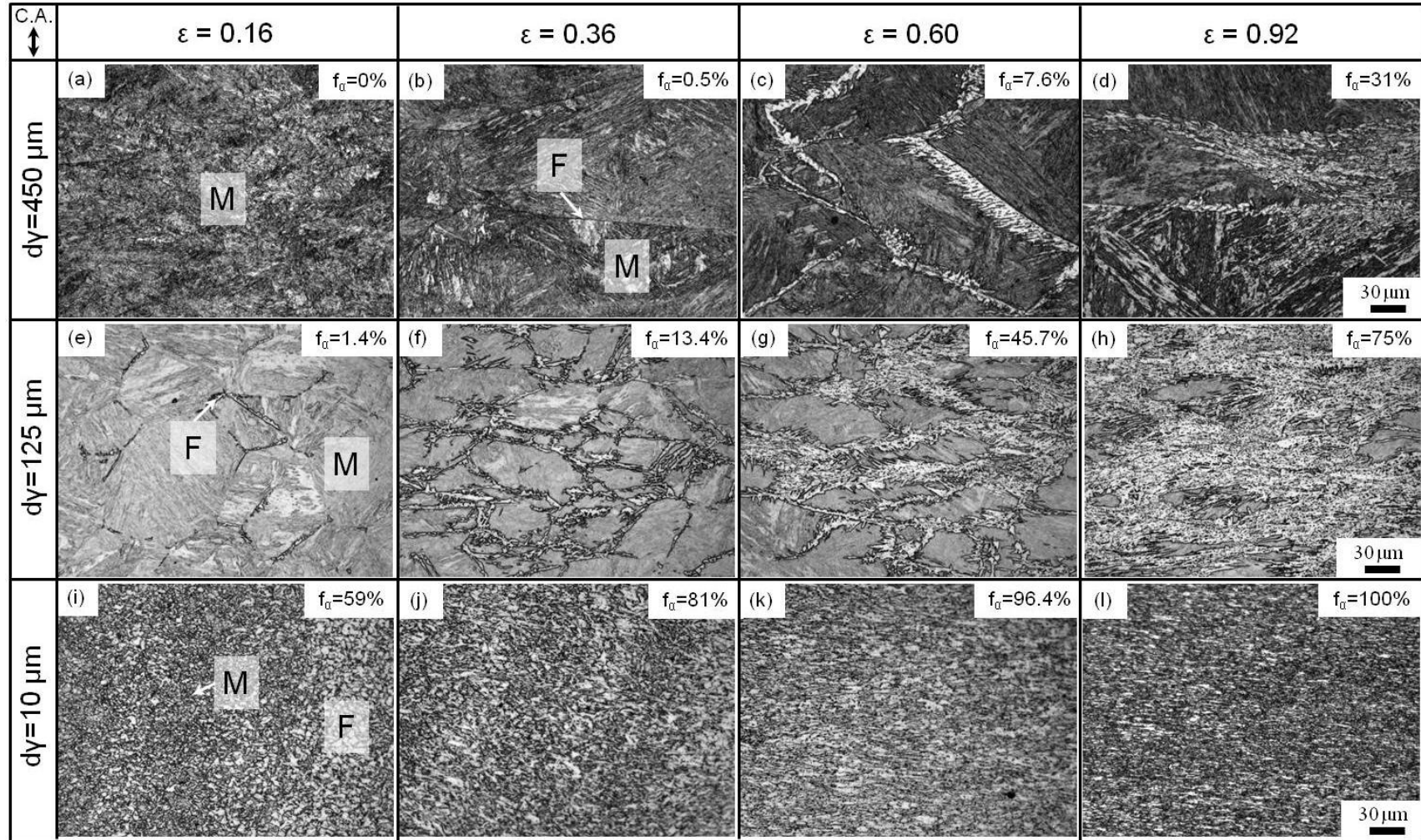


Fig. 2.14 OM images of the specimens with different austenite grain sizes from 450 μm to 10 μm deformed to different strains from 0.16 to 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. “F” and “M” indicate ferrite and martensite, respectively. The volume fractions of ferrite are shown on the top right corners of the micrographs, respectively. Compression axis is along the vertical direction of the images.

Figure 2.15 summarizes changes in the volume fraction of dynamically transformed ferrite in the specimens deformed to various strains (a) at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C , (b) at a strain rate of 10^{-2} s^{-1} at different temperatures from 600°C to 480°C , and (c) at a strain rate of 10^{-2} s^{-1} at 520°C with different austenite grain sizes from $10 \mu\text{m}$ to $450 \mu\text{m}$. The austenite grain size of the specimens in (a) and (b) was $125 \mu\text{m}$. The volume fraction of dynamically transformed ferrite increases with an increase of strain under all the deformation conditions. The figures substantially show the kinetics of DT as a function of strain. In fact, under a constant strain rate, the strain can be converted to the time at that temperature. In **Fig. 2.15(b)** and **(c)**, the time held at that temperature (including the holding time till the onset of deformation, 60 s) is indicated on the upper horizontal axis of the figures. When the strain rate is changed, it takes different periods of time for an identical strain and the situation is a little bit complicated. Such cases will be discussed later. As a function of strain, the lower the strain rate (**Fig. 2.15(a)**), the lower the deformation temperature (**Fig. 2.15(b)**) and the finer the austenite grain size (**Fig. 2.15(c)**), the volume fraction of dynamically transformed ferrite becomes higher. The critical strains for the onset of DT obtained from the double differentiation method shown in **Fig. 2.11** are marked by blue arrows in **Fig. 2.15**. The fractions of ferrite at the critical strains fall into a range of 2-10%, which indicates that fairly successful estimation of the onset of DT could be done by the double differentiation method described in the last section.

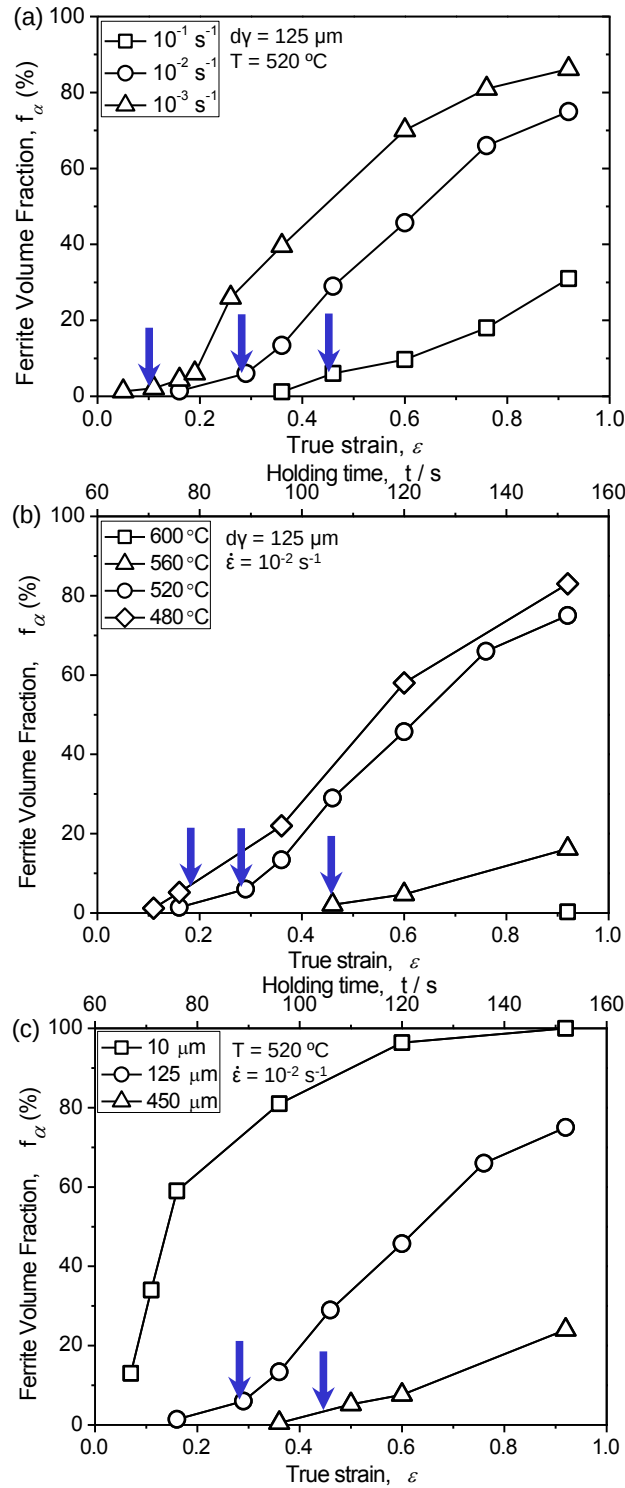


Fig. 2.15 Changes in volume fraction of ferrite in the specimens deformed to various strains (a) at strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C , (b) at a strain rate of 10^{-2} s^{-1} at temperatures from 600°C to 480°C , and (c) at a strain rate of 10^{-2} s^{-1} at 520°C with austenite grain sizes from $10 \mu\text{m}$ to $450 \mu\text{m}$. The austenite grain size in (a) and (b) is $125 \mu\text{m}$. The holding time on the upper horizontal axis of (b) and (c) includes the holding time till the onset of deformation (60 s) and the time converted from strain at a constant strain rate (10^{-2} s^{-1}). Blue arrows mark critical strains for the onset of DT obtained in **Fig. 2.11**.

It was confirmed that the volume fraction of ferrite increases with decreasing the strain rate (**Fig. 2.15(a)**). When the strain rate is changed, however, a lower strain rate corresponds to longer time at that temperature under a certain strain. It is, therefore, not appropriate to conclude that the kinetics of DT (formation rate of ferrite as a function of time) is accelerated by lowering the strain rate. **Figure 2.16** shows changes in the volume fraction of dynamically transformed ferrite in the specimens deformed to various strains at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C , plotted as a function of holding time. Here, the holding time means the total time at 520°C including both static holding period (60 s) till the onset of deformation and deformation period (converted from the applied strain and strain rate), which is also the case in the rest of this thesis. The solid diamond symbols represent the volume fraction of statically transformed ferrite in the specimens statically held at 520°C for different periods of time without deformation. The transformation kinetics of DT is much faster than that of ST. The incubation period till the onset of DT is shortened at higher strain rate and the kinetics of transformation is faster at higher strain rate according to the slope of the curve. But the final volume fraction of ferrite at the same strain of 0.92 increases with decreasing the strain rate, which is quite different from some of the results previously reported [23]. On the other hand, the results shown in **Fig. 2.15(b)** and **Fig. 2.16** coincide with the result reported by Park et al. [31] that DT occurs above a critical

Zener-Hollomon parameter (i.e., DT tends to occur at lower temperature and higher strain rate) very well.

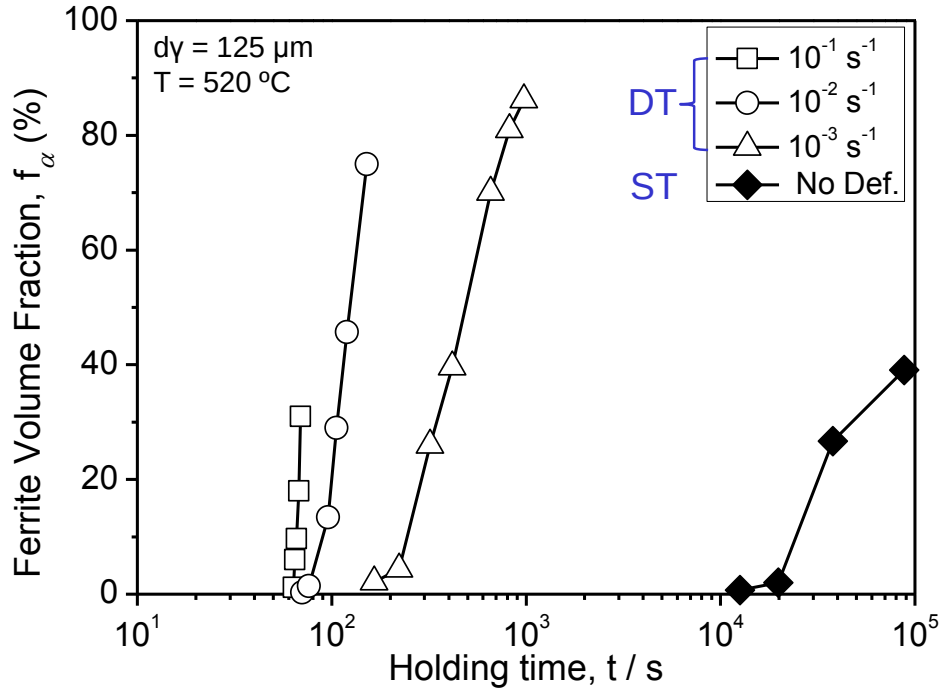


Fig. 2.16 Change in the volume fraction of dynamically transformed ferrite in the specimens deformed to various strains up to 0.92 at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 °C, plotted as a function of holding time at 520 °C. The holding time includes both the static holding time till the onset of deformation (60 s) and the deformation period calculated from the applied strain and strain rate. The solid diamond symbols represent the volume fraction of statically transformed ferrite in the specimens statically held at 520 °C for different periods of time.

The effect of strain rate on the formation of ferrite by DT is further examined under other conditions, i.e., at different deformation temperatures (**Fig. 2.17**) or with different austenite grain sizes (**Fig. 2.18**). **Figure 2.17** shows OM images of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at different strain rates from 10^0 s^{-1} to 10^{-3}

s^{-1} at different temperatures from 600 °C to 480 °C. At 600 °C, no ferrite was observed at the strain rate of 10^0 s^{-1} or 10^{-1} s^{-1} . Ferrite started to form along prior austenite grain boundaries with decreasing the strain rate to 10^{-2} s^{-1} (**Fig. 2.17(c)**) and its volume fraction increased at a lower strain rate of 10^{-3} s^{-1} (**Fig. 2.17(d)**). At other temperatures, i.e., 560 °C, 520 °C and 480 °C, the changes in the volume fraction of ferrite exhibit the same tendency, that is, the volume fraction of ferrite increases with decreasing the strain rate.

Figure 2.18 shows OM images of the specimens with different austenite grain sizes from 450 μm to 10 μm deformed to a strain of 0.92 at different strain rates from 10^0 s^{-1} to 10^{-3} s^{-1} at 520 °C. In the case of a coarse austenite grain size (450 μm , **Fig. 2.18(a)-(d)**), the volume fraction of ferrite increases with decreasing the strain rate. It shows the same tendency in the smaller austenite grain sizes (**Fig. 2.18(e)-(h)** and **Fig. 2.18(i)-(l)**). Therefore, it could be concluded that with different austenite grain sizes, the volume fraction of ferrite increases with decreasing the strain rate.

$d\gamma = 125 \mu\text{m}$, $\epsilon = 0.92$

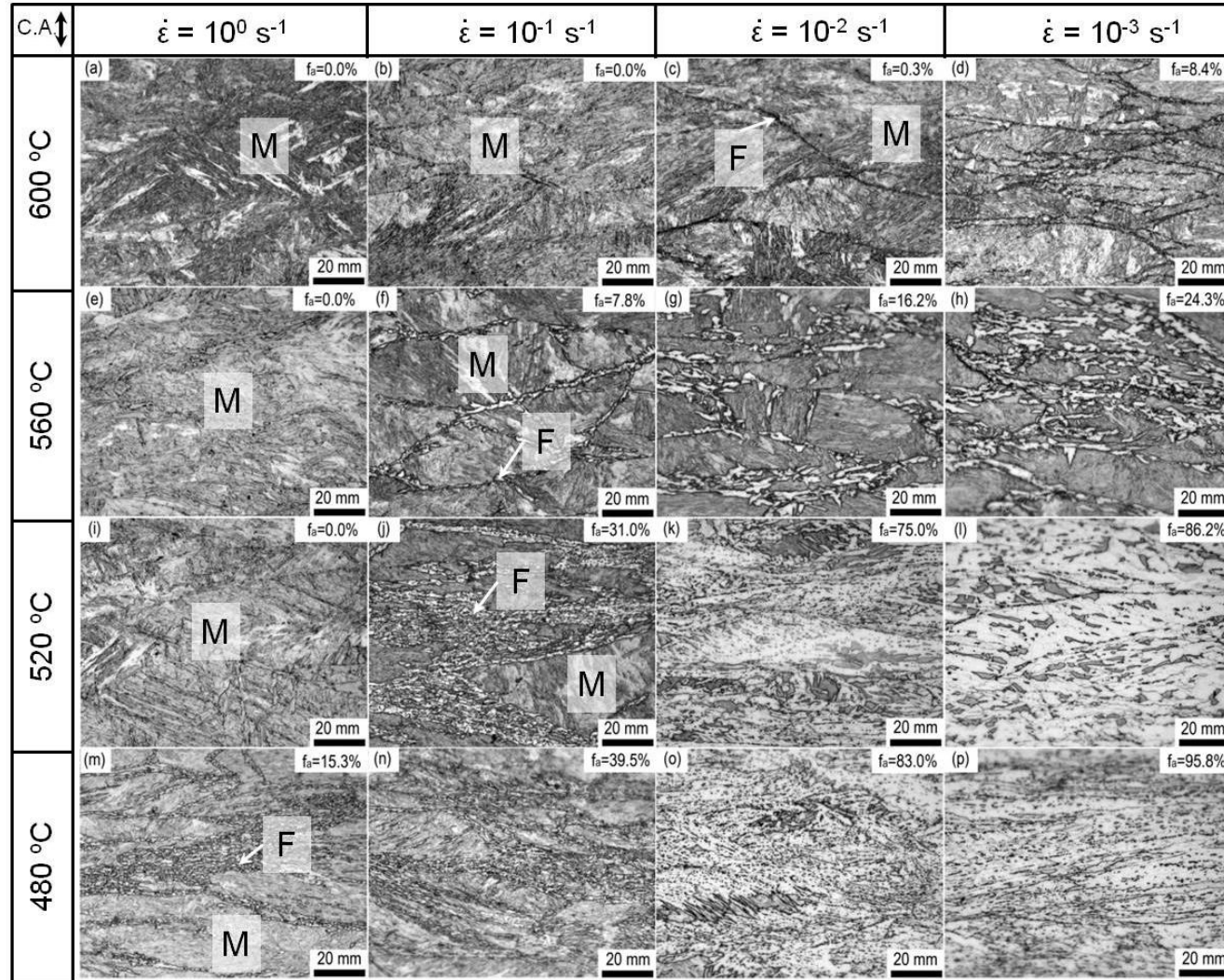


Fig. 2.17 OM images of the specimens with an austenite grain size of $125 \mu\text{m}$ deformed to a strain of 0.92 at different strain rates from 10^0 s^{-1} to 10^{-3} s^{-1} at different temperatures from $600 \text{ }^\circ\text{C}$ to $480 \text{ }^\circ\text{C}$. “F” and “M” represent ferrite and martensite, respectively. Compression axis is along the vertical direction of the images.

$T = 520\text{ }^{\circ}\text{C}$, $\varepsilon = 0.92$

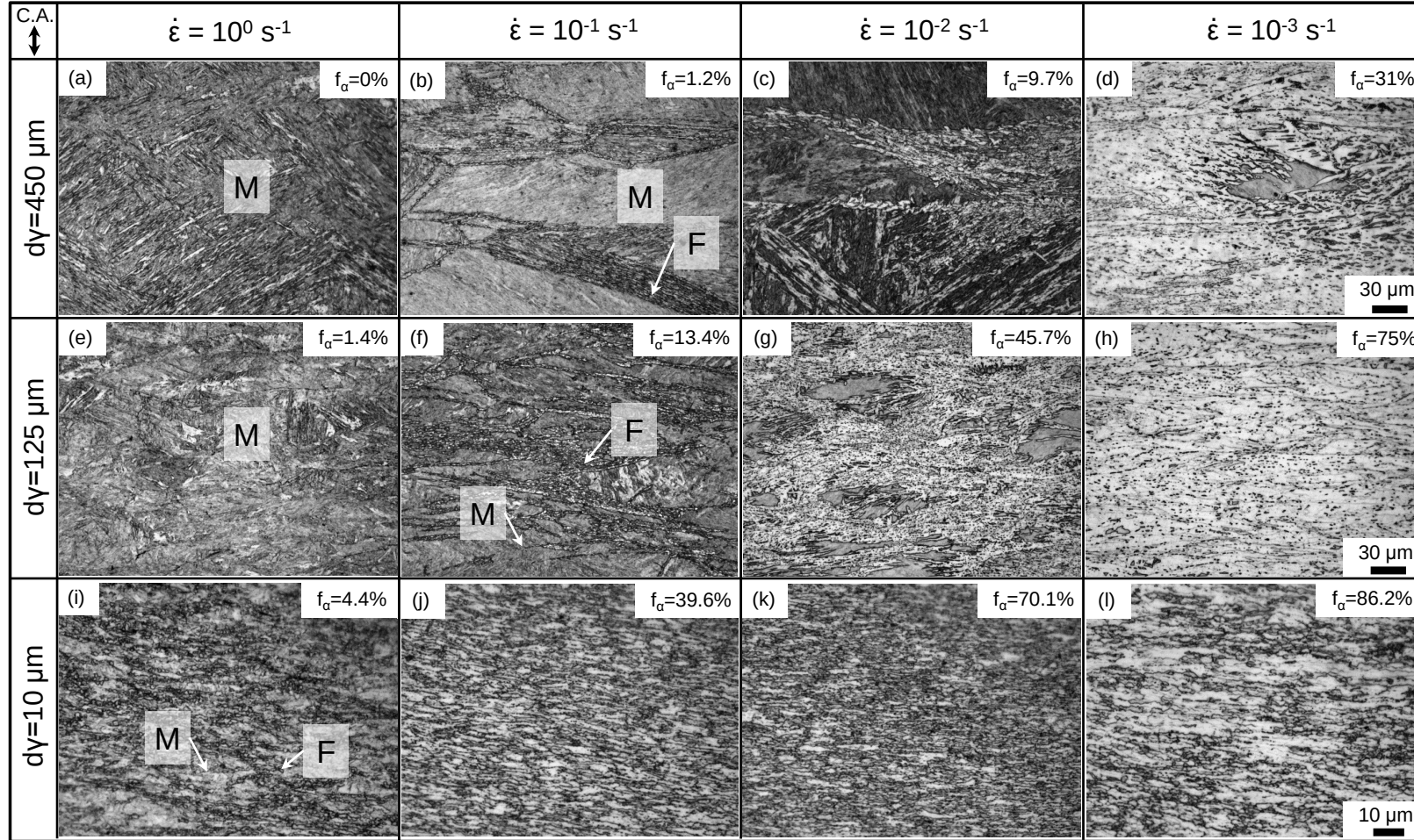


Fig. 2.18 OM images of the specimens with different austenite grain sizes from 450 μm to 10 μm deformed to a strain of 0.92 at strain rates from 10^0 s^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$. “F” and “M” represent ferrite and martensite, respectively. Compression axis is along the vertical direction of the images.

Figure 2.19 summarizes the changes in the volume fraction of DT ferrite as a function of the strain rate in the specimens deformed to a strain of 0.92 (a) at different temperatures and (b) with different austenite grain sizes. Both figures show the same tendency that, the lower strain rate leads to the higher fraction of ferrite.

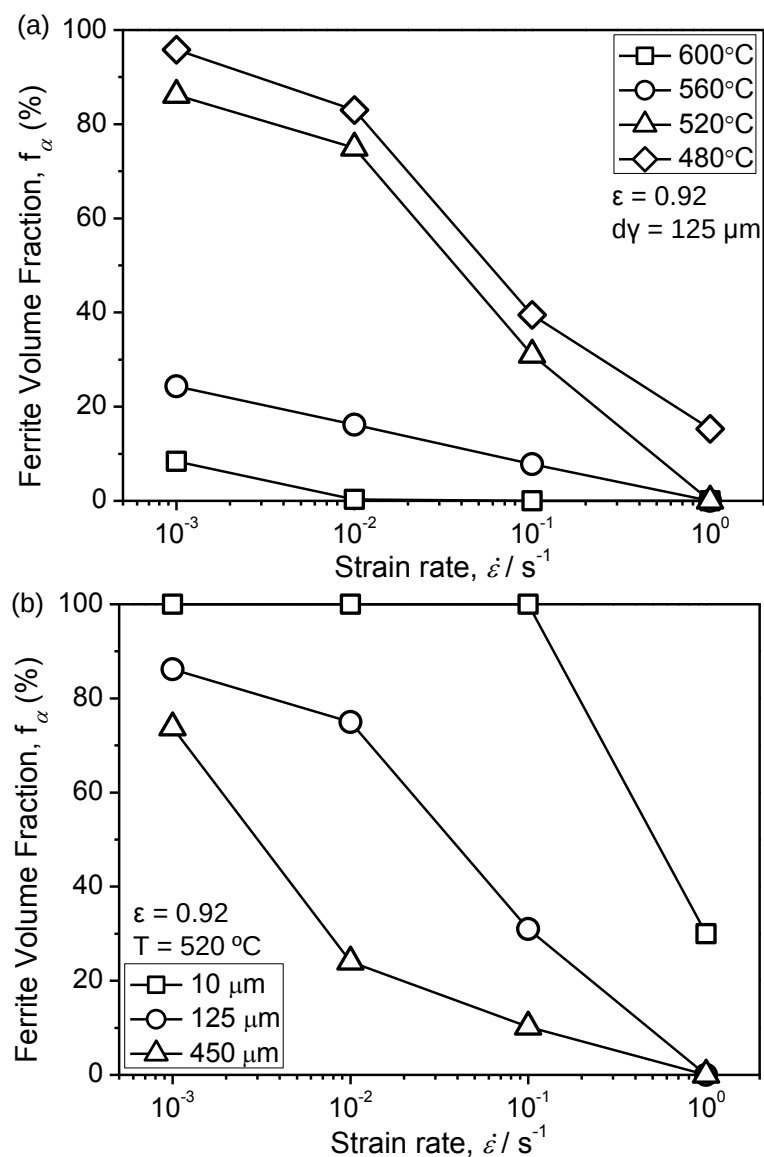
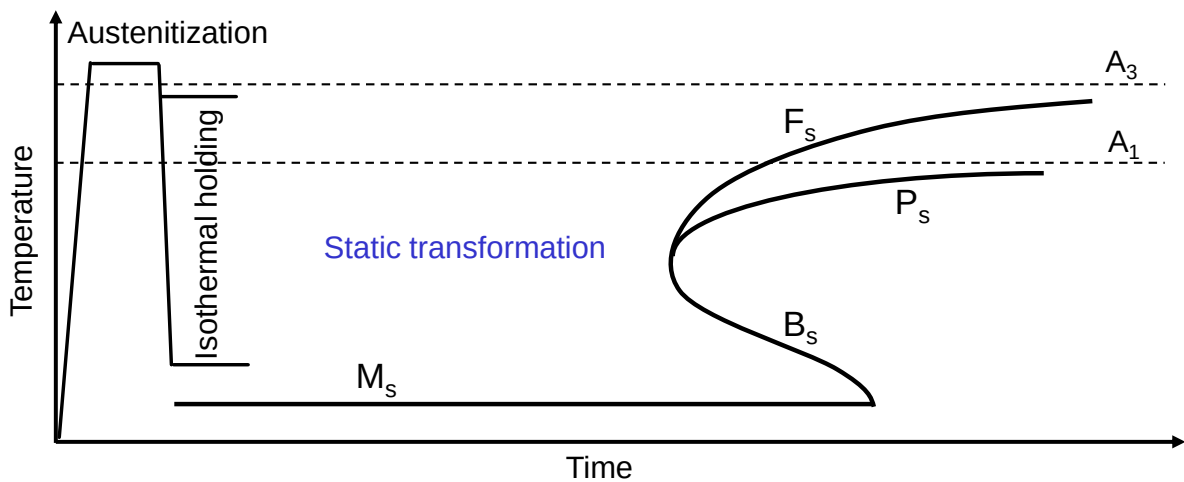


Fig. 2.19 Changes in the volume fraction of ferrite as a function of strain rate in the specimens deformed to a strain of 0.92 (a) at different temperatures from 600 °C to 480 °C and (b) with different austenite grain sizes. The austenite grain size in (a) was 125 μm , and the deformation temperature in (b) was 520 °C.

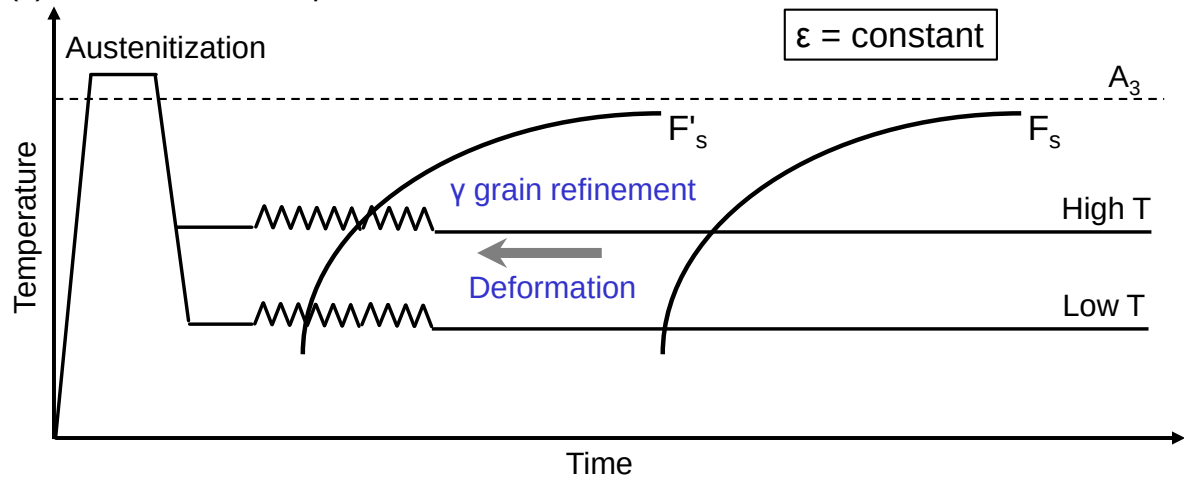
The effects of thermomechanical parameters (i.e., strain, strain rate, deformation temperature and austenite grain size) on the kinetics of ferrite transformation are discussed below. Generally speaking, when austenite is isothermally held at a specific temperature between A_3 and A_1 , static ferrite transformation (ST) from austenite eventually occurs (**Fig. 2.20(a)**). Temperature has a profound influence on the kinetics of ferrite transformation. At low temperatures, chemical driving force for ST is high, but diffusivity of atoms is low. High driving force leads to high nucleation density for ferrite, but Low diffusivity results in low growth rate of ferrite. Consequently, the kinetics of ferrite transformation (involving both nucleation and growth) exhibits a typical C-curve in TTT diagram. At a specific temperature, deformation of austenite can introduce large amount of defects and deformation structures (like microbands, etc.), which can increase the nucleation density for ferrite and thus accelerate the kinetics of transformation (**Fig. 2.20(b)**). With decreasing deformation temperature (above nose temperature in the kinetic C-curve), both driving force and nucleation density (introduced by supercooling and deformation) increase, which will decrease the required strain for the onset of DT and accelerate the kinetics (**Fig. 2.15(b)**). Austenite grain size can also affect the kinetics of ferrite transformation (**Fig. 2.20(b)**). Austenite grain boundary is the most preferential nucleation site for ferrite. Fine austenite grains provide more grain boundary areas in a given volume of material [36] and thus increase the nucleation density for ferrite transformation. Therefore, grain refinement of

austenite can decrease the required strain for the onset of DT and accelerate its kinetics (**Fig. 2.15(c)**). The effect of strain rate on the kinetics of DT is illustrated in **Fig 2.20(c)**. For a given thermomechanical condition (i.e. with the same austenite grain size, to the same strain and at the same deformation temperature), a high strain rate introduces more defects into austenite, which will increase the nucleation density for ferrite transformation. This will shorten the incubation time for the occurrence of DT (**Fig. 2.16**). However, increasing strain rate will decrease the deformation time (under a constant strain), allowing less time for ferrite to nucleate and grow during the deformation. Therefore, smaller volume fraction of ferrite could be observed at high strain rates under a constant strain (**Fig. 2.16** and **Fig. 2.19**). On the contrary, low strain rates may not generate as many nucleation sites as that at high strain rates, but the nucleated ferrite has longer time to grow, which leads to a smaller critical strain for the onset of DT and a higher ferrite fraction (**Fig. 2.15(a)**). Although the effect of strain rate on the kinetics of DT has been a subject of debate, it is confirmed in the present study systematically done under various conditions that the volume fraction of ferrite increases with decreasing the strain rate.

(a) General view of TTT in steels



(b) Effect of ϵ , T and $d\gamma$ on DT



(c) Effect of $\dot{\epsilon}$ on DT

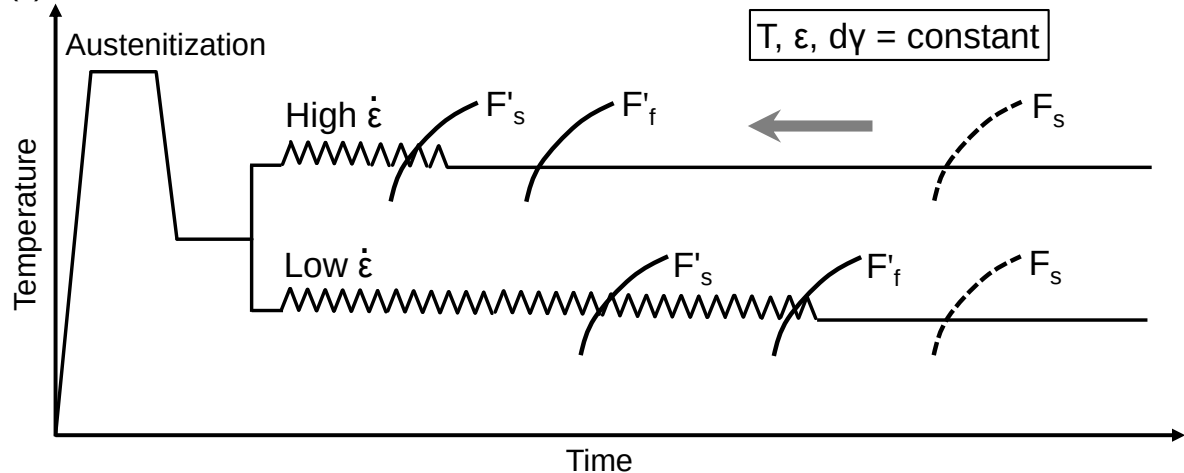


Fig. 2.20 Schematic illustration showing the effect of strain, strain rate, deformation temperature and austenite grain size on kinetics of ferrite transformation. F_s , P_s , B_s and M_s indicate static initiation of ferrite, pearlite, bainite and martensite transformation, respectively. F'_s and F'_f indicate start and finish of DT. Applied strain (ϵ) is constant in (b). Temperature (T), strain (ϵ) and austenite grain size ($d\gamma$) are constant in (c).

2.3.4 Microstructural features of dynamically transformed ferrite

The OM images of ferrite in the last section provided a whole view of microstructure evolution in dynamic ferrite transformation under various thermomechanical conditions in a relatively large scale. In this section, the effects of strain rate, deformation temperature and austenite grain size on the ferrite microstructure evolution are furthermore examined by SEM and EBSD measurements in detail.

The effect of strain rate on the ferrite microstructure evolution is revealed in **Fig. 2.21**, which shows SEM images of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$. “F” and “M” represent ferrite and martensite, respectively, which is the case for all SEM images in this chapter hereafter. White broken lines in **Fig. 2.21(a)** and **(b)** mark prior austenite grain boundaries (PAGBs). The dynamically transformed ferrite shows different morphologies depending on the strain rate. At a higher strain rate of 10^{-1} s^{-1} (**Fig. 2.21(a)**), the ferrite shows fine grains formed along the PAGBs and there are also relatively coarse grains growing into martensite (i.e., austenite at 520 $^{\circ}\text{C}$). But the grain size difference between two areas (i.e., near PAGBs and at interiors of austenite grains) is relatively small. At a lower strain rate of 10^{-2} s^{-1} (**Fig. 2.21(b)**), dynamically transformed ferrite has two typical morphologies. Fine and nearly equiaxed grains are mainly formed along the PAGBs, while the ferrite growing into martensite (prior austenite) mainly shows coarse morphologies

elongated to the direction perpendicular to the compression axis (C.A.). In the vicinity of the phase boundaries between the coarse ferrite and martensite (prior austenite), there are no new ferrite grains formed, indicating that grain growth is more dominant than nucleation in this area. With decreasing the strain rate to 10^{-3} s^{-1} (**Fig. 2.21(c)**), fine-grain areas become hard to find, and most of the ferrite exhibit coarse and elongated morphologies.

For obtaining more detailed information of the microstructures shown in **Fig. 2.21**, EBSD analysis of the specimens was carried out. **Figure 2.22** shows EBSD grain boundary maps of the specimens with an austenite grain size of $125 \text{ }\mu\text{m}$ deformed to a strain of 0.92 at a strain rate of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at $520 \text{ }^{\circ}\text{C}$. Low-angle boundaries (LABs) with misorientation of $2\text{--}15^{\circ}$ and high-angle boundaries (HABs) with misorientation above 15° are drawn in red and blue lines, respectively. In order to clearly present the microstructure of dynamically transformed ferrite, non-ferrite phases (i.e. martensite and/or retained austenite in the present study) are painted in black in the maps, which is the case for all EBSD maps shown in this chapter hereafter. The method for distinguishing ferrite from martensite and austenite was previously mentioned in **Fig. 2.4**. At a strain rate of 10^{-1} s^{-1} (**Fig. 2.22(a)**), the dynamically transformed ferrite consists of elongated ferrite grains and equiaxed ultrafine ferrite (UFF) grains surrounded by HABs (as marked by black arrows). Subgrains surrounded by LABs were observed within the elongated ferrite grains (as marked by green arrows), which indicates that the elongated grains have deformed structures.

Equiaxed UFF grains and elongated grains containing subgrains could also be observed at lower strain rates (**Fig. 2.22(b)** and **(c)**). The difference in grain size between equiaxed UFF and elongated ferrite is relatively small at a strain rate of 10^{-1} s^{-1} (**Fig. 2.22(a)**). With decreasing the strain rate, the elongated grains become coarser and contain more subgrains (**Fig. 2.22(b)** and **(c)**), indicating preferential grain growth under lower strain rate.

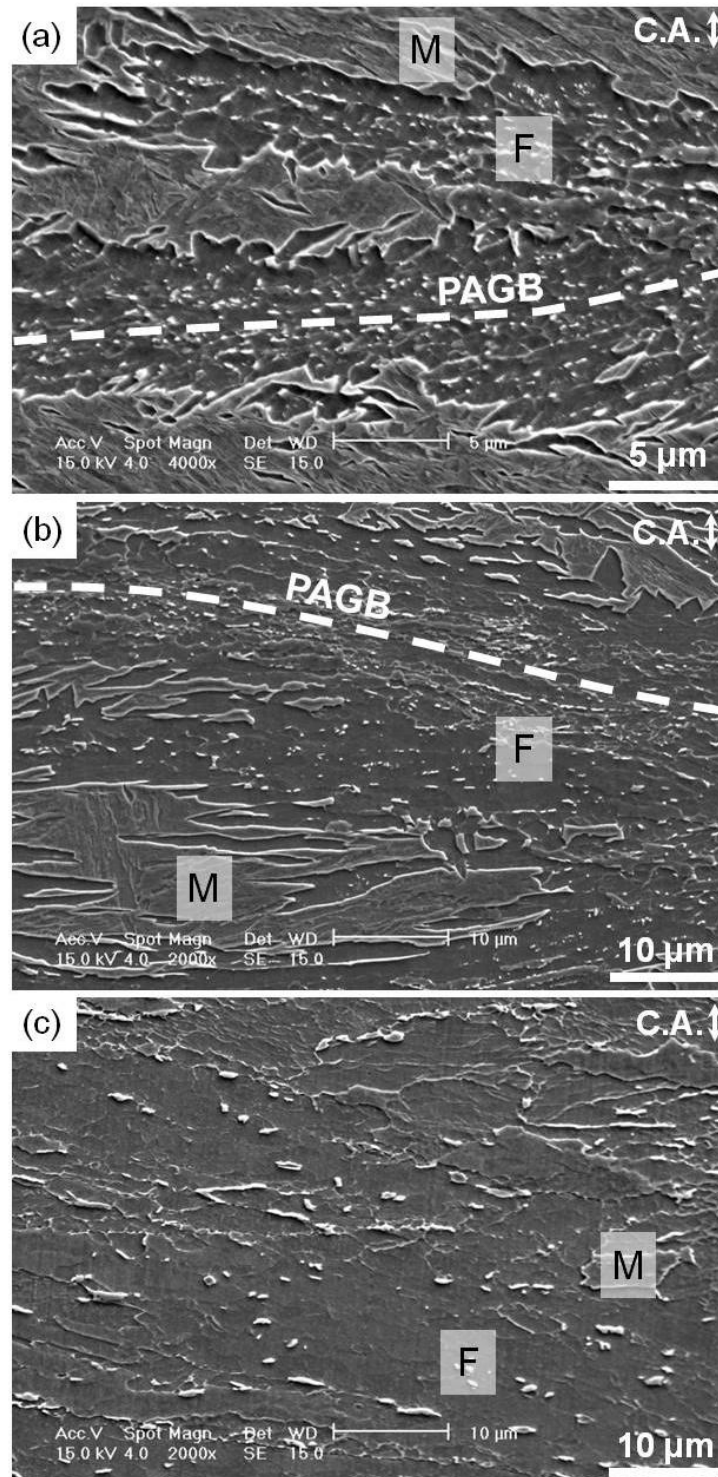


Fig. 2.21 SEM images of the specimens deformed to a strain of 0.92 at a strain rate of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at 520 °C. Prior austenite grain size was 125 μm. White broken lines represent prior austenite grain boundaries (PAGBs). “F” and “M” indicate ferrite and martensite, respectively. Compression axis is along the vertical direction of the images.

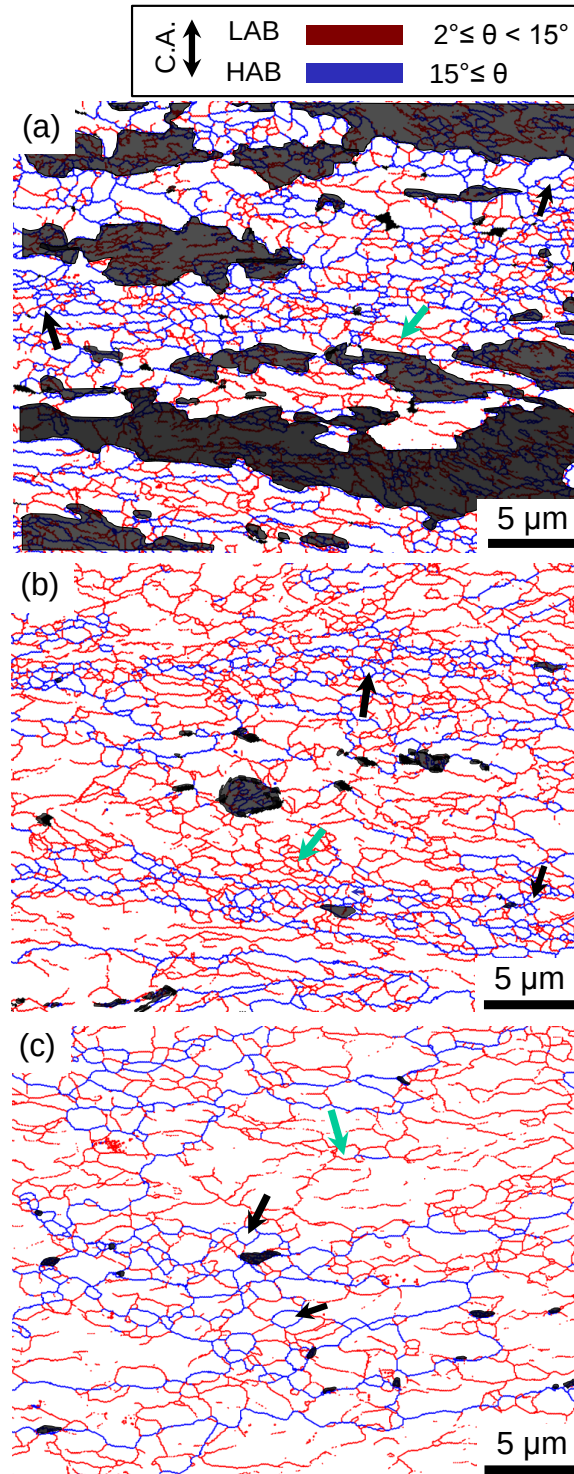


Fig. 2.22 EBSD grain boundary maps of the specimens with austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of (a) 10⁻¹ s⁻¹, (b) 10⁻² s⁻¹ and (c) 10⁻³ s⁻¹ at 520 °C. Non-ferrite phases (i.e., martensite and/or retained austenite) are painted in black in the maps. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. Black and green arrows mark equiaxed UFF grains and subgrains, respectively. Compression axis is along the vertical direction of the maps.

The effect of deformation temperature on the ferrite microstructure is revealed in **Fig. 2.23**, which shows SEM microstructures of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (a) 600 $^{\circ}\text{C}$, (b) 560 $^{\circ}\text{C}$, (c) 520 $^{\circ}\text{C}$ and (d) 480 $^{\circ}\text{C}$. White broken lines in **Fig. 2.23(b)** and **(c)** indicate prior austenite grain boundaries (PAGBs). At 600 $^{\circ}\text{C}$, only a small amount (around 0.3%) of fine equiaxed grains are formed along PAGBs. At 560 $^{\circ}\text{C}$, the ferrite formed along PAGBs becomes much coarser and exhibits elongated morphologies. At 520 $^{\circ}\text{C}$, the ferrite is composed of fine equiaxed grains along PAGBs and coarse elongated grains growing into martensite (prior austenite). With decreasing the temperature to 480 $^{\circ}\text{C}$, ferrite seems to exhibit a fine-grained morphology, although it is hard to distinguish the details from the SEM image.

For obtaining more detailed information of the microstructures shown in **Fig. 2.23**, EBSD analysis of the specimens was carried out. **Figure 2.24** shows EBSD grain boundary maps of the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (a) 600 $^{\circ}\text{C}$, (b) 560 $^{\circ}\text{C}$, (c) 520 $^{\circ}\text{C}$ and (d) 480 $^{\circ}\text{C}$. Microstructures of dynamically transformed ferrite differ at different deformation temperatures. At 600 $^{\circ}\text{C}$ (**Fig. 2.14(a)**), a small amount of equiaxed ferrite grains surrounded by HABs are aligned possibly along grain boundaries of prior austenite, indicating that the specimen is at an early stage of transformation. At 560 $^{\circ}\text{C}$ (**Fig. 2.24(b)**),

the ferrite mostly shows coarse and elongated morphologies. Only a few equiaxed UFF grains can be observed in **Fig. 2.24(b)**, as marked by black arrows. Considering the volume fraction of ferrite under the deformation conditions at 560 °C (16.2% from **Fig. 2.13(d)**), the specimen is on the stage where dynamically transformed ferrite is growing after the primary nucleation along PAGBs like that shown in (a). With decreasing the temperature to 520 °C (**Fig. 2.24(c)**), the fraction of the equiaxed UFF grains increases. However, with decreasing the temperature to 480 °C furthermore (**Fig. 2.14(d)**), the fraction of equiaxed UFF grains decreases. Most of the ferrite grains exhibit elongated morphology and contain LABs.

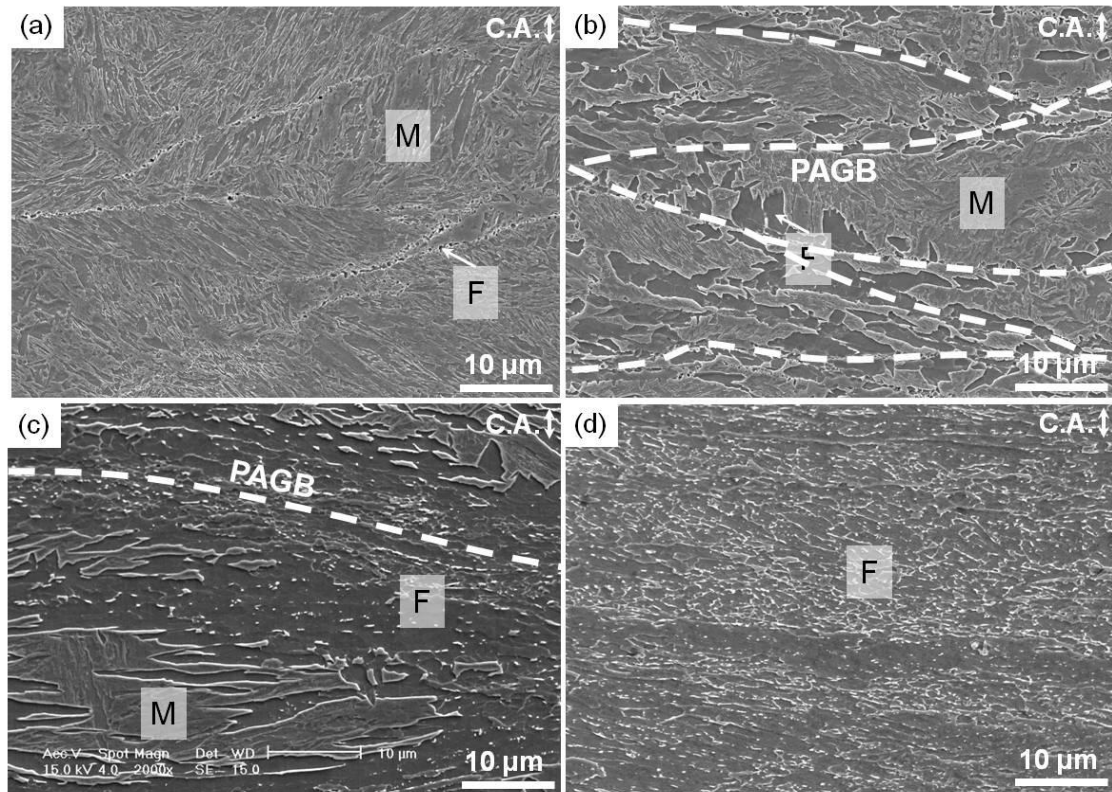


Fig. 2.23 SEM images of the specimens deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (a) 600 °C, 560 °C, (c) 520 °C and (d) 480 °C. Prior austenite grain size was 125 μm. White broken lines represent prior austenite grain

boundaries (PAGBs). “F” and “M” indicate ferrite and martensite, respectively. Compression axis is along the vertical direction of the images.

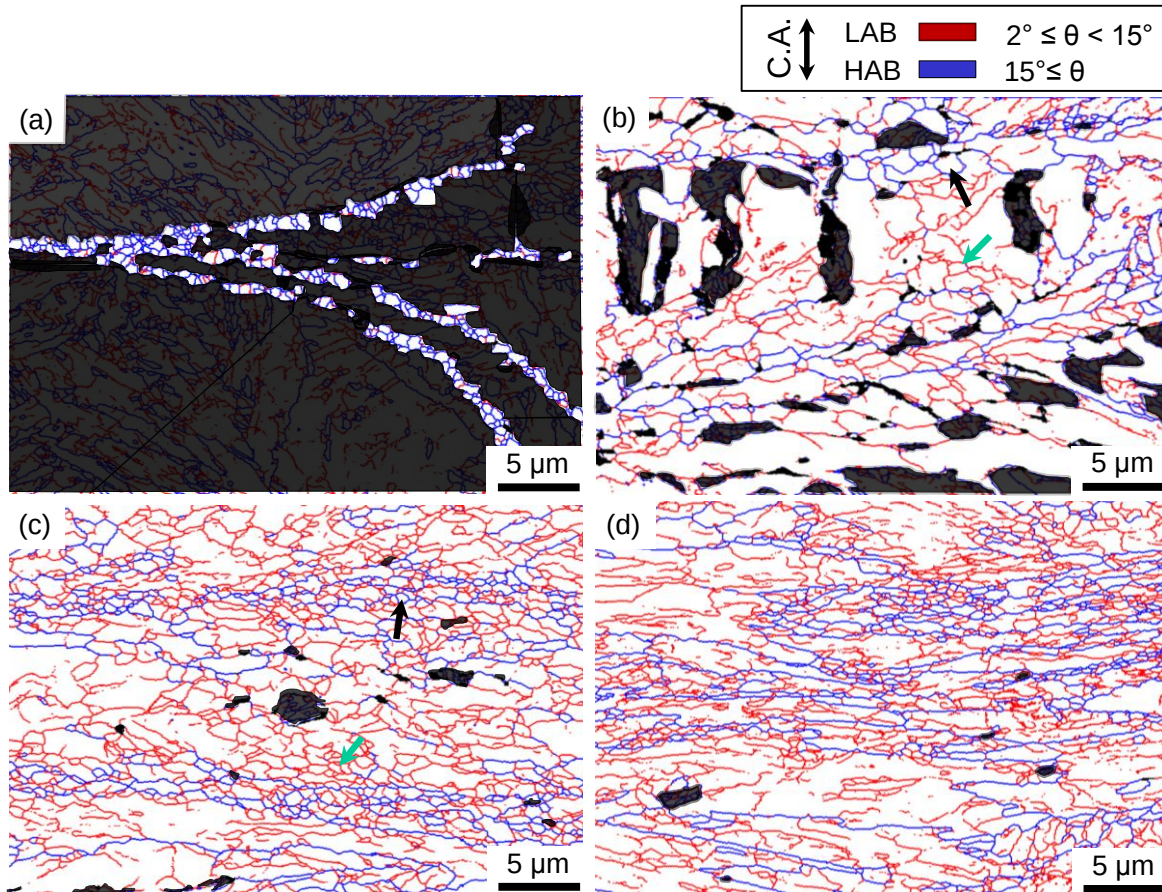


Fig. 2.24 EBSD grain boundary maps of the specimens with austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (a) 600 $^{\circ}\text{C}$, (b) 560 $^{\circ}\text{C}$, (c) 520 $^{\circ}\text{C}$ and (d) 480 $^{\circ}\text{C}$. Non-ferrite phases (i.e. martensite and/or retained austenite) are painted in black in the maps. Low-angle boundaries (LAB) with misorientation of 2-15 $^{\circ}$ and high-angle boundaries (HAB) with misorientation above 15 $^{\circ}$ are drawn in red and blue lines, respectively. Black and green arrows mark equiaxed UFF grains and subgrains, respectively. Compression axis is along the vertical direction of the maps.

The effect of austenite grain size on the ferrite microstructure is revealed in **Fig. 2.25**, which shows SEM microstructures of the specimens with different austenite grain sizes of (a) 450 μm , (b) 125 μm and (c) 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at

520 °C. The ferrite transformed from coarse austenite grains (grain size of 450 µm, **Fig. 2.25(a)**) exhibits coarse and elongated morphology. Note that the volume fraction of ferrite in the whole specimen is around 24% (**Fig. 2.14(d)**), indicating that the specimen is at a relatively early stage of transformation. With decreasing the austenite grain size to 125 µm (**Fig. 2.25(b)**), fine and nearly equiaxed grains are observed along PAGBs. With decreasing the austenite grain size to 10 µm (**Fig. 2.25(c)**), ferrite mostly exhibit a fine-grained morphology.

Detailed information of the microstructures shown in **Fig. 2.25** is revealed in **Fig. 2.26**, which shows EBSD grain boundary maps of the specimens with different austenite grain sizes of (a) 450 µm, (b) 125 µm and (c) 10 µm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 °C. In the case of coarse austenite (grain size of 450 µm, **Fig. 2.26(a)**), the dynamically transformed ferrite exhibit a coarse and elongated morphology and contains a large amount of subgrains surrounded by LABs. Equiaxed UFF grains surrounded by HABs could be observed (as marked by a black arrow), but the fraction is very small. In the case of medium grain-sized austenite (grain size of 125 µm, **Fig. 2.26(b)**), ferrite consist of equiaxed UFF grains and elongated coarse grains containing LABs. The fraction of the equiaxed UFF grains significantly increases in the case of fine-grained austenite (grain size of 10 µm, **Fig. 2.26(c)**). It could be concluded from **Fig. 2.26** that with decreasing the austenite grain size, the volume fraction of equiaxed UFF increases.

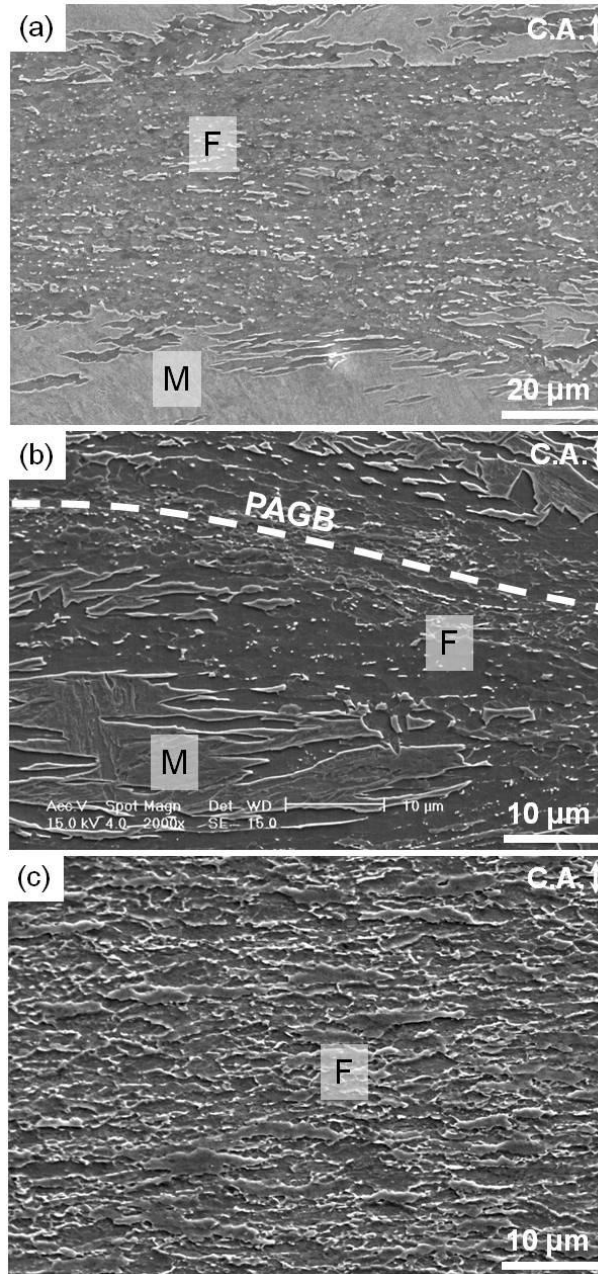


Fig. 2.25 SEM images of the specimens with different austenite grain sizes of (a) 450 μm, (b) 125 μm and (c) 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 °C. White dotted lines represent prior austenite grain boundaries (PAGBs). “F” and “M” indicate ferrite and martensite, respectively. Compression axis is along the vertical direction of the images.

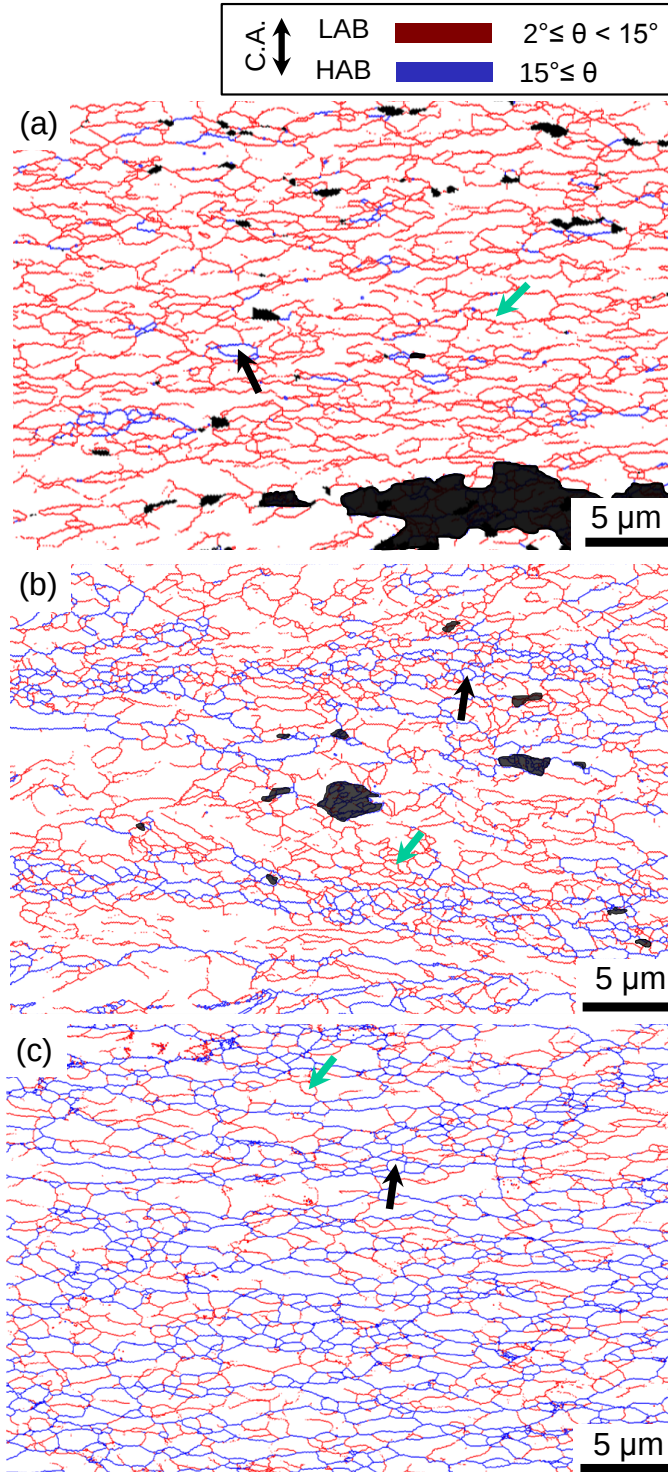


Fig. 2.26 EBSD grain boundary maps of the specimens with austenite grain sizes of (a) 450 μm, (b) 125 μm and (c) 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 °C. Non-ferrite phases (i.e. martensite and/or retained austenite) are painted in black. Low-angle boundaries (LAB) with misorientation of 2-15 ° and high-angle boundaries (HAB) with misorientation above 15 ° are drawn in red and blue lines, respectively. Black and green arrows mark equiaxed UFF grains and subgrains, respectively. Compression axis is along the vertical direction of the maps.

Equiaxed UFF and elongated coarse ferrite containing subgrains were observed in most of the specimens deformed under different conditions (**Fig. 2.21-Fig. 2.26**). **Figure 2.27** summarizes changes in the volume fractions of dynamically transformed ferrite and equiaxed UFF grains in the specimens deformed at different strain rates (a), at different temperatures (b) and with different austenite grain sizes (c). Basically there are three regions in each figure, i.e., the region of martensite and retained austenite (γ), the region of coarse elongated ferrite (α) and the region of equiaxed UFF. In the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at 520 $^{\circ}\text{C}$ (**Fig. 2.27(a)**), with decreasing the strain rate, the volume fraction of dynamically transformed ferrite increases, while the fraction of equiaxed UFF decreases, showing a different strain rate dependency. In the specimens with an austenite grain size of 125 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} (**Fig. 2.27(b)**), low temperatures enhance formation of ferrite. The volume fraction of equiaxed UFF shows a peak value at 520 $^{\circ}\text{C}$. That means neither too high nor too low deformation temperature can enhance the formation of equiaxed UFF. In the specimens deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$ (**Fig. 2.27(c)**), fine austenite grain size enhances formation of both dynamically transformed ferrite and equiaxed UFF grains.

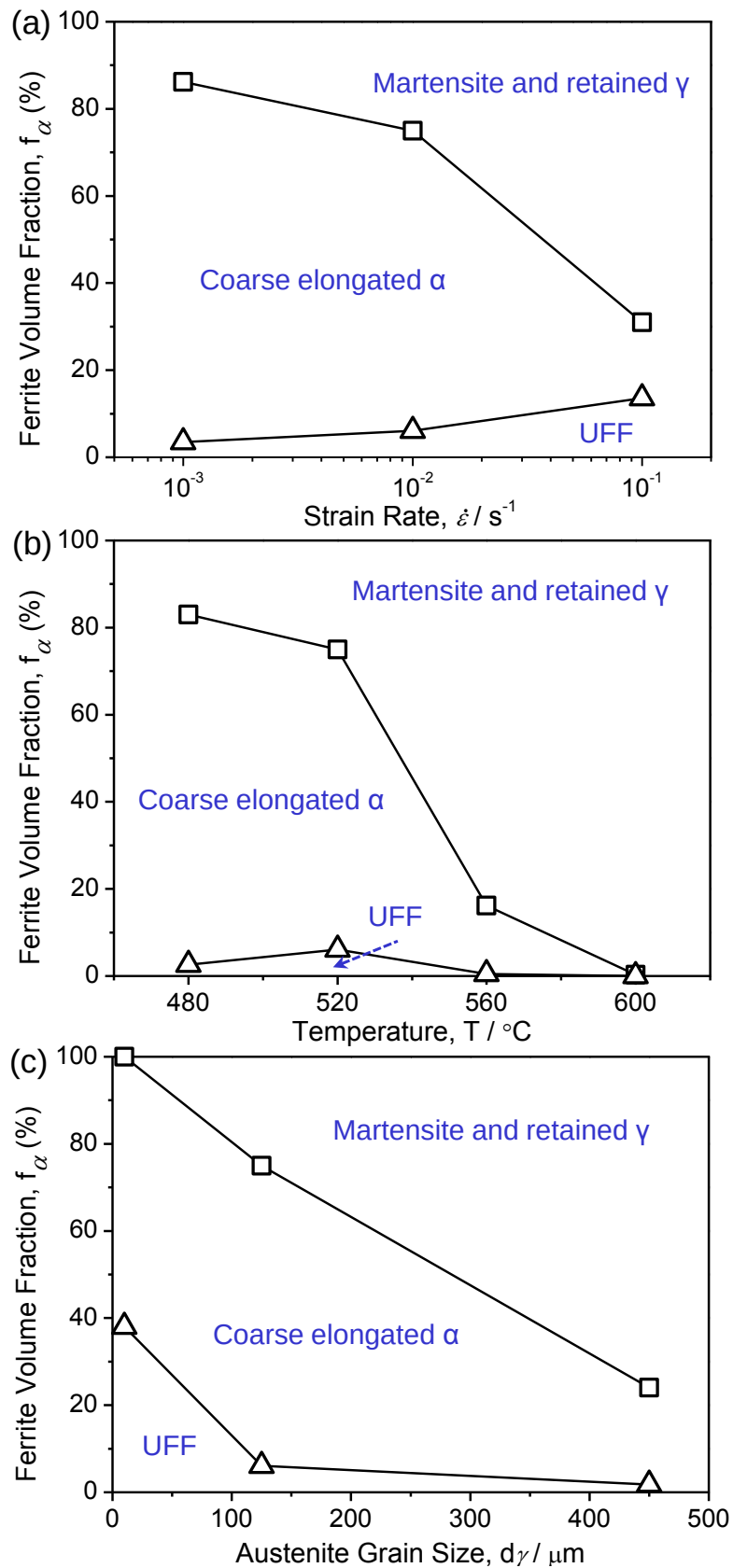


Fig. 2.27 Changes in the volume fractions of ferrite and equiaxed UFF in the specimens deformed at different strain rates (a), at different temperatures (b) and with different austenite grain sizes (c).

To examine at what stages of DT the equiaxed UFF grains and coarse elongated grains are formed, an example showing the microstructural evolution of ferrite under different strains is presented in **Fig. 2.28**, which reveals EBSD grain boundary maps of the specimens with an austenite grain size of 125 μm deformed to different strains of (a) 0.11, (b) 0.36, (c) 0.60 and (d) 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. Green broke lines in (a) and (b) indicate prior austenite grain boundaries (PAGBs). At a strain of 0.11 (**Fig. 2.28(a)**), very fine and nearly equiaxed ferrite grains are formed along a PAGB. With increasing the strain to 0.36 (**Fig. 2.28(b)**), the ferrite grains grow into prior austenite grain and exhibit an irregular morphology. A small amount of LABs are observed within the coarse ferrite grains, indicating a deformed structure. The fraction of LABs increases with increasing the strain to 0.60 (**Fig. 2.28(c)**). At this strain, equiaxed UFF grains surrounded by HABs start to form along the grain boundaries of coarse ferrite (as marked by blue arrows in **Fig. 2.28(c)**). The fraction of the equiaxed UFF increases with increasing the strain to 0.92 (**Fig. 2.28(d)**). It should be noted that the ferrite structure in **Fig. 2.28(d)** seems to be finer than that in **Fig. 2.28(c)**, which indicates another grain refinement process of ferrite during deformation.

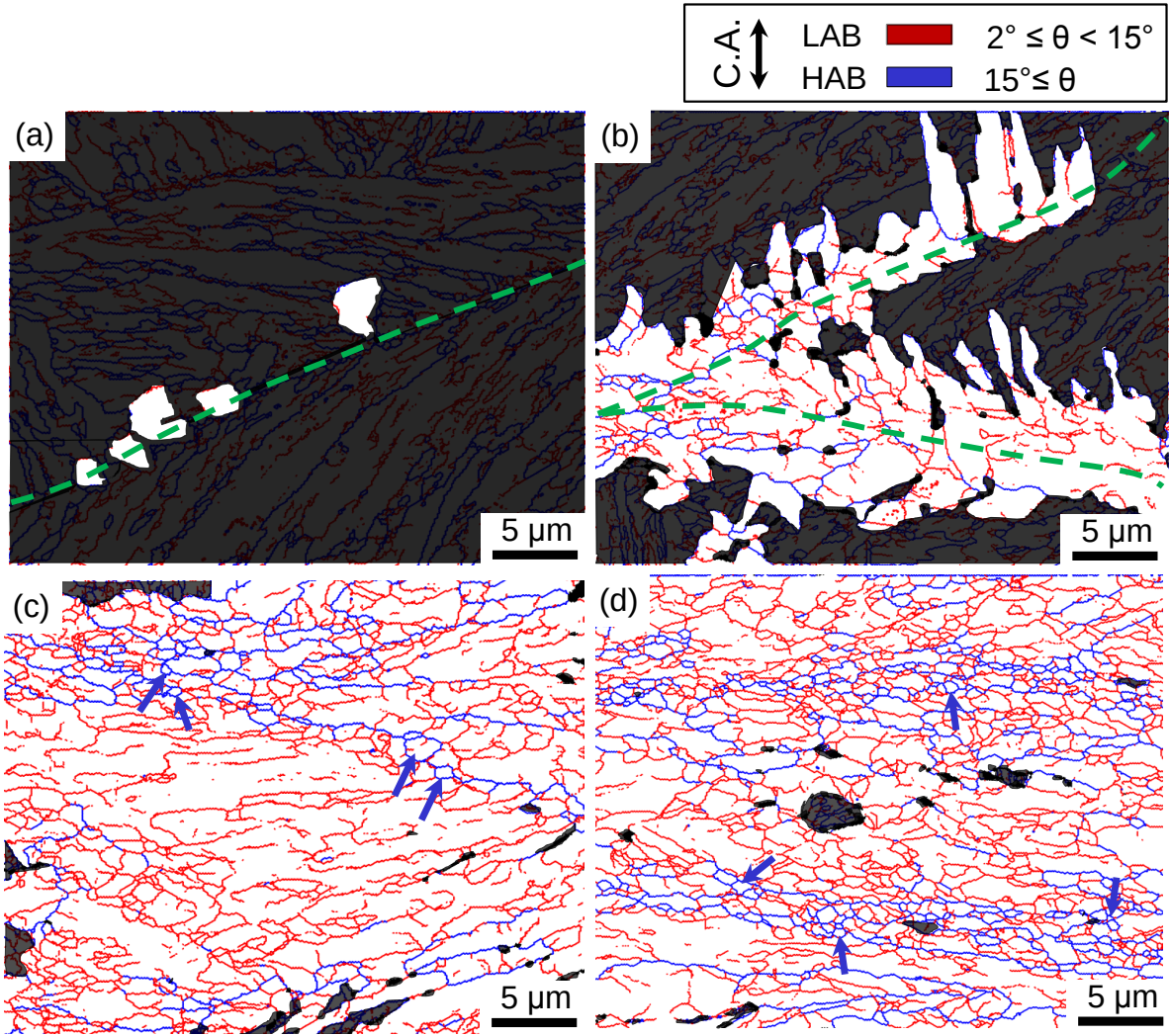


Fig. 2.28 EBSD grain boundary maps of the specimens with an austenite grain size of 125 μm deformed to a strain of (a) 0.11, (b) 0.36, (c) 0.60 and (d) 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. Green broken lines in (a, b) indicate prior austenite grain boundaries (PAGBs). Non-ferrite phases (i.e., martensite and/or retained austenite) were painted in black. Low-angle boundaries with misorientation of $2\text{--}15^{\circ}$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Blue arrows in (c, d) mark equiaxed UFF grains. Compression axis is along the vertical direction of the maps.

The changes in ferrite grain size as a function of strain in the specimens deformed under various conditions are further investigated. **Figure 2.29** shows changes of ferrite grain size: (a)-(b) at different strain rates from 10^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$; (c)-(d) at a strain rate of 10^{-2} s^{-1} at different temperatures from 600 $^{\circ}\text{C}$ to 480 $^{\circ}\text{C}$; (e)-(f) at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$

with different austenite grains. The austenite grain size in (a)-(d) was 125 μm . The grain sizes along the compression axis were measured by linear interception method on the EBSD grain boundary maps counting all boundaries with misorientations larger than 2° (defined as d^{All} in **Fig. 2.29(a), (c) and (e)**) or only counting high-angle grain boundaries with misorientations larger than 15° (defined as d^{HAGB} in **Fig. 2.29(b), (d) and (f)**). For the ferrite obtained at different strain rates (**Fig. 29(a) and (b)**), the grain size firstly increases and then decreases with further straining, exhibiting peak grain size at different strains depending on the strain rate. The peak grain size occurs earlier at a lower strain rate in **Fig. 2.29 (a)**, but in **Fig. 2.29(b)** the peak grain size occurs earlier at a higher strain rate. According to the methods for measuring grain size and the representative microstructure in **Fig. 2.28**, the decrease of grain size (d^{HAGB}) in **Fig. 2.29(b)** is probably due to the formation of equiaxed UFF grains. This indicates that the equiaxed UFF grains are preferentially formed at high strain rates. The changes in ferrite grain size of the specimens deformed at different temperatures (**Fig. 2.29(c) and (d)**) or with different austenite grain sizes (**Fig. 2.29(e) and (f)**) show similar tendencies to that observed in **Fig. 2.29(a) and (b)**. That is, the grain size firstly increases, and then decreases after a certain strain. The grain sizes of ferrite obtained at 560 °C in **Fig. 2.29(c, d)** and in coarse austenite grains (450 μm) in **Fig. 2.29(f)** keep increasing during deformation, which are mainly because the specimens were at early stage of transformation where grain growth is rather dominant (as shown in **Fig. 2.23(b)** for ferrite

obtained at 560 °C, and **Fig. 2.25(a)** for ferrite obtained in coarse austenite (450 µm)). The decrease of ferrite grain size during transformation has never been observed in ST. In ST (**Fig. 2.5**), the grain size of ferrite monotonously increases with the progress of transformation. In DT, however, the grain size decreases during the deformation (**Fig. 2.29(b)**), which is probably due to the formation of equiaxed UFF grains (**Fig. 2.28(c)** and **(d)**).

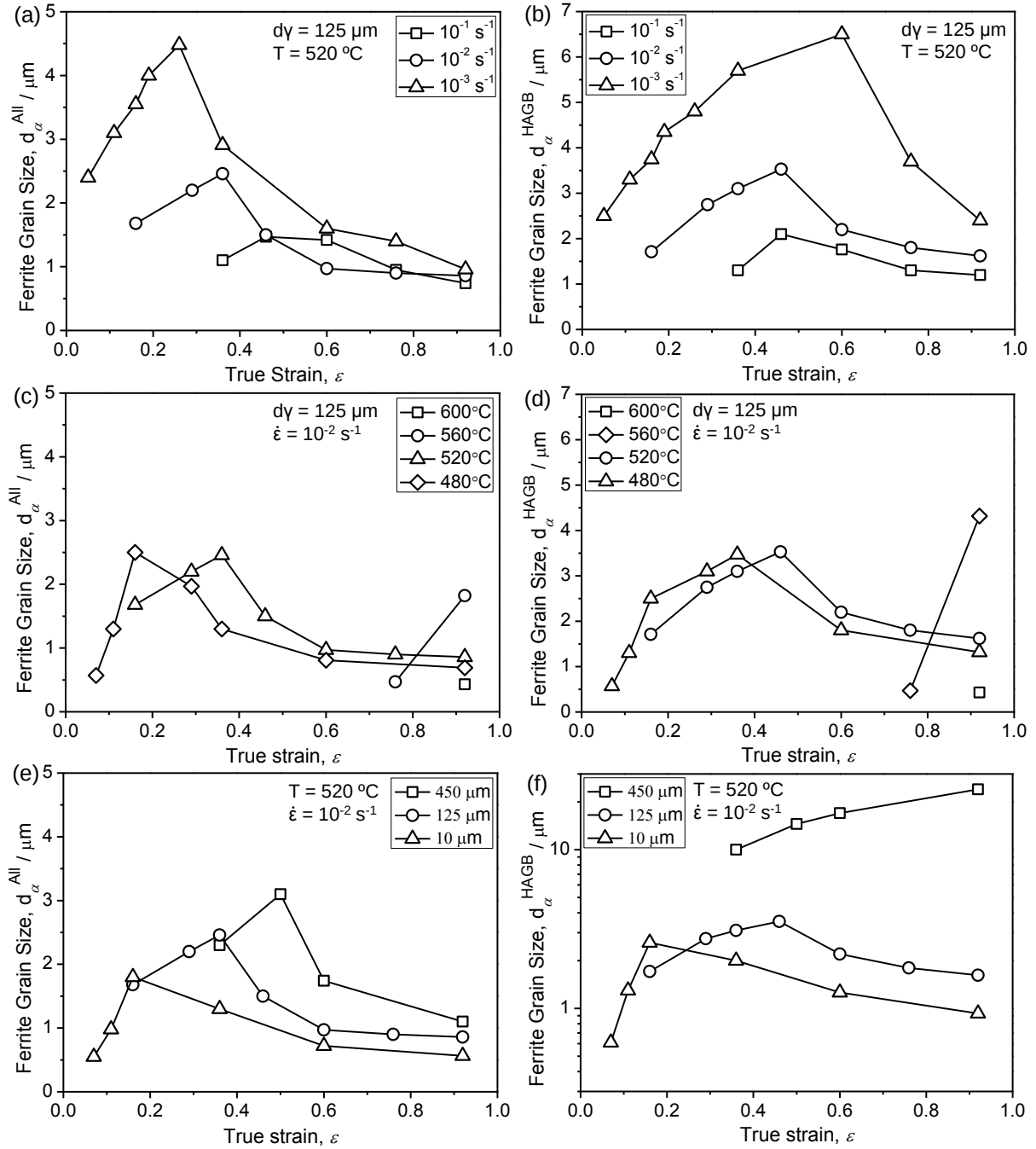


Fig 2.29 Changes in the ferrite grain sizes of the specimens deformed (a)-(b) at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C , (c)-(d) at a strain rate of 10^{-2} s^{-1} at different temperatures from 600 to 480°C , and (e)-(f) at a strain rate of 10^{-2} s^{-1} at 520°C with different austenite grain sizes. The austenite grain size in (a-d) is $125 \mu\text{m}$. The grain sizes along the compression axis were measured by linear interception method on the EBSD grain boundary maps counting all boundaries with misorientations larger than 2° (defined as d_{α}^{All}) or only counting boundaries with misorientations larger than 15° (defined as d_{α}^{HAGB}).

As mentioned above, the characteristic change in the grain size of ferrite shown in **Fig. 2.29** is probably due to the formation of equiaxed UFF grains. Here, the formation of UFF grains is discussed. The UFF grains have two kinds of features listed below:

- 1) Under a certain strain, the UFF grains exhibit more equiaxed aspect ratio than the ideal shape change by deformation, although it is difficult to exactly determine at which stage of deformation they are formed.
- 2) The UFF grains are much finer than the coarse and elongated grains (deformed grains), and are surrounded by HABs completely.

Here, the fine equiaxed grains formed near PAGBs at very early stage of DT are not counted as equiaxed UFF grains.

Equiaxed UFF grains are surely observed in present study, and the formation of UFF grains seems to be greatly affected by thermomechanical parameters (**Fig. 2.27**). Until now, the mechanism of UFF formation is one of the major areas under debate. Therefore, a systematic investigation on the mechanism of UFF formation will be particularly conducted in **Chapter 4**.

Not only the formation of euqiaxed UFF grains, but also the other microstructural features of ferrite are significantly affected by the thermomechancial processing parameters (i.e., strain rate, deformation temperature and austenite grain size). A detailed examination of the effects of these parameters on both nucleation and growth of ferrite grains is conducted

below.

1) Effect of strain rate on nucleation and growth of ferrite. Ferrite obtained at a high strain rate mostly consists of fine grains (**Fig. 2.22(a)**), indicating that the grain growth of ferrite is suppressed at a high strain rate. This is mainly due to two factors. Firstly, a larger amount of defects introduced by deformation of austenite at high strain rate can act as potential nucleation sites for the ferrite transformation, leading to an early impingement between ferrite grains after short distances of their growth (especially the grains formed along PAGBs, **Fig. 2.21(a)**). Secondly, the time for the ferrite grain growth is limited at high strain rate. Therefore, the formation of ferrite at high strain rate is a nucleation-dominant process. On the contrary, during deformation of austenite at low strain rate, ferrite nucleated near PAGBs grows into austenite grains. As is shown in **Fig. 2.21(b)**, the ferrite obtained at a strain rate of 10^{-2} s^{-1} consists of fine grains along the PAGBs and very coarse grains growing into martensite (i.e., retained austenite until cooling). The grain growth behavior is rather pronounced at this low strain rate. It is noteworthy that there is not much newly nucleated ferrite formed along the phase boundaries between the growing ferrite and martensite. This means that even though new nucleation sites are continuously generated during deformation of retained austenite, some of them are consumed by the rapid growth of initially nucleated ferrite. Therefore, the formation of ferrite at low strain rate is a nucleation-dominant process.

2) Effect of deformation temperature on nucleation and growth of ferrite. The morphology of ferrite differs depending on temperatures. At a high temperature of 600 °C (**Fig. 2.23(a)**), a small amount of ferrite nucleated mainly along PAGBs at an early stage of transformation, exhibiting fine and equiaxed morphology (**Fig. 2.24(a)**). At 560 °C, ferrite is mainly composed of coarse and elongated grains, indicating a pronounced grain growth at this temperature (**Fig. 2.23(b)**). The grain growth behavior of dynamically transformed ferrite is suppressed at low deformation temperatures (i.e., 520 °C and 480 °C, as shown in **Fig. 2.29(c)** and **(d)**). There are two reasons discussed below: Firstly, deformation at low deformation temperatures introduces more defects and thus can enhance the kinetics of ferrite transformation. Fast transformation kinetics (**Fig. 2.15(b)**) leads to an early impingement between ferrite grains, which is in favor of suppression of grain growth. Secondly, the low diffusivity of atoms at low temperatures naturally suppresses grain growth in long-distance. Therefore, it can be concluded that low deformation temperatures enhance the nucleation of ferrite and suppress the grain growth, which can lead to finer ferrite structure (**Fig. 2.23**).

3) Effect of austenite grain size on nucleation and growth of ferrite. Austenite grain boundary is the most preferential nucleation sites for ferrite. Fine austenite grains size (e.g., 10 µm) means larger area of austenite grain boundaries, which can increase the nucleation density for ferrite transformation and accelerate the transformation kinetics. Fast transformation kinetics can lead to an early impingement between ferrite grains.

Consequently, the grain growth of ferrite is suppressed, resulting in a finer ferrite structure (**Fig. 2.26(c)** and **Fig. 2.29(f)**). On the contrary, the nucleation density for ferrite transformation is small in coarse austenite grains (450 μm). Without sufficient impingement, the nucleated ferrite grows rapidly, resulting in very coarse grain morphology (**Fig. 2.25(a)**). Therefore, it could be concluded that ferrite transformation from fine austenite grains is a nucleation-dominant process, while ferrite transformation from coarse austenite grains is rather a growth-dominant process.

The effects of strain rate, deformation temperature and austenite grain size on the nucleation and growth behaviors of ferrite could be summarized in **Table 2.2**. It is clearly shown that high strain rates, low deformation temperatures and fine prior austenite grains enhance nucleation for ferrite and suppress ferrite grain growth, which can lead to finer ferrite structures (**Fig. 2.22, 2.24 and 2.26**).

Table 2.2 Effects of thermomechanical parameters on DT (nucleation and growth)

Parameters DT	Strain rate		Temperature		Austenite grain size	
	High	Low	High	Low	Coarse	Fine
Nucleation	O	×	×	O	×	O
Growth	×	O	O	×	O	×

O – Enhanced; × – Suppressed.

2.4 Conclusions

Occurrence of dynamic transformation (DT) from austenite to ferrite, and the effects of thermomechanical processing parameters on the kinetics of DT and ferrite microstructures were investigated in an Fe-10Ni-0.1C alloy. The main results are summarized as follows:

- 1) Occurrence of DT was confirmed through analyzing the softening behaviors on true stress-true strain curves using a double differentiation method in association with microstructure observations. Critical strains for the onset of DT, estimated by the double differentiation method under various conditions, were obtained. The volume fraction of ferrite experimentally observed at the critical strains fell into a range of 2-10%, indicating a good applicability of this method for determining the onset of DT.
- 2) Lower deformation temperatures and finer austenite grains resulted in smaller critical strain for the onset of DT and accelerated its kinetics. High strain rates also accelerated the transformation kinetics, but low strain rates decreased the critical strain for the onset of DT and led to a higher fraction of ferrite after a certain amount of compression deformation, which was mainly due to the longer deformation time.
- 3) DT showed a different grain growth behavior from ST. The grain size of dynamically transformed ferrite firstly increased and then decreased after a certain strain, which corresponded to the formation of ultrafine ferrite (UFF) grains.
- 4) High strain rates, low deformation temperatures and fine prior austenite grains enhanced

nucleation for ferrite and suppressed ferrite grain growth, resulting in finer ferrite structures.

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Chapter 3: Difference between DT and ST from deformed austenite

3.1 Introduction

It has been previously reported that kinetics of ferrite transformation is greatly accelerated under the thermomechanical conditions involving dynamic transformation (DT), i.e., deformation and transformation progress concurrently [1-12]. Some works suggest that the deformation of austenite introduces a large amount of lattice defects and deformation microstructures which act as preferential nucleation sites for ferrite transformation, leading to the acceleration of transformation kinetics. From this point of view, the mechanism of DT seems the same as that of static transformation (ST) from deformed austenite [13-16]. However, in the case of ST from deformed austenite, deformation of austenite has completed before the initiation of ferrite transformation. In the case of DT, on the other hand, deformation is continuously applied during ferrite transformation, so that it seems reasonable to consider that the nucleation and growth of ferrite are different between DT and ST from deformed austenite. Therefore, the comparison of transformation kinetics between the two phenomena (DT and ST from deformed austenite) is necessary and important to understand the nature of DT.

Most of the works to date have presented that the ferrite formed by DT exhibits equiaxed grain morphologies [17-26], which is similar to the ferrite formed by ST from deformed

austenite [13-16]. Some researchers have proposed that during the thermomechanical processes including DT, plenty of lattice defects and deformation microstructures (e.g., microband, etc.) introduced by deformation act as preferential nucleation sites for ferrite transformation and the nucleation density of ferrite significantly increases. The high nucleation density suppresses grain growth of ferrite by impingement of ferrite grains to each other, leading to the formation of equiaxed ultrafine ferrite (UFF). However, in **Chapter 2**, it has been confirmed that dynamically transformed ferrite is composed of both equiaxed UFF grains and elongated coarse grains. The elongated coarse grains contain subgrains surrounded by low-angle boundaries, which indicates that the coarse ferrite grains have been deformed during compression after the nucleation. On the other hand, the deformation of transformed ferrite never occurs in the case of ST from deformed austenite. The difference in ferrite microstructures between DT and ST (especially the formation of equiaxed UFF in DT) is quite unclear and requires further investigations.

In this chapter, systematic experiments were designed aiming to investigate the difference in the transformation kinetics and ferrite microstructures between DT and ST from deformed austenite.

3.2 Experimental procedure

3.2.1 Material

The material used in this study is the same alloy (Fe-10Ni-0.1C) as that studied in **Chapter 2**, of which chemical composition and TTT diagram were shown in **Table 2.1** and **Fig. 2.3**, respectively. The as-received hot-rolled plate 15 mm thick was cold-rolled to a thickness of 11.2 mm with a reduction of 25%, then homogenized at 1100 °C for 172,800 s in vacuum followed by water-quenching. Cylindrical specimens with a height of 12 mm and a diameter of 8 mm were machined from the homogenized plate and installed in a thermomechanical processing simulator (Thermecmasto-Z, Fuji Electronic Industrial Co. Ltd.) for conducting the thermomechanical processes.

3.2.2 Thermomechanical processes

Four kinds of thermomechanical processing routes were designed to systematically investigate the static and dynamic ferrite transformation behaviors, as shown in **Fig. 3.1**. There are two routes for static transformation (ST1 and ST2) and another two routes for dynamic transformation (DT1 and DT2). All the specimens were austenitized at 1000 °C for 300 s, resulting in an average austenite grain size of 125 μm. For investigating ferrite transformation in the route-ST2, it requires a condition under which ferrite transformation cannot occur during plastic deformation. According to the results in **Chapter 2**, after

austenite was deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C, no ferrite was observed in the water-quenched specimen. Therefore, this deformation condition is selected in present chapter for conducting the route-ST2. In order to compare the kinetics of transformation and ferrite microstructure between the four routes, the deformation temperature (520 °C) and applied strain (0.92) is same in the route-ST2, route-DT1 and route-DT2.

The route-ST1 is for simulating static ferrite transformation during isothermal holding. In the route-ST1 (**Fig. 3.1(a)**), the specimens austenitized at 1000 °C were furnace-cooled to 520 °C at a cooling rate of 0.2 °C s^{-1} , and then isothermally held for different periods of time ranging from 3600 s to 88,200 s followed by water-quenching.

The route-ST2 is for simulating static ferrite transformation from deformed austenite. In the route-ST2 (**Fig. 3.1(b)**), the austenitized specimens were cooled by nitrogen gas to 520 °C at a cooling rate of 30 °C s^{-1} , held for 60 s to homogenize the temperature of the cylindrical specimens, uniaxially compressed to a strain of 0.92 at a strain rate of 10^0 s^{-1} and then isothermally held at 520 °C for different periods of time from 0 s to 12,600 s followed by water-quenching. It was confirmed that no ferrite transformation happened before the completion to a strain of 0.92.

The route-DT1 is for simulating dynamic transformation. In the route-DT1 (**Fig. 3.1(c)**), the austenitized specimens were cooled by nitrogen gas to 520 °C at a rate of 30 °C s^{-1}

s^{-1} , isothermally held for different periods of time from 500 s to 19,800 s in order to consume the incubation period of ferrite transformation, and then uniaxially compressed to a strain of 0.92 at a strain rate of 10^0 s^{-1} followed by water-quenching. The strain rate in route- DT1 is same as that in the route-ST2. The difference between the route-DT1 and route-ST2 is that, the isothermal holding time before deformation varies in the route-DT1. The consumption of incubation period of ferrite transformation aims to enhance the possibility of DT during subsequent compression.

The route-DT2 is also for simulating dynamic transformation. In the route-DT2 (**Fig. 3.1(d)**), the austenitized specimens were cooled by nitrogen gas to 520°C at a rate of 30°C s^{-1} , held for 60 s to homogenize the temperature of the cylindrical specimens, and then uniaxially compressed to a strain of 0.92 at different strain rates ranging from 10^0 to 10^{-3} s^{-1} followed by water-quenching. The difference of the route-DT2 from the route-DT1 is that, the isothermal holding time is constant (60 s) in this route, but the strain rate is different. Since the applied strain is same in route-DT1 and route-DT2, therefore the deformation time (converted from the strain and strain rate) in the route-DT2 is different from that in the route-DT1. The aim of this design is to examine the effect of strain rate (or deformation time) on DT.

Mica and glass powder were used as the insulator and lubricant to minimize the friction between the contact surfaces of the anvils and the specimen during straining. The water injection was precisely controlled to minimize the time interval until the initiation of cooling.

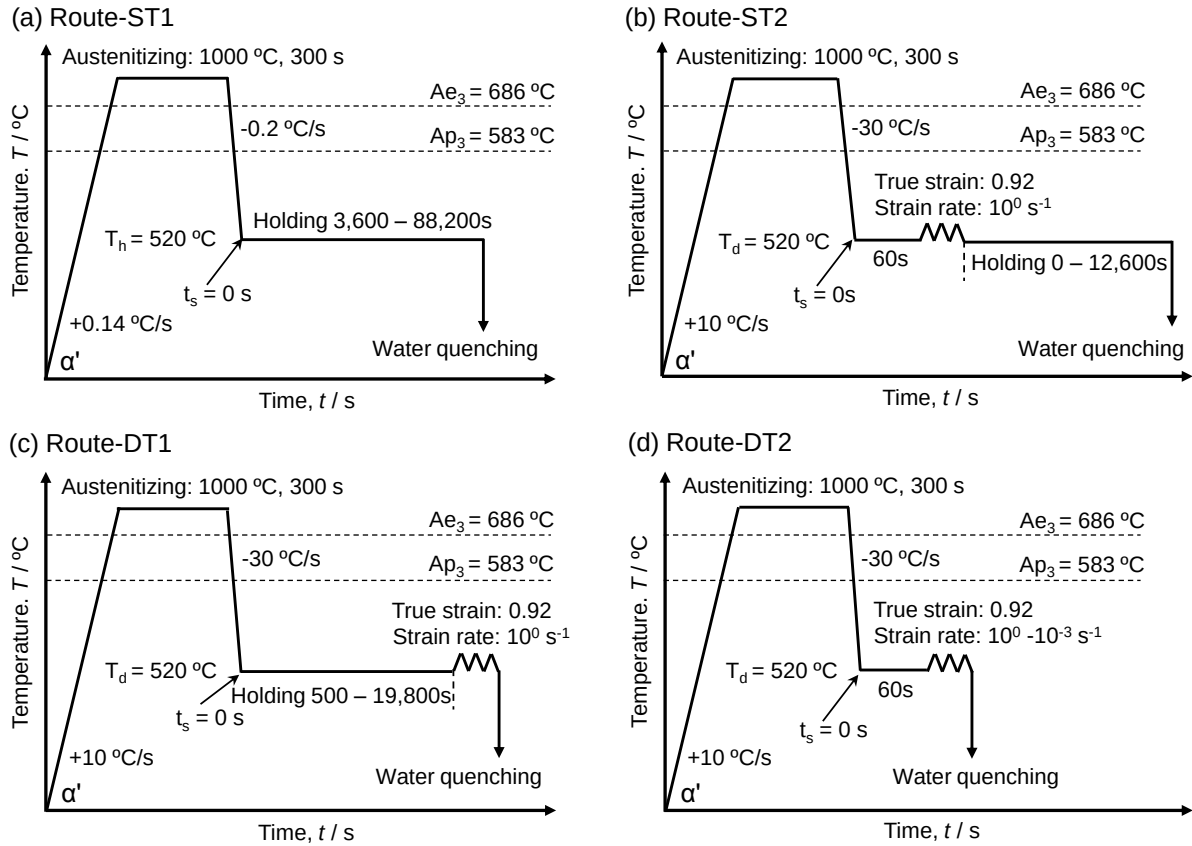


Fig. 3.1 Schematic illustrations showing four different thermomechanical routes carried out in this chapter. (a) and (b) show two routes for static transformation (ST1 and ST2). (c) and (d) show two routes for dynamic transformation (DT1 and DT2). The applied strain, deformation temperature and austenite grain size are same in the four routes.

3.2.3 Microstructure observation

Microstructures at the center of the cylindrical specimens on the sections parallel to the compression axis were characterized by optical microscopy (OM), a field-emission type scanning electron microscope (FE-SEM, FEI XL30S FEG) and electron back-scattering diffraction (EBSD) analysis performed in the FE-SEM. The specimen was mechanically polished and etched using a 3% nital solution for the OM and SEM observations. For the EBSD measurement, the specimens were mechanically polished and then electrically polished in a solution of 10% HClO_4 and 90% CH_3COOH at 20 °C. The EBSD measurement was performed using TSL OIM Data Collection system, and the obtained EBSD data were analyzed by TSL OIM Analysis software.

3.3 Results and discussion

3.3.1 Difference in transformation kinetics

In this section, differences in ferrite transformation kinetics between the four routes (i.e., the route-ST1, route-ST2, route-DT1 and route-DT2) are investigated at the same temperature (520 °C). No strain is applied in the route-ST1. The same amount of strain (logarithmic strain of 0.92; 60% compression reduction) is applied in the route-ST2, route-DT1 and route-DT2. The strain rate is the same (10^0 s^{-1}) in the route-ST2 and route-DT1.

The microstructures of the specimens processed in the four routes are mainly presented by OM images from **Fig. 3.2** to **Fig. 3.5**. **Figure 3.2** shows OM images of the specimens austenitized at 1000 °C and then isothermally held at 520 °C for different periods of time ((a) 12,600 s, (b) 19,800 s, (c) 37,800 s and (d) 88,200 s) followed by water-quenching in the route-ST1. “F” and “M” indicate ferrite (light area) and martensite (dark area), respectively. Point-counting method was used for measuring the volume fraction of ferrite in OM images, which was introduced in **Chapter 2**. The volume fraction of ferrite is shown on the top right corner of each image, which is the case for all OM images in this chapter hereafter. The statically transformed ferrite exhibits polygonal morphology. With increasing the isothermal holding time from 12,600 s (**Fig. 3.2(a)**) to 88,200 s (**Fig. 3.2(d)**), the volume fraction of ferrite increases from 1.0% to 39.6%, indicating very sluggish kinetics of static

ferrite transformation.

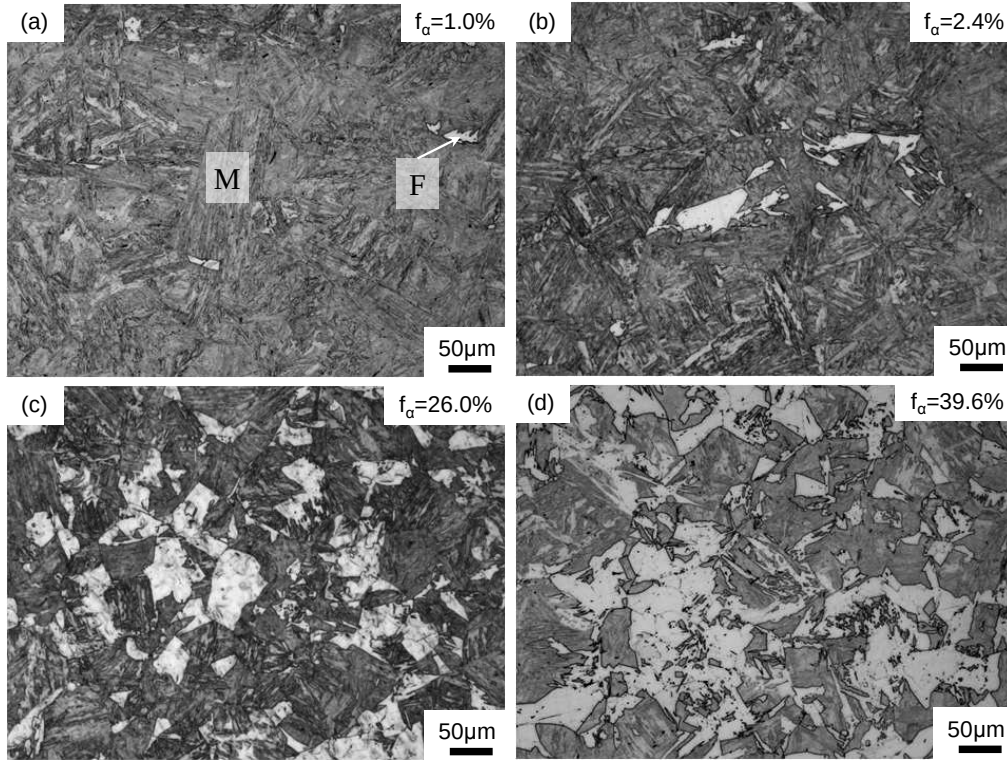


Fig. 3.2 OM images of the specimens austenitized at 1000 °C and then isothermally held at 520 °C for (a) 12,600 s, (b) 19,800 s, (c) 37,800 s and (d) 88,200 s followed by water-quenching in the route-ST1. “F” and “M” indicate ferrite (light area) and martensite (dark area), respectively. The volume fraction of (statically transformed) ferrite is shown on the top right corner of each image.

Figure 3.3 shows OM images of the specimens austenitized at 1000 °C, deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C and then isothermally held at 520 °C for (a) 0 s, (b) 9 s, (c) 92 s and (d) 916 s followed by water-quenching in the route-ST2. For the specimen water-quenched right after deformation (**Fig. 3.3(a)**), only martensite is observed, which confirms that (dynamic) ferrite transformation has not occurred till the end of the compression deformation. After isothermal holding for 9 s (**Fig. 3.3(b)**), a small amount of

nearly equiaxed ferrite appears mainly along the prior austenite grain boundaries (PAGBs). With increasing the isothermal holding time to 92 s (**Fig. 3.3(c)**) and 916 s (**Fig. 3.3(d)**), the volume fraction of ferrite increases to 55% and 79%, respectively. Ferrite growing into martensite (prior austenite) exhibits coarse morphology. As the fraction of ferrite was only 2.4% even after 19,800 s holding (**Fig. 3.2(b)**) in the route-ST1 (isothermal holding without deformation), the kinetics of static ferrite transformation is significantly accelerated by deformation of austenite in the route-ST2.

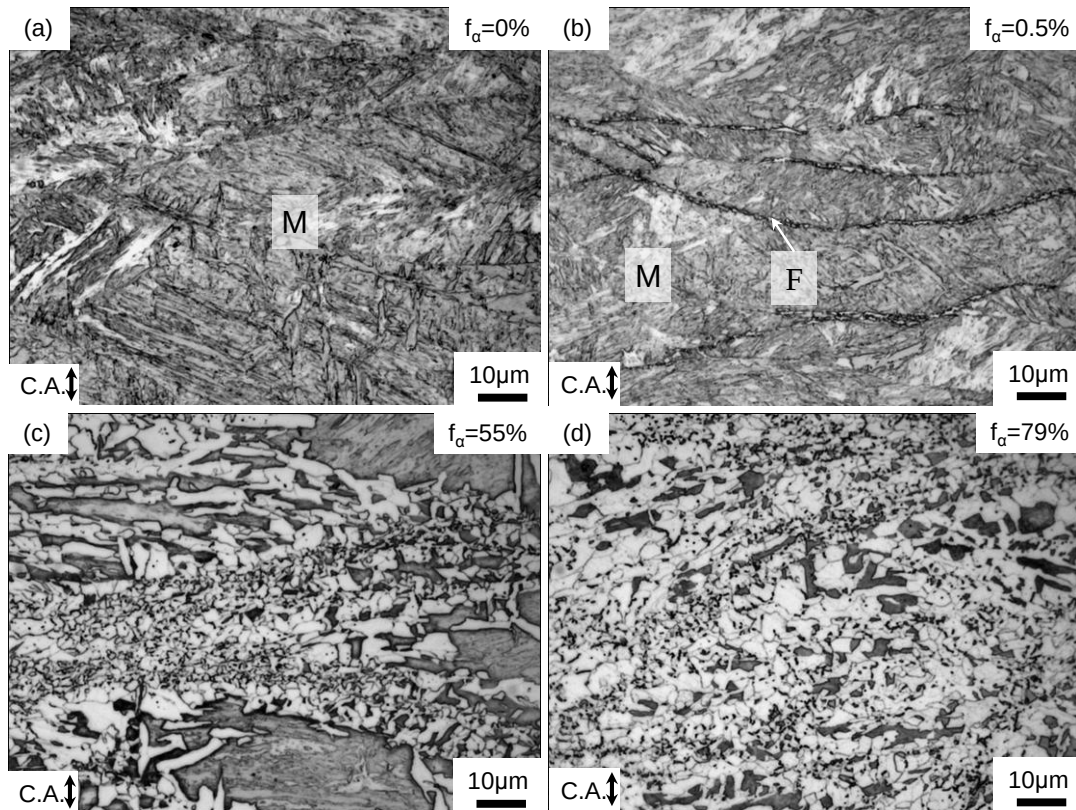


Fig. 3.3 OM images of the specimens austenitized at 1000 °C, deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C and then isothermally held at 520 °C for (a) 0 s, (b) 9 s, (c) 92 s and (d) 916 s followed by water-quenching in the route-ST2. “F” and “M” indicate ferrite and martensite, respectively. The volume fraction of ferrite is shown on the top right corner of each image. Compression axis is along the vertical direction of the images.

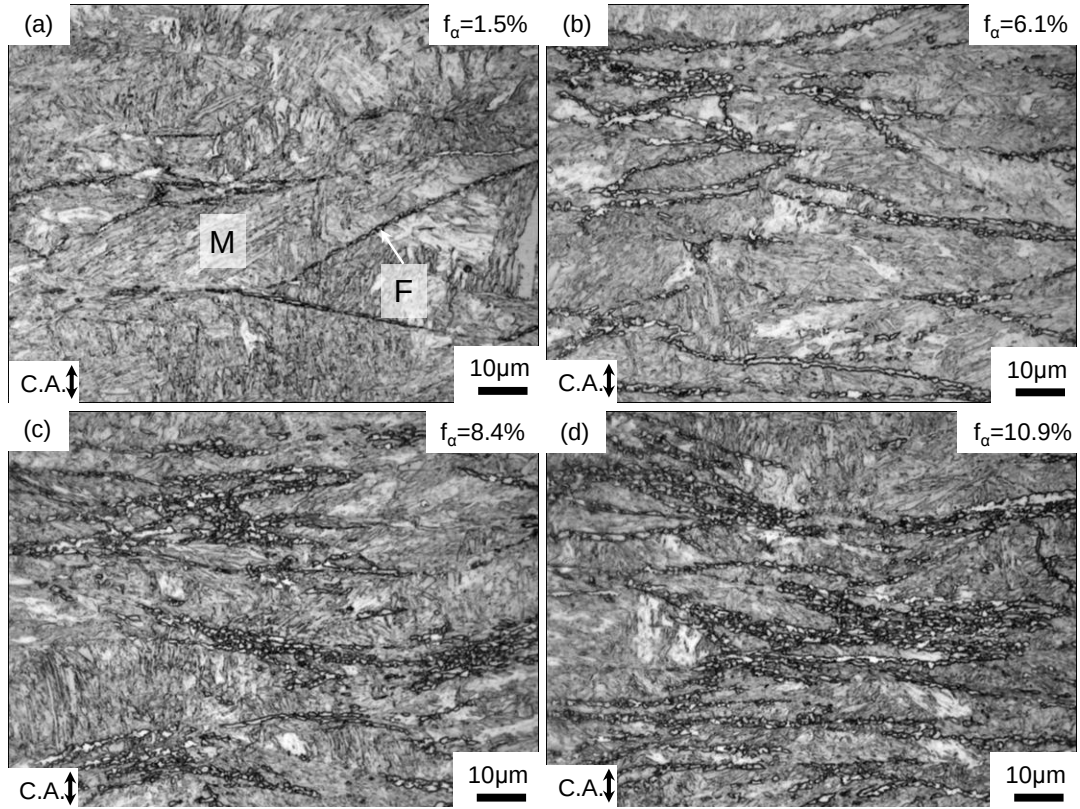


Fig. 3.4 OM images of the specimens austenitized at 1000 °C, isothermally held at 520 °C for (a) 500 s, (b) 3,600 s, (b) 12,600 s and (d) 19,800 s, and then deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C followed by water-quenching in the route-DT1. “F” and “M” indicate ferrite and martensite, respectively. The volume fraction of ferrite is shown on the top right corner of each image. Compression axis is along the vertical direction of the images.

Figure 3.4 shows OM images of the specimens austenitized at 1000 °C, isothermally held at 520 °C for (a) 500 s, (b) 3,600 s, (b) 12,600 s and (d) 19,800 s, and then deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C followed by water-quenching in the route-DT1. For the specimen deformed after isothermal holding for 500 s (**Fig. 3.4(a)**), a small amount (1.5%) of nearly equiaxed ferrite is formed along PAGBs. They are dynamically transformed ferrite grains. With increasing the isothermal holding time (before deformation) from 3600 s (**Fig. 3.4(b)**) to 12,600 s (**Fig. 3.4(c)**) and 19,800 s (**Fig. 3.4(d)**),

the dynamically transformed ferrite keep equiaxed morphology, and the volume fraction of ferrite slightly increases from 6.1% to 8.4% and 10.9%. The result indicates that isothermal holding before deformation has no significant effect on enhancing the kinetics of DT.

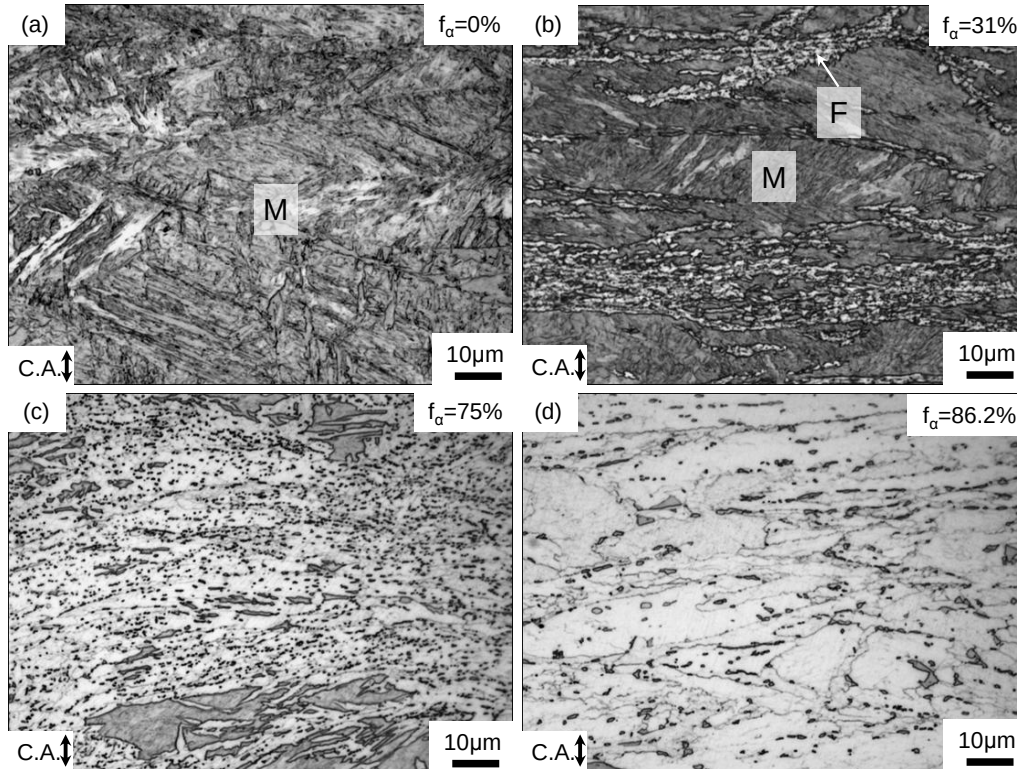


Fig. 3.5 OM images of the specimens austenitized at 1000 °C, held at 520 °C for 60 s, and then deformed to a strain of 0.92 at strain rates of (a) 10^0 s^{-1} (b) 10^{-1} s^{-1} , (c) 10^{-2} s^{-1} and (d) 10^{-3} s^{-1} at 520 °C in the route-DT2. “F” and “M” indicate ferrite and martensite, respectively. The volume fraction of ferrite is shown on the top right corner of each image. Compression axis is along the vertical direction of the images.

Figure 3.5 shows OM images of the specimens austenitized at 1000 °C, isothermally held at 520 °C for 60 s, and then deformed to a strain of 0.92 at different strain rates of (a) 10^0 s^{-1} , (b) 10^{-1} s^{-1} , (c) 10^{-2} s^{-1} and (d) 10^{-3} s^{-1} at 520 °C followed by water-quenching in the route-DT2. At a high strain rate of 10^0 s^{-1} (**Fig. 3.5(a)**), no ferrite is formed, which was

already confirmed in **Fig. 3.3(a)**. At a lower strain rate of 10^{-1} s^{-1} (**Fig. 3.5(b)**), fine ferrite could be observed with a volume fraction of 31%. With decreasing the strain rate to 10^{-2} s^{-1} (**Fig. 3.5(c)**) and 10^{-3} s^{-1} (**Fig. 3.5(d)**), ferrite becomes coarse and the volume fraction of ferrite increases to 75% and 86.2%, respectively. The result indicates that lowering the strain rates enhances the ferrite formation significantly.

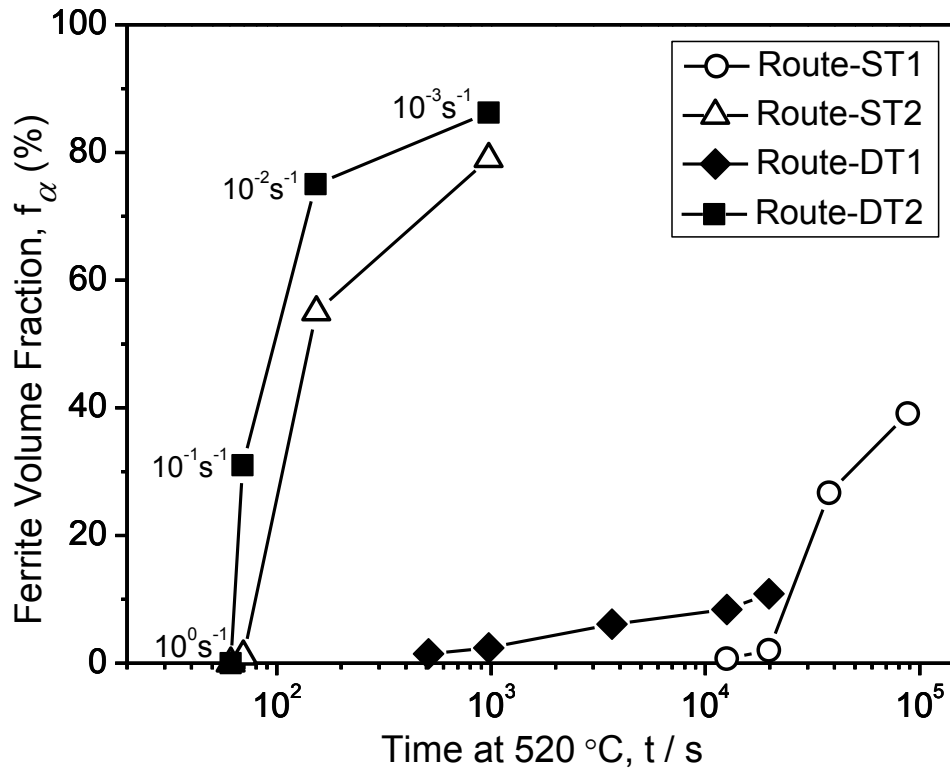


Fig. 3.6 Change in the volume fraction of ferrite transformed in the four processing routes as a function of time. The time means the total time at 520 °C including both isothermal holding time and deformation time (converted from applied strain and strain rate). Open and solid symbols represent the volume fraction of statically and dynamically transformed ferrite, respectively.

In order to compare the transformation kinetics of ferrite (the formation rate of ferrite) between the four routes, the volume fraction of ferrite is plotted as a function of time in **Fig.**

3.6. Here, the time means the total time at 520 °C including both isothermal holding time and deformation time (converted from applied strain and strain rate). In **Fig. 3.6**, open and solid symbols represent the volume fraction of statically and dynamically transformed ferrite, respectively. In the route-ST1, the kinetics of static transformation (without deformation) is very sluggish. Ferrite transformation initiates at 12,600 s, and the volume fraction of ferrite slightly increases to 39.6% after isothermal holding for 88,200 s. Compared to the route-ST1, the kinetics of static transformation is significantly accelerated by the deformation of austenite in the route-ST2. Ferrite transformation starts at around 70 s (i.e., isothermal holding for 60 s before deformation, 1 s during deformation, and 9 s after the deformation), and the volume fraction of ferrite reaches to 79% at 977 s. The result indicates that deformation of austenite greatly enhances subsequent static ferrite formation. In the route-DT1, even though the specimens are isothermally held for relatively long periods from 500 s to 19,800 s before deformation, the volume fraction of dynamically transformed ferrite just slightly increases from 1.5% to 10.9% after a strain of 0.92. Note that only after isothermally holding for 19,800 s at 520 °C without deformation, the volume fraction of statically transformed ferrite becomes already 2.4% in the route-ST1. The results indicate that consumption of incubation time by isothermal holding before deformation (route-DT1) has no significant effect on accelerating the transformation kinetics. In the route-DT2, no ferrite is formed at a high strain rate of 10^0 s^{-1} , corresponding to the total time of 61 s (60 s in

isothermal holding and 1 s in deformation) at 520 °C. Decreasing the strain rate significantly enhances the ferrite formation, even though the isothermal holding time before deformation is only 60 s. Compared with the route-DT1, as the applied strain is the same (0.92) in the route-DT2, decreasing strain rate corresponds to increasing deformation time at 520 °C. It indicates that, for DT, time for deformation is more important to enhance the ferrite formation than the isothermal holding time before deformation. Furthermore, the comparison between the route-DT2 and the route-ST2 gives us a very interesting aspect. At the same total time at 520 °C, the volume fraction of dynamically transformed ferrite obtained in the route-DT2 is always higher than that of the statically transformed ferrite in the route-ST2.

To explore the difference in ferrite transformation kinetics between the route-DT 2 and the route-ST 2, detailed examinations of nucleation and growth of ferrite grains in two routes were conducted. In order to facilitate the discussion, comparison between the route-ST2 and route-DT2 is concentrated on one condition, that is, within the same transformation time of 153 s in both routes. **Figure 3.7** shows grain average misorientation (GAM) maps, obtained by EBSD measurements of the specimens (a) deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} and then isothermally held for 92 s at 520 °C in the route-ST2, and (b) deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 °C immediately followed by water-quenching in the route-DT2. The total periods at 520 °C are (a) 153 s and (b) 153 s.

The GAM map, as introduced in **Chapter 2**, can be used to evaluate misorientation within each grain. Non-ferrite phases (i.e., martensite and/or retained austenite) are painted in black. The methods for distinguishing ferrite from martensite or retained austenite were explained in **Chapter 2**. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. Statically transformed ferrite obtained in the route-ST2 shows equiaxed grain morphologies surrounded by HABs and contains a small amount of LABs (**Fig. 3.7(a)**). The average GAM value of ferrite is around 0.32 °, indicating low average misorientation within the ferrite grains. Dynamically transformed ferrite in the route-DT2 shows mostly elongated coarse grains and partly a small amount of equiaxed grains surrounded by HABs (as marked by black arrows in **Fig. 3.7(b)**). The elongated grains contain a large amount of LABs, suggesting deformed structures inside the grains. The average GAM value of ferrite is around 0.50 °, indicating that the average misorientation within the dynamically transformed ferrite is much higher than that within statically transformed ferrite. The elongated ferrite grains in **Fig. 3.7(b)** are much coarser than the grains in **Fig. 3.7(a)**, indicating more pronounced grain growth of ferrite in the route-DT2. Accordingly, there are larger numbers of ferrite grains in **Fig. 3.7(a)** than **Fig. 3.7(b)**, which indicates that the nucleation density in the route-ST2 is higher than that in the route-DT2.

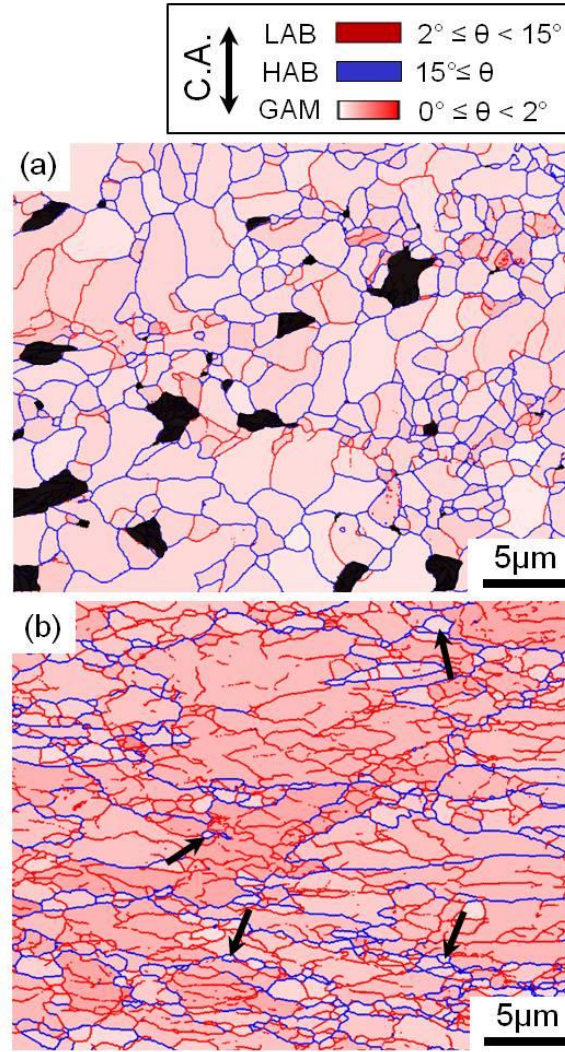


Fig. 3.7 Grain average misorientation (GAM) maps of the specimens (a) deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} and then isothermally held for 92 s at 520 °C in the route-ST2, and (b) deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 °C immediately followed by water-quenching in the route-DT2. Total period for (a) and (b) is same (i.e., 153 s). Non-ferrite phases (i.e., martensite and/or retained austenite) are painted in black. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. Black arrows in (b) mark equiaxed grains. Compression axis is along the vertical direction of the maps.

The number of ferrite grains was measured by counting the grains surrounded by HABs in GAM maps. The apparent nucleation density of ferrite was then evaluated by dividing the number of ferrite grains by the total area of the grains in GAM maps. This method has

been widely used for evaluating the nucleation of ferrite grains [13, 15]. **Figure 3.8** shows changes in the apparent nucleation density of ferrite in the route-ST2 and the route-DT2 as a function of time. The apparent nucleation density decreases with increasing the transformation time in both routes, corresponding to a grain growth process during the transformation. At the same time period at 520 °C, the apparent nucleation density in the route-DT2 is much lower than that in the route-ST2. Note that the volume fraction of ferrite in the route-DT2 is higher than that in the route-ST2. Therefore, it is reasonable to conclude that the fast transformation kinetics in the route-DT2 (**Fig. 3.6**) is not due to the high nucleation density (**Fig. 3.8**), but mainly due to the faster grain growth rate under deformation (**Fig. 3.7(b)**).

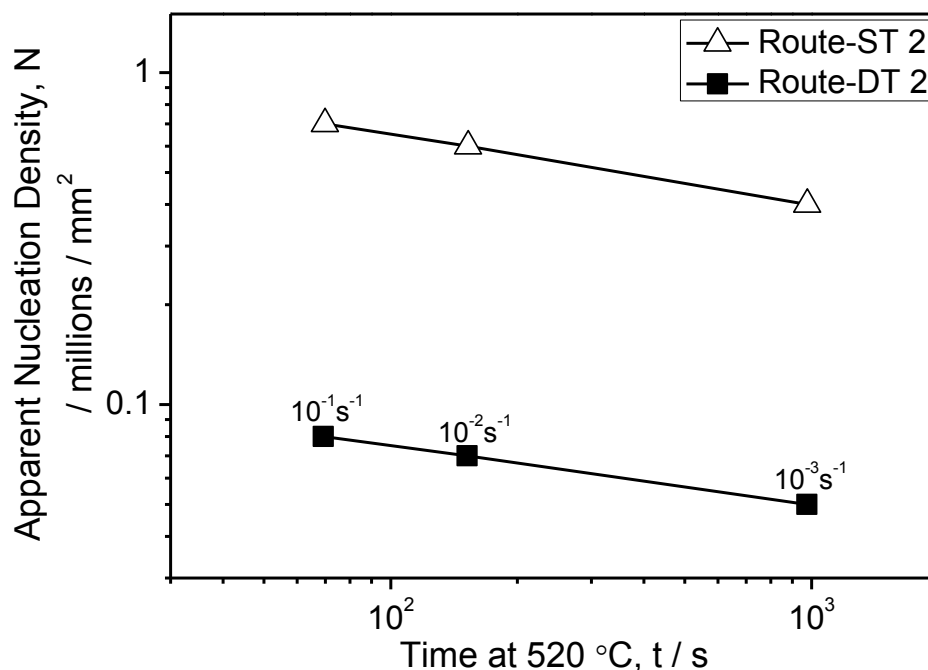


Fig. 3.8 Changes in apparent nucleation density in the route-ST2 and the route-DT2 as a function of time at 520 °C.

The reason why the grain growth rate in the route-DT2 is faster than that in the route-ST2 is considered below. The grain growth rate depends on the velocity of α/γ interface during austenite (γ) to ferrite (α) transformation. High α/γ interface velocity means high grain growth rate of ferrite. The interface velocity v_α is considered to be proportional to the product of the interface mobility M and the driving force for the transformation $\Delta G_{\alpha\gamma}$ ($\Delta G_{\alpha\gamma} < 0$) [27, 28]:

$$v_\alpha = M\Delta G_{\alpha\gamma} \quad (3.1)$$

The interface mobility exhibits an Arrhenius-type temperature dependence:

$$M(T) = M_0 \exp(-Q/RT) \quad (3.2)$$

where M_0 is the pre-exponential factor and Q is the activation energy for interface migration. The driving force $\Delta G_{\alpha\gamma}$ in equation (3.1) is given by

$$\Delta G_{\alpha\gamma} = \Delta G_{\alpha\gamma}^{chem} + \Delta G_\gamma^{def} + [\Delta G_{\alpha\gamma}^{def}(f_\alpha) + \Delta G_{\alpha\gamma}^{int}(f_\alpha)] \quad (3.3)$$

where $\Delta G_{\alpha\gamma}^{chem}$ ($\Delta G_{\alpha\gamma}^{chem} < 0$) is the molar chemical Gibbs energy difference between ferrite and austenite, ΔG_γ^{def} ($\Delta G_\gamma^{def} < 0$) is the molar stored energy in deformed austenite, $\Delta G_{\alpha\gamma}^{def}(f_\alpha)$ ($\Delta G_{\alpha\gamma}^{def}(f_\alpha) > 0$) is the summation of molar elastic and plastic misfit energies between α and γ at the interface, and $\Delta G_{\alpha\gamma}^{int}(f_\alpha)$ ($\Delta G_{\alpha\gamma}^{int}(f_\alpha) > 0$) is the molar free energy of α/γ interface. The driving force $\Delta G_{\alpha\gamma}$ consists of the terms $\Delta G_{\alpha\gamma}^{chem}$ and ΔG_γ^{def} which favor the transformation, and the terms $\Delta G_{\alpha\gamma}^{def}(f_\alpha)$ and $\Delta G_{\alpha\gamma}^{int}(f_\alpha)$ which obstruct the transformation. It is reasonable to assume that the chemical compositions of ferrite and

austenite are the same between the route-ST2 and route-DT2. Since the chemical driving force $\Delta G_{\alpha\gamma}^{chem}$ only depends on the temperature (T), the values are considered to be the same both in the route-ST2 and the route-DT2 at the same deformation temperature (520 °C). Both $\Delta G_{\alpha\gamma}^{def}(f_{\alpha})$ and $\Delta G_{\alpha\gamma}^{int}(f_{\alpha})$ primarily depend on the fraction of transformed ferrite. With the progress of transformation, the number of α/γ interface layers increases, so that the values of $\Delta G_{\alpha\gamma}^{def}(f_{\alpha})$ and $\Delta G_{\alpha\gamma}^{int}(f_{\alpha})$ increase with increasing the ferrite fraction [29]. Therefore, their values are considered to be higher in the route-DT2 than the route-ST2, because the volume fraction of transformed ferrite in the route-DT2 is always higher at the same holding time at 520 °C (**Fig. 3.6**).

The stored energy in austenite ΔG_{γ}^{def} in equation (3.3), can be calculated by the following formula [30], assuming that the stored energy is mostly due to dislocations accumulated in the crystal,

$$\Delta G_{\gamma}^{def} = \mu b^2 \rho \quad (3.4)$$

where μ is the shear modulus, ρ is the dislocation density, and b is the magnitude of Burgers vector. It is also well known that the flow stress σ of deformed austenite is proportional to the square root of the dislocation density, as shown in the following equation (Bailey-Hirsch equation) [31, 32],

$$\sigma = N\alpha\mu b\rho^{1/2} \quad (3.5)$$

where N is the Taylor factor of polycrystalline austenite, and α is a numerical constant.

Thus, combining equation (3.4) and (3.5), the stored energy in austenite could be described as,

$$\Delta G_Y^{def} = \sigma^2 / \mu N^2 \alpha^2 \quad (3.6)$$

In equation (3.6), $\mu N^2 \alpha^2$ is constant, so that ΔG_Y^{def} is proportional to the square of the flow stress σ^2 .

Figure 3.9 shows true stress-true strain curves of the specimens deformed to a strain of 0.92 at strain rates from 10^0 s^{-1} to 10^{-3} s^{-1} at 520 °C. As is shown, the flow stress increases with increasing the strain rate. The flow stresses of the specimens deformed in the route-DT2 are lower than that in the route-ST2. This means that the stored energy of austenite ΔG_Y^{def} (mechanical driving force) in the route-DT2 is lower than that in the route-ST2 (equation (3.6)).

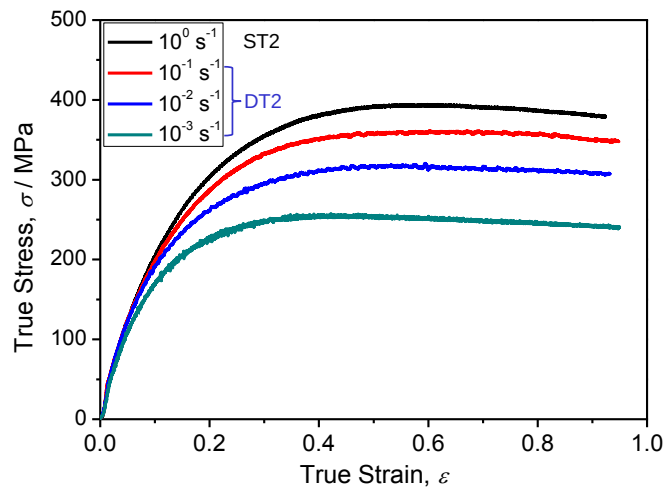


Fig. 3.9 True stress-true strain curves of the specimens deformed to a strain of 0.92 at strain rates from 10^0 s^{-1} to 10^{-3} s^{-1} at 520 °C.

Here the discussions about all parameters in equation (3.3) are summarized: for the chemical driving force $\Delta G_{\alpha\gamma}^{chem}$, it is equivalent in both routes; for the mechanical driving force ΔG_{γ}^{def} , it is lower in the route-DT2 than in the route-ST2; for the term $[\Delta G_{\alpha\gamma}^{def}(f_{\alpha}) + \Delta G_{\alpha\gamma}^{int}(f_{\alpha})]$, it is higher in the route-DT2, but these two parameters obstruct the transformation (positive values). Therefore, the total driving force $\Delta G_{\alpha\gamma}$ is smaller in the route-DT2 than that in the route-ST2.

According to equation (3.1), the interface velocity v_{α} (related to the grain growth rate, **Fig. 3.7**) is higher, but the driving force $\Delta G_{\alpha\gamma}$ is smaller in the route-DT2, so that the interface mobility M must be higher in the route-DT2 than that in the route-ST2. In other words, the faster transformation kinetics in the route-DT2 is mainly due to the high interface mobility M . It has been simulated by a kinetic Monte Carlo Method [33] that under uniaxial compressive stress, presence of lattice defects (vacancy, etc.) in the vicinity of α/γ interface can lower the planar density of atoms, provide extra free volume at the moving α/γ interface, and thereby lowers the extent of local rearrangement necessary to move the interface (to drive the transformation). For the DT phenomenon in the present study (i.e., in the route-DT2), the compression deformation is applied during transformation and can continuously introduce lattice defects into austenite. During the migration of α/γ interfaces (i.e., ferrite grain growth) under compression, the defects in the vicinity of α/γ interfaces can decrease the activation energy Q for the migration of the interface, resulting

in higher interface mobility (equation (3.2)) and thus faster interface velocity (grain growth rate). Consequently, the transformation kinetics is more accelerated under compression deformation. Therefore, the faster transformation kinetics in the route-DT2 is mainly due to the deformation applied during transformation. This is considered to be an essential difference between DT (route-DT2) and ST from deformed austenite (route-ST2).

3.3.2 Difference in ferrite microstructure

In the last section, OM images of the specimens processed in the route-ST1, route-ST2, route-DT1 and route-DT2 (**Figs. 3.2-3.5**) have provided a whole view of the microstructure evolution of ferrite in the four routes. In this section, more detailed microstructural features of ferrite transformed in the four routes are investigated by SEM and EBSD.

Since the static ferrite formed in the route-ST1 is very coarse, OM image is sufficiently clear to present the feature of ferrite (**Fig. 3.2**). Therefore, only one SEM image is shown here as an example.

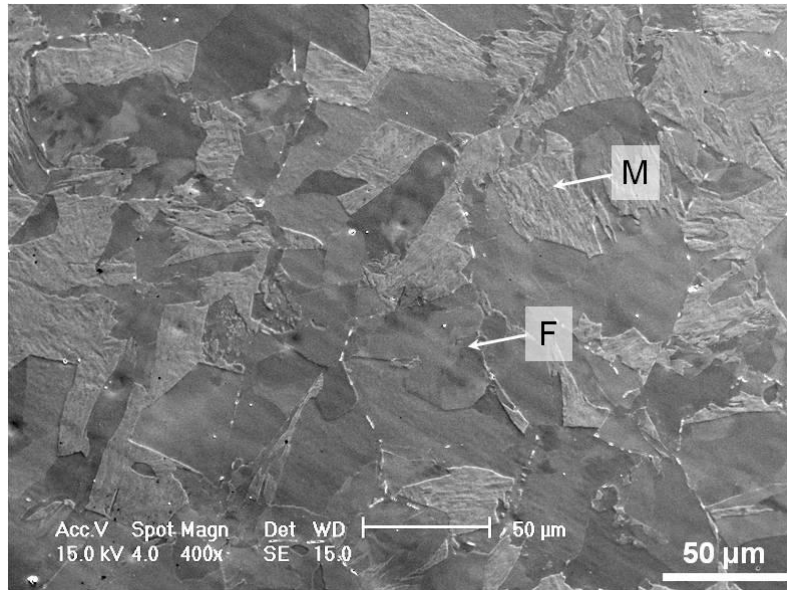


Fig. 3.10 SEM image of the specimen processed in the route-ST1: Austenitized at 1000 °C, and isothermally held at 520 °C for 88,200 s followed by water-quenching. “F” and “M” indicate ferrite and martensite, respectively.

Figure 3.10 shows a SEM image of the specimen austenitized at 1000 °C and isothermally held at 520 °C for 88,200 s followed by water-quenching. “F” and “M” (as marked in the images) indicate ferrite and martensite, respectively. The static ferrite exhibits mostly polygonal morphologies with large grain sizes. The mean grain size of ferrite is 18.4 μm. The microstructure indicates that the ferrite experiences pronounced coarsening during static transformation.

Figure 3.11 shows SEM images of the specimens austenitized at 1000 °C, deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C and then isothermally held at 520 °C for (a) 9 s, (b) 92 s, (c) 916 s and (d) 3600 s followed by water-quenching (route-ST2). White broken lines in (b)-(d) indicate prior austenite grain boundaries (PAGBs), which is the case

for all SEM images in this chapter hereafter. In the specimen held at 520 °C for 9 s after deformation (**Fig. 3.11(a)**), a small amount of fine and nearly equiaxed ferrite grains are mainly formed along PAGBs. After holding at 520 °C for 92 s, the microstructure (**Fig. 3.11(b)**) reveals fine and equiaxed ferrite grains along PAGBs and some coarse ferrite grains growing into martensite (austenite till water-quenching). When the holding time increases to 916 s (**Fig. 3.11(c)**) and 3600 s (**Fig. 3.11(d)**), statically transformed ferrite exhibits similar morphologies, that is, fine equiaxed grains formed mainly along PAGBs and coarse grains growing into martensite (prior austenite).

Figure 3.12 shows SEM images of the specimens processed in the route-DT1. The specimens were austenitized at 1000 °C, isothermally held at 520 °C for different periods ((a) 500 s, (b) 3,600 s, (b) 12,600 s and (d) 19,800 s), and then deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C followed by water-quenching. In the specimen deformed after holding for 500 s (**Fig. 3.12(a)**), a small amount of fine and equiaxed ferrite grains are observed mainly along PAGBs. With increasing the holding time to 3,600 s (**Fig. 3.12(b)**) and 19,800 s (**Fig. 3.12(d)**), ferrite keeps fine equiaxed morphology (with grain size around 0.65 μm) and the volume fraction of ferrite slightly increases to 6.1% and 10.9%, respectively, indicating that these specimens are at the early stages of transformation. It is concluded that ferrite formed at early stages of dynamic transformation in the route-DT1 exhibits mostly fine and equiaxed morphology.

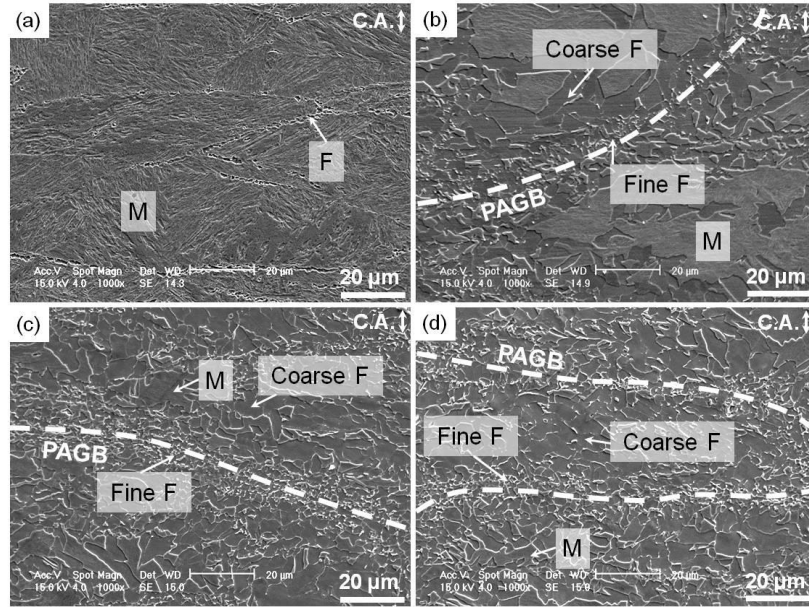


Fig. 3.11 SEM images of the specimens austenitized at 1000 °C, deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C and then isothermally held at 520 °C for (a) 9 s, (b) 92 s, (c) 916 s and (d) 3600 s followed by water-quenching (route-ST2). “F” and “M” indicate ferrite and martensite, respectively. White broken lines in (b)-(d) indicate prior austenite grain boundaries (PAGBs). Compression axis is along the vertical direction of the images.

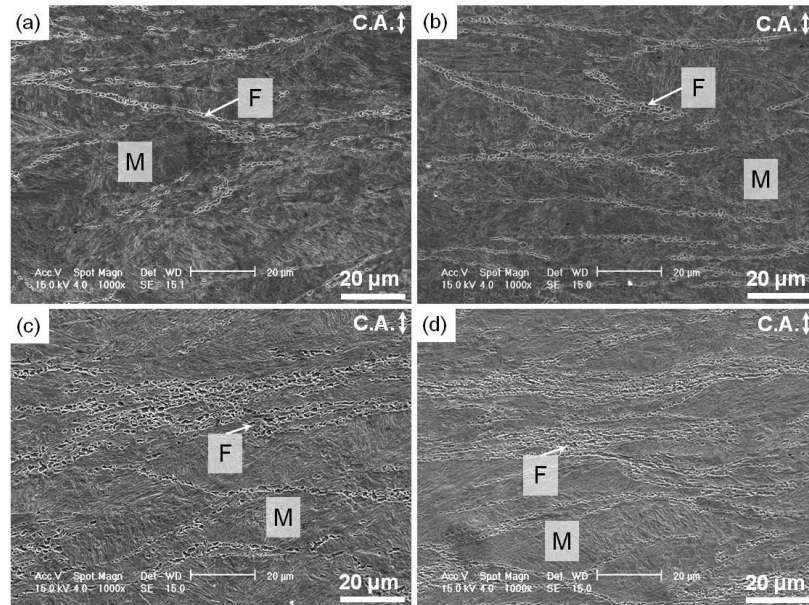


Fig. 3.12 SEM images of the specimens processed in the route-DT1. The specimens were austenitized at 1000 °C, isothermally held at 520 °C for different periods ((a) 500 s, (b) 3,600 s, (b) 12,600 s and (d) 19,800 s), and then deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C followed by water-quenching. “F” and “M” indicate ferrite and martensite, respectively. Compression axis is along the vertical direction of the images.

Figure 3.13 shows SEM images of the specimens processed in the route-DT2. The specimens were austenitized at 1000 °C, held at 520 °C for 60 s, and then deformed to a strain of 0.92 at a strain rate of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at 520 °C followed by water-quenching. Interestingly, the dynamically transformed ferrite shows different morphologies depending on the strain rate. At a higher strain rate of 10^{-1} s^{-1} , the microstructure (**Fig. 3.13(a)**) shows fine ferrite grains along PAGBs and relatively coarse grains growing into martensite (prior austenite). The grain size difference between two areas (i.e., near PAGBs and at interiors of martensite) is relatively small. At a lower strain rate of 10^{-2} s^{-1} (**Fig. 3.13(b)**), dynamically transformed ferrite has two typical morphologies. Fine and nearly equiaxed grains are mainly formed along the PAGBs, while the ferrite growing into martensite (prior austenite) mainly shows coarse morphologies elongated to the direction perpendicular to the compression axis (C.A.), indicating a pronounced grain growth. With decreasing the strain rate furthermore to 10^{-3} s^{-1} (**Fig. 3.13(c)**), fine-grain areas almost disappear, and most of the ferrite exhibit coarse and elongated morphologies.

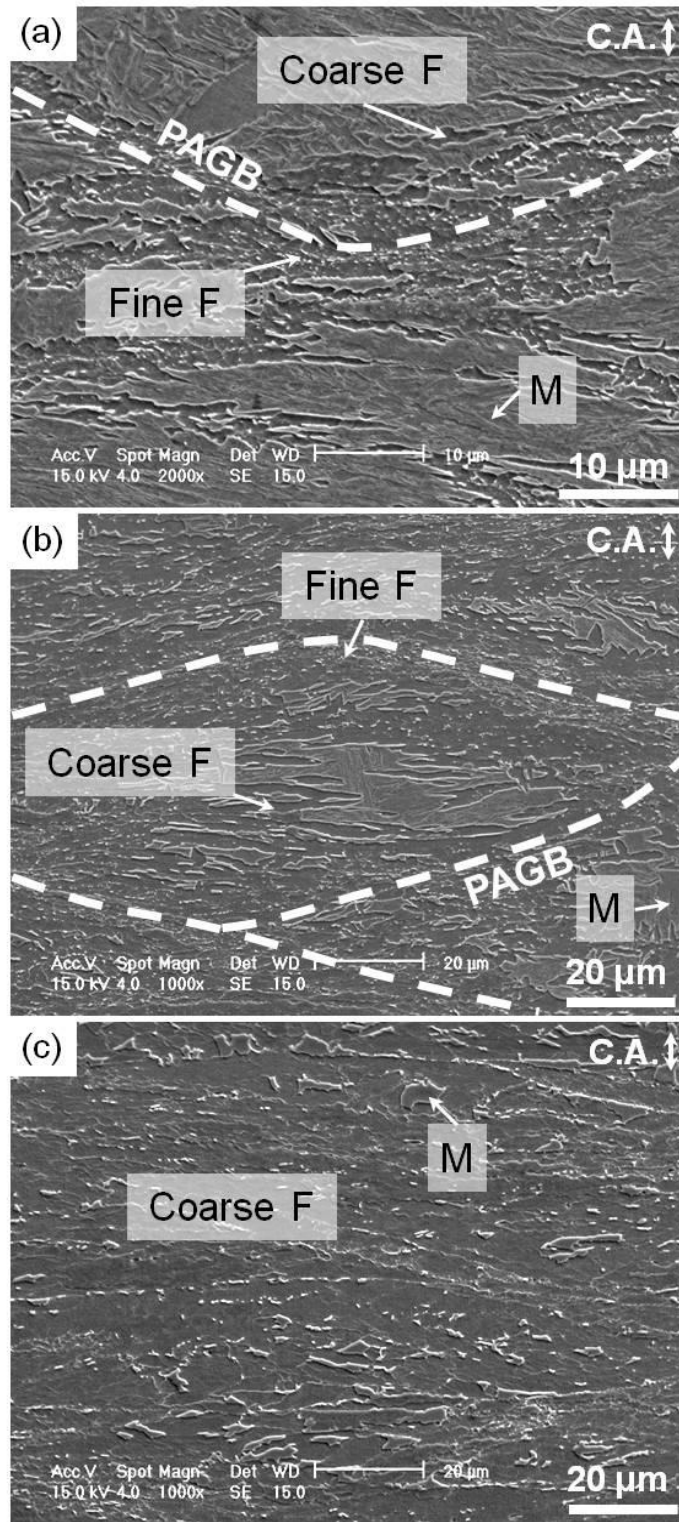


Fig. 3.13 SEM images of the specimens processed in the route-DT2. The specimens were austenitized at 1000 °C, held at 520 °C for 60 s, and then deformed to a strain of 0.92 at a strain rate of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at 520 °C followed by water-quenching. “F” and “M” indicate ferrite and martensite, respectively. White broken lines in (a) and (b) indicate prior austenite grain boundaries (PAGBs). Compression axis is along the vertical direction of the images.

The SEM images of statically or dynamically transformed ferrite have shown that the ferrite microstructure changes at different stages of transformation in ST from deformed austenite (e.g., **Fig. 3.11(a)** and **(b)**) or at different strain rates in DT (e.g., **Fig. 3.13**). In order to investigate the difference in ferrite microstructures between DT and ST (from deformed austenite), the specimens should be deformed under the same conditions. Therefore, the route-ST2 and the route-DT1 were specially selected, because the deformation temperature (520 °C), applied strain (0.92) and strain rate (10^0 s^{-1}) are the same in the two routes. The microstructures of ferrite obtained in the two routes were examined in the specimens processed under the conditions as following: in the route-ST2, the specimen was firstly deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C and then held at 520 °C for 12,600 s; in the route-DT1, the specimen was firstly held at 520 °C for 12,600 s and then deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C. The total holding periods in the two routes are same. The detailed information of ferrite microstructures in the two routes is presented in **Fig. 3.14** and **Fig. 3.15**.

Figure 3.14 shows (a) SEM image, (b) EBSD grain boundary map, (c) inverse pole figure (IPF) map and (d) IPF of the specimen austenitized at 1000 °C, held at 520 °C for 60 s, deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C and then isothermally held for 12,600 s followed by water-quenching (route-ST2). The colors in the IPF map (c) indicate the crystallographic orientation parallel to the compression axis, according to the key

stereographic triangle shown above the figures. The IPF shown in (d) reveals the relative intensity of crystallographic orientation parallel to the compression axis (compression texture). The SEM image (**Fig. 3.14(a)**) shows that the microstructure of statically transformed ferrite is composed of equiaxed ultrafine ferrite (UFF) grains and equiaxed coarse grains. The equiaxed UFF grains preferentially form in the vicinity of PAGBs. The equiaxed UFF grains and equiaxed coarse grains are surrounded mainly by HABs (**Fig. 3.14(b)**), although a small amount of LABs are observed in ferrite grains. The difference in grain size between UFF grains and coarse grains is not so large. The IPF map (**Fig. 3.14(c)**) and IPF (**Fig. 3.14(d)**) indicate that the statically transformed ferrite exhibits mostly random crystallographic orientations.

Figure 3.15 shows (a) SEM image, (b) grain boundary map, (c) IPF map and (d) IPF of the specimen austenitized at 1000 °C, isothermally held at 520 °C for 12,600 ks, and then deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C followed by water-quenching (route-DT1). The volume fraction of ferrite is around 8.4%, indicating that the specimen is at an early stage of transformation. The dynamically transformed ferrite is mainly formed in the vicinity of PAGBs (**Fig. 3.15(a)**), exhibiting equiaxed UFF grains mostly surrounded by HABs (**Fig. 3.15(b)**). The IPF map (**Fig. 3.15(c)**) and IPF (**Fig. 3.15(d)**) show that the ferrite grains have relatively random crystallographic orientations, which is quite similar to the statically transformed ferrite shown in **Fig. 3.14(c)** and **(d)**.

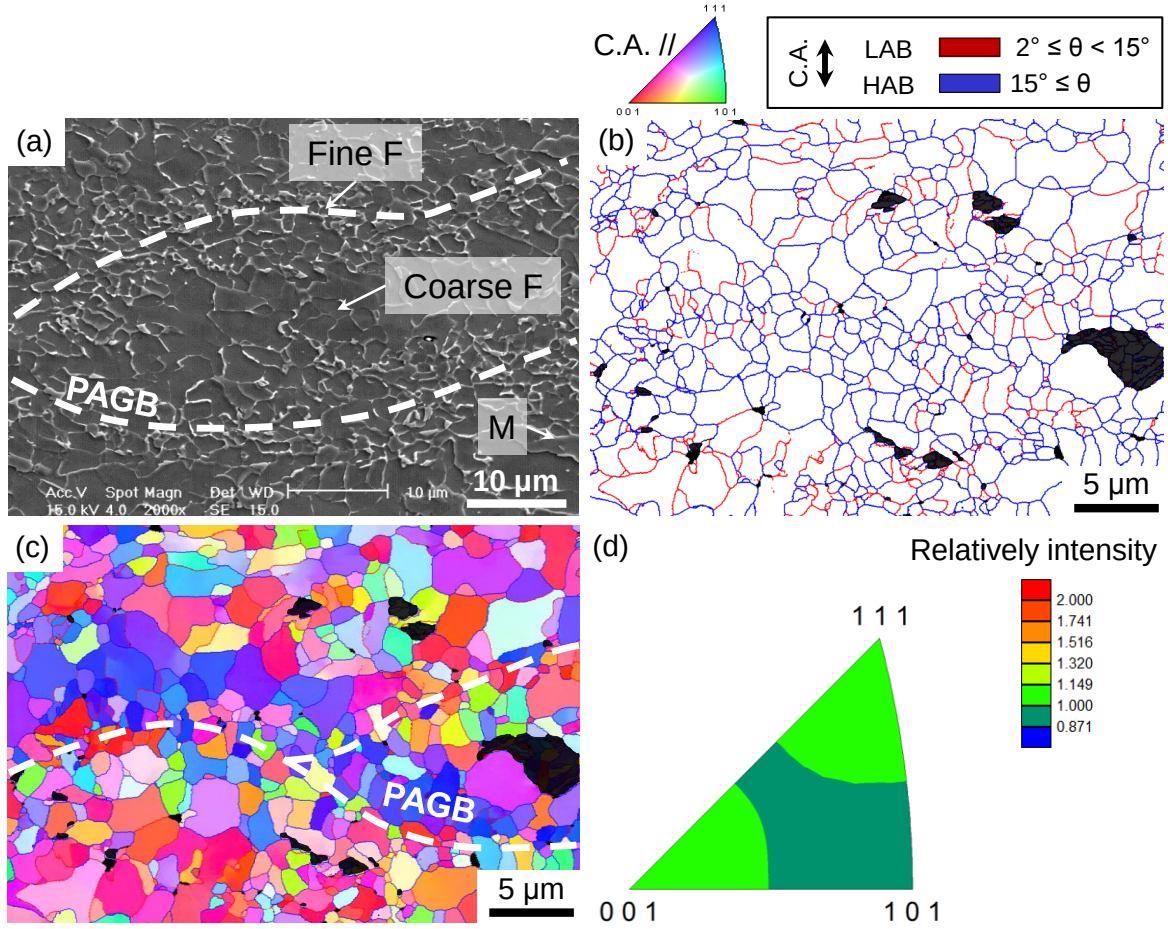


Fig. 3.14 (a) SEM image, (b) EBSD grain boundary map, (c) inverse pole figure (IPF) map and (d) IPF of the specimen austenitized at 1000 °C, held at 520 °C for 60 s, deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C and then isothermally held for 12,600 s followed by water-quenching (route-ST2). “F” and “M” in (a) indicate ferrite and martensite, respectively. Non-ferrite phases (i.e. martensite and/or retained austenite) in (b) and (c) are painted in black. In (b, c), low-angle boundaries (LABs) with misorientation of $2\text{--}15^\circ$ and high-angle boundaries (HABs) with misorientation above 15° are drawn in red and blue lines, respectively. The colors in the IPF map (c) indicate the crystallographic orientation parallel to the compression axis, according to the key stereographic triangle shown above the figures. The IPF shown in (d) reveals the relative intensity of crystallographic orientation parallel to the compression axis (compression texture). Compression axis is along the vertical direction of the images.

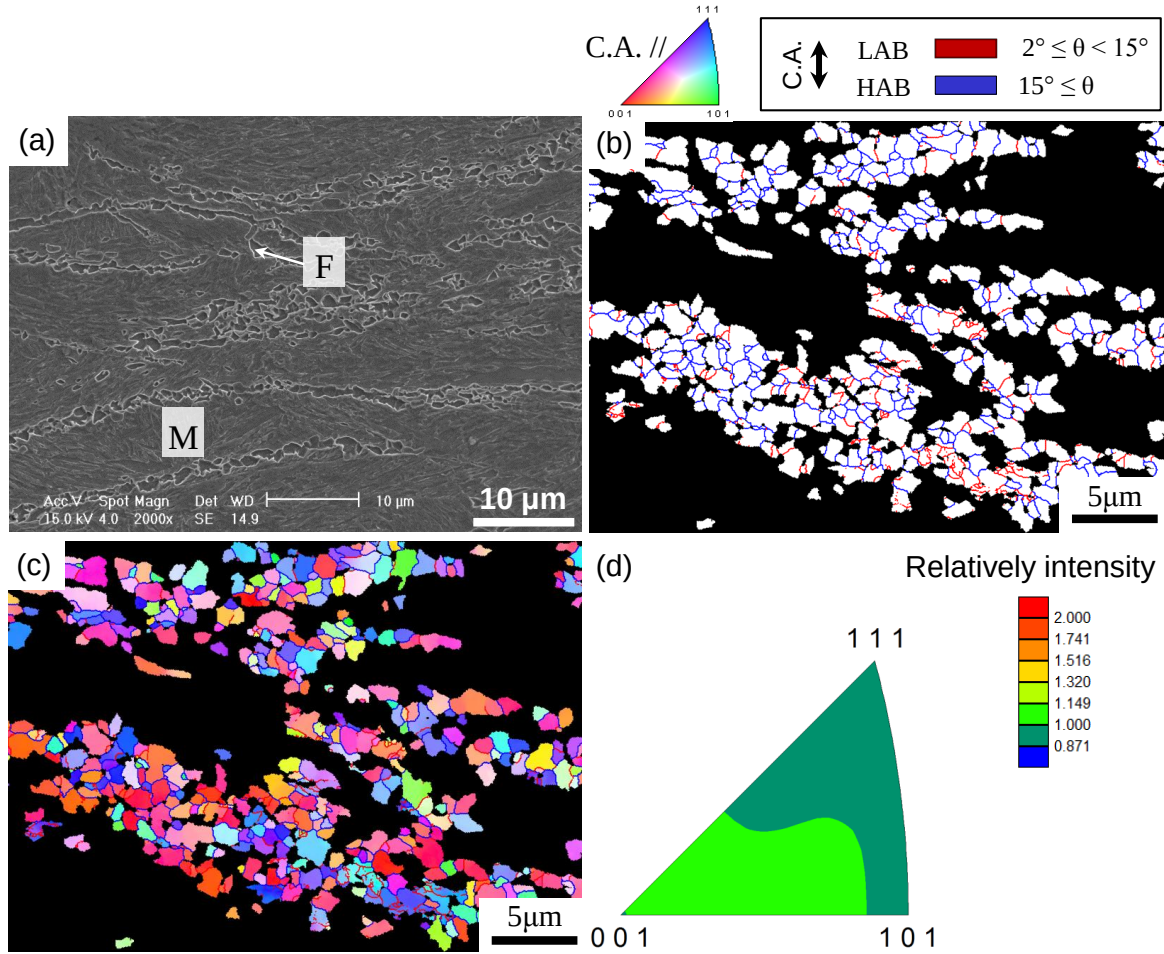


Fig. 3.15 (a) SEM image, (b) EBSD grain boundary map, (c) IPF map and (d) IPF of the specimen austenitized at 1000 °C, isothermally held at 520 °C for 12,600 ks, and then deformed to a strain of 0.92 at a strain rate of 10^0 s^{-1} at 520 °C followed by water-quenching (route-DT1). “F” and “M” in (a) indicate ferrite and martensite, respectively. Non-ferrite phases (i.e. martensite and/or retained austenite) in (b) and (c) are painted in black. In (b, c), low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. The colors in the IPF map (c) indicate the crystallographic orientation parallel to the compression axis, according to the key stereographic triangle shown above the figures. The IPF shown in (d) reveals the relative intensity of crystallographic orientation parallel to the compression axis (compression texture). Compression axis is along the vertical direction of the images.

It should be noted that the volume fraction of statically transformed ferrite obtained in the route-ST2 is 92% (**Fig. 3.14(a)**), while that of the dynamically transformed ferrite in the route-DT1 is only 8.4% (**Fig. 3.15(a)**). The small fraction of ferrite shows a morphology at an early stage of transformation, but cannot fully exhibit the microstructural features of dynamically transformed ferrite. In order to increase the volume fraction of ferrite obtained in the route-DT1, higher strain is required. Since high strains (e.g., logarithmic equivalent strain above 3) cannot be applied in the current compression equipment, the experiments were conducted through torsion deformation under the same deformation condition (i.e., same temperature, strain rate and austenite grain size). In the torsion deformation simulator, a bar specimen can be subjected to torsion deformation by rotating one end of the specimen, as is schematically shown in **Fig. 3.16**. The strain rate and deformation temperature could be well controlled through this process. The torsion specimens 4 mm in gage length and 8 mm in diameter were austenitized at 1000 °C for 300 s to obtain an austenite grain size of 125 μm, cooled to 520 °C, isothermally held for 12,600 s, and then deformed in torsion to an equivalent strain of 0.93 and 3.2 at a strain rate of 10⁰ s⁻¹ at the radial position of 0.9R (R: the radius of the gage part) followed by water-quenching. The equivalent strain ($\epsilon_{eq.}$) in torsion deformation was calculated using an equation shown below [34]:

$$\epsilon_{eq.} = \frac{2\pi RN}{L\sqrt{3}} \quad (3.7)$$

Where R is the radius, L is the gauge length, N is the number of rotation, π is a constant.

Microstructures on the longitudinal section parallel to the torsion axis at 0.9R position were characterized by electron back-scattering diffraction (EBSD) analysis performed in a field-emission type scanning electron microscope (FE-SEM, FEI XL30S FEG).

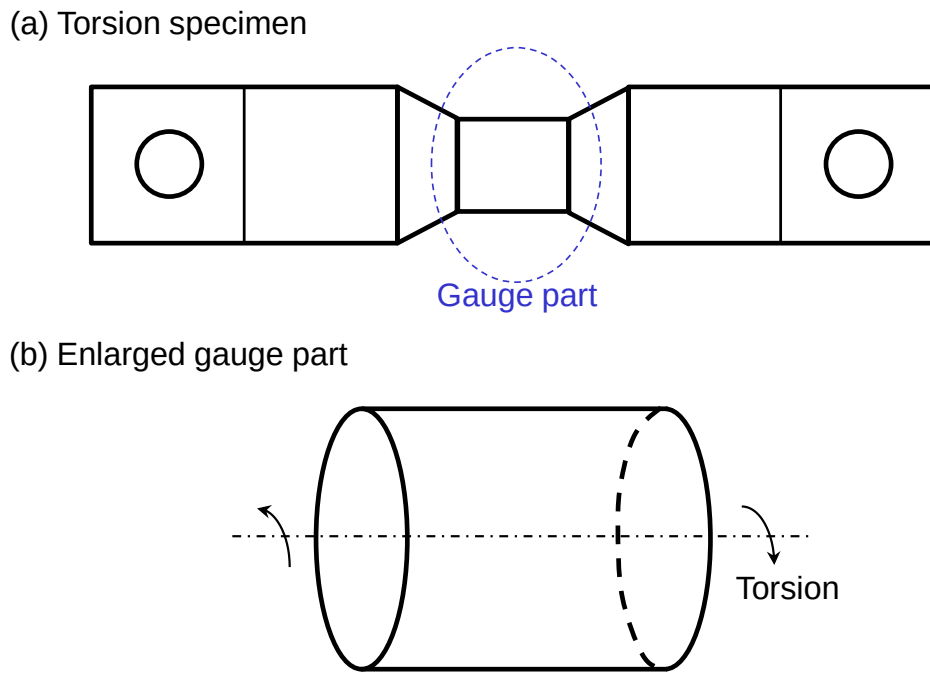


Fig. 3.16 Schematic illustration of the torsion deformation.

Microstructures of the specimens deformed by torsion are shown in **Fig. 3.17** and **Fig. 3.18**. **Figure 3.17** shows IPF maps (a, c) and corresponding IPFs (b, d) of the specimen austenitized at 1000 °C, isothermally held at 520 °C for 12,600 s, and then deformed in torsion to a strain of 0.93 at a strain rate of 10^0 s^{-1} followed by water-quenching (corresponding to the route-DT1). The colors in the IPF map (a) and (c) indicate the crystallographic orientation parallel to the shear direction (SD) and axis direction (AD), respectively. The key stereographic triangle corresponding to the colors is shown above **Fig. 3.17(a)**. The IPFs

shown in (b) and (d) reveal the relative intensity of crystallographic orientation parallel to the shear direction (SD) and axis direction (AD), respectively. The volume fraction of ferrite in this specimen is around 10%, indicating that the specimen is at an early stage of transformation. The volume fraction of ferrite obtained by torsion deformation (10%) is quite similar to that obtained by compression (8.4%) deformation under the similar equivalent strain (0.93 in torsion and 0.92 in compression). The ferrite mainly formed along PAGBs and consists of fine and equiaxed grains surrounded by HABs. Ferrite grains show nearly random orientation distribution, although a weak $\langle 001 \rangle$ texture parallel to the shear direction (SD) is observed in **Fig. 3.17(b)**. It can be concluded that the dynamically transformed ferrite grains formed at the early stage of transformation exhibits fine equiaxed morphology having relatively random crystallographic orientations. Such features are similar to those of the ferrite obtained by compression deformation (**Fig. 3.15**). Therefore, it is reasonable to examine the microstructural features of ferrite by torsion deformation in the route-DT1.

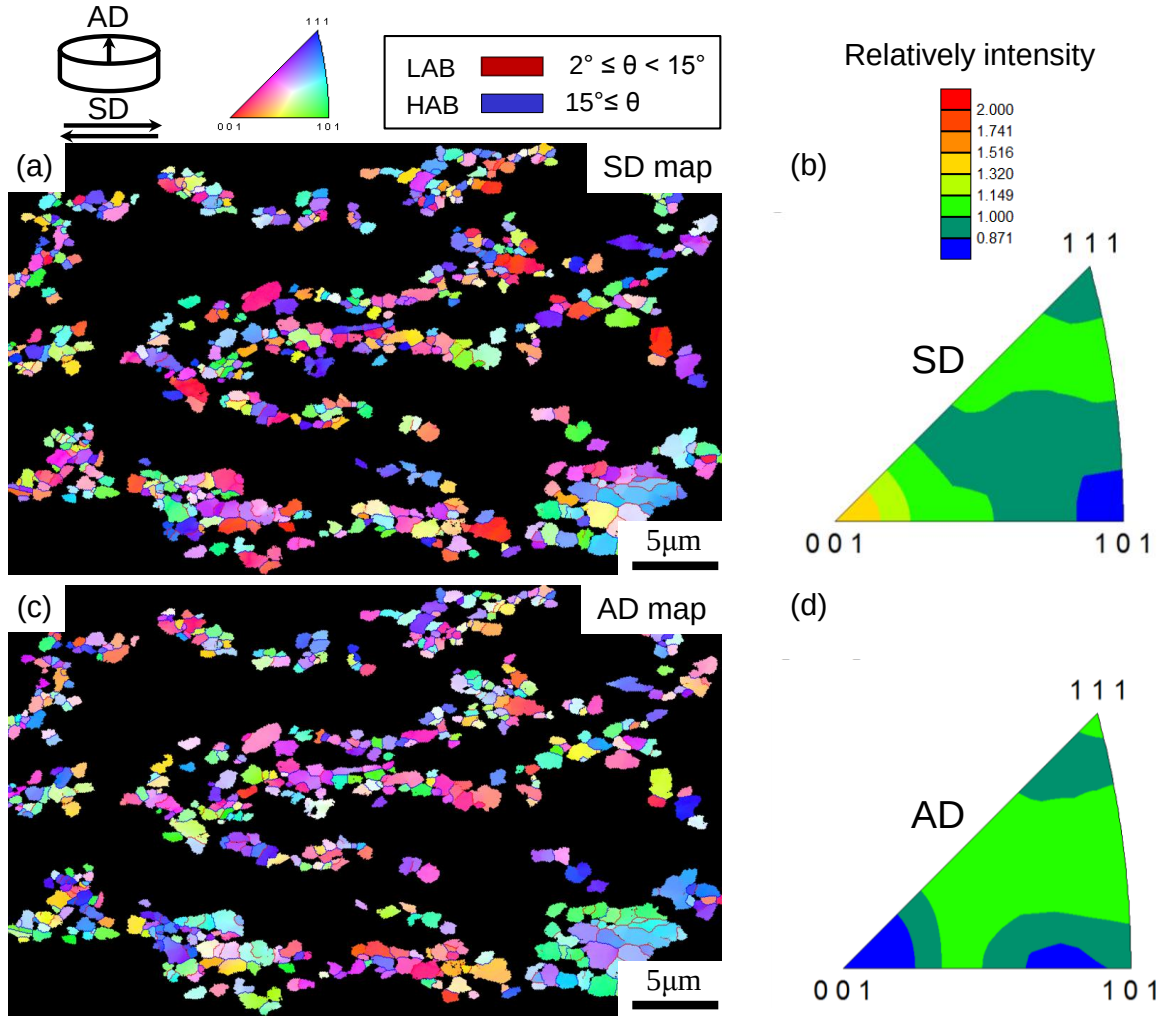


Fig. 3.17 IPF maps (a, c) and corresponding IPFs (b, d) of the specimen austenitized at 1000 °C, isothermally held at 520 °C for 12,600 s, and then deformed in torsion to a strain of 0.93 at a strain rate of 10^0 s^{-1} followed by water-quenching (corresponding to the route-DT1). Non-ferrite phases (i.e. martensite and/or retained austenite) in (a) and (c) are painted in black. The colors in the IPF map (a) and (c) indicate the crystallographic orientation parallel to the shear direction (SD) and axis direction (AD), respectively. The key stereographic triangle corresponding to the colors is shown above (a). In (a, c), low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. The IPFs shown in (b) and (d) reveal the relative intensity of crystallographic orientation parallel to the shear direction (SD) and axis direction (AD), respectively. Shear direction is along the horizontal direction of the maps.

Figure 3.18 shows a SEM image, and EBSD grain boundary maps (b, c) of the specimen austenitized at 1000 °C, isothermally held at 520 °C for 12,600 s, and then deformed in torsion to a equivalent strain of 3.2 at a strain rate of 10^0 s^{-1} followed by water-quenching (corresponding to the route-DT1, up to large strain (3.2)). In the SEM image (**Fig. 3.18(a)**), a white broken line marks a PAGB. “F” and “M” indicate ferrite (dark area) and martensite (light area), respectively. The dynamically transformed ferrite is mainly formed along PAGBs. The volume fraction of ferrite is 36%. The area for EBSD analysis is marked by a yellow rectangle in the SEM image. **Figure 3.18(b)** shows the grain boundary map corresponding to the area marked in **Fig. 3.18(a)**. The dynamically transformed ferrite is composed of equiaxed grains surrounded by HABs (as marked by green arrows) and elongated grains including many subgrains surrounded by LABs (as marked by black ellipse). The subgrains in the elongated grains indicate that they are deformed structures. The equiaxed grains formed by DT could be observed in the vicinity of PAGBs (**Fig. 3.18(c)**), but they are more preferentially formed near the vicinity of grain boundaries of the elongated coarse grains, not specifically along PAGBs. The equiaxed grains surrounded by HABs are highlighted in green color in **Fig. 3.18(c)**. It becomes clear that most of the equiaxed grains are formed along grains boundaries of elongated coarse ferrite. The thickness of the equiaxed grain regions is much thicker than that in ST.

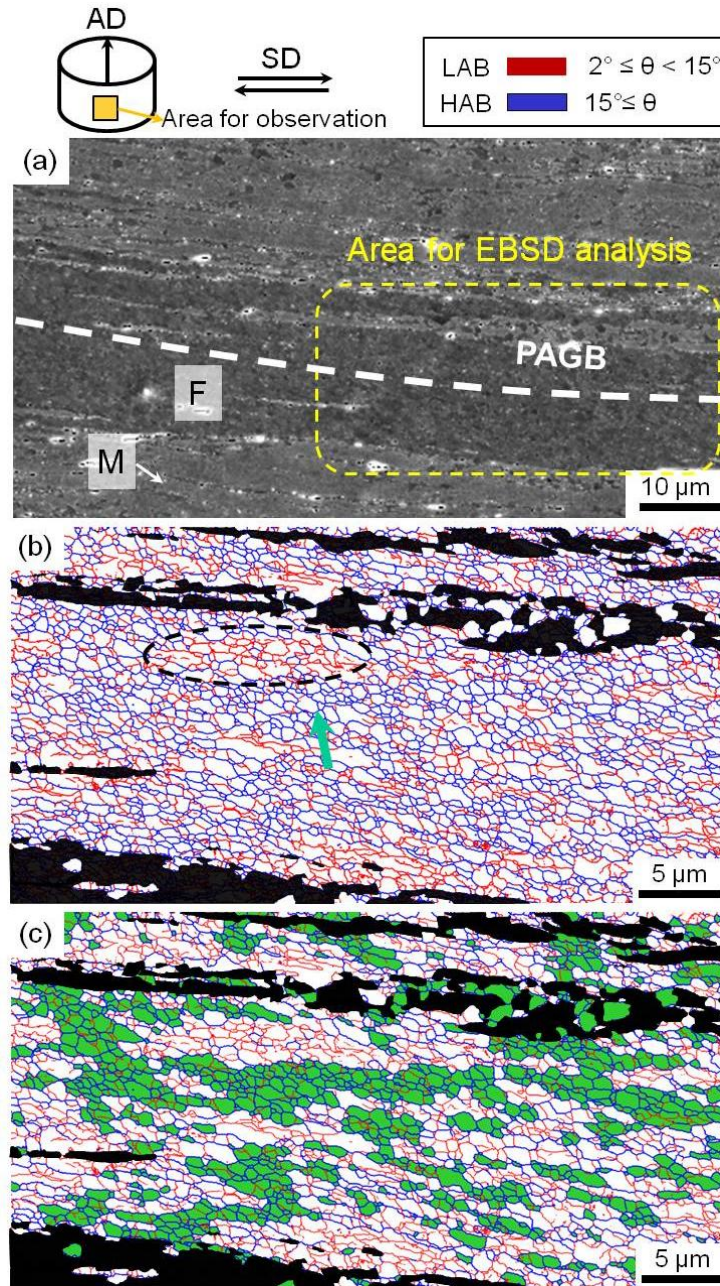


Fig. 3.18 SEM image (a) and EBSD grain boundary maps (b,c) of the specimen austenitized at 1000 °C, isothermally held at 520 °C for 12,600 s, and then deformed in torsion to a equivalent strain of 3.2 at a strain rate of 10^0 s^{-1} followed by water-quenching (corresponding to the route-DT1). White broken lines indicate prior austenite grain boundaries (PAGBs). “F” and “M” indicate ferrite and martensite, respectively. Yellow rectangle in (a) marks the area for EBSD analysis in (b). Green arrow and black ellipse in (b) mark equiaxed and elongated grains, respectively. Equiaxed grains are highlighted in green color in (c). In (b, c), non-ferrite phases (i.e. martensite and/or retained austenite) are painted in black. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. Shear direction is along the horizontal direction of the maps.

Other features of the two kinds of DT ferrite grains (i.e., equiaxed grains and elongated grains shown in **Fig. 3.18(c)**) are shown in **Fig. 3.19**. **Figure 3.19** reveals distributions of grain size (a, c) and misorientation angle (b, d) of the equiaxed grains (a, b) and the elongated grains (c, d). The grain size of equiaxed ferrite (**Fig. 3.19(a)**) is very fine and shows a narrow distribution (from 0.12 μm to 1.6 μm). The mean grain size of the equiaxed grains is 0.6 μm . The equiaxed grains are mostly surrounded by HABs (**Fig. 3.19(b)**). On the other hand, the elongated grains have a broad distribution of the grain sizes ranging from 0.24 μm to 3.85 μm (**Fig. 3.19(c)**). The mean grain size of the coarse grains is 1.4 μm . The fraction of LABs in the elongated grains is very high (66%, **Fig. 3.19(d)**). These results confirm that the elongated coarse grains have been deformed during their growth after nucleation.

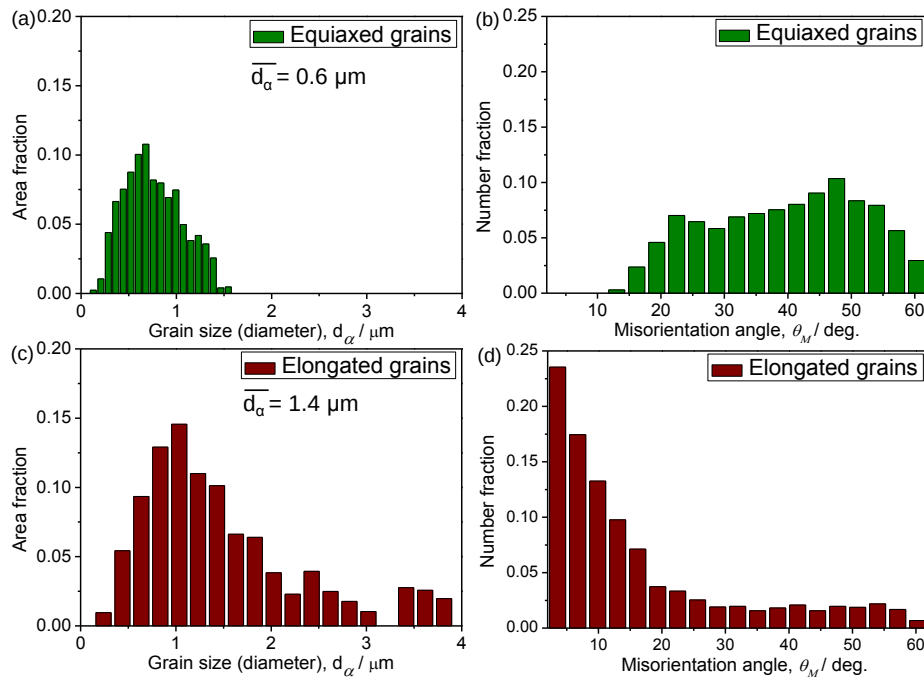


Fig. 3.19 Distributions of grain size (a, c) and misorientation angle (b, d) of the equiaxed grains (a, b) and the elongated grains (c, d) corresponding to **Fig. 3.18(c)**.

Since the elongated coarse grains exhibit more deformed structures than the equiaxed UFF grains (**Fig. 3.19(b)** and **(d)**), the difference in their crystallographic orientations is investigated. **Figure 3.20** shows IPF maps (a, c) and corresponding IPFs (b, d) of the elongated coarse grains (a, b) and equiaxed UFF grains (c, d) in **Fig. 3.18(c)**. The colors in the IPF map (a) and (c) indicate the crystallographic orientation parallel to the shear direction (SD). The key stereographic triangle corresponding to the colors is shown above (a). The IPFs shown in (b) and (d) reveal the relative intensity of crystallographic orientation parallel to the shear direction (SD). The IPF of the elongated coarse grains (**Fig. 3.20(b)**) indicates a strong crystallographic orientation of $\langle 001 \rangle$ parallel to the shear direction ($\langle 001 \rangle // \text{SD}$), which is a typical shear texture in BCC crystals [35]. On the other hand, the IPF of the equiaxed UFF grains (**Fig. 3.20(d)**) shows that $\langle 001 \rangle // \text{SD}$ texture is weak but $\langle 111 \rangle // \text{SD}$ texture is strong. It can be concluded that equiaxed UFF grains (**Fig. 3.20(c)**) exhibit quite different crystallographic texture from elongated coarse grains (**Fig. 3.20(a)**).

Figure 3.21 shows IPF maps (a, c) and corresponding IPFs (b, d) of the elongated coarse grains (a, b) and equiaxed UFF grains (c, d) in **Fig. 3.18(c)**. The colors in the IPF map (b) and (d) indicate the crystallographic orientation parallel to the axis direction (AD). The key stereographic triangle corresponding to the colors is shown above (a). The IPFs shown in (b) and (d) reveal the relative intensity of crystallographic orientation parallel to the axis direction (AD). The IPF of the elongated coarse grains (**Fig. 3.21(b)**) indicates a strong

texture around $\langle 212 \rangle // AD$. On the other hand, the IPF of the equiaxed UFF grains (**Fig. 3.21(d)**) shows more random orientation distribution than the coarse elongated grains, although a weak concentration around $\langle 212 \rangle // AD$ is observed.

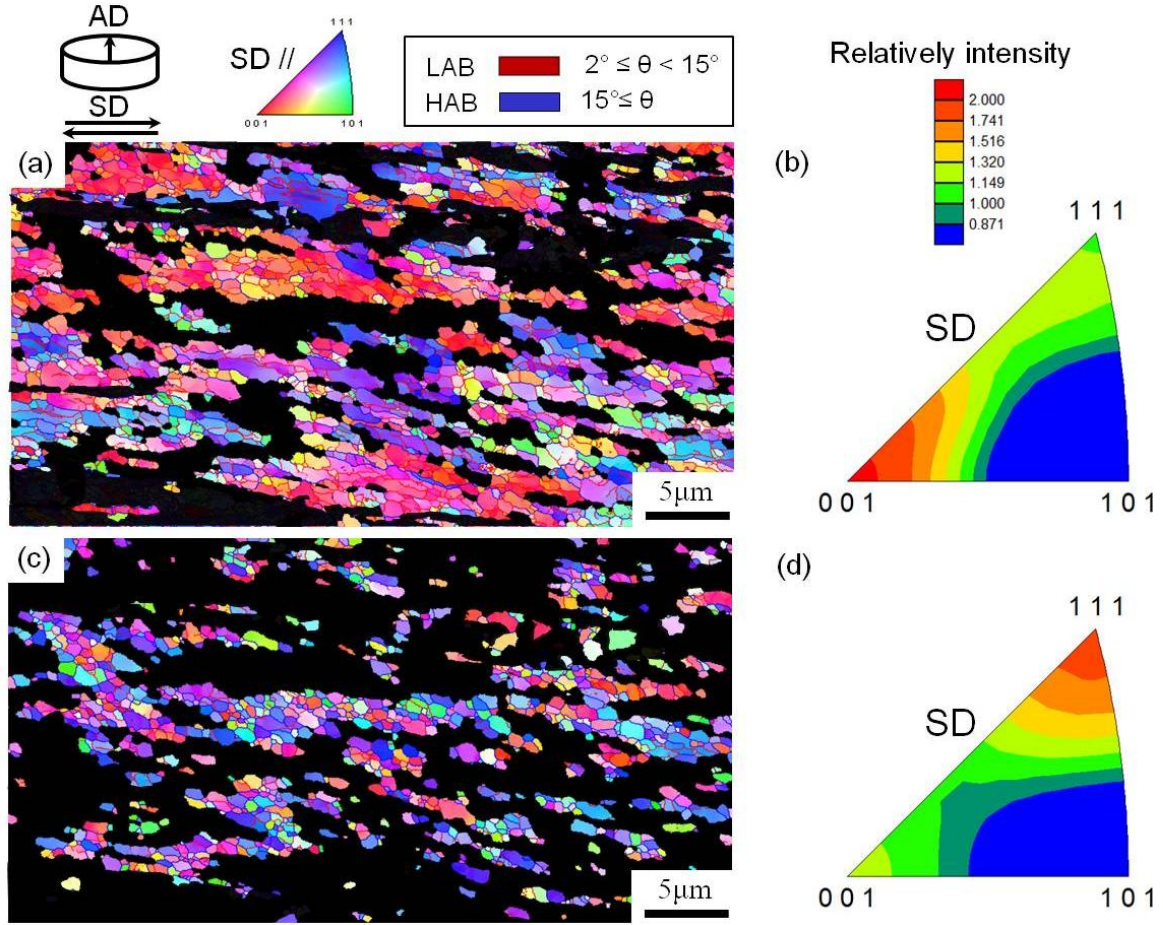


Fig. 3.20 IPF maps (a, c) and corresponding IPFs (b, d) of the elongated coarse grains (a, b) and equiaxed UFF grains (c, d) in **Fig. 3.18(c)**. The colors in the IPF map (a) and (c) indicate the crystallographic orientation parallel to the shear direction (SD). The key stereographic triangle corresponding to the colors is shown above (a). In (a, c), low-angle boundaries (LABs) with misorientation of $2\text{--}15^\circ$ and high-angle boundaries (HABs) with misorientation above 15° are drawn in red and blue lines, respectively. The IPFs shown in (b) and (d) reveal the relative intensity of crystallographic orientation parallel to the shear direction (SD). Shear direction is along the horizontal direction of the maps.

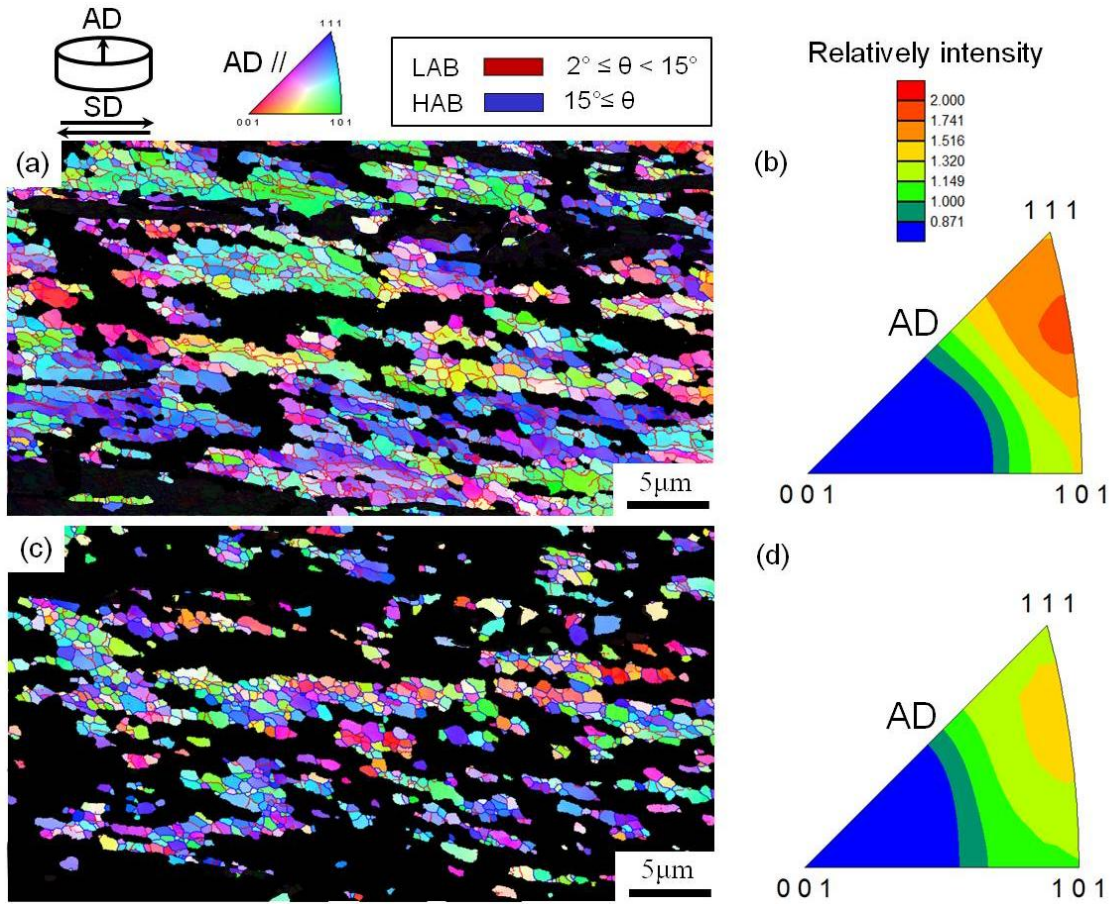


Fig. 3.21 IPF maps (a, c) and corresponding IPFs (b, d) of the elongated coarse grains (a, b) and equiaxed UFF grains (c, d) in **Fig. 3.18(c)**. The colors in the IPF map (a) and (c) indicate the crystallographic orientation parallel to the axis direction (AD). The key stereographic triangle corresponding to the colors is shown above (a). In (a, c), low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. The IPFs shown in (b) and (d) reveal the relative intensity of crystallographic orientation parallel to the axis direction (AD). Shear direction is along the horizontal direction of the maps.

Figure 3.15 – Fig. 3.21 have shown the microstructural features of dynamically transformed ferrite obtained in the route-DT1. Compared to the ferrite statically transformed from deformed austenite in the route-ST2, the dynamically transformed ferrite shows quite different microstructures and textures. The differences in the ferrite microstructures are summarized into three aspects described below:

1) The morphology of ferrite. The statically transformed ferrite is composed of equiaxed UFF grains and equiaxed coarse grains mainly surrounded by HABs (**Fig. 3.14(b)**). Only a small amount of LABs could be observed in ferrite grains. The dynamically transformed ferrite is composed of equiaxed UFF grains and elongated coarse grains (**Fig. 3.18(c)**). The equiaxed UFF grains are mostly surrounded by HABs, although a very small amount of LABs could be observed in these grains. A number of subgrains surrounded by LABs are formed in the elongated coarse grains (**Fig. 3.18(c)**), which indicates that the dynamically transformed ferrite is further deformed during their growth after the formation (nucleation) (**Fig. 3.19(c) and (d)**).

2) The distribution of equiaxed UFF grains. The equiaxed UFF grains formed by ST are mainly distributed along PAGBs (**Fig. 3.14(a) and (c)**). It is believed that the formation of equiaxed UFF by ST is mainly due to the impingement between the ferrite grains at the early stage of the transformation [13, 36]. Without sufficient impingement effect, the ferrite grains grow into the interior of martensite (prior austenite) and become coarse (as shown in **Fig. 3.14(a) and (c)**). The equiaxed UFF grains formed by DT could be observed in the vicinity of PAGBs (**Fig. 3.18(c)**), but they are more preferentially formed near the vicinity of grain boundaries of the elongated coarse grains, not specifically along PAGBs. As a result, the thickness of the UFF regions is much thicker than that in ST.

3) The crystallographic orientation of ferrite. The statically transformed ferrite shows

relatively random crystallographic orientations (**Fig. 3.14(c)** and **(d)**) under compression deformation. Basically the crystallographic textures of ferrite obtained in compression and torsion deformations cannot be directly compared. However, the dynamically transformed ferrite still shows some new features of crystallographic orientations. At a strain of 0.93 (in torsion), the ferrite formed at the early stage of transformation (**Fig. 3.17(a)** and **(c)**) mainly shows fine and equiaxed morphology. The fine equiaxed grains exhibit a weak $\langle 001 \rangle // SD$ texture (**Fig. 3.17(b)**) and mostly random orientation distribution along AD (**Fig. 3.17(d)**). With increasing the strain to 3.2, the ferrite is composed of equiaxed UFF grains and elongated coarse grains (**Fig. 3.18(c)**). The elongated coarse grains show a strong crystallographic texture of $\langle 001 \rangle // SD$ (**Fig. 3.20(b)**) and $\langle 212 \rangle // AD$ (**Fig. 3.21(b)**). It is reasonable to consider that the fine equiaxed grains appeared along PAGBs at early stages of transformation (deformation) grow into untransformed austenite to form the coarse and elongated ferrite grains during subsequent deformation. In that case, the $\langle 001 \rangle // SD$ and $\langle 212 \rangle // AD$ are supposed to be a kind of deformation texture formed in torsion. Interestingly, the equiaxed UFF grains formed at a larger strain of 3.2 shows a strong $\langle 111 \rangle // SD$ texture (**Fig. 3.20(d)**) and weak $\langle 212 \rangle // AD$ texture (**Fig. 3.21(d)**). That is, the crystallographic feature of the equiaxed UFF grains (formed by DT) is not only different from the statically transformed ferrite (**Fig. 3.14(d)**), but also quite different from the elongated coarse grains (**Fig. 3.20(b)** and **Fig. 3.21(b)**) and the fine equiaxed grains (**Fig.**

3.17 (b) and (d)) dynamically transformed at early stages of transformation. It is suggested that the equiaxed UFF grains observed in the later stage of deformation (in the route-DT1) may be formed by a different process (mechanism).

Based on the above discussion, the difference between ST from deformed austenite and DT could be schematically illustrated in **Fig. 3.22**. In the case of ST from deformed austenite (**Fig. 3.22(1)**), austenite is firstly deformed, and then ferrite transformation occurs during isothermal holding. Equiaxed UFF grains preferentially form along austenite grain boundaries as well as some intragranular substructures (like microband, etc.) at the early stage of transformation (**Fig. 3.22(b)**). With increasing the holding time, some nucleated ferrite grains grow into austenite and exhibit coarse morphology (**Fig. 3.22(c)**). The ferrite initially nucleated near austenite grain boundaries keeps fine and equiaxed morphology due to the impingement effect between grains. Eventually, the statically transformed ferrite is composed of fine grains near austenite grain boundaries and coarse grains in the interior of austenite. In the case of DT (**Fig. 3.22(2)**), austenite is firstly held to consume the incubation period of ferrite transformation. Therefore, ferrite transformation could occur during the deformation of austenite. Equiaxed UFF grains preferentially form along austenite grain boundaries as well as some intragranular substructures (like microband, etc.) at the early stage of transformation (**Fig. 3.22(e)**), which is similar to ST (**Fig. 3.22(b)**). However, some initially nucleated grains grow into austenite grains and are deformed during

continuous deformation, exhibiting coarse and elongated morphology (**Fig. 3.22(f)**). Subgrains surrounded by low-angle boundaries are formed within the coarse elongated grains. The different feature of DT from ST is that, equiaxed UFF grains could be formed not only along austenite grain boundaries, but also along the grain boundaries of coarse elongated ferrite grains (**Fig. 3.22(f)**). And the thickness of UFF grains increases with increasing deformation. This illustration implies that the formation mechanism of UFF grains in DT is different from that in ST. The microstructural evolution of ferrite grains during deformation requires further investigation.

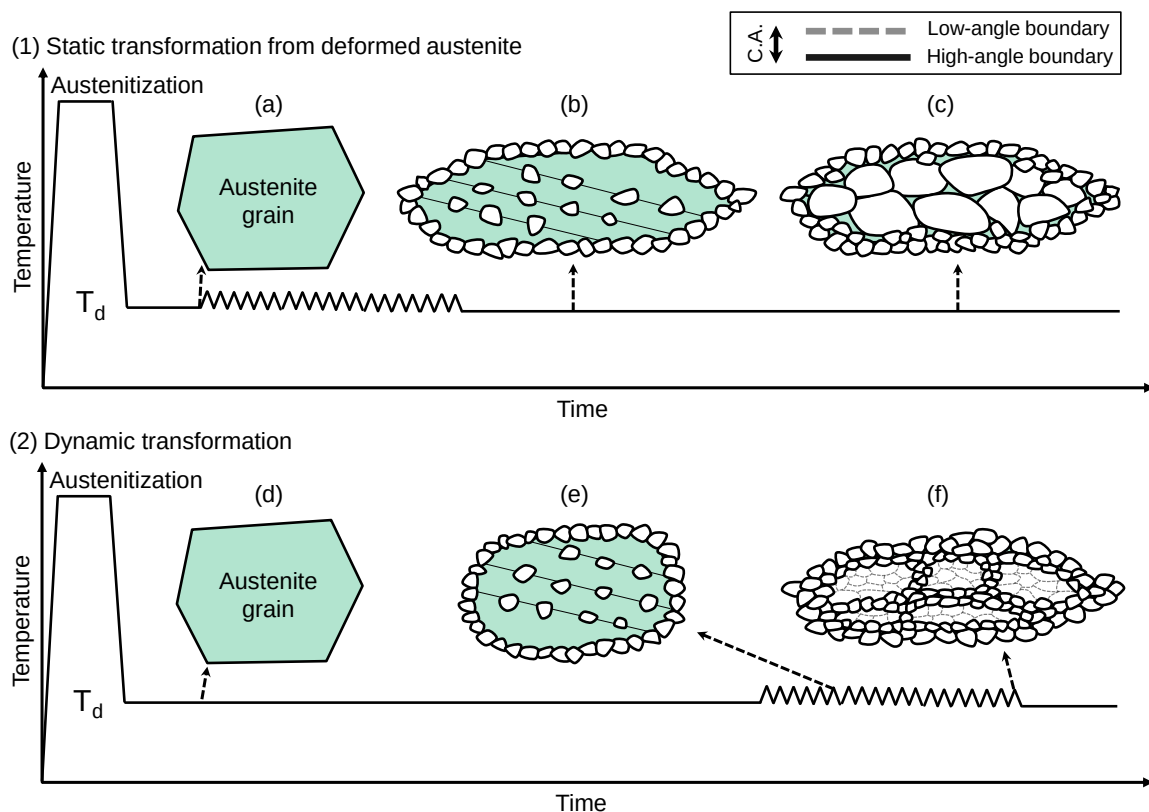


Fig. 3.22 Schematic illustration showing the difference between static transformation (ST) from deformed austenite (1) and dynamic transformation (2). The applied strain, strain rate, deformation temperature and austenite grain size are same in (1) and (2).

3.4 Conclusions

The differences in transformation kinetics and ferrite microstructures between static transformation (ST) and dynamic transformation (DT) were investigated through four different thermomechanical processing routes in this chapter. The main results are summarized as follows:

- 1) For ST, the transformation kinetics was greatly accelerated by the deformation of austenite. For DT, consuming incubation period before transformation could not effectively enhance ferrite formation, while decreasing strain rate (i.e., increasing deformation time) could greatly enhance ferrite formation.
- 2) Within the same transformation time, the volume fraction of dynamically transformed ferrite was higher than the statically transformed ferrite. The faster transformation kinetics (in DT) was considered to be due to the enhanced α/γ interface mobility during deformation.
- 3) The statically transformed ferrite was composed of equiaxed ultrafine ferrite (UFF) grains and equiaxed coarse grains. The equiaxed UFF grains were mainly formed in the vicinity of the prior austenite grain boundaries. Most of the statically transformed ferrite grains were surrounded by HABs, and exhibit relatively random crystallographic orientation distribution.
- 4) The dynamically transformed ferrite was composed of equiaxed UFF grains surrounded by HABs and elongated coarse grains. The elongated coarse grains contained a number of

subgrains surrounded by LABs, which indicates that they are deformed grains. The equiaxed UFF grains could be formed not only along PAGBs, but also along the grain boundaries of coarse elongated ferrite grains, and the number of UFF grains increases with increasing the strain, so that the thickness of the UFF regions is much thicker than that of the statically formed fine ferrite grains. The UFF grains show different crystallographic orientation (strong $\langle 111 \rangle // \text{SD}$ texture and weak $\langle 212 \rangle // \text{AD}$ texture) from the elongated coarse grains (strong $\langle 001 \rangle // \text{SD}$ texture and $\langle 212 \rangle // \text{AD}$ texture). The results suggest that the formation mechanism of the UFF grains in DT is not only different from that in ST, but also different from that of the elongated coarse grains formed in DT.

3.5 References

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Chapter 4: Mechanism of ultrafine ferrite (UFF) formation

4.1 Introduction

Dynamic transformation (DT), especially DT from austenite (γ ; FCC phase) to ferrite (α ; BCC phase) in steels has been extensively studied in the last 30 years [1-8]. Equiaxed ultrafine ferrite (UFF) grains (1-3 μm) could be obtained in the thermomechanical processes where DT occurs [9-13]. Some of the works suggest that plenty of lattice defects and deformation microstructures introduced into austenite by deformation could greatly increase the nucleation density for ferrite and lead to an early impingement between ferrite grains nucleated and grown, which is the main cause for the equiaxed UFF formation [14-16]. However, DT is different from static transformation from deformed austenite, in which ferrite grains are formed after deformation of austenite, so that the static ferrite should keep nearly equiaxed morphology [17-22]. In DT, on the other hand, ferrite grains are formed during deformation of austenite. They are expected to be elongated, because of the continuous deformation after nucleation. However, only equiaxed UFF grains are often observed in the microstructure after thermomechanical processes including DT [23-25]. Such a contradictory situation including the formation of equiaxed UFF has been confirmed in **Chapter 3** of the present study as well.

In order to understand how the equiaxed UFF grains form, we should pay an attention not only to the features of the deformed austenite, but also, and more importantly, to the behavior of transformed ferrite during subsequent deformation. From such a viewpoint, another possible mechanism for the formation of the equiaxed UFF grains is dynamic recrystallization (DRX). It has been suggested in some studies that after the ferrite transformation from austenite, ferrite grains experience DRX for forming the equiaxed UFF structures [26-28]. However, up to now, the actual role of DRX in the grain refinement of ferrite formed by DT is still unclear. A major difficulty to investigate the behavior of ferrite grains is that the transformation in commercial steels occurs in fast kinetics at relatively high temperatures, so that it is substantially not possible to track the change of microstructures during subsequent deformation and transformation.

For clarifying the formation mechanisms of UFF during the thermomechanical processes under which DT can happen, a 10Ni-0.1C steel was designed in this study to decrease the A_{e3} temperature and to slow down the ferrite transformation kinetics. The first section of this chapter is an attempt to carefully observe the evolution of ferrite grains during their first formation through DT as well as during subsequent deformation. A special attention is paid to understand the contribution of DRX to the equiaxed UFF formation. Considering this motivation, an investigation on DRX behavior of statically transformed ferrite (fully ferrite phase) is conducted in the second section of this chapter. It provides a possibility to

compare the DRX behaviors (if they really occur) between dynamically transformed ferrite and statically transformed ferrite under the same deformation condition. In the third section, the similarity and difference between the DRX of dynamically transformed ferrite and the DRX of statically transformed ferrite is compared. In the fourth section, the effects of several important thermomechanical parameters (e.g., austenite grain size and deformation temperature) on the DRX behaviors of dynamically transformed ferrite were particularly examined.

4.2 Experimental procedure

The material used in this study is the same alloy (Fe-10Ni-0.1C) as that used in **Chapter 2**, of which chemical composition and TTT diagram were shown in **Table 2.1** and **Fig. 2.3**, respectively. The as-received hot-rolled plate 15 mm thick was cold-rolled to a thickness of 11.2 mm with a reduction of 25%, and then homogenized at 1100 °C for 172,800 s in vacuum. Two procedures were designed for the sample preparation. In the first case, the homogenized plate was water-quenched after the homogenization at 1100 °C to have a martensite structure. In the other case, the homogenized plate was firstly water-quenched to obtain martensite, then austenitized again at 750 °C for 900 s or at 900 °C for 1,800 s, and slowly furnace-cooled to obtain a fully ferrite microstructure with a mean grain size of 6.7 μm and 17 μm , respectively. Cylindrical specimens with a height of 12 mm and a diameter of 8 mm were machined from the two kinds of plate and installed in a thermomechanical processing simulator (Thermecmastor-Z, Fuji Electronic Industrial Co. Ltd.) for conducting thermomechanical processes.

Figure 4.1 shows the thermomechanical processes using compression deformation of (a) austenite and (b) ferrite, respectively. In the case of deformation of austenite (named route-1 for short), the specimens were austenitized at 1200 °C for 300 s, at 1000 °C for 300 s or at 700 °C for 900 s to obtain different austenite grain sizes (d_γ) of around 450 μm , 125 μm or 10 μm , respectively. The austenitized specimens were cooled to the deformation

temperatures ranging from 600 °C to 440 °C at a rate of 30 °C s⁻¹ by nitrogen gas, held for 60 s to homogenize the temperature of the cylindrical specimens, and then uniaxially compressed to various strains ranging from 0.05 to 0.92 (5% to 75% reduction in height) at different strain rates ranging from 10⁻¹ s⁻¹ to 10⁻³ s⁻¹ followed by water-quenching for minimizing static change of microstructure during cooling. In the case of deformation of a fully ferrite structure for investigating fundamental DRX behaviors of ferrite (named route-2 for short), the specimens were heated from room temperature to the deformation temperatures from 480 °C to 560 °C at a rate of 10 °C s⁻¹, held for 60 s to homogenize the temperature, and then uniaxially compressed to various strains ranging from 0.16 to 1.39 at different strain rates ranging from 10⁻¹ s⁻¹ to 10⁻³ s⁻¹ followed by water-quenching. Mica and glass powder were used as the insulator and lubricant to minimize the friction between the contact surfaces of the anvils and the specimen during straining. The water injection was precisely controlled to minimize the time interval until the initiation of cooling after the deformation.

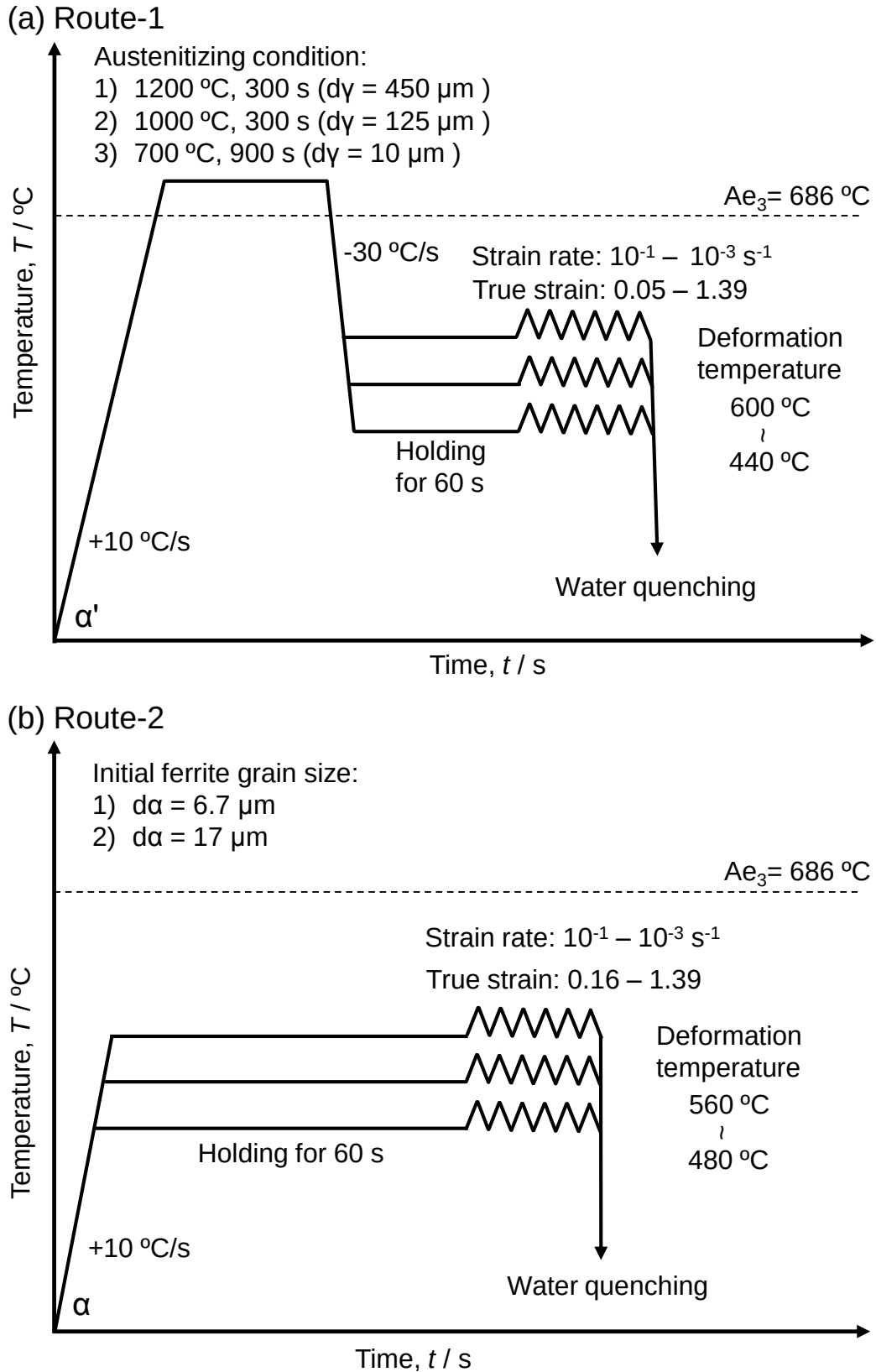


Fig. 4.1 Schematic illustrations showing the thermomechanical processes in compression deformation of (a) austenite and (b) ferrite. The processes (a) and (b) are hereafter called route-1 and route-2, respectively.

Microstructures at the center of the cylindrical specimens on the sections parallel to the compression axis were characterized by optical microscopy (OM), a field-emission type scanning electron microscope (FE-SEM, FEI XL30S FEG), electron back-scattering diffraction (EBSD) analysis performed in the FE-SEM and a transmission electron microscope (TEM, Philips CM200FEG). The specimen was mechanically polished and etched using a 3% nital solution for the OM observations. For the EBSD measurement, the specimens were mechanically polished and then electrically polished in a solution of 10% HClO_4 and 90% CH_3COOH at 20 °C. The EBSD measurement was performed using TSL OIM Data Collection system, and the obtained EBSD data were analyzed by TSL OIM Analysis software. For the TEM observations, thin-foil specimens were prepared by twin-jet electropolishing using the same solution as that used for EBSD.

4.3 Results and discussion

4.3.1 Occurrence of DRX of dynamically transformed ferrite

As mentioned in **Chapter 2**, the kinetics of static transformation is sufficiently slow at 520 °C (TTT diagram, **Fig. 2.3**), which provides a possibility to investigate the features of DT in detail. Therefore, most of the investigations were conducted at 520 °C in the present study. In this section, the microstructure evolution of ferrite grains is observed during their first formation through DT as well as during subsequent deformation. The formation mechanisms of equiaxed UFF grains (including the contribution of DRX) are particularly investigated.

Figure 4.2 shows true stress-true strain curves of the specimens with an austenite grain size of 125 µm deformed in the route-1 at different strain rates from 10^{-1} to 10^{-3} s^{-1} at 520 °C. The flow curves display a region of power-law type work hardening at early stages followed by a steady-state regime. The later stage does not perfectly show a steady-state but slight decrease in the flow stress (softening) is observed. According to the previous discussion in **Chapter 2**, this softening in the true stress-true strain curves indicates the occurrence of DT from austenite to ferrite.

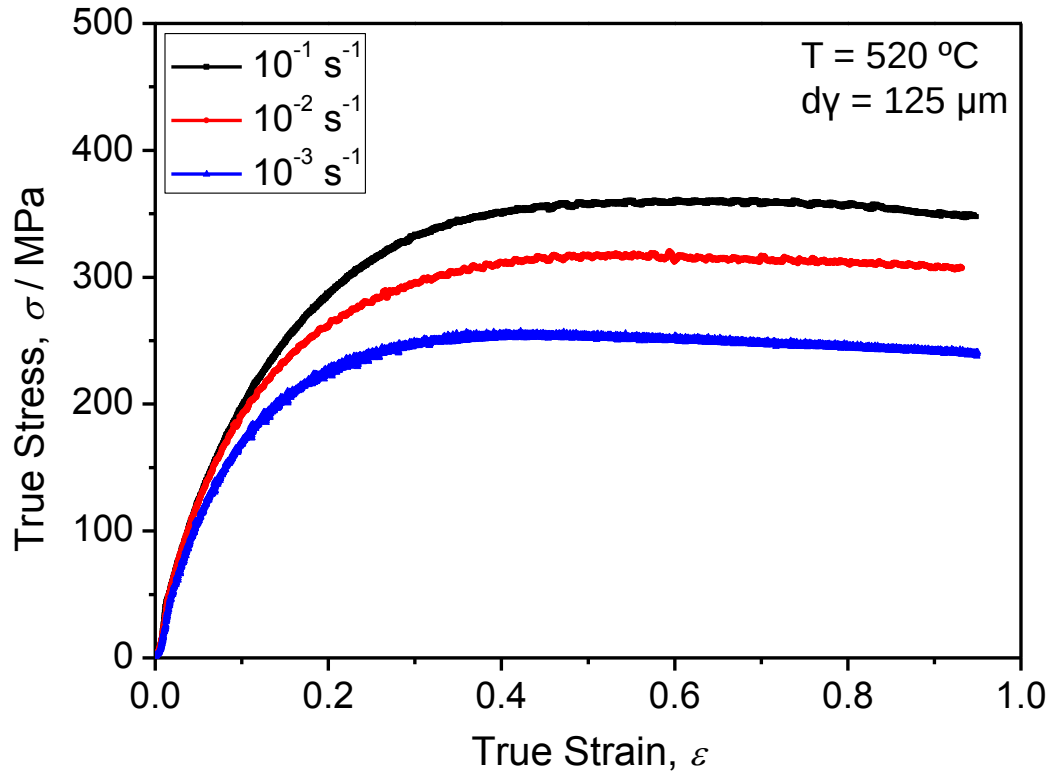


Fig. 4.2 True stress-true strain curves of the specimens with an austenite grain size of 125 μm deformed in the route-1 at strain rates ranging from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$.

In order to analyze the microstructure evolution during the whole deformation process, the microstructures of the specimens deformed to different strains and then water-quenched were observed in detail. A true stress-true strain curve of a specimen with an austenite grain size of 125 μm deformed in the route-1 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$ is shown in **Fig. 4.3**. The triangle symbols indicate the positions where the microstructures were observed using several specimens deformed under the same condition.

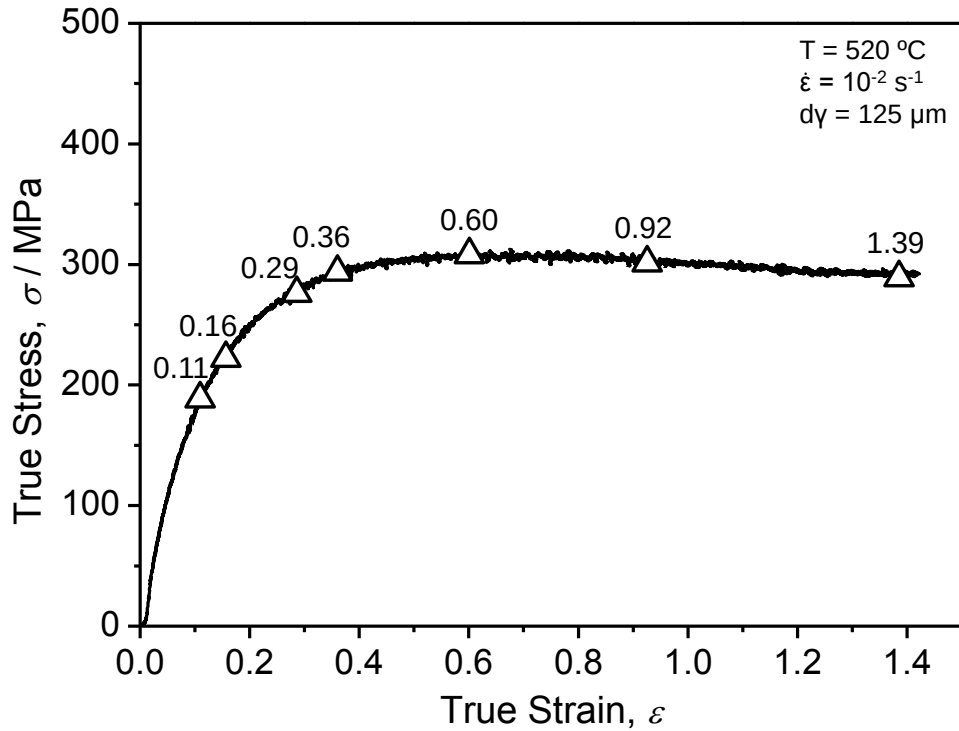


Fig. 4.3 True stress-true strain curve of the specimen with an austenite grain size of 125 μm deformed in the route-1 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. The triangle symbols indicate the positions where the microstructures were observed using several specimens deformed under the same condition.

Figure 4.4 shows OM images of the specimens corresponding to **Fig. 4.3**. The volume fraction of ferrite is shown on the top right corner of each OM image. “F” and “M” indicate ferrite and martensite, respectively. At a strain of 0.11 (**Fig. 4.4(a)**), a small amount of ferrite initially forms along the prior austenite grain boundaries (PAGBs). Since the isothermal holding time (60 s) plus deformation time (11 s) is much shorter than the incubation time for the onset of static transformation (12,600 s) at 520 $^{\circ}\text{C}$, the ferrite grains observed in these images are dynamically transformed. With increasing the strain to 0.29 (**Fig. 4.4(b)**), the ferrite grows into martensite (prior austenite). The volume fraction of

ferrite increases from 0.4% to 89.3% with increasing the strain from 0.11 to 1.39 (**Fig. 4.4(a)-(f)**).

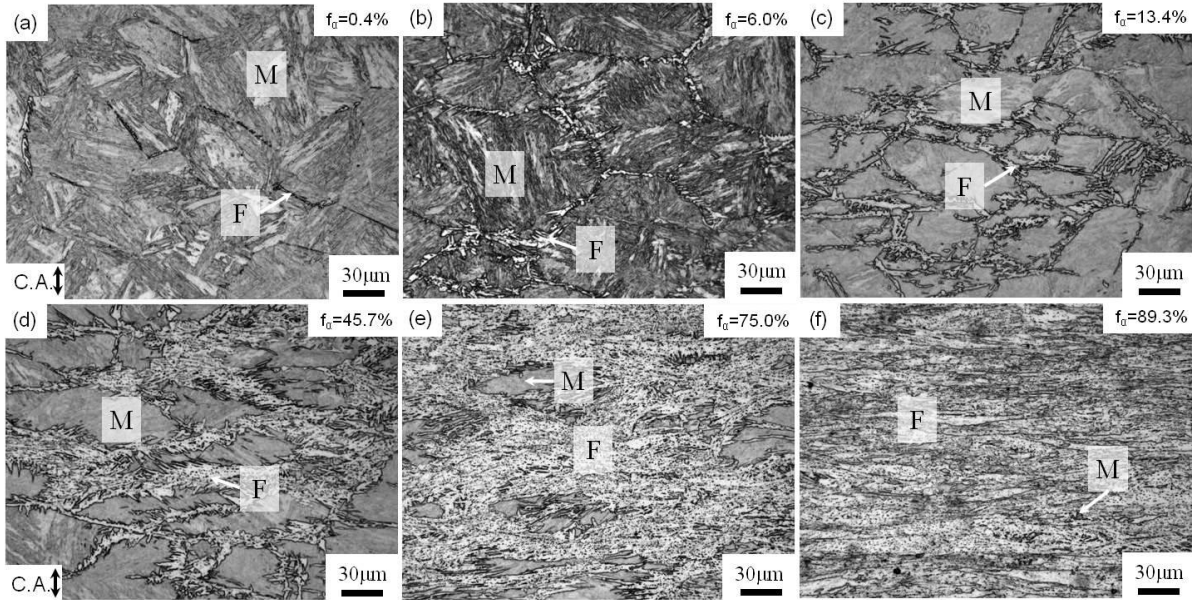


Fig. 4.4 OM images of the specimens with an austenite grain size of 125 μm deformed to various strains of (a) 0.11, (b) 0.29, (c) 0.36, (d) 0.60, (e) 0.92 and (f) 1.39 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. The strains correspond to the triangle symbols shown in Fig. 4.3. “F” and “M” indicate ferrite and martensite, respectively. The volume fraction of ferrite is shown on the top right corner of each OM image. Compression axis is along the vertical direction of the images.

Detailed microstructural features of dynamically transformed ferrite corresponding to **Fig. 4.4** are presented in **Fig. 4.5**. **Figure 4.5 (a)-(g)** shows grain average misorientation (GAM) maps obtained from EBSD measurements of the specimens. **Figure 4.5(h)** is an inverse pole figure (IPF) map corresponding to **Fig. 4.5(g)**. The GAM maps, which show average misorientation angle among all pairs of adjacent points in each grain, can be used to evaluate the degree of misorientation inside the grain. The range of the GAM value is from 0° to 2° ;

where a higher value indicates higher misorientation. The IPF map is used to show crystal orientations parallel to the compression axis. In order to depict the microstructure evolution of ferrite, non-ferrite phases (i.e., martensite and/or retained austenite in the present study) are painted in black, which is the case for all EBSD maps in this chapter hereafter. The methods for distinguishing ferrite from austenite and martensite were introduced in **Chapter 2**. White broken lines in **Fig. 4.5(a)-(d)** indicate prior austenite grain boundaries (PAGBs). Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively, which is the case for all EBSD maps in this chapter hereafter. At a strain of 0.11 (**Fig. 4.5(a)**), very fine and nearly equiaxed ferrite grains are formed along a PAGB. With increasing the strain to 0.16 (**Fig. 4.5(b)**), the ferrite grains grow into prior austenite grain and exhibit an irregular morphology. It is noteworthy that there are no HABs within the irregular shaped ferrite shown in **Fig. 4.5(b)**, which indicates that the ferrite grains formed along a PAGB have nearly the same orientation (i.e., the same variant). At a strain of 0.29 (**Fig. 4.5(c)**), in addition to continuous coarsening of ferrite, a small amount of LABs are observed within the coarse ferrite grains, indicating a deformed structure is introduced. The fraction of LABs increases with increasing the strain to 0.36 (**Fig. 4.5(d)**). With further straining, equiaxed UFF grains surrounded by HABs start to form along the grain boundaries of coarse ferrite (as marked by blue arrows in **Fig. 4.5(e)**). It should be noted that the fine grains formed at a

very early stage of transformation, or the fine grains formed near the ferrite and martensite (prior austenite) phase boundaries were not counted as the equiaxed UFF grains in this study. This excludes any possibility that the equiaxed UFF grains could be statically transformed from the deformed austenite during water-quenching. The detailed definition of the equiaxed UFF was introduced in **Chapter 2**. The fraction of the equiaxed UFF increases with increasing the strain to 0.92 (**Fig. 4.5(f)**) and 1.39 (**Fig. 4.5(g)**). At the strain of 1.39, the microstructure is composed of elongated coarse ferrite grains and equiaxed UFF grains (as marked by blue arrows). Subgrains surrounded by either complete LABs or partial LAB and HAB are formed within the elongated coarse grains. It clearly indicates that the ferrite grains experience further deformation after their nucleation. The defects introduced into ferrite by deformation would increase the misorientation within the grains. Here, the misorientation within each grain is evaluated by GAM value. As introduced before, a higher GAM value (dark red color) indicates higher misorientation. Qualitatively speaking, the elongated coarse grains exhibit high misorientation (dark red color) within the grains, indicating a more deformed structure. On the contrary, most of the equiaxed UFF grains show lower GAM values (light red color) than the elongated coarse grains. Quite a few subgrains (especially those surrounded by partial LAB and HAB, as marked by dotted circles in **Fig. 4.5(g)** and **(h)**) show similar crystallographic orientations to the elongated coarse grains (**Fig. 4.5(h)**), but have relatively low GAM values (light red color) like the equiaxed

UFF grains (**Fig. 4.5(g)**).

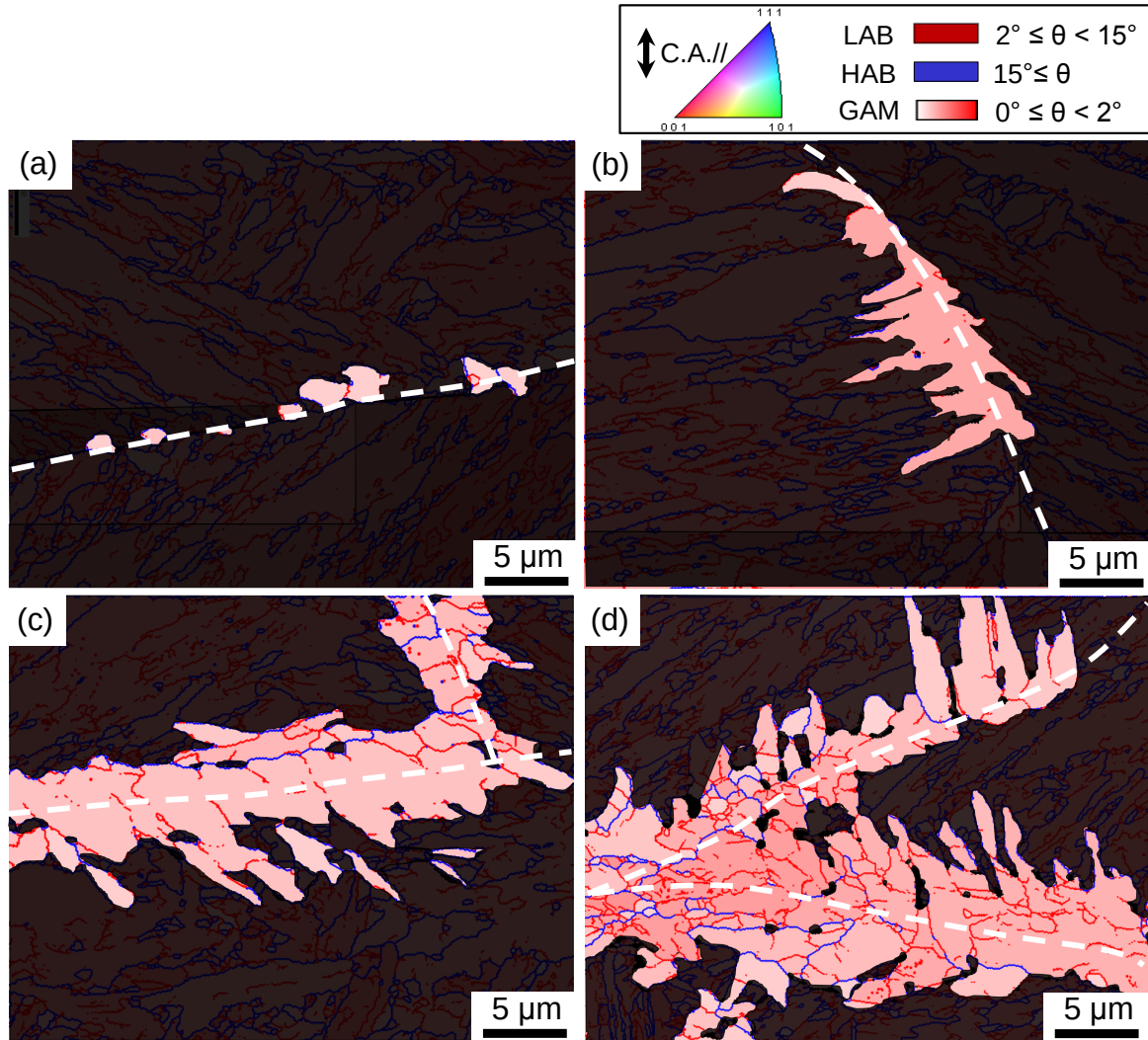


Fig. 4.5 Grain average misorientation (GAM) maps obtained from EBSD measurements of the specimens with an austenite grain size of $125 \mu\text{m}$ deformed to a strain of (a) 0.11, (b) 0.16, (c) 0.29, (d) 0.36, (e) 0.60, (f) 0.92 and (g) 1.39 at a strain rate of 10^{-2} s^{-1} at 520°C . (h) is an inverse pole figure (IPF) map corresponding to (g). The colors in the IPF map (h) indicate the crystallographic orientation parallel to the compression axis, according to the key stereographic triangle shown above the figures. White broken lines in (a)-(d) indicate prior austenite grain boundaries (PAGBs). Non-ferrite phases (i.e., martensite and/or retained austenite) were painted in black. Low-angle boundaries with misorientation of $2-15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Blue arrows in (e)-(g) and black dotted circles in (g)-(h) mark equiaxed UFF grains and subgrains, respectively. Compression axis is along the vertical direction of the maps.

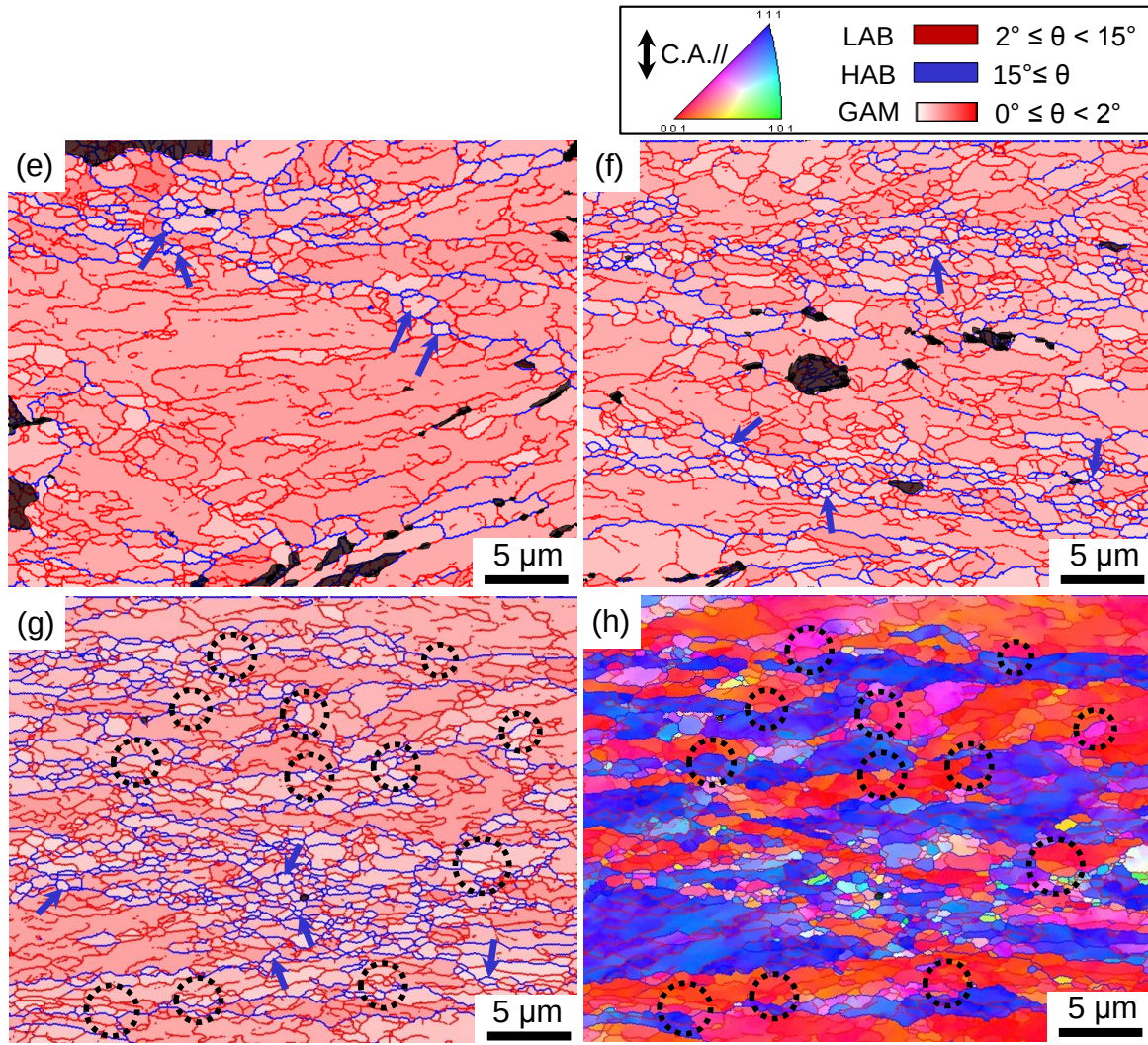


Fig. 4.5 (continued)

The quantitative analysis of the GAM values of the equiaxed UFF grains, subgrains and elongated coarse grains is summarized in **Fig. 4.6**. Note that the subgrains here only include the ones surrounded by both partial LAB and HAB, as is shown in **Fig. 4.5(g)**, and these subgrains are mainly formed in the vicinity of grain boundaries of elongated coarse ferrite. **Figure 4.6(a)**, **(b)** and **(c)** show the distribution of GAM values of the equiaxed UFF grains, subgrains and elongated coarse grains, respectively. The average GAM values of the three types of grains are plotted in **Fig. 4.6(d)**. The elongated coarse grains have an average

GAM of 0.78° , indicating relatively high misorientations within the grains. This indicates that the elongated coarse grains have experienced larger plastic strains. The average GAM values of the equiaxed UFF grains and subgrains are 0.37° and 0.43° , respectively, which are much lower than that of the elongated coarse grains (i.e., deformed grains). The low GAM values of the equiaxed UFF grains as well as some subgrains indicate low misorientations within these grains, which imply that they may appear at later stages of deformation or/and may have experienced a kind of restoration process during deformation.

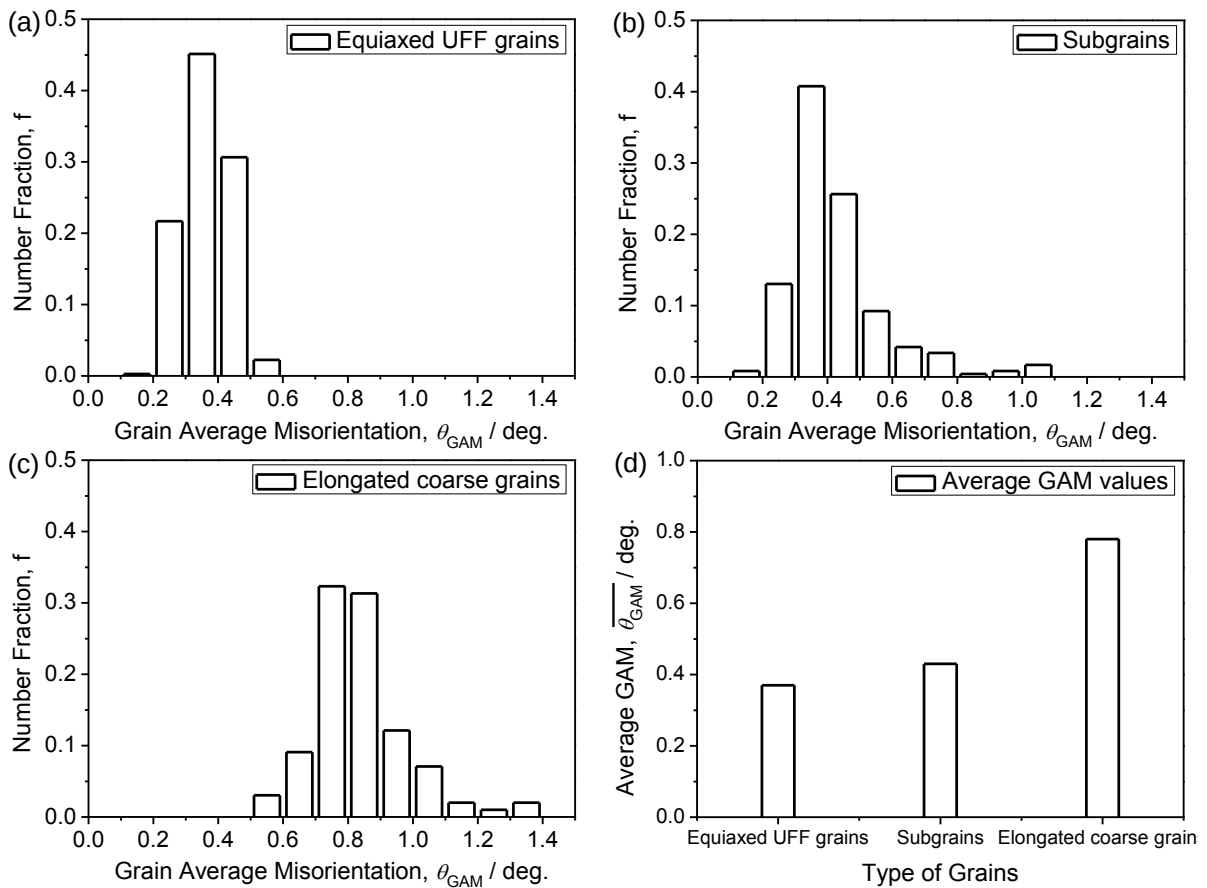


Fig. 4.6 Distribution of GAM values of (a) equiaxed UFF grains, (b) subgrains and (c) elongated coarse grains in **Fig. 4.5(g)**. (d) Average GAM values of the three types of grains.

Since the elongated coarse grains exhibit more deformed structures than the equiaxed UFF grains (**Fig. 4.5(g)** and **Fig.4.6(d)**), the difference in the crystallographic orientations between the two kinds of grains is furthermore investigated. **Figure 4.7** shows IPF maps (a, c) and corresponding IPFs (b, d) of the elongated coarse grains (a, b) and equiaxed UFF grains (c, d) shown in **Fig. 4.5(h)**. The colors in the IPF maps (a, c) indicate the crystallographic orientation parallel to the compression axis, according to the key stereographic triangle shown above the figures. The IPFs shown in (b, d) reveal the relative intensity of crystallographic orientation parallel to the compression axis (compression texture). The IPF of the elongated coarse grains (**Fig. 4.7(b)**) indicates strong $\langle 001 \rangle // CA$ and $\langle 111 \rangle // CA$ textures, which are typical compression textures in BCC crystals [29]. On the other hand, the IPF of the equiaxed UFF grains (**Fig. 4.7(d)**) shows a more random orientation distribution than the elongated coarse grains, although weak concentrations to $\langle 001 \rangle // CA$ and $\langle 111 \rangle // CA$ are observed. It indicates that these equiaxed UFF grains may not be formed through the same formation process as the elongated coarse grains.

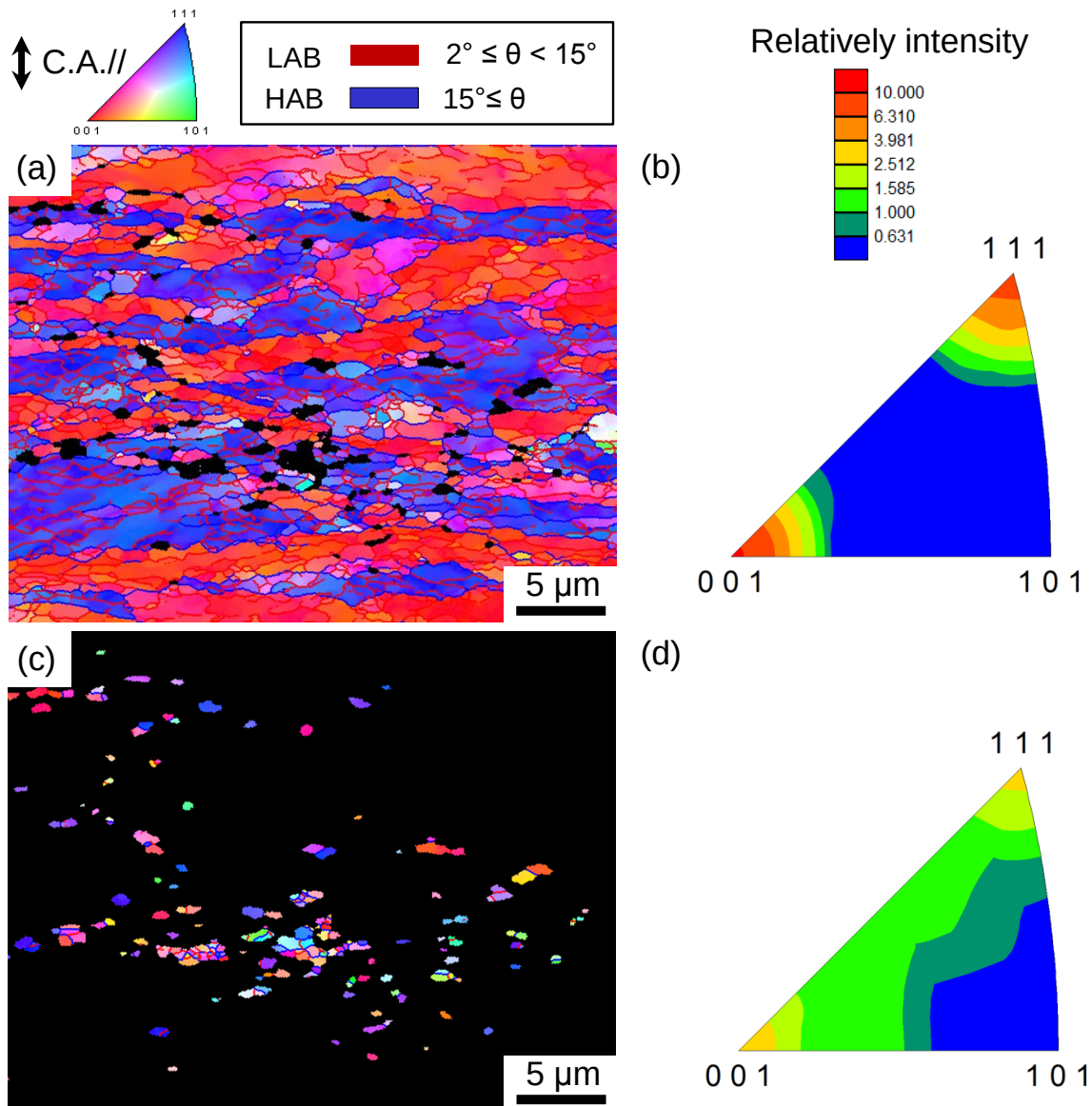


Fig. 4.7 IPF maps (a, c) and IPFs (b, d) of the elongated coarse ferrite grains (a, b) and equiaxed UFF grains (c, d) shown in **Fig. 4.5(h)**. The colors in the IPF maps (a, c) indicate the crystallographic orientation parallel to the compression axis, according to the key stereographic triangle shown above the figures. The IPFs shown in (b, d) reveal the relative intensity of crystallographic orientation parallel to the compression axis (compression texture). Low-angle boundaries with misorientation of $2\text{--}15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively, in (a) and (c). Compression axis is along the vertical direction of the maps.

More detailed information about the equiaxed UFF grains is shown in **Fig. 4.8**, which includes (a) TEM image and (b) corresponding misorientation map determined by

Kikuchi-line analysis in the specimen same as that shown in **Fig. 4.5(g)**. Bold black and broken grey lines depict HABs with misorientation above 15° and LABs with misorientation below 15° , respectively. **Figure 4.8(c)** is an enlarged area marked in **Fig. 4.8(a)** and **(b)**. As shown in the misorientation map (**Fig. 4.8(b)**), along the grain boundaries (depicted by bold black lines) of the elongated coarse ferrite, equiaxed UFF grains and subgrains coexist. The equiaxed UFF contains a certain amount of dislocations (**Fig. 4.8(c)**) and has the similar grain size to the subgrains. The only difference is that the equiaxed UFF grains are surrounded by complete HABs but the subgrains are surrounded by either complete LABs (with misorientation from 0.9° to 13.9°) or partial LAB and HAB. It is already confirmed that the dynamically transformed ferrite is furthermore deformed during growth after nucleation and contains subgrains. During the deformation of ferrite, it is reasonable to consider that higher concentration of deformation (or inhomogeneous deformation) would occur near grain boundaries (i.e., high-angle boundaries) of ferrite. The deformation concentration or inhomogeneous deformation could increase the misorientation angle of the subgrains [30] and form equiaxed UFF surrounded by HABs finally. In order to prove this assumption, the change of misorientation angles of ferrite grains during deformation should be furthermore examined.

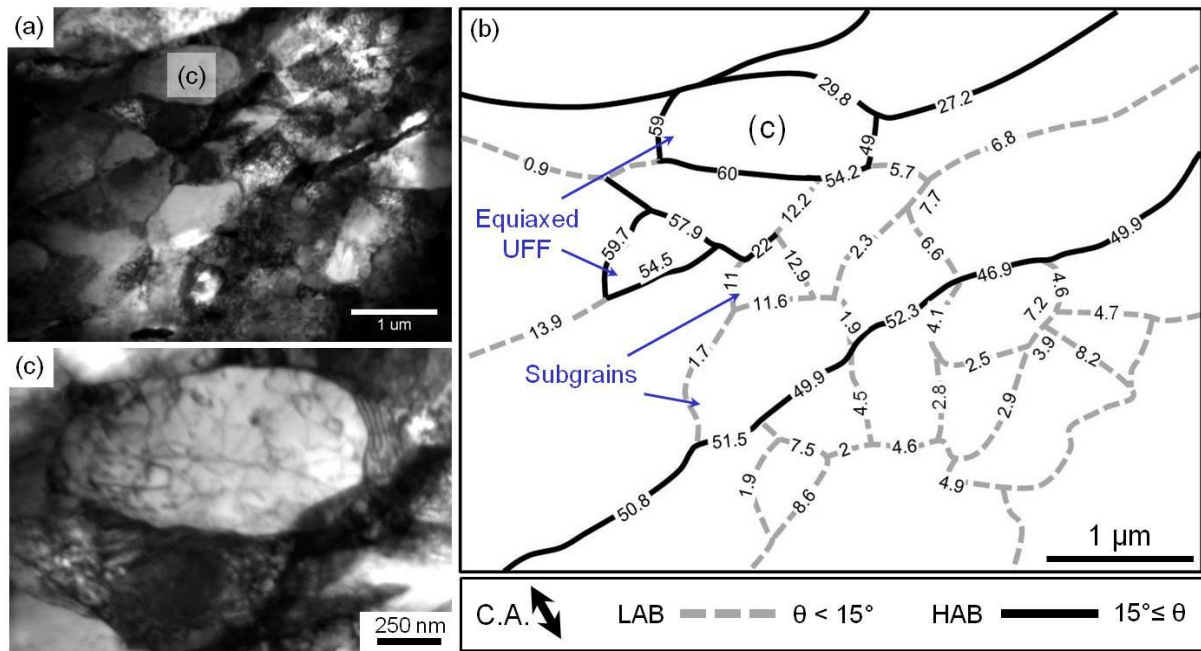


Fig. 4.8 (a) TEM image and (b) corresponding misorientation map determined by Kikuchi-line analysis of the specimen with an austenite grain size of 125 μm deformed to a strain of 1.39 at a strain rate of 10^{-2} s^{-1} at 520 °C. (c) An enlarged area marked in (a) and (b). Low-angle boundaries with misorientation below 15° and high-angle boundaries with misorientation above 15° are drawn in bold black and broken grey lines, respectively. Compression axis is marked in the figure.

Figure 4.9(a) shows changes in the distribution of boundary misorientation angles, obtained by EBSD analysis of the specimens with an austenite grains size of 125 μm deformed to various strains from 0.46 to 1.39 at a strain rate of 10^{-2} s^{-1} at 520 °C. The relative frequency of HABs becomes higher with increasing the strain. The change in average misorientation and fraction of HABs corresponding to **Fig. 4.9(a)** are plotted in **Fig. 4.9(b)** as a function of strain. With increasing the strain, the average misorientation angle of ferrite grains increases. That means the deformation inhomogeneity in ferrite increases with straining, leading to an increase in the fraction of HABs.

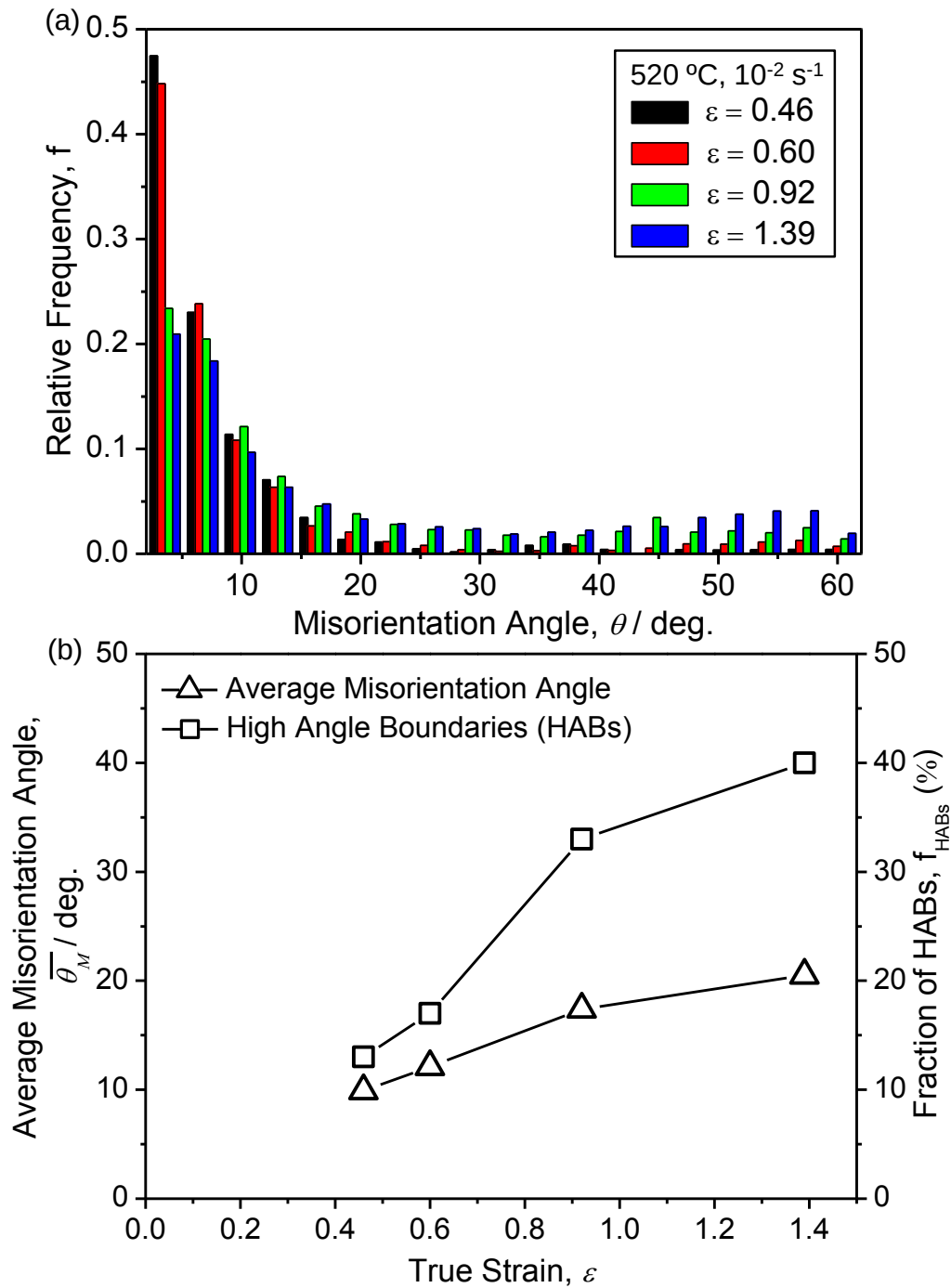


Fig. 4.9 (a) Distribution of boundary misorientation angles obtained by EBSD analysis of ferrite and (b) changes in average misorientation angle and fraction of HABs in the specimens with an austenite grains size of 125 μm deformed at a strain rate of 10^{-2} s^{-1} at 520 °C.

Accordingly, the changes in volume fraction and grain size of the equiaxed UFF, which is surrounded by complete HABs, are shown in **Fig. 4.10**. The UFF starts to appear at a strain

around 0.46, and its fraction increases with further deformation. During the formation of equiaxed UFF, the grain size of the UFF does not show an obvious change, stably keeping around 400 nm. Since the equiaxed UFF grains and subgrains have the similar grain size (around 400 nm) and the fraction of HABs increases with straining, it is suggested that deformation increases the misorientation angles of subgrains and consequently forms the equiaxed UFF grains.

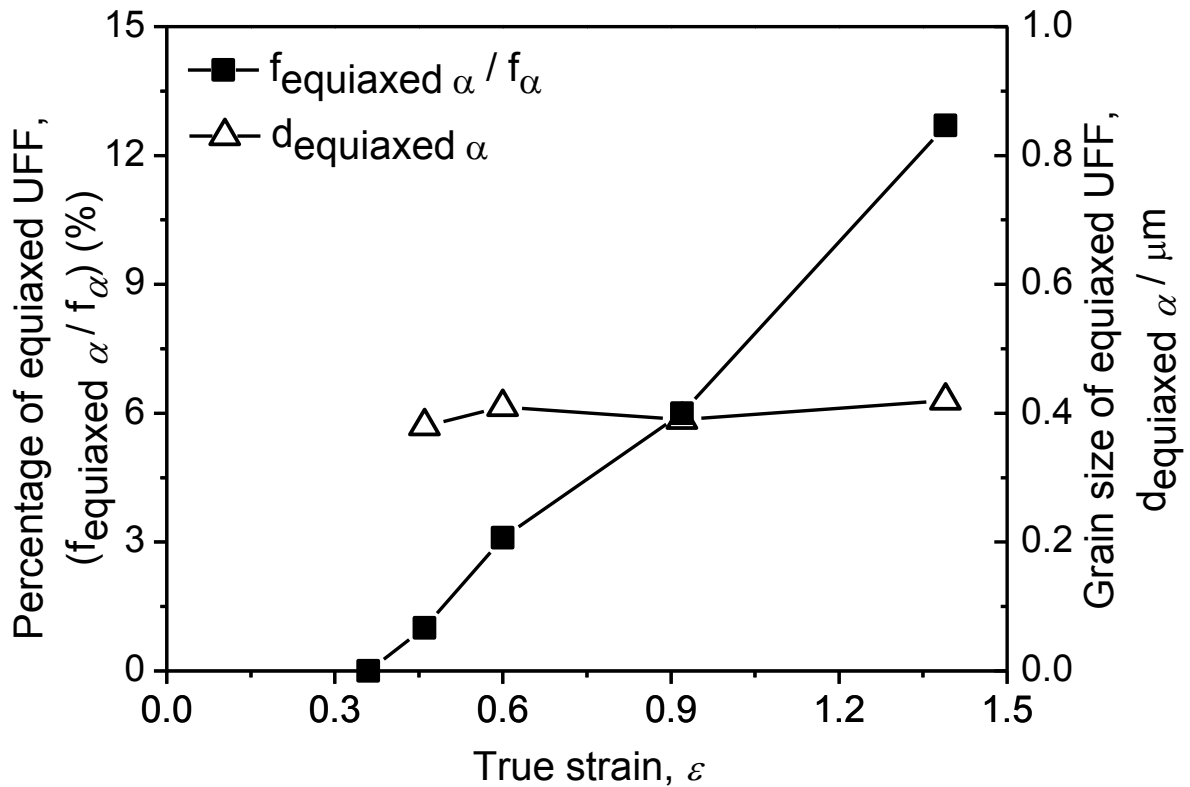


Fig. 4.10 Changes in volume fraction and grain size of equiaxed UFF in the specimens with an austenite grains size of 125 μm deformed at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$.

The evolution of ferrite grains during the thermomechanical processing at 520 $^{\circ}\text{C}$, including the formation process of equiaxed UFF grains, is expressed in another form in **Fig.**

4.11, which shows changes in (a) grain size and volume fraction of the dynamically transformed ferrite and (b) volume fraction of the equiaxed UFF. The grain sizes of ferrite along the compression axis was measured by linear interception method on the EBSD maps only counting boundaries with misorientation larger than 15° (defined as d^{HAGB}). Basically there are two regions in **Fig. 4.11(a)**: a region prior to the onset of DT (No-DT region) and a region after the onset of DT (DT-region). In the DT-region, there is another stage where equiaxed UFF forms. There are two critical strains: one is for the onset of DT and the other is for the initiation of UFF formation. The grain size of dynamically transformed ferrite firstly increases, indicating a grain growth process at a relatively early stage of transformation, but then decreases after a certain strain, which is totally different from the conventional grain growth behavior in static ferrite transformation [17-22]. The decrease in the ferrite grain size suggests another grain refinement process happening during deformation. As shown in **Fig. 4.11(a)** and **(b)**, the decrease in the ferrite grain size accords well with the development of UFF. Therefore, it could be concluded that the equiaxed UFF grains only appear after a certain strain after the onset of DT. It is the formation of equiaxed UFF that leads to the grain refinement of ferrite.

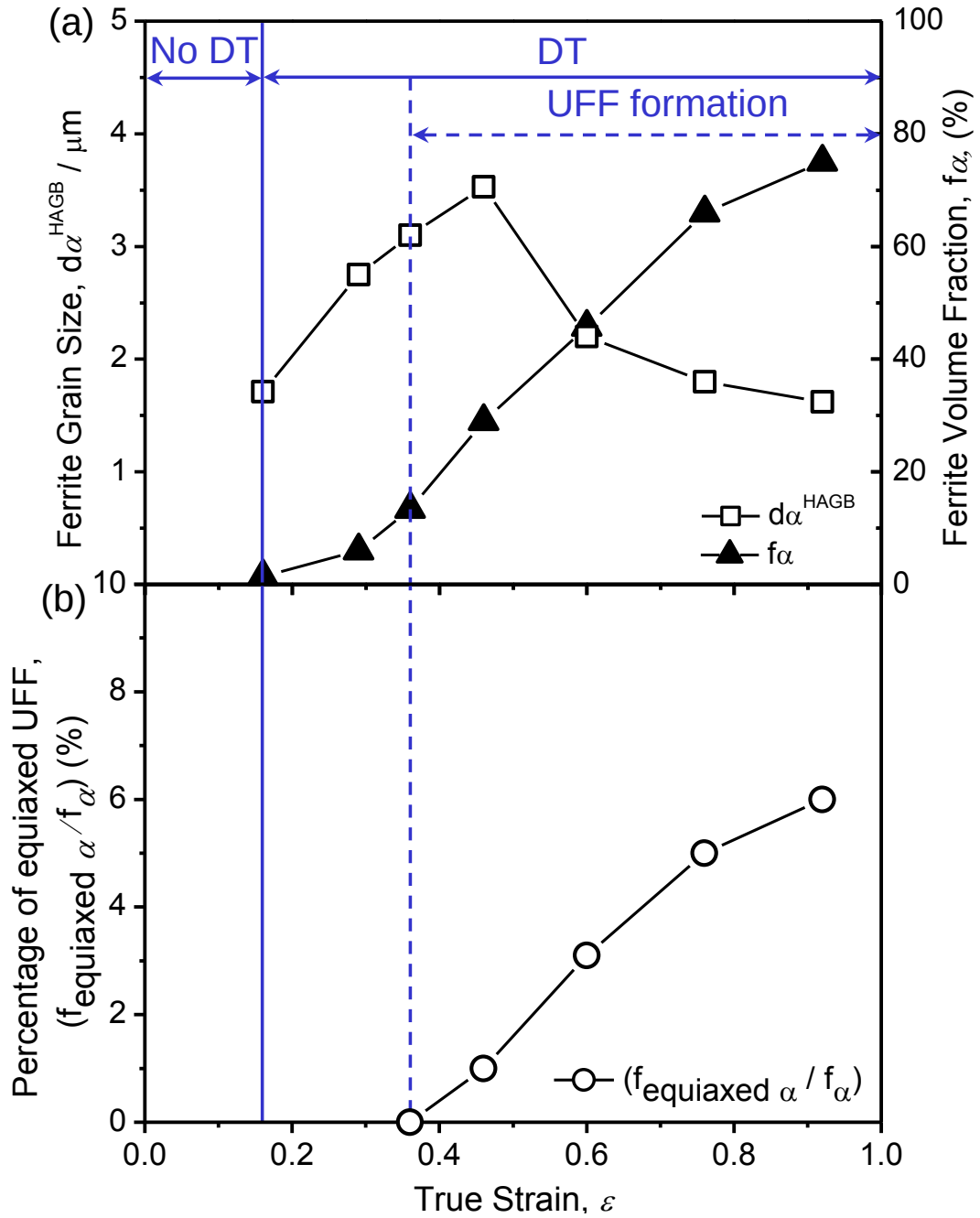


Fig. 4.11 Changes in (a) ferrite grain size and volume fraction of the dynamically transformed ferrite and (b) volume fraction of the equiaxed UFF in the specimens with an austenite grain size of $125 \mu\text{m}$ deformed at strain rate of 10^{-2} s^{-1} at $520 \text{ }^{\circ}\text{C}$. The ferrite grain size was measured by linear interception method on the EBSD maps only counting boundaries with misorientation above 15° (defined as d^{HAGB}).

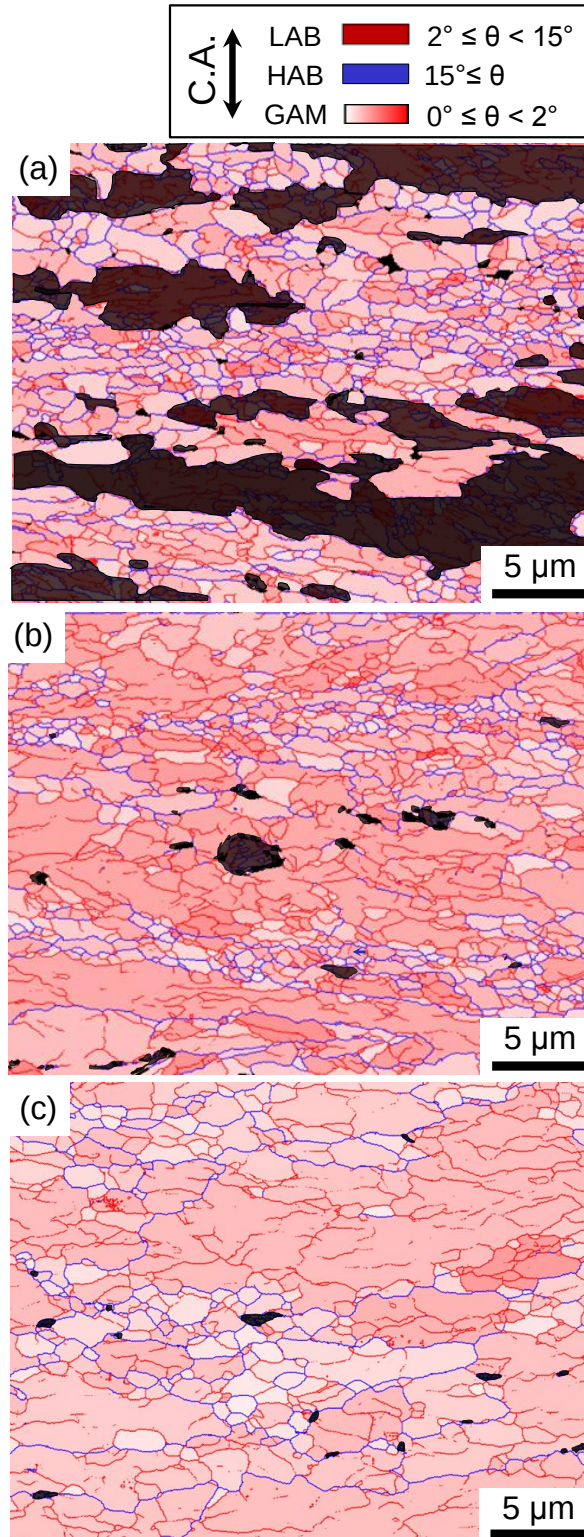


Fig. 4.12 GAM maps of the specimens with an austenite grain size of $125 \mu\text{m}$ deformed to a strain of 0.92 at 520°C at various strain rates of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} . Non-ferrite phases (i.e., martensite and/or retained austenite) were painted in black. Low-angle boundaries with misorientation of $2\text{--}15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Compression axis is along the vertical direction of the maps.

All the above description of DT behavior comes from one deformation condition, that is, the deformation at a strain rate of 10^{-2} s^{-1} at 520°C . **Figure 4.12** shows GAM maps of the specimens deformed to a strain of 0.92 at various strain rates of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at 520°C . At all strain rates (**Fig. 4.12(a)-(c)**), the microstructures consist of elongated coarse ferrite grains and equiaxed UFF grains, showing a similar feature to that described above.

Figure 4.13(a) shows distribution of boundary misorientation angles, obtained by the EBSD analysis of the ferrite corresponding to **Fig. 4.12**. The relative frequency of HABs becomes higher with increasing the strain rate. The changes in average misorientation and fraction of HABs corresponding to **Fig. 4.13(a)** are plotted in **Fig. 4.13(b)** as a function of strain rate. With increasing the strain rate, the average misorientation angle of ferrite grains increases. That means the deformation inhomogeneity in ferrite becomes higher at a higher strain rate, leading to an increase in the fraction of HABs. It indicates that high strain rate enhances the formation of HABs under a constant strain.

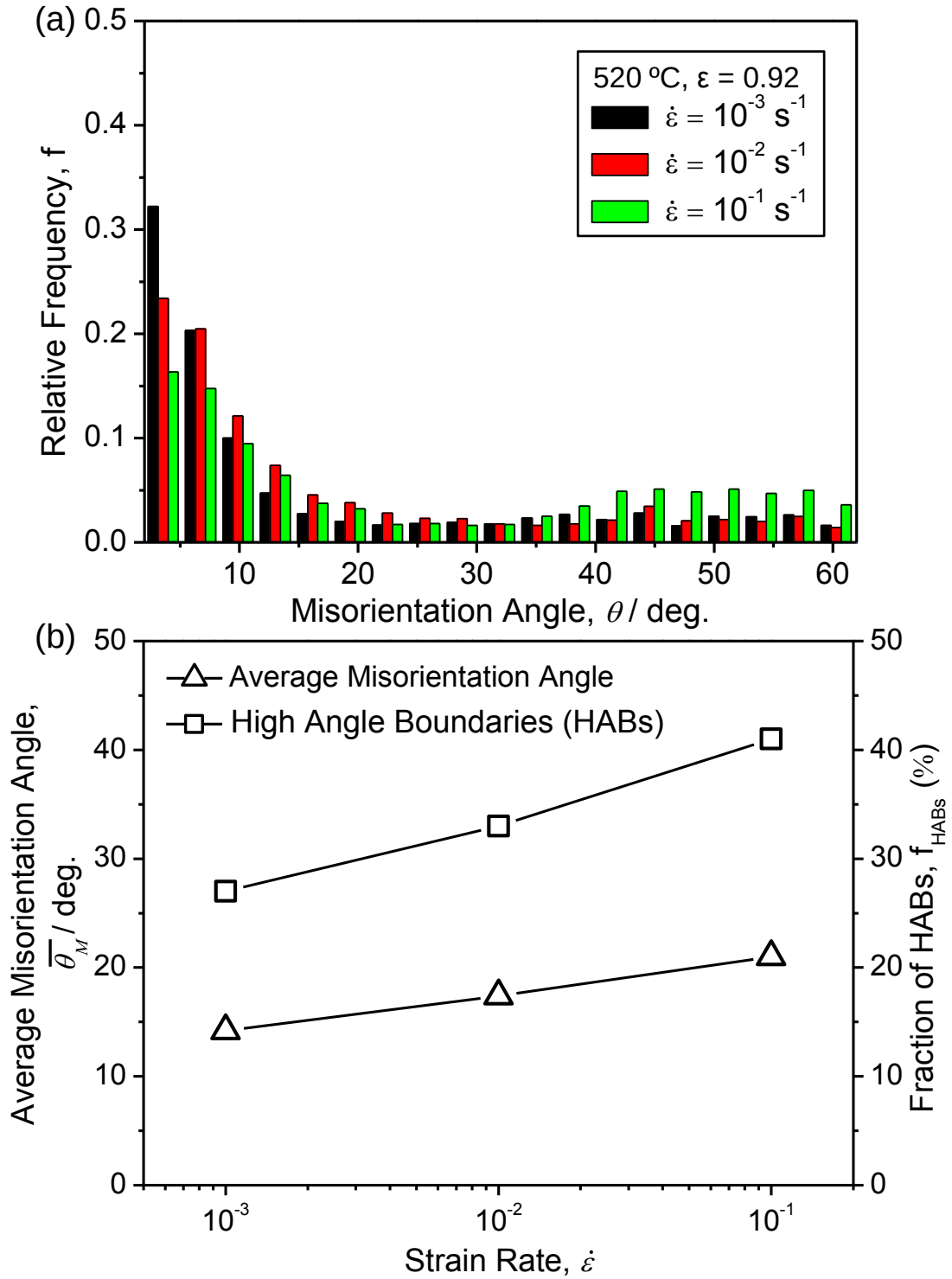


Fig. 4.13 (a) Distribution of boundary misorientation angles obtained by EBSD analysis of ferrite and (b) changes in average misorientation angle and fraction of HABs in the specimens with an austenite grains size of 125 μm deformed to a strain of 0.92 at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 °C.

Figure 4.14 summarizes changes in (a) volume fraction of dynamically transformed ferrite, (b) mean grain size of ferrite and (c) the volume fraction of equiaxed UFF (within the whole transformed ferrite) obtained at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C .

The volume fractions of dynamically transformed ferrite (**Fig. 4.14(a)**) increase with straining. At the same strain, the ferrite volume fraction increases with decreasing the strain rate. The grain size of ferrite (**Fig. 4.14(b)**) firstly increases and then decreases with increasing the strain at all three strain rates. At each strain rate, the grain size decreases after a certain strain, which corresponds well with the formation of equiaxed UFF (**Fig. 4.14(c)**). The grain size and the critical strain are larger at lower strain rate (**Fig. 4.14(b)**), although the onset strain of UFF formation does not differ so much at different strain rates (**Fig. 4.14(c)**). The fraction of equiaxed UFF increases with increasing the strain, and is larger at higher strain rate (**Fig. 4.14(c)**). Here it could be confirmed that the equiaxed UFF grains are formed at various strain rates, and the tendencies shown in **Fig. 4.14** are all similar to each other. It is the formation of equiaxed UFF grains that leads to the decrease of ferrite grain size during deformation.

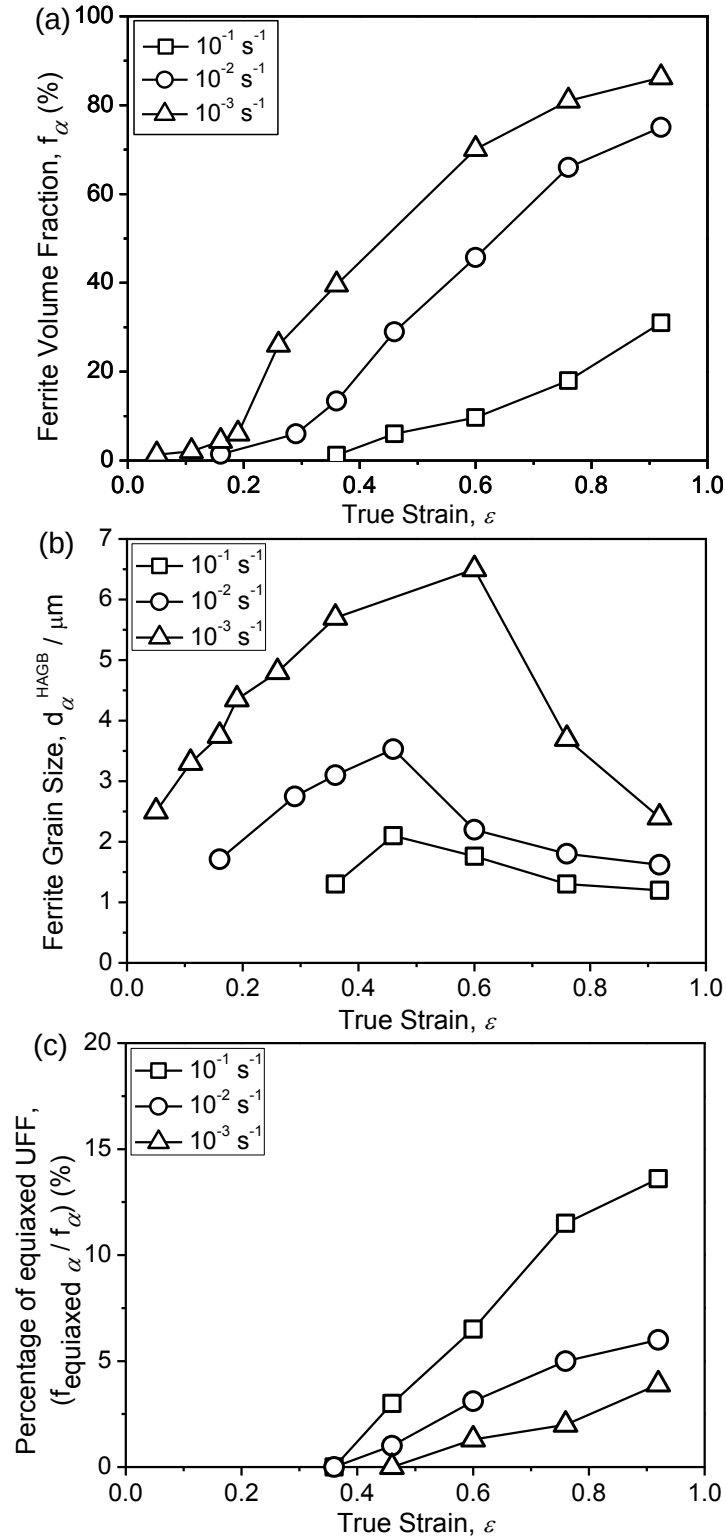


Fig. 4.14 Changes in (a) volume fraction of dynamically transformed ferrite, (b) mean grain size of ferrite and (c) volume fraction of the equiaxed UFF (within the whole transformed ferrite) in the specimens with an austenite grain size of 125 μm deformed at different strain rates ranging from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$. The grain sizes of ferrite along the compression axis was measured by linear interception method on the EBSD maps only counting boundaries with misorientation larger than 15° (defined as d^{HAGB}).

The results on microstructure evolution of ferrite during the dynamic transformation process (in the route-1) suggest the most possible mechanism of the equiaxed UFF formation: after the formation by DT, ferrite is further deformed, which leads to the formation of LABs inside the ferrite grains (**Fig. 4.5(c)**), and subgrains surrounded by LABs are formed during the deformation of ferrite (**Fig. 4.5(d)-(g)**). Higher concentration of deformation (or inhomogeneous deformation) would occur near grain boundaries (i.e., high-angle boundaries) of ferrite. The deformation concentration or inhomogeneous deformation could increase the misorientation angle of the subgrains (**Fig. 4.9**) and form equiaxed UFF surrounded by HABs finally (**Fig. 4.10**). The equiaxed UFF grains contain a certain amount of dislocations (**Fig. 4.8(c)**) and show nearly random crystallographic texture, which is different from the elongated coarse ferrite originated from DT (**Fig. 4.7**). The features of the UFF formation accord well with those of DRX of ferrite [31-33].

There are two main aspects which can lead to the occurrence of DRX of dynamically transformed ferrite. Firstly, ferrite is softer than austenite at elevated temperatures [34, 35]. Once ferrite is formed, subsequently the applied deformation tends to more concentrate on the softer phase (i.e., ferrite) in the specimen composed of dual-phase structures (of ferrite and austenite). The microstructure showed in **Fig. 4.5** confirms that the ferrite is surely deformed after the formation. It is expected that the plastic deformation of ferrite could be quite large due to the strain concentration. Secondly, during the nucleation and growth

process in dynamic ferrite transformation, ferrite could be deformed at any stages of transformation, even when they maintain very fine grain sizes. It is widely believed that decreasing the grain size of ferrite can not only decrease the critical strain for the onset of DRX, but also accelerate the DRX kinetics [36, 37]. As shown in **Fig. 4.14(b)**, the grain size of ferrite in the whole transformation process is not bigger than 7 μm . That means the transformed ferrite could be deformed in the grain sizes around several microns or even finer, which would significantly shorten the incubation time (strain) for initiating DRX. Therefore, the high strain concentration and fine ferrite grain size might contribute to the occurrence of DRX in the route-1.

The thermomechanical processes including DT (i.e., the route-1) could be schematically illustrated in **Fig. 4.15**. F_s and F_f indicate the starting and finishing points of ferrite transformation, respectively. R_s and R_f indicate the starting and finishing points of recrystallization of ferrite, respectively. As is well known, in diffusional phase transformations, lower temperature (above the nose temperature of kinetic C-curve) accelerates transformation kinetics due to the higher chemical driving force under larger supercooling. But in (static) recrystallization from deformed matrix, higher temperature enhances the recrystallization kinetics because of higher diffusivity under a constant driving force given by the deformation. During the deformation at a specific temperature, when ferrite is dynamically transformed from austenite, it will be further deformed. High strain

concentration on the fine ferrite grains leads to the initiation of DRX, resulting in the further grain refinement of ferrite. According to the present study, the evolution of UFF grains from the microstructure firstly formed by DT strongly suggests the occurrence of DRX. It is the concurrent behaviors of DT and DRX that leads to the formation of equiaxed UFF grains during one thermomechanical process.

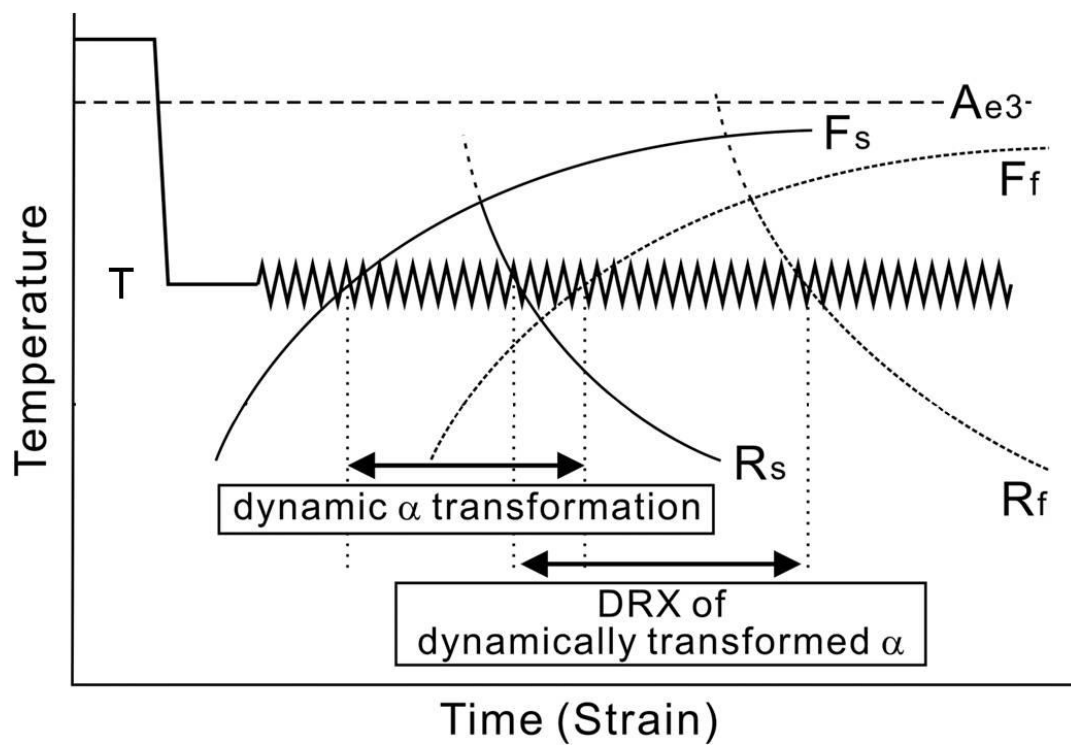


Fig. 4.15 Schematic illustration showing the occurrence of DRX during a thermomechanical process including DT at a constant temperature and strain rate.

4.3.2 Occurrence of DRX of statically transformed ferrite

In the previous section, the evolution of UFF grains from the microstructure firstly formed by DT strongly suggests the occurrence of DRX (in the route-1). In this section, investigations are conducted to confirm if DRX could occur during the compression deformation of fully ferrite phase (in the route-2) in the present material. In order to compare the similarity and difference in DRX behaviors between the two thermomechanical routes, experiments in the route-2 are carried out under the same deformation condition as that in the route-1. Furthermore, the effect of initial ferrite grain size on DRX behavior is also examined.

Figure 4.16 shows true stress-true strain curves of the specimens with (a) coarse ferrite (grain size of 17 μm) and (b) fine ferrite (grain size of 6.7 μm) deformed in the route-2 at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$. The fully ferrite phase with different grain sizes were obtained by furnace-cooling, as was introduced in **Section 4.2**. The flow curves display early strain-hardening followed by steady state. However, perfect steady-state is not achieved but slight softening is observed in every case. The softening in the true stress-true strain curves suggests the occurrence of dynamic recrystallization, as similar stress-strain curves have been reported in DRX of ferrite [38, 39].

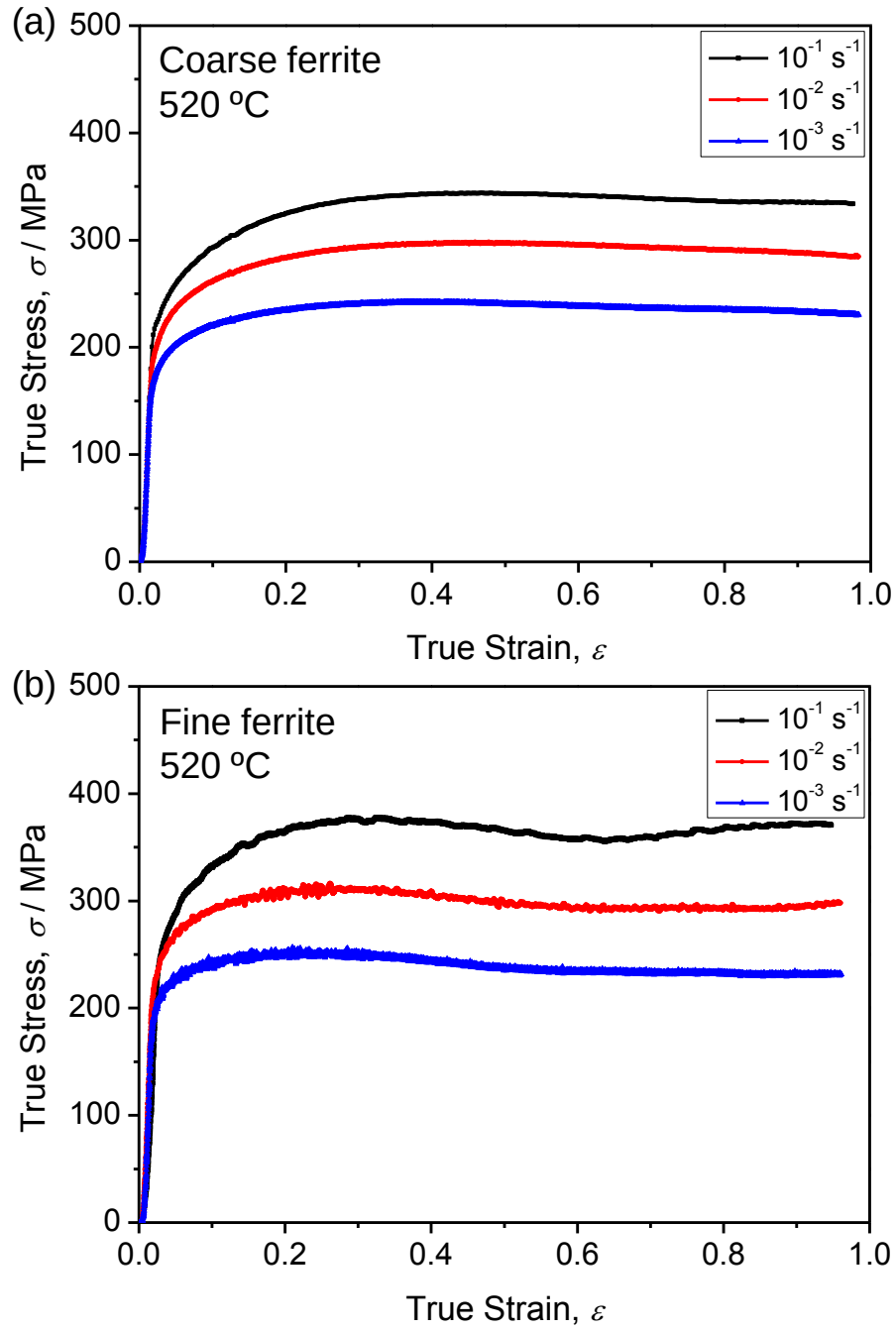


Fig. 4.16 True stress-true strain curves of the specimens initially having (a) coarse ferrite grain size of 17 μm and (b) fine ferrite grain size of 6.7 μm deformed in the route-2 at strain rates ranging from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 °C.

Microstructural evolution of ferrite is investigated by interrupting the deformation at several stages and observing microstructures. **Figure 4.17** is a true stress-true strain curve of the specimen with initial ferrite grain size of 6.7 μm deformed in the route-2 at a strain rate

of 10^{-2} s^{-1} at 520°C . Triangle symbols on the true stress-true strain curve indicate the positions where the microstructures were observed.

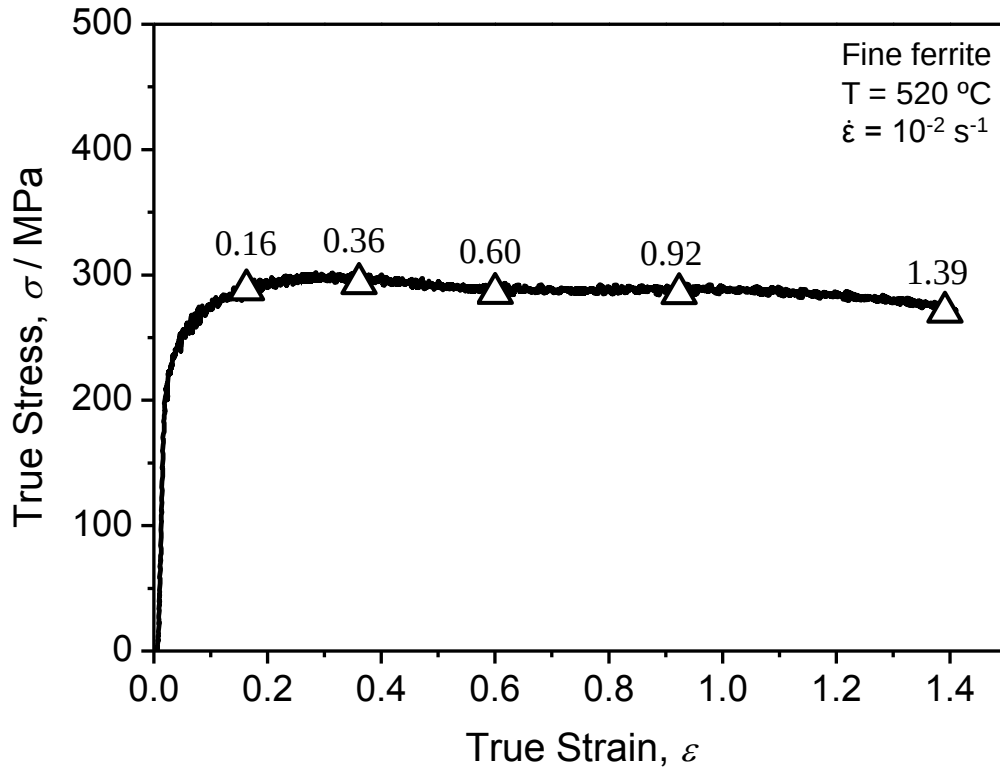


Fig. 4.17 True stress-true strain curve of the specimen with fine initial ferrite (grain size of $6.7 \mu\text{m}$) deformed in the route-2 at a strain rate of 10^{-2} s^{-1} at 520°C . The triangle symbols indicate the positions where the microstructures were observed.

The microstructures of ferrite corresponding to **Fig. 4.17** are presented in **Fig. 4.18**, which shows GAM maps obtained from EBSD measurements of the specimens deformed to a strain of (a) 0.16, (b) 0.36, (c) 0.60, (d) 0.92 and (e) 1.39. (f) is an IPF map corresponding to (e). At a strain of 0.16 (**Fig. 4.18(a)**), the microstructure consists of mostly coarse (initial) ferrite grains with a small amount of LABs inside. With increasing the strain to 0.36 (**Fig. 4.18(b)**), the fraction of LABs within the deformed initial grains increases and a few equiaxed fine

grains surrounded by HABs (as marked by blue arrows) start to form along the grain boundaries of coarse ferrite. The fraction of the equiaxed fine grains increases with increasing the strain from 0.60 (**Fig. 4.18(c)**) to 1.39 (**Fig. 4.18(e)**). At a strain of 1.39, the ferrite microstructure is composed of equiaxed fine grains and elongated coarse grains. Subgrains surrounded by either complete LABs or partial LAB and HAB are formed within the elongated coarse grains. The deformation can introduce defects in ferrite and thus to increase the misorientation within the grains. The misorientation within each grain can be evaluated by GAM values. Qualitatively speaking, the elongated coarse grains mainly exhibit high misorientation (dark red color) within the grains. On the contrary, most of the equiaxed fine grains show lower GAM values (light red color) compared to the elongated coarse grains. Quite a few of the subgrains (especially those surrounded by partial LAB and HAB, as marked by dotted circles in **Fig. 4.18(e)** and **(f)**) show the similar crystallographic orientations to the elongated coarse grains (**Fig. 4.18(f)**), but have relatively low GAM values (light red color) similar to the equiaxed fine grains (**Fig. 4.18(e)**). Those microstructural characteristics are quite similar to those of the microstructures obtained in the route-1 (**Fig. 4.5**).

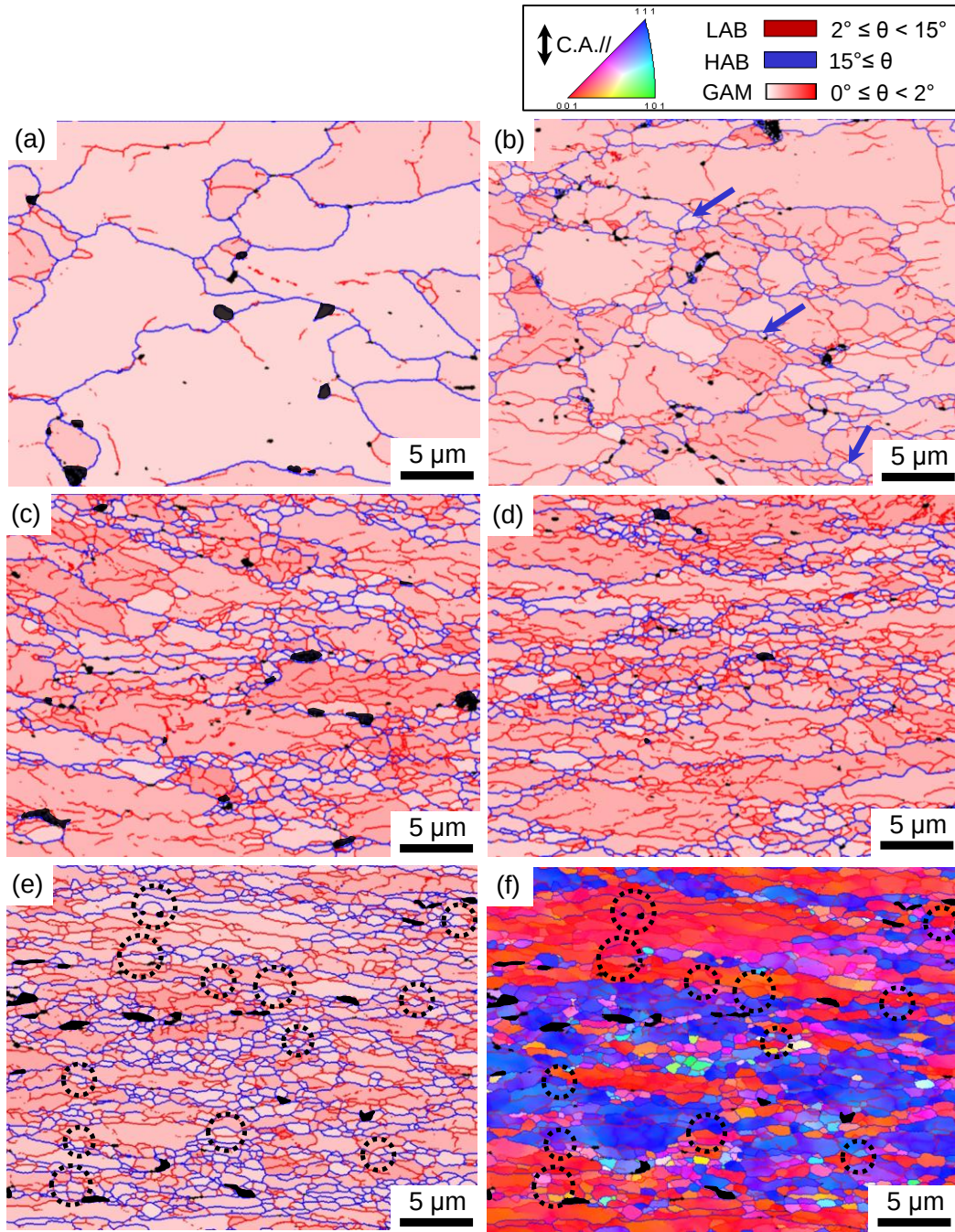


Fig. 4.18 Grain average misorientation (GAM) maps of the specimens with an initial ferrite grain size of $6.7\ \mu\text{m}$ deformed to a strain of (a) 0.16, (b) 0.36, (c) 0.60, (d) 0.92 and (e) 1.39 at $10^{-2}\ \text{s}^{-1}$ and $520\ ^\circ\text{C}$. (f) is an IPF map corresponding to (e). The colors in the IPF map (f) indicate the crystallographic orientation parallel to the compression axis, according to the key stereographic triangle shown above the figures. Non-ferrite phase (i.e., reversely transformed austenite during deformation of ferrite) was painted in black. Low-angle boundaries with misorientation of $2\text{--}15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Blue arrows in (b) and black dotted circles in (e)-(f) mark equiaxed UFF grains and subgrains, respectively. Compression axis is along the vertical direction of the maps.

The result of quantitative analysis of the GAM values of equiaxed fine grains, subgrains and elongated coarse grains is shown in **Fig. 4.19**. It should be noted that the subgrains only include the ones surrounded by partial LAB and HAB, as is shown in **Fig. 4.18(e)**, and these subgrains are mainly formed in the vicinity of grain boundaries of elongated coarse ferrite. **Figure 4.19(a), (b) and (c)** show the distribution of GAM values of the equiaxed fine grains, subgrains and elongated coarse grains, respectively. The average GAM values of the three types of grains are plotted in **Fig. 4.19(d)**. The elongated coarse grains have an average GAM of 0.72° , indicating relatively high misorientation within the grains. This indicates that the elongated coarse grains have experienced larger plastic strains. The average GAM values of the equiaxed fine grains and subgrains are 0.37° and 0.41° , respectively, which are much lower than that of the elongated coarse grains (i.e., deformed grains). The low GAM values of the equiaxed fine grains as well as the subgrains indicate small misorientations within these grains, which imply that they may have experienced a kind of restoration process during deformation.

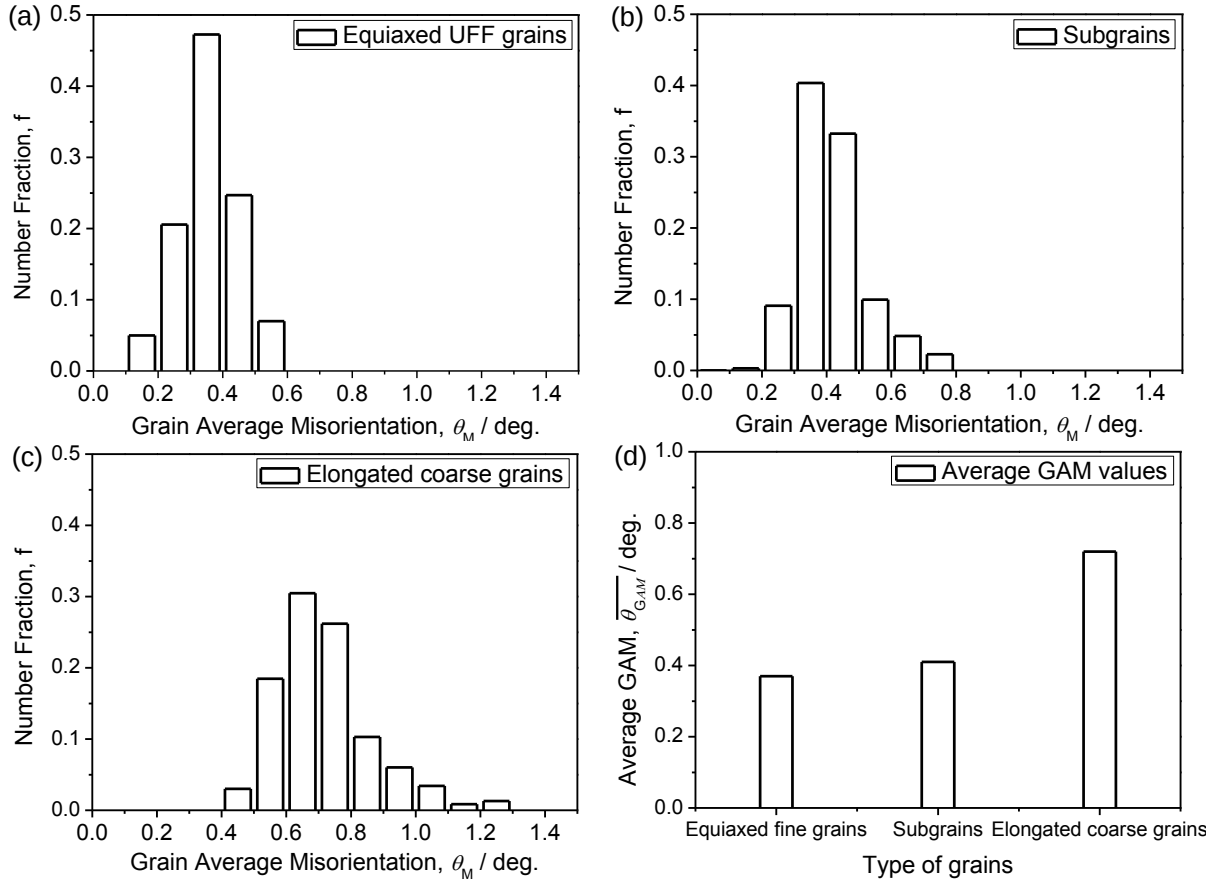


Fig. 4.19 Distributions of GAM values of (a) equiaxed fine grains, (b) subgrains and (c) elongated coarse grains in the specimen corresponding to **Fig. 4.18(e)**. (d) Average GAM values of the three types of grains.

Since the elongated coarse grains exhibit more deformed structures than the equiaxed fine grains (**Fig. 4.18(e)** and **Fig. 4.19(d)**), the difference in the crystallographic orientations between the two kinds of grains is furthermore investigated. **Figure 4.20** shows IPF maps (a, c) and IPFs (b, d) of the elongated coarse grains (a, b) and equiaxed fine grains (c, d) shown in **Fig. 4.18(f)**. The IPF of the elongated coarse grains (**Fig. 4.20(b)**) indicates strong $\langle 001 \rangle // CA$ and $\langle 111 \rangle // CA$ textures, which are typical compression textures in BCC crystals [29]. On the other hand, the IPF of the equiaxed fine grains (**Fig. 4.20(d)**) shows a more

random orientation distribution than the elongated coarse grains, although weak concentrations to $\langle 001 \rangle // CA$ and $\langle 111 \rangle // CA$ are observed.

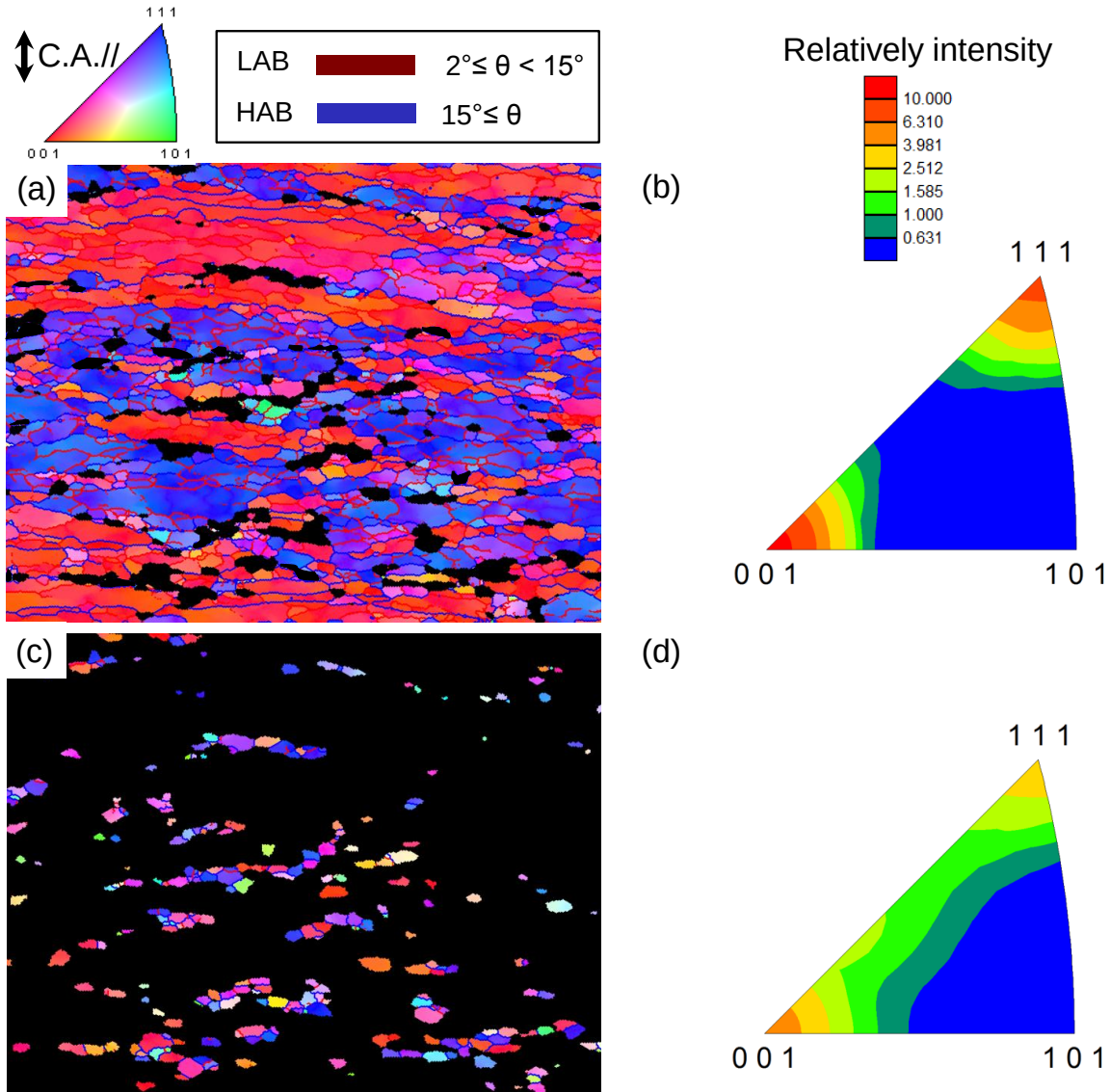


Fig. 4.20 IPF map (a, c) and IPF (b, d) of the elongated coarse ferrite grains (a, b) and equiaxed fine grains (c, d) shown in **Fig. 4.18(f)**. The colors in the IPF map (a, c) indicate the crystallographic orientation parallel to the compression axis, according to the key stereographic triangle shown above the figures. The IPF shown in (b, d) reveals the relative intensity of crystallographic orientation parallel to the compression axis (compression texture). Low-angle boundaries with misorientation of $2\text{--}15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively, in (a) and (c). Compression axis is along the vertical direction of the maps.

Another noteworthy feature of the equiaxed fine ferrite is that, the grain size of the equiaxed fine ferrite is quite similar to that of some subgrains (as highlighted by dotted circles in **Fig. 4.18(e)**). The equiaxed fine grains also have similar GAM values as the subgrains (**Fig. 4.18(e)** and **Fig. 4.19(d)**). The main difference between two types of grains is that the equiaxed fine grains are surrounded by HABs but the subgrains are surrounded either by complete LABs or partial LAB and HAB. During the deformation of ferrite, it is reasonable to consider that higher concentration of deformation (or inhomogeneous deformation) would occur near grain boundaries (i.e., high-angle boundaries) of ferrite. The deformation concentration (or inhomogeneous deformation) could increase the misorientation angle of the subgrains [30], especially those near the grain boundaries of ferrite. Finally equiaxed fine grains surrounded by complete HABs are formed. In order to prove this assumption, the change of misorientation angles of ferrite grains during deformation should be furthermore examined.

Figure 4.21(a) shows changes in distribution of boundary misorientation angles, obtained by EBSD analysis of the specimens with initial ferrite grain size of 6.7 μm deformed to various strains from 0.36 to 1.39 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. The relative frequency of HABs becomes higher with increasing the strain, implying a statistical transition of the misorientation of ferrite grains from low angle to high angle. The change in average misorientation and fraction of HABs corresponding to **Fig. 4.21(a)** are plotted in **Fig. 4.21(b)**

as a function of strain. With increasing the strain, the average misorientation angle of ferrite grains increases. That means the deformation inhomogeneity in ferrite increases with straining, leading to an increase in the fraction of HABs.

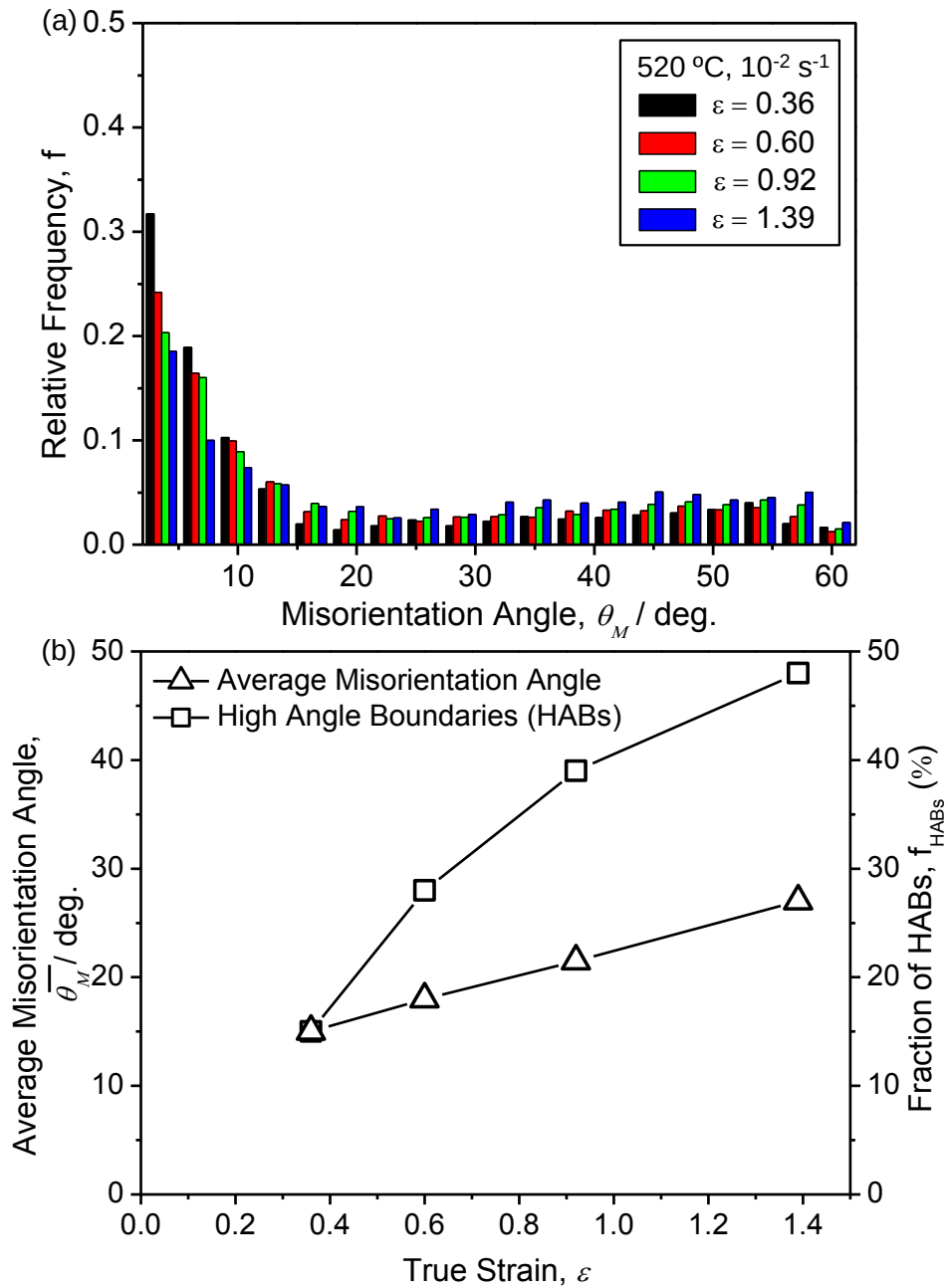


Fig. 4.21 (a) Distribution of boundary misorientation angles of ferrite and (b) changes in average misorientation angle and fraction of HABs in the specimens with a ferrite grain size of $6.7 \mu\text{m}$ deformed at a strain rate of 10^{-2} s^{-1} at $520 \text{ }^\circ\text{C}$.

Accordingly, the changes in volume fraction and grain size of the equiaxed fine ferrite, which is surrounded by complete HABs, are shown in **Fig. 4.22**. The equiaxed fine grains are firstly observed at a strain around 0.36, and its fraction increases with further straining. During the formation of equiaxed fine grains, the grain size of the equiaxed fine ferrite does not obviously change, but stably keeps a mean grain size around 600 nm. Since the equiaxed fine grains and subgrains have the similar grain size (around 600 nm) and the fraction of HABs increases with straining, it is suggested that deformation increases the misorientation angles of subgrains and consequently forms the equiaxed fine grains.

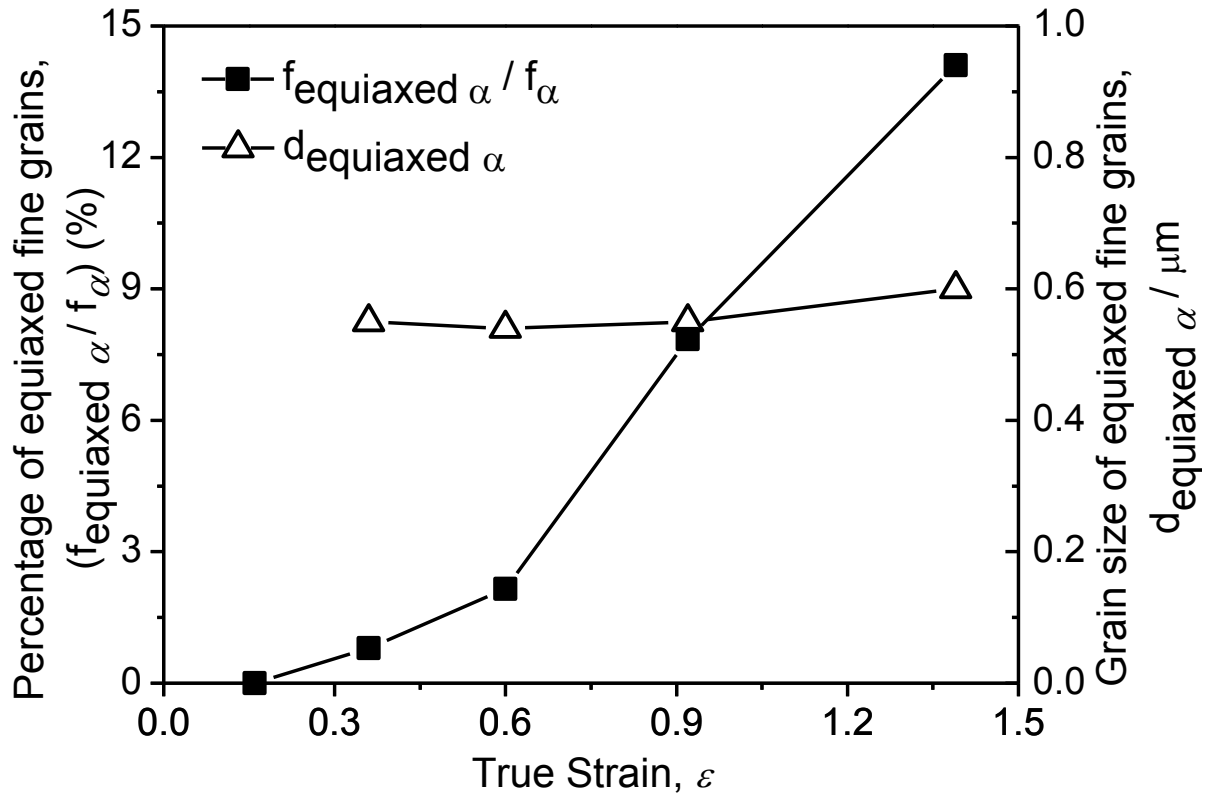


Fig. 4.22 Changes in volume fraction and grain size of equiaxed fine ferrite grains in the specimens with a ferrite grains size of 6.7 μm deformed at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$.

All the above description of ferrite microstructure evolution is obtained from the results under one deformation condition, that is, the specimen with initial ferrite grain size of $6.7\ \mu\text{m}$ was deformed at a strain rate of $10^{-2}\ \text{s}^{-1}$ at $520\ ^\circ\text{C}$. **Figure 4.23** shows GAM maps of the specimens with (a-c) coarse initial ferrite grain size of $17\ \mu\text{m}$ and (d-f) fine ferrite grain size of $6.7\ \mu\text{m}$ deformed to a strain of 0.92 at various strain rates at $520\ ^\circ\text{C}$. In the case of coarse initial grain size, equiaxed fine grains are hard to form at a high strain rate of $10^{-1}\ \text{s}^{-1}$ (**Fig. 4.23(a)**). With decreasing the strain rate to $10^{-2}\ \text{s}^{-1}$ and $10^{-3}\ \text{s}^{-1}$, the volume fraction of equiaxed UFF grains increases (as marked by blue arrows). Compared to the specimens with coarse initial grain size, equiaxed fine grains are much more easily formed in the specimens with fine initial ferrite grain size (as marked by blue arrows in **Fig. 4.23(d)-(f)**). The result indicates that decreasing the ferrite grain size enhances the formation of euqiaxed fine grains. The volume fraction of equiaxed fine grains increases with decreasing the strain rate, suggesting that low strain rate enhances the formation of equiaxed fine ferrite. On the other hand, the grain size of equiaxed fine ferrite increases with decreasing the strain rate.

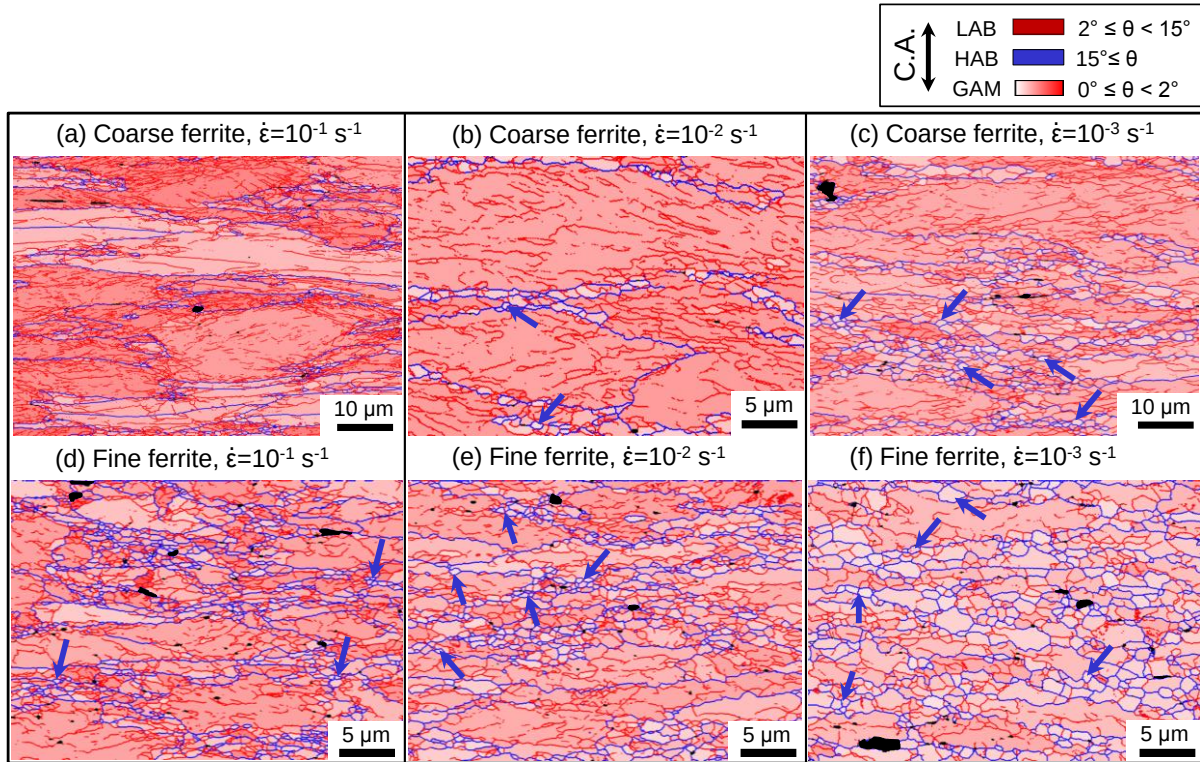


Fig. 4.23 GAM maps of the specimens with (a)-(c) coarse initial ferrite grain size of 17 μm and (d)-(f) fine initial ferrite grain size of 6.7 μm deformed to a strain of 0.92 at various strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$. Low-angle boundaries with misorientation of $2-15^{\circ}$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Blue arrows in (b)-(f) mark equiaxed UFF grains. Compression axis is along the vertical direction of the maps.

Figure 4.24 shows distributions of boundary misorientation angle (a, c) and changes in average misorientation angle as well as fraction of HABs (b, d) of the specimens with coarse initial grains (a, b) and fine initial grains (c, d) deformed to a strain of 0.92 at strain rates from 10^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$. It is shown that the relative frequency of HABs increases with decreasing the strain rate. The average misorientation angle as well as the fraction of HABs increases with decreasing the strain rate. The result also confirms the tendency that low strain rates enhance the formation of HABs, i.e., the formation of equiaxed fine grains.

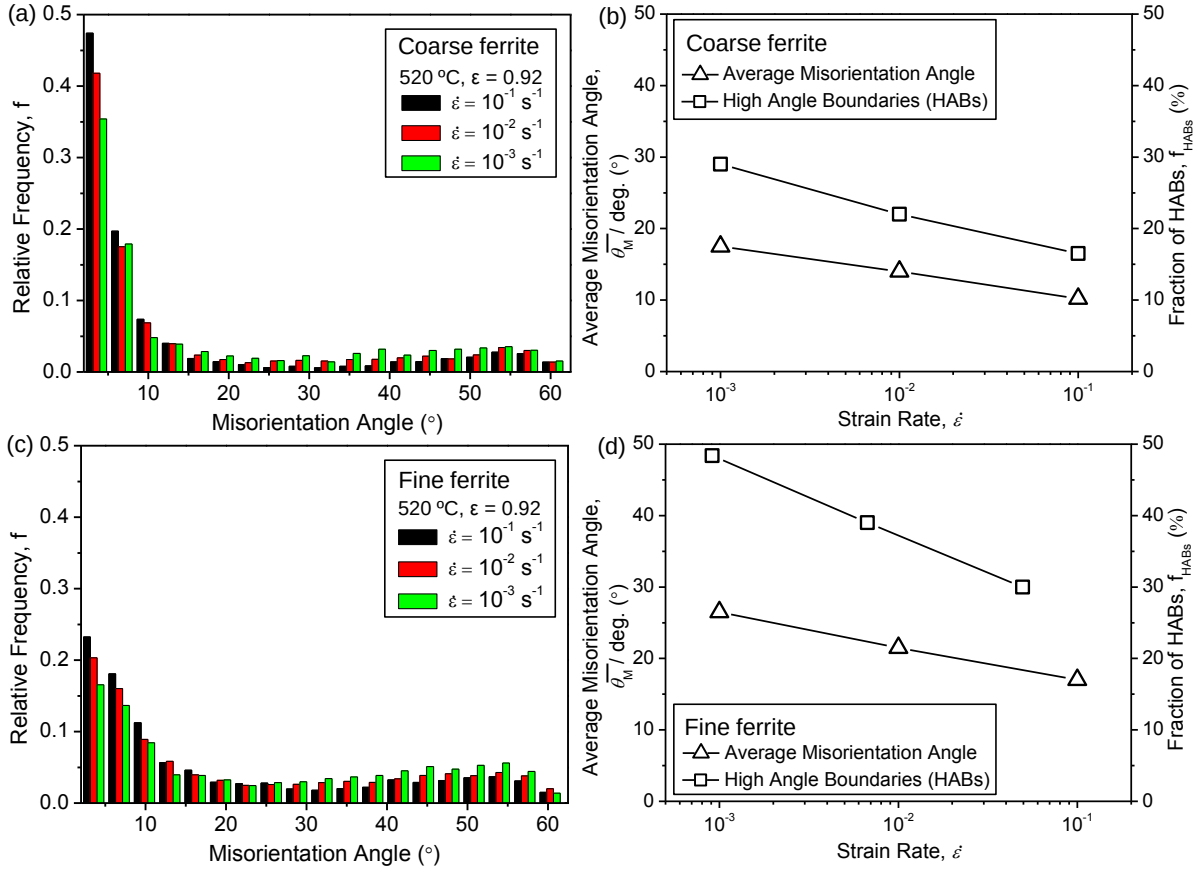


Fig. 4.24 Distributions of boundary misorientation angle (a, c) and changes in average misorientation angle as well as fraction of HABs (b, d) of the specimens with coarse initial ferrite grain of 17 μm (a, b) and fine initial ferrite grain of 6.7 μm (c, d) deformed to a strain of 0.92 at strain rates from 10^{-1} to 10^{-3} s^{-1} at 520 °C.

Figure 4.25 quantitatively summarizes changes in the volume fraction of equiaxed fine grains in the specimens with coarse initial ferrite (a, b) and fine initial ferrite (c, d) deformed at different strain rates from 10^{-1} to 10^{-3} s^{-1} at 520 °C, as a function of strain and time. In the present study, the time at the deformation temperature includes both isothermal holding time (60 s) till the initiation of deformation and deformation time (converted from the applied strain and strain rate). From a viewpoint of strain (**Fig. 4.25(a)** and **(c)**), equiaxed fine grains appear after a critical strain at each strain rate, and the fraction of equiaxed fine grains

increases with straining. The critical strain for the onset of equiaxed fine grains decreases with decreasing the strain rate. From a viewpoint of time (i.e., kinetics of fine grain formation, as shown in **Fig. 4.25(b)** and **(d)**), the formation of equiaxed fine grains is earlier at higher strain rate, and the formation rate of equiaxed fine grains is higher at higher strain rate, which is known from the slope of the curves. However, the final fraction of equiaxed fine grains (at an identical strain) is higher at lower strain rate, indicating that low strain rates enhance the formation of equiaxed fine grains. Compared to the specimens with coarse initial grain size (**Fig. 4.25(a)** and **(b)**), the critical strain for the onset of equiaxed fine grains is decreased (**Fig. 4.25(c)**) in the specimens with finer initial grain size. The finer initial grain size also accelerates the formation rate of equiaxed fine grains.

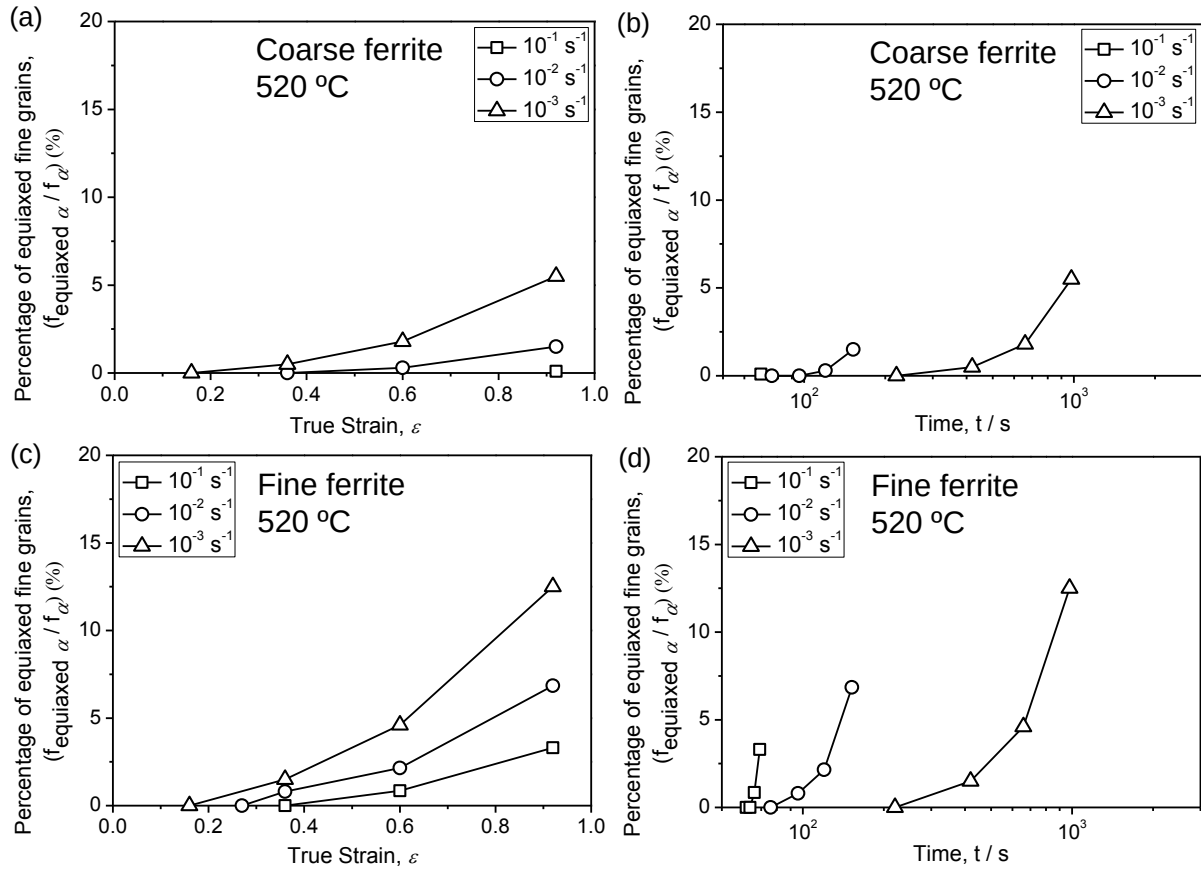


Fig. 4.25 Changes in the volume fraction of equiaxed UFF grains in the specimens with (a, b) coarse initial ferrite grain size of 17 μm and (c, d) fine initial ferrite grain size of 6.7 μm deformed at different strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 °C as a function of strain (a, c) and time (b, d).

It is confirmed that the equiaxed fine grains (with grain sizes around 600 nm) are formed during the deformation of fully ferrite (initial grain size of 6.7 μm or 17 μm) in the route-2. The microstructure evolution of ferrite during deformation suggests a possible formation mechanism of the equiaxed fine grains: the deformation of ferrite results in the formation of LABs inside the ferrite grains (**Fig. 4.18**), and subgrains surrounded by LABs are formed during the deformation. Higher concentration of deformation (or inhomogeneous deformation) would occur near grain boundaries (i.e., high-angle boundaries) of ferrite. The

deformation concentration could increase the misorientation angle of the subgrains (**Fig. 4.21**) and form equiaxed fine grains surrounded by HABs finally (**Fig. 4.22**). The equiaxed fine grains show nearly random crystallographic orientation distributions (textures), which is different from the elongated (deformed) coarse grains (**Fig. 4.20**). Furthermore, fine initial ferrite grain size decreases the critical strain for the formation of equiaxed fine grains and accelerates its kinetics (**Fig. 4.25**). The feature of the ferrite behavior during deformation accords well with that of DRX of ferrite. Therefore, it is reasonable to conclude that DRX of ferrite occurs in the route-2 and the formation mechanism of the equiaxed fine grains is DRX.

4.3.3 Similarity and difference in DRX between dynamically and statically transformed ferrite

4.3.3.1 Similarity

The dynamically recrystallized ferrite obtained in the route-2 shows the similar morphologies as those of the UFF grains obtained in the route-1. The similarities in morphologies of ferrite formed in two different routes are summarized below.

1) In both routes, ferrite microstructure is a mixture of elongated coarse grains and equiaxed UFF grains (i.e., dynamically recrystallized grains), exhibiting a partially DRX structure. The elongated coarse grains have higher GAM values (0.78° in the route-1 or 0.72° in the route-2 at a strain of 1.39), and show strong $\langle 001 \rangle // CA$ and $\langle 111 \rangle // CA$ textures (**Fig. 4.7(b)** and **Fig. 4.20(b)**). The dynamically recrystallized grains have lower average GAM value (0.37° at a strain of 1.39 in both routes) than the elongated coarse grains and show nearly random orientation distributions (**Fig. 4.7(d)** and **Fig. 4.20(d)**).

2) The grain sizes of dynamically recrystallized ferrite keep nearly an identical value during deformation, e.g., 400 nm in the route-1 (**Fig. 4.10**) and 600 nm in the route-2 (**Fig. 4.22**)) at a strain rate of 10^{-2} s^{-1} at 520°C . In both routes, high concentration of deformation (inhomogeneous deformation) occurs particularly near the grain boundaries of ferrite, which increases misorientation angle of the subgrains (**Fig. 4.9** and **Fig. 4.21**) and form equiaxed UFF grains (in the route-1) or equiaxed fine grains (in the route-2) surrounded by HABs.

4.3.3.2 Difference

To quantify the difference of the DRX behaviors between the route-1 (deformation of austenite) and the route-2 (deformation of ferrite), **Fig. 4.26** shows changes in volume fraction of dynamically recrystallized ferrite (UFF ferrite) formed at various strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520°C in (a) the route-1 and (b) the route-2. In both routes the fraction of dynamically recrystallized grains increases with increasing the strain. However, the formation of dynamically recrystallized grains interestingly shows an opposite strain rate dependency between two routes. In the route-1 (**Fig. 4.26(a)**), high strain rates enhance the formation of dynamically recrystallized grains, which is opposite to the tendency in the route-2 (**Fig. 4.26(b)**). As clearly shown in **Fig. 4.26(c)**, in the route-2 (initial ferrite grain size of $6.7 \mu\text{m}$), the fraction of dynamically recrystallized ferrite increases with decreasing the strain rate, which is a common feature of DRX behavior reported before [40, 41]. However, in the route-1 (prior austenite grain size of $125 \mu\text{m}$), the fraction of dynamically recrystallized ferrite becomes higher at higher strain rate, which is totally different from the tendency in conventional DRX described above and must be an interesting characteristic of DRX of dynamically transformed ferrite.

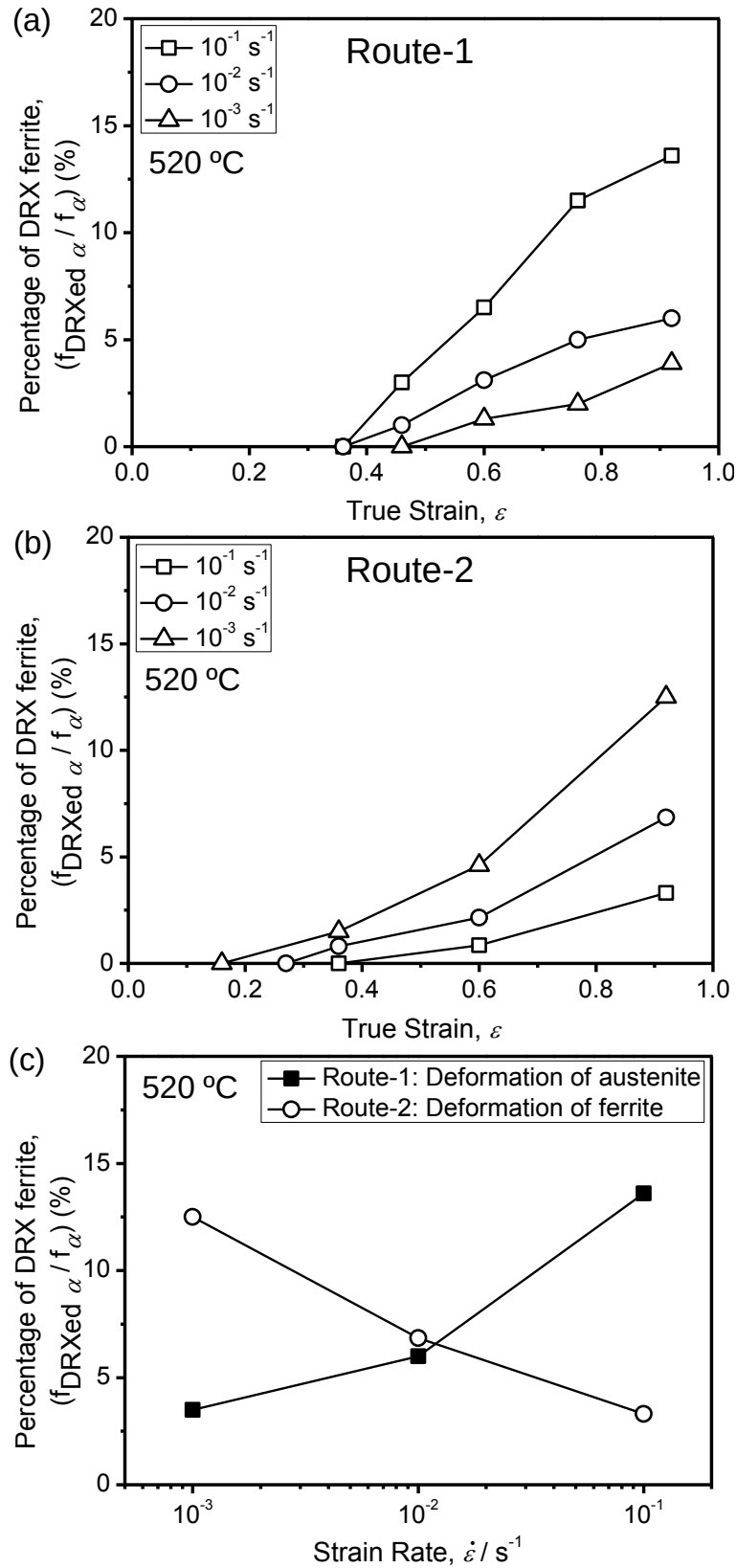


Fig. 4.26 Changes in the volume fraction of dynamically recrystallized grains (UFF grains) obtained in (a) the route-1 and (b) the route-2 at strain rates from 10^{-1} s^{-1} to 10^{-3} s^{-1} at 520 °C, as a function of applied strain. (c) Changes in volume fraction of dynamically recrystallized grains (UFF grains) obtained in two routes as a function of strain rate.

The characteristic strain rate dependency of DRX behavior in the route-1 is probably due to two different phenomena sequentially occurring in the thermomechanical process. Firstly dynamic transformation from austenite to ferrite occurs, and then DRX of ferrite occurs during subsequent deformation. The volume fraction of dynamically transformed ferrite in the dual-phase microstructures (of austenite and ferrite) differs at each strain rate (**Fig. 4.14(a)**). This makes it difficult to evaluate the factors which can affect DRX of ferrite (i.e., deformation concentration and ferrite grain size, as discussed in **Section 4.3.1**). Therefore, it is necessary to choose the specific deformation condition, in which the volume fractions of transformed ferrite are the same at different strain rates, for further investigations.

Figure 4.27 shows changes in (a) volume fraction and (b) grain size of dynamically transformed ferrite in the route-1 at different strain rates at 520 °C. **Figure 4.27(c)** is a combined figure of (a) and (b), in which the ferrite grain size is plotted as a function of ferrite volume fraction. In **Fig. 4.27(c)**, the change in ferrite grain size at different stages of transformation could be clearly demonstrated. With an increase of ferrite volume fraction (i.e., with the progress of dynamic transformation), the ferrite grain size firstly increases and then decreases after a critical volume fraction at each strain rate. As is marked by the arrow in **Fig. 4.27(c)**, the critical volume fraction decreases with increasing the strain rate. As is discussed before, the decrease in grain size is mainly due to the formation of equiaxed UFG grains (i.e., dynamically recrystallized grains). That means the DRX occurs at an earlier

stage of transformation at a higher strain rate. To compare the ferrite microstructures obtained at different strain rates, one condition was selected for further investigation (as marked by red dotted rectangles in **Fig. 4.27(c)**). The volume fraction of dynamically transformed ferrite is 6% at all three strain rates. The required strains ($\Delta\varepsilon$) to obtained 6% ferrite at each strain rate are marked in **Fig. 4.27(a)**. $\Delta\varepsilon$ is calculated from the strain where DT occurs to the strain where 6% ferrite is formed. As is shown in **Fig. 4.27(a)**, similar amount of strain ($\Delta\varepsilon = 0.14, 0.13, 0.1$ at strain rates of $10^{-3}, 10^{-2}$ and 10^{-1} s^{-1} , respectively) is required to obtain 6% ferrite, although the strain for the onset of DT is different at different strain rates.

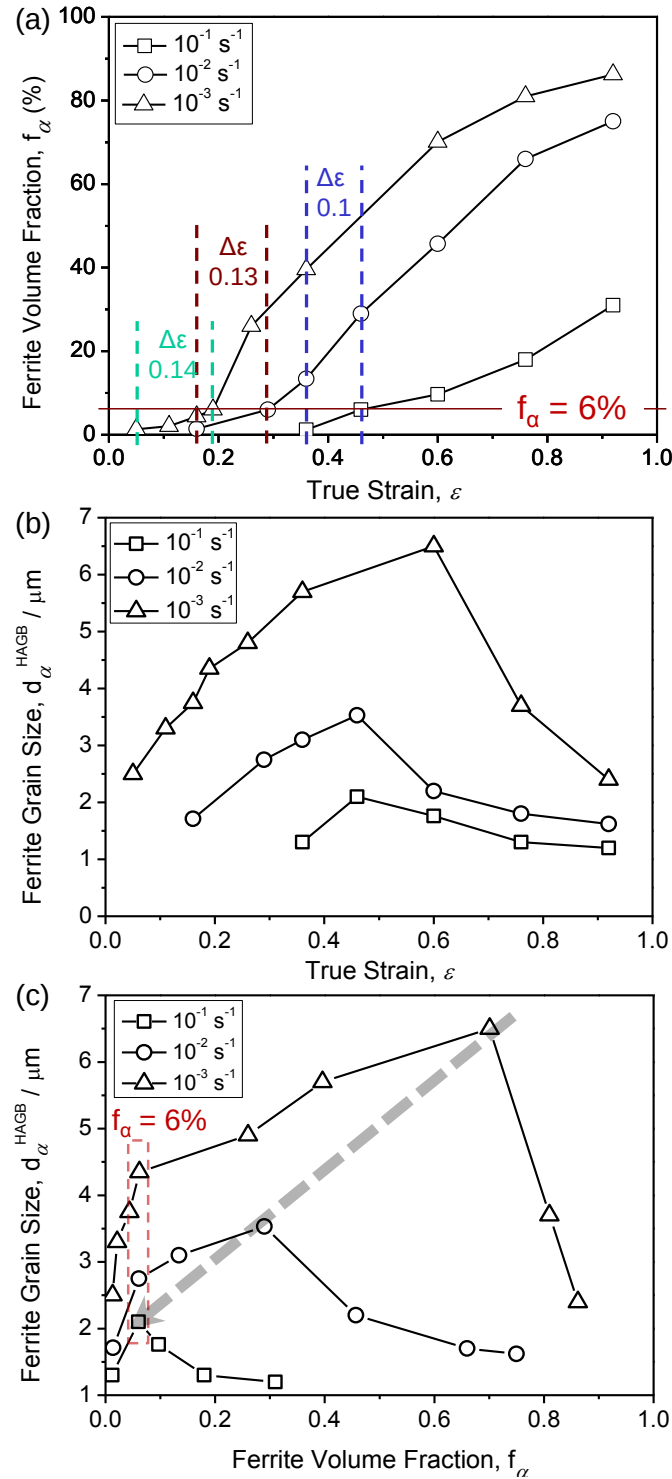


Fig. 4.27 Changes in (a) volume fraction and (b) grain size of dynamically transformed ferrite in the route-1 at different strain rates at 520 °C. (c) A combination of (a) and (b), which shows change in the ferrite grain size as a function of the ferrite volume fraction. Red dotted rectangle in (c) marks the condition for further investigation at different strain rates. $\Delta\epsilon$ is the required strain for obtaining 6% ferrite, which is calculated from the strain where DT occurs to the strain where 6% ferrite is formed. Green, red and blue broken lines in (a) mark $\Delta\epsilon$ at a strain rate of 10^{-3} s^{-1} , 10^{-2} s^{-1} and 10^{-1} s^{-1} , respectively.

Figure 4.28 shows GAM maps of the specimens having 6% (in volume fraction) of ferrite dynamically transformed at strain rates of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at 520°C . The ferrite obtained at a high strain rate (10^{-1} s^{-1} , **Fig. 4.28(a)**) is composed of relatively fine grains and contains a number of LABs. The ferrite obtained at lower strain rates of 10^{-2} s^{-1} (**Fig. 4.28(b)**) and 10^{-3} s^{-1} (**Fig. 4.28(c)**) is mainly composed of coarse grains, indicating more pronounced grain growth. The fraction of LABs within ferrite grains decreases with decreasing the strain rate. The GAM maps, which show the misorientation between a pair of adjacent points within each grain, can be used to evaluate the degree of accumulated strain (amount of lattice defects) in the grains. High GAM value (i.e., high misorientation within the grain) corresponds to larger accumulated strain. Qualitatively speaking, the GAM value of ferrite is higher (dark red color) at a higher strain rate of 10^{-1} s^{-1} , and the red color becomes lighter at lower strain rate. The average GAM values of ferrite obtained at three strain rates are shown in **Fig. 4.28(d)**. The GAM value decreases with decreasing the strain rate. The higher GAM value of ferrite at a higher strain rate indicates larger accumulated strain in ferrite grains, i.e., larger amount of lattice defects.

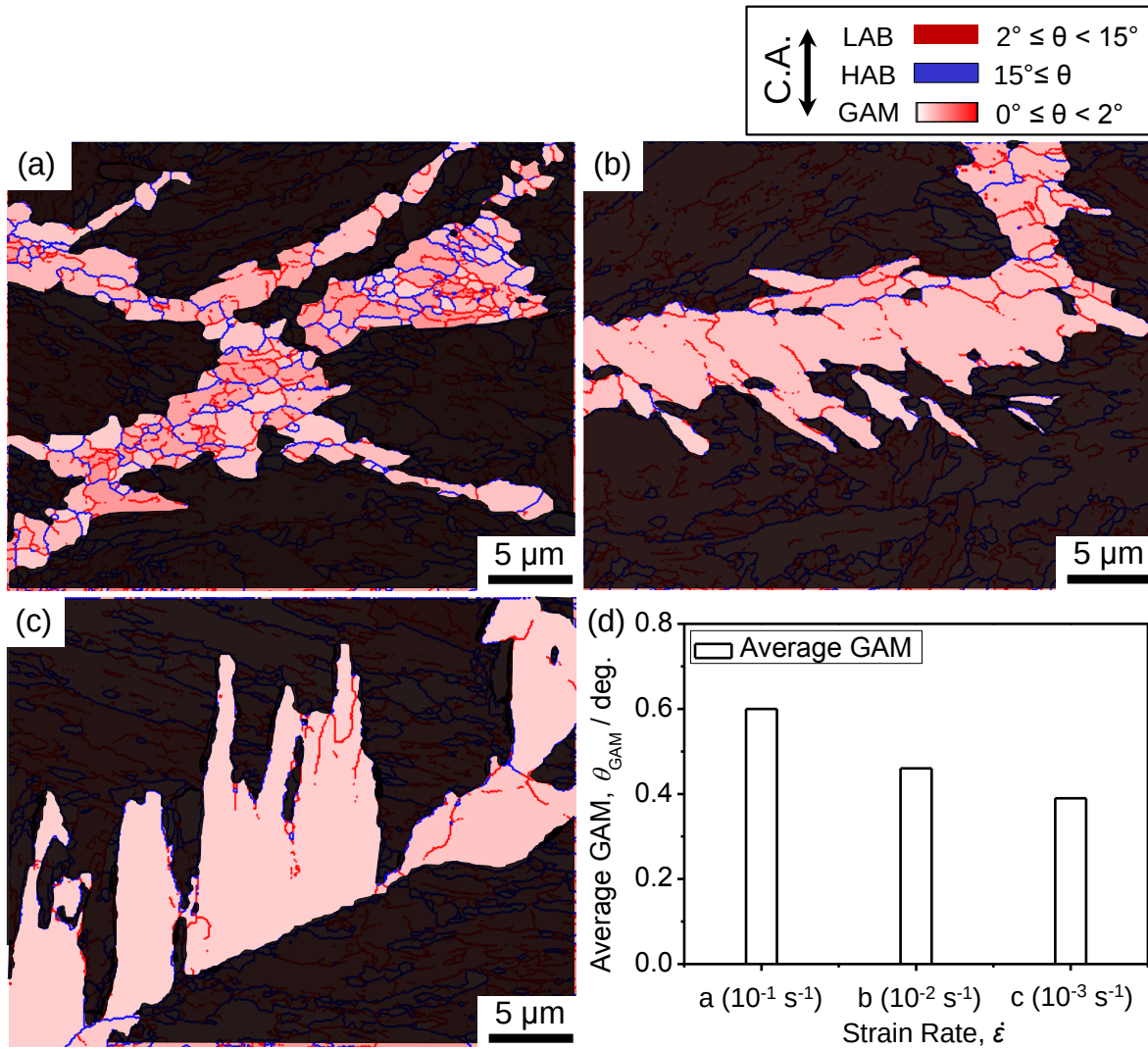


Fig. 4.28 GAM maps of the ferrite dynamically transformed in the route-1 at strain rates of (a) 10^{-1} s^{-1} , (b) 10^{-2} s^{-1} and (c) 10^{-3} s^{-1} at 520 °C. The applied strains are (a) 0.1, (b) 0.13, and (c) 0.14. The volume fraction of dynamically transformed ferrite is the same (6%) in all the specimens. (d) Average GAM values of the ferrite in (a)-(c). Non-ferrite phases (i.e., martensite and/or retained austenite) are painted in black. Low-angle boundaries with misorientation of 2-15° and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Compression axis is along the vertical direction of the maps.

The flow stresses to give 6% volume fraction of ferrite at different strain rates are evaluated in **Fig. 4.29**, which shows true stress-true strain curves of the specimens deformed at different strain rates at 520 °C. The open and solid circles marked on the curves indicate

the initiation of DT and the point of 6% ferrite formation, respectively. It is clearly shown that the applied stress on the dual phase microstructures of austenite and ferrite for achieving 6% ferrite formation is higher at higher strain rate.

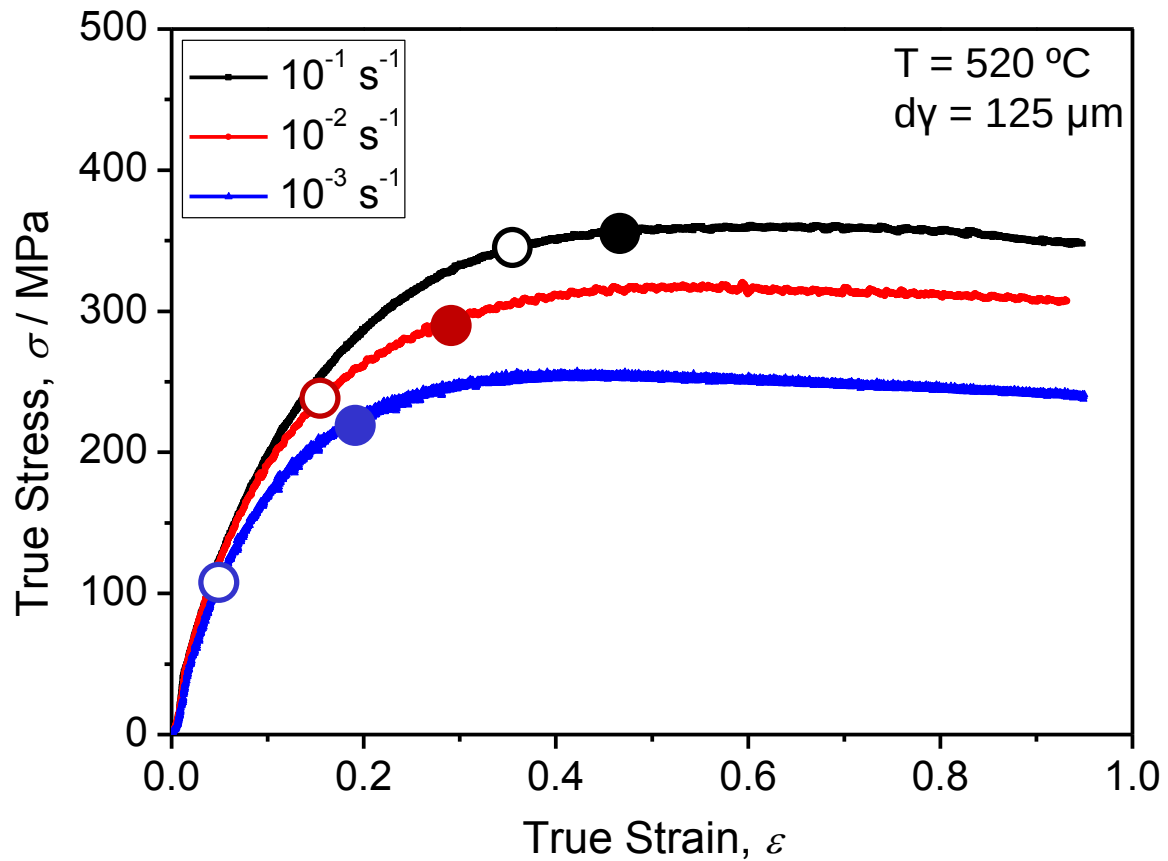


Fig. 4.29 True stress-true strain curves of the specimens with an austenite grain size of 125 μm deformed at different strain rates from 10^{-1} to 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$ (in the route-1). Open and solid circles marked on the curves indicate the initiation of DT and the point of the 6% ferrite formation, respectively.

The factors that could affect the strain rate dependency of DRX behavior in the route-1 are summarized below:

1) Amount of lattice defects in ferrite. Higher strain rate naturally results in larger amount of stored defects. It has also been confirmed that the ferrite obtained at higher strain rate (e.g., 10^{-1} s^{-1} , in **Fig 4.28**) has higher GAM value. That means the fraction of lattice defects (or accumulated strain) in the ferrite is higher. A higher fraction of lattice defects could increase the nucleation density for DRX of ferrite. Therefore, the critical strain for onset of DRX would be decreased and the kinetics of DRX would be accelerated at higher strain rate.

2) Deformation concentration on ferrite. From the onset of DT to the formation of 6% ferrite, the required strain is similar at different strain rates (**Fig. 4.27(a)**). However, the critical strain for the onset of DT is higher at higher strain rate (as shown by open circles in **Fig. 4.29**). That means the austenite is more work-hardened at higher strain rate before the onset of DT. So that austenite is harder at higher strain rate before the formation of ferrite (i.e., 6% of ferrite). Furthermore, during the formation of 6% ferrite, the dynamic recovery (DRV) of austenite and ferrite is different at different strain rates. **Figure 4.30** schematically illustrates how the difference in hardness changes at different strain rates. It is well known that recovery (DRV) of austenite (FCC phase) is much slower than that of ferrite (BCC phase) [42, 43]. During the deformation at high strain rates, the time for DRV is very limited. Therefore, the difference in hardness between austenite and ferrite at that temperature becomes larger with increasing the strain rate. Accordingly, the deformation

can be more concentrated in the softer phase (i.e., ferrite) at higher strain rate. Moreover, the flow stress applied on the dual phase of austenite and ferrite is higher at higher strain rate (Fig. 4.29). The high applied stress may increase the degree of deformation concentration in ferrite at higher strain rate. Therefore, the three aspects, i.e., harder austenite before the formation of ferrite (6%), larger difference in hardness between austenite and ferrite during the formation of ferrite and the higher applied stress, contribute to the higher deformation concentration on ferrite at higher strain rate.

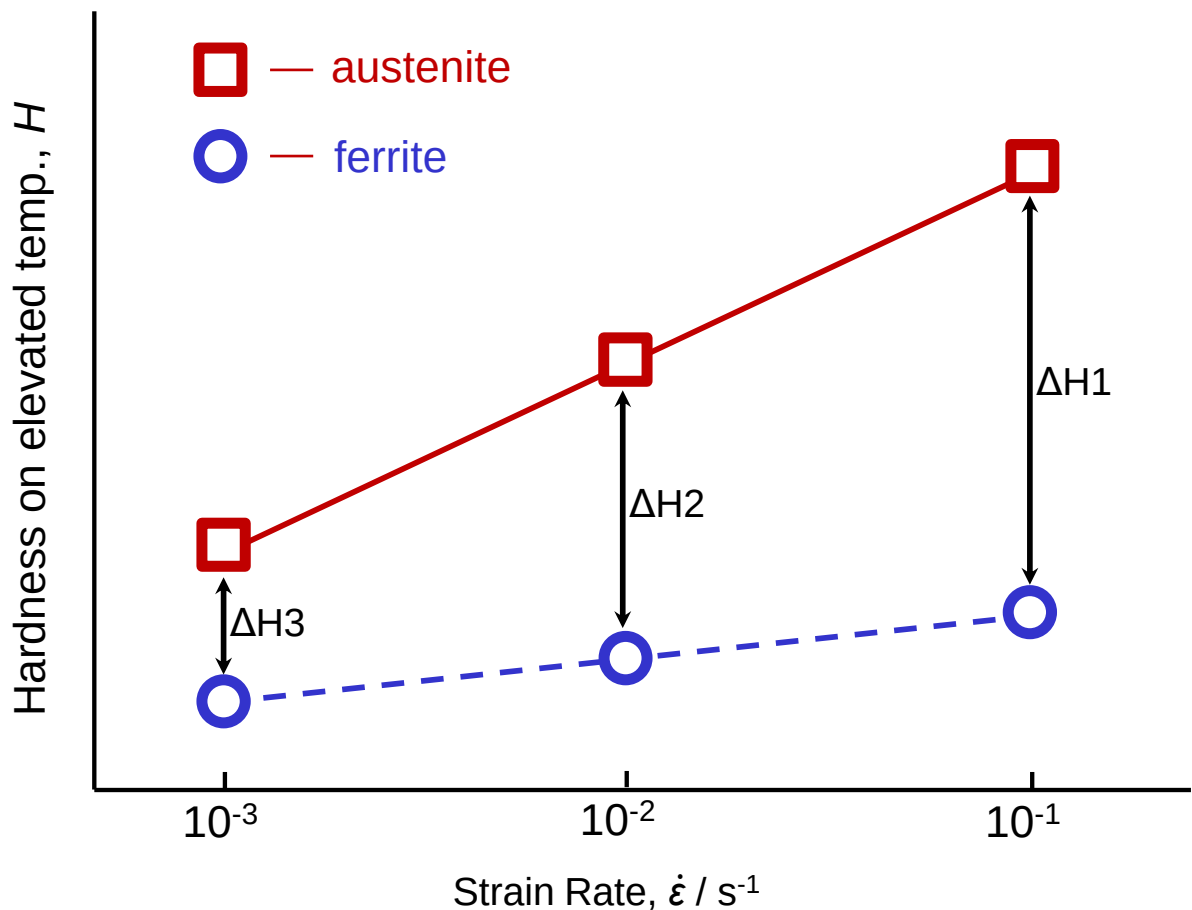


Fig. 4.30 Schematic illustration showing the difference in change of hardness between austenite and transformed ferrite at an elevated temperature in the route-1 as a function of strain rate.

3) Grain size of ferrite. The grain size of dynamically transformed ferrite is smaller at higher strain rate (**Fig. 27(c)** and **Fig. 28**). Decreasing the grain size can decrease the critical strain for the onset of DRX and accelerate its kinetics [35, 36].

Therefore, these three factors, i.e. larger amount of defects in ferrite, higher deformation concentration on ferrite and smaller grain size of ferrite, mainly contribute to the higher volume fraction of dynamically recrystallized ferrite at higher strain rate in route-1(**Fig. 4.26(c)**).

As shown in **Fig. 4.26(c)**, DRX in the route-1 shows a different strain rate dependency from that in the route-2. The different strain rate dependency of DRX in two routes is schematically explained in **Fig. 4.31**. In the illustration corresponding to the route-2 (**Fig. 4.31(a)**), the applied strain and deformation temperature are constant at different strain rates. R_s and R_f indicate the starting and finishing points of dynamic recrystallization, respectively. The flow stress is higher at higher strain rate (**Fig. 4.16**), and more lattice defects are stored in ferrite grains to increase the driving force and the density of nucleation sites for recrystallization. Thus the incubation time of DRX would be shorter at higher strain rate, leading to an earlier initiation of DRX (**Fig. 4.25(b)** and **(d)**). At the same strain, lower strain rate allows longer time (**Fig. 4.31(a)**) for the progress of recrystallization. Since the amount of stored energy is lower at lower strain rate due to a progress of recovery during longer deformation time, the nucleation density of DRX is smaller so that the grain growth of

recrystallized grains is enhanced at lower strain rate. Therefore, lower strain rate can lead to higher volume fraction (**Fig. 4.25(b)** and **(d)**) of dynamically recrystallized grains with larger grain size (**Fig. 4.23(f)**). On the other hand, the strain rate dependency of DRX in the route-1 (**Fig. 4.31(b)**) is different from that in the route-2, because dynamic transformation behaviors control subsequent DRX behaviors. In **Fig. 4.31(b)**, F_s and F_f indicate starting and finishing points of dynamic transformation, respectively. R_s and R_f indicate starting and finishing points of recrystallization, respectively. The applied strain and deformation temperature are constant at different strain rates. During deformation at higher strain rate, larger amount of lattice defects are stored in austenite, leading to higher nucleation density for ferrite transformation. The higher density of nucleation sites leads to faster transformation kinetics and formation of finer ferrite grains by impingement effect between ferrite grains. Deformation concentration into softer ferrite may become larger at higher strain rate due to different DRV behaviors in austenite and ferrite (**Fig. 4.30**). The finer ferrite grains and more concentrated deformation in ferrite lead to the earlier initiation and faster kinetics of DRX (**Fig. 4.31(b)**). Consequently, larger fraction of dynamically recrystallized grains is obtained at higher strain rate (**Fig. 26(c)**). This is the mechanism for the characteristic strain rate dependence of DRX kinetics in the route-1, which is totally opposite to the conventional strain rate dependence of DRX in the route-2.

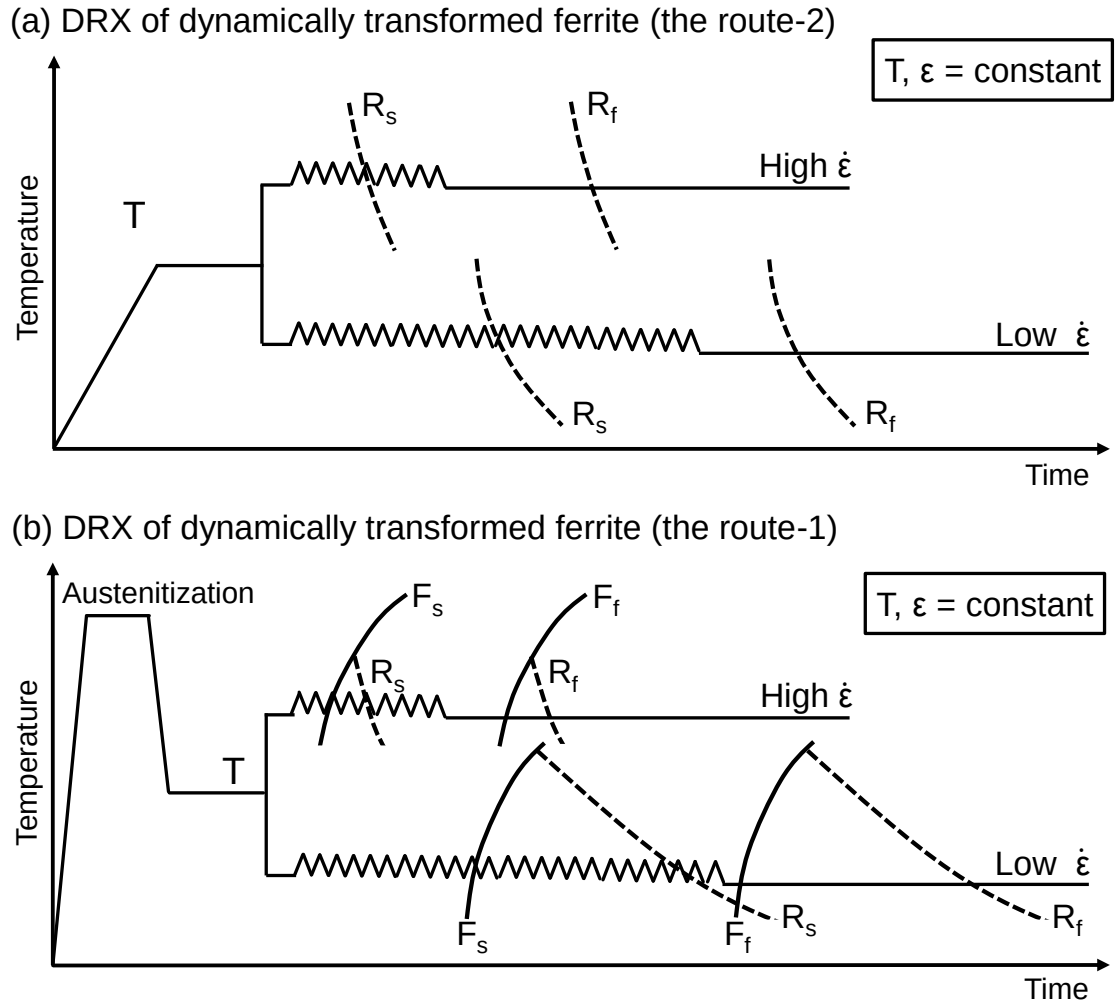


Fig. 4.31 Schematic illustration showing strain rate dependency of DRX behaviors in (a) route-2 and (b) route-1. DRX of statically transformed ferrite and DRX of dynamically transformed ferrite occur in route-2 and route-1, respectively. R_s and R_f indicate starting and finishing points of recrystallization, respectively. F_s and F_f indicate starting and finishing points of dynamic transformation, respectively. The applied strain and deformation temperature are constant in this illustration.

The difference in strain rate dependency of DRX between the route-1 and the route-2 found in **Fig. 4.26** has been clarified. However, there is still one unclear issue about the DRX behavior in the route-1. As illustrated in **Fig. 4.31(b)**, the dynamic transformation kinetics changes at different strain rates. Such a difference in the transformation kinetics may have an important influence on the DRX behavior. It is unclear whether the strain rate

dependency of DRX in the route-1 could be changed by transformation kinetics. Therefore, the strain rate dependency of DRX in the route-1 is further investigated under different transformation kinetics.

There are mainly two different ways to change the dynamic transformation kinetics, i.e., changing deformation temperature or changing austenite grain size. Since temperature can greatly affect DRX behaviors as well [44], it is not a good idea to change the deformation temperature. Therefore, the transformation kinetics is changed by changing austenite grain size. As was discussed in **Chapter 2**, fine austenite grain size leads to faster transformation kinetics. **Figure 4.32** shows EBSD grain boundary maps of the specimens with austenite grain sizes of (a)-(c) 450 μm and (d)-(f) 10 μm deformed in the route-1 to a strain of 0.92 at different strain rates at 520 $^{\circ}\text{C}$. In the case of coarse austenite grain size (450 μm), equiaxed UFG grains formed by DRX are observed at a high strain rate of 10^{-1} s^{-1} (**Fig. 4.32(a)**). It is really hard to find dynamically recrystallized grains at lower strain rates, i.e., 10^{-2} s^{-1} and 10^{-3} s^{-1} . Instead, there are a lot of subgrains surrounded by LABs in the deformed ferrite grains in **Fig. 4.32(b)** and **(c)**. In this case (slow dynamic transformation kinetics from coarse austenite), high strain rate enhances DRX of ferrite. In the case of fine austenite grain size (10 μm), dynamically recrystallized grains are observed at all strain rates (**Fig. 4.32(d)-(f)**). The fraction of dynamically recrystallized grains increases with decreasing the strain rate. That is, low strain rate enhances DRX of ferrite in the case of fast transformation kinetics

from fine austenite. It is therefore concluded that the strain rate dependency of DRX is totally changed by changing austenite grain size.

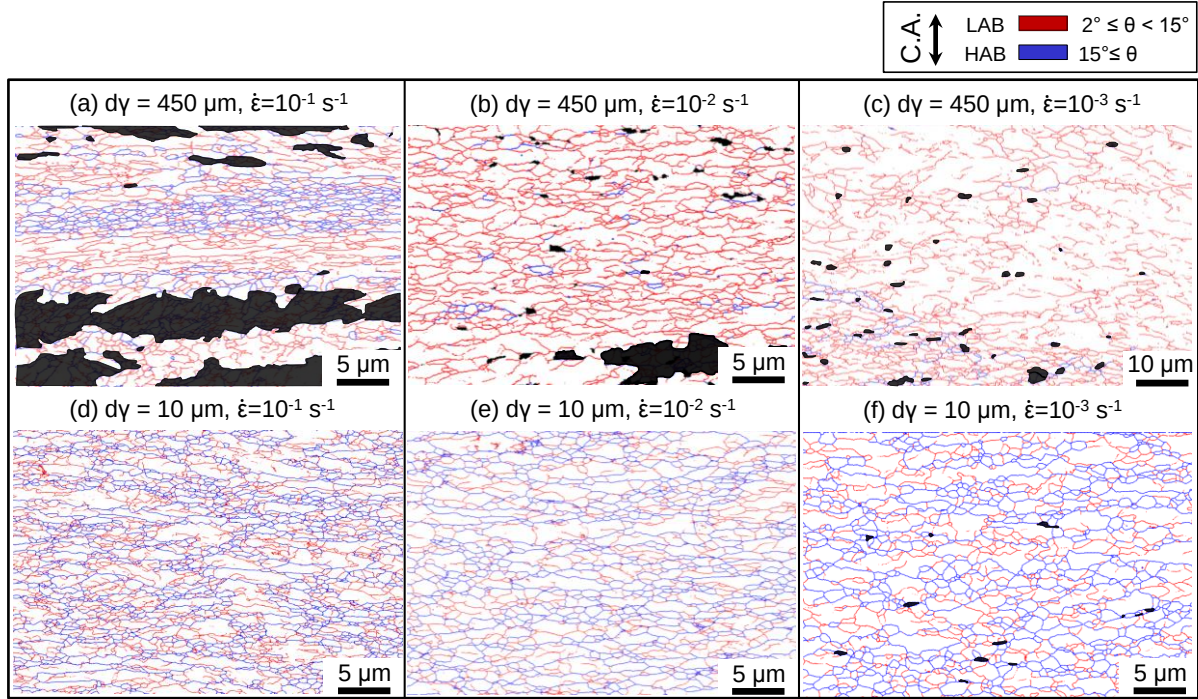


Fig. 4.32 EBSD grain boundary maps of the specimens with austenite grain sizes of 450 μm (a-c) and 10 μm (d-f) deformed to a strain of 0.92 at a strain rate of (a, d) 10^{-1} s^{-1} , (b, e) 10^{-2} s^{-1} and (c, f) 10^{-3} s^{-1} at 520 $^{\circ}\text{C}$ in the route-1. Non-ferrite phase (i.e., martensite and/or retained austenite) were painted in black. Low-angle boundaries with misorientation of $2-15^{\circ}$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Compression axis is along the vertical direction of the maps.

Figure 4.33 summarizes the strain rate dependence of (a) volume fraction of dynamically transformed ferrite and (b) volume fraction of dynamically recrystallized ferrite in the specimens with different austenite grain sizes deformed to an identical strain of 0.92 at 520 $^{\circ}\text{C}$ (in the route-1). The open reverse-triangle in (b) indicates the volume fraction of dynamically recrystallized ferrite formed in the route-2 at the same temperature and strain

rates. At each strain rate (**Fig. 4.33(a)**), the kinetics of DT is greatly enhanced by decreasing austenite grain size, which has been already confirmed in **Chapter 2**. In the specimens with relatively coarse austenite grain size (i.e., 450 μm and 125 μm), the volume fraction of dynamically recrystallized grains increases with increasing the strain rate (**Fig. 4.33(b)**). With decreasing the austenite grain size to 10 μm , on the contrary, the volume fraction of dynamically recrystallized grains decreases with increasing the strain rate, which shows the same strain rate dependency as that in the route-2 (indicated by open reverse-triangle in **Fig. 4.33(b)**). The fast kinetics of transformation (from fine austenite grains) leads to two different possibilities which may affect the subsequent DRX behavior. Firstly, the fast transformation kinetics can lead to an early impingement between ferrite grains [40] at all the three strain rates. Therefore, the difference in ferrite grain size between different strain rates might become smaller. Secondly, if austenite quickly transforms to complete ferrite at an early stage of deformation, most of the plastic strain would be applied on ferrite in compression deformation. In that case, the difference of the strain concentration effect on ferrite between different strain rates becomes weaker. Therefore, when the dynamic transformation completes fast, both possibilities make the DRX behavior similar to that in the route-2. The phenomenon that the strain rate dependency of DRX could be changed by transformation kinetics may provide some inspirations to the future research on ferrite grain refinement by DT and DRX.

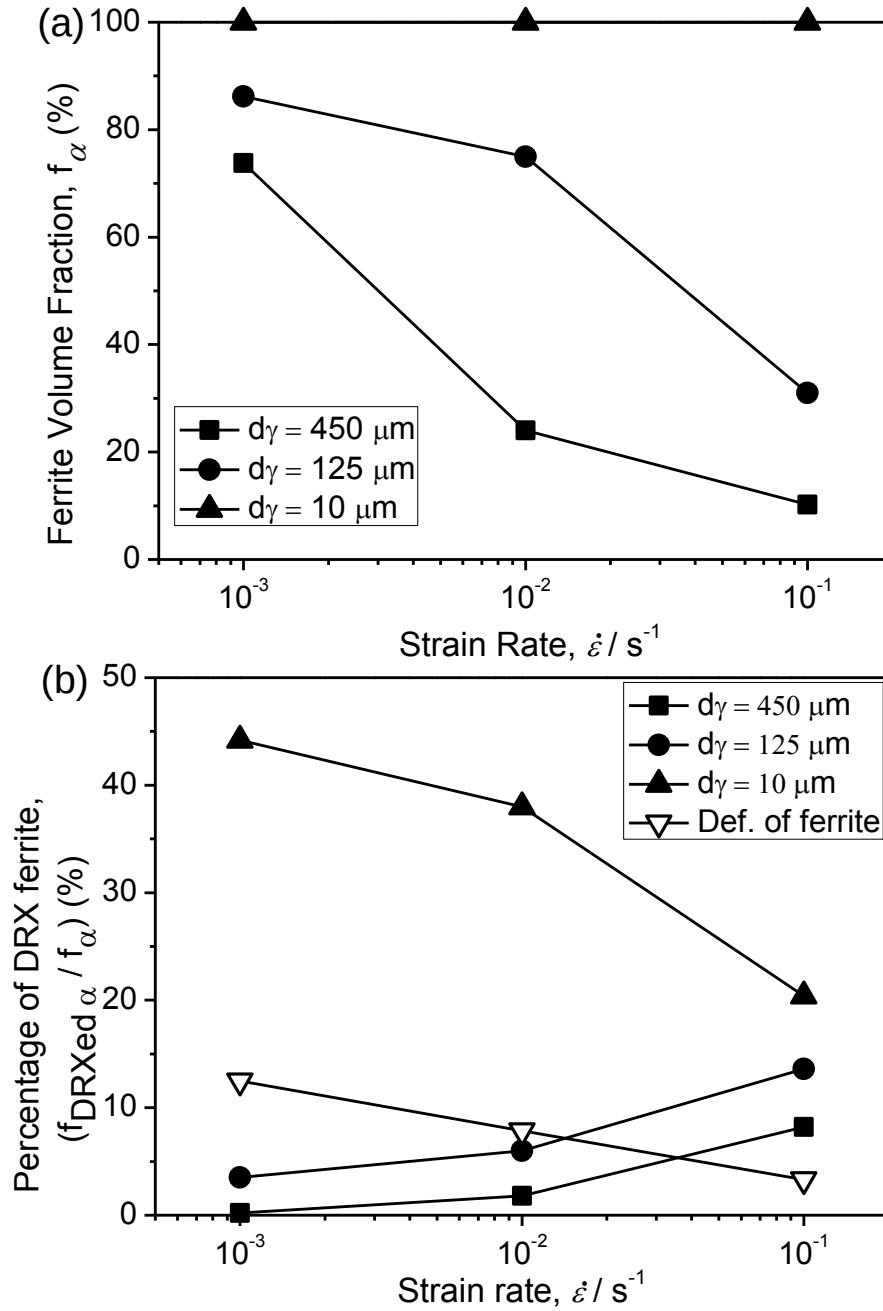


Fig. 4.33 Strain rate dependence of (a) volume fraction of dynamically transformed ferrite and (b) volume fraction of dynamically recrystallized ferrite in the specimens with different austenite grain sizes from 450 μm to 10 μm deformed in the route-1 to an identical strain of 0.92 at 520 $^\circ\text{C}$ as a function of strain rate. The open reverse-triangle in (b) represents the volume fraction of dynamically recrystallized ferrite in the specimens with an ferrite grain size of 6.7 μm deformed in the route-2 at the same temperature and strain rates as those in the route-1.

4.3.4 DRX of ferrite formed by DT under different thermomechanical conditions

It was concluded in **Chapter 2** that DT is affected by thermomechanical parameters (i.e., strain, strain rate, austenite grain size or deformation temperature). It is reasonable to think that DRX of dynamically transformed ferrite will also be affected by these parameters. The effects of strain and strain rate on DRX of dynamically transformed ferrite have been systematically investigated in **Section 4.3.1** and **4.3.3**. In this section, the effects of other two important parameters (i.e., austenite grain size and deformation temperature) on the DRX of dynamically transformed ferrite will be particularly examined.

4.3.4.1 Effect of austenite grain size on DRX of dynamically transformed ferrite

For investigating the effects of austenite grain size on DRX of ferrite, deformation temperature and strain rate should be constant. According to the results in **Chapter 2**, the transformation behavior in different austenite grains could be clearly revealed at a strain rate of 10^{-2} s^{-1} at 520°C . Therefore, the recrystallization behavior of ferrite at this strain rate and temperature is particularly examined.

Figure 4.34 shows true stress-true strain curve of the specimens with different austenite grain sizes from $450 \mu\text{m}$ to $10 \mu\text{m}$ deformed at a strain rate of 10^{-2} s^{-1} at 520°C . The curve of coarse austenite (grain size of $450 \mu\text{m}$) shows a typically power-law type work-hardening.

The curve of medium austenite (125 μm) displays early strain-hardening followed by a decreasing region, indicating the occurrence of DT and/or DRX of ferrite according to the previous discussion in **Chapter 2 and Chapter 4**. The softening in true stress-true strain curve is enhanced during the deformation of fine austenite (10 μm).

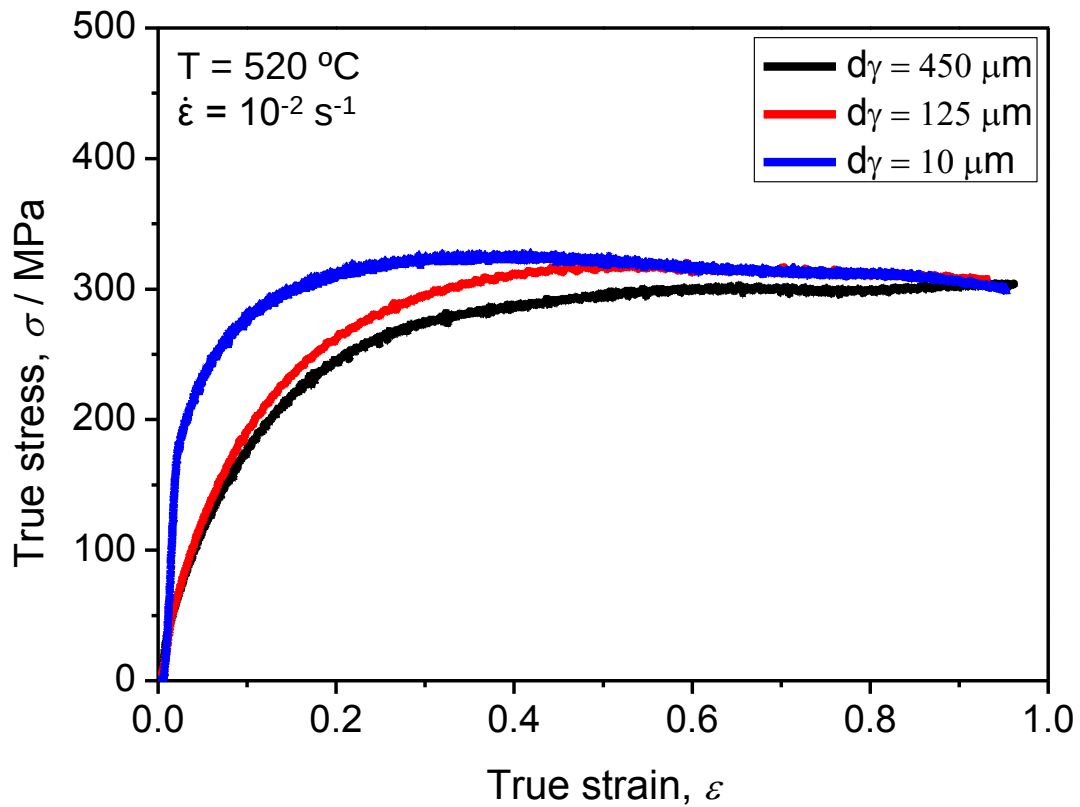


Fig. 4.34 True stress-true strain curves of the specimens with different austenite sizes deformed at a strain rate of 10^{-2} s^{-1} at 520°C .

The effect of austenite grain size on the formation of ferrite is shown in OM images in **Fig. 4.35**, which reveals microstructures in the specimens with different austenite grain sizes from 450 μm to 10 μm deformed to different strains from 0.16 to 0.92 at a strain rate of 10^{-2} s^{-1} at 520°C (corresponding to **Fig. 4.34**). “F” and “M” indicate ferrite and martensite,

respectively, which is the case to all the images in this chapter hereafter. In the case of coarse austenite grain size (450 μm , **Fig. 4.35(a)-(d)**), the progress of DT is relatively slow. A very small amount of ferrite was observed along prior austenite grain boundaries (PAGBs) at a strain of 0.36. At the same strain of 0.36, the volume fraction of dynamically transformed ferrite increased markedly with decreasing the austenite grain size. This tendency could also be seen in the specimens deformed to other strains. Therefore, it could be concluded that the progress of DT is accelerated by decreasing austenite grain size.

$T = 520\text{ }^{\circ}\text{C}$, $\dot{\epsilon} = 10^{-2}\text{ s}^{-1}$

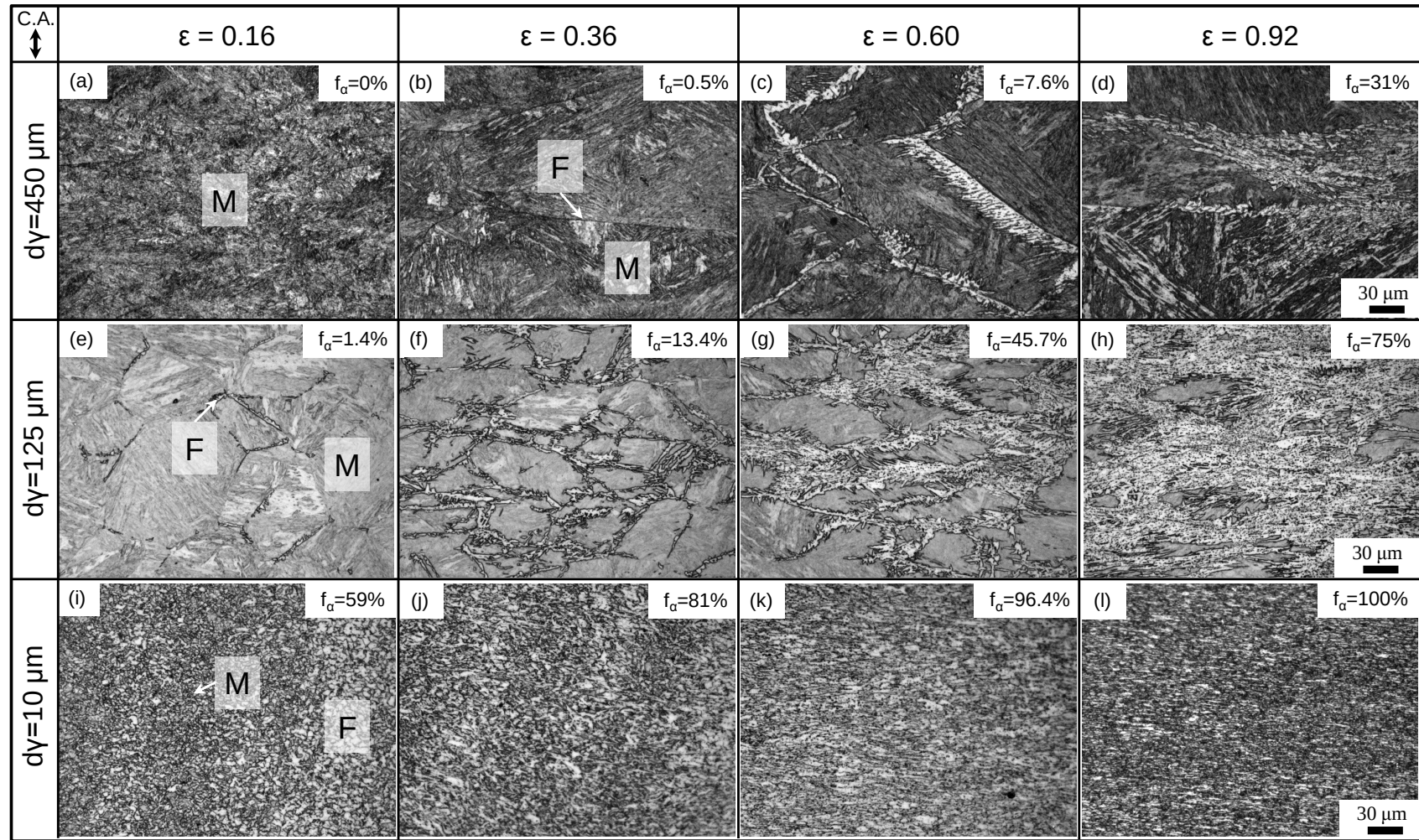


Fig. 4.35 OM images of the specimens with different austenite grain sizes from 450 μm to 10 μm deformed to different strains from 0.16 to 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. “F” and “M” indicate ferrite and martensite, respectively. The volume fractions of ferrite are shown on the top right corners of the micrographs, respectively. Compression axis is along the vertical direction of the image.

The detailed microstructural features of ferrite are further examined in **Fig. 4.36**, which reveals EBSD grain boundary maps of the microstructures corresponding to **Fig. 4.35(d, h, l)**. Non-ferrite phases (i.e. martensite and/or retained austenite) are painted in black, which is the case to all EBSD maps in this chapter hereafter. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. In the case of coarse austenite (450 μm, **Fig. 4.36(a)**), the ferrite exhibit a coarse and elongated morphology and contains a large amount of subgrains surrounded by LABs (as marked by green arrows). Equiaxed UFF grains surrounded by HABs could be observed (as marked by a black arrow), but the fraction is very small. According to the discussion in **Chapter 4**, the equiaxed UFF grains observed in **Fig. 4.36** are formed due to DRX. In the case of medium austenite (125 μm, **Fig. 4.36(b)**), ferrite consist of equiaxed UFF grains (i.e., dynamically recrystallized grains) and elongated coarse grains containing LABs. The fraction of the dynamically recrystallized grains significantly increases in the case of fine austenite (10 μm, **Fig. 4.36(c)**). It could be concluded from **Fig. 4.36** that with decreasing the austenite grain size, the DRX of transformed ferrite is enhanced.

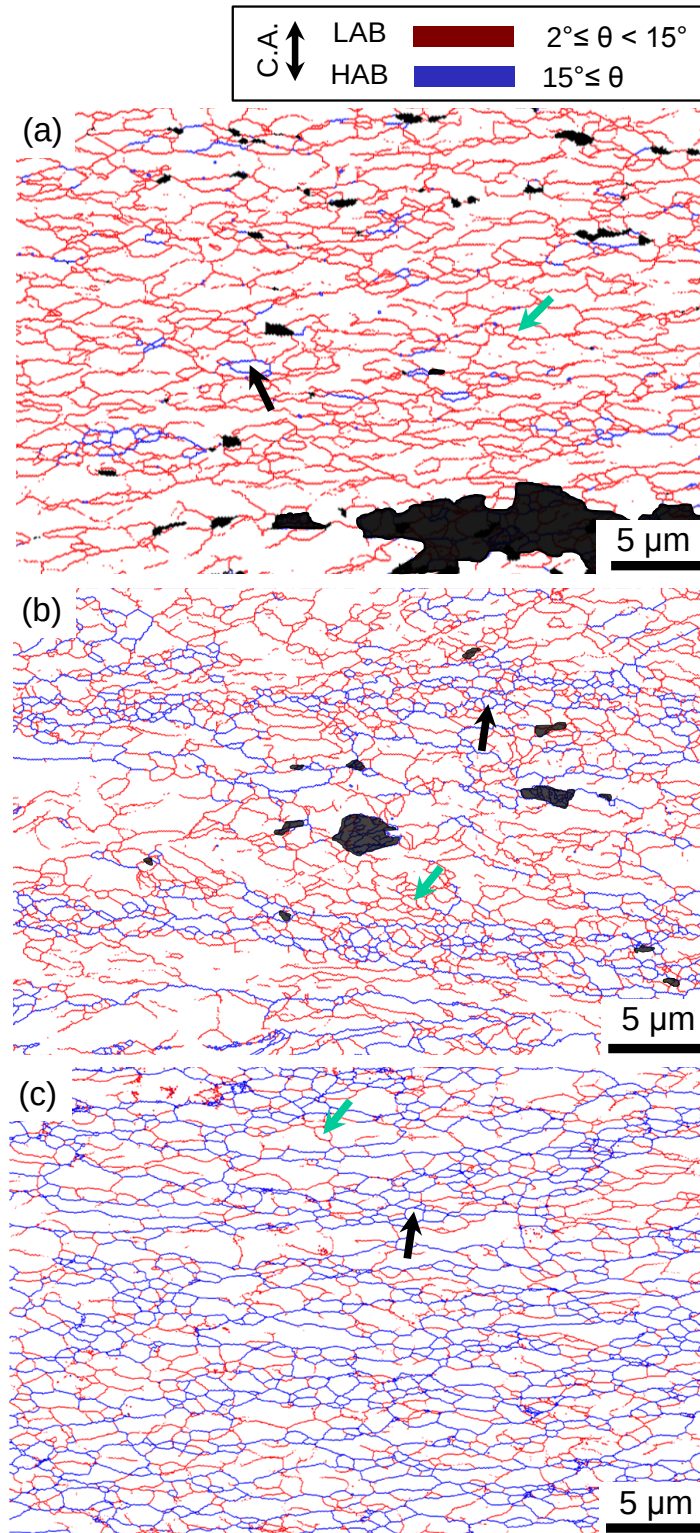


Fig. 4.36 Grain boundary maps of the specimens with different austenite grain sizes of (a) 450 μm , (b) 125 μm and (c) 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at 520 $^\circ\text{C}$. Non-ferrite phases (i.e. martensite and/or retained austenite) are covered in black. The low-angle boundaries with misorientation of 2-15 $^\circ$ and the high-angle boundaries with misorientation above 15 $^\circ$ are drawn in red and blue lines, respectively. Compression axis is along the vertical direction of the maps.

The other features of the microstructure in **Fig. 4.36** are shown in **Fig. 4.37**, which reveals distributions of grain size (a, c, e) and misorientation angle (b, d, f) of the ferrite transformed from coarse austenite (a, b), medium austenite (c, d) and fine austenite (e, f). The grain size of ferrite in **Fig. 4.37(a)** is large and shows a very broad distribution from 1.4 μm to 46.5 μm . The mean grain size of the ferrite is 24 μm . The fraction of LABs in ferrite grains is very high (97% in **Fig. 4.37(b)**). With decreasing the austenite grain size to 125 μm (**Fig. 4.37(c)**) and 10 μm (**Fig. 4.37 (e)**), the grain size of ferrite becomes smaller (1.62 μm in **Fig. 4.37(c)** and 0.93 μm in **Fig. 4.37(e)**) and the distribution of grain size becomes much narrower. The fraction of LABs in ferrite decreases with decreasing austenite grain size (**Fig. 4.37(d)**) and **Fig. 4.37(f)**). The results indicate that fine austenite grain size enhances the formation of HABs. Consequently, the mean grain size of ferrite is decreased.

Figure 4.38 shows changes in the volume fraction of (a) dynamically transformed ferrite and (b) dynamically recrystallized ferrite in the specimens with different austenite grain sizes deformed at a strain rate of 10^{-2} s^{-1} at 520 $^{\circ}\text{C}$. With decreasing the austenite grain size, the formation of ferrite is greatly enhanced (**Fig. 4.38(a)**). Especially in the case of fine austenite (10 μm), the transformation reaches the completion after applying a strain of 0.92. The critical strain for the onset of DRX decreases with decreasing the austenite grain size (**Fig. 4.38(b)**). The fraction of dynamically recrystallized ferrite increases with an increase of the strain. The changes in volume fraction of dynamically transformed ferrite and

dynamically recrystallized ferrite are plotted as a function of austenite grain size (**Fig. 4.38(c)**). It is clearly shown that fine austenite leads to higher fraction of transformed ferrite.

Correspondingly, the faction of dynamically recrystallized grains increases.

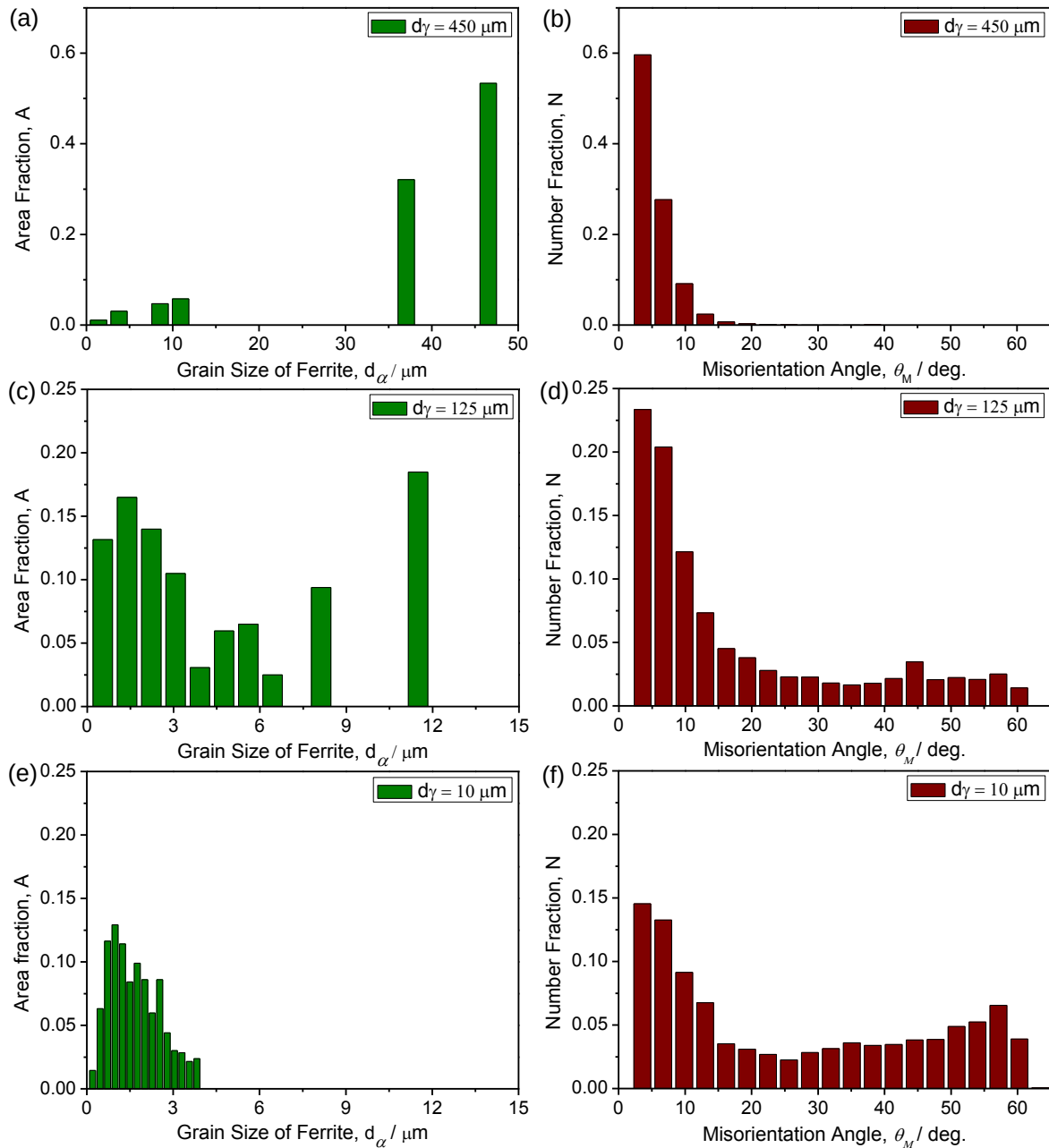


Fig. 4.37 Distributions of grain size (a, c, e) and misorientation angle (b, d, f) of the ferrite transformed from coarse grain-sized austenite (a,b), medium grain-sized austenite (c, d) and fine grain-sized austenite (e, f).

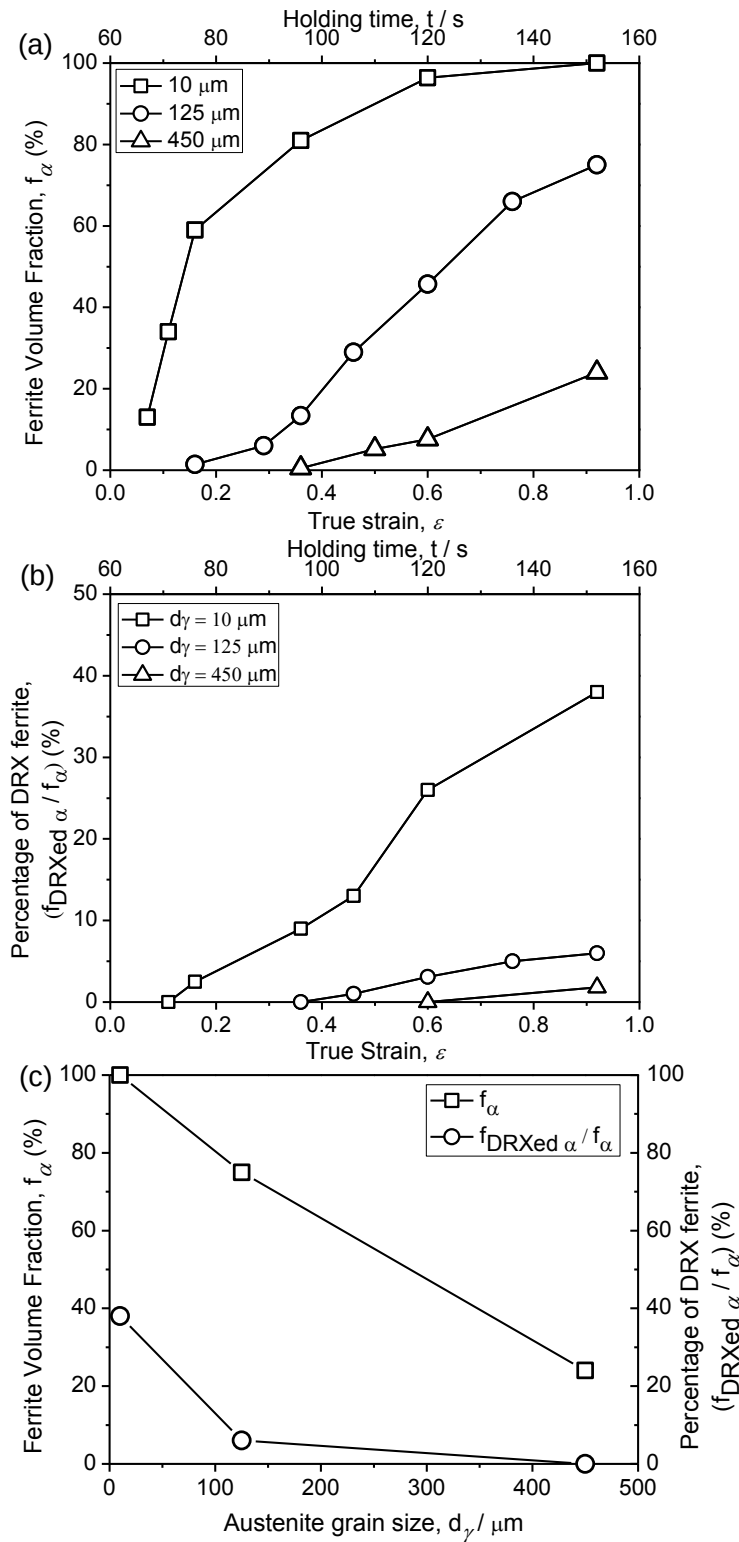


Fig. 4.38 Changes in (a) volume fraction of ferrite and (b) volume fraction of dynamically recrystallized ferrite in the specimens with different austenite grain sizes deformed at a strain rate of 10^{-2} s^{-1} at 520°C . The holding time on the upper horizontal axis of (a) and (b) includes the holding time till the onset of deformation (60 s) and the time converted from strain at a constant strain rate (10^{-2} s^{-1}). (c) Changes in the volume fraction of dynamically transformed ferrite and dynamically recrystallized ferrite as a function of austenite grain size.

The effect of prior austenite grain size on DRX of ferrite can be schematically illustrated in **Fig. 4.39**. The applied strain, strain rate and deformation temperature are constant in this illustration. F_s and F_f represent the starting and finishing points of ferrite transformation, respectively. R_s and R_f represent the starting and finishing points of ferrite recrystallization, respectively. As is well known, austenite grain boundaries are the most preferential nucleation sites for ferrite. In the case of coarse austenite (**Fig. 4.39(a)**), the density of nucleation sites for ferrite transformation is small, resulting in slow transformation kinetics. DT starts (F_s) at relatively late stage of deformation. Without sufficient impingement effect between ferrite grains, the transformed ferrite experience pronounced grain growth (**Fig. 4.36(a)**). Consequently, the start of DRX of ferrite (R_s) is retarded. The ferrite structure exhibits coarse and elongated grains containing large amount of LABs, i.e., mainly a deformed structure (**Fig. 4.36(a)**). In the case of fine austenite (**Fig. 4.39(b)**), larger fraction of austenite grain boundaries increases the nucleation density for ferrite, resulting in earlier start of transformation (F_s) and faster transformation kinetics (**Fig. 4.38(a)**). The fast transformation kinetics results in finer ferrite due to the impingement effect between ferrite grains. The finer ferrite grain size decreases the critical strain for the onset of DRX, therefore the recrystallization of ferrite starts (R_s) at much earlier stage of deformation. Furthermore, when the transformation kinetics is fast, more strain would be concentrated on ferrite from an early stage of deformation. As is shown in the true stress-true strain curves

(**Fig. 4.34**), the slope of the stress-strain curve of the specimen with fine austenite grain size is very sharp. It means that austenite (FCC) quickly transformed to ferrite (BCC) and thus the strain concentrates on ferrite at an early stage of deformation. Therefore, the high strain concentration on fine ferrite grains enhances the formation of dynamically recrystallized grains (**Fig. 4.38(b)**), i.e., accelerate the kinetics of DRX (**Fig. 5.39(b)**).

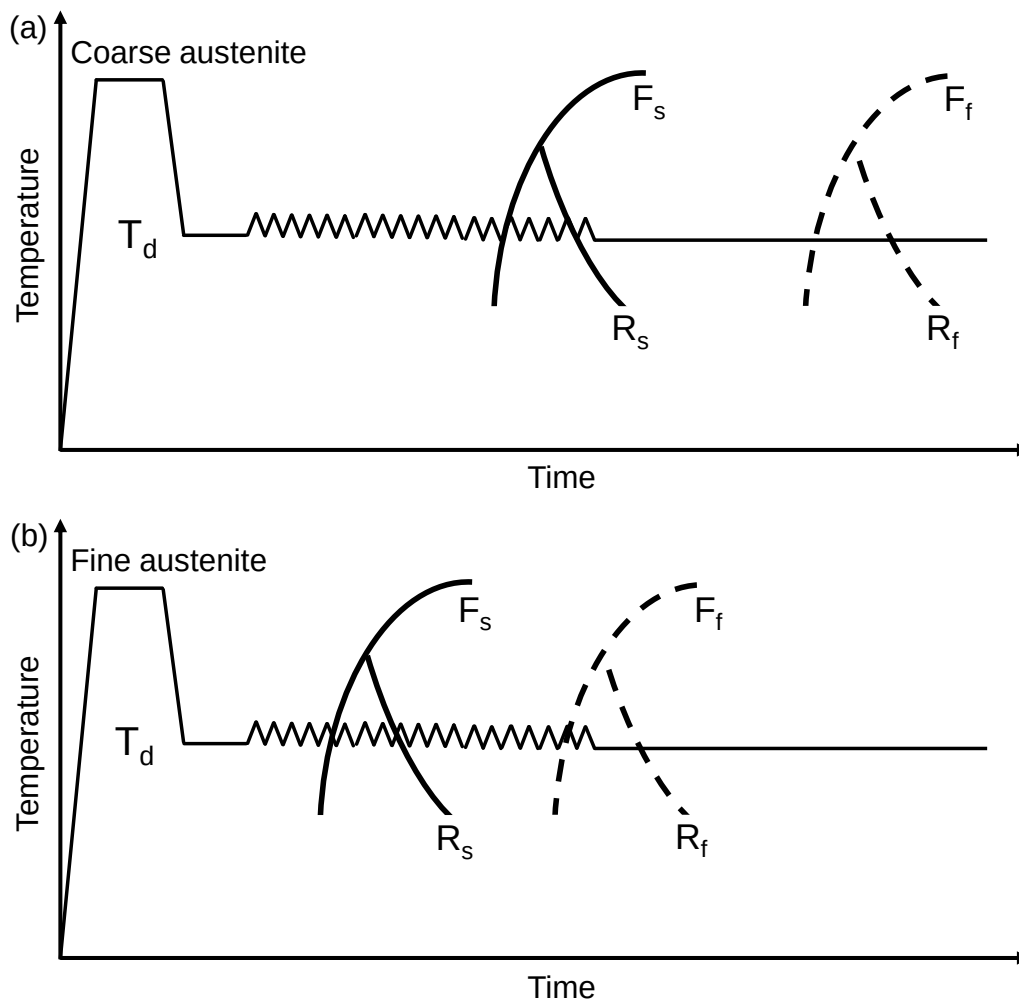


Fig. 4.39 Schematic illustration showing the effect of prior austenite grain size on the DRX of ferrite. F_s and F_f represent the starting and finishing points of ferrite transformation, respectively. R_s and R_f represent the starting and finishing points of ferrite recrystallization, respectively. The applied strain, strain rate and deformation temperature are constant in this illustration.

4.3.4.2 Effect of temperature on DRX of dynamically transformed ferrite

For investigating the effect of deformation temperature on DRX of dynamically transformed ferrite, the applied strain (0.92), strain rate (10^{-2} s^{-1}) and austenite grain size $10 \text{ }\mu\text{m}$ are constant in this section.

Figure 4.40 shows SEM images of the specimens with an austenite grain size of $10 \text{ }\mu\text{m}$ deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (a) $600 \text{ }^{\circ}\text{C}$, (b) $560 \text{ }^{\circ}\text{C}$, (c) $520 \text{ }^{\circ}\text{C}$ and (d) $440 \text{ }^{\circ}\text{C}$. “F” and “M” indicate ferrite and martensite, respectively. At $600 \text{ }^{\circ}\text{C}$, only a small amount (around 16%) of fine equiaxed grains is formed along PAGBs. At $560 \text{ }^{\circ}\text{C}$, the fraction of ferrite significantly increases. The ferrite grains become much coarser and exhibit elongated morphologies. With decreasing the temperature to $520 \text{ }^{\circ}\text{C}$ (**Fig. 4.40(c)**) and $440 \text{ }^{\circ}\text{C}$ (**Fig. 4.40(d)**), the microstructures are completely composed of ferrite, and the ferrite seems to exhibit fine-grained morphologies, although it is hard to distinguish the details from the SEM images.

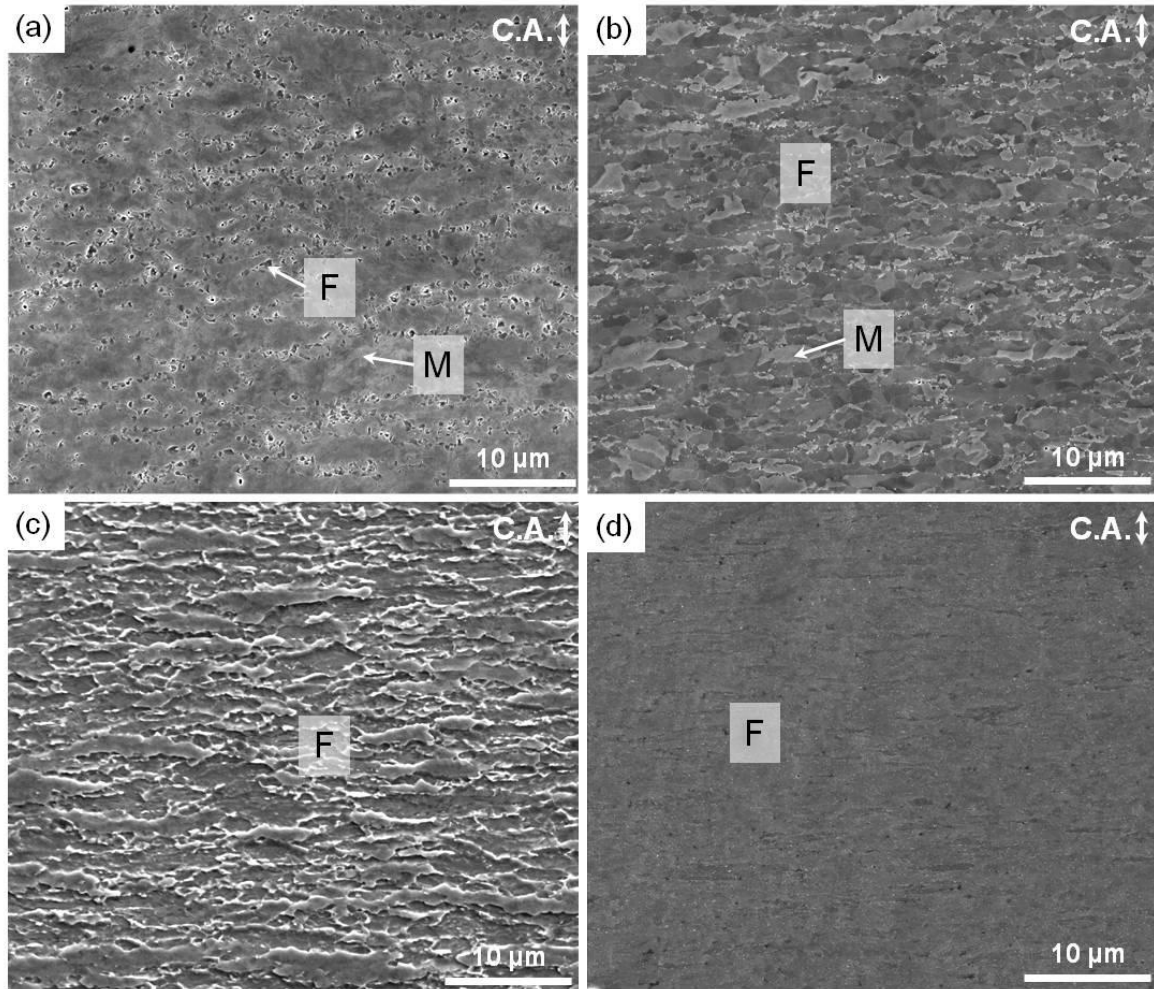


Fig. 4.40 SEM images of the specimens with an austenite grain size of 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (a) 600 $^{\circ}\text{C}$, (b) 560 $^{\circ}\text{C}$, (c) 520 $^{\circ}\text{C}$ and (d) 440 $^{\circ}\text{C}$. “F” and “M” indicate ferrite and martensite, respectively. Compression axis is along the vertical direction of the images.

The detailed microstructural features of ferrite are revealed in **Fig. 4.41**, which shows EBSD grain boundary maps of ferrite corresponding to that in **Fig. 4.40**. The ferrite microstructures differ at different deformation temperatures. At 600 $^{\circ}\text{C}$ (**Fig. 4.41(a)**), a small amount of equiaxed ferrite grains surrounded by HABs are aligned along PAGBs, indicating that the specimen is at an early stage of transformation. It is hard to conclude that these ferrite grains are formed due to DRX. At 560 $^{\circ}\text{C}$ (**Fig. 4.41(b)**), the ferrite mostly

shows coarse and elongated morphologies. The volume fraction of ferrite is 52% at this temperature, but only a few of equiaxed UFF grains can be observed, as marked by green arrows. It means that the specimen is on the stage where dynamically transformed ferrite is growing after the primary nucleation along PAGBs like that shown in **Fig. 4.41(a)**. According to the definition of equiaxed UFF grains in **Chapter 2** and the discussion in **Chapter 4**, it can be concluded that the equiaxed UFF grains obtained at temperatures from 560 °C to 440 °C are due to DRX. With decreasing the deformation temperature to 520 °C (**Fig. 4.41(c)**), the fraction of dynamically recrystallized grains increases compared to that at 560 °C. The coarse ferrite grains also contain larger fraction of LABs, exhibiting a more deformed structure. With decreasing the temperature to 440 °C furthermore (**Fig. 4.41(d)**), the fraction of dynamically recrystallized grains (as marked by green arrows) decreases. Most of the grains are elongated and contain a large amount of LABs. The HABs surrounding ferrite grains are more like geometrically necessary boundaries (GNB), which are formed due to the deformation [45] at relatively low temperatures.

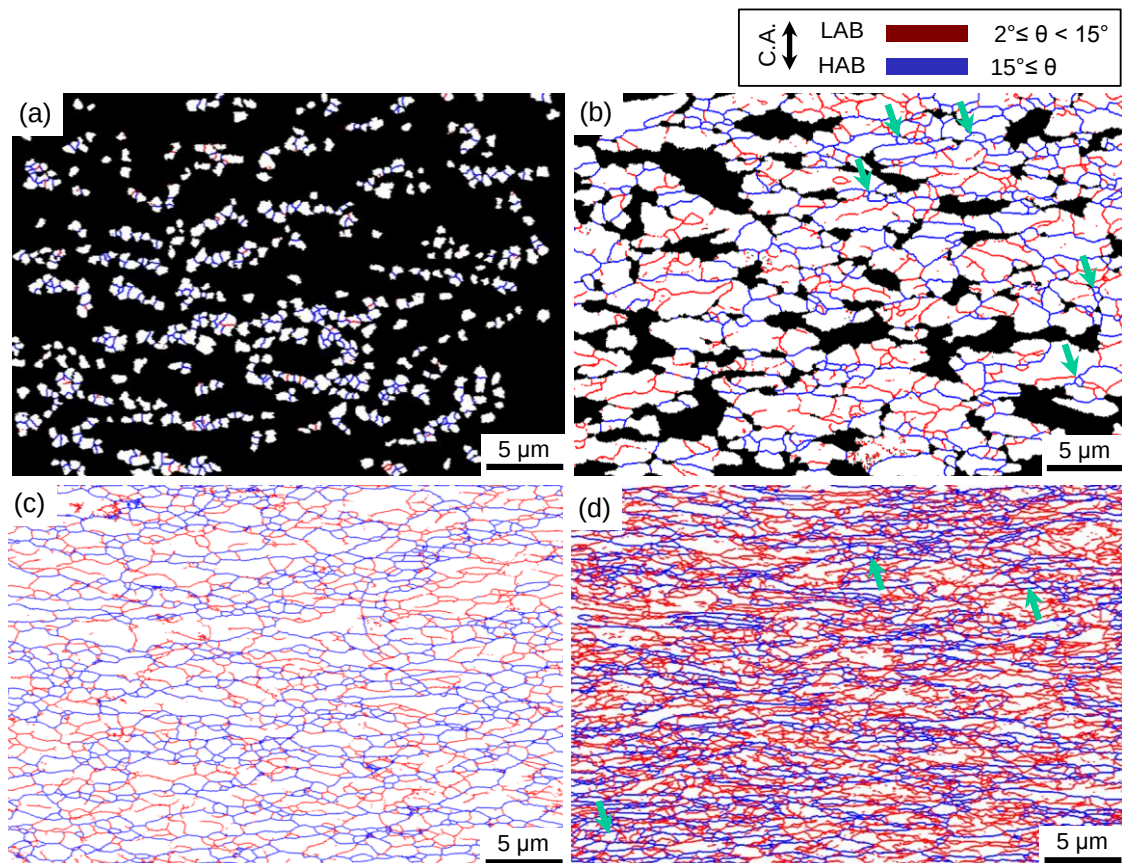


Fig. 4.41 Grain boundary maps of the specimens with an austenite grain size of 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at various deformation temperatures of (a) 600 $^{\circ}\text{C}$, (b) 560 $^{\circ}\text{C}$, (c) 520 $^{\circ}\text{C}$ and (d) 440 $^{\circ}\text{C}$. Non-ferrite phases (i.e. martensite and/or retained austenite) were covered in black. The low-angle boundaries with misorientation of 2-15 $^{\circ}$ and the high-angle boundaries with misorientation above 15 $^{\circ}$ are drawn in red and blue lines, respectively. Green arrows mark the dynamically recrystallized grains in (b) and (d). Compression axis is along the vertical direction of the maps.

Another feature of ferrite grains in **Fig. 4.41** is revealed in **Fig. 4.42**, which shows distributions of the grain size of ferrite obtained at (a) 600 $^{\circ}\text{C}$, (b) 560 $^{\circ}\text{C}$, (c) 520 $^{\circ}\text{C}$ and (d) 440 $^{\circ}\text{C}$. The ferrite grain size distributes in a very narrow range at 600 $^{\circ}\text{C}$ (**Fig. 4.42(a)**). The small fraction of grains (16%) and narrow distribution of grain size accords with the feature of ferrite at the early stage of transformation. Therefore, the equiaxed fine ferrite grains obtained at 600 $^{\circ}\text{C}$ are not considered to be formed through DRX. With decreasing

the temperature from 560 °C to 440 °C (**Fig. 4.42(b)-(c)**), the grain size of ferrite becomes finer.

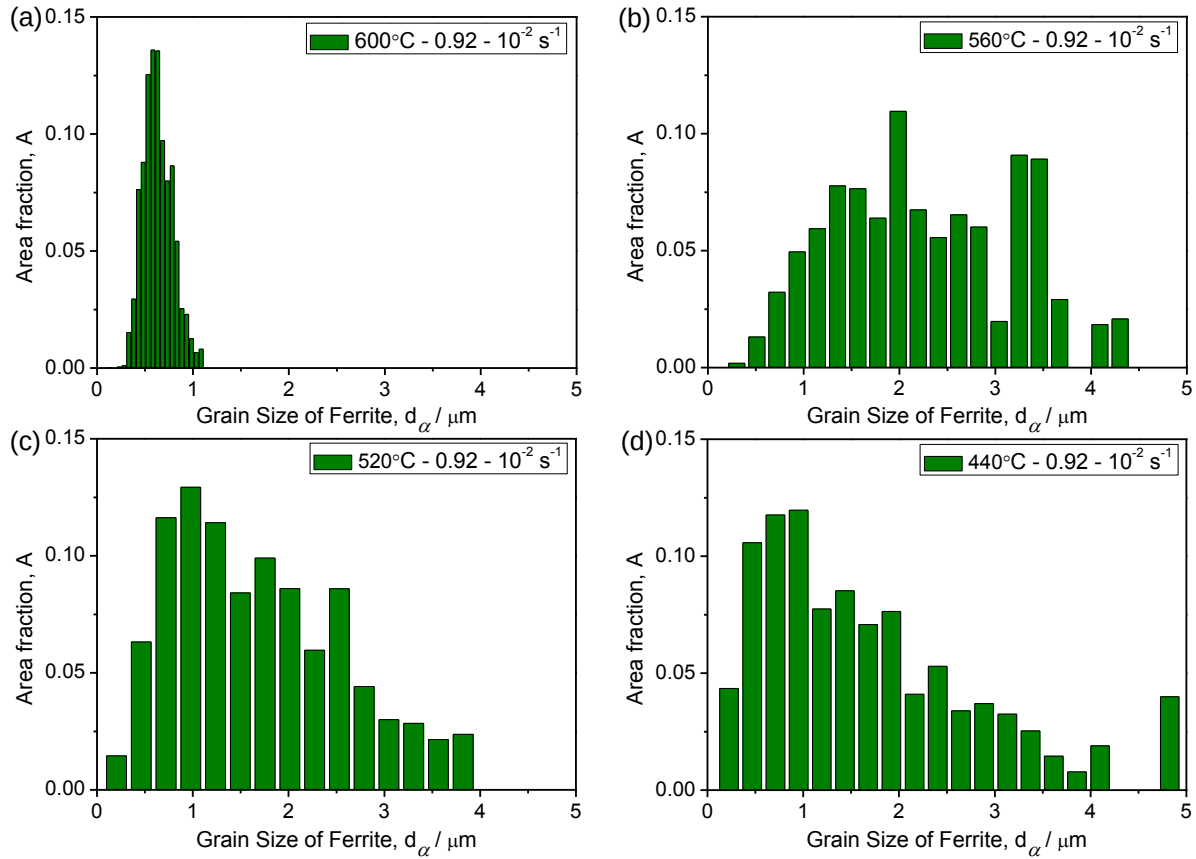


Fig. 4.42 Distribution of grain size, obtained by EBSD analysis of the specimens deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures of (a) 600 °C, (b) 560 °C, (c) 520 °C and (d) 440 °C.

In order to show the effect of deformation temperature on the DRX of ferrite, **Figure 4.43** summarizes the changes in grain size, volume fraction of dynamically transformed ferrite and volume fraction of dynamically recrystallized ferrite as a function of deformation temperatures. The grain size along the compression axis was measured by linear interception method on the EBSD maps only counting high-angle boundaries with

misorientations above 15°. At the temperatures from 440 °C to 520 °C, the ferrite transformation (open rectangles) is completed after a strain of 0.92. With increasing the temperature to 600 °C, the fraction of ferrite decreases markedly. The grain size (open circles) of ferrite increases from 440 °C to 560 °C, and then decreases with further increasing the temperature to 600 °C. The fraction of dynamically recrystallized ferrite (solid triangles) increases with increasing the temperature from 440 °C to 520 °C, but then decrease with further increasing the temperature to 600 °C, exhibiting a peak value at 520 °C. The DRX of dynamically transformed ferrite does not show a monotonous temperature dependency as that reported before [46, 47], which is an interesting phenomenon observed for the first time.

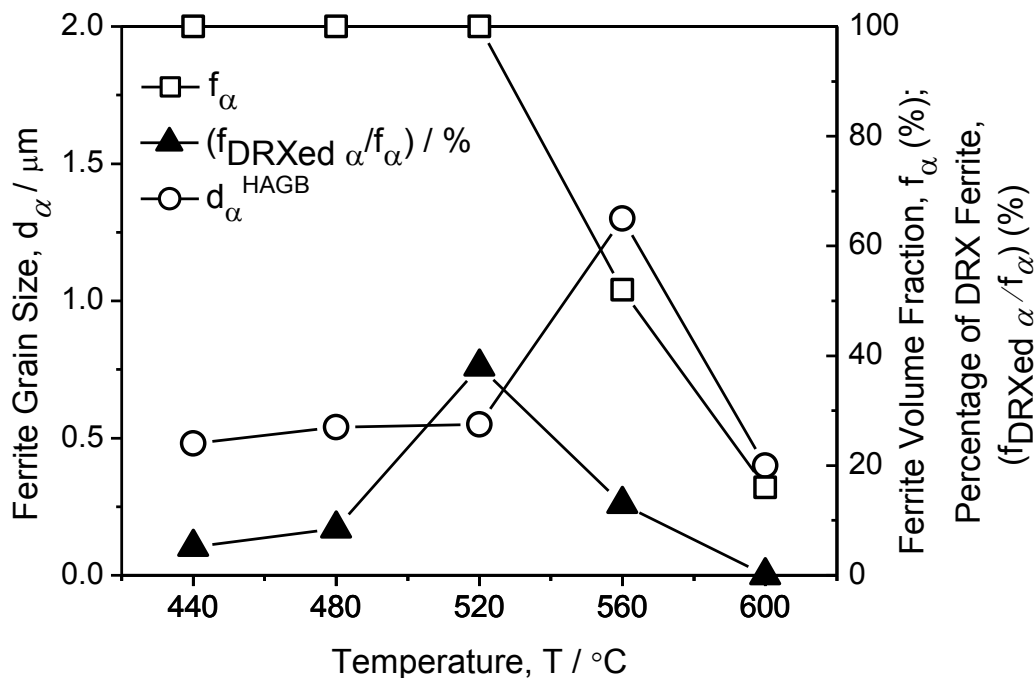


Fig. 4.43 Changes in grain size, volume fraction of dynamically transformed ferrite and volume fraction of dynamically recrystallized ferrite as a function of deformation temperatures. The grain size was measured by linear interception method on the EBSD maps only counting high-angle boundaries with misorientation above 15°.

For DRX of single ferrite phase, it has been reported that increasing the deformation temperature will enhance the occurrence of DRX [46, 47]. However, for DRX of ferrite transformed from austenite by DT, it shows a different temperature dependency. The effect of deformation temperature on the DRX of dynamically transformed ferrite is schematically illustrated in **Fig. 4.44**. It should be noted that the applied strain, strain rate and austenite grain size are constant in this illustration. F_s and R_s represent ferrite transformation and recrystallization, respectively. It was discussed that the equiaxed fine ferrite grains formed at 600 °C is probably not due to DRX. Therefore, the discussion about transformation and recrystallization behaviors will be only concentrated on the temperatures ranging from 560 °C to 440 °C. It is believed that lowering deformation temperature (above nose temperature in kinetic C-curve) shortens the incubation time for the onset of DT and accelerates its kinetics [48, 49]. At a high temperature (e.g., 560 °C), the driving force and nucleation density for ferrite are low, but the diffusivity of atoms is high. Therefore, the grain growth of ferrite is rather dominant (**Fig. 4.41(b)**). The transformed ferrite coarsens very fast, resulting in a large grain size which retards the onset of DRX [50]. Consequently, only a few dynamically recrystallized grains could be observed (**Fig. 4.41(b)** and **Fig. 4.43**). At a lower deformation temperature (e.g., 520 °C), both the driving force and the nucleation density for ferrite transformation increase, which lead to faster transformation kinetics. The fast transformation kinetics results in finer ferrite grain size due to the sufficient impingement

effect between ferrite grains. Furthermore, the strain concentration on ferrite becomes higher at fast transformation kinetics. The high strain concentration and fine ferrite grain size will greatly enhance the initiation of DRX and accelerate its kinetics. Therefore, more dynamically recrystallized grains could be observed at a lower temperature of 520 °C (**Fig. 4.41(c)** and **Fig. 4.43**). However, when the deformation temperature is further decreased to a very low value (e.g., 440 °C), the fraction of dynamically recrystallized grains becomes smaller (**Fig. 4.41(d)** and **Fig. 4.43**). That means the kinetics of DRX is not promoted, but rather retarded. The transformation kinetics is surely faster at lower temperature (above nose temperature in kinetic C-curve), but the diffusivity of atoms is so low that the thermal activation of DRX is suppressed. Even though the ferrite is still deformed after transformation, the deformation would introduce more GNB (**Fig. 4.41(d)**) rather than enhance DRX. The DRX behavior of dynamically transformed ferrite exhibits a C-curve according to the deformation temperature, which means there is an optimized deformation temperature for the progress of DRX, to be more specific, for the grain refinement of ferrite. This would be academically and industrially important to the future research.

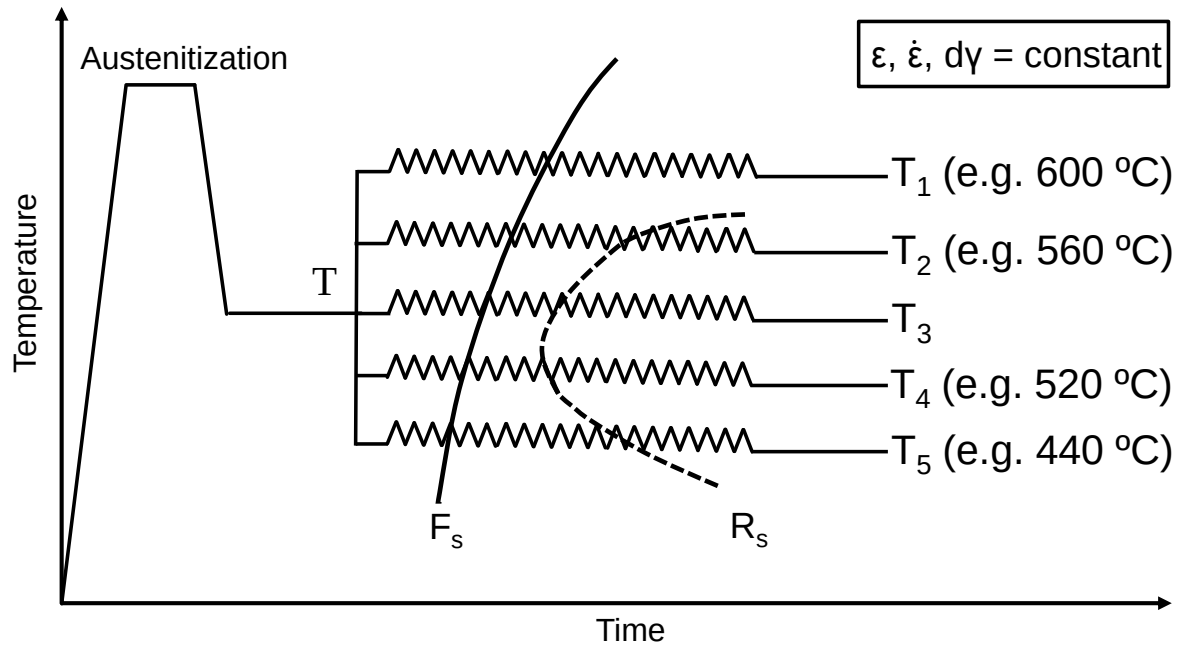


Fig. 4.44 Schematic illustration showing the DRX behavior of dynamically transformed ferrite at different deformation temperatures. F_s and R_s represent ferrite transformation and recrystallization, respectively. The applied strain, strain rate and austenite grain size are constant in this illustration.

4.4 Conclusions

A 10Ni-0.1C steel was used to investigate the microstructural evolution of ferrite in compression deformation of austenite (route-1) or full ferrite (route-2) under the same deformation conditions. The main results obtained are summarized as follows:

- (1) In the route-1, ferrite was continuously deformed after it was formed from austenite by dynamic transformation (DT). The occurrence of dynamic recrystallization (DRX) of ferrite was confirmed during the subsequent deformation. The deformation concentration in ferrite, which was caused by the difference in strength between austenite and ferrite at elevated temperatures, and the fine ferrite grain size formed by DT enhanced the initiation of DRX.
- (2) The volume fraction of dynamically recrystallized ferrite increased with increasing the strain, and the grain size of dynamically recrystallized ferrite stably kept a constant value under an identical deformation condition. Dynamically recrystallized ferrite grains contained a number of dislocations and exhibited nearly random textures.
- (3) The occurrence of DRX of ferrite was also confirmed in the route-2. The volume fraction of dynamically recrystallized ferrite grains increased with increasing the strain, and the grain size stably kept a constant value under an identical deformation condition. Decreasing the initial ferrite grain size shortened the incubation period till the initiation of DRX and accelerated its kinetics. The similarity in the morphologies of equiaxed UFF

grains obtained in the route-2 and the route-1 corroborates the occurrence of DRX of ferrite and its importance in ferrite grain refinement in the thermomechanical processes.

(4) In the route-1, the fraction of dynamically recrystallized ferrite increased with increasing the strain rate, showing an opposite strain rate dependency to that in the route-2. Three factors, i.e., large amount of lattice defects, large deformation concentration in softer ferrite in the austenite and ferrite dual phase structures, and fine ferrite grain size contributed to the larger fraction of dynamically recrystallized ferrite at higher strain rate in the route-1.

(5) The strain rate dependency of DRX in the route-1 changed depending on the kinetics of dynamic transformation. Fast transformation kinetics decreased the differences in deformation concentration and ferrite grain size between different strain rates. Consequently, the strain rate dependency of DRX in the route-1 became similar to that in the route-2 when the transformation kinetics was fast.

(6) A fine austenite grain size could accelerate the kinetics of DT thus to decrease the required strain for the onset of DRX and accelerate its kinetics.

(7) The DRX of dynamically transformed ferrite was retarded at a high temperature mainly due to the low nucleation density for ferrite transformation and high grain growth rate. DRX of ferrite was also retarded at very low temperatures. Equiaxed UFF grains cannot be formed due to the low thermal activation for initiating DRX. Instead, lamellar boundaries (i.e. GNB) were preferentially generated. The DRX behavior of dynamically transformed

ferrite exhibited a C-curve according to the deformation temperature. There is an optimized deformation condition for promoting DRX of ferrite, which is very important for grain refinement of ferrite.

4.5 References

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Chapter 5: Potential strategies for fabricating UFF through simple TMCP routes

5.1 Introduction

Grain refinement is one of the most effective ways to simultaneously improve strength and toughness in steels. If ultrafine grained (UFG) ferritic steels with mean grain sizes smaller than 1 μm were achieved in relatively simple chemical compositions, they would have large potential and great benefits to replace some kinds of steels employing additional alloying elements and/or complicated heat treatments. Therefore, extensive attention has been concentrated on developing novel methods to fabricate UFG ferritic steels in the past decades [1-10]. Among these methods, thermo-mechanical controlled processing (TMCP), such as cold rolling and annealing of martensitic steels [5], dynamic recrystallization (DRX) of ferrite during warm deformation [6, 7] and so on [8-10], can effectively achieve grain refinement of ferrite by employing relatively low accumulated strains in the range of $\varepsilon=1\sim3.6$ (in logarithmic true strain). These TMCP methods have large potential for mass production. Among the metallurgical mechanisms occurring in TMCP, dynamic transformation (DT), which is ferrite transformation occurring during deformation of austenite, can achieve a very fine ferrite grain size around 1~3 μm through simple processing routes [8]. Therefore, in the past decades, TMCP including DT has drawn considerable attention around the world [11-16].

However, to fabricate ultrafine ferrite (UFF) structures, there are still some barriers to overcome. With regard to the TMCP route including DT, UFF grains have been only achieved on the surface layers of plates [17]. To obtain a relatively homogeneous UFF structure through the thickness of materials, it usually requires large deformation (usually the accumulated strain is over 2) [18, 19]. The high required strain for fabricating UFF is the main obstacle to the industrial application of the TMCP route including DT.

It has been concluded in **Chapter 4** that DRX of ferrite occurs during the TMCP route including DT. Ferrite is firstly transformed from austenite and then continuously deformed, which leads to the initiation of DRX. The grain size of dynamically transformed ferrite firstly increases, and then decreases after the occurrence of DRX. That means the occurrence of DRX leads to a further grain refinement of ferrite. This inspires an idea that if DT and DRX behaviors could be well controlled during TMCP, the grain refinement of ferrite would be enhanced and the required strain for achieving UFF structure might be decreased. Therefore, in this chapter, potential strategies for fabricating UFF structure through TMCP routes using DT and DRX are explored.

5.2 Experimental procedure

5.2.1 Material

The material used in this study is the same alloy (Fe-10Ni-0.1C) as that used in other chapters, of which chemical composition and TTT diagram are shown in **Table 2.1** and **Fig.**

2.3. Ms temperature of the specimens with prior austenite grain size of 10 μm is 250 $^{\circ}\text{C}$.

The as-received hot-rolled plate 15 mm thick was cold-rolled to a thickness of 11.2 mm with a reduction of 25%, and then homogenized at 1100 $^{\circ}\text{C}$ for 172.8 ks in vacuum followed by water quenching. Cylindrical specimens with a height of 12 mm and a diameter of 8 mm were machined from the homogenized plate and installed in a thermomechanical processing simulator (Thermecmator-Z, Fuji Electronic Industrial Co. Ltd.) for conducting physical simulations of different TMCP routes. Mica and glass powder were used as the insulator and lubricant to minimize the friction between the contact surfaces of the anvils and the specimen during straining. The water injection was precisely controlled to minimize the time interval until the initiation of cooling.

5.2.2 Thermomechanical processes

Four kinds of TMCP routes were designed in this chapter. As is shown in **Fig. 5.1**, the TMCP route-1 is for comparison, the TMCP route-2 and the TMCP route-3 are the newly designed TMCP routes for obtaining UFF, and the TMCP route-4 is for examining the microstructures at different stages of the TMCP route-3. The austenitizing condition is the same in all four routes. According to the conclusion in **Chapter 4**, there was an optimized deformation temperature (around 520 °C) for enhancing DRX of ferrite. Therefore, the major compression deformations in the TMCP routes-1, 2 and 3 were conducted at 520 °C.

In the TMCP route-1 (**Fig. 5.1(a)**), the specimens were austenitized at 700 °C for 900 s to obtain a fine austenite grain size of 10 μm, cooled by nitrogen gas to 520 °C at a rate of 30 °C s⁻¹, held for 60 s to homogenize the temperature, and then uniaxially compressed with a reduction of 60% in height (logarithmic equivalent strain of 0.92) at a strain rate of 10⁻² s⁻¹ followed by water quenching. This is a standard route using single step deformation for comparison.

The second route is composed of two-step deformation at different temperatures. In the TMCP route-2 (**Fig. 5.1(b)**), the austenitized specimens were firstly cooled to 410 °C at a rate of 30 °C s⁻¹, deformed to 10% in reduction (equivalent strain of 0.11) at a strain rate of 10⁰ s⁻¹, reheated up to 520 °C at a rate of 55 °C s⁻¹ and then deformed to 50% in reduction (equivalent strain of 0.81) at a strain rate of 10⁻² s⁻¹ followed by water quenching. The first

10% deformation at 410 °C is for effectively accumulating lattice defects such as dislocations in austenite, which is called pre-deformation hereafter.

The third route (TMCP route-3) is also composed of two-step deformation at different temperatures. As is shown in **Fig. 5.1(c)**, the austenitized specimens were cooled by nitrogen gas to 300 °C (above the Ms temperature (250 °C)) at a rate of 30 °C s⁻¹, deformed to 10% in reduction at a strain rate of 10⁰ s⁻¹, then reheated up to 520 °C at a rate of 55 °C s⁻¹ and deformed to 50% reduction at a strain rate of 10⁻² s⁻¹ followed by water quenching. The pre-deformation in the TMCP route-3 was carried out at lower temperature than in the TMCP route-2.

It should be noted that the total strain in the TMCP routes-1, 2 and 3 is same (0.92, 60% in reduction). The only difference between three routes is the fact that, in the TMCP route-2 and TMCP route-3, the austenitized specimens were pre-deformed to 10% in reduction at relatively low temperatures of 410 °C and 300 °C, respectively. But in the TMCP route-1, the whole 60% reduction was conducted in a single step at 520 °C.

In order to verify the microstructures at different stages in the three routes, microstructures were observed in the specimens water-quenched immediately after the isothermal holding at 520 °C for 60 s in the TMCP route-1 (marked as (a1) in **Fig. 5.1(a)**) or after the pre-deformation of 10% at 410 °C in the TMCP route-2 (marked as (b1) in **Fig. 5.1(b)**). Because the pre-deformation of austenite in the TMCP route-3 was conducted at

300 °C, which was close to the Ms temperature (250 °C), deformation-induced martensite transformation may occur. Therefore, the TMCP route-4 (**Fig. 5.1(d)**) was designed to carefully check the microstructures of the specimens water-quenched right after austenitization (marked as (d1)), or pre-deformation of 10% at 300 °C (marked as (d2)), or isothermal holding at 520 °C for 4 s and 96 s (marked as (d3) and (d4), respectively).

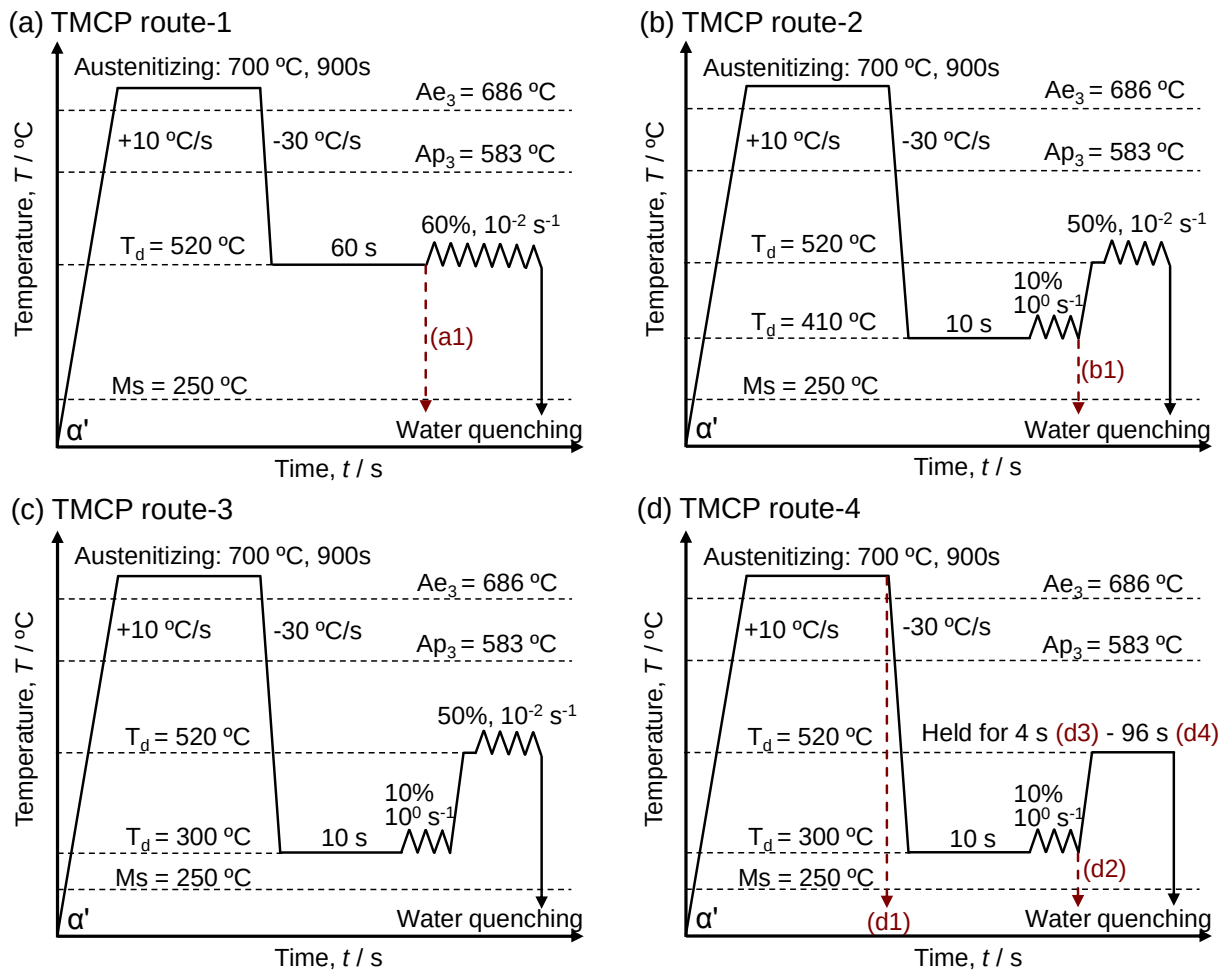


Fig. 5.1 Schematic illustrations showing the thermomechanical processes in compression deformation of austenite. (a), (b), (c) and (d) represents the TMCP route-1, TMCP route-2, TMCP route-3 and TMCP route-4, respectively. (a1), (b1) and (d1)-(d4) mark the positions where the microstructures were examined.

5.2.3 Microstructure observation

Microstructures at the center of the specimens on the section parallel to the compression axis were characterized by a field-emission type scanning electron microscope (FE-SEM, FEI XL30S FEG), electron back-scattering diffraction (EBSD) analysis performed in the FE-SEM and a transmission electron microscope (TEM, Philips CM200FEG). The specimens were mechanically polished and etched using a 3% nital solution for the SEM observation. For the EBSD measurement, the specimens were mechanically polished and then electrically polished in a solution of 10% HClO_4 and 90% CH_3COOH at 20 °C. The EBSD measurement was performed using TSL OIM Data Collection system, and the obtained EBSD data were analyzed by TSL OIM Analysis software. For the TEM observations, thin-foil specimens were prepared by twin-jet electropolishing using the same solution as that used for EBSD.

5.2.4 Miniature tensile test

Sheet-type specimens for tensile test were machined from the compressed samples, as shown in **Fig. 5.2**. The tensile direction was perpendicular to the compression direction as marked on the right hand side of the figure. The top and bottom surfaces of the compressed samples were mechanically polished. The center position of the compressed samples was chosen to be the gauge part of the tensile test specimen. The dimension of the gauge part is

2.0 mm (length) $\times$ 1.0 mm (width) $\times$ 0.5 mm (thickness). A CCD camera taking 10 images per second was used to precisely measure the displacement during the tensile test. Tensile test was conducted at an initial strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$ at room temperature.

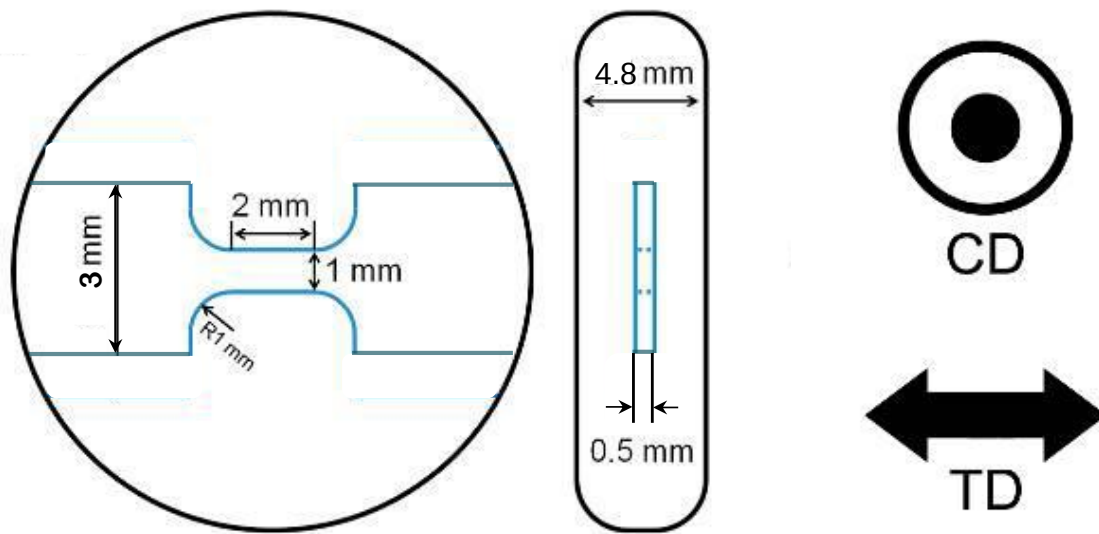


Fig. 5.2 Illustrations of top and side views of the cylindrical specimen deformed in compression to a strain of 0.92 to fabricate a tensile test sample. The dimension of the tensile test sample is 2.00 mm (length) $\times$ 1.0 mm (width) $\times$ 0.5 mm (thickness). Directions of the compression in thermomechanical test and the tensile test are shown on the right hand side.

A specimen with coarse ferrite grains (17000 nm) statically transformed by very slow furnace-cooling after the austenitization and a specimen of an ultra-low carbon interstitial free (IF) steel with submicron grain size (920 nm) fabricated by annealing a SPD (severe plastic deformation) processed (ARB processed) material [20] were used for comparing the mechanical properties.

5.3 Results and discussion

5.3.1 UFF formation by two-step deformation of austenite

In this section, the microstructures obtained from the TMCP route-1 and TMCP route-2 were observed. The difference in formation of UFF structures between two routes was particularly examined.

Figure 5.3 shows SEM images of the specimens water-quenched after (a1) isothermal holding at 520 °C for 60 s in the TMCP route-1 (corresponding to the point (a1) in **Fig. 5.1(a)**) and (b1) pre-deformation to a reduction of 10% at 410 °C in the TMCP route-2 (corresponding to the point (b1) in **Fig. 5.1(b)**). “F” and “M” represent ferrite and martensite, respectively. The microstructure in **Fig. 5.3(a1)** is fully martensitic, indicating that there was no static transformation from austenite to ferrite occurred before the deformation in the TMCP route-1. On the other hand, in the TMCP route-2, there is a small amount of ferrite (around 7% in area fraction) after the pre-deformation of austenite at 410 °C (**Fig. 5.3(b1)**). It is unclear whether the small amount of ferrite was dynamically transformed from austenite during deformation at 410 °C or statically transformed after the deformation. Anyway, the microstructures before deformation at 520 °C were mostly austenite in both routes.

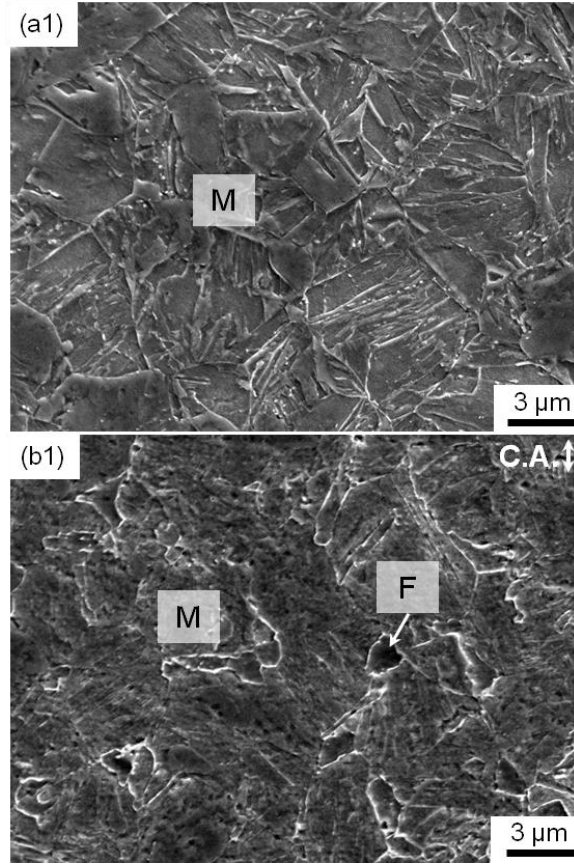


Fig. 5.3 SEM images of the specimens water-quenched after (a1) isothermal holding at 520 °C for 60 s in the TMCP route-1 (corresponding to the point (a1) in **Fig. 5.1(a)**) and (b1) pre-deformation to a reduction of 10% at a strain rate of 10^0 s^{-1} at 410 °C in the TMCP route-2 (the point (b1) in **Fig. 5.1(b)**). “F” and “M” indicate ferrite and martensite, respectively. Compression axis in (b1) is along the vertical direction of the micrograph.

The final microstructures obtained in the TMCP route-1 and TMCP route-2 are shown in **Fig 5.4**. **Figure 5.4(a)** is an EBSD grain boundary map of the specimen deformed to 60% reduction at a strain rate of 10^{-2} s^{-1} at 520 °C (TMCP route-1). Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines, respectively. It shows a microstructure composed of fine equiaxed ferrite grains and coarse elongated ferrite grains. The coarse elongated grains are divided by a number of low-angle boundaries. They are clearly the

ferrite firstly formed by DT and then deformed in subsequent compression. On the other hand, the existence of the equiaxed grains indicates the occurrence of DRX of ferrite [21-24].

The distribution of boundary misorientation angles corresponding to **Fig. 5.4(a)** shows that the fraction of high-angle boundaries in the specimen deformed in the TMCP route-1 is about 56% (**Fig. 5.4(c)**), revealing a partially recrystallized microstructure. The grain sizes along the compression axis were measured by linear interception method on the EBSD maps counting all boundaries (with misorientations higher than 2° , defined as d^{All}) or only high-angle boundaries (with misorientations higher than 15° , defined as d^{HAGB}). The mean grain sizes (d^{All} and d^{HAGB}) of ferrite obtained in the TMCP route-1 are 565 nm and 930 nm, respectively. The microstructure of the specimen deformed in the TMCP route-2 (**Fig. 5.4(b)**) also exhibits fine equiaxed ferrite grains and coarse elongated ferrite grains as well as **Fig. 5.4(a)**. The distribution of boundary misorientation angles corresponding to **Fig. 5.4(b)** shows that the fraction of high-angle boundaries increases from 56% to 70% (**Fig. 5.4(d)**) in the TMCP route-2, indicating that DRX of transformed ferrite progressed much more than that in the TMCP route-1. Accordingly, finer ferrite grains were formed with mean grain sizes (d^{All} and d^{HAGB}) of 416 nm and 550 nm, respectively. As shown in the TEM image (**Fig. 5.4(e)**), the DRX grains obtained in the TMCP route-2 exhibit ultrafine equiaxed morphology and contain a certain amount of dislocations. It is noteworthy that the grain size obtained in the TMCP route-2 is much finer than that of the equiaxed ferrite grains (~ 1

μm) fabricated through SPD (ARB to an equivalent strain of 4.8) and subsequent annealing process in an ultra-low carbon IF steel previously reported [25].

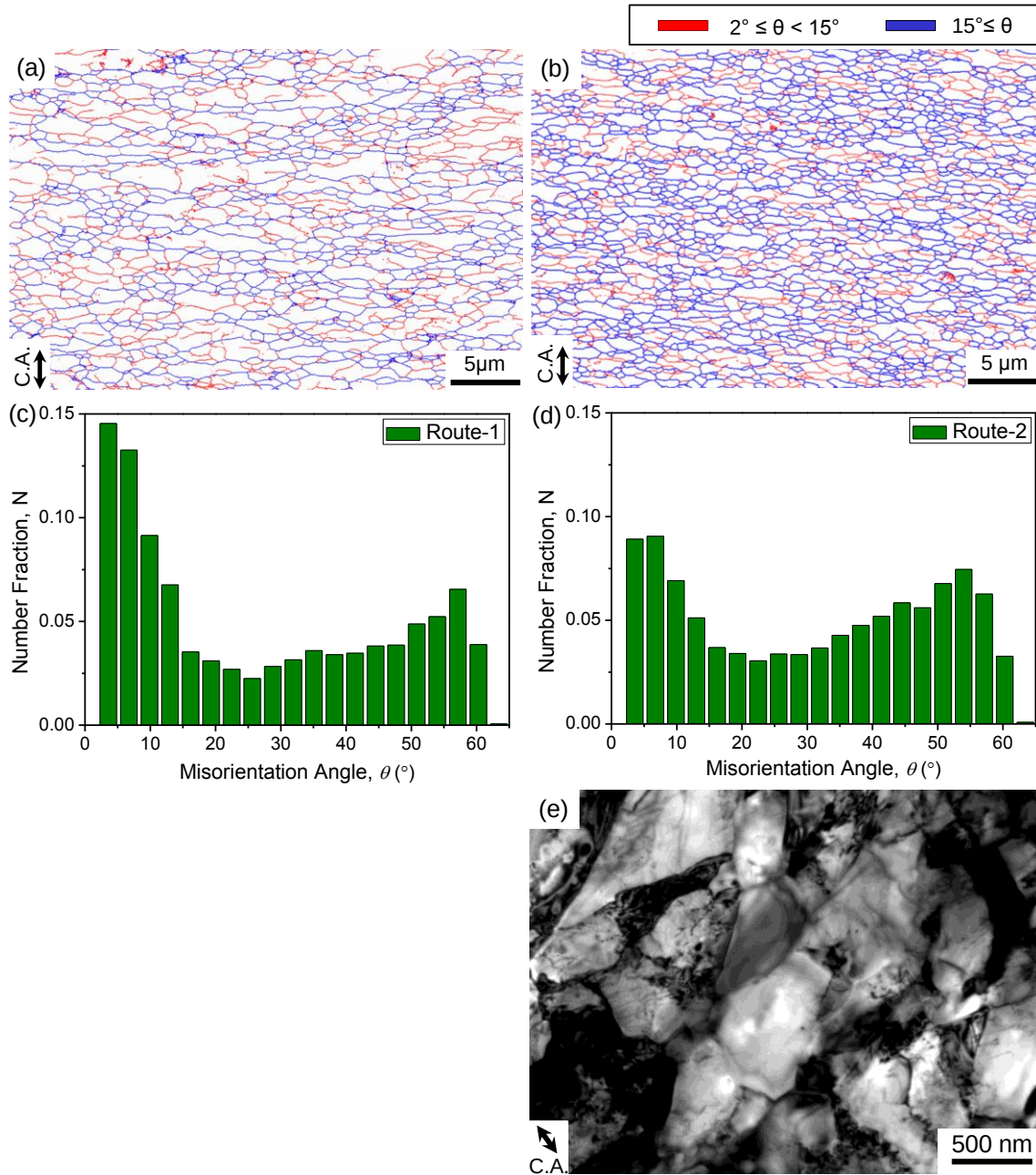


Fig. 5.4 (a, b) EBSD grain boundary maps of the specimens deformed in the TMCP route-1 (a) and TMCP route-2 (b). Low-angle boundaries with misorientation of $2\text{--}15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. (c, d) Distributions of boundary misorientation angles corresponding to (a) and (b), respectively. (e) TEM image of the specimen deformed in the TMCP route-2.

In the TMCP route-2, it is considered that DT mainly progressed during the 50% deformation at 520 °C. The microstructural evolution of ferrite in the TMCP route-2 is revealed in **Fig. 5.5**, which shows EBSD grain boundary maps of the specimen pre-deformed to 10% at 410 °C and then further deformed to (a) 5%, (b) 20%, (c) 35% and (d) 50% at a strain rate of 10^{-2} s^{-1} at 520 °C. In order to depict the microstructure evolution of ferrite, non-ferrite phases (i.e., martensite and/or retained austenite) are painted in black, which is the case for all EBSD maps in this chapter hereafter. The methods for distinguishing ferrite from austenite and martensite were introduced in **Chapter 2**. At a reduction of 5% (**Fig. 5.5(a)**), ferrite grains observed are coarse and contain a small amount of LABs, indicating a deformed morphology. With increasing the reduction to 20% (**Fig. 5.5(b)**), the fraction of LABs increases and equiaxed UFF grains (i.e., DRX grains) are observed. The fraction of dynamically recrystallized grains increases with increasing the reduction to 35% (**Fig. 5.5(c)**) and 50% (**Fig. 5.5(d)**). Consequently, a homogeneous UFF structure could be obtained, as shown in **Fig. 5.5(d)**. The microstructural evolution from **Fig. 5.5(a)** to **(d)** indicates that ferrite is continuously refined from coarse grains at the beginning of the transformation to UFF grains after the completion of deformation.

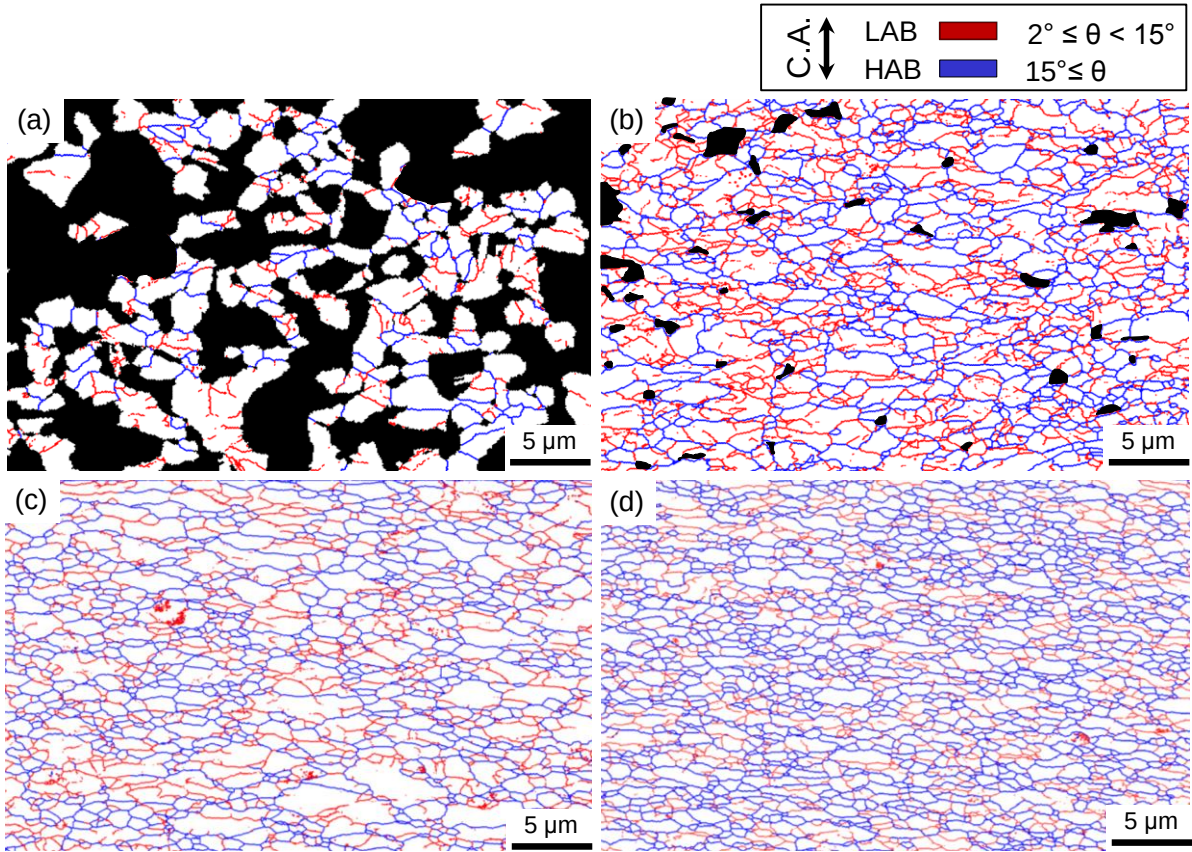


Fig. 5.5 EBSD grain boundary maps of the specimens pre-deformed to a reduction of 10% at 410 °C and further deformed to a reduction of (a) 5%, (b) 20%, (c) 35% and (d) 50% at a strain rate of 10^{-2} s^{-1} at 520 °C followed by water quenching (TMCP route-2). Non-ferrite phase (i.e. martensite and/or retained austenite) were painted in black. Low-angle boundaries with misorientation of $2-15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in red and blue lines, respectively. Compression axis is along the vertical direction of the maps.

Figure 5.6 summarizes the changes in the ferrite grain size, volume fraction of dynamically transformed ferrite and the volume fraction of dynamically recrystallized ferrite obtained in the TMCP route-2. The ferrite transformation progresses very fast and reaches completion around the strain of 0.6. It indicates that the lattice defects introduced by pre-deformation of austenite at lower temperature (410 °C) provide more nucleation sites for ferrite transformation at 520 °C, leading to the acceleration of DT. Consequently the grain

size of dynamically transformed ferrite becomes finer due to the enhanced nucleation and early impingement between ferrite grains nucleated in high density. Fine ferrite grains decrease the critical strain for initiating DRX and also accelerate the kinetics of DRX [23]. The fraction of dynamically recrystallized grains (fine grains) increases with the progress of transformation, and continues to increase after the completion of transformation (at a strain around 0.6).

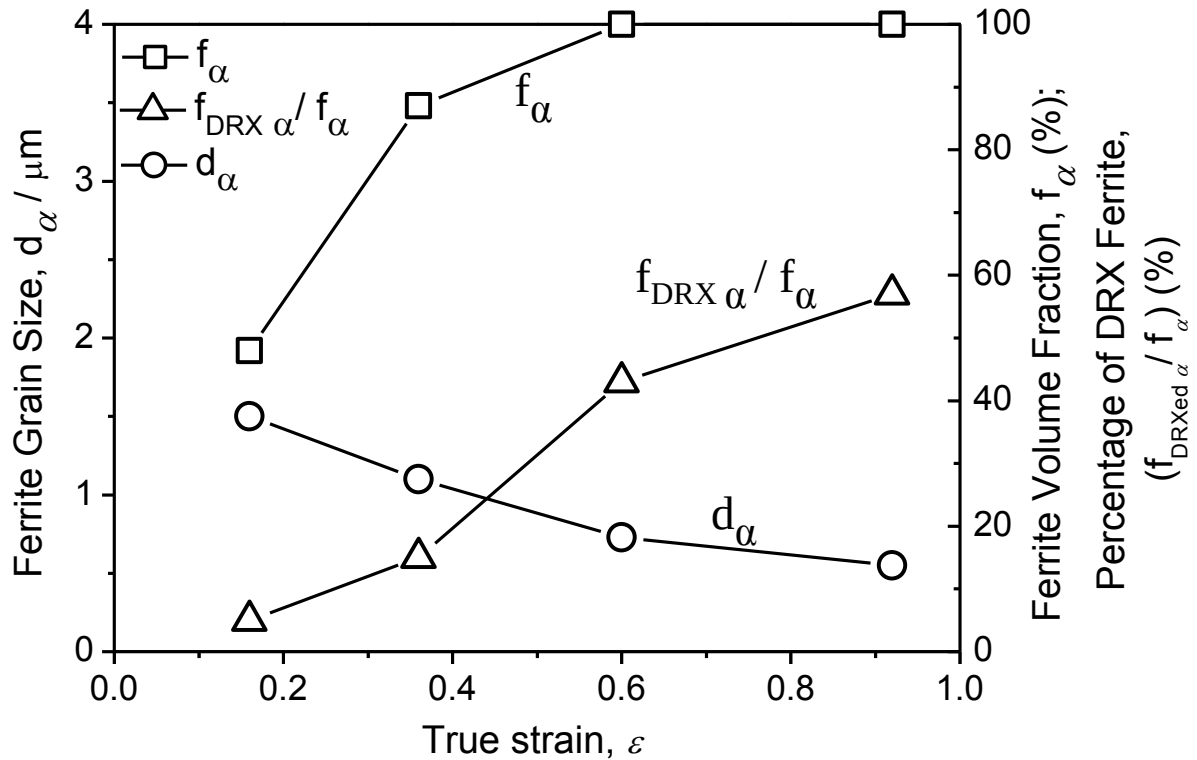


Fig. 5.6 Changes in ferrite grain size, volume fraction of dynamically transformed (DT) ferrite and volume fraction of dynamically recrystallized (DRX) ferrite as a function of compression strain in the TMCP route-2. The grain size was measured by linear interception method on the EBSD maps only counting high-angle boundaries with misorientation above 15°.

Figure 5.7 shows (a) TEM image and (b) corresponding misorientation map determined by Kikuchi-line analysis of the ultrafine ferrite (UFF) structure (grain size of 550 nm) obtained in the TMCP route-2. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in red and blue lines in (b), respectively. The microstructure is composed of equiaxed UFF grains surrounded by complete HABs and subgrains surrounded by LABs and partial HABs, indicating a similar DRX structure to that shown in **Chapter 4**. This also confirms that the UFF structure obtained in the TMCP route-2 is formed by DRX mechanism, that is, the strain concentration or inhomogeneous deformation introduces dislocations into transformed ferrite (**Fig. 5.7(c)**) and thus increases the misorientation angle of the subgrains. Finally equiaxed UFF grains surrounded by HABs (**Fig. 5.7(d)**) are formed.

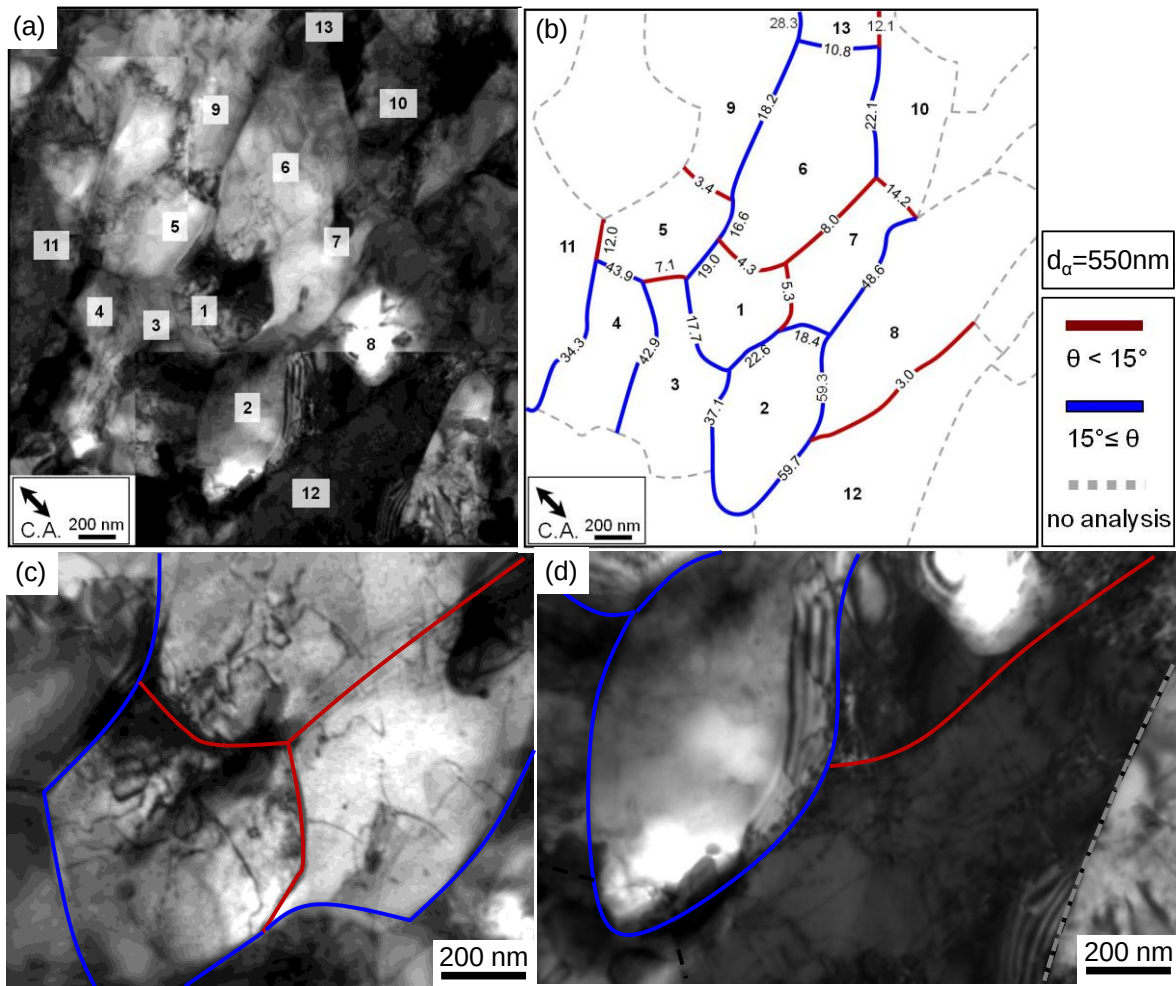


Fig. 5.7 (a) TEM image and (b) corresponding misorientation map determined by Kikuchi-line analysis of the specimen pre-deformed to a reduction of 10% at 410 °C and further deformed to a reduction of 50% at a strain rate of 10^{-2} s^{-1} at 520 °C followed by water quenching (TMCP route-2). (c) and (d) show the enlarged areas in (a). (c) Grains 1, 6 and 7. (d) Around grain 2. LABs with misorientation below 2° and HABs with misorientation above 15° are drawn in red and blue lines, respectively. Broken black lines in (b) represent the boundaries not analyzed. Compression axis is indicated in (a) and (b).

5.3.2 UFF formation by two-step deformation of austenite and martensite

In this section, the microstructure obtained in the TMCP route-3 was examined. The mechanism for obtaining UFF grains in the TMCP route-3 was particularly discussed. The efficiency in obtaining UFF grains by the TMCP route-3 is compared with the TMCP route-1 and TMCP route-2.

First of all, the microstructures at different stages of the TMCP route-3 are shown in **Fig. 5.8**, which reveals SEM images of the specimens quenched after austenitization at 700 °C for 900 s (d1) (corresponding to the point (d1) in **Fig. 5.1(d)**), or pre-deformation to a reduction of 10% at 300 °C (d2) (corresponding to the point (d2) in **Fig. 5.1(d)**), or isothermal holding at 520 °C for 4 s (d3) or 96 s (d4) (corresponding to the points (d3) and (d4) in **Fig. 5.1(d)**). “F”, “M” and “TM” represent ferrite, martensite, and tempered martensite, respectively. The microstructure shown in **Fig. 5.8(d1)** is fully martensitic, indicating that the specimen was complete austenite with grain size around 10 µm after the austenitization at 700 °C for 900 s. The specimen pre-deformed at 300 °C also shows a fully martensitic morphology (**Fig. 5.8(d2)**). However, it cannot be concluded that it was complete austenite phase after pre-deformation, because the deformation temperature (300 °C) was close to the Ms temperature (250 °C). There is a possibility that deformation induced martensitic transformation occurred during the pre-deformation at 300 °C. From **Fig. 5.8(d2)**, it is difficult to distinguish deformation-induced martensite from thermally-induced martensite

(i.e., austenite before water-quenching). The pre-deformed specimen isothermally held at 520 °C for 4 s (**Fig. 5.8(d3)**) shows martensite and tempered martensite as well as a small amount of ferrite concurrently. The tempered martensite (with a volume fraction of 61%) consists of extremely small cementite particles embedded within a continuous ferrite matrix. With increasing the isothermal holding time to 96 s, the fraction of martensite decreases and more ferrite grains are observed around martensite (**Fig. 5.8(d4)**). This microstructural evolution confirms the occurrence of deformation-induced martensitic transformation during the pre-deformation at 300 °C, which means that the microstructure after pre-deformation in the TMCP route-3 was composed of deformation induced martensite and deformed austenite. When the pre-deformed dual phased structure was isothermally held at 520 °C, the deformation induced martensite was tempered and ferrite transformation from deformed austenite occurred. Austenite was progressively consumed so that the fraction of ferrite increased with increasing the holding time, and correspondingly the fraction of martensite after water-quenching decreased (**Fig. 5.8(d3)** and **(d4)**).

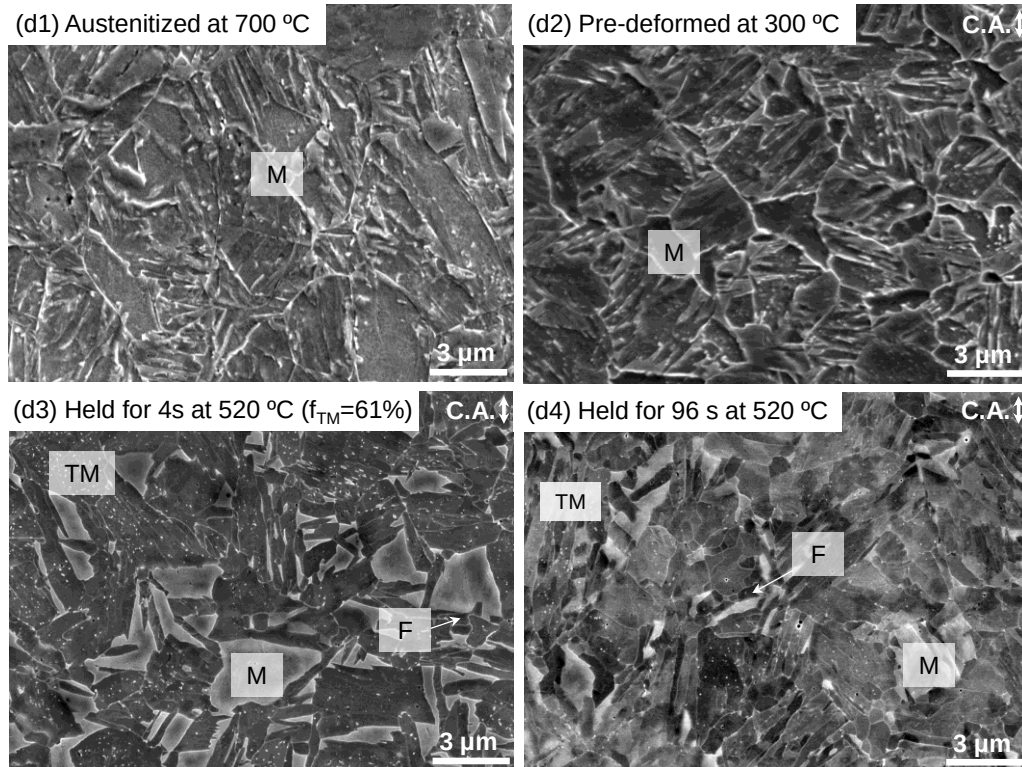


Fig. 5.8 SEM images of the specimens processed in the TMCP route-3. Water-quenched (d1) after austenitization at 700 °C for 900 s (corresponding to the point (d1) in **Fig. 5.1(d)**), (d2) pre-deformation to a reduction of 10% at a strain rate of 10^0 s^{-1} at 300 °C (the point (d2) in **Fig. 5.1(d)**), and isothermal holding at 520 °C for (d3) 4 s or (d4) 96 s (the points (d3) and (d4) in **Fig. 5.1(d)**). “F”, “M” and “TM” indicate ferrite, martensite and tempered martensite, respectively. Compression axis in (d2 – d4) is along the vertical direction of the micrographs.

The final microstructure obtained in the TMCP route-3 is revealed in **Fig. 5.9(a)**, which shows an inverse pole figure (IPF) map combined with an image quality (IQ) map obtained by EBSD analysis of the specimen. Low-angle boundaries (LABs) with misorientation of 2-15 ° and high-angle boundaries (HABs) with misorientation above 15 ° are drawn in white and black lines, respectively. **Figure 5.9(a)** shows a microstructure composed of mainly fine dynamically recrystallized ferrite grains and some portion of coarse elongated ferrite. The coarse and elongated grains are divided by a number of LABs (**Fig. 5.9(a)**), and contain

considerable amount of dislocations inside, as is shown by a TEM image in **Fig. 5.9(b)**. The mean grain sizes (d^{All} and d^{HAGB}) of ferrite are 373 nm and 460 nm, respectively. The corresponding distribution of boundary misorientation angle (**Fig. 5.9(c)**) shows that the fraction of high angle boundaries is around 75%. It should be noted that it is the first time to date to obtain such an equiaxed and uniform ferrite structure with ultrafine grain size (**Fig. 5.9(d)**) without severe plastic deformation (only 60% in total reduction) in low-C steel.

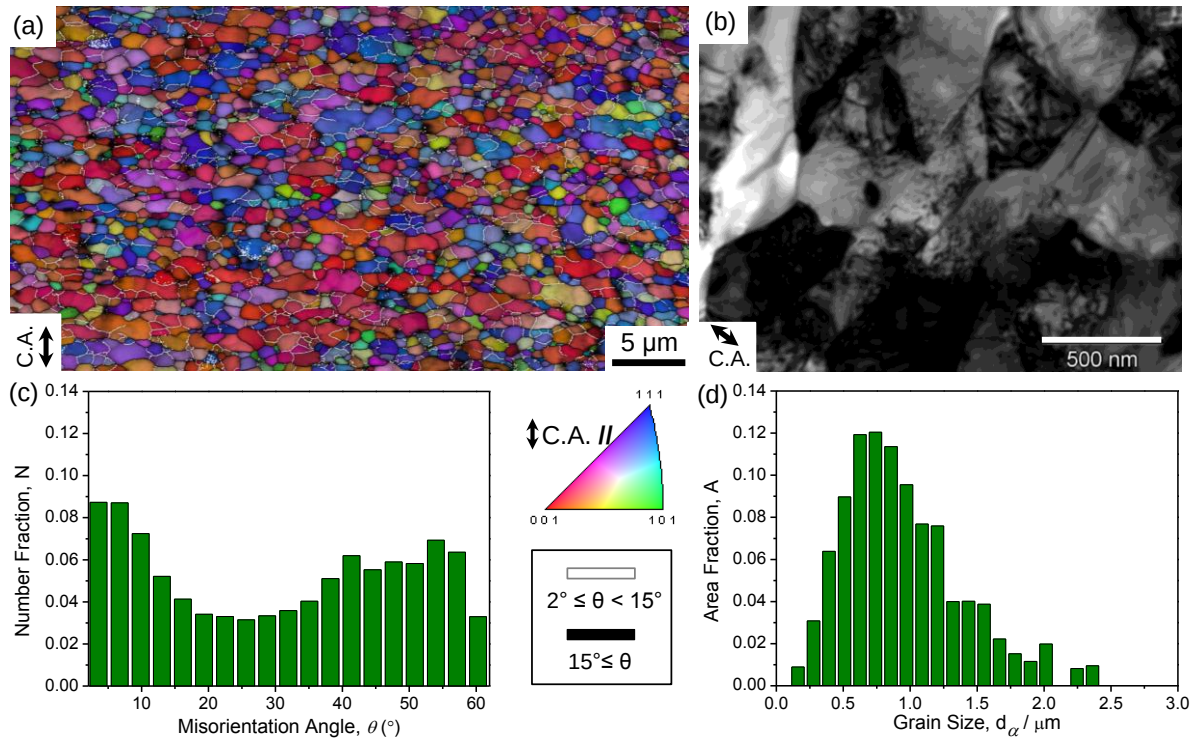


Fig. 5.9 (a) EBSD inverse pole figure (IPF) map combined with image quality (IQ) map, (b) TEM image, (c) corresponding distribution of boundary misorientation angles and (d) corresponding grain size distribution of the specimen pre-deformed to a reduction of 10% at 300 $^\circ\text{C}$ and further deformed to a reduction of 50% at a strain rate of 10^{-2} s^{-1} at 520 $^\circ\text{C}$ followed by water-quenching (TMCP route-3). Low-angle boundaries with misorientation of $2\text{--}15^\circ$ and high-angle boundaries with misorientation above 15° are drawn in white and black lines in (a), respectively. Compression axis is indicated in (a) and (b).

In the TMCP route-3, it has been confirmed that the specimen consists of deformation-induced martensite and deformed austenite after the pre-deformation at 300 °C (**Fig. 5.8**). Therefore, during the further deformation to a 50% reduction at 520 °C, there are mainly two possible mechanisms progressing with deformation. One is DT from the deformed austenite and the other is DRX of tempered martensite and dynamically transformed ferrite.

It seems difficult to precisely measure the austenite grain size after the formation of deformation-induced martensite at 300 °C, but it is reasonable to think that the austenite grains would become finer (less than 10 µm) after the formation of martensite. Additionally, the austenite deformed at 300 °C had deformation structures including high density of dislocations. Fine austenite grain size and lattice defects introduced by pre-deformation can provide more nucleation sites for ferrite transformation at 520 °C. Consequently the grain size of dynamically transformed ferrite would become finer due to the enhanced nucleation as well as early impingement between ferrite grains and with deformation-induced martensite. Fine transformed ferrite grains shorten the incubation time for the onset of DRX and accelerate its kinetics [26]. Furthermore, ferrite is softer than austenite [27]. Compared to austenite, the deformation-induced martensite can act as a much hard phase during the deformation of the multi-phased structure at 520 °C. The strain concentration would enhance the progress of not only DT but also DRX of ferrite. Moreover, warm deformation

of martensite phase at 520 °C should also be considered to contribute to the grain refinement. The martensite transformed at 300 °C will be deformed at 520 °C. Because martensite phase has high density of dislocations, supersaturated carbon atoms and fine substructures, it is well believed that these characteristics make the martensite to be a suitable initial structure to enhance recrystallization (including DRX) for realizing fine grain structures [28-30]. The mechanisms discussed above proceed simultaneously during the deformation of multi-phased specimen at 520 °C. Consequently, DRX of ferrite is enhanced, and plays an important role in the formation of UFF grains (**Fig. 5.9**).

Compared with the ferrite structure obtained in the TMCP route-1 (**Fig. 5.4(a)**) and TMCP route-2 (**Fig. 5.4(b)**), the ferrite structure obtained in the TMCP route-3 (**Fig. 5.9**) is finer. The efficiency in ferrite grain refinement between the three TMCP routes could be compared in **Fig. 5.10**, which shows the true stress-true strain curves of the specimens during compression in the three TMCP routes. The black, red and blue lines represent the curves obtained in the TMCP route-1, TMCP route-2 and TMCP route-3, respectively. The stress-strain curve (black color) obtained in the TMCP route-1 shows initial work-hardening followed by a slight decrease in the flow stress. It suggests a softening due to the occurrence of DT and DRX, which is confirmed by the corresponding microstructure in **Fig. 5.4(a)**. The stress-strain curve (red color) obtained in the TMCP route-2 shows initial work-hardening to a high stress around a strain of 0.11. This is caused by the

pre-deformation (true strain of 0.11) at low temperature (410 °C), which results in work-hardened austenite. In the subsequent deformation at 520 °C, the work-hardened austenite is deformed and the stress-strain curve exhibits a peak stress followed by a larger decrease in the flow stress than that in the TMCP route-1. It is confirmed by **Fig. 5.5** that the softening in stress-strain curves is caused by the occurrence of DT and DRX during deformation of austenite. The pre-deformation of austenite at 410 °C in the TMCP route-2 introduces large amount of defects and deformation structures into austenite. The nucleation density for ferrite transformation increases and thus DT and subsequent DRX are enhanced at 520 °C. The stress-strain curve (blue color) obtained in the TMCP route-3 shows a much higher stress around a strain of 0.11, which is caused by the pre-deformation (true strain of 0.11) at lower temperature (300 °C), resulting in work-hardened austenite and deformation-induced martensite (with volume fraction around 61%, **Fig. 5.8 (d3)**). In the subsequent deformation at 520 °C, the work-hardened austenite and martensite were deformed and the stress-strain curve exhibits a peak stress followed by a larger decrease in the flow stress compared to those in the TMCP route-1 and TMCP route-2. The more softened stress-strain curve in the TMCP route-3 indicates that deformation of the dual-phase structure composed of work-hardened austenite and martensite could more enhance DT and DRX of ferrite. Accordingly, finer ferrite structure could be obtained (**Fig. 5.9**).

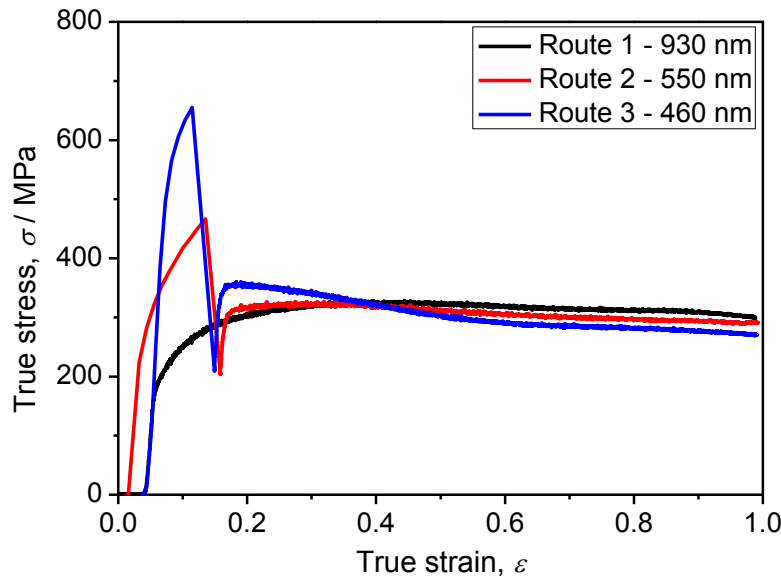


Fig. 5.10 True stress-true strain curves of the specimens during compression in three TMCP routes. The black, red and blue lines represent the TMCP route-1, TMCP route-2 and TMCP route-3, respectively.

The newly designed TMCP route-3 is confirmed to be more effective in grain refinement of ferrite. Based on the mechanism of grain refinement discussed above, it is suggested finer ferrite structure could be achieved through changing the thermomechanical parameters in the TMCP route-3. **Figure 5.11** is just an example for showing this possibility for further grain refinement through a modified TMCP route-3 with higher deformation in the first step. The EBSD grain boundary map (a) and TEM image (b) reveal the ferrite structure in the specimen deformed to 30% in reduction at 300 °C and further deformed to 45% in reduction at a strain rate of 10^{-1} s^{-1} at 520 °C. The mean grain size (d^{All} and d^{HAGB}) along the compression direction is 272 nm and 350 nm, respectively, which is finer than any reported ferrite structures fabricated under the deformation with a reduction no larger than 75% (true strain of 1.39).

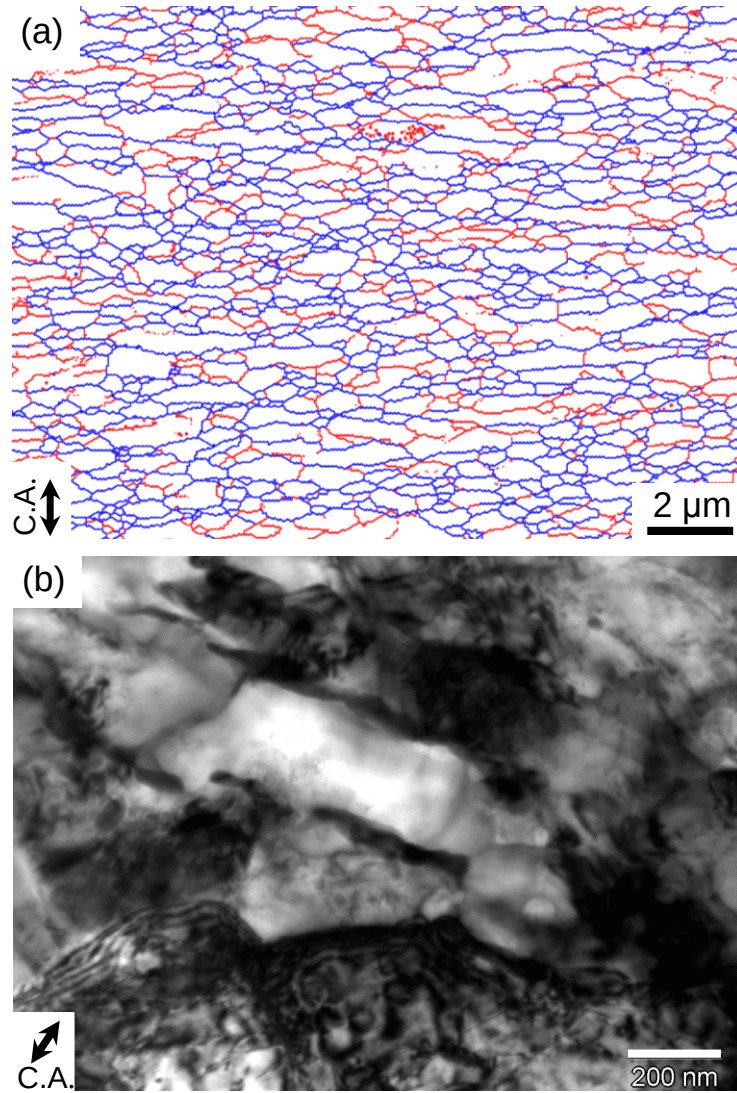


Fig. 5.11 EBSD grain boundary map (a) and TEM image (b) of the specimen pre-deformed to a reduction of 30% at a strain rate of 10^0 s^{-1} at 300 °C and further deformed to a reduction of 45% at a strain rate of 10^{-1} s^{-1} at 520 °C followed by water-quenching (modified TMCP route-3). Low-angle boundaries with misorientation of 2-15° and high-angle boundaries with misorientation above 15° are drawn in red and blue lines in (a), respectively. Compression axis is indicated in each figure.

Table 5.1 lists the microstructural parameters of the ferrite structures obtained from three types of TMCP routes. It is indicated that if kinetics of DT could be enhanced, for instance, by pre-deformation at low temperature, then the DRX of ferrite could be enhanced and ultrafine ferrite structure could be obtained. It is expected that the present two-step routes

are applicable to practical industry, since the degree of deformation at lower temperature is very low (just 10%) and natural heating may happen in materials with large dimensions, such as plates, due to large heat capacity of the materials.

Table 5.1 Microstructural parameters of ferrite obtained in three types of TMCP routes

TMCP routes	Type	ε	f_{α}	f_{HAGB}	d_{α}^{All}	d_{α}^{HAGB}
Def. of γ (route-1)	One-step	0.92	100%	56%	565 nm	930 nm
Def. of γ (route-2)	Two-step	0.11+0.81	100%	70%	416 nm	550 nm
Def. of γ and α' (route-3)	Two-step	0.11+0.81	100%	75%	372 nm	460 nm
Def. of γ and α' (modified route-3)	Two-step	0.35+1.04	100%	71%	273 nm	350 nm

5.3.3 Mechanical property of UFF

In this section, the mechanical properties of the UFF structure obtained in the TMCP routes are investigated. Since the dimensions of the specimens for tensile test are relatively small (gauge size: 2.0 mm in length, 1.0 mm in width and 0.5 mm in thickness) in the present study, it is necessary to examine the reliability of the tensile test using such small specimens.

Figure 5.12(a) shows engineering stress-strain curves of the specimens composed of statically transformed (ST) ferrite grains. Two different tensile specimens with a small gage size (2.0 mm in length, 1.0 mm in width and 0.5 mm in thickness) and a large gage size (10.0 mm in length, 5.0 mm in width and 1.0 mm in thickness) were used. The yield strength, tensile strength as well as elongation are quite similar between the specimens with different gage sizes (**Fig. 5.12(a)**). Therefore, it can be concluded that the small-size tensile specimens cut from compressed materials could provide reliable mechanical properties.

Figure 5.12(b) shows engineering stress-strain curves of the UFF specimens obtained from the TMCP route-2 (grain size of 550 nm) and the TMCP route-3 (grain size of 460 nm and 350 nm), as well as the specimen with coarse ferrite grains (17000 nm = 17 μ m) obtained by static transformation from undeformed austenite in slow furnace-cooling, and the UFG IF steel (920 nm) obtained by ARB and subsequent annealing process. An engineering stress-strain curve of the specimen with full martensite phase of Fe-10Ni-0.1C alloy (the present material) is also shown in the figure. The specimen with coarse ferrite grains

exhibits yield strength of 360 MPa and tensile strength of 482 MPa with an uniform elongation of 16% and total elongation of 39%. The UFF specimens show significantly higher yield strength of 770-953 MPa and tensile strength of 810-973 MPa. The yield strength of the UFF specimen with a grain size of 350 nm (953 MPa) is even close to that of martensite (1100 MPa). The UFF structure obtained by the new TMCP routes can maintain large tensile ductility as well: uniform elongation of 8-10% and total elongation of 23-29%.

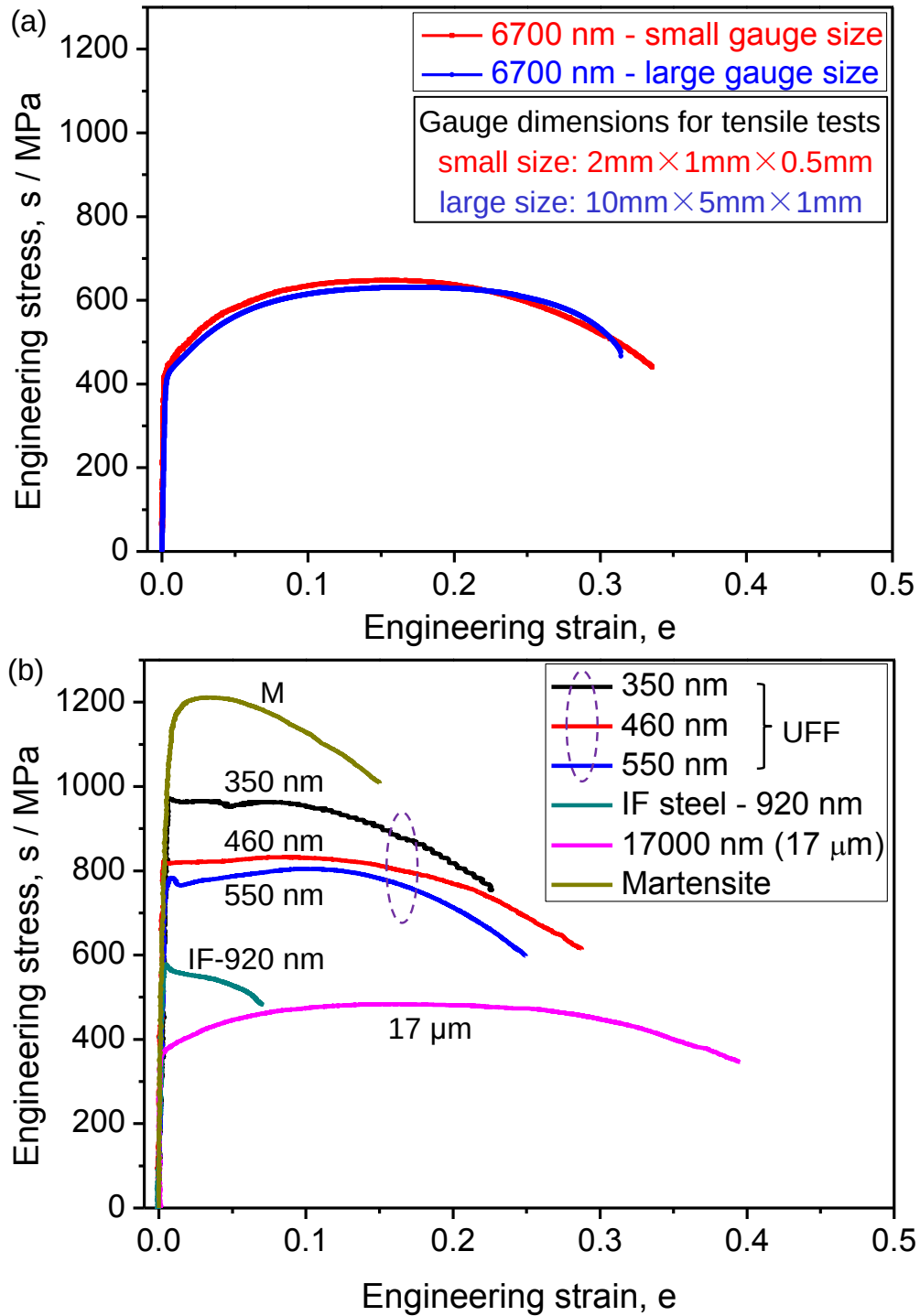


Fig. 5.12 (a) Engineering stress-strain curves of the specimens with coarse grained (6700 nm) ferrite (Fe-10Ni-0.1C alloy) having small and large gauge sizes. (b) Engineering stress-strain curves of the specimens with UFF structures (grain size of 350-550 nm), coarse grained (17000 nm) ferrite and martensite in the Fe-10Ni-0.1C alloy, as well as that of the ultrafine grained (920 nm) IF steel. Tensile tests were conducted at room temperature at an initial strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$.

Figure 5.13 shows SEM images of the fracture surfaces in the tensile specimens with (a) statically transformed coarse ferrite with grain size of 6700 nm, (b) UFF of 550 nm grain size, (c) UFF of 460 nm and (d) UFF of 350 nm. It is revealed that the fracture surfaces of all the UFF specimens consist of very fine and spherical dimples, which are typical characteristics of ductile fracture.

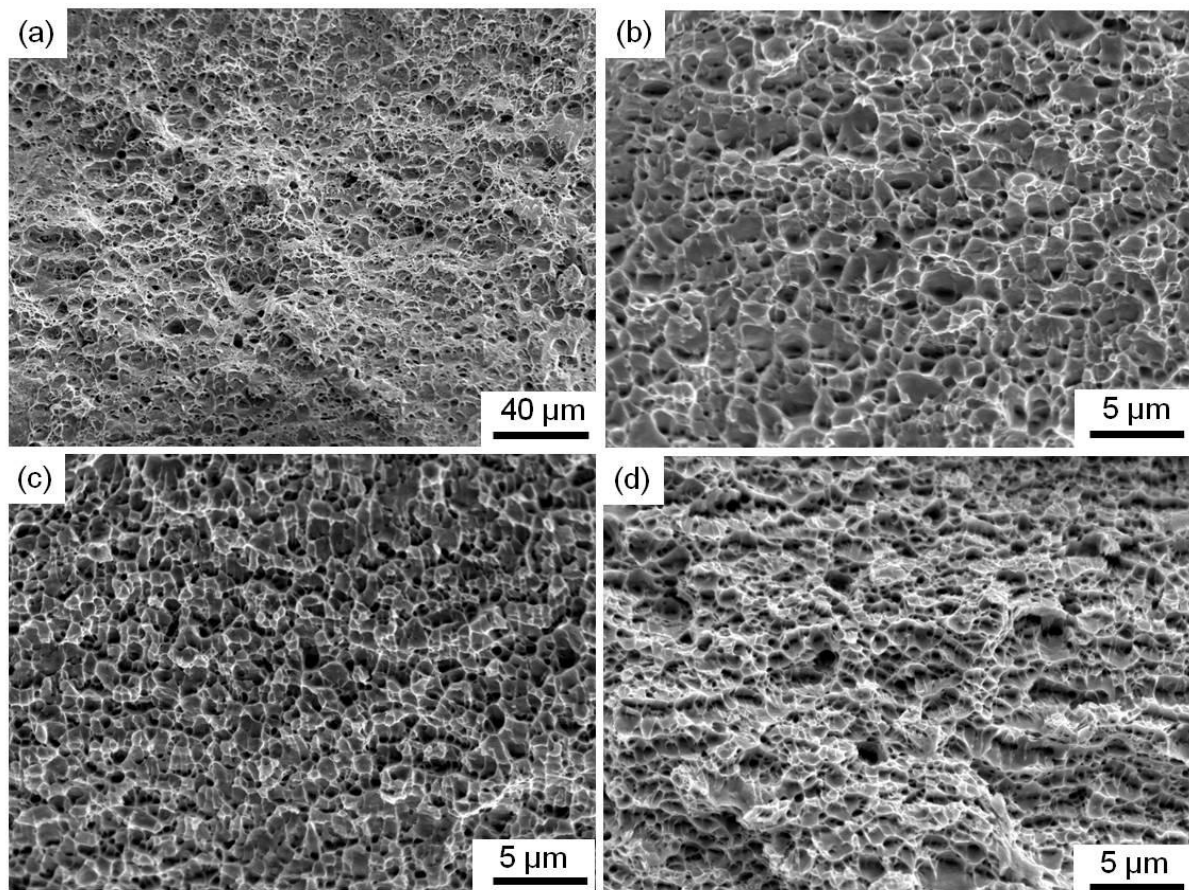


Fig. 5.13 SEM images showing fracture surfaces of the specimens with (a) statically transformed ferrite with grain size of 6700 nm, (b) UFF of 550 nm grain size, (c) UFF of 460 nm and (d) UFF of 350 nm.

Figure 5.14 summarizes the yield strength (a) and uniform elongation (b) of the specimens with UFF structures reported in previous works [31-35] and that obtained by the new TMCP routes in the present study (shown as solid triangle symbol). Compared with the UFF structure obtained in previous works, the UFF structures obtained in the present study have the smallest grain size under the same deformation. The UFF structures obtained in the present study show ultra-high yield strengths, which are comparable to those obtained either by SPD or heavy warm deformation. Qualitatively speaking, in the materials with sub-micrometer grain sizes (for instance, the UFG IF steel), it is usually hard to maintain a considerable uniform elongation because of the early plastic instability during tensile test [36]. It is, on the other hand, rather surprising that the present UFF ferrite obtained through the new TMCP routes maintains large uniform elongation of 8-10%.

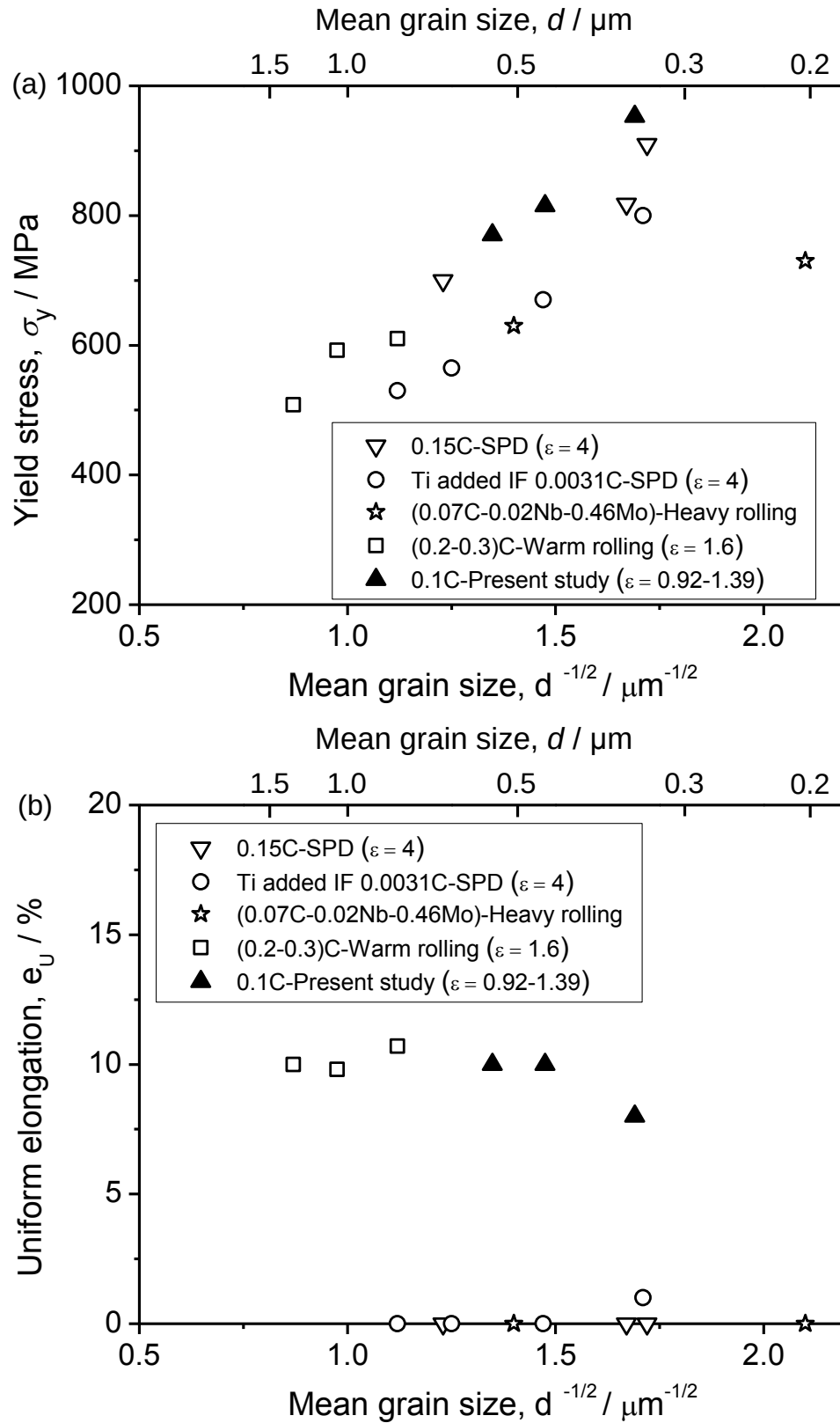


Fig. 5.14 Yield strength and uniform elongation of the specimens with UFF structures (grain size of 350-550 nm) obtained in the present study (solid triangle symbols), compared with the UFF structure reported in previous works [31-35].

5.4 Conclusions

Potential strategies for fabricating UFF structure were explored in the present chapter. Fairly homogeneous UFF structures were obtained through two thermo-mechanically controlled processes (TMCP) composed of two-step deformation at different temperatures. The main results are summarized as follows:

- 1) Pre-deformation of austenite at low temperature (410 °C) introduced a large amount of defects to enhance the DT and subsequent DRX at an elevated temperature (520 °C), resulting in an UFF structure with a mean grain size of 550 nm.
- 2) Pre-deformation of austenite at low temperature (300 °C) led to the formation of martensite partially, which could more enhance the subsequent DT and DRX during deformation at elevated temperature (520 °C), resulting in an UFF structure with a mean grain size of 350-460 nm.
- 3) The UFF structure exhibited ultra-high strength (770-953 MPa of yield strength and 810-973 MPa of tensile strength) and large tensile ductility (8-10% of uniform elongation and 23-29% of total elongation).
- 4) It is noteworthy that the present process uses only 60% compression (equivalent strain of 0.92) in total to obtain the UFF structures with grain sizes around 400 nm. The TMCP routes proposed in this study is expected to be applicable to practical steel industries.

5.5 References

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Chapter 6: Summary and Conclusions

In the present dissertation, a Fe-10Ni-0.1C alloy was used to investigate dynamic ferrite transformation (DT) behaviors during thermo-mechanically controlled process. Several important features of DT have been clarified. The main results obtained in each chapter are summarized as follows.

In **Chapter 1**, background and purpose of present study were introduced.

In **Chapter 2**, occurrence of DT was confirmed through analyzing the softening behaviors in stress-strain curves using a double differentiation method in association with microstructure observations. Critical strains for the onset of DT estimated by the double differentiation method were obtained. The volume fraction of ferrite experimentally observed at the critical strains fell into a range of 2-10%, indicating a good applicability of this method for determining the onset of DT.

The effects of thermomechanical processing parameters on the kinetics of DT and ferrite microstructures were investigated. Low deformation temperature and fine austenite grain size decreased the critical strain for onset of DT and accelerate its kinetics. High strain rates also accelerated the kinetics of DT, but low strain rates decreased the critical strain for onset of DT and led to a higher fraction of ferrite. High strain rates, low deformation temperatures and fine austenite grains enhanced the nucleation for ferrite and suppressed the ferrite grain growth, resulting in fine ferrite structures. The grain size of dynamically transformed ferrite

firstly increased and then decreased after a certain strain, which corresponded to the formation of UFF grains.

In **Chapter 3**, the differences in transformation kinetics and ferrite microstructures between static transformation (ST) from deformed austenite and DT were investigated. For ST, kinetics of ferrite transformation was greatly accelerated by the deformation of austenite. For DT, consuming incubation period before transformation could not effectively enhance ferrite formation, while decreasing strain rate (i.e., increasing deformation time) could greatly enhance ferrite formation. At the same temperature and strain, the kinetics of ferrite transformation was more accelerated by DT than ST (from deformed austenite), which is mainly due to the enhanced α/γ interface mobility during deformation.

The dynamically transformed ferrite exhibited different morphologies from statically transformed ferrite. The statically transformed ferrite was composed of equiaxed UFF grains and equiaxed coarse grains. The equiaxed UFF grains were mainly formed in the vicinity of the prior austenite grain boundaries (PAGBs). Most of the statically transformed ferrite grains were surrounded by high-angle boundaries (HABs), and exhibit relatively random crystallographic orientation distribution. The dynamically transformed ferrite was composed of equiaxed UFF grains and elongated coarse grains. The elongated coarse grains contained a number of subgrains surrounded by low-angle boundaries (LABs), which indicates that they are deformed grains. The equiaxed UFF grains could be formed not only

along PAGBs, but also along the grain boundaries of coarse elongated ferrite grains, and the number of UFF grains increases with increasing the strain, so that the thickness of the UFF regions is much thicker than that of the statically formed fine ferrite grains. The UFF grains show different crystallographic orientation ($\langle 111 \rangle // \text{SD}$ texture) from the elongated coarse grains ($\langle 001 \rangle // \text{SD}$ texture and $\langle 212 \rangle // \text{AD}$ texture). The results suggest that the formation mechanism of the UFF grains in DT is not only different from that in ST, but also different from that of the elongated coarse grains formed in DT.

In Chapter 4, to clarify the mechanism of UFF formation, microstructural evolution during the deformation of dynamically transformed ferrite (route-2) and statically transformed ferrite (route-1) were investigated under the same conditions. Occurrence of dynamic recrystallization (DRX) of ferrite initially formed by DT (in the route-2) was confirmed during the continuous deformation. The deformation concentration in ferrite, which was caused by the difference in strength between austenite and ferrite at elevated temperatures, and the fine ferrite grain size formed by DT enhanced the initiation of DRX. The volume fraction of dynamically recrystallized ferrite increased with increasing the strain, and the grain size of ferrite stably kept a constant value under an identical deformation condition. Dynamically recrystallized ferrite grains contained a number of dislocations and exhibited nearly random textures.

DRX of statically transformed ferrite (in the route-1) was also confirmed to occur. The

volume fraction of dynamically recrystallized ferrite grains increased with increasing the strain, and the grain size stably kept a constant value under an identical deformation condition. Decreasing the initial ferrite grain size shortened the incubation period till the initiation of DRX and accelerated its kinetics.

DRX of dynamically transformed ferrite (in the route-2) showed an opposite strain rate dependency to that of statically transformed ferrite (in the route-1). Three factors, i.e., large amount of lattice defects, large deformation concentration in softer ferrite in the austenite and ferrite dual phase structures, and fine ferrite grain size contributed to the larger fraction of dynamically recrystallized ferrite at higher strain rate in the route-1. The strain rate dependency of DRX in the route-1 changed depending on the kinetics of dynamic transformation. Fast transformation kinetics decreased the differences in deformation concentration and ferrite grain size between different strain rates. Consequently, the strain rate dependency of DRX in the route-1 became similar to that in the route-2 when the transformation kinetics was fast.

The effects of austenite grain size and deformation temperature on DRX of dynamically transformed ferrite were further investigated. A fine austenite grain size could accelerate the kinetics of DT thus to decrease the required strain for the onset of DRX and accelerate its kinetics. The DRX of dynamically transformed ferrite was retarded at a high temperature mainly due to the low nucleation density for ferrite transformation and high grain growth rate.

DRX of ferrite was also retarded at very low temperatures. Equiaxed UFF grains cannot be formed due to the low thermal activation for initiating DRX. The DRX behavior of dynamically transformed ferrite exhibited a C-curve according to the deformation temperature. There is an optimized deformation condition for promoting DRX of ferrite, which is very important for grain refinement of ferrite.

In **Chapter 5**, based on the results obtained in **Chapter 4**, two new TMCP routes were proposed in this chapter for fabricating UFF through DT and DRX. Equiaxed UFF structures with mean grain size of 350-550 nm were obtained through the new TMCP routes without heavy deformation (only 60%-75% compression). The UFF exhibited ultra-high strength (770-953 MPa of yield strength and 810-973 MPa of tensile strength) and large tensile ductility (8-10% of uniform elongation and 23-29% of total elongation). The new TMCP routes proposed in present study are expected to be applicable to practical steel industries.

Chapter 6 concluded the whole research findings in the present research work.

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List of publications

International journal papers

- 1) **L. J. Zhao**, N. Park, Y. Z. Tian, S. Chen, A. Shibata and N. Tsuji. Formation of ultrafine grained ferrite in low-carbon steel through thermomechanical processing using dynamic transformation and dynamic recrystallization. *Metallurgical and Materials Transactions A*, 2014, submitted.
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- 6) **L. J. Zhao**, N. Park, Y. Z. Tian, A. Shibata and N. Tsuji. Effects of thermomechanical parameters on kinetics of dynamic ferrite transformation during thermo-mechanically controlled Process. *To be submitted*
- 7) **L. J. Zhao**, N. Park, Y. Z. Tian, A. Shibata and N. Tsuji. Effects of thermomechanical parameters on microstructural evolution of dynamically transformed ferrite during thermo-mechanically controlled Process. *To be submitted*.

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