

**Formation Mechanisms of Fine/Ultra-fine Grains
in Metals Highly Deformed by Torsion at Various
Temperatures and Strain Rates**

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Formation Mechanisms of Fine/Ultra-fine Grains in Metals Highly Deformed by Torsion at Various Temperatures and Strain Rates



by

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Chapter 1: Background and purpose

1.1. Introduction

Conventional hot deformation is one of the most common methods to fabricate metallic products. Traditionally, the microstructures formed during hot deformation are categorized in two types; that is, (1) dynamic recrystallization (DRX) structure composed of nearly equiaxed grains often observed at higher temperature and lower strain rate and (2) pancaked or deformed structure at lower temperature and higher strain rate. In the latter case, dynamic recovery (DRV) is the main restoration mechanism during the deformation since only subgrains could form inside the initial deformed grains. However, such a microstructural categorization was often based on the limited amount of strain ($\varepsilon < 2$) which was common in most of the hot deformation processes.

However, in later 1980s, it was found out through applying intense plastic deformation (even at ambient temperature) that another new grain structures with grain sizes below 1 μm could be achieved. The microstructure evolution in such a method (nowadays known as SPD: severe plastic deformation) is believed to be governed by the phenomena so-called grain subdivision ¹⁻⁴, which will be explained in a later section. Compared to the DRX grains, much higher strains are required for the ultra-fine grains formed through the grain subdivision process.

Such a major breakthrough by SPD, anyway, has aroused a lot of curiosity especially from scientific viewpoints. In other words, there have been a lack of universal understanding regarding the formation of new grain structures during plastic deformation under various conditions. Accordingly, the purpose of the present study is to systematically examine a wide range of the high-strain deformation conditions (i.e., various deformation temperatures and strain rates), under which different types of microstructure evolution (leading to new fine grain structures) can be investigated. This chapter firstly explains the background of different deformation processes and related mechanisms. Then, the motivation behind the current study is introduced in details.

1.2. Advantage of grain refinement

Grain size in polycrystalline metals is one of the most important parameters to have a great impact on their properties. Therefore, controlling the grain size has become one of the priorities in designing the thermo-mechanical processing of metallic products ⁵. In general, refining the grain size favors most of mechanical properties ^{6,7}. For instance, the grain size (D) – yield stress (σ_y) relationship can be defined according to the well-known Hall-Petch ^{8,9} relationship (**Eq. 1.1**):

$$\sigma_y = \sigma_0 + k_y D^{-0.5} \quad (1.1)$$

where σ_0 is the lattice friction stress and k_y is a constant. Thus, decreasing the grain size increases the strength of polycrystalline metals.

On the other hand, in recent decades, enormous efforts have been made to fabricate a special class of structural materials, i.e. bulk ultra-fine grained (UFG) metals with submicron grain sizes (more information can be found here ^{6,7,10,11}). This type of innovative materials exhibit some superior properties including improved toughness and extraordinary high strength ^{12,13}.

For example, **Figs. 1.1a-f** show TEM microstructures of an ultra-low carbon interstitial free (IF) steel with various mean grain sizes ranging from 0.2 μm to 13 μm . Different grain sizes in the IF steel specimens were achieved by accumulative roll bonding (ARB) for 5 cycles and subsequent annealing process at various temperatures. **Figure 1.1g** shows true stress - true strain curves of the IF steel with various grain sizes (d_t). It is clear that with decreasing the grain size, the yield strength increased. In **Fig. 1.2**, mechanical properties obtained from **Fig. 1.1g**, i.e., the yield stress and tensile strength are plotted as a function of minus square root of the grain size (d_t). It could be said that strength held Hall-Petch relationship (**Eq. 1.1**) and particularly the yield strength greatly increased by grain refinement. However, the Hall-Petch slopes (k) were different between the yield stress and the tensile strength in the coarse grain region with the grain size above 1 μm .

Nowadays, it is well known that ARB and other types of high-strain deformation so-called severe plastic deformation (SPD) are promising techniques to fabricate the UFG-ed structures ^{6,10,11}. Details of the SPD processes are described in the next section.

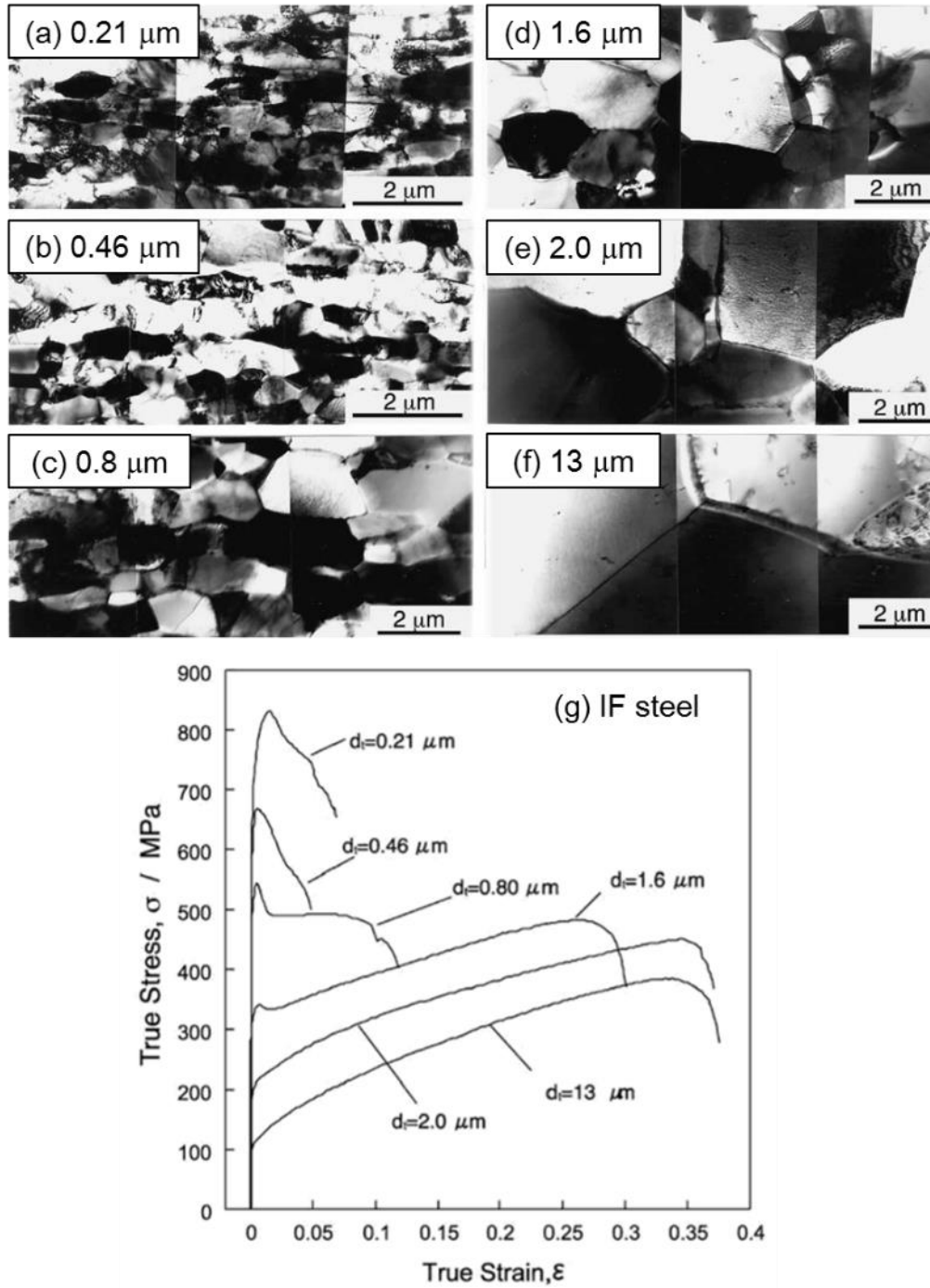


Figure 1.1. (a-f) TEM microstructures of an IF steel annealed for 1.8 ks at various temperatures (400 °C - 700 °C) after 5 cycles of ARB at 500 °C. (g) True stress – true strain curves of the IF steel with various grain sizes (d_i)¹².

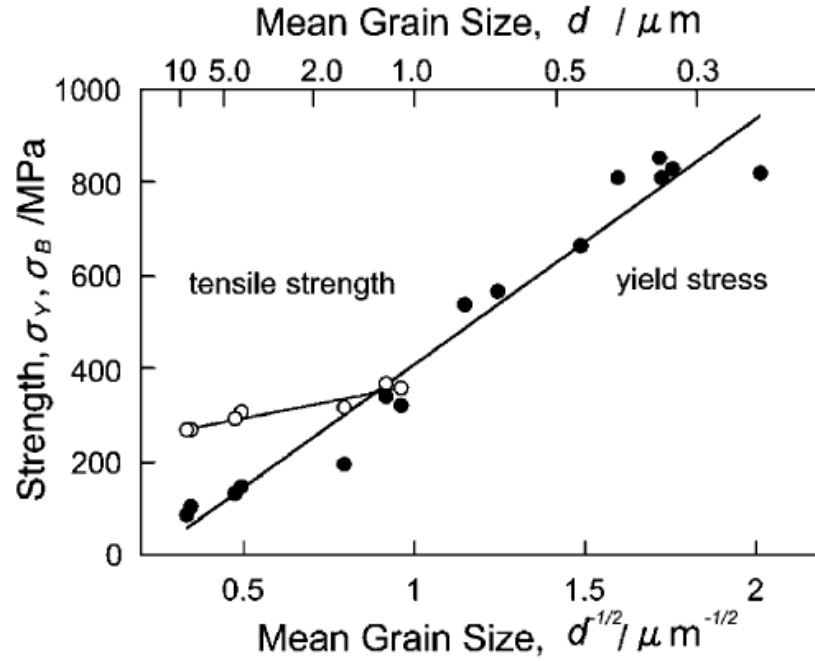


Figure 1.2. Grain size dependence of strength of the IF steel with various grain sizes, previously shown in Fig. 1.1.

1.3. Severe plastic deformation (SPD)

1.3.1. Methods

In general, SPD can be defined as a method to introduce a very large plastic strain to bulky materials without significant dimensional change in the specimens ¹⁴. The schematic plots in **Fig. 1.3** ¹¹ show the principals of common SPD processes. The corresponding equations shown in **Fig. 1.3** define relationships between the equivalent strain (ϵ) and n as the number of rotations, passes or cycles in different SPD methods. These methods are explained furthermore in this section.

Background of SPD processing can be traced back to a classical work carried out by P.W. Bridgman ¹⁵ in 1930s. This can be considered as the first attempt to apply a high pressure accompanied with torsion deformation on bulk metals. This process evolved further into a method nowadays known as the high pressure torsion (HPT) (**Fig. 1.3a**), where high hydrostatic compressive pressure is substantially applied to the material to avoid fracture during high strain deformation by torsion.

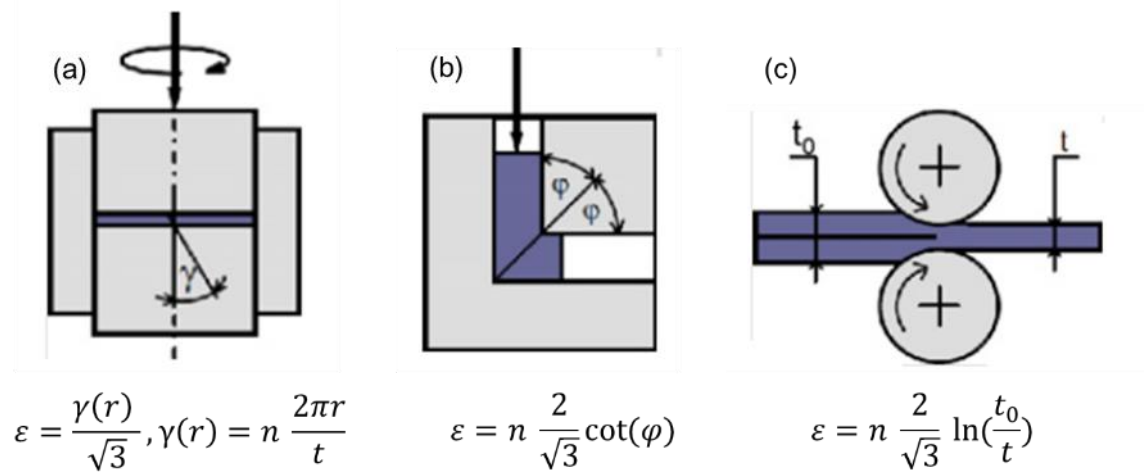


Figure 1.3. Schematic illustrations of well-known SPD processes as well as corresponding equations to calculate the equivalent plastic strain (ε) in each method (the original figures after A. Azushima et al. ¹¹): (a) high pressure torsion or HPT (γ : shear strain, n : number of rotations, r : radial distance from the disk center, t : thickness of the disk), (b) equal-channel angular pressing or ECAP (n : number of passes through the die, φ : angle of intersection of two channels), and (c) accumulative roll bonding or ARB (n : number of cycles, t_0 : initial thickness of the sheet, t : thickness after roll-bonding).

However, it should be noted that it was only after late 1980s (more details here ¹⁶) when enormous interests in the refined microstructures of SPD processed specimens were expressed. The main reason was simply due to the development advanced methods for nanostructure observations such as high resolution transmission electron microscopy (HRTEM), electron back-scattered diffraction (EBSD), etc., which made it possible to characterize the UFG structures with sub-micrometer scales ⁷.

Another important step in the history of SPD processing was when V.M. Segal et al. ¹⁷ developed a technique known as the equal-channel angular pressing (ECAP) or extrusion (ECAE) (**Fig. 1.3b**). Here the specimen passes through an angular channel in a die, experiencing large/uniform shear strain without changing its cross section. This method has become one of the most popular techniques among the SPD processes. N. Tsuji et al. ^{18,19} developed another innovative intense plastic deformation so-called the accumulative roll bonding (ARB) (**Fig. 1.3c**), which is especially capable of producing larger-size bulk UFG structures.

In addition to these popular SPD methods, many other novel high-stain plastic deformation processes have been proposed, such as repetitive corrugation and straightening (RCS) ²⁰, severe torsion straining (STS) ²¹, etc.

1.3.2. Grain subdivision phenomena

In general, formation of UFGs during the intense plastic deformation in polycrystalline metals can be understood in terms of *grain subdivision* ¹⁻⁴. Here, the substantial grain refinement is caused by the generation of various kinds of deformation-induced boundaries. The process of grain subdivision is illustrated in **Fig. 1.4** ²². Plastic deformation in metals can happen by dislocation slips. In polycrystalline metals, the slip patterns (amounts and combinations of activated slip systems) change from one grain (crystal) to another grain and can be different even within an identical grain.

In other words, the prior grain splits into several smaller units with different slip patterns combinations of slip systems. Due to the dislocation glide process, lattice rotation to different crystallographic orientations occurs in different domains that had different slip patterns activated. This indicates that the resultant misorientation must be accommodated by the presence of boundaries whose misorientation gradually increases with increasing the applied strain. The boundaries are called geometrically necessary boundaries (GNBs) (as shown in **Fig. 1.4**). There are also other boundaries called incidental dislocation boundaries (IDBs). The latter form by random trapping of dislocations (or statistically stored dislocations) ¹⁻⁴. With further straining, the boundary (especially GNBs) spacing decreases while their misorientation

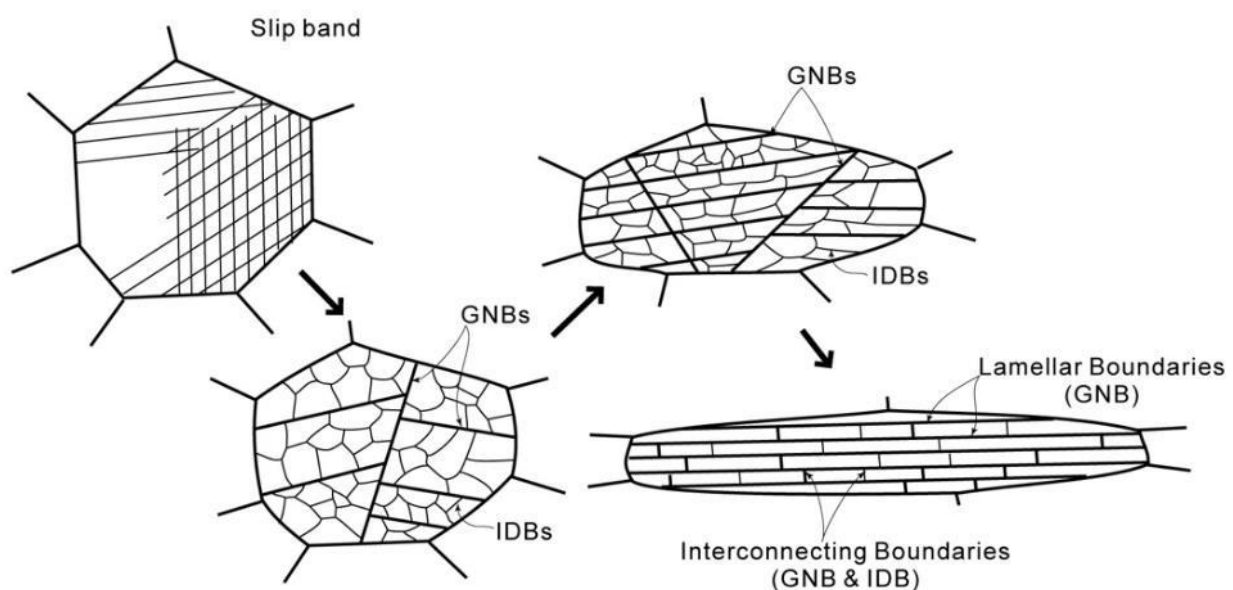


Figure 1.4. Schematic illustration showing the *grain subdivision* mechanism during plastic deformation ²².

increases ¹⁻⁴. As a result, the lamellar structure forms eventually at high strains, where the lamellar boundaries are GNBs (**Fig. 1.4**).

In **Fig. 1.5a**, the TEM microstructure shows an arrangement of GNBs and IDBs in a pure nickel processed by cold-rolling with 20% rolling reduction ⁴. **Figure 1.5b** shows a power-law relationship between the misorientation angle and the equivalent strain in cold-rolled Al and Ni ^{3,23}. It can be seen that the slope of the plot is significantly larger for GNBs. In other words, at large strains, misorientation across GNBs reaches above 15 degree (a criterion to define high angle boundaries). As explained in the previous section, very high strains can be achieved by SPD methods.

A representative microstructure evolution in SPD is shown in **Fig. 1.6** ²⁴. EBSD grain boundary maps show the microstructures of a 1100-Al, ARB processed by various cycles at room temperature. With increasing the number of ARB cycles, the spacing of high angle boundaries (illustrated by the green lines) decreased. After 6 cycles, an uniform UFG structure dominated by the lamellar high angle boundaries formed. It is noteworthy that the spacing of high angle boundaries in **Fig. 1.6** tended to saturate with increasing the ARB cycles.

N. Tsuji et al. ²⁵ studied the saturation of grain size in different metals (aluminium, iron, etc.) processed by ARB. As an example, an 1100-Al was deformed to different

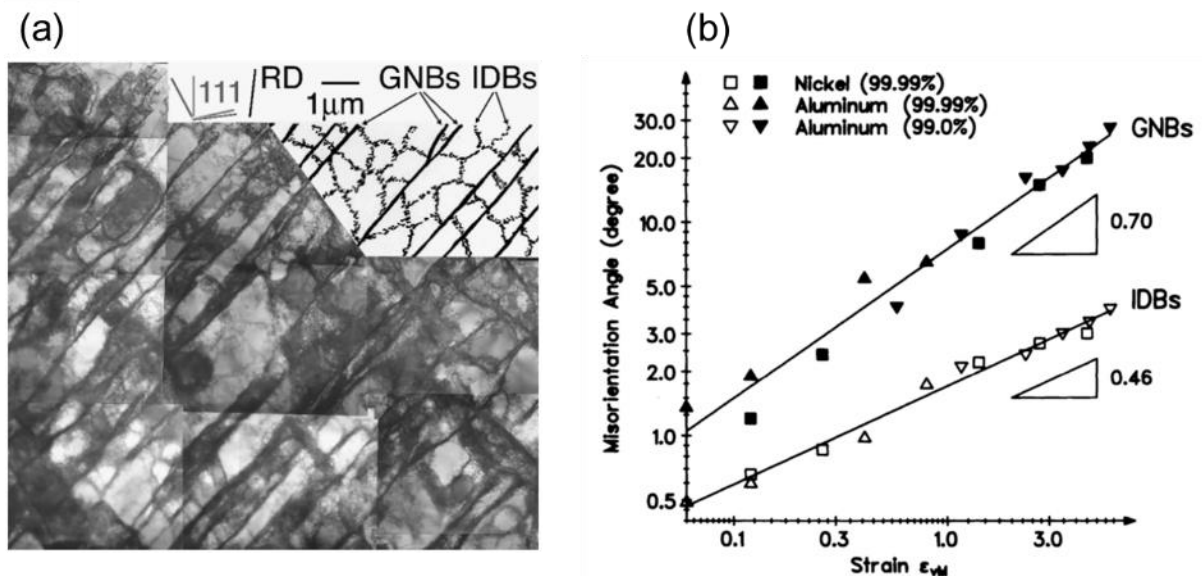


Figure 1.5. (a) Transmission electron micrograph associated to a pure Ni cold-rolled with 20% rolling reduction, showing arrangement of dislocation boundaries as solid lines (GNBs) and speckled line (IDBs) ⁴, and (b) misorientation angle as a function of equivalent strain plotted for IDBs and GNBs for cold-rolled Al and Ni ^{3,23}.

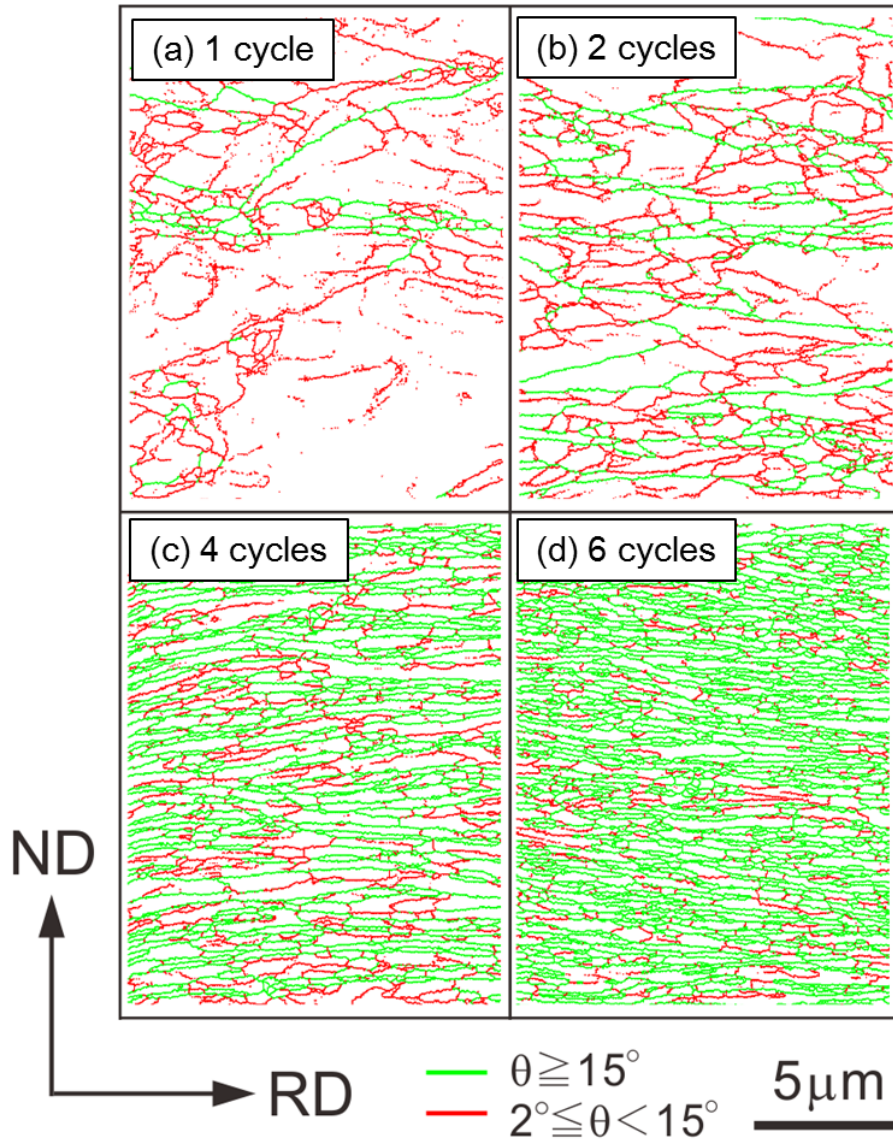


Figure 1.6. EBSD boundary maps in a 1100-Al ARB processed at room temperature by various cycles ²⁴. Observed from the transverse direction (TD) of the sheets.

ARB cycles and TEM observation was carried out to measure the mean grain thickness (or the interval of lamellar boundaries along the normal direction (ND)). **Fig. 1.7** shows the mean grain thickness plotted as a function of the macroscopic equivalent strain applied by the ARB process. The geometrically expected thickness of initial grains was also calculated from the total reduction in thickness (strain) and shown as a broken line in **Fig. 1.7**. Firstly, the measured grain size greatly decreased at early stages of ARB, however the rate of grain refinement decreased at higher strains.

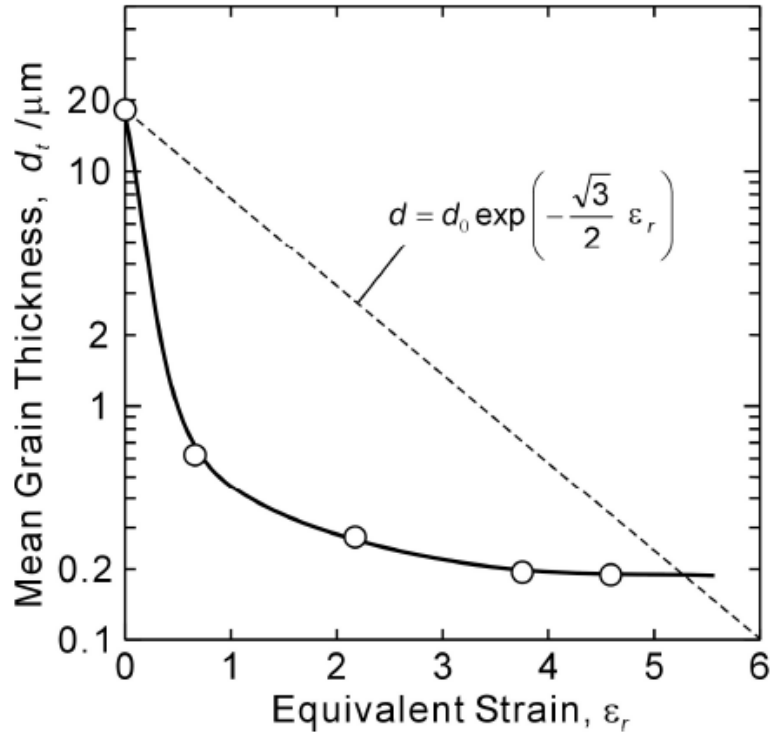


Figure 1.7. Mean grain thickness (interval of lamellar boundaries) in a 1100-Al as a function of ARB strain ²⁵.

Eventually, the grain size of the UFG 1100-Al saturated and became nearly close to the geometrically expected thickness of initial grains. Similar tendency of grain refinement was also confirmed in other metals such as IF steel and copper. One possible explanation for the grain size saturation could be given by considering the effect of adiabatic heating, especially in heavy rolling at low temperature. In other words, microstructure coarsening could happen due to a possible temperature rise in ARB (or SPD) processed specimen, causing a limitation on further refinement of the grains ²⁵. However, this topic requires more investigation to be clarified.

1.4. Conventional hot deformation

Hot deformation, such as hot rolling, hot forging, hot extrusion, etc., is a widely used industrial method to fabricate many metallic products. On the other hand, it is a common task to adjust microstructural parameters during the hot deformation

processing, especially to refine the grain size by controlling the deformation conditions. However, comparing to the SPD processes, the size of grain structures obtained in the thermo-mechanical processes at elevated temperatures is much larger (above 1 μm).

At elevated temperatures, thermally activated phenomena, so-called restoration mechanisms including recovery, recrystallization and grain growth, become dominant. As a result, in addition to the strain, the microstructure evolution is dependent on both deformation temperature (T) and strain rate ($\dot{\epsilon}$). Zener-Hollomon (Z) parameter ²⁶ is an effective parameter for understanding the hot deformation conditions including both temperature and strain rate. The Z parameter is defined as:

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) \quad (1.2)$$

where Q is the apparent activation energy of the deformation and R (8.31 Jmol⁻¹K⁻¹) is the gas constant. Using Z parameter is convenient for discussing deformation behaviors (including flow stresses) and microstructure evolution in hot deformation.

1.4.1. Dynamic restoration mechanisms

The deformed microstructure includes high density of point defects (like vacancies) and dislocations that act as a driving force for the restoration mechanisms such as recovery, recrystallization, and grain growth. Annihilation and rearrangement of dislocations into lower energy configurations happen during recovery ²⁷. These events involve dislocation movement such as dislocation glide, climb, and cross-slip. On the other hand, recrystallization might be considered as a process in which “a *crystallite of low internal energy grows into the deformed (or recovered) matrix from which the recrystallized grain is separated by high angle grain boundary*” ²⁷. In one point of view, recovery and recrystallization are competing processes to decrease the stored energy in deformed materials. In another viewpoint, recovery plays an important role to form “a *crystallite of low internal energy*” or recrystallization nucleus. Therefore, it might be somehow difficult to make a clear division between recovery and recrystallization ²⁷.

Recovery and recrystallization can occur during deformation at high temperatures. In other words, the restoration processes occur simultaneously with strain hardening during hot deformation. In that case, the processes are called dynamic recovery

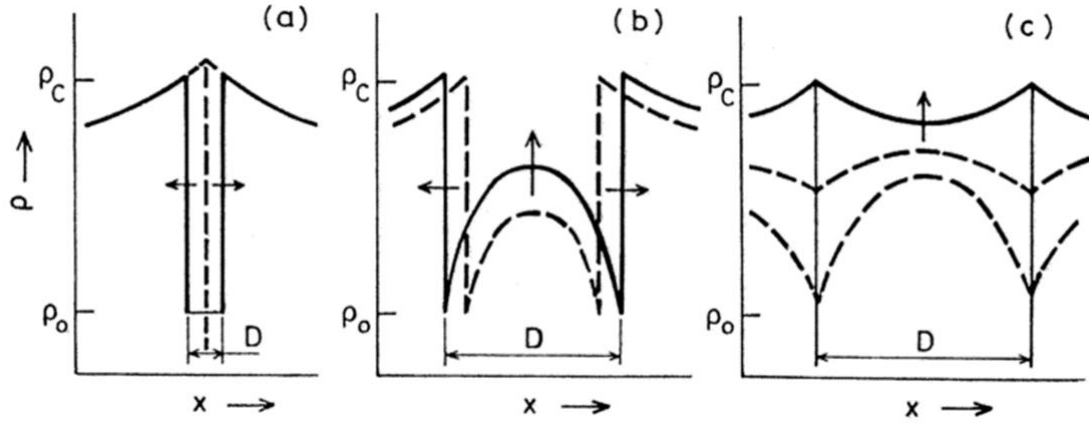


Figure 1.8. A model describing the development of a DRX grain (after T. Sakai et al. ²⁸): (a) DRX nucleus, (b) growing DRX grains, and (c) DRX grain undergoing strain hardening. ρ_0 and ρ_c are the initial dislocation density and the critical density required for DRX, respectively.

(DRV) and dynamic recrystallization (DRX). **Figure 1.8** represents a model ²⁸ to describe the microstructure evolution under a DRX condition. In general, a DRX grain can be expressed in three stages; that is, a DRX nucleus with a low dislocation density compared to that of surrounding deformed (strain-hardened) matrix (**Fig. 1.8a**), a growing DRX grain (**Fig. 1.8b**), and a DRX grain again being deformed (**Fig. 1.8c**). Here, ρ_0 and ρ_c in **Fig. 1.8** are the initial dislocation density and the critical density required for the occurrence of DRX, respectively.

DRX events are normally initiated by serration or bulging of initial grain boundaries and new grains form along the initial grain boundaries, which is considered as "nucleation" of DRX grains. Providing that DRX leads to the grain refinement (DRX grains smaller than the prior grains), a typical type of DRX structure so-called necklace structure can be realized. The development of such a structure is simply illustrated in **Fig. 1.9** ²⁹. The onset of DRX occurs at a critical strain (ϵ_c) smaller than the peak strain (ϵ_p) on the stress-strain curve. The first layer of necklaced DRX grains form along initial grain boundaries and the next layers form consecutively, expanding into the grain interior. As a result, the flow stress drops until it reaches a steady-state condition. However, this illustration is somehow ideal to describe the typical DRX behavior. Actual DRX events may happen in a more complicated way and somewhat different from that drawn in **Fig.1.9**, depending on the kind of materials and deformation conditions. Even for the necklace-type model, it was claimed ²⁹ that the

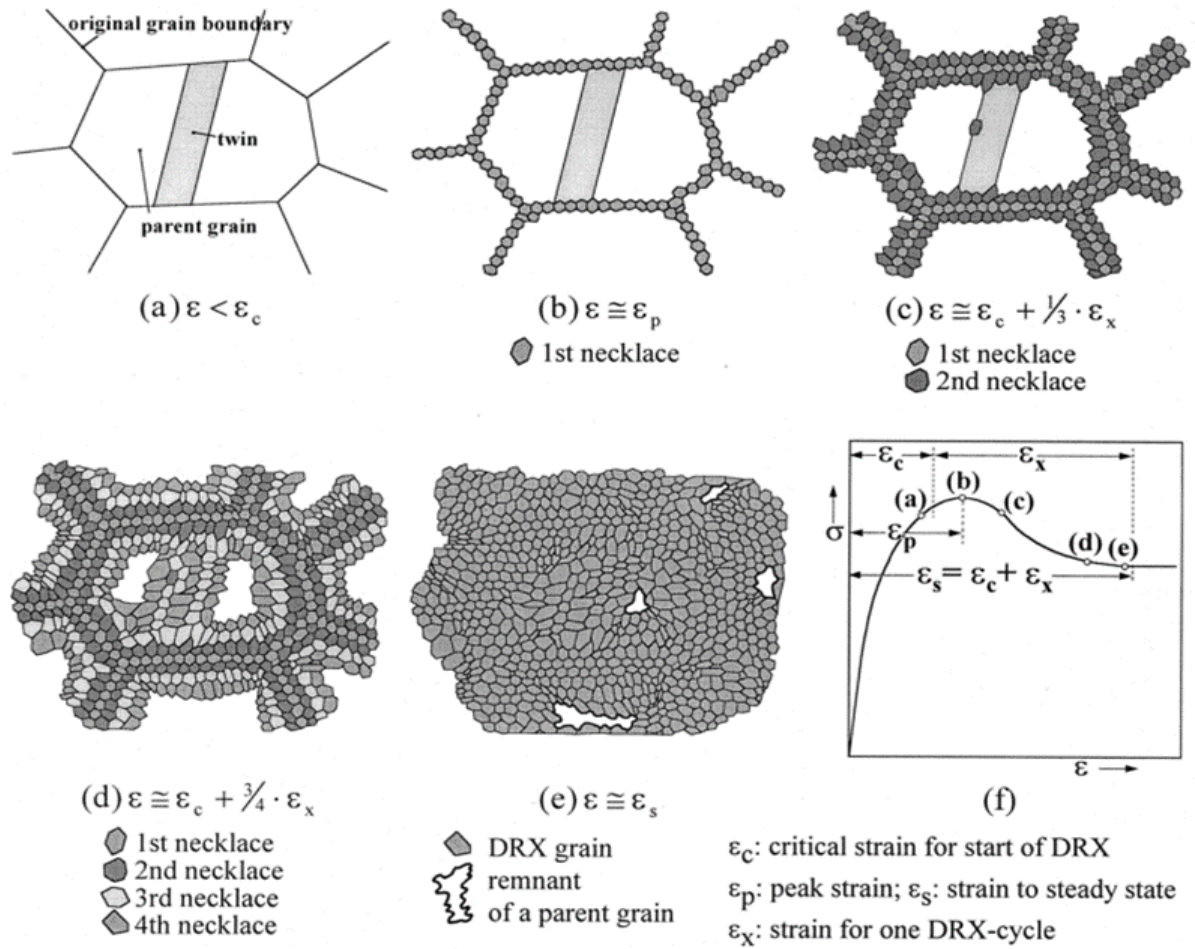


Figure 1.9. (a-e) A model illustrating the development of consecutive necklace formation of DRX grains, starting from prior grain boundaries and expanding into grain interior. (f) The corresponding flow stress showing different strains including the critical strain (ϵ_c) for the onset of DRX, the peak strain (ϵ_p) and the strain to start the steady state (ϵ_s) (D. Pong et al. ²⁹).

mechanism of the first DRX layer could be different from that during the subsequent expansion of DRX.

Figure 1.10 shows an actual example at early stage of necklace-like DRX grains in a pure copper deformed in a compression test ($\epsilon \sim 1$) at 300°C and a strain rate of 0.16 s⁻¹ ³⁰⁻³². The size of DRX grains in the necklace regions was dependent on the deformation conditions, so that the grain size decreased with increasing the Zener-Hollomon (Z) parameter; i.e. with decreasing the temperature and increasing the strain rate. Additionally, the OIM (orientation imaging microscopy) map in **Fig. 1.10b** provides more details about the misorientation data. It was suggested that near the

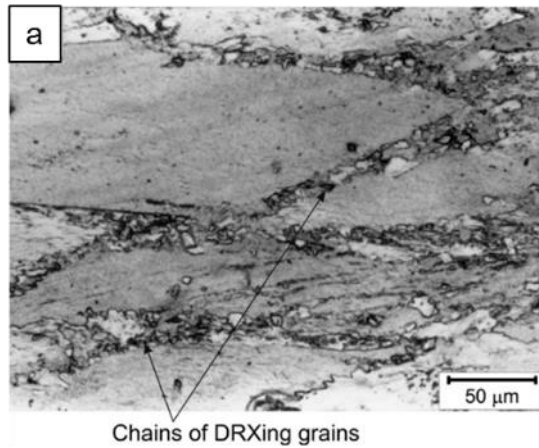
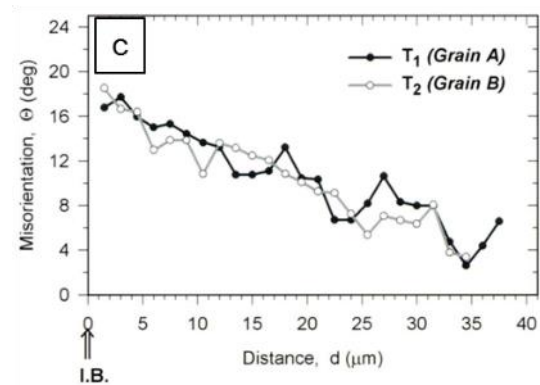
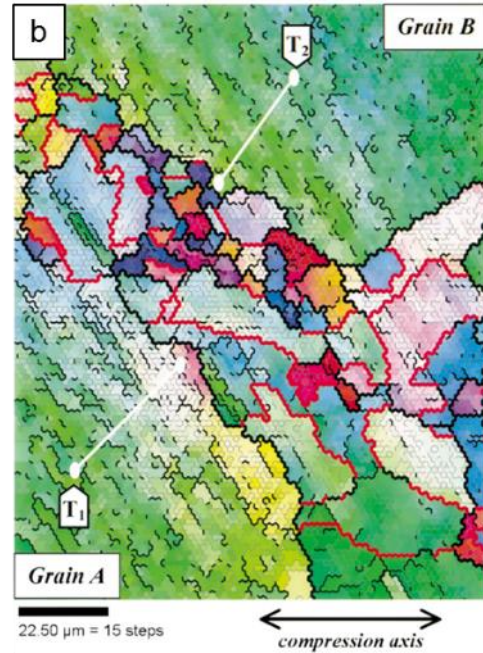


Figure 1.10. (a) A necklace-like DRX structure forming during a compression of a pure copper at 300°C. (b) OIM map of the DRX region in the pure copper (99.9999%) deformed in compression under 300°C and 0.16 s⁻¹ condition. Boundaries are shown by thin black lines ($\theta > 4^\circ$), thick black lines ($\theta > 15^\circ$), and red lines (twin boundaries). (c) Misorientations measured along the lines T1 and T2. I.B. as the initial boundary between the parent grains and the DRX region ^{30–32}.



DRX region (inside the parent grains), strain/orientation gradients developed (**Fig. 1.10c**) and therefore bulging could repeatedly operate.

The illustration presented in **Fig. 1.11** is one of the most popular models proposed as a DRX mechanism by T. Sakai et al. ³¹. Here, the bulging of the serrated segments of initial grain boundaries accompanied with the formation of geometrically necessary boundary (at lower temperature) or annealing twin boundary (at higher temperature), are mainly responsible for the nucleation of DRX grains. Such a process can be additionally assisted by local strain gradients caused by grain boundary sliding or shearing.

1.4.2. Stress - grain size plot

During the elevated temperature deformation when dynamic recovery is possible,

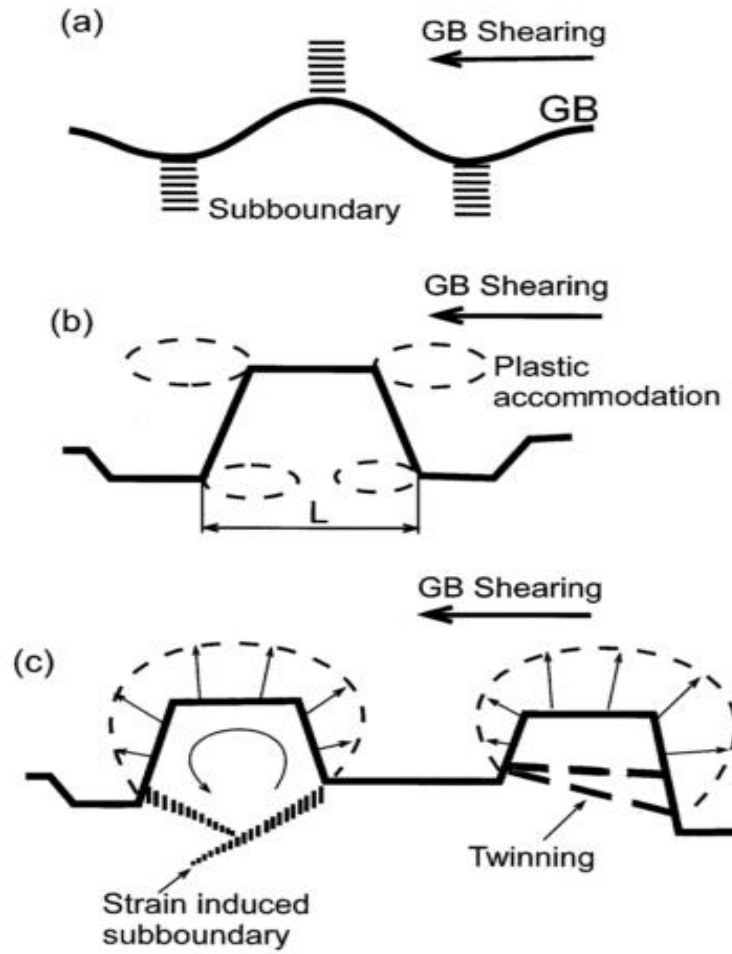


Figure 1.11. Nucleation of DRX grains: (a) boundary corrugation with a development of sub-boundaries, (b) grain boundary sliding or shearing leading to inhomogeneous local strains, and (c) bulging of serrated grain boundaries accompanied with the evolution of sub-boundaries or annealing twins behind ³¹.

it is generally accepted that there is a strong relationship between the steady-state subgrain size (d) and the flow stress (σ). Sherby et al. ³³ proposed that the subgrain size and the flow stress are reversely proportional; that is, $\sigma d = \text{constant}$. Furthermore, Takeuchi et al. ³⁴ suggested a normalized relationship between the subgrain size and the flow stress as shown in **Eq. 1.3**:

$$(\sigma/G)(d/b) = K \quad (1.3)$$

where σ is normalized by shear modulus (G) and d is normalized by Burgers vector (b). Here, K is a constant for a certain type of materials; e.g. $K \sim 10$ for metals, and

$K = 25 \sim 80$ for ionic crystals having NaCl structure. In another research, Derby et al. ³⁵ plotted the subgrain sizes versus the stress in various types of metals and minerals, which is depicted in **Fig. 1.12a**.

Similar to the subgrain size, there have been several attempts to define a relationship between the steady-state grain size (D) and the flow stress using the experimental data. In general, such an empirical relationship can be expressed as **Eq. 1.4**:

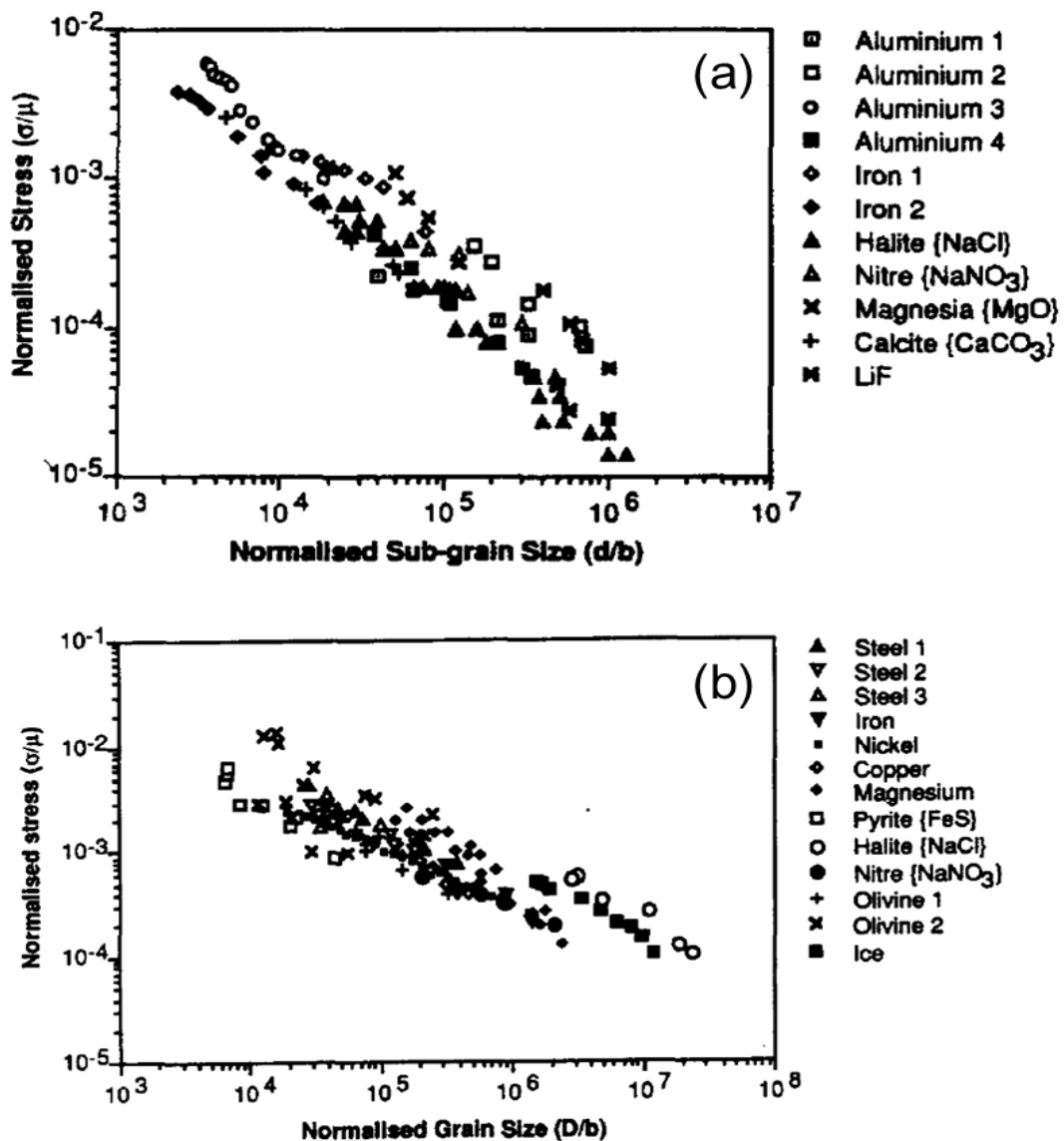


Figure 1.12. (a) The mean subgrain size formed by dynamic recovery (normalized by Burgers vector: b) versus the flow stress at high temperature deformation (normalized by shear modulus: G). (b) Similar plot for the mean steady state grain size formed by dynamic recrystallization ³⁵.

$$\sigma D^N = K \quad \text{or} \quad \sigma = K D^{-N} \quad (1.4)$$

where K is a constant and N can take values approximately in the range of $0.4 < N < 1.0$, depending on the type of materials ³⁵. This relationship or other similar forms are quite important since they can be used for better understanding of the formation mechanisms of fine grains during deformation at elevated temperatures, nevertheless their interpretation has been still argued.

Twiss et al. ³⁶ suggested an universal relationship between the normalized grain size and the normalized flow stress for a number of materials, according to **Eq. 1.5**:

$$(\sigma/G)(D/b)^{0.8} \approx 15 \quad (1.5)$$

Similarly, σ is normalized by shear modulus (G) and D is normalized by Burgers vector (b). However, there were some criticisms ³⁷ about the physical meaning of **Eq. 1.5**, as the exponent 0.8 was roughly chosen and it actually changed in a range of $0.4 \sim 0.9$. Another important point was to discriminate between materials showing different mechanisms of recrystallization; i.e., rotational recrystallization and migration recrystallization (terminologies used for minerals ³⁸). In rotational recrystallization, the subgrain rotation leads to the progressive increase in misorientation angles of sub-boundaries until high angle grain boundaries form. As a result, the sizes of subgrains and recrystallized grains are nearly equal to each other. On the other hand, migration recrystallization involves the migration of boundaries towards the grain interiors (matrix) with higher dislocation density. Therefore, this type of recrystallization occurs in the materials in which dislocation distribution is highly heterogeneous. However, it is uncertain whether the subgrain rotation and the grain boundary migration can be considered as totally independent processes ³⁹. (Comparable metallurgical concepts for the rotational and migration recrystallization seem to have been referred as continuous and discontinuous recrystallization, respectively.)

Later, Derby et al. ³⁵ tried to more justify the possibility of the universal relationship. Considering some assumptions, they applied the normalized plot for a range of minerals and metals, which is shown in **Fig. 1.12b**. It was found that the experimental data in **Fig. 1.12b** fell within a remarkably narrow range bounded within $K = 1, 10$, and N was estimated around $2/3$ (**Eq. 1.6**).

They suggested a dynamic recrystallization model in order to provide a physical basis to explain the derived empirical relationship. Finally, it was claimed that the DRX model considering a dynamic balance between the formation rate of the deformation substructure and the mean migration velocity of recrystallizing grain boundaries can predict the empirical relationship (**Eq. 1.6**).

$$1 < (\sigma/G)(D/b)^{-2/3} < 10 \quad (1.6)$$

The data used in **Fig. 1.12b** was later redrawn by Sakai et al.⁴⁰ using only the data belonging to the fcc-metals and hcp-magnesium, which is represented in **Fig. 1.13**. Considering only fcc-metals showing conventional DRX behaviors, it can be seen that the data fell even in a much narrower range bounded by solid lines in **Fig. 1.13** ($K = 1$,

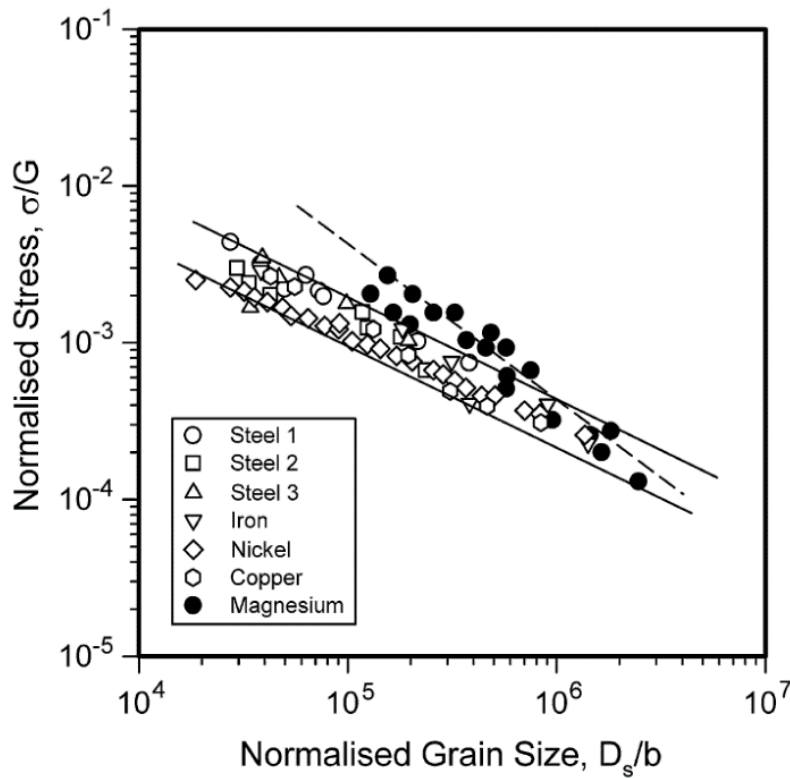


Figure 1.13. Normalized grain size versus normalized flow stress in hot-deformation for metallic materials⁴⁰ (from the original plot by Derby³⁵ shown in Fig. 1.12).

2). It is interesting that the magnesium data is approximated by the broken line with a different gradient of $0.9 \sim 1$.

1.4.3. Case of easy-recovery metals

In spite of the fact that both DRV and DRX are restoration mechanisms, sometimes they are considered as competitive processes, particularly in case of easy-recovery fcc metals with high stacking fault energy such as aluminum and ferritic steels with bcc structure. As a result, it is generally presumed that the occurrence of DRX is suppressed by the enhanced DRV ^{41,42} in such metals, because of the decrease of the driving force (dislocation density) for recrystallization. Nevertheless, evidences for DRX have been claimed even in ferritic steels and aluminum (DRV-type metals) by some researchers, particularly in case of high purity, such as the classical work by Glover et al. ^{43,44} on a vacuum-melted iron and other studies on ultra-low carbon IF steel ^{45–47} and high purity ($> 99.999\%$) aluminum ^{48–50}.

Figure 1.14 shows flow stress-strain curves of an IF steel deformed at 850 °C with

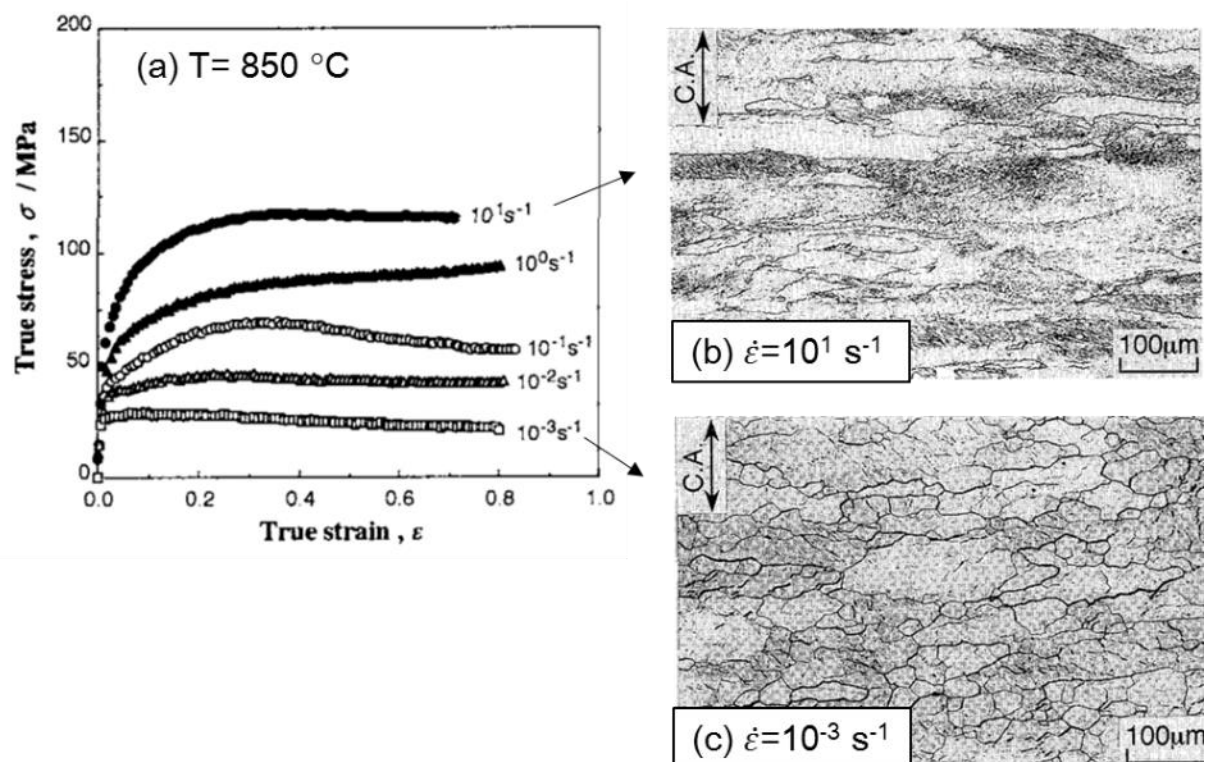


Figure 1.14. (a) Stress-strain curves of an IF steel deformed at 850°C with various strain rates. (b, c) Optical microstructures of the deformed IF steel specimens ($\epsilon = 0.8$) at two different strain rates ^{45,46}.

various strain rates ^{45,46}. As can be seen, such a flat-type flow stress (with slight softening: barely showing a peak stress) is typical in easy-recovery metals. Nonetheless, the formation of DRX grains under low Z conditions, i.e., at low strain rate and high temperature (e.g. **Fig. 1.14c**) was confirmed by microstructural and crystallographic evidences. It was also pointed out ^{45,46} that a noticeable feature of DRX in ferrite is inhomogeneity of recrystallization (with some initial grains hard to recrystallize possibly due to their particular orientation).

Moreover, there is another concept so-called geometrically DRX (GDRX) which

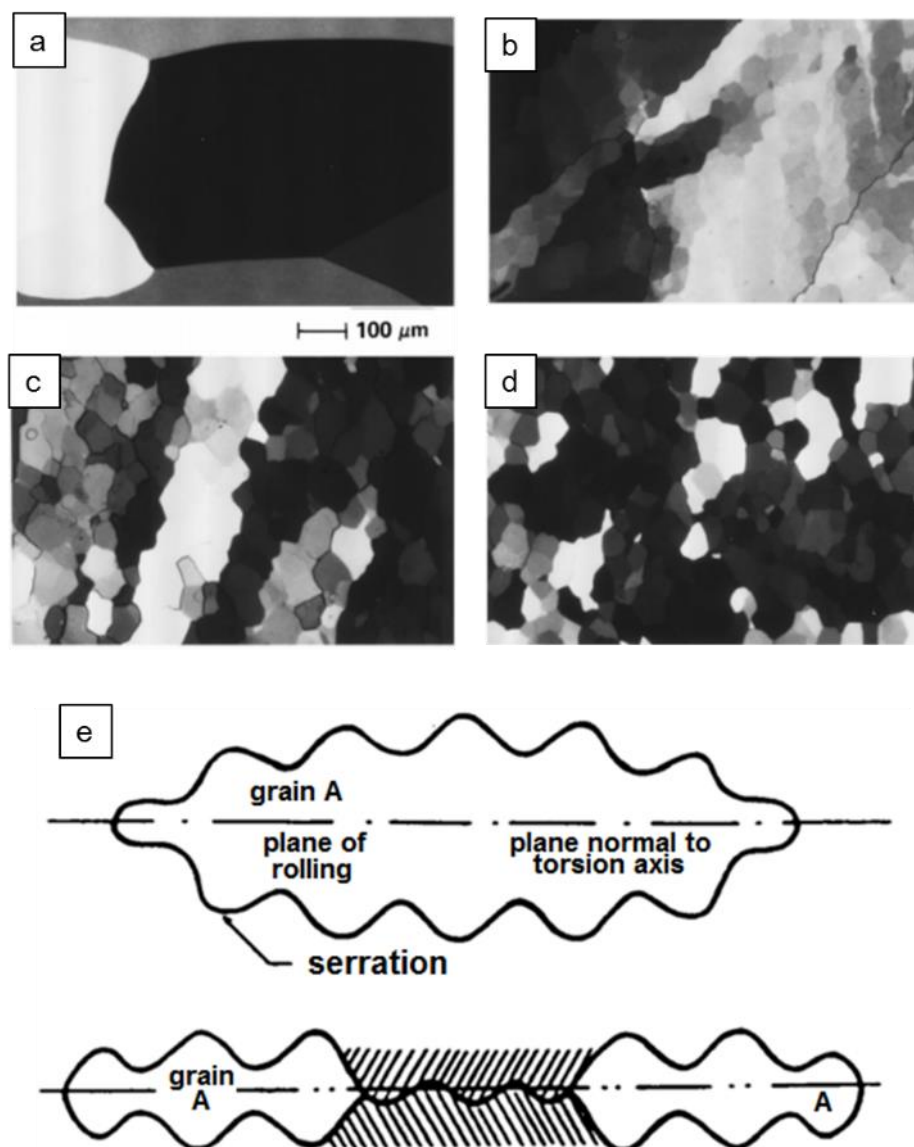


Figure 1.15. (a-d) Polarized optical micrographs of a torsion-deformed pure aluminum deformed at 370°C and 0.0005s^{-1} to different strains of (a) 0, (b) 0.6, (c) 4, and (d) 16. (e) A model showing opposite serrations making contact: “pinch off” ⁵⁴⁻⁵⁷.

was firstly introduced by McQueen et al. ^{51–53}, which has been used to explain the microstructure evolution at elevated temperatures and large strains, mainly in high stacking fault metals such as aluminum ^{54–57} (**Fig. 1.15**). Firstly, the high angle boundaries become serrated (**Fig. 1.15b**) due to the connected sub-boundaries (which easily form in aluminum). The wavelength of serrations is roughly similar to the size of subgrains. The grain thickness becomes smaller due to the geometrical shape change, i.e., grain elongation and thinning, with increasing the total strain (**Fig. 1.15c**). Eventually when the grain thickness becomes comparable to the subgrain size, impingement of the serrated boundaries on the opposite side may happen (**Fig. 1.15e**), resulting in the formation of equiaxed grains with a size similar to the subgrain size ^{54–57} (**Fig. 1.15d**). In a few literatures ⁵⁸, GDRX was also mentioned as a possible event in ferrite during hot deformation.

1.5. High-strain warm deformation

1.5.1. Challenges

Extensive researches on SPD processes have been mostly carried out at room temperature. However, the formation of fine or ultra-fine grains at elevated temperatures (warm deformation) has not been well understood yet. As most of SPD methods (e.g. ECAP ¹⁷, ARB ^{18,19}, etc.) are batch processes or difficult to be carried out isothermally at elevated temperatures (HPT¹⁵, ECAP, ARB), it is quite difficult to accurately control the deformation parameters such as temperature and strain rate in SPD. Despite this fact, several attempts have been made, using various interrupted [e.g. ^{59–63}] or even continuous [e.g. ^{64–67}] methods, to study the highly-deformed microstructures at elevated temperatures. However, there are still disagreements and uncertainties in the results, often due to the complex microstructure evolution under such conditions. As a result, this topic still needs to be furthermore investigated.

Figure 1.16 shows microstructure examples of an ARB-processed IF steel ⁵⁹ deformed by several cycles at 500 °C (**Figs. 1.16a-b**), compared with those processed at room temperature (**Figs. 1.16c-d**). In the 500°C-ARB, stacked sheets were firstly held in an air furnace set at 500 °C for 10 minutes and then immediately deformed at an average (nominal) strain rate of 19 s⁻¹. The rolled specimen was water-quenched

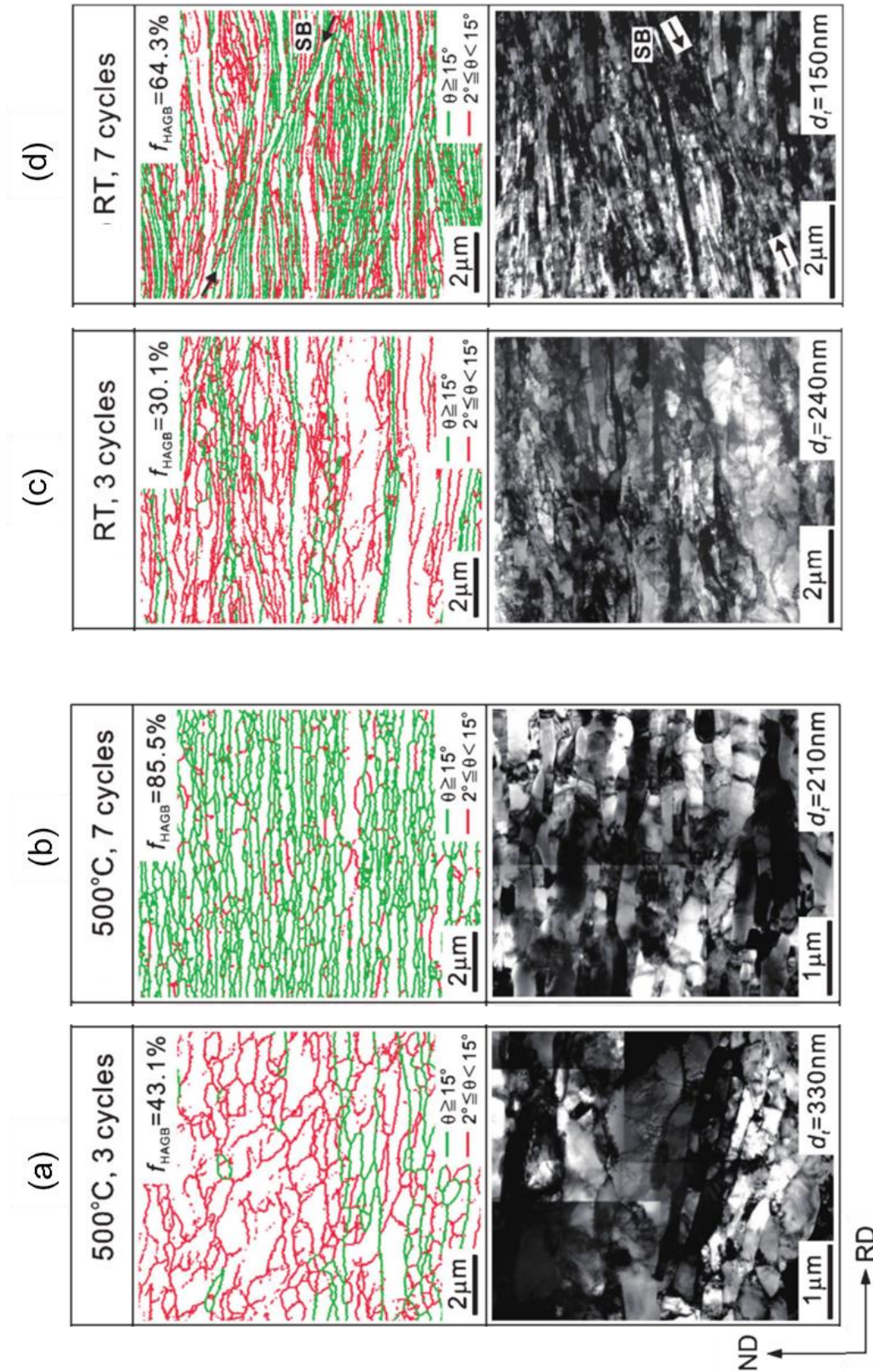


Figure 1.16. EBSD grain boundary maps and TEM images of an IF steel ARB-processes by several cycles at (a, b) 500°C and at (c, d) room temperature (SB: shear band) ⁵⁹.

quickly after the deformation and prepared for subsequent furnace heating and rolling processes.

In **Fig. 1.16**, it can be seen that the boundary spacing became larger with

increasing the deformation temperature. However, no extensive grain growth of particular grains was observed under the warm condition (500 °C). It was then suggested ⁵⁹ that the evolution of UFGs can be attributed to a balance of grain subdivision and dynamic recovery plus short-range boundary migration. However, there was a risk of static restoration as well as difficulty in temperature control in such an interrupted process.

Continuous torsion deformation of aluminum at constant strain rates and temperatures was recently studied by S. Khamsuk et al. ⁶⁸ in our group. For instance, **Fig. 1.17** shows obtained microstructures in the aluminum (1100-Al) specimens, highly torsion-deformed to an equivalent strain of 5.4 continuously, under various Z conditions (i.e. at various temperatures and strain rates). As shown by the EBSD boundary maps, ultra-fine lamellar structures formed under high Z conditions (e.g. **Fig.**

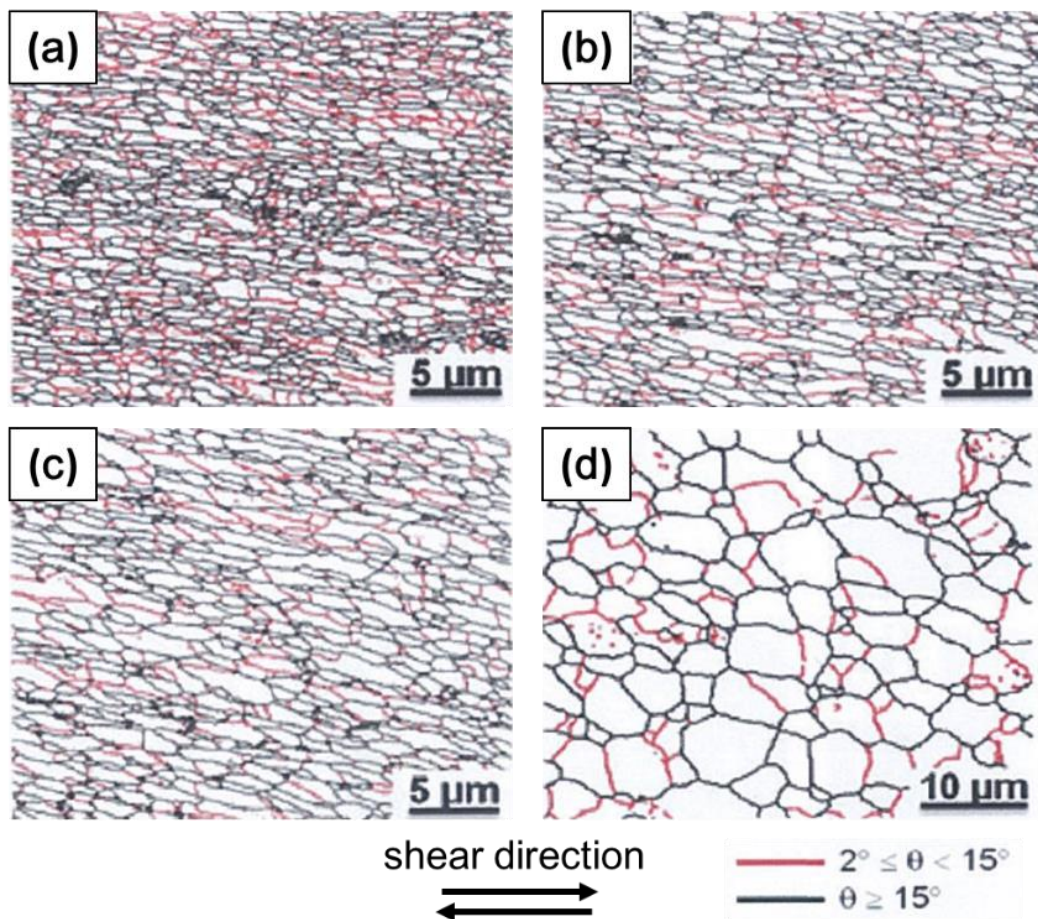


Figure 1.17. EBSD boundary maps of an aluminum (1100-Al) torsion-deformed to an equivalent strain of 5.4 continuously: (a) $Z = 2.2 \times 10^{26} \text{ s}^{-1}$ (25 °C, 0.1 s^{-1}), (b) $Z = 7.0 \times 10^{19} \text{ s}^{-1}$ (100 °C, 0.01 s^{-1}), (c) $Z = 1.7 \times 10^{16} \text{ s}^{-1}$ (200 °C, 0.1 s^{-1}), and (d) $Z = 1.7 \times 10^{12} \text{ s}^{-1}$ (300 °C, 0.01 s^{-1}) ⁶⁸.

1.17a) were nearly parallel to the shear direction. However, the size and morphology of microstructures changed into coarse equiaxed grains under low Z conditions (e.g. **Fig. 1.17d**).

It was found ⁶⁸ that dislocation cross-slip at lower temperatures (higher Z) and grain boundary migration accompanied with dislocation climb at higher temperatures (lower Z) were the important mechanisms in the torsion deformed aluminum. However, detailed study of microstructure evolution was not the main concern of that research. Additionally, the nature of aluminum alloys having relatively low melting temperature made it difficult to get clear results on the UFG evolution in a wide range of deformation temperatures.

Figure 1.18 demonstrates a microstructure evolution in a multiple warm-forging at 500°C (**Figs. 1.18a-c**) and 600°C (**Figs. 1.18d-e**) of a 304 austenitic (fcc) stainless steels studied by A. Belyakov et al. ^{63,69,70}. At high strains ($\varepsilon : 4 \sim 6$), the flow stress approached a saturation level. Accordingly, a great degree of grain refinement could be achieved leading to ultra-fine grained microstructures with grain sizes below 1 μ m.

Additionally, **Fig. 1.19** shows warm rolled microstructures in a 304 austenitic stainless steel deformed by moderate strains ($\varepsilon \sim 2$) ^{71,72}. As shown by the TEM images (**Figs. 1.19b-c**), micro-bands were frequently found in the microstructure at this moderate strain. In both cases (**Fig. 1.18** and **Fig. 1.19**), the development of the new ultra-fine grains was mainly attributed to the continuous development of the deformation-induced boundaries, which was possibly similar to grain subdivision. Such a microstructure evolution could continuously lead to DRX-like grains at high strains. As a result, sometimes the terminology so-called continuous dynamic recrystallization or CDRX is used for the evolution of UFGs under such a high-strain warm deformation, in contrast to the conventional DRX (or discontinuous DRX), where nucleation and growth of recrystallized grains are observed as the formation mechanism.

Moreover, it was shown ^{63,69} that the size of the grains in severe warm deformation increased with increasing the deformation temperature (decreasing Z). It has been sometimes claimed that conventional (discontinuous) DRX was activated. Similar results and viewpoints were also reported for the other types of fcc-metals such as copper ^{62,73} and nickel ^{74,75}. This will be more discussed in the next section by considering the stress-grain size relationship.

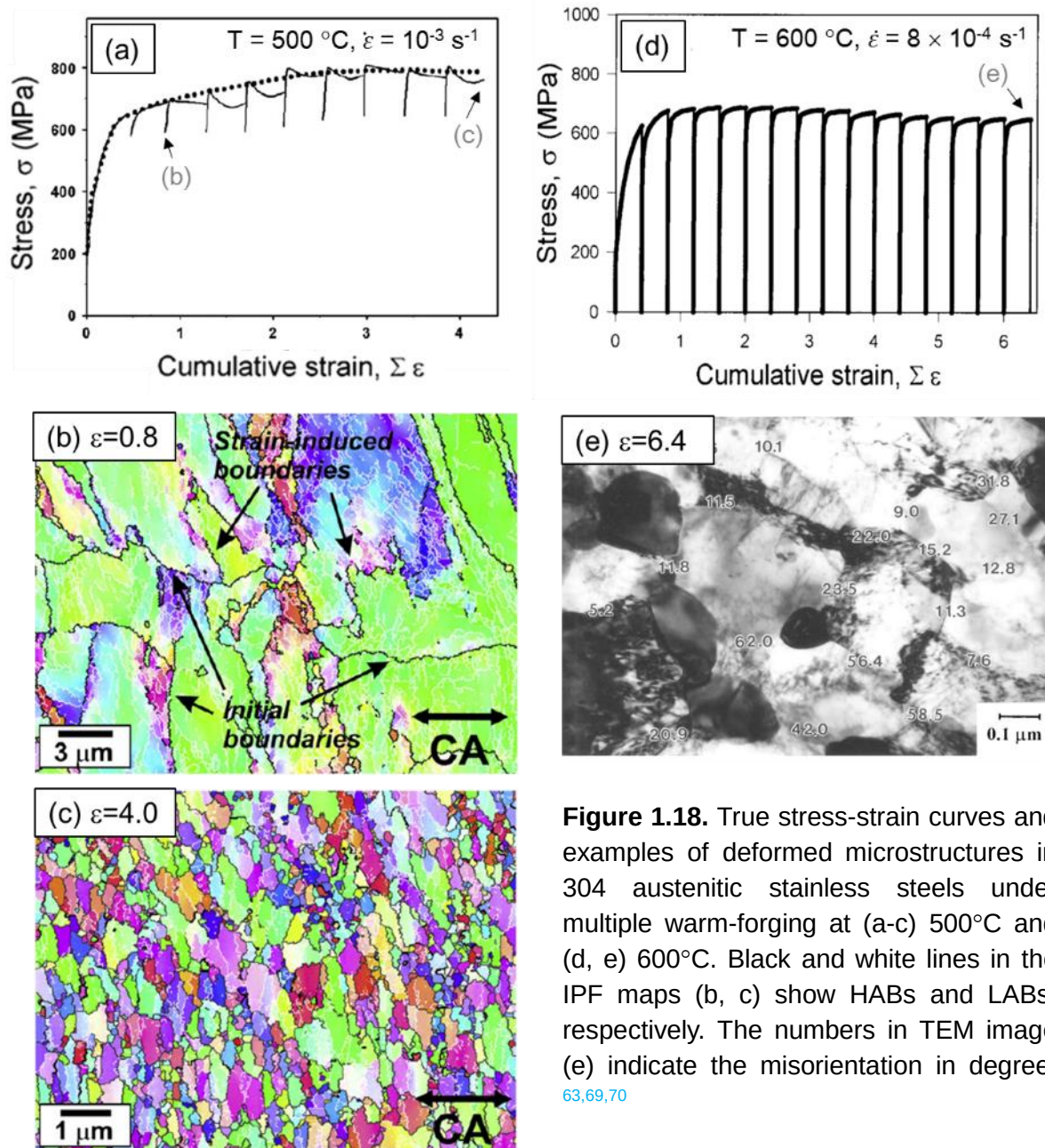


Figure 1.18. True stress-strain curves and examples of deformed microstructures in 304 austenitic stainless steels under multiple warm-forging at (a-c) 500°C and (d, e) 600°C. Black and white lines in the IPF maps (b, c) show HABs and LABs, respectively. The numbers in TEM image (e) indicate the misorientation in degree. [63,69,70](#)

1.5.2. Continuous and discontinuous DRX

As explained previously in **Section 1.4.2.**, the stress - grain size relationship has been widely used for understanding the hot deformation of materials. Recently, similar plot has been even more expanded from the range of hot deformation to the range of warm or even low-temperature deformation for metallic materials. **Figure 1.20** is a typical example of this type of plot for different fcc-metals such as austenitic steel [61](#), copper [62,73](#) and nickel [74,75](#) alloys. The aim was to explain the change in the formation mechanisms of new grains.

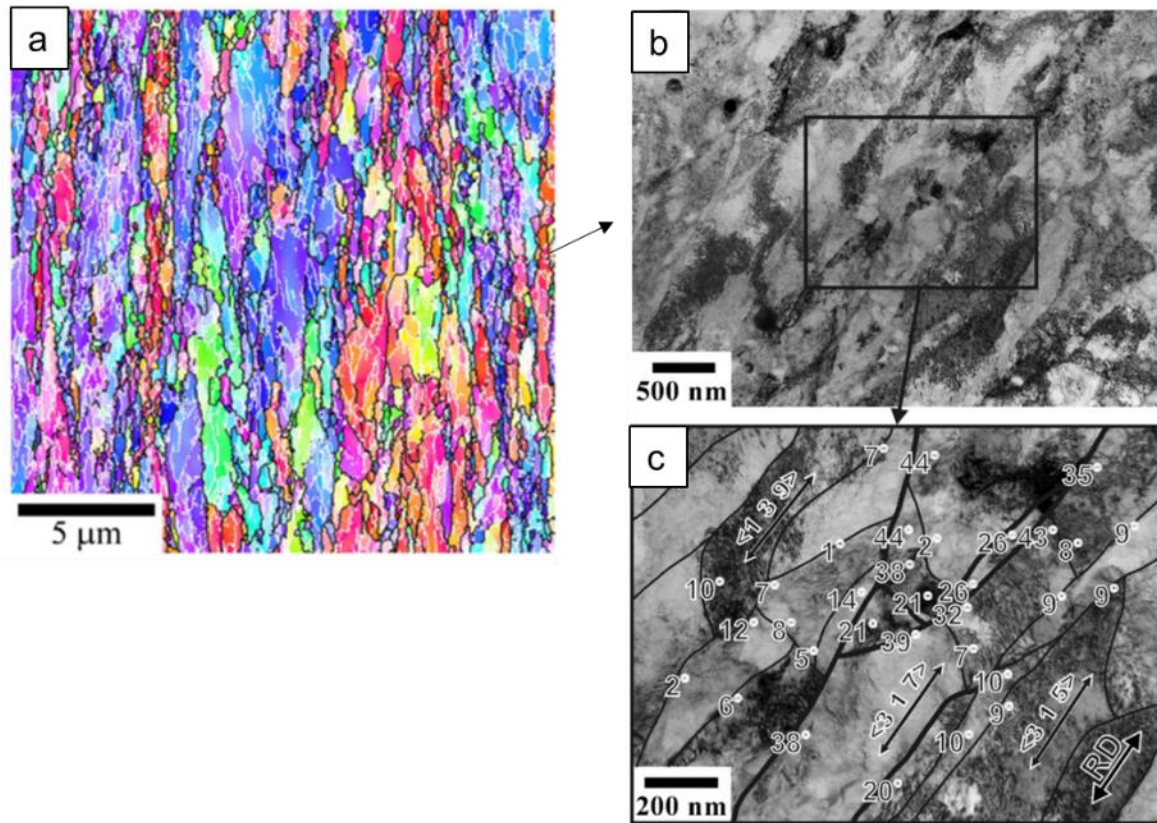


Figure 1.19. Deformed microstructures in a S304H-type stainless steel after multiple rolling to a strain of 2 at 600°C. Black and white lines show HABs and LABs, respectively. The numbers in TEM image indicate the misorientation in degree (RD: rolling direction) ^{71,72}.

As can be seen, in the hot deformation region (or lower stress level), slopes of the curves for different fcc-metals are almost identical, ≈ 0.75 . However, there is a change in the slope from ≈ 0.75 to ≈ 0.3 in these plots with a shift from hot to warm deformation regions. Apparently, the stress became less sensitive to the size of the grains in the warm deformation. This change in the gradient was claimed to be due to the change in the mechanisms of microstructure evolutions, e.g. from discontinuous (conventional) DRX to continuous DRX where grain sizes became closer to the subgrain sizes.

It should be noted that, unlike the fcc-metals that are mostly not recovery-type, there has been no attempt to apply the similar analysis shown in **Fig. 1.20** for the hot and warm deformation of other types of metals such as bcc-iron. One reason could be due to the fact that ferrite is typically considered as an easy-recovery metal without typical DRX. Under any scenario, it is of interest to use the same method to

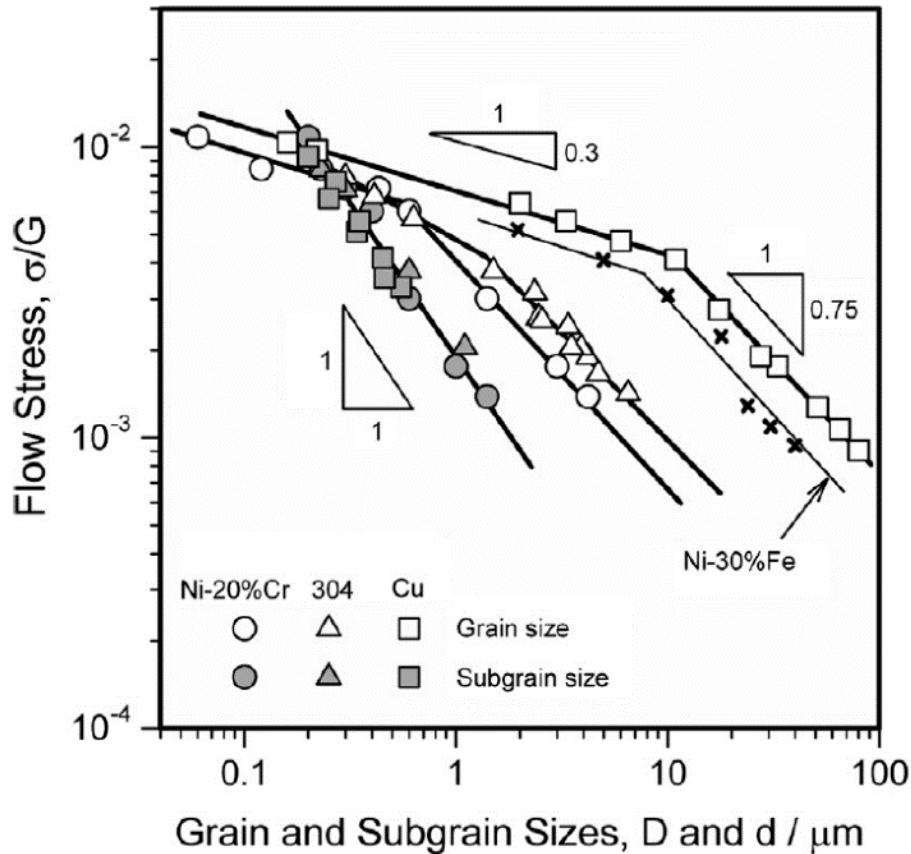


Figure 1.20. The relationship between the normalized flow stress and DRX grain size for typical DRX metals; 304 austenitic steel ⁶¹, pure copper ^{62,73} and nickel ^{74,75} alloy.

summarize the data of grain size in bcc-iron and compare the data with those shown in **Fig. 1.20**.

Critically speaking, the term continuous DRX (used in this section) is sometimes ambiguous since the nature of nucleation stage in DRX or even conventional recrystallization which is probably governed by recovery has not yet been well understood due to technical difficulties. In one point of view, the phenomenon called continuous DRX in warm deformation might be explained by considering well-known mechanisms such as grain subdivision and restoration mechanisms instead. For example, the TEM microstructure shown in **Fig. 1.19c** formed during warm deformation is similar to the deformation microstructure at low temperature (e.g. **Fig. 1.5a**). As a result, it can be expected that the grain subdivision play a dominant role under warm deformation. Simultaneously, restoration processes such as recovery and boundary

migration (either as uniform grain growth or extensive growth of particular grains) can be partly responsible for the microstructure evolution. In other words, the microstructure evolution in warm deformation might be dominated by a combination of grain subdivision and restoration phenomena, which will be investigated based on experimental results in this thesis.

1.6. Motivation/aim/method

Based on the background introduced in the previous sections, the phenomena such as grain subdivision and DRX have been well-known as the mechanisms of fine grain formations during deformation at warm or hot temperatures for several decades. However, these two refining mechanisms have been separately confirmed under different deformation conditions. It is necessary to know how the transition between different conditions occurs. Such a viewpoint is expressed in **Fig. 1.21**, where the microstructure evolution is arranged simply as a function of strain (ε) and Zener-Hollomon parameter (Z). Here, the various conditions under which plastic deformation can lead to different types of known or unknown microstructures are demonstrated. In the high Z region (e.g. SPD at low temperature), the formation of new grain structure can be understood by the grain subdivision phenomenon. By increasing the strain, the fraction of HABs (f_{HABs}) increases. Eventually a UFG lamellar structure dominated by HABs forms at high strains. On the other hand, DRX is another refining process that is dominant in the low Z region (e.g. hot deformation: $T > 0.5 T_m$). The DRX leads to a fine equiaxed structure which needs much lower amount of strain to form, compared to that in the grain subdivision. As shown previously in **Fig. 1.9**, DRX initiates at a critical strain (ε_{cr}) and completes at a strain of the initiation of steady-state (ε_s) on the flow stress-strain curve. In **Fig. 1.21**, two characteristic lines associated to the DRX initiation (ε_{cr}) and DRX completion (ε_s) are drawn in the low Z region. The slopes of the lines indicate that both ε_{cr} and ε_s increase with increasing Z .

Concerning the $\varepsilon - Z$ plot in **Fig. 1.21**, there is still a lack of universal understanding for the formation of new grains during the plastic deformation over a wider range of Z conditions. It is of interest to understand how the microstructures

formed under low Z conditions change into the microstructures formed under high Z conditions. In particular, there are still many uncertainties about the microstructure evolution in high-strain warm deformation (or transition Z) at constant temperature and strain rate which needs to be furthermore investigated.

In brief, the present study aims to investigate the formation of ***fine or ultra-fine grains*** fabricated by ***high-strain deformation*** at different temperatures and strain rates, i.e. under ***various Z conditions***. Moreover, the study is carried out for different types of metals. For example, the Z -dependence behaviors and microstructure evolution in the ***easy-recovery metals*** and in the ***conventional DRX metals*** and their similarities and differences would be clarified.

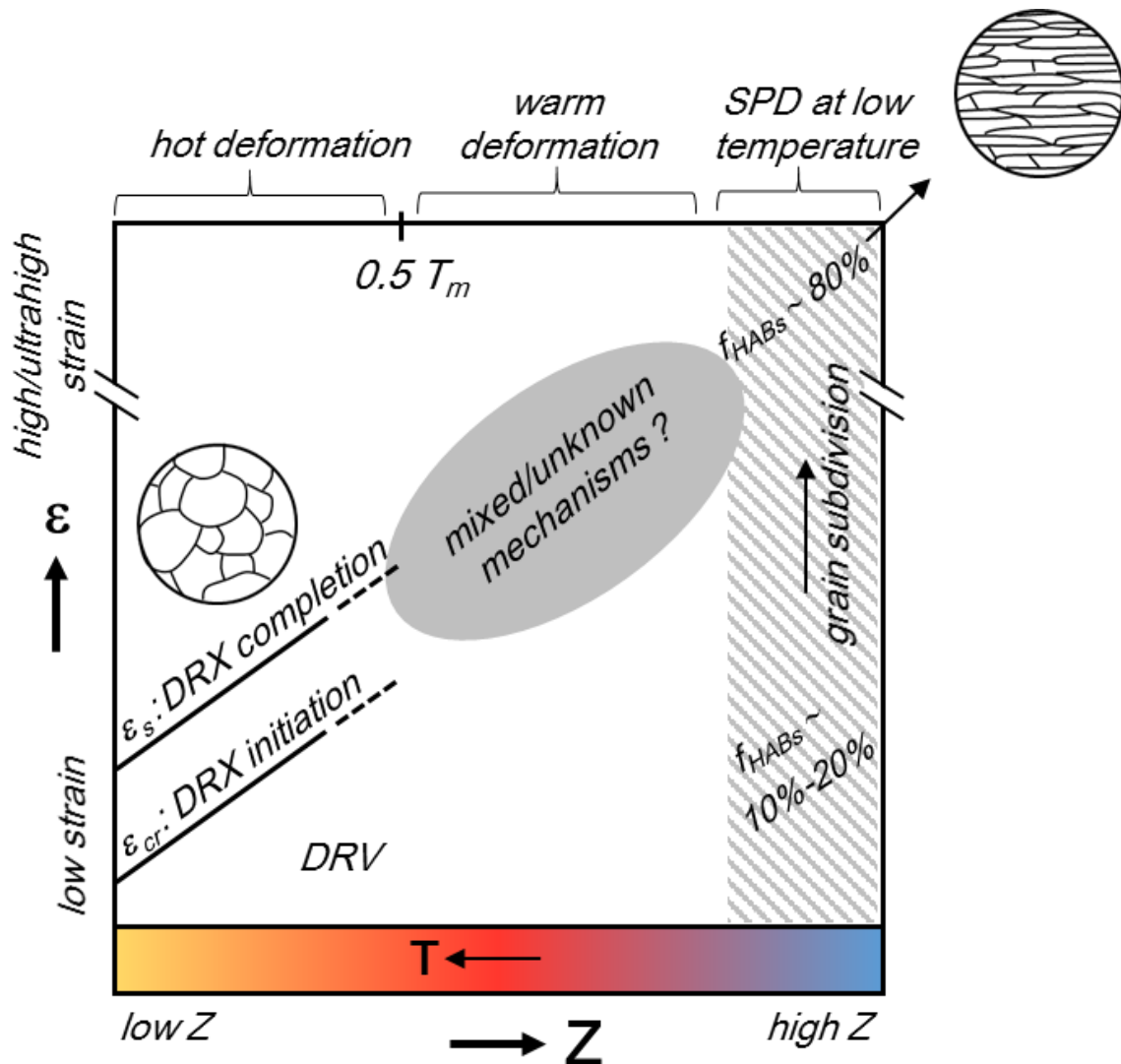


Figure 1.21. A ϵ (strain) - Z (Zener-Hollomon) plot, illustrating various conditions at which plastic deformation can lead to different types of microstructures.

Accordingly, a set of systematic processes was designed in the current research, in order to deform different metals, to sufficiently high strains at various constant temperatures and strain rates. Such a systematic study requires the continuous SPD process which makes the accurate and continuous control of temperature and strain rates possible. Torsion deformation is a popular method to simulate hot deformation of metals with the advantage of accurate control of deformation conditions. Even though the torsion deformation is not categorized as a typical SPD process, it is possible to deform the specimen up to desired high strains continuously by torsion, preventing the risk of static recovery and recrystallization which normally happen in other types of interrupted (or batch) plastic deformation tests, such as ECAP and ARB at high temperatures.

As candidate materials, firstly an ultra-low carbon interstitial free (IF) steel is examined. The IF steel has a ferrite single-phase stable in a wide temperature range (below $\sim 900\text{ }^{\circ}\text{C}$) which is suitable for this study. As mentioned before, bcc-ferrite in the IF steel has an easy-recovery character. For comparison, the same deformation approach is taken for a typical DRX metal, i.e, SUS 304 austenitic stainless steel, which has a fcc-phase for wide temperature ranges.

1.7. Outlines of the dissertation

The contents of this dissertation are divided in five chapters. The first chapter “**background and purpose**”, has been already described here. The grain refinement through deformation under various conditions reported in previous literatures has been explained. It has been pointed out that it is necessary to get an universal understanding of the formation of new grain structures during plastic deformation to high strains. The second and third chapters are dedicated to the results found in the torsion deformation of the easy-recovery IF steel. More specifically, the second chapter, “**Formation of fine or ultrafine grains in bcc-ferrite during high-strain torsion deformation at various temperatures and strain rates**”, mainly focuses on the effect of Z parameter (T , $\dot{\epsilon}$) on the microstructure evolution. Microstructural parameters together with the flow stress behavior in the IF steel deformed by torsion under various low, medium, and high Z conditions are discussed. On the other hand, in the third chapter, “**Effect of strain on microstructure evolution in torsion-**

deformed bcc-ferrite", the development of various grain structures is examined and discussed at different (low-to-high-via medium) strains. The contents of the forth chapter, "**Formation of new grain structures in SUS 304 stainless steel during high-strain torsion deformation at various temperature and strain rate**", are concentrated on the microstructure evolution during the high-strain torsion deformation in the 304 austenitic stainless steel. The aim is to describe the microstructure evolution in the fcc-metal and discuss the similarities and differences with the bcc-IF steel with easy-recovery nature. Finally, a summary of the important results and findings in this research is presented in the fifth chapter, "**Summary and conclusions**".

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Chapter 2: Formation of fine or ultrafine grains in bcc-ferrite during high-strain torsion deformation at various temperatures and strain rates

2.1. Introduction

Based on the aim of the current study introduced in the Chapter 1, an interstitial free (IF) steel was firstly chosen for investigations, as a type of easy-recovery metal (in the ferrite region). In the Chapter 2, the IF steel was deformed continuously to high strains (equivalent strain, ε : 5 ~ 7) in series of systematically designed torsion tests. The purpose of this chapter is to investigate the formation of fine or ultrafine grained ferrite at various temperatures and strain rates; i.e., under various Z (Zener-Hollomon) conditions. Accordingly, the material's behaviors are examined in a wide Z range and expressed in terms of flow stress-strain behaviors, microstructural parameters (such as grain size, morphology, fraction of HABs), and textures. The results are compared with the data available in other literatures on ferrite processing in steels. Finally, the Z -dependency of the UFG ferrite formation is discussed and categorized into different types.

The IF steel is considered to be free of interstitial atoms (C, N, etc.), since it has an ultra-low carbon (ULC) and nitrogen contents generally below several-tens mass ppm and additionally carbon and nitrogen atoms in the IF steel are stabilized as carbides and nitrides by adding strong carbide/nitride forming elements such as titanium and niobium. The single-phase ferrite in the IF steel is available in a wide temperature range below ~ 900 °C, the A_3 temperature.

2.2. Experimental procedures

2.2.1. Material

A titanium-added IF steel with an ultra-low carbon content (0.002 wt%) was used

in the present study. **Table 2.1** shows the chemical composition of the as-received IF steel. The material was received in a form of square bars ($15\text{ mm} \times 15\text{ mm}$) with 300 mm-length cut from a hot-rolled and air-cooled plate (**Fig. 2.1a**). The torsion specimens with a 4 mm gauge length and 8 mm gauge diameter (**Figs. 2.1b, 2.1c**) were made from the as-received material, in a way that the specimen axis was parallel to the rolling direction (RD) of the original hot-rolled plate. The initial microstructure of the IF steel is shown in **Fig. 2.2**, which was a fully recrystallized microstructure with an average grain size around $60\text{ }\mu\text{m}$.

Table 2.1. Chemical composition (wt%) of the as-received IF steel (balance: Fe)

C	Si	Mn	P	S	Ti	Al	N
0.002	0.01	0.12	0.003	0.003	0.034	0.028	0.002

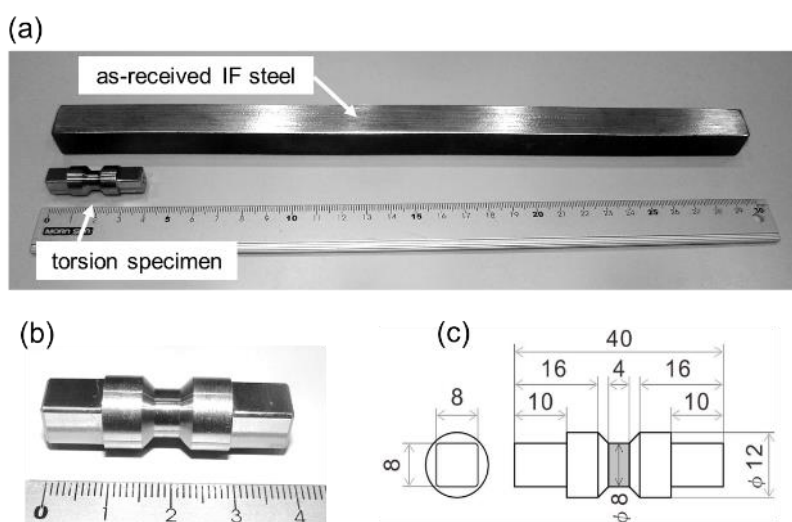


Figure 2.1. (a) As-received IF steel bar, and (b, c) shape and dimension of the torsion specimen (mm) which was cut from the as-received bar.

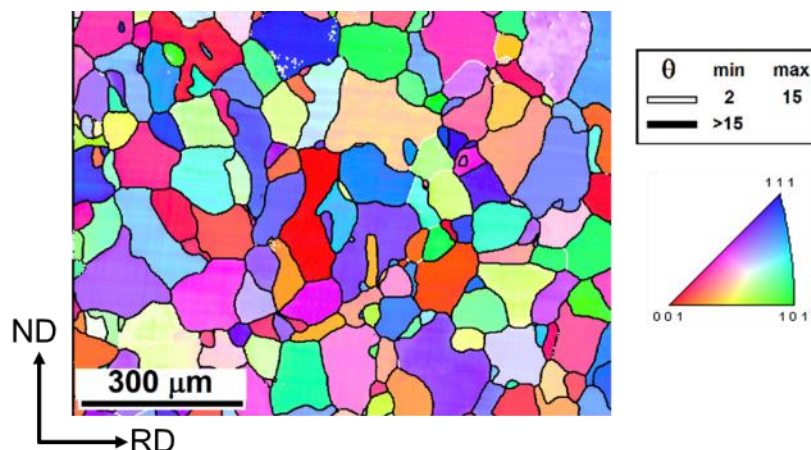


Figure 2.2. EBSD orientation map of the Initial microstructure of the IF steel.

2.2.2. Torsion test

For the torsion deformation, a thermo-mechanical processing simulator (Thermecmasto-TS) equipped with a high-frequency induction heating system was used. It was the free-end type torsion machine whose outside and inside views are shown in **Fig. 2.3**.

Torsion tests were carried out at a wide range of deformation temperatures in a ferrite single-phase region; i.e., below the A_3 (bcc-to-fcc phase transformation) temperature ($< 900\text{ }^{\circ}\text{C}$). The temperature range includes the deformation at $850\text{ }^{\circ}\text{C}$ ($0.62 T_m$), $750\text{ }^{\circ}\text{C}$ ($0.56 T_m$), $650\text{ }^{\circ}\text{C}$ ($0.51 T_m$), $550\text{ }^{\circ}\text{C}$ ($0.45 T_m$), and $400\text{ }^{\circ}\text{C}$ ($0.37 T_m$), where T_m is the melting temperature of pure iron in Kelvin scale. Under an argon (Ar) atmosphere, the torsion specimens were deformed at different strain rates: 0.01 s^{-1} , 0.1 s^{-1} , and 1 s^{-1} . Higher deformation rates ($> 1\text{ s}^{-1}$) were not used in this study in order to reduce the risk of post-dynamic (static) recrystallization during cooling after the deformation as much as possible.

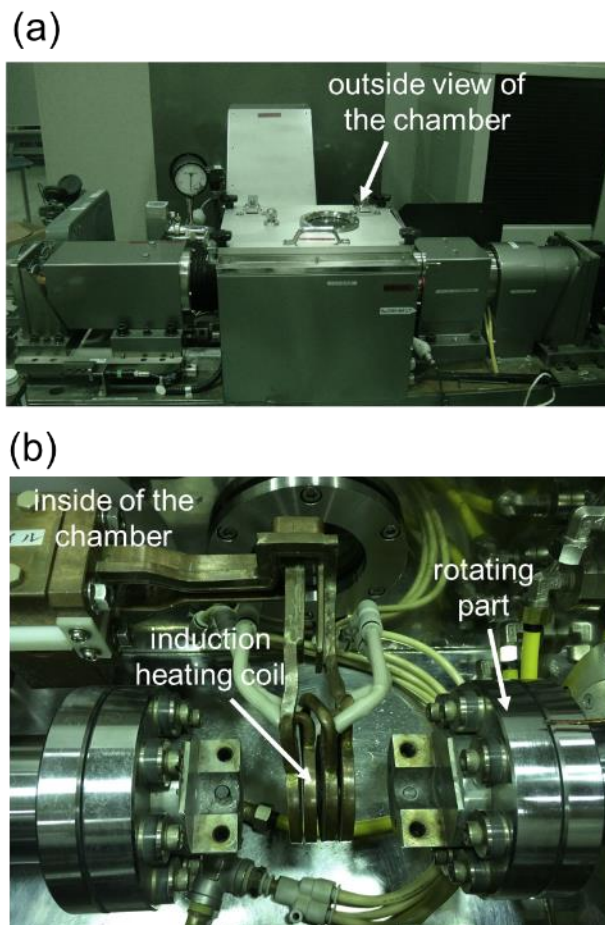


Figure 2.3. (a) Outside and (b) inside views of the torsion machine used in this study.

Figure. 2.4 illustrates the applied thermo-mechanical processing to deform the torsion specimens. After heating by induction heating at a rate of $2\text{ }^{\circ}\text{C s}^{-1}$, the specimens were held at the test temperature for 5 minutes for homogenizing the temperature of the specimens, and then they were deformed to high strains (5 ~ 7) by torsion at various strain rates. Then, a water-jet system was used to quench the specimens immediately after the torsion deformation. The quenching period was estimated to be within 1 second, which could avoid any post-deformation phenomena statically taking place as much as possible.

At higher temperatures ($\geq 650\text{ }^{\circ}\text{C}$), the specimens could be torsion-deformed up to two rotations (equivalent strain ≈ 7.2) or more, without any sign of cracks or major dimensional changes. At lower temperatures (i.e. $400^{\circ}\text{C} - 550^{\circ}\text{C}$), however, the torsion test was carried out up to 1.5 rotation (equivalent strain ≈ 5.4) in the maximum, mainly because of the initiation of surface cracking after this stage.

Torque-rotation data obtained during the torsion test were converted into shear stress – shear strain data by employing Fields and Backofen analysis ¹. **Equation**

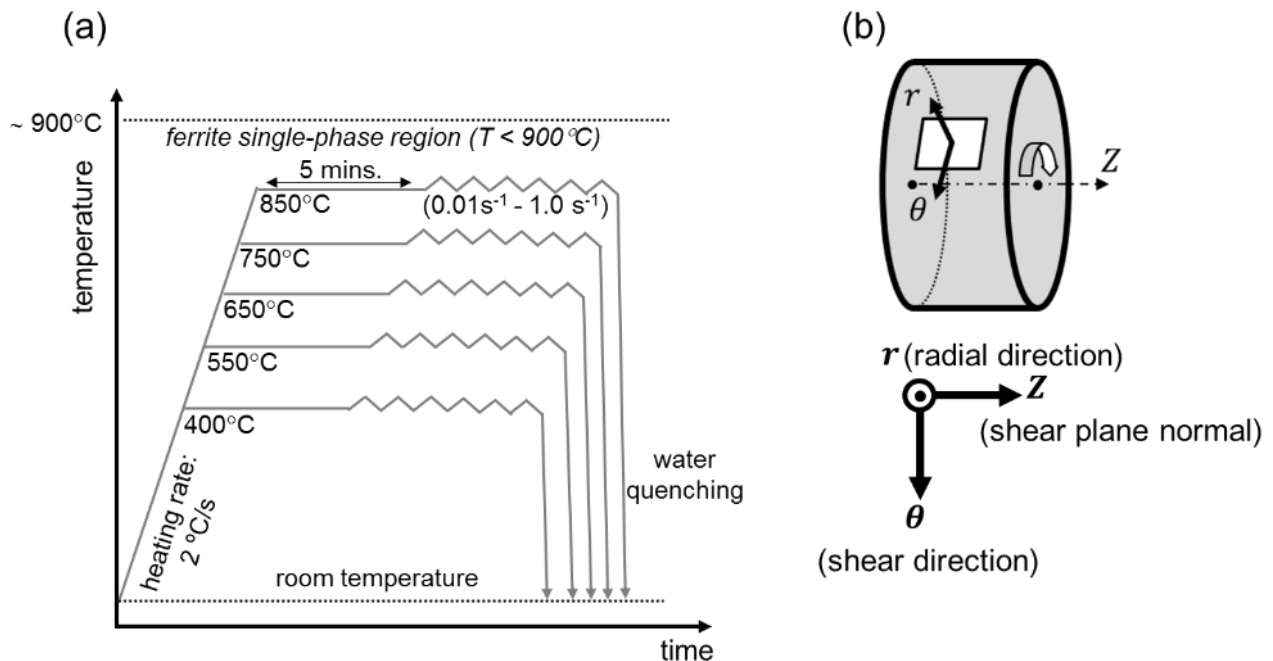


Figure 2.4. (a) Thermo-mechanical processing schedules in the ferrite single-phase region together with (b) configuration of the torsion specimen; a tangential section (indicated by the white rectangle) on the gauge part of the specimen was used for the microstructure observation; r is the radial direction perpendicular to the observed section, Z and θ are the shear plane normal direction and the shear direction, respectively.

2.1 and **Eq. 2.2** show the relationships of shear strain (γ) - rotation number (N) and shear stress (τ) - torque (Γ), respectively;

$$\gamma = \frac{2\pi R}{L} N \quad (2.1)$$

$$\tau = \frac{\Gamma}{2\pi R^3} (3+n+m) \quad (2.2)$$

where R is the radius of the torsion specimen, L is the gauge length, n is the strain hardening coefficient (~ 0 ; at the peak and steady-state condition) and m is the strain rate sensitivity (e.g., 0.1 at 650 °C and 0.2 at 850 °C in this study). Subsequently, von Mises criteria was used to attain the equivalent stress (σ) and equivalent strain (ε) according to **Eq. 2.3** and **Eq. 2.4**;

$$\varepsilon = \frac{\gamma}{\sqrt{3}} \quad (2.3)$$

$$\sigma = \sqrt{3}\tau \quad (2.4)$$

It should be noted that for the equivalent strain (ε) and accordingly the equivalent strain rate ($\dot{\varepsilon}$) mentioned in this study, the specimen surface (i.e., the maximum radial position) was taken as the reference position for the calculation.

2.2.3. Microstructure observation

Similar to the most common way for microstructure observations in torsion specimens, a tangential section close to the surface (at $0.9 \times$ radius) was chosen for the observation (**Fig. 2.4b**). Electron Back-scattering Diffraction (EBSD) measurements were mainly employed to study the microstructure evolution in the current study. In addition, transmission electron microscopy (TEM) was used, especially to measure the subgrain size.

For the EBSD scanning, the surface (normal to the r direction) was prepared by mechanical polishing and subsequent final polishing by a 0.04 and 0.02 μm colloidal silica suspension. Then, EBSD measurements were carried out in a field emission scanning electron microscope (FE-SEM; JEOL JSM 7100F) operated at 15 kV with 15 mm working distance. For each deformation condition, a sufficient size of area was scanned at an appropriate step size. Finally, EBSD data were analyzed using Orientation Imaging Microscopy (TSL OIM, version 7.2.1) software in order to

characterize the microstructural parameters such as grain size, boundary fraction, etc. Thin-foil specimens for TEM were made by twin-jet electro-polishing, using a 10% HClO_4 - 90% CH_3COOH solution. TEM observation was carried out in a TEM microscope (JEOL JEM 2010) operated at 200 kV.

2.3. Results

2.3.1. Flow stress behavior

Figure 2.5 shows flow stress-strain curves of the IF steel torsion-deformed within the ferrite single-phase region, at a wide range of deformation temperatures from 850°C to 400°C and different strain rates; i.e., 0.01 s⁻¹ (**Fig. 2.5a**), 0.1 s⁻¹ (**Fig. 2.5b**), and 1.0 s⁻¹ (**Fig. 2.5c**). At higher temperatures, the specimen could be deformed up to very high strains, but the torsion test was stopped at the equivalent strain of 7.2 simply because the steady state was achieved already. At lower temperatures (i.e., 400°C - 550°C), however, the torsion test could be carried out only up to the equivalent strain of 5.4, mainly because of the initiation of surface cracking after this stage.

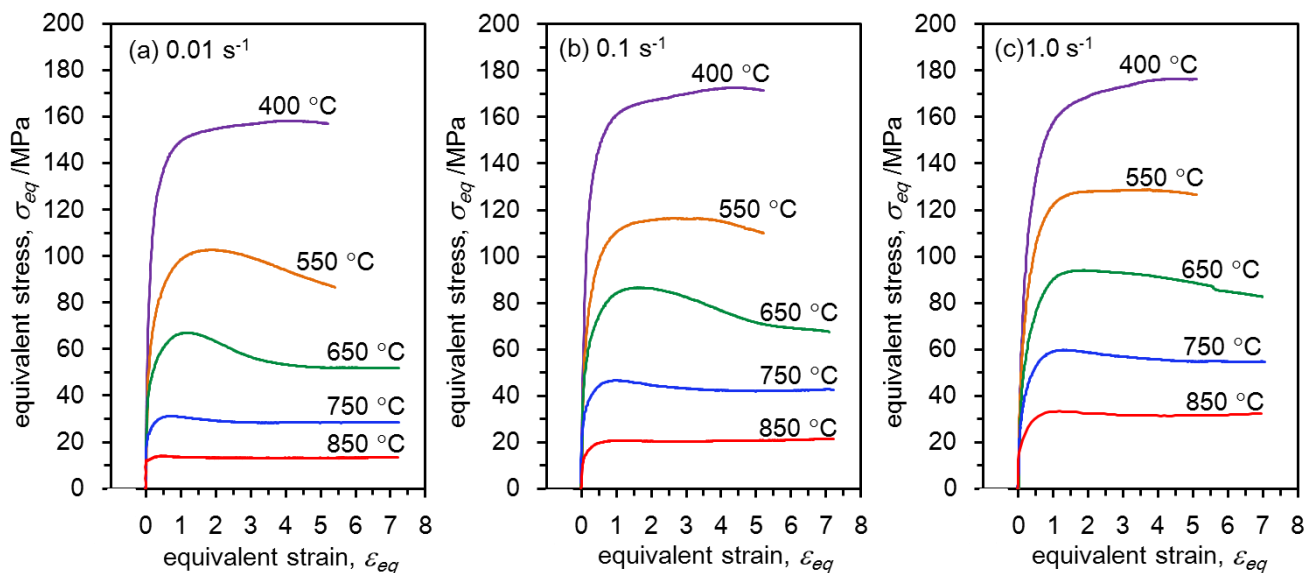


Figure 2.5. Torsion flow stress-strain curves (von Mises equivalent stress vs. equivalent strain) of the IF steel highly deformed (ϵ : 5 ~ 7) at different temperatures (400 °C - 850 °C) and strain rates (0.01 s⁻¹ - 1.0 s⁻¹), all in the ferrite single-phase region.

It can be seen in **Fig. 2.5** that the stress levels increased with either decreasing the deformation temperature or increasing the strain rate. At the deformation temperatures of 850°C and 750°C (relatively high temperatures), the flow stress showed a work hardening part at the beginning, and soon reached the maximum stress, subsequently followed by a slight stress drop and eventually a steady state. In this case, the difference between the maximum (peak) stress and the steady-state stress was almost negligible. As reported in other works ²⁻⁷, this flat-type flow stress without a distinguishable peak and stress-drop, can be considered as a typical behavior of the easy-recovery metals.

On the other hand, decreasing the deformation temperature to 650 °C resulted in a broader peak, which required larger strains to achieve the steady-state. However, the type of flow stress-strain curves eventually changed into a work hardening type (e.g., at 400 °C) which merely reached a plateau even at large strains, in other words, no distinct peak found on the flow stress-strain curves. As mentioned before, the possibility of deformation (adiabatic) heating and its effect on the flow stress were tried to be removed by keeping the deformation rate below 1 s⁻¹ as well as using an advanced induction heating system to adjust the temperature of the specimen with the set temperature. As a result, no temperature rise was detected by the thermo-couple welded on the middle of the side surface of the specimens during the torsion test except for the case of 400°C/1.0 s⁻¹ (with a temperature increase of ~ 25 °C, which was taken into account for the estimation of the Z parameter).

In further analysis shown in later parts, the maximum stress (σ_m) and the stress at the final strain (σ^*) or at a constant strain (e.g., σ_2 at $\varepsilon = 2$) determined on the stress-strain curves were used.

- *Flow stress analysis*

In order to establish a relationship between the Z parameter and the stress (σ), some empirical equations have been proposed previously ⁸. That is, a hyperbolic equation (**Eq. 2.5**) applied for a wide stress range, a power-law equation (**Eq. 2.6**) applied for a low stress level, and an exponential form (**Eq. 2.7**) applied for a high stress level have been proposed;

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) = A [\sinh(\alpha\sigma)]^n \quad (2.5)$$

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) = \dot{A} \sigma^{\dot{n}} \quad (2.6)$$

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) = \dot{A} \exp(\beta \sigma) \quad (2.7)$$

where A , $\dot{A}$, $\dot{A}$, n , $\dot{n}$, α , and β are constants. It should be noted that the relationship between α (**Eq. 2.5**) known as a stress multiplier with $\dot{n}$ (**Eq. 2.6**) and β (**Eq. 2.7**) can be expressed as $\alpha = \beta / \dot{n}$.

The power-law function is the common equation for creep analysis which usually breaks down at high stress. On the other hand, the exponential equation is suitable for the hot working condition ($T > 0.5 T_m$) which also breaks down at high temperature below a certain strain rate. However, the hyperbolic function includes both types of equations previously mentioned. For the current study, the hyperbolic sine correlation (**Eq. 2.5**) was mainly applied for the analysis since it was assumed to satisfy a wide range of stress level. Here, the α in the hyperbolic equation is an adjustable constant that brings $\alpha\sigma$ into a correct range, so that the $\ln(\dot{\epsilon})$ vs. $\ln[\sinh(\alpha\sigma)]$ plot becomes linear and parallel (at constant T)⁹. This type of plot will be explained furthermore below.

By taking the natural logarithm on both sides, **Eq. 2.5** can be expressed as **Eq. 2.8**. As a result, the values of n (stress exponent) and S ($= Q / RT$) can be defined according to **Eq. 2.9** at a constant T and **Eq. 2.10** at a constant $\dot{\epsilon}$, respectively;

$$\ln(\dot{\epsilon}) + \frac{Q}{RT} = \ln(A) + n \ln[\sinh(\alpha\sigma)] \quad (2.8)$$

$$n = \left. \frac{\partial \ln(\dot{\epsilon})}{\partial \ln[\sinh(\alpha\sigma)]} \right|_T \quad (2.9)$$

$$S \text{ or } \left(\frac{Q}{Rn} \right) = \left. \frac{\partial \ln[\sinh(\alpha\sigma)]}{\partial (1/T)} \right|_{\dot{\epsilon}} \quad (2.10)$$

In case of employing other types of equations (**Eq. 2.6** and **Eq. 2.7**) instead of the hyperbolic type, the same approach can be taken. The details for all types of equations including the values of parameters such as $\dot{n}$, β and various types of Q are shown in **Table 2.2**.

On the other hand, knowing the values of $\dot{n}$ and β is necessary to calculate the stress multiplier α according to **Eq. 2.11**;

$$\alpha = \beta / \dot{\epsilon} = \left(\frac{\partial \ln(\dot{\epsilon})}{\partial \sigma} \right) \bigg|_T / \left(\frac{\partial \ln(\dot{\epsilon})}{\partial \ln(\sigma)} \right) \bigg|_T \quad (2.11)$$

Table 2.2. Constitutive equations and related parameters.

equation type	constant T	constant $\dot{\epsilon}$
$Z = \dot{\epsilon} \exp(Q/RT) = A [\sinh(\alpha\sigma)]^n$	$n = \frac{\partial \ln(\dot{\epsilon})}{\partial \ln[\sinh(\alpha\sigma)]} \bigg _T$	$S = \left(\frac{Q}{Rn} \right) = \frac{\partial \ln[\sinh(\alpha\sigma)]}{\partial (1/T)} \bigg _{\dot{\epsilon}}$
$Z = \dot{\epsilon} \exp(Q/RT) = \dot{A} \sigma^{\dot{n}}$	$\dot{n} = \frac{\partial \ln(\dot{\epsilon})}{\partial \ln(\sigma)} \bigg _T$	$S = \left(\frac{Q}{R\dot{n}} \right) = \frac{\partial \ln(\sigma)}{\partial (1/T)} \bigg _{\dot{\epsilon}}$
$Z = \dot{\epsilon} \exp(Q/RT) = \dot{A} \exp(\beta\sigma)$	$\beta = \frac{\partial \ln(\dot{\epsilon})}{\partial \sigma} \bigg _T$	$S = \left(\frac{Q}{R\beta} \right) = \frac{\partial \sigma}{\partial (1/T)} \bigg _{\dot{\epsilon}}$

Using the hyperbolic type equation, n could be attained at a constant T by measuring the slopes of the linear regressions in the $\ln(\dot{\epsilon})$ vs. $\ln[\sinh(\alpha\sigma)]$ plot. In the same way, S (or Q/Rn) at a constant $\dot{\epsilon}$ could be estimated using the $\ln[\sinh(\alpha\sigma)]$ vs. $(1/T)$ plot. For instance, such constant- T and constant- $\dot{\epsilon}$ plots are provided in **Fig. 2.6**, using the data associated to the maximum stress (σ_m) (**Figs. 2.6a** and **2.6b**) and the final stress (σ^*) (**Figs. 2.6c** and **2.6d**).

It can be seen from **Fig. 2.6** that the n values (shown above the plots in **Figs. 2.6a** and **2.6c**) changed within a range of 5 ~ 10 for the entire range of the deformation temperature (from 850 °C to 400 °C). The n value tends to increase with increasing the flow stress. In the same way, the S value changed within a range of 10 ~ 4 (**Figs. 2.6b** and **2.6d**). In this case, the point-to-point S values on the constant- $\dot{\epsilon}$ plot are shown for a detailed comparison, which shows a tendency to decrease with decreasing the deformation temperature. For both n and S values, it is interesting that the variation ranges are narrower in case of using the final stress than that using the maximum stress.

Using such an approach shown in **Fig. 2.6**, the average n , S , and therefore the average Q ($= R(n)(S)$) values could be estimated over any deformation conditions. This was firstly conducted over two theoretical regions, i.e., 850 °C - 650 °C ($> 0.5 T_m$) and 650 °C - 400 °C ($< 0.5 T_m$). However, this simple classification does not necessarily indicate the ranges of different mechanisms governing the deformation.

As a result, for the upper temperature range, the activation energies were estimated as 449 kJmol⁻¹ (σ_m), 416 kJmol⁻¹ (σ_2), and 360 kJmol⁻¹ (σ^*). Similarly, these values for the lower temperature range were estimated as 352 kJmol⁻¹ (σ_m), 362 kJmol⁻¹ (σ_2), and 359 kJmol⁻¹ (σ^*).

One possible reason for the larger Q in the upper temperature range might be the activation of boundary migration with increasing the temperature. However, a great care should be taken for the interpretation of the activation energy, since it should be noted that the Q value measured by this method is solely based on the flow stress analysis and often called an apparent activation energy for the deformation. In any

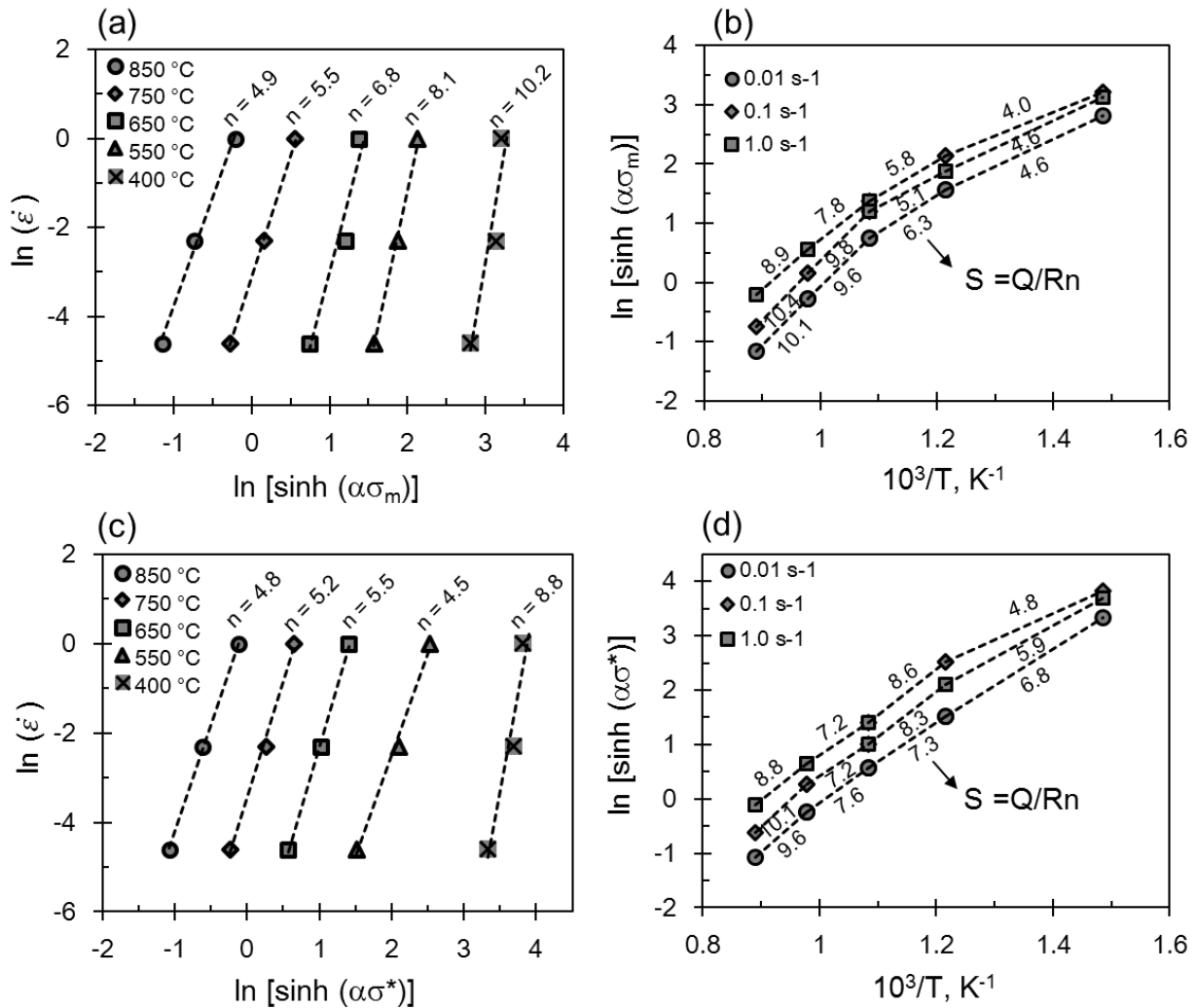


Figure 2.6. Applying the maximum stress: σ_m (a, b) and the final stress: σ^* (c, d) to plot $\ln(\dot{\epsilon})$ vs. $\ln[\sinh(\alpha\sigma)]$ diagrams at constant T (a, c), and $\ln[\sinh(\alpha\sigma)]$ vs. $(1/T)$ diagrams at constant $\dot{\epsilon}$ (b, d).

case, the average Q in this research was estimated as 383 kJmol^{-1} , which was quite close to the Q for a torsion-deformed IF steel reported by Oudin et al. (372 kJmol^{-1})⁴.

Furthermore, in order to calculate the stress multiplier: α (based on **Eq. 2.11**), the values of n and β can be obtained by measuring the slopes of linear regressions of the $\ln(\dot{\epsilon})$ vs. $\ln(\sigma)$ plot (**Fig. 2.7a**) and the $\ln(\dot{\epsilon})$ vs. σ plot (**Fig. 2.7b**), respectively. As a result, based on some assumptions, α was estimated as $0.022 \text{ MPa}^{-1} (\sigma_m)$, $0.024 \text{ MPa}^{-1} (\sigma_2)$, and $0.026 \text{ MPa}^{-1} (\sigma^*)$. The average α value (0.024 MPa^{-1}) in the current research was close to the value of α reported in torsion and compression deformations of ferritic steels by other researchers^{4,10}.

For comparison, the α , n and Q values reported by some other researchers are represented in **Table 2.3**. Comparatively, n took relatively large values in the current study since the warm conditions are also included in the analysis. On the other hand, it can be seen that the Q values associated to the IF steels and ferritic stainless steel ($350 \sim 400 \text{ kJmol}^{-1}$) are larger than $Q = 280 \text{ kJmol}^{-1}$ reported¹¹ for a pure iron ($\sim 99.95\%$). This indicates that the presence of impurity or other alloying elements probably resulted in such a higher activation energy¹².

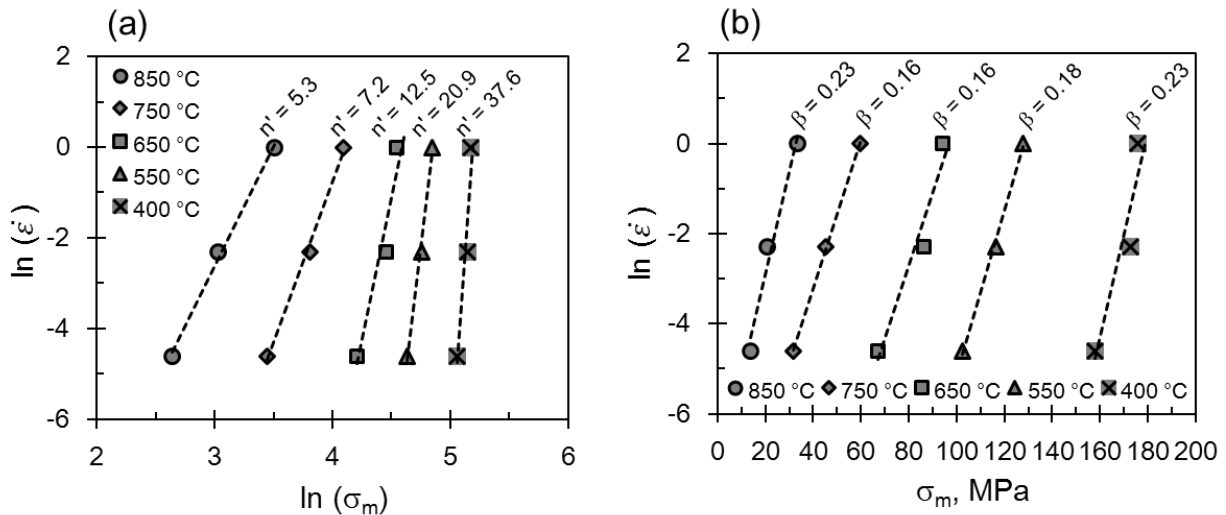


Figure 2.7. Applying σ_m to plot (a) $\ln(\dot{\epsilon})$ vs. $\ln(\sigma)$ diagram, and (b) $\ln(\dot{\epsilon})$ vs. σ diagram, both at constant T .

Table 2.3. The values of α , n and apparent activation energy (Q) obtained in this research and other literatures.

Refs.	α	n	Q (kJmol ⁻¹)	conditions
Current study	0.024	5 ~ 10	383	torsion deformed IF steel; 400°C-850°C
A. Oudin et al. ⁴	0.2	3.8	372	torsion deformed IF steel; 765°C-850°C
G. Glover et al. ¹¹	-	-	280	torsion deformed pure iron; 550°C-880°C
S.V. Mehtonen et al. ¹⁰	0.022	3.7	397	comp. of ferritic stainless steel; 950°C-1050°C

Table 2.4. The activation energy reported for some thermally-activated phenomena in similar materials.

process	Q (kJmol ⁻¹)
Self-diffusion in pure ferritic iron ¹³	251 (ferromagnetic)
	239 (paramagnetic)
Grain boundary diffusion ¹⁴	174
Mobility of grain boundary in static recrystallization; 70%-90% cold rolled IF steel/annealed at 600°C-675°C ¹⁵	237-365

Additionally, **Table 2.4** summarizes the activation energies for some thermally-activated processes in bcc-ferrite. Similarly, the activation energy for hot deformation was quite larger than the Q values for any single atomic-scale phenomena such as self-diffusion of iron atom in ferritic (bcc) lattice (251 kJmol⁻¹ ¹³) or grain boundary diffusion (174 kJmol⁻¹ ¹⁴). As a result, considering the apparent activation energy does not seem to be a simple or straightforward solution to discuss the atomic-scale events governing the deformation.

Later in this study, the Z -dependence of different parameters, e.g., the flow stress or the grain size, will be discussed. For that reason, the average Q (383 kJmol⁻¹) was used to determine the Z parameter. Even though assuming a single and constant Q over the entire range of deformation does not seem precise, such a single/constant Q value is taken for the purpose of simple discussion from a practical viewpoint.

- Stress vs. Z parameter

In **Fig. 2.8**, the stress data obtained in this study (e.g. σ_m and σ^*) together with the data collected in two other studies on the hot deformation of IF steel ^{4,7} were plotted

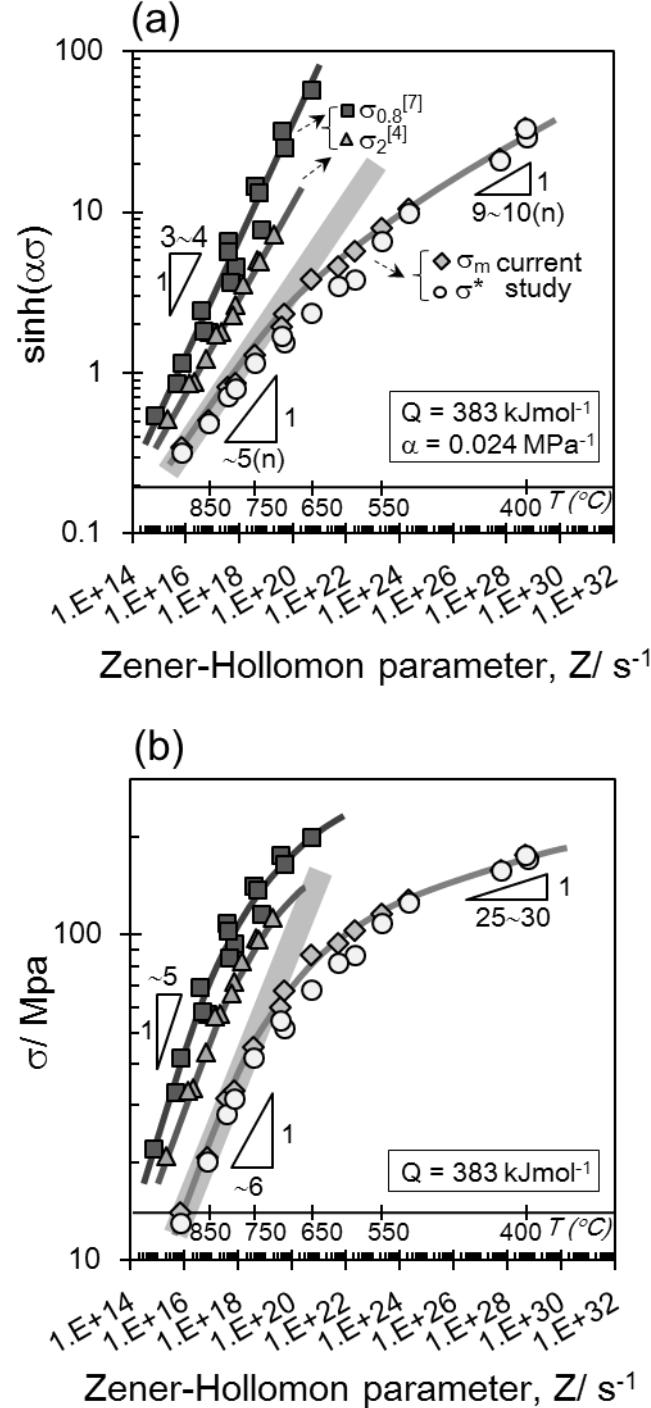


Figure 2.8. Z -dependence of the stress, using (a) the hyperbolic and (b) the power-law correlations (current study; σ_m as the maximum stress and σ^* as the stress at the final strain, other studies; $\sigma_{0.8}$ as the stress at $\varepsilon = 0.8$ in hot compression ⁷, $\sigma_{2.0}$ as the stress at $\varepsilon = 2.0$ in hot torsion ⁴). The equivalent temperatures are shown only for a constant strain rate of 0.1 s^{-1} .

as a function of the Z parameter, in the forms of $\sinh(\alpha\sigma) - Z$ (**Fig. 2.8a**) and $\sigma - Z$ (**Fig. 2.8b**). For an applicable comparison, the same Q (383 kJmol⁻¹) and α (0.024 MPa⁻¹) were assumed for all plots.

Over the lower Z region in **Fig. 2.8a** (> 650 °C), single lines could be fitted on the data obtained in this study ($n \sim 5$) and those in others ($n: 3 \sim 4$), indicating that the hyperbolic sine relationship could well predict the stress variations at least under the hot deformation conditions (with low Z). However, over the wider Z conditions, a slight deviation from the linearity could be observed ($n \rightarrow 9 \sim 10$), which was more noticeable in case of the σ_m plot. In fact, this deviation was accompanied with the change in the shape of the stress-strain curves, which happened under some medium conditions (~ 650 °C), as was previously shown in **Fig. 2.5**.

For comparison, a similar plot in the form of $\sigma - Z$ was considered in **Fig. 2.8b**. To make a simple analysis, the same Q value (383 kJmol⁻¹) was used here. It should be mentioned that the power-law form is often used for the low stress conditions. As a result, the deviation from the linearity (or power-law break down) was more obvious in **Fig. 2.8b**, when approaching towards high Z (high stress) conditions. In any case, as was suggested by the slopes in the plots (in **Figs. 2.8a, 2.8b**), the stress progressively increased with increasing Z ; however, the variation in the low Z region was much larger than that in the high Z region. Accurate or not, one can consider this behavior as a sign of gradual change in the deformation mechanisms (e.g., from thermal to athermal modes) [16–19](#). This will be more investigated in the following sections, where microstructures associated to different Z conditions are explained.

2.3.2. Microstructure vs. Z parameter

Figure 2.9 exhibits typical microstructures on the θ - Z planes of the IF steel highly deformed (5 ~ 7) at 850°C (**Figs. 2.9a, 2.9b**), 750°C (**Figs. 2.9c, 2.9d**), 650°C (**Figs. 2.9e, 2.9f**), 550°C (**Figs. 2.9g, 2.9h**), and 400°C (**Figs. 2.9i, 2.9j**), at two different strain rates of 0.01 s⁻¹ (upper maps) and 1.0 s⁻¹ (lower maps). The equivalent Z value for each condition is also provided. In the EBSD grain boundary maps, green and blue lines represent low angle boundaries (LABs; with misorientation (θ), $2^\circ \leq \theta < 15^\circ$) and high angle boundaries (HABs; $\theta \geq 15^\circ$), respectively. For the microstructures shown in **Fig. 2.9**, the applied strains were high enough to consider that they reached steady-state conditions from a microstructural point of view.

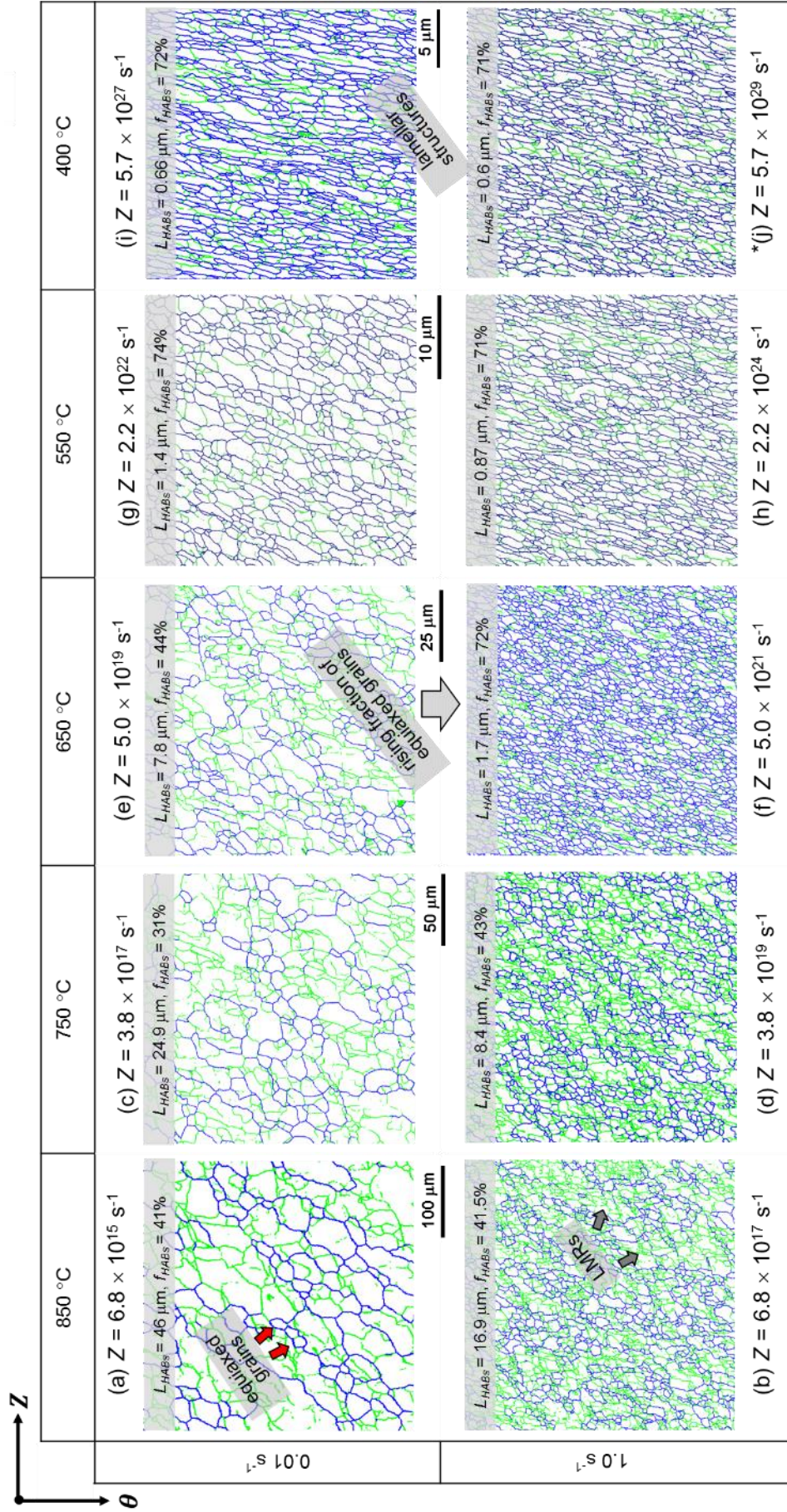


Figure 2.9. EBSD grain boundary maps showing typical microstructures of the IF steel highly deformed ($\varepsilon : 5 \sim 7$) at strain rates of 0.01 s⁻¹ (upper maps) and 1.0 s⁻¹ (lower maps) at (a, b) 850°C, (c, d) 750°C, (e, f) 650°C, (g, h) 550°C, and (i, j) 400°C. In the description of the boundary maps; HABs ($\theta \geq 15^\circ$) are expressed as blue lines and LABs ($2^\circ \leq \theta < 15^\circ$) as green lines, excluding the boundaries with the misorientation $< 2^\circ$ due to the inaccuracy of data. Low misorientation regions are indicated as LMRs. In addition the equivalent Z values for various conditions are provided. *(j) The modified Z value due to the deformation heating was estimated $\sim$

In general, decreasing the temperature or increasing the strain rate (increasing Z) enhanced grain refinement. As a result, a range of fine and ultrafine grained structures with the HAB-spacing (L_{HABs}) of $46\ \mu\text{m} \sim 0.6\ \mu\text{m}$ was attained. The common microstructures at lower Z conditions (e.g., **Figs. 2.9a-d**) consisted of the fine equiaxed grains of which fraction was, however, relatively small in the whole microstructures. The microstructures at lower Z displayed heterogeneity with rather low fractions of HABs ($f_{HABs} < 45\%$). This was somewhat different from typical DRX microstructures often observed in austenitic steel ²⁰ or copper ¹⁹ having fcc structure with medium $\sim$ low stacking fault energies. In those fcc-materials, DRX grains occupy the entire matrix at even lower strains than those applied in the present investigations. In the IF steel with easy-recovery nature, instead, a large fraction of coarse matrices including LABs were observed. From now on, the coarse matrices including LABs are referred as low misorientation regions (LMRs).

Approaching to the higher Z conditions, a transition in the microstructure could be clearly observed. Firstly, the fractions of the fine equiaxed grains noticeably increased with the increase in Z (e.g., compare **Fig. 2.9e** with **Fig. 2.9f**). In other words, the area of LMRs significantly reduced and the microstructures became more homogeneous with larger f_{HABs} ($> 70\%$). Eventually, at the highest Z conditions (e.g., **Figs. 2.9i** and **2.9j**), lamellar microstructures elongated along the shear direction formed, although a few equiaxed grains still existed within the lamellae. It should be noted that conditions with larger Z (than that in **Fig. 2.9j**) were not tried in this study because of the sample failure and the risk of substantial adiabatic heating during the deformation.

The microstructural parameters quantitatively evaluated from **Fig. 2.9** are summarized in **Fig. 2.10a**, where f_{HABs} was plotted as a function of the Z parameter. The fraction of HABs, f_{HABs} (or correspondingly the fraction of fine equiaxed grains surrounded by HABs) increased from $30\% \sim 40\%$ under the low Z conditions to $70\% \sim 75\%$ under the high Z conditions, and then saturated. Simultaneously, the grain shape became more and more elongated gradually (**Fig. 2.10b**). The rise in f_{HABs} was accelerated under some intermediate Z conditions that were mainly associated to the deformation conditions around $650\ ^\circ\text{C}$.

Furthermore, the grain size was measured within the entire Z range. Due to the microstructure heterogeneity explained before (especially in the low- Z microstructures shown in **Fig. 2.9**), two types of grain sizes were considered; (1) L_{HABs} : the average

spacing of all HABs existed in the matrix and (2) $D_{GOS < 2^\circ}$: the average diameter of the fine equiaxed grains with the grain orientation spread (GOS) $< 2^\circ$. To calculate the value of GOS based on a definition used in the TSL OIM software, firstly the

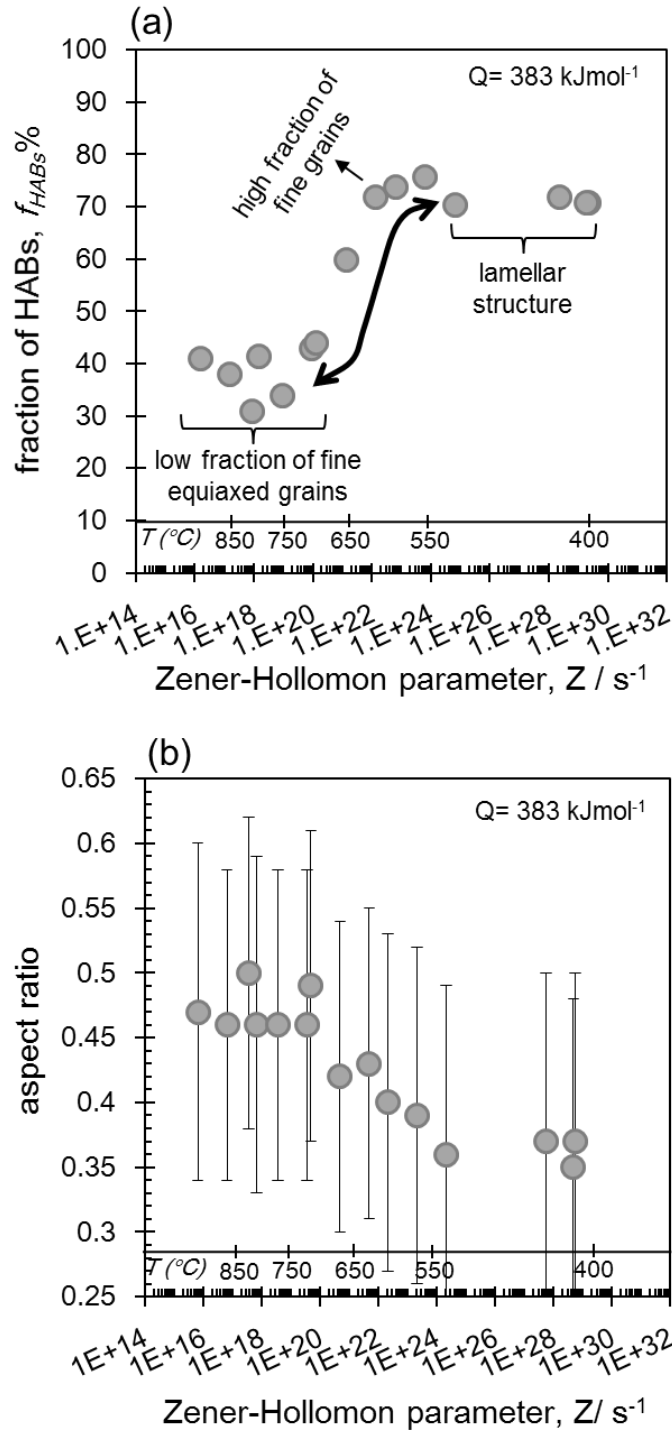


Figure 2.10. Z-dependence of (a) the fraction of HABs (f_{HABs}) and (b) the aspect ratio of the grain shape. The corresponding temperatures to one constant strain rate (0.1 s^{-1}) are also indicated for reference.

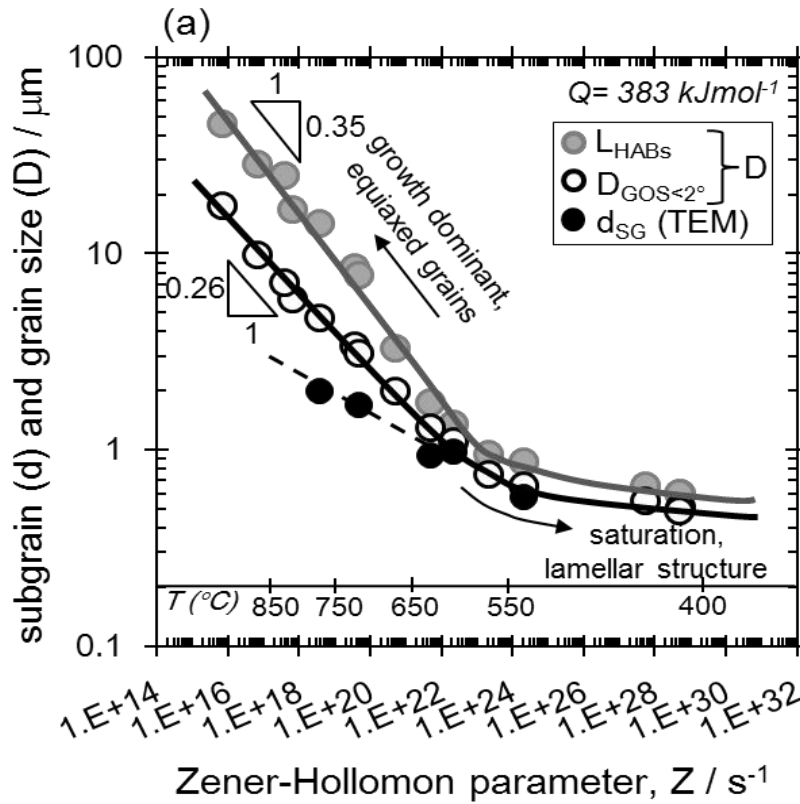
misorientation between the average orientation of a grain and the orientation of each individual measurement point within that grain is calculated. The average of these misorientations is then determined and assigned to the grain. The GOS parameter can express the extension of deformation inside a grain. For example, the GOS value in a newly generated grain (e.g. a (dynamically) recrystallized grain) is lower than that in a deformed and elongated grain. In the current study, the criterion $GOS < 2^\circ$ was found to be appropriate to extract the fine equiaxed grains from the remaining deformed matrix such as LMRs.

Fig. 2.11a demonstrates the variation of the grain size (L_{HABs} and $D_{GOS<2^\circ}$) within the entire Z range. Additionally, subgrain sizes (d_{SG}) under some deformation conditions are added in **Fig. 2.11a**. The value of d_{SG} was determined by measuring the spacing of all boundaries in the substructures observed in TEM micrographs (**Figs. 2.11b, 2.11c**).

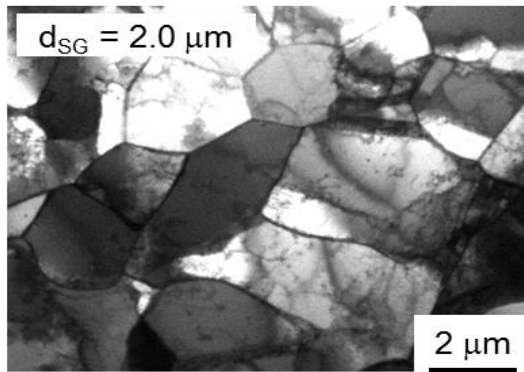
Figure 2.11a suggests that the $D - Z$ relationship could be represented by power-law relationships; i.e., $D_{GOS<2^\circ} \propto Z^{-0.26}$ or $L_{HABs} \propto Z^{-0.35}$. However, these relationships were applicable over the lower Z region, where the grain size was larger than the subgrain size and the grain shapes were rather equiaxed. In that case, it is reasonable to assume that the migration of HABs and the grain growth play a key role in the microstructural evolution.

Nonetheless, the power-law relationship eventually broke down under large Z conditions, where the grain size (D) became nearly the same as the subgrain size (d_{SG}). This was accompanied with the appearance of lamellar structures (**Fig. 2.11c**). Further grain refinement was significantly limited or seemed to approach an apparent saturation. This could be clearly found by comparing the lamellar microstructures in **Figs. 2.9i** and **2.9j**, where increasing the deformation rate could not remarkably refine the lamellar spacing. In other words, the microstructure evolution was apparently less sensitive to the change in the deformation conditions (or Z parameter) under very high Z conditions.

To find out what phenomenon controls the grain size, a simple approach using the diffusion distance in the bcc-ferrite is taken in this section. Here, both lattice (bulk) diffusion and grain boundary diffusion are considered. **Equation 2.12** and **Eq. 2.13** define the lattice diffusion coefficient (D_l) and the diffusion distance (L_l) for a period of t at a temperature T , respectively;



(b) $Z = 3.7 \times 10^{18}$



(c) $Z = 2.1 \times 10^{24}$

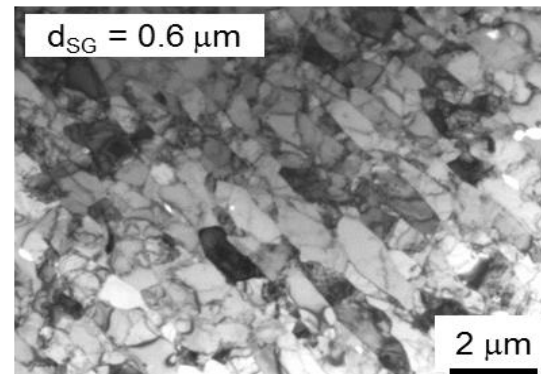


Figure 2.11. (a) Z -dependence of the grain size (L_{HABs} : average spacing of all HABs or $D_{GOS<2^\circ}$: average diameter of the fine grains with the grain orientation spread: $GOS < 2^\circ$). Subgrain sizes: d_{SG} for some deformation conditions are also indicated (filled circles). The corresponding temperatures to one constant strain rate (0.1 s^{-1}) are also shown. (b, c) Examples of TEM micrographs under (b) a low- Z condition and (c) a high- Z condition, used to determine the subgrain sizes (d_{SG}).

$$D_l = D_l^0 \exp(-Q_l/RT) \quad (2.12)$$

$$L_l = \sqrt{D_l t} \quad (2.13)$$

where, D_l^0 is the pre-exponential factor (a constant; $2 \times 10^{-4} \text{ m}^2 \text{ s}^{-1}$) ¹³, Q_l is the activation energy for the lattice diffusion (251 kJ mol^{-1}) ¹³, L_l is the lattice diffusion distance, and t is the time. Similarly, **Eq. 2.14** and **Eq. 2.15** describe the diffusion coefficient (D_{gb}) and the diffusion distance (L_{gb}) for the grain boundary diffusion:

$$\delta * D_{gb} = \delta * D_{gb}^0 \exp(-Q_{gb}/RT) \quad (2.14)$$

$$L_{gb} = \sqrt{D_{gb} t} \quad (2.15)$$

where Q_{gb} is the activation energy for the grain boundary diffusion (174 kJ mol^{-1}) ¹⁴. Here, δ is the grain boundary width and $\delta * D_{gb}^0$ was taken as $1.1 \times 10^{-12} \text{ m}^3 \text{ s}^{-1}$ ²¹.

Based on such assumptions, the lattice diffusion distance (L_l) and the grain boundary diffusion distance (L_{gb}) for a period of time in the torsion deformation were calculated. **Figure 2.12** shows the values of grain sizes ($D_{GOS < 2^\circ}$) determined in the current study, plotted as a function of the lattice diffusion distance and the grain boundary diffusion distance. The dashed line in **Fig. 2.12** denotes the equality of the grain size and the diffusion distance. It is interesting that the grain size is roughly comparable to the boundary diffusion distance, especially for a part of data of which the grain size is above $1 \mu\text{m}$. On the other hand, the grain size is much larger than the equality line for the lattice diffusion distance, indicating that the lattice diffusion does not control the grain size.

Such an analysis was previously carried out by Murty et al. ²² and similar results to that in the present study were found in a low (0.15%) carbon steel highly deformed in a single-press compression test. It is concluded from the present consideration that the grain boundary diffusion is the controlling mechanism to determine the grain size during the high-strain deformation.

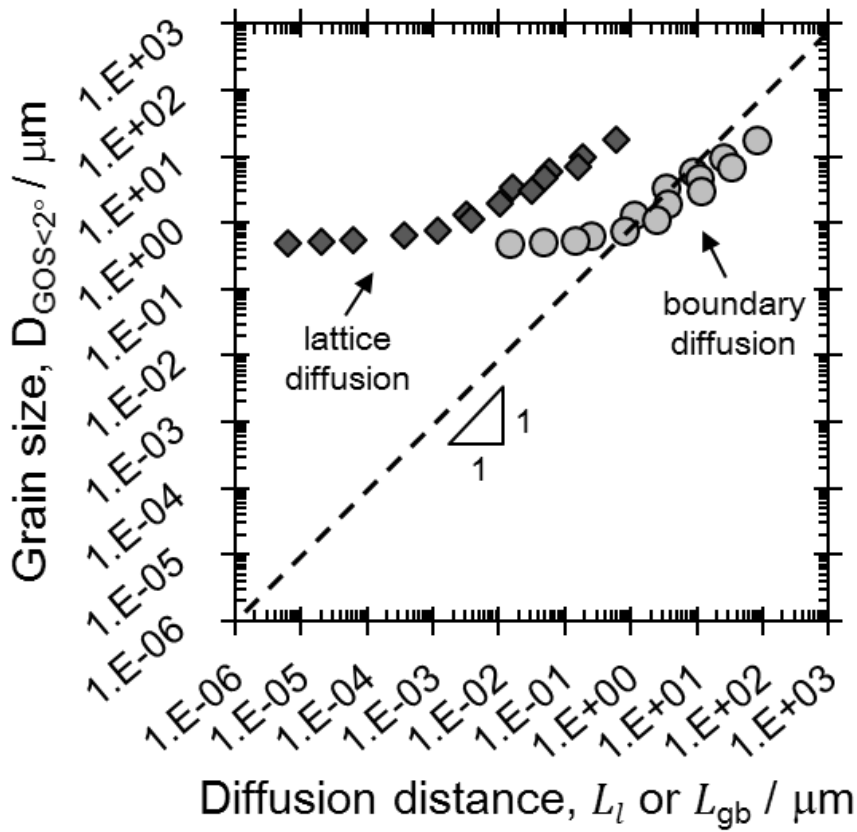


Figure 2.12. The grain size ($D_{GOS<2^\circ}$: diameter of fine equiaxed grains) as a function of the lattice diffusion distance (L_l) or as a function of the grain boundary diffusion distance (L_{gb}). The dashed line denotes the equality of the grain size and the diffusion distance.

2.3.3. Stress – grain size relationship

The variations of the flow stress (**Fig. 2.8**) and the grain size (**Fig. 2.11**) as a function of Z parameter were explained in the **Sections 2.3.1** and **2.3.2**. On the other hand, Z is defined purely by the deformation condition ($\dot{\epsilon}$, T), considering a constant Q for the entire Z range. Here, therefore, a direct relationship between the flow stress and the grain size, both of which are the material's properties, is considered in this section. Such a plot might reveal more straightforward view in the microstructure evolution during the high strain (torsion) deformation at different temperatures and strain rates.

Accordingly, the $\sigma - D$ plot shown in **Fig. 2.13a** illustrates such a relationship

obtained in this study, using two different definitions of the grain size (i.e., L_{HABs} and $D_{GOS<2^\circ}$). In **Fig. 2.13a**, considering the difference in the slope in the $\sigma - D$ plot (exponent N in $\sigma = D^{-N}$), three regions divided by red dashed lines can be seen. In the high flow stress (or high Z) region, where the lamellar structures (e.g. **Fig. 2.13b**) were observed, the grain size was close or almost equal to the subgrain size. The N value in this region was estimated around 1.0 ~ 1.15. With decreasing the flow stress (or Z), the grain size became larger than the subgrain size gradually. Therefore, the medium stress (or medium Z) region existed, of which N was around 0.3 ~ 0.5. It seemed that, under the medium stress, the lamellar structures became

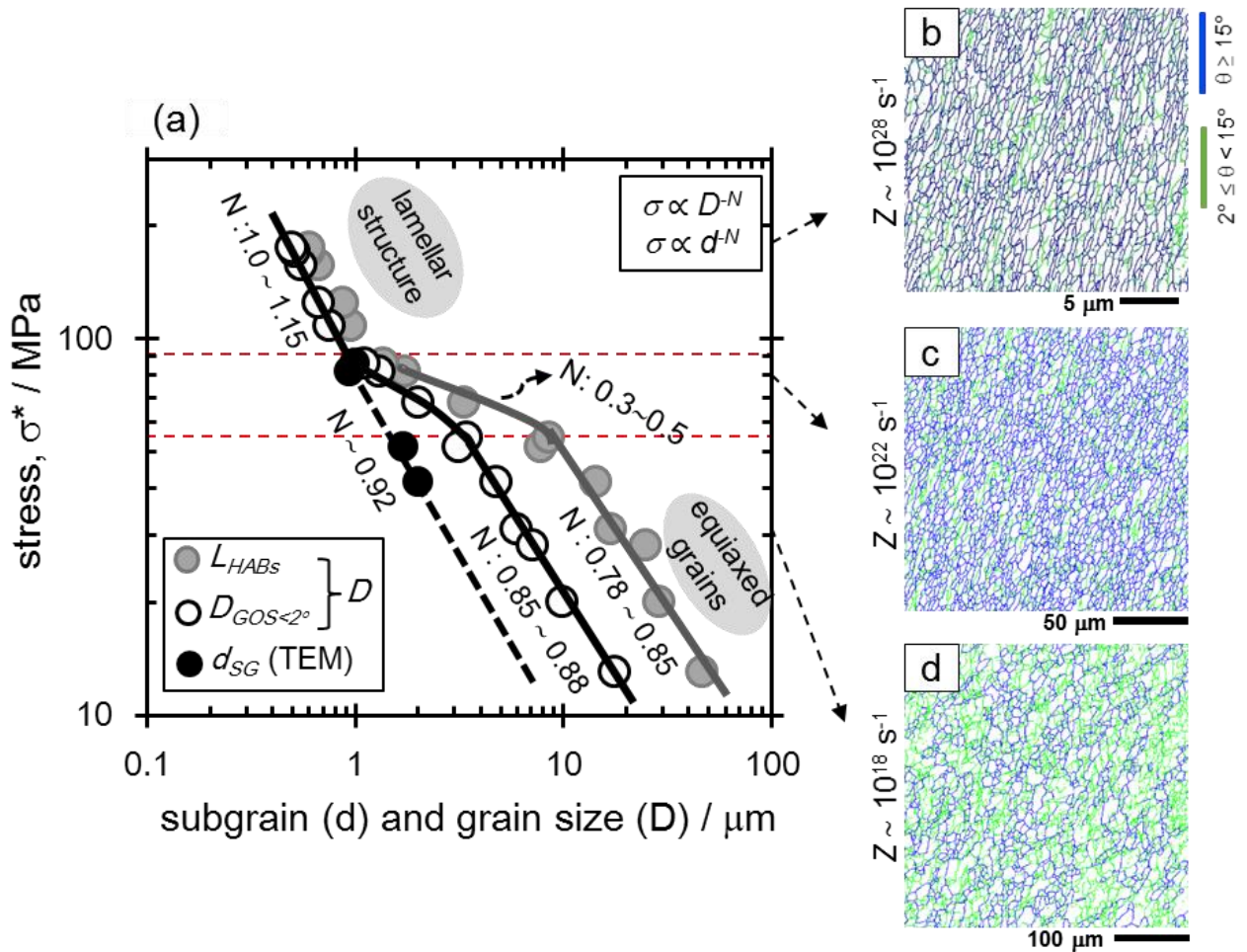


Figure 2.13. (a) Flow stress (σ^*) plotted as a function of grain size (D : L_{HABs} or $D_{GOS<2^\circ}$) and subgrain size (d_{SG}). The red dashed lines divide the graph into the high stress, medium stress, and low stress regions, of which N value ($\sigma \propto D^{-N}$) differs. (b-d) Typical microstructures (boundary maps) corresponding to (b) the high stress or high Z region, (c) the medium stress or medium Z region, and (d) the low stress or low Z region.

unstable and replaced by the fine equiaxed grains (**Fig. 2.13c**). Finally, another σ - D relationship was established in the low stress (or low Z) region where N was estimated around 0.78 ~ 0.88. The equiaxed grains surrounded by HABs (**Fig. 2.15d**) were coarser, however their fraction was small and the microstructure was heterogeneous owing to the formation of LMRs.

Based on to the σ - D plot shown in **Fig. 2.13**, all Z conditions under which the IF steel was deformed in the present study could be categorized into three regions. The summary of such a categorization is represented in **Table 2.5**, in which the deformation conditions are associated to the high Z region, the medium Z region, and the low Z region and highlighted by different colors.

Table 2.5. Summary of the deformation conditions (temperature and strain rate) applied for the IF steel in the present study. All conditions are categorized into the high Z (σ), the medium Z (σ), and the low Z (σ) regions with associated N values ($\sigma = D^{-N}$).

strain rate, $\dot{\epsilon} / \text{s}^{-1}$	Zener-Hollomon parameter, Z / s^{-1}					Z / s^{-1}	σ / MPa	N ($\sigma = D^{-N}$)
	400 °C	550 °C	650 °C	750 °C	850 °C			
10^0	*4.8×10 ²⁸	2.1×10 ²⁴	4.9×10 ²¹	3.7×10 ¹⁹	6.7×10 ¹⁷	High Z > 10 ²³	High σ > 90	1.0 ~ 1.15
10^{-1}	5.6×10 ²⁸	2.1×10 ²³	4.9×10 ²⁰	3.7×10 ¹⁸	6.7×10 ¹⁶	Medium Z 10 ¹⁹ ~ 10 ²³	Medium σ 45 ~ 90	0.3 ~ 0.5
10^{-2}	5.6×10 ²⁷	2.1×10 ²²	4.9×10 ¹⁹	3.7×10 ¹⁷	6.7×10 ¹⁵	Low Z < 10 ¹⁹	Low σ < 45	0.78 ~ 0.88

* Z was modified due to the deformation heating.

2.3.4. Microtexture analysis

EBSD measurements were also used to conduct a microtexture analysis of the specimens torsion-deformed under various Z conditions. Some representative results are displayed in **Fig. 2.14** and **Fig. 2.15**. The schematic ODF plots (for $\varphi_2 = 0^\circ$ and $\varphi_2 = 45^\circ$ sections) in **Fig. 2.14a** illustrate important ideal orientations in torsion deformation of bcc metals ²³. The notations for different orientations and their Miller indexes, with respect to the shear plane normal direction and the shear direction are also indicated. **Figures. 2.14b, 2.14c, and 2.14d** display the ODF plots of the IF steel specimens torsion-deformed under three different (low Z , medium Z , and high Z)

conditions, which correspond to **Figs. 9b** ($Z = 6.7 \times 10^{17} \text{ s}^{-1}$), **9f** ($Z = 4.9 \times 10^{21} \text{ s}^{-1}$), and **9j** ($Z = 4.8 \times 10^{28} \text{ s}^{-1}$), respectively.

The intensity in the ODF plot at the low Z condition (**Fig. 2.14b**) was quite high (> 20), indicating that the specimen had a certain texture. This was associated to the microstructure (**Fig. 2.9b**) in which typical DRX grains were not found but a mixture of LMRs and equiaxed grains was observed. Here, D2 orientation: $\{\bar{1}\bar{1}2\} \langle 111 \rangle$ was found to be the major texture component in **Fig. 2.14b**. There were also other minor orientations such as D1, E1/E2, etc. Similar results have been reported in previous studies on torsion deformation of ferritic steels including an IF steel ^{23–27}, where D2

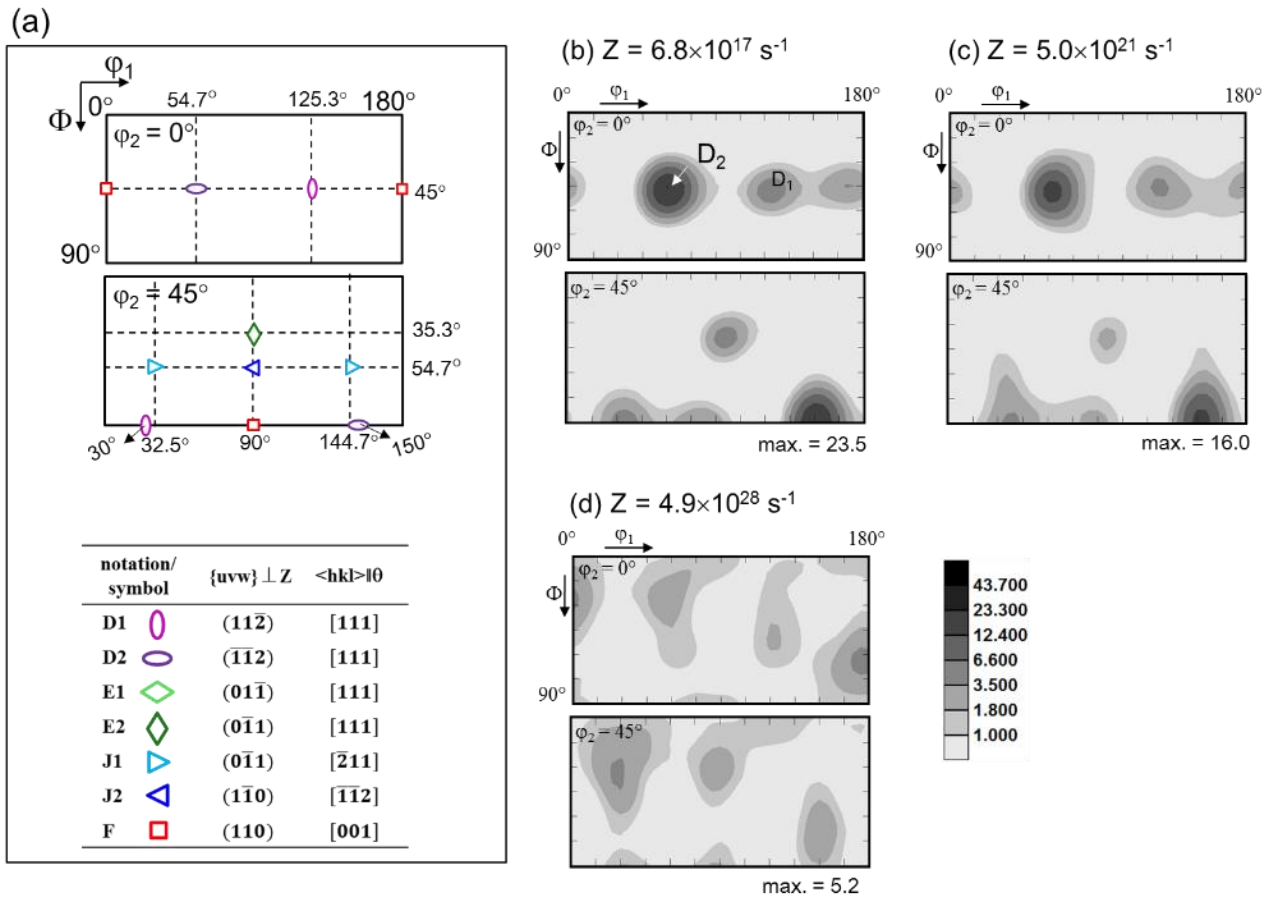


Figure 2.14. (a) Schematic ODF (orientation distribution function) plots at constant ϕ_2 sections (0° , 45°) including the ideal crystallographic orientations in torsion texture of bcc metals ^{23,38} together with the representative angles, notation, and the symbol related to each orientation; (b)-(d) ODF plots associated to three different deformation conditions whose microstructures were previously shown in Figs. 2.9b ($Z = 6.8 \times 10^{17} \text{ s}^{-1}$), 2.9f ($Z = 5.0 \times 10^{21} \text{ s}^{-1}$), and 2.9j ($Z = 4.9 \times 10^{28} \text{ s}^{-1}$).

orientation dominated at the expense of other orientations. Similarly, in **Fig. 2.14c**, the D orientation was found under the medium Z condition. However, the intensity of D decreased significantly with increasing Z, especially under the high Z condition (**Fig. 2.14d**).

In the same way, local orientation maps corresponding to the specimens shown in **Fig. 14** are represented in **Fig. 2.15**. Some typical orientation components are expressed in different colors in the microstructures. For example, the low Z microstructure (**Fig. 2.15a**) clearly shows a dominance of the D2 component (dark purple), occupying a high area fraction (~ 35%). It was found that those areas with the D orientation mostly corresponded to the coarse matrix or LMRs. On the other hand, increasing Z (**Fig. 2.15b** and **2.15c**) suppressed the D2-oriented regions (LMRs) to a great extent. **Figure 2.15d** provides the detailed fractions of all ideal orientations expressed in different colors in **Fig. 15a-c**, over the entire Z conditions. The areas with sufficiently large sizes were scanned for increasing the statistical accuracy, although these data were obtained in local (microtexture) measurements and therefore might be slightly scattered compared with macroscopic textures measured by XRD. Nevertheless, it is clear that the lower Z conditions resulted in the stronger D2 domination. In addition to the D2 component, D1 and E1/E2 were found to be the secondary major orientation components.

In conventional DRX-type metals such as copper ²⁸, DRX grains at a low Z condition quickly grow into the deformed grains, where the dislocation density and its gradient are large. As a result, DRX led to a texture randomization after a certain strain. However, DRX under the low-Z conditions was restricted in the present IF steel (a recovery-type metal), so that a micro-texture evolution different from DRX-type metals was realized (**Fig. 2.14** and **Fig. 2.15**). In fact, a strong texture intensity was found in the highly deformed specimens. The microtexture results and how they develop during the torsion deformation will be discussed in the next chapter (Chapter 3).

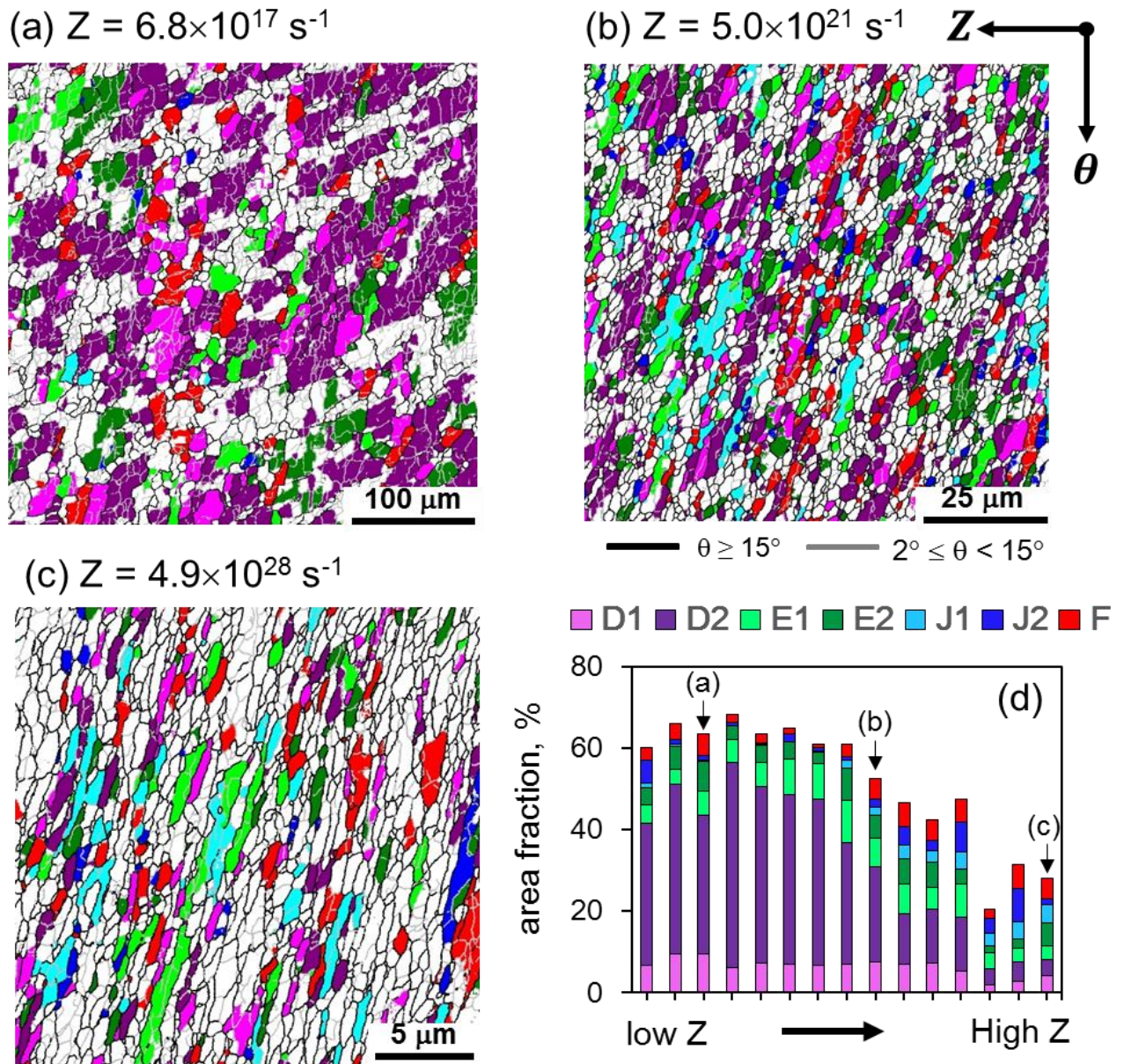


Figure 2.15. (a)-(c) Orientation maps (with various highlighting colors illustrating the distribution of different crystallographic orientations) associated to three different deformation conditions whose microstructures were previously shown in Figs. 2.9b ($Z = 6.8 \times 10^{17} \text{ s}^{-1}$), 2.9f ($Z = 5.0 \times 10^{21} \text{ s}^{-1}$), and 2.9j ($Z = 4.9 \times 10^{28} \text{ s}^{-1}$). Here, the HABs and the LABs are shown by black and gray lines, respectively. (d) Orientation fraction chart over the entire Z conditions studied in the present study (details regarding the Miller indexes of the orientations as provided in Fig. 2.14).

2.4. Discussion

2.4.1. Material's behavior as a function of Z parameter

In the present study, the flow stress decreased with decreasing Z (**Figs. 2.5, 2.8**), i.e., with increasing the temperature and decreasing the strain rate. However, the σ - Z relationship (**Fig. 2.8**) did not follow an identical function in the wide Z range, proved by both hyperbolic and power-law types relationships. In other words, the σ variations took different n ($Z = A [\sinh(\alpha\sigma)]^n$) and $\dot{n}$ ($Z = \dot{A} \sigma^{\dot{n}}$) values ($n = 1/\text{slope}$) when shifting from high Z to low Z or vice versa.

The n and $\dot{n}$ became smaller (resulting in larger slope) at low Z , indicating that the flow stress started to decrease faster under the lower Z conditions. Such a deviation from an identical function in the σ - Z relationship was also reported in other metals such as copper ²⁹ and 304 austenitic stainless steel ¹⁶. Such a deviation suggested a transition in terms of high- Z -to- low- Z conditions, warm -to- hot deformation, or athermal -to- thermal behaviors, under which diffusion-assisted phenomena can be accelerated and can act as restoration mechanisms.

Similar to the Z -dependence of σ (**Fig. 2.8**), the Z -dependence of the grain size (**Fig. 2.11a**) followed different functions under a wide range of Z conditions. In the low- Z region in **Fig.11a**, the grain size was greatly sensitive to the deformation conditions and could be efficiently refined with increasing Z . However, the $D - Z$ relationship started to deviate from the tendency in the low- Z region with further increase in Z . The $D - Z$ relationship eventually led to the new saturation regime in which the grain refinement was slowed down significantly. This corresponded to the formation of the UFG lamellae (**Fig.11c**) with grain sizes below 1 μm .

It can be said that at low Z a higher degree of grain growth is generally expected. In fact, the boundaries formed during deformation can migrate easier at high temperatures and low strain rates, once they became HABs with high mobility. Therefore, in the low- Z region in **Fig.11a**, the grain size became larger than the subgrain size. It was shown (**Fig.12**) that the grain size was comparable to the grain boundary diffusion distance (L_{gb}). This similarity suggested that the boundary diffusion might be the main mechanism to control the migration of HABs and therefore determine the grain size.

At high Z , the boundary mobility (or diffusion) became more and more inhibited and the grain size became nearly equal to the subgrain size (**Fig. 11a**). The grain

size (or the lamellar spacing) in the new regime saturated at high Z . The reason(s) why the grain size did not decrease to further extent with increasing Z is not clear still. One possible explanation can be given by considering the effect of adiabatic heating. Especially during deformation at high Z , adiabatic heating can significantly raise the temperature of deformed specimen. As a result, grain coarsening can happen and prevent further grain refinement. However, the risk of substantial adiabatic heating was tried to be removed in the present study by using an advanced induction heating system to adjust the temperature of the specimen.

In the current study, the torsion deformation at larger Z was not successful due to the failure of specimens above a certain amount of strain. In this section, another $D - Z$ plot (**Fig. 2.16**) is provided, in which the variation of grain size is illustrated in a broader range of Z . The data shown in **Fig. 2.16** were gathered from other studies including a variety of deformation methods such as (hot) compression ⁷, (hot or warm) torsion in the current study, (warm) plane-strain compression ^{21,30}, ARB ²⁸ and HPT ^{31,32}. It is noteworthy that all the data were obtained in bcc-ferrite single-phase regions of pure iron ^{31,32}, ultra-low carbon steel ^{7,21,28}, and low carbon steel (0.15% C) ³⁰. Another assumption was to use the same activation energy for all the data to unify the Z parameter. In **Fig. 2.16**, the Q value of 250 kJmol^{-1} , which was known as the activation energy for the self-diffusion of iron atom in ferrite ¹³ was used.

The Z -dependence of the grain size greatly changed in such a broad range of deformation conditions shown in **Fig. 2.16**; $D \propto Z^{-(0.3 \sim <0.05)}$. It can be seen that the grain size associated to the current study (displayed by red circles) followed a general tendency (shown by the shaded area) in the $D - Z$ plot. In **Fig. 2.16**., the grain refinement was firstly enhanced by the increase in Z , however, the refining slowed down at grain sizes below $\sim 1 \mu\text{m}$. At very large Z , the grain size of UFG ferrite processed by ARB or HPT was almost independent of the Z parameter ($D \propto Z^{(<0.05)}$). It seems that no matter how large Z is possible by SPD processes, the saturation behavior imposes a limitation of the minimum grain size one can achieve.

It is possible that the saturation of the grain size in SPD processed metals can be caused by a combination of various phenomena. One might be the grain coarsening due to adiabatic heating ³³ which was mentioned before. Some others can be briefly named as deformation localization and structure coarsening within shear bands ²⁸, removal of thin lamellae by grain boundary triple-junction motions ^{34,35}, and stress-driven boundary migration in thin lamellae ³⁶.

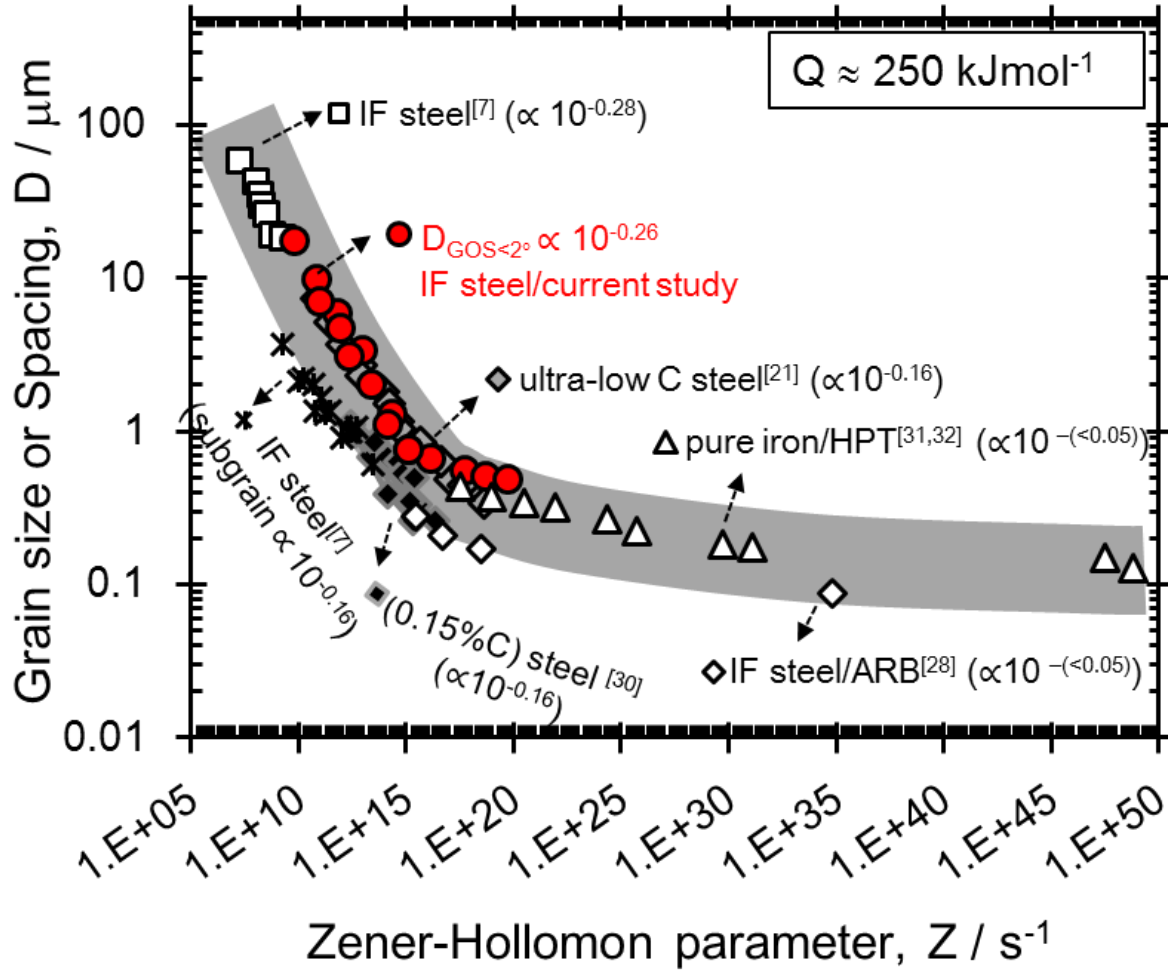


Figure 2.16. Variation of grain size as a function of Z , obtained from a variety of deformation methods under various conditions (from room temperature to warm or high temperatures) in bcc-iron with different chemical compositions (pure iron, low, and ultra-low carbon steels) ^{7,21,28,30–32}.

2.4.2. Stress – grain size relationship

The variation of the stress as a function of the grain size (shown in **Fig. 13a**) proved to be a more straightforward method to understand the microstructure evolution in the IF steel deformed in high strain (torsion) deformation under various conditions. With respect to the σ - D plot shown in **Fig. 13**, three different regions with different N ($\sigma = D^{-N}$) were realized; i.e., a high stress (Z) region, a medium stress (Z) region, and a low stress (Z) region. In the high stress (Z) region, lamellar structures (**Fig. 13b**) with grain sizes below 1 μm were found. This type of UFGs

dominated by elongated HABs was nearly the same as that often observed in a SPD-processed materials (e.g. **Fig. 1.6** in the Chapter 1), in which the grain subdivision is the dominant mechanism for the microstructure evolution. The N value was around 1.0 ~ 1.15, close to the N value (0.92) found in the extrapolated line on the stress (σ) – subgrain size (d) data.

In the medium stress (Z) region, the enhanced mobility of HABs (the enhanced diffusivity) led to the grain size larger than the subgrain size and the microstructure with rather equiaxed shapes (**Fig. 13c**) formed. The N value (0.3 ~ 0.5) in the narrow medium region changed drastically until the low stress (Z) region was realized (N : 0.78 ~ 0.88), in which the grain growth was dominant and the grain size became larger than 10 μm . The formation of equiaxed grains under the low stress (Z) conditions is sometimes discussed in terms of dynamic recrystallization (DRX) ³⁷. However, the heterogeneous microstructure shown in **Fig. 13d** was different from a typical DRX structure. In fact, the microstructure was a mixture of equiaxed grains surrounded by HABs and low misorientation regions (LMRs) with coarse and irregular shapes. Formation of such a heterogeneous microstructure seems to be a characteristic behavior of the easy-recovery ferrite in the IF steel deformed in high strain (torsion) deformation under low Z conditions.

According to the 3-stage variation found in the σ - D plot in **Fig. 13**, all the deformation conditions applied for the IF steel were categorized into three regions (**Table 2.5**). In the next chapter (Chapter 3), more details regarding the microstructure evolution under each condition will be discussed.

As the last point in this section, the stress and the grain size data in (vacuum-melted) pure irons studied by G. Glover et al. ¹¹ were reproduced and shown in **Fig. 2.17**. For comparison, the data associated to the IF steel in the present study are also represented by grey lines in **Fig. 2.17**. The pure iron ¹¹ was deformed by torsion within a ferrite single-phase region. G. Glover et al. ¹¹ reported the occurrence of conventional DRX in bcc-ferrite owing to the high purity (> 99.94%) in the vacuum-melted iron. As shown in **Fig. 17**, the N values associated to the pure iron were estimated as 0.7 in the σ - D plot and 1.1 in the σ - d plot. The grain size in the pure iron was available only for the low-stress conditions, of which N (0.7) was slightly smaller than N (0.78 ~ 0.88) obtained in the IF steel. Most importantly, a much larger grain size could be achieved in the pure iron than in the present IF steel. This suggests that the mobility of grain boundaries is very sensitive to the purity level

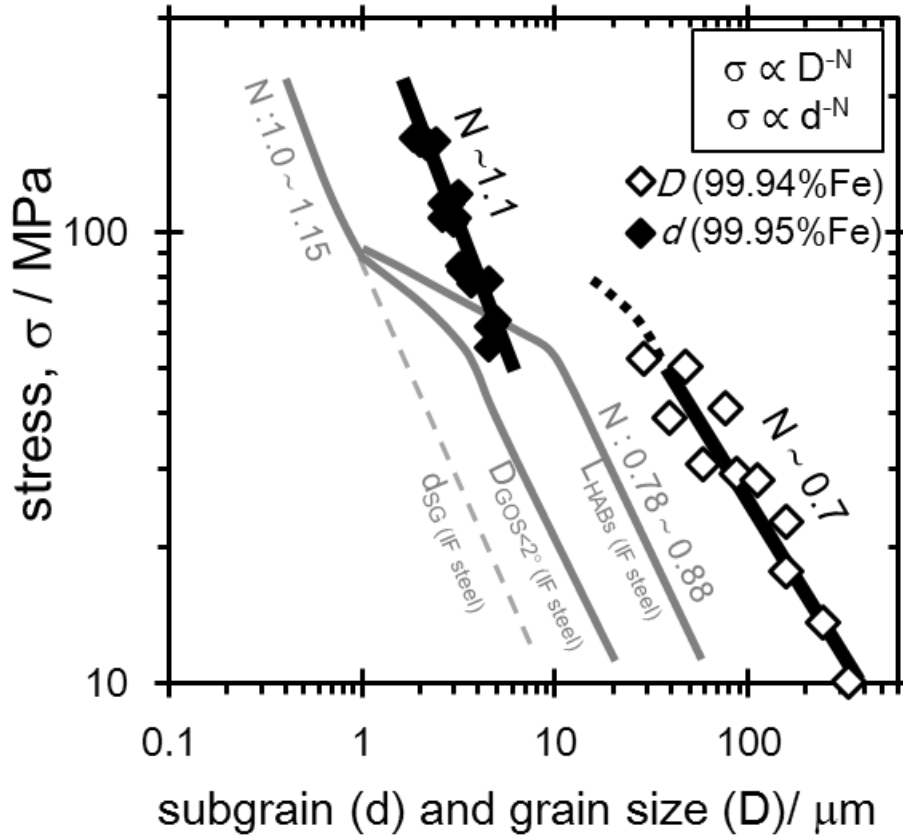


Figure 2.17. Stress as a function of grain size (D) and subgrain size (d), obtained in torsion deformation of vacuum-melted (v.m.) pure irons ¹¹. The lines and N values shown in grey color are associated to the IF steel in the present study, which were previously introduced in Fig. 2.13.

of the material. In the IF steel, some fine precipitates (e.g., titanium carbide and nitride) could impede the boundary migration. It can be concluded that in the easy-recovery ferrite, where the occurrence of DRX is somewhat difficult due to the enhanced DRV, the impurity level plays an important role deciding the type of microstructure evolution.

2.5. Conclusions

The formation of **fine or ultrafine grains** in high-strain (torsion) deformation (ε : 5 ~ 7) under various Z (temperature and strain rate) conditions was studied in single-phase **bcc-ferrite** of an IF steel. Approaching from high- Z to low- Z conditions or vice versa, several noticeable changes in the deformation regime were realized. The followings are the main results and new findings:

Different types of microstructures from **UFG lamellae** to **fine equiaxed grains** with various grain sizes from ~ **0.6 μm** to ~ **46 μm** were successfully achieved. With decreasing Z (increasing temperature and decreasing strain rate), material's properties such as the **flow stress (σ)** and the **grain size (D)**, respectively decreased and increased. However, the Z -dependences of σ and D followed different functions over the wide Z range in the present study. Under low Z where diffusion-assisted phenomena could accelerate, σ and D were greatly dependent on the Z parameter (temperature and strain rate). Under very high Z , however, σ and D became apparently less sensitive to the change in the deformation conditions.

The **σ - D relationship** ($\sigma = D^{-N}$) revealed that the entire Z conditions could be divided into three regions; i.e., a **high σ or high Z region** (N : 1.0 ~ 1.15), a **low σ or low Z region** (N : 0.78 ~ 0.88), and a narrow **transition** in between (N : 0.3 ~ 0.5) or **medium σ or medium Z region**. In the high Z region, the UFG lamellae were realized, of which the microstructure was dominated by elongated HABs ($f_{\text{HABs}} > 70\%$), similar to microstructures resulted from **grain subdivision** often found in SPD-processed metals. In the low Z region, **grain growth** was dominant, leading to the coarse and equiaxed grains, of which fraction was, however, relatively small in the whole microstructures. The microstructure was a mixture of equiaxed grains surrounded by HABs and some low misorientation regions (LMRs). Such a heterogeneous microstructure with a low fraction of HABs ($f_{\text{HABs}} < 45\%$) seemed to be a **characteristic behavior of easy-recovery ferrite** in the low Z region. Additionally, the microstructure in the medium Z region could be considered as a transition microstructure, in which HABs dominated the matrix like the UFG lamella at high Z but the grain morphology was rather equiaxed like the equiaxed grains at low Z .

2.6. References

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Chapter 3: Effect of strain on microstructure evolution in torsion-deformed bcc-ferrite

3.1. Introduction

As explained in Chapter 2, microstructures of highly torsion-deformed ferrite in the IF steel could be categorized into three types based on the relationship between the flow stress (σ) and the grain size (D) (the σ - D plot shown in **Fig. 2.13**) and other results: i.e., the microstructures developed under low Z conditions (low σ), medium Z (medium σ , or transition) conditions, and high Z (high σ) conditions. The aim of this chapter is to understand how they evolve during the deformation, selecting representative microstructures formed under those conditions. In other words, the effect of strain, from low to very high strains, on the evolution of new grain structures is investigated.

In this chapter, firstly the effect of strain on the microstructure evolution is explained for some medium Z (σ) conditions; i.e., $10^{19} \text{ s}^{-1} \sim 10^{23} \text{ s}^{-1}$ (45 MPa $\sim$ 90 MPa) (see **Table 2.5**). Then, the result is compared with those obtained under high Z (σ) and low Z (σ) conditions; i.e., **above** 10^{23} s^{-1} (> 90 MPa) and **below** 10^{19} s^{-1} (< 45 MPa), respectively. In particular, the generation of new grains and their differences in morphologies, sizes, fractions, etc. are explained. Finally, based on the results as well as those acquired in Chapter 2, the formation of fine/ultra-fine grained ferrite evolved under various Z (σ) conditions is discussed.

3.2. Experimental procedures

The titanium-added IF steel with an ultra-low carbon content (0.002 wt%), same as that used in Chapter 2, was used in the present study. The initial microstructure of the IF steel was a fully recrystallized microstructure with an average grain size around 60 μm . The chemical composition and the initial microstructure of the as-received IF

steel have been presented in Chapter 2 (section 2.2.1).

Torsion specimens with a 4 mm gauge length and 8 mm gauge diameter were made from the as-received material. For the torsion deformation, a thermo-mechanical processing simulator (Thermecmastor-TS) equipped with a high-frequency induction heating system was used. More details about the torsion test, the specimen size, and the thermomechanical processing simulator (Thermecmastor-TS) used have been provided in Chapter 2 (section 2.2.2).

In this chapter, some certain Z conditions in the ferrite single-phase region of the IF steel were chosen to carry out the torsion tests by different strains from low to very high amounts. The applied thermo-mechanical processing is schematically shown in **Fig. 3.1**. After the deformation to a certain strain planned, the specimen was water quenched immediately in order to freeze microstructure as much as possible.

The details regarding the specimen preparation for the microstructure analysis have been explained in Chapter 2 (section 2.2.3). The microstructural observations were carried out by electron backscattering diffraction (EBSD) measurements using a field emission scanning electron microscope (FE-SEM; JEOL JSM 7100F) operated at

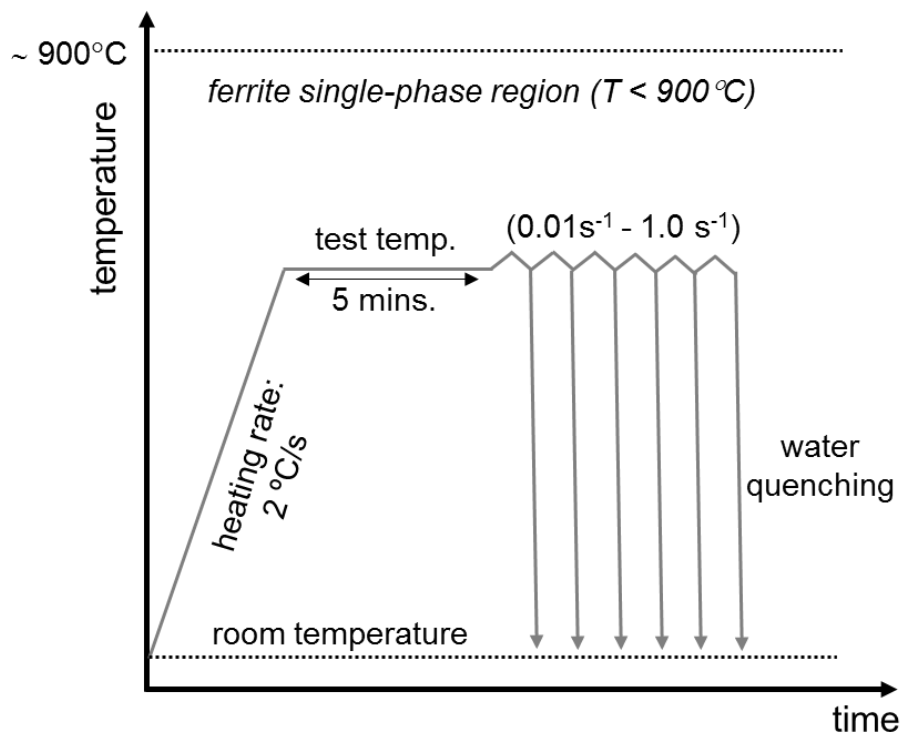


Figure 3.1. Thermo-mechanical processing schedules in the ferrite single-phase region used in this chapter.

15 kV with 15 mm working distance. Additionally, the substructures were examined carefully using electron channelling contrast imaging (ECCI) technique, carried out in a field emission scanning electron microscope (FE/SEM- JEOL JSM 7800F).

As mentioned before, the torsion tests were conducted under some selected conditions shown below, corresponding to three kinds of representative microstructures formed under low Z , medium Z , or high Z conditions. Details of the procedures are as follows:

(1) Firstly, the IF steel was deformed under some medium Z conditions ($10^{19} \text{ s}^{-1} \sim 10^{23} \text{ s}^{-1}$). More specifically, those conditions included three different pairs of deformation temperature and strain rate; 650 °C/ 0.01 s^{-1} ($Z = 4.9 \times 10^{19} \text{ s}^{-1}$), 650 °C/ 1.0 s^{-1} ($Z = 4.9 \times 10^{21} \text{ s}^{-1}$), and 550 °C/ 0.01 s^{-1} ($Z = 2.1 \times 10^{22} \text{ s}^{-1}$).

(2) Torsion test was also tried under a high Z condition ($> 10^{23} \text{ s}^{-1}$). Here, only one condition, i.e., 400 °C/ 0.01 s^{-1} ($Z = 5.6 \times 10^{27} \text{ s}^{-1}$) was examined since different strain rates or temperatures did not make a great difference in the microstructure evolution under high Z conditions, as was shown in **Figs. 2.9.i** and **2.9.j** in Chapter 2.

(3) Low Z conditions ($< 10^{19} \text{ s}^{-1}$) or high deformation temperature just below the bcc-to-fcc phase transformation temperature (A_3) were taken, to compare the microstructure evolution with those under the conditions described in (1) and (2). More specifically, the conditions included 850 °C/ 0.01 s^{-1} ($Z = 6.7 \times 10^{15} \text{ s}^{-1}$), 850 °C/ 1.0 s^{-1} ($Z = 6.7 \times 10^{17} \text{ s}^{-1}$), and 750 °C/ 0.01 s^{-1} ($Z = 3.7 \times 10^{17} \text{ s}^{-1}$).

3.3. Results

3.3.1. Evolution of substructures

Figure 3.2a shows flow stress-strain curves of the IF steel highly deformed by torsion under certain Z conditions (different pairs of deformation temperature and strain rate). Under the high Z condition, after a work hardening part, the curve reached a plateau with no stress drop at high strains. Under the medium Z conditions, after a work hardening part, the curves showed a broad peak or maximum stress (σ_m at ε_m) followed by softening at higher strains. The amount of stress drop after the maximum stress were estimated to be less than 23%.

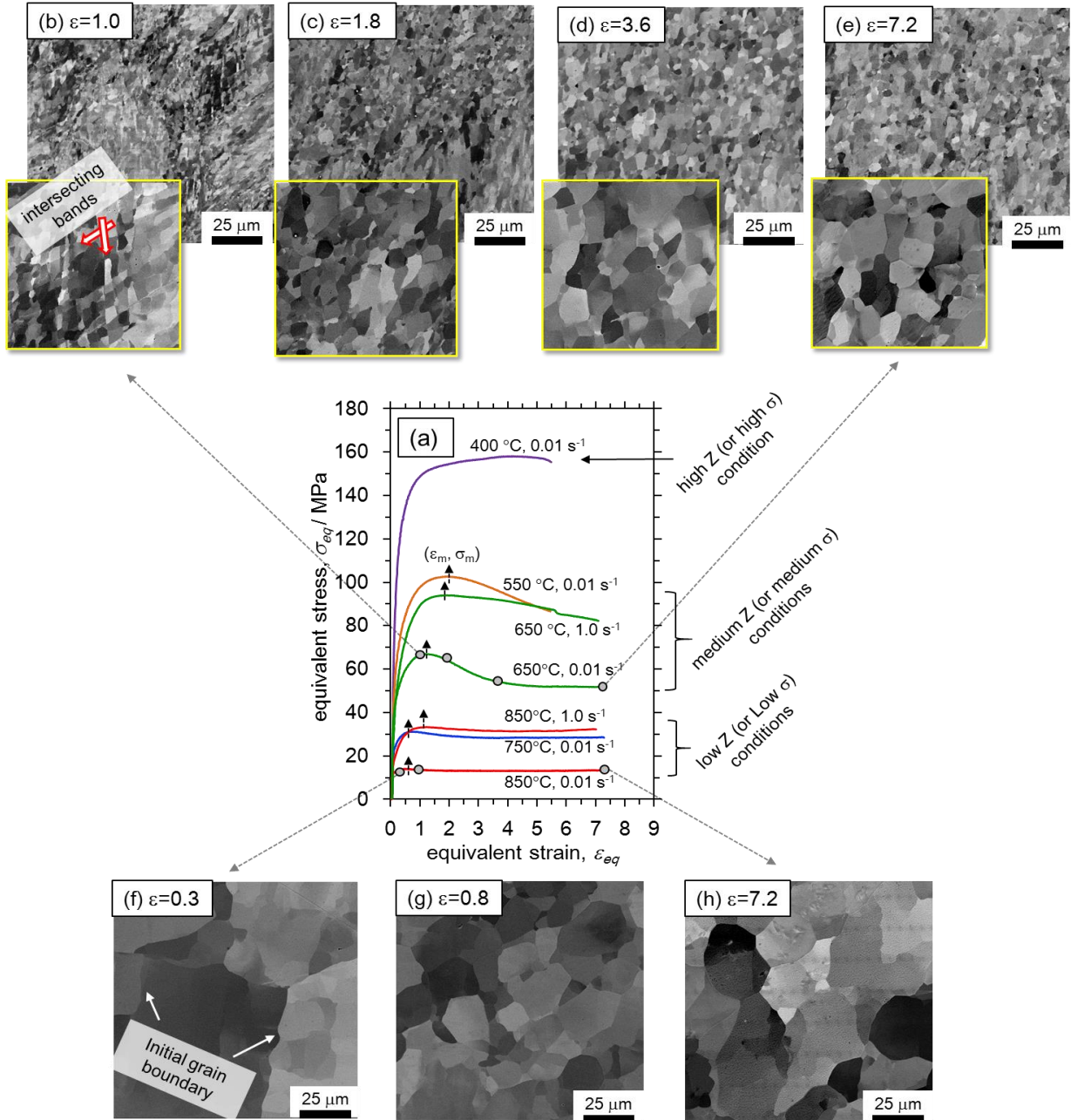


Figure 3.2. (a) Flow stress-strain diagrams in torsion of the IF steel under some certain Z conditions (different pairs of deformation temperature and strain rate) in the ferrite single-phase region ($< 900\text{ }^{\circ}\text{C}$), together with (b-h) representative electron channeling contrast images (ECCI) for the deformation conditions at (b-e) $650^{\circ}\text{C}/0.01\text{s}^{-1}$ (a medium-Z condition) and (f-h) $850^{\circ}\text{C}/0.01\text{s}^{-1}$ (a low- Z condition). The strains at which the microstructure observations were carried out are indicated by grey-circle symbols on the stress-strain curves in (a).

On the other hand, under the low Z conditions, the peak strain (ϵ_m) was smaller compared to that under the medium Z conditions. In these cases, the stress drop after σ_m was so small ($< 8\%$) that there was barely a difference between the maximum stress and the stress at high strains. Such a rather flat-type curve under the low Z

conditions is normally considered as a typical behavior of easy-recovery metals such as bcc-iron ^{1–6}.

ECC images of representative substructures at early stages of deformation as well as at large strains ($\varepsilon \sim 7$) are inserted in **Fig. 3.2**. Under a medium Z condition, cell blocks (**Fig. 3.2b**) could be observed at lower strains, in the form of banded structures. Their spacing and morphology were found to be different depending on the initial grains. The banded sub-structures gradually changed (**Figs. 3.2c-e**) into relatively equiaxed and coarser subgrains at high strains. On the other hand, under a low Z condition, the substructure size was much larger (**Figs. 3.2f-h**) than that under the medium Z condition; only a weak trace of banded substructure was found even at early stage (**Fig. 3.2f**), where the formation of equiaxed subgrains inside the initial grains seemed to be greatly accelerated due to the enhanced DRV.

The replacement of the elongated and banded substructures by the equiaxed ones with increasing the applied strain was also reported somewhere else ^{7–9}. This should be considered as a key point to understand the microstructure evolution, which will be discussed furthermore in the next sections.

3.3.2. Microstructure evolution as a function of strain in deformation under medium Z condition: $10^{19} \text{ s}^{-1} \sim 10^{23} \text{ s}^{-1}$

Microstructures at different (low to moderate and high) strains were comprehensively examined using the EBSD measurements. The results of deformation under the medium Z condition are discussed in this section.

• 3.3.2.1. $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ (650 °C/ 0.01 s⁻¹)

Figure 3.3 shows microstructural changes at different strains under the deformation condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ (650 °C/ 0.01 s⁻¹). The deformation was stopped at various strains displayed on the flow stress-strain curve (marked by open circles) in **Fig. 3.3a**, and the quenched specimens were analyzed by EBSD. **Figures 3.3b** through **3.3f** demonstrate representative microstructures at different strains. Here, two types of EBSD maps are displayed. The upper half of **Figs. 3.3a-f** shows only high angle boundaries (HABs; with misorientation $\theta \geq 15^\circ$) as blue lines, while the lower half contains low angle boundaries (LABs; $2^\circ \leq \theta < 15^\circ$) as green lines as well as HABs. The latter displays distribution and density of all boundaries with misorientation above 2° .

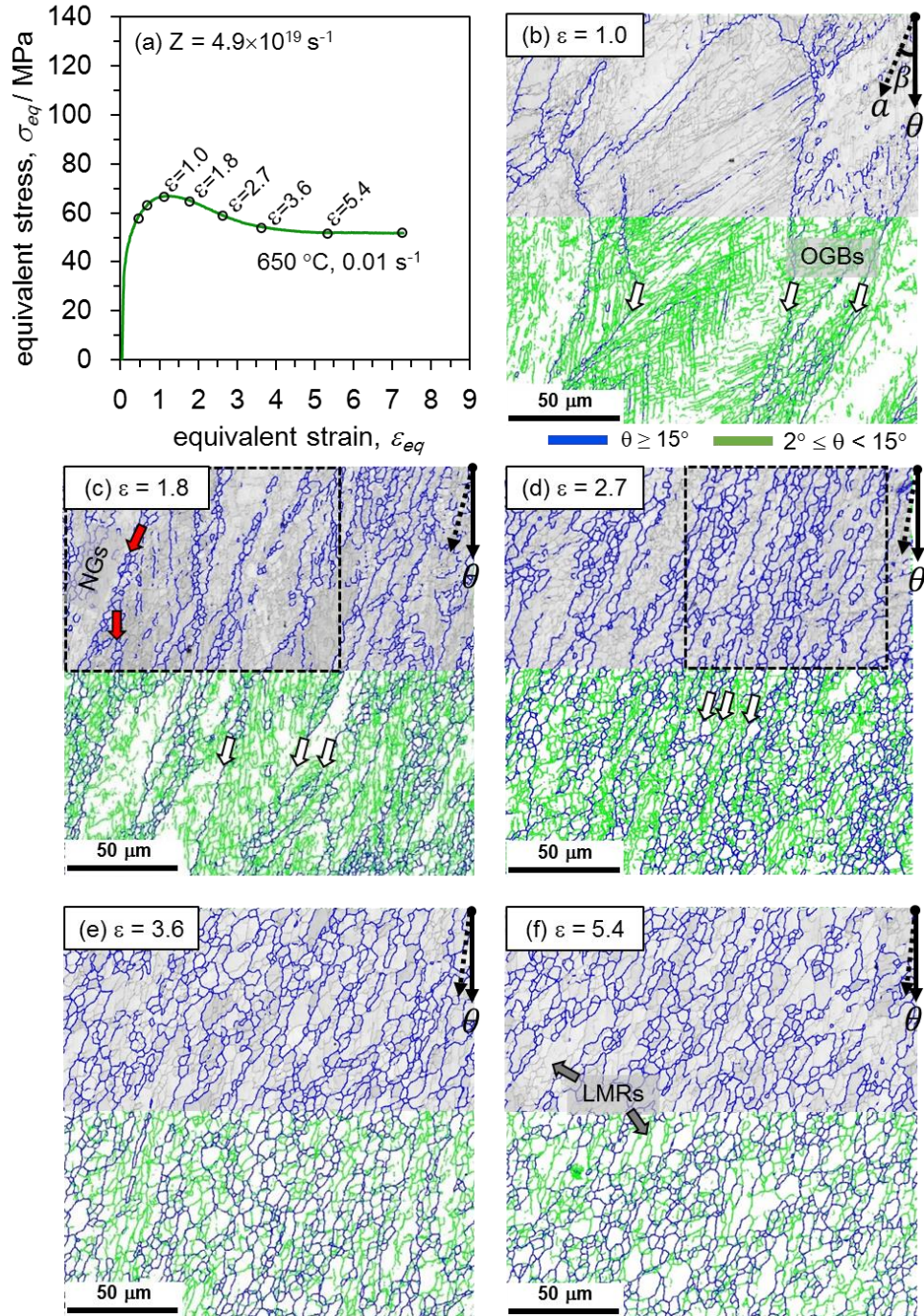


Figure 3.3. (a) Flow stress-strain diagram under the deformation condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ ($650^\circ\text{C} / 0.01 \text{ s}^{-1}$). Microstructure observations were conducted at different strains indicated by the open circle. (b-f) Representative torsion-deformed microstructures at the strains of (b) $\epsilon = 1.0$, (c) $\epsilon = 1.8$, (d) $\epsilon = 2.7$, (e) $\epsilon = 3.6$, and (f) $\epsilon = 5.4$. In each EBSD map (b-f), the upper half shows HABs (with misorientation $\theta \geq 15^\circ$) drawn by blue lines superimposed on the image quality (IQ) map, while the lower half includes LABs ($2^\circ \leq \theta < 15^\circ$) drawn by green lines in addition to HABs. Small-angle boundaries with misorientation below 2° are excluded here for removing the inaccuracy of EBSD measurement and analysis. Moreover, β indicates the angle between the shear direction (θ) and the major axis (a) of original grains after the corresponding shear deformation ($\tan 2\beta = 2/\sqrt{3}\epsilon$). Abbreviations: OGBs as original grain boundaries (pointed out by white arrows in (b), (c) and (d)), NGs as new grains (red arrows in (c)), and LMRs as low misorientation regions (gray arrows in (f)).

It could be seen that deformed initial grains at early stages were gradually replaced by finer and rather equiaxed grains at moderate to high strains. Such a microstructure evolution was accompanied with the stress drop (by $\sim 20\%$) on the flow stress-strain curve. In fact, around the peak at $\varepsilon = 1.0$ (**Fig. 3.3b**), the original grains were subdivided by high density of deformation-induced boundaries although most of them were LABs at this stage. They often showed the trace of band-like substructures previously observed in the ECC image (**Fig. 3.2b**). With further straining to $\varepsilon = 1.8$ (**Fig. 3.3c**) and $\varepsilon = 2.7$ (**Fig. 3.3d**), the original grains (of which boundaries were indicated by white arrows) were more elongated and the grains became thinner. Simultaneously, the fraction of newly formed HABs increased noticeably, in the form of scattered HAB segments and fine grains (pointed out by red arrows). The fine grains generally formed near original grain boundaries or inside original grains. The regions inside the rectangular frames marked in **Figs. 3.3c and 3.3d** are selected for a detailed explanation on microstructures, which will be discussed later in **Fig. 3.4**.

In **Fig. 3.3**, the vertical direction coincides with the shear direction (θ) in the torsion deformation, and the axis (**a**) shown by dotted arrows indicates the direction of the major axis of original grains after the corresponding shear deformation, assuming that the initial shape of the grains is a circle, they are deformed into elliptical shapes by shear and the axis (**a**) corresponds to the major axis of the ellipses. Based on the theory of simple shear deformation, the axis (**a**) can be determined by measuring β ($\tan 2\beta = 2/\sqrt{3}\varepsilon$ ¹⁰), i.e., the angle between the shear direction (θ) and the axis (**a**). As can be seen, the original grain boundaries (which could be noticed till $\varepsilon = 2.7$) was almost parallel to the (**a**) direction, showing a good agreement with the theory of simple shear deformation.

Approaching to the steady-state condition on the stress-strain curve, near $\varepsilon = 3.6$, it seemed that the spacing between original grain boundaries came close enough so that a mixed microstructure composed of the fragmented initial grains and the newly generated fine grains was observed (**Fig. 3.3e**). Further deformation to higher strains (e.g. **Fig. 3.3f**) did not necessarily raise the fraction of HABs. It should be noted, on the other hand, some low misorientation regions (LMRs) inside which LABs mainly existed could be clearly observed in **Fig. 3.3f**. This could be attributed to strengthening of a special texture, which will be furthermore explained in the section 3.3.2.4 (**Fig. 3.9**).

Inverse pole figure (IPF) maps obtained by EBSD of the selected regions in **Fig.**

3.3 are provided in **Fig. 3.4**, indicating post-peak conditions at $\varepsilon = 1.8$ (**Figs. 3.4a-g**) and $\varepsilon = 2.7$ (**Fig. 3.4h**). In the IPF maps, the black lines show HABs while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. As represented by the stereographic triangle, different colors in the IPF maps indicate the crystallographic orientation of each measured point with respect to the radial direction in torsion. In **Fig. 3.4a**, it can be seen that original grains were split into a numerous number of finer units with different orientations (different colors) by different types of dislocation boundaries (such as cell walls and deformation bands). Misorientation profiles along some directions (A-A', B-B' and C-C') in the IPF map are also shown in **Figs. 3.4b-c**. The profiles along A-A' and B-B' display the misorientation gradients across and along some banded morphologies in the deformed microstructure. Here, inside the original grain including the lines A-A' and B-B' showed a higher grade of fragmentation, particularly across the deformation-induced bands (i.e. the profile along A-A'). On the other hand, some other grains contained smoother misorientation gradients inside, as suggested by the misorientation profile along C-C'. In fact, depending on the orientations of the initial grains, various types of fragmentation could be found in different grains.

Additionally, the initial boundaries were serrated under this medium Z condition. The serration is due to the interaction between initial grain boundaries and connected sub-boundaries formed during deformation, leading to a tension force at the triple junction and subsequent local boundary migration. Some regions in **Fig. 3.4a** outlined by white circles are magnified and shown in **Figs. 3.4.e-g**. As shown by the magnified IPF maps, a fraction of fine grains with rather equiaxed shapes pointed out by red arrows could be observed. These grains could be either adjacent to the original grain boundaries (**Fig. 3.4.e**), along some deformation bands (**Fig. 3.4.f**), or isolated inside the grains (**Fig. 3.4.g**). The formation process of some of these grains (e.g. the ones formed near original grain boundaries) had some similarities to conventional DRX process. However, conventional DRX and related phenomena (such as bulging of initial boundaries into deformed grains) have been expected to happen under lower Z (higher temperature) conditions and at much lower strains.

A large fraction of newly formed equiaxed grains were surrounded by HABs, indicating that the boundaries surrounding equiaxed grains were progressively increasing their misorientation during deformation. It seemed that the grain subdivision and continuous fragmentation could essentially contribute to the

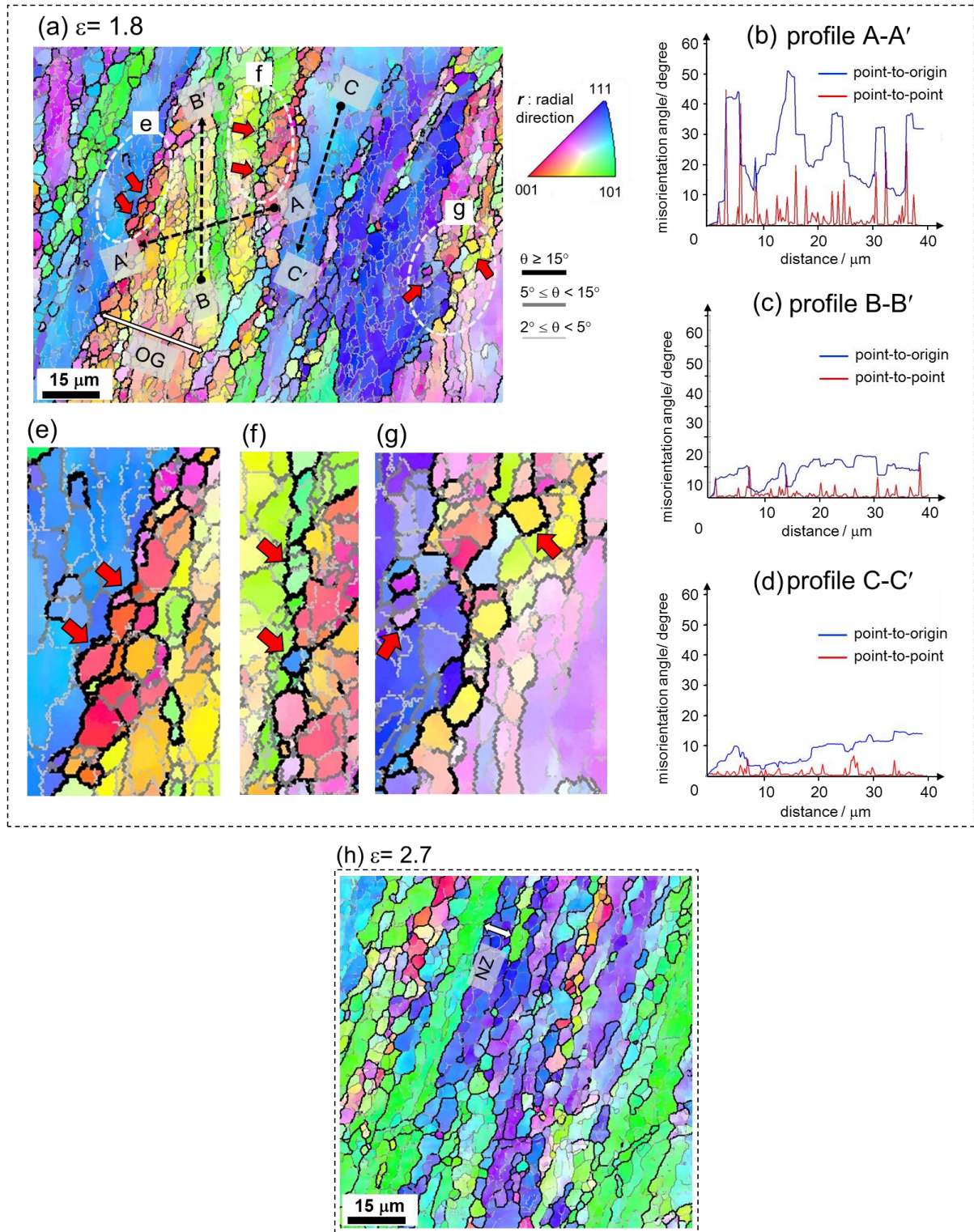


Figure 3.4. EBSD inverse pole figure (IPF) maps of some regions selected in Fig. 3.3. The IF steel specimen was deformed under the condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ (650 $^\circ\text{C}$ / 0.01 s^{-1}). (a-g) $\varepsilon = 1.8$ and (h) $\varepsilon = 2.7$. In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. Red arrows point out newly formed equiaxed grains, OG the original grain, NZ the narrow zone. Misorientation profiles along the lines A-A', B-B', and C-C' marked in (a) are also indicated in (b), (c) and (d).

generation of those equiaxed grains. Such a process could be accelerated near initial grain boundaries or deformation bands, where different slip patterns (i.e. different combinations and amounts of operated slip systems) resulted in different rotation fields. In addition, some boundary arrangements with short-range migration of boundaries could help them to get more equiaxed morphologies.

Such a microstructure evolution progressed furthermore at the strain of 2.7 (**Fig. 3.4h**), where the trace of stretched (original) grains was still visible. Due to the nature of simple shear deformation in torsion, spacing between original boundaries (narrow zone) became thinner and finally reached the sizes comparable to the size of subgrains. Therefore, at a certain stage, it was possible that some boundary interactions led to the division of elongated original grains into several finer grains, which was similar to the phenomenon referred as GDRX (geometrical dynamic recrystallization) reported in some literatures [11,12](#).

- 3.3.2.2. $Z \sim 10^{22} \text{ s}^{-1}$ (650 °C / 1.0 s^{-1} and 550 °C/ 0.01 s^{-1})

The formation of new grain structures was also investigated under other medium Z conditions; that is, $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ (650 °C / 1.0 s^{-1}) and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$ (550 °C/ 0.01 s^{-1}). The microstructure evolution under these conditions, whose Z values were rather increased from that in the former section, is shown in **Fig. 3.5**.

Figure 3.5a shows stress-strain curves under the deformation conditions of $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$. The stress-strain curve under the condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ discussed in the former section is also indicated in a thin line. Similar to the result shown in the former section, at the early stage ($\epsilon = 1.8$) of deformation, the original grains, of which boundaries were shown by white arrows, were subdivided by deformation-induced boundaries, particularly in banded morphologies (**Figs. 3.5b** and **3.5e**). However, comparing to the previous condition shown in **Fig. 3.3c** (with lower Z), the number of generated HABs per original grain significantly increased at the same strain of 1.8. In other words, the deformed grains were highly misoriented in **Figs. 3.5b** and **3.5e**. Applying further strains (**Figs. 3.5c,d** and **3.5f,g**) resulted in the appearance of the equiaxed grains (marked by red arrows) that eventually occupied most of the entire matrix. The regions inside the rectangular frames marked in **Figs. 3.5b,c,d** and **3.5e,f** are selected for a detailed explanation on microstructures.

Figure 3.6 represents results of some microstructural examinations in the local regions indicated in **Fig.3.5**, under the two kinds of medium Z conditions. The IPF

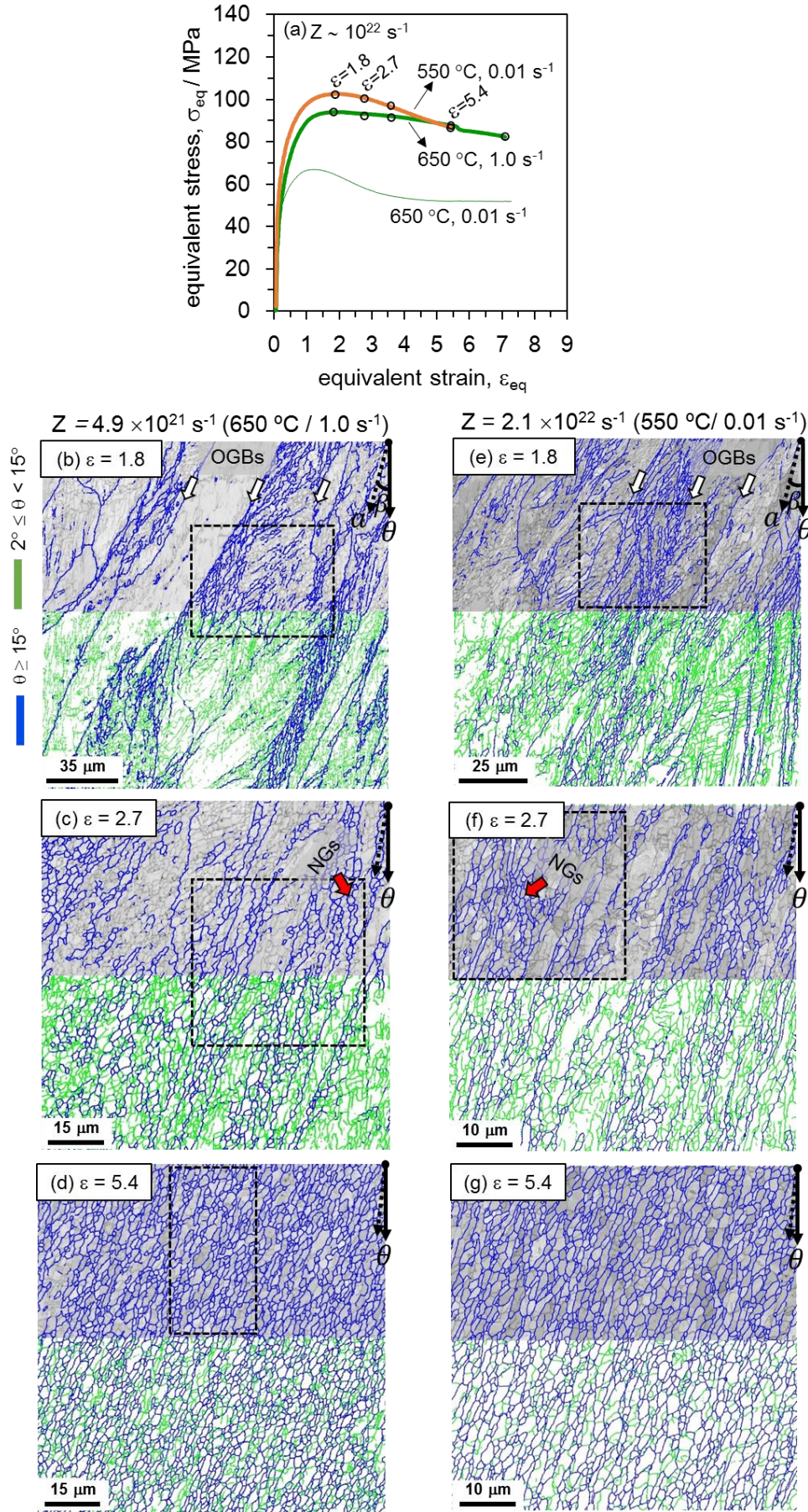


Figure 3.5. (a) Flow stress-strain curves under the deformation conditions of $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ ($650^\circ\text{C} / 1.0 \text{ s}^{-1}$) and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$ ($550^\circ\text{C} / 0.01 \text{ s}^{-1}$). The curve under the condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ ($650^\circ\text{C} / 0.01 \text{ s}^{-1}$) discussed in Fig. 3.3. is also indicated in a thin line. Microstructure observations were conducted at different strains indicated by the open circle. Representative torsion-deformed microstructures under the (b)-(d) $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ and (e)-(g) $4.9 \times 10^{19} \text{ s}^{-1}$ conditions at the strains of (b, e) $\epsilon = 1.8$, (c, f) $\epsilon = 2.7$, and (d, g) $\epsilon = 5.4$. In each EBSD map (b-g), the upper half shows HABs (with misorientation $\theta \geq 15^\circ$) drawn by blue lines superimposed on the image quality (IQ) map, while the lower half includes LABs ($2^\circ \leq \theta < 15^\circ$) drawn by green lines in addition to HABs. Moreover, β indicates the angle between the shear direction (θ) and the major axis (a) of original grains after the corresponding shear deformation. Abbreviations: OGBs as original grain boundaries (pointed out by white arrows in (b) and (e)). NGs as new grains (red arrows in (c) and (f)).

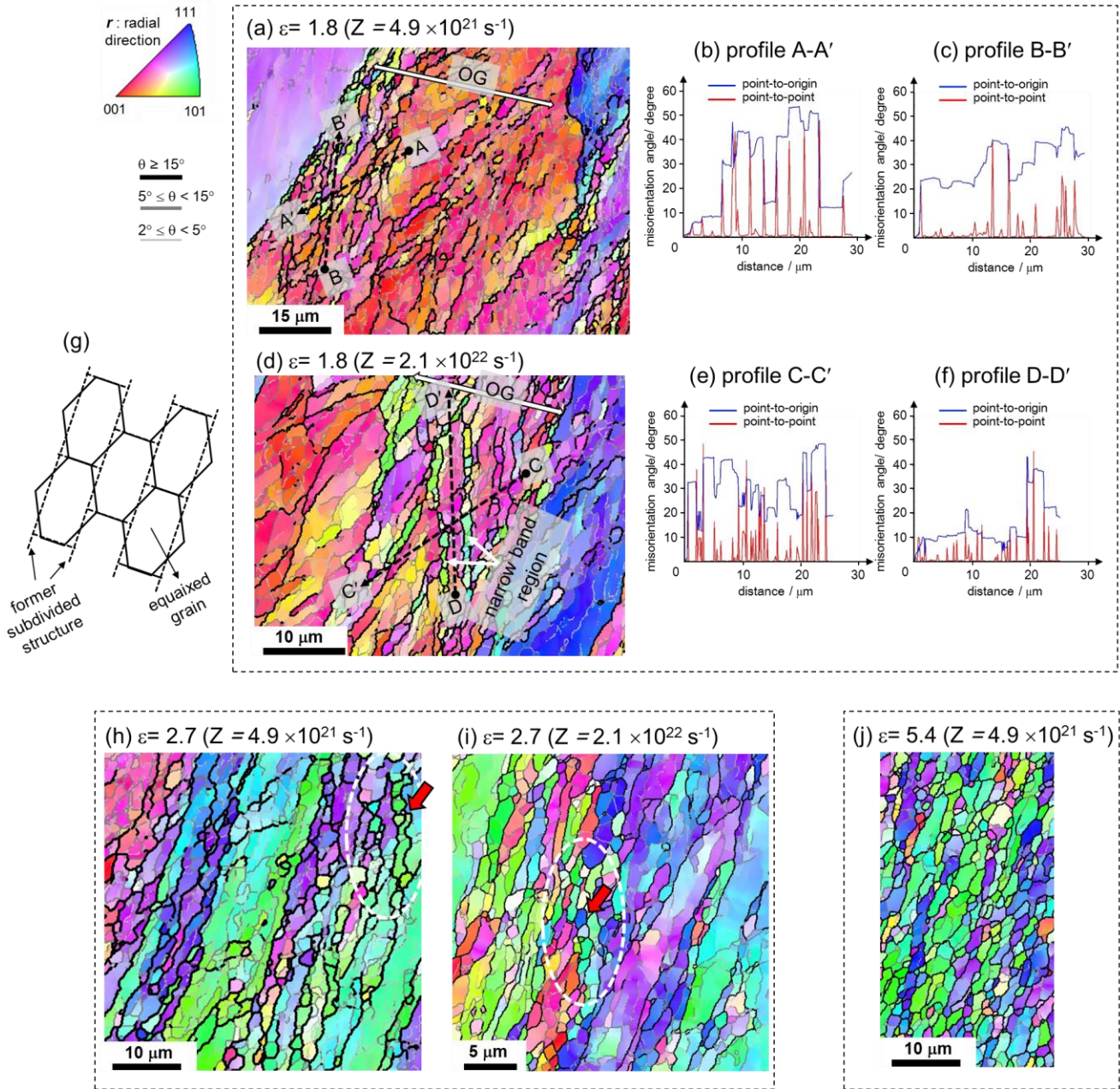


Figure 3.6. EBSD inverse pole figure (IPF) maps of some regions selected in Fig. 3.5. The IF steel specimen deformed the conditions of $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$. (a, d) $\varepsilon = 1.8$, (h, i) $\varepsilon = 2.7$, and (j) $\varepsilon = 5.4$ under the conditions of (a, h, j) $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ and (d, i) $Z = 2.1 \times 10^{22} \text{ s}^{-1}$. In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. Red arrows point out newly formed equiaxed grains, OG the original grain. Misorientation profiles along the lines A-A', B-B', C-C', and D-D' marked in (a) and (d) are also indicated (c, d, e, f). (g) A model illustrating the formation of equiaxed grains within subdivided structures such as narrow bands marked in (d).

maps at $\varepsilon = 1.8$ (**Figs. 3.6a, 3.6b**) showed that some original grains were significantly subdivided. Misorientation profiles along the lines A-A' and B-B' (marked in **Fig. 3.6a**)

or C-C' and D-D' (marked in **Fig. 3.6b**) are represented in **Figs. 3.4c-f**. It can be seen that misorientation values were large and they frequently changed within the measured distances. In other words, the misorientation profiles shown in **Figs. 3.4c-f** suggested that the inside of original grains including the lines A-A' and B-B' or C-C' and D-D' (**Figs. 3.6a, 3.6b**) had a high degree of subdivision. An abundant number of HAB segments were generated, mainly in the form of parallel or intersecting bands. Even though initial boundaries were less corrugated under these conditions, it seemed that some equiaxed features were about to emerge within such microstructural heterogeneities (e.g. narrow band regions shown in **Fig. 3.6b**) induced by the higher-Z deformation. As an example, a simple model of microstructure evolution from a subdivided (or banded) structure into equiaxed grains is schematically illustrated in **Fig. 2.6g**. Narrow bands could be split into finer grains by triple junction motion and local boundary migration. Boundary migration and growth could happen in order to reduce the boundary energy, particularly within the highly subdivided regions.

The IPF maps in **Figs. 2.6h** and **2.6i** display the microstructure evolution at $\varepsilon = 2.7$, under the two Z conditions. A noticeable fraction of equiaxed grains surrounded by HABs formed within subdivided regions; e.g., the regions outlined by white circles in **Figs. 3.6h** and **3.6i**. The appearance of several equiaxed grains in row (pointed out by red arrows in **Figs. 3.6h** and **3.6i**) could be a result of such processes explained before by the illustration in **Fig. 2.6g**; i.e., triple junction motion and local boundary migration within narrow bands. Finally, the IPF map shown in **Fig. 3.6j** is a representative microstructure of the IF steel torsion-deformed by a high strain of 5.4, under the medium Z condition. Eventually, the subdivided microstructure with numerous bands and HAB segments was replaced by fine equiaxed grains.

- **3.3.2.3. Boundary evolution**

Figure 3.7a shows HAB fractions (f_{HABs}) evolved in the microstructures as a function of the applied strain under the medium Z conditions explained in the previous sections 3.3.2.1 and 3.3.2.2. For comparison, **Fig. 3.7b** displays f_{HABs} as a function of the Z parameter, which was presented in Chapter 2 (**Fig. 2.10a**). Additionally, changes in length densities of LABs and HABs (l_{LABs} and l_{HABs}) in the microstructures, as well as the fraction of HABs, $f_{HABs} (= l_{HABs} / (l_{LABs} + l_{HABs}))$ are provided in **Figs. 7c-e**. It should be noted that the boundary length could be measured by counting the number of boundary segments in the EBSD boundary maps (e.g. **Fig. 3.3** and **Fig. 3.5**)

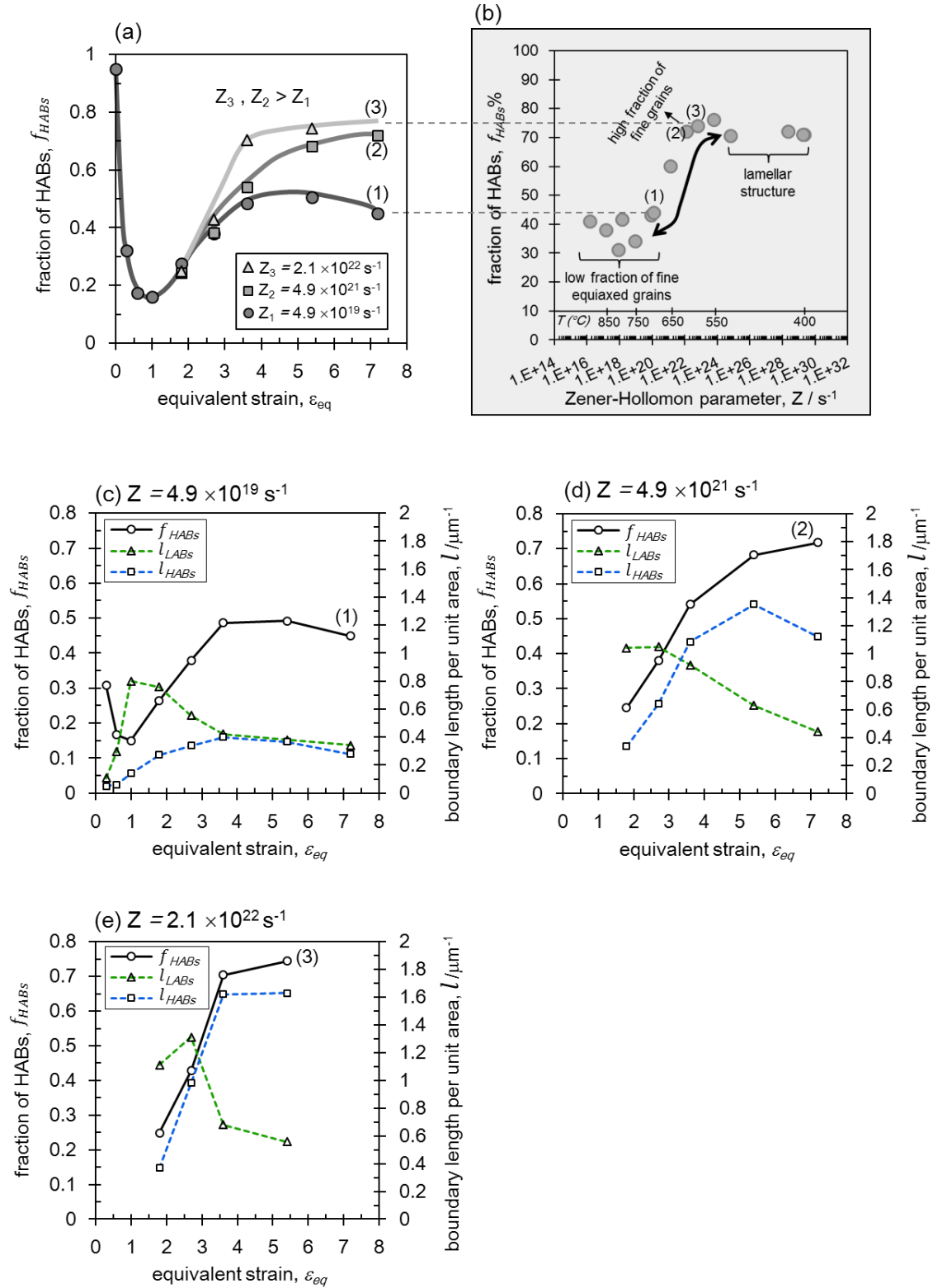


Figure 3.7. (a) Fractions of HABs (f_{HABs}) as a function of equivalent strain applied in the torsion deformation under the medium Z conditions. (b) Fraction of HABs (f_{HABs}) as a function of the Z parameter under a wide range of Z conditions in the present study (Chapter 2, Fig. 2.10a). (c-e) Fractions of HABs (f_{HABs}) together with the density (length per unit area, μm^{-1}) of LABs (l_{LABs}) and HABs (l_{HABs}) under the different medium Z conditions: (c) $Z = 4.9 \times 10^{19} \text{ s}^{-1}$, (d) $Z = 4.9 \times 10^{21}$, and (e) $Z = 2.1 \times 10^{22} \text{ s}^{-1}$.

and knowing the step size in EBSD scanning. Then, the measured boundary length per unit area was considered as the length density.

In **Fig. 3.7a**, it can be seen that f_{HABs} quickly decreases at the beginning of deformation, and starts to increase after an equivalent strain about 1, followed by a saturation at high strains. It is clear that the higher Z conditions led to the higher fractions of HABs (> 70%) in the saturation stage. This result quantitatively explained the microstructure evolution previously shown in **Fig. 3.3** and **Fig. 3.5**, where increasing Z provided more misoriented areas (higher degree of subdivision), and subsequently resulted in the higher fractions of equiaxed grains surrounded by HABs. This can be also the reason why the f_{HABs} - Z plot (**Fig. 3.7b**) showed a noticeable increase in f_{HABs} around the medium Z conditions.

It should be noted that f_{HABs} in the starting material having a fully recrystallized microstructure was about 95%. Therefore, in **Fig. 3.7a**, a sudden drop of f_{HABs} at early stage ($\varepsilon < 1$) was attributed to the decrease of HABs owing to the introduction of deformation microstructures (as is shown in the l_{LABs} diagram, **Fig. 3.7c**). In **Fig. 3.7c** ($Z = 4.9 \times 10^{19} \text{ s}^{-1}$), however, l_{LABs} decreased (and f_{HABs} increased) again at an equivalent strain about 1. This can be generally considered as the result of the evolution of HABs with the progress of grain subdivision. The tendency of boundary evolution under the condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ could be also in the other medium Z conditions; i.e., $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ (**Figs. 3.7d**) and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$ (**Fig. 3.7e**).

Figure 3.8 illustrates distributions of misorientation angles of boundaries at various strains under three different medium- Z conditions, $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ (**Figs. 3.8a-c**), $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ (**Figs. 3.8d-f**), and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$ (**Fig. 3.8g-i**). The green and blue colors used to highlight the graph columns emphasize the distribution ranges of LABs ($2^\circ \leq \theta < 15^\circ$) and HABs ($\theta \geq 15^\circ$), respectively. It can be said that the microstructures at the lower strain ($\varepsilon = 1.8$) had a wide distribution of misorientation angles, where the fraction of LABs was high and the average misorientation value (θ_{avg}) was below 15° . With further straining (e.g. $\varepsilon = 2.7, 5.4$), the distribution of misorientation angles gradually shifted toward the higher values, and the fractions of LABs and HABs dropped and increased, respectively. This process seemed to be accelerated under the higher Z conditions (**Figs. 3.8d-f** and **Figs. 3.8g-i**), where θ_{avg} at $\varepsilon = 5.4$ reached the values of 28.9° and 31.9° under the conditions of $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$, respectively.

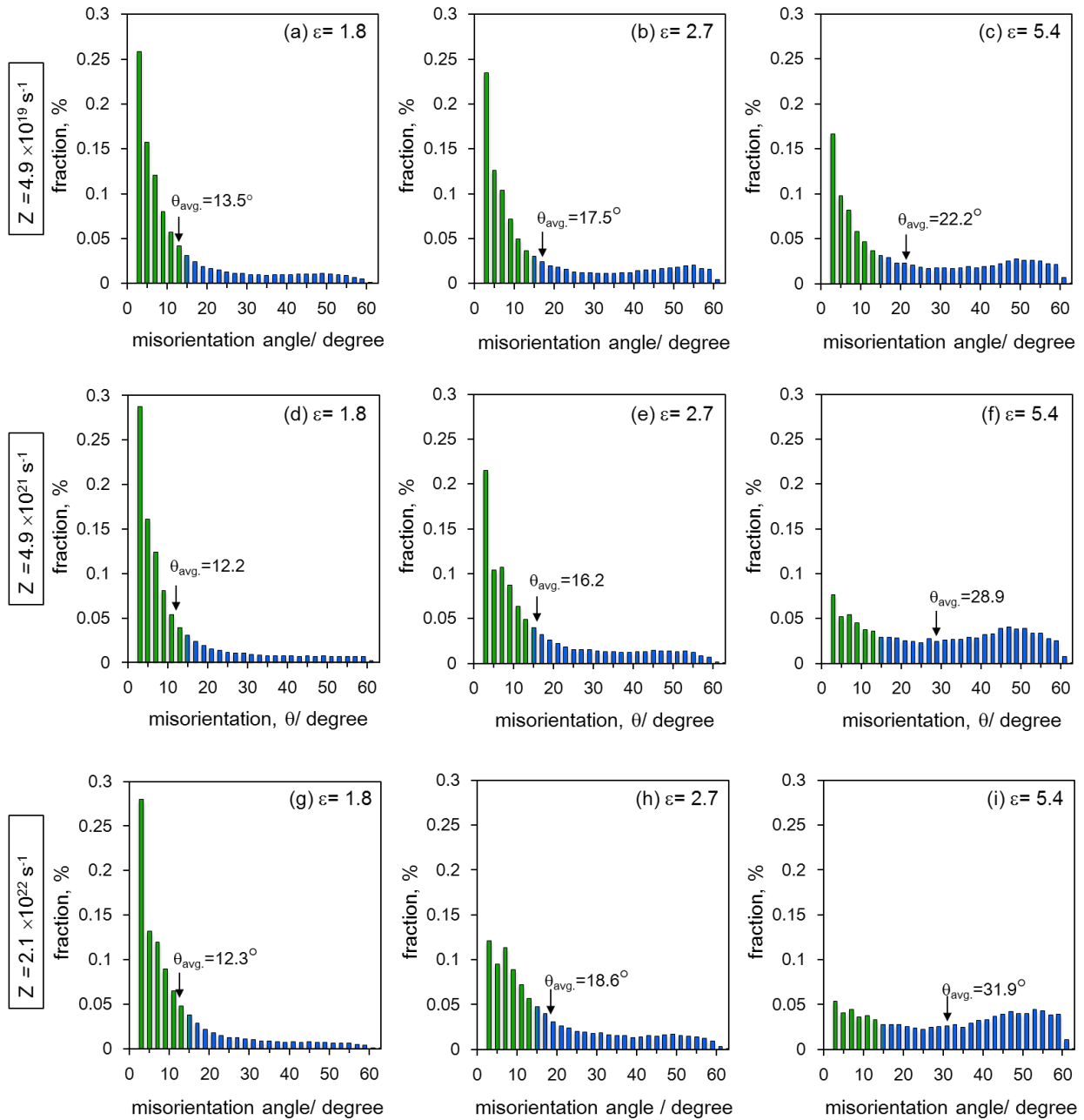


Figure 3.8. Distribution of misorientation angles of boundaries at various strains under three different medium-Z conditions. (a-c) $Z = 4.9 \times 10^{19} \text{ s}^{-1}$, (d-f) $Z = 4.9 \times 10^{21} \text{ s}^{-1}$, and (g-i) $Z = 2.1 \times 10^{22} \text{ s}^{-1}$. The green and blue colors of the graph columns emphasize the distribution ranges of LABs ($2^\circ \leq \theta < 15^\circ$) and HABs ($\theta \geq 15^\circ$), respectively.

• 3.3.2.4. Microtexture evolution

In the previous chapter, it was found that D2 orientation: $\{\bar{1}\bar{1}2\} \langle 111 \rangle$ was the major texture component in the IF steel highly deformed by torsion under medium and low Z

conditions (see **Fig. 2.15d** in Chapter 2). A microtexture analysis was also carried out in this chapter to find out how such a microtexture evolved during the deformation. The results obtained from the EBSD analysis are represented in **Fig. 3.9** and **Fig. 3.10**. The schematic ODF plot ($\varphi_2 = 0^\circ$ and $\varphi_2 = 45^\circ$ sections) showing the positions of typical shear texture components is again provided in **Fig. 3.9a** as a reference.

Figures 3.9b to 3.9d display the ODF plots associated to the specimens deformed under the condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$, corresponding to **Figs. 3.3c** ($\varepsilon = 1.8$), **3.3d** ($\varepsilon = 2.7$), and **3.3f** ($\varepsilon = 5.4$), respectively. With increasing the applied strain, it can be seen that the texture intensity gradually increased, where D2 orientation became the major texture component.

The texture evolution in the torsion-deformed bcc-metals (e.g. IF steel) has been studied by several researchers ^{13–17}. The result in the current study where D2 orientation dominated at high strains was in agreement with the previous reports. It was also discussed in previous literatures ^{13,14,17} that the positive or negative sense of shear determines whether D1 or D2, respectively, was the preferred orientation. However, no simple crystal plasticity model solely based on the crystallographic slip process could accurately predicted such a result: i.e., the dominance of D orientation at the expense of other ideal orientations. Thus, it has been concluded that the texture result is attributed to the formation and growth of the regions with the D2 orientation ^{13,14,17}.

Figures 3.9e-g show corresponding orientation maps of the specimens deformed to different strains ($\varepsilon = 1.8, 2.7$, and 5.4), where the existence of various orientations was highlighted by different colors. These maps demonstrate how the fraction and distribution of orientations change within the microstructure. For example, with increasing the applied strain, regions with orientation close to the D2 component (highlighted in dark-purple) developed at the expense of other orientations (e.g. J1/J2 and E1/E2). Simultaneously, the interval of original grain boundaries became narrower due to the grain elongation and thinning (**Fig. 3.9f**). As a result, the dark-purple regions seemed to eventually coalesce at higher strains (e.g. **Fig. 3.9g**). This also led to the appearance of some large LMRs, whose morphology gave a microstructure inhomogeneity under the lower Z conditions.

Figures 3.10 shows orientation maps of the specimens deformed to different strains ($\varepsilon = 1.8, 2.7$, and 5.4) under the conditions of $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$, whose microstructures were previously shown in **Figs. 3.5**. It can be seen that

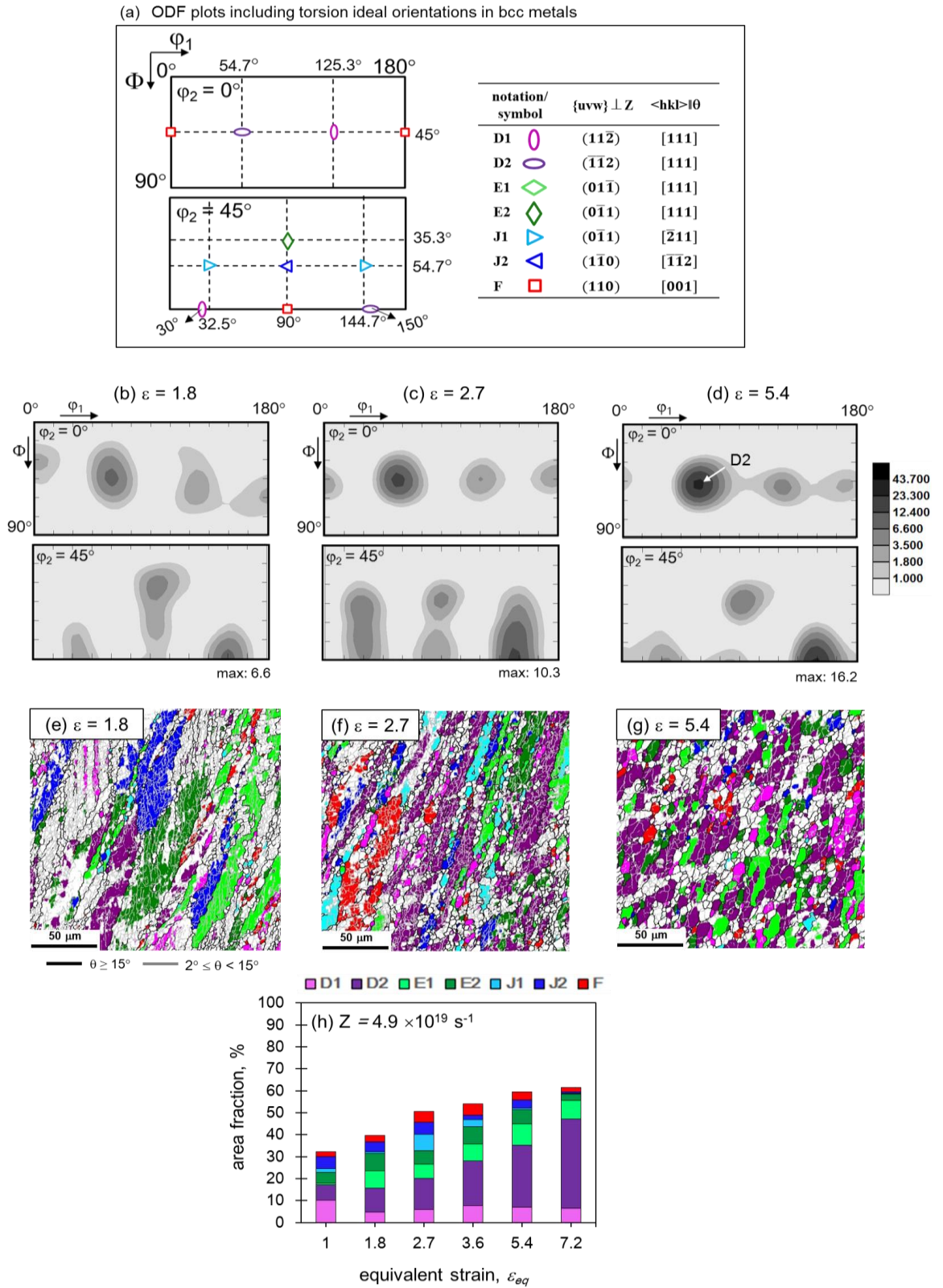


Figure 3.9. (a) Schematic ODF (orientation distribution function) plots at constant ϕ_2 sections (0° , 45°) including the ideal crystallographic orientations in (bcc) torsion [13,25](#) together with the representative angles, notation, and the symbol related to each orientation. (b-d) ODF plots associated to the deformation condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ whose microstructures were previously shown in Figs. 3.3c ($\varepsilon = 1.8$), 3.3d ($\varepsilon = 2.7$), and 3.3f ($\varepsilon = 5.4$). (e-g) Corresponding orientation maps with various highlighting colors illustrating the distribution of different crystallographic orientations: (e) $\varepsilon = 1.8$, (f) $\varepsilon = 2.7$, and (g) $\varepsilon = 5.4$. Here, HABs and LABs are shown by black and gray lines, respectively. (h) Orientation fraction chart as a function of equivalent strain.

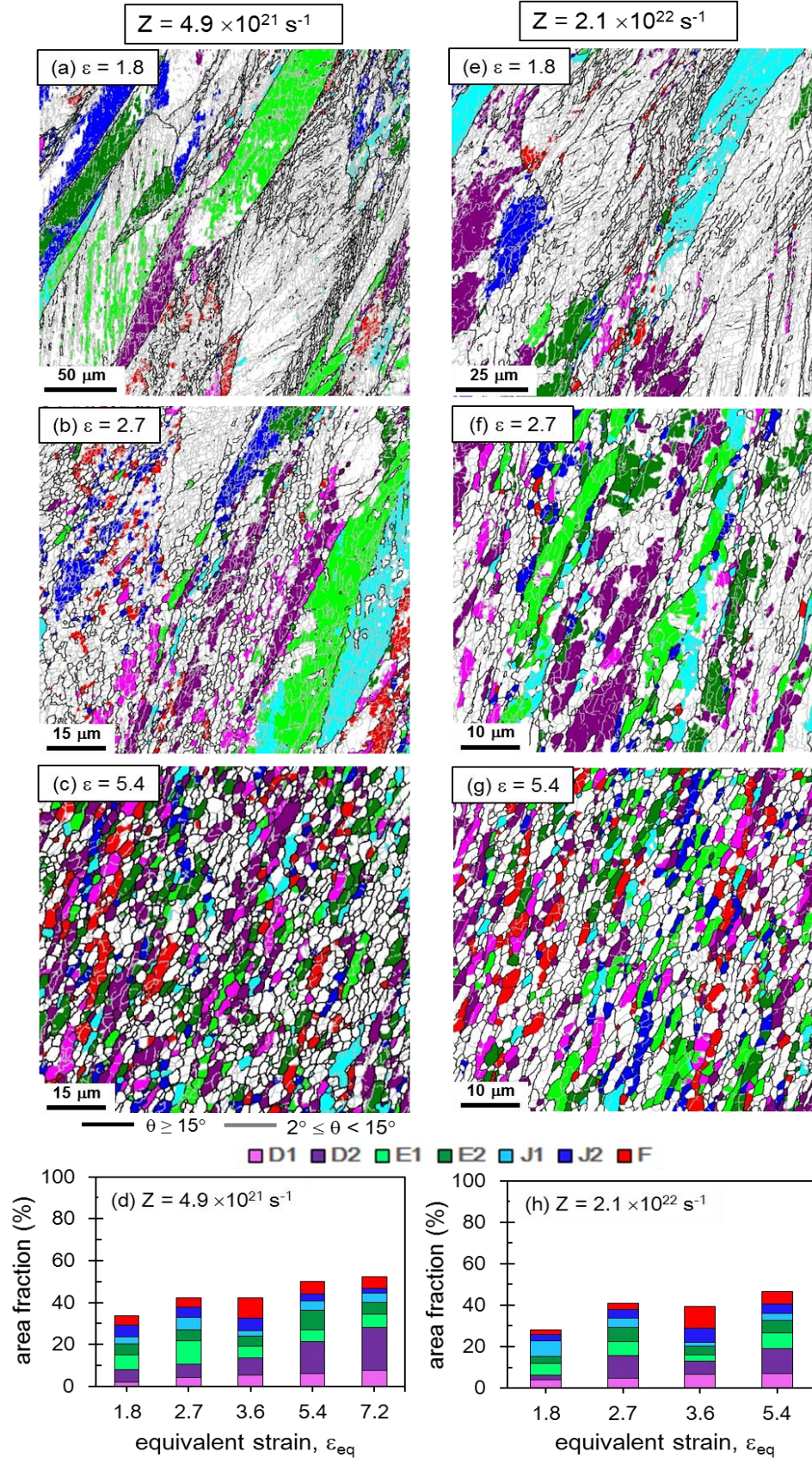


Figure 3.10. (a-c, e-g) Orientation maps (with various highlighting colors illustrating the distribution of different crystallographic orientations) associated to the deformation conditions of (a-c) $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ whose microstructures were previously shown in Figs. 3.5b ($\epsilon = 1.8$), 3.5c ($\epsilon = 2.7$), and 3.5d ($\epsilon = 5.4$) and (e-g) $Z = 2.1 \times 10^{22} \text{ s}^{-1}$ whose microstructures were previously shown in Figs. 3.5e ($\epsilon = 1.8$), 3.5f ($\epsilon = 2.7$), and 3.5g ($\epsilon = 5.4$). Here, HABs and LABs are shown by black and gray lines, respectively. (d, h) Orientation fraction charts as a function of equivalent strain under the deformation conditions of $Z = 4.9 \times 10^{21} \text{ s}^{-1}$ (d) and $Z = 2.1 \times 10^{22} \text{ s}^{-1}$ (h). (Details regarding the Miller indexes of the orientations as provided in Fig. 3.9).

the fraction of D2-oriented regions even at the high strain of 5.4 (**Figs. 3.10c** and **3.10g**) was restricted to some extent, compared to that under the condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ (**Fig. 3.9g**). In other words, more homogeneous distribution of various orientations could be found within the matrix in **Figs. 3.10c** and **3.10g**. The detailed information regarding the fractions of ideal orientations at various strains are provided in **Figs. 3.10e** ($Z = 4.9 \times 10^{21} \text{ s}^{-1}$) and **3.10h** ($Z = 2.1 \times 10^{22} \text{ s}^{-1}$). Although the fraction of D2 orientation increased slightly with increasing the strain, the domination of D2 was suppressed with increasing Z (compared with **Fig. 3.9h**).

D (and E) orientation has the lowest Taylor-factor in torsion (simple shear) among all ideal orientations in the shear texture [13,14,17](#). In brief, its formation was enhanced at higher strains particularly under lower Z conditions. The latter (the effect of Z) indicated that the dominance of D2 orientation should be related to any thermally activated events. For example, in regions with orientation close to D2 having low Taylor factor, dislocation density could be smaller leading to a lower stored energy in those regions. It might be possible that boundaries surrounding D-oriented regions migrated and grew at the expense of other orientations. However, more detailed study is necessary for clarifying the process in future.

3.3.3. Microstructure evolution as a function of strain in deformation under high Z condition: $Z > 10^{23} \text{ s}^{-1}$

As previously explained in Chapter 2, the lamellar boundary structure was the typical microstructure after high strain deformation under high Z conditions. **Figure 3.11** demonstrates the evolution of such microstructures from early deformation stages under the condition of $Z = 5.6 \times 10^{27} \text{ s}^{-1}$ (400 °C/ 0.01 s^{-1}). The flow stress-strain curve in **Fig. 3.11a** showed a work-hardening part reaching a plateau without any stress drop. The deformation was stopped at various strains displayed on the flow stress-strain curve (marked by open circles) in **Fig. 3.11a**. **Figures 3.3b** through **3.3d** demonstrate representative microstructures (EBSD boundary maps) at different strains. With increasing the strain, fraction of deformation induced-HABs (f_{HABs}) gradually increased. At the high strain of 5.4 (**Figs. 3.11d**), the UFG lamellae dominated by HABs ($f_{HABs} > 70\%$) elongated along the shear direction formed. Such a microstructure evolution was nearly the same as that often observed in SPD-processed materials (e.g. see **Fig. 1.6** in Chapter 1), in which the grain subdivision is the dominant mechanism for the microstructure evolution.

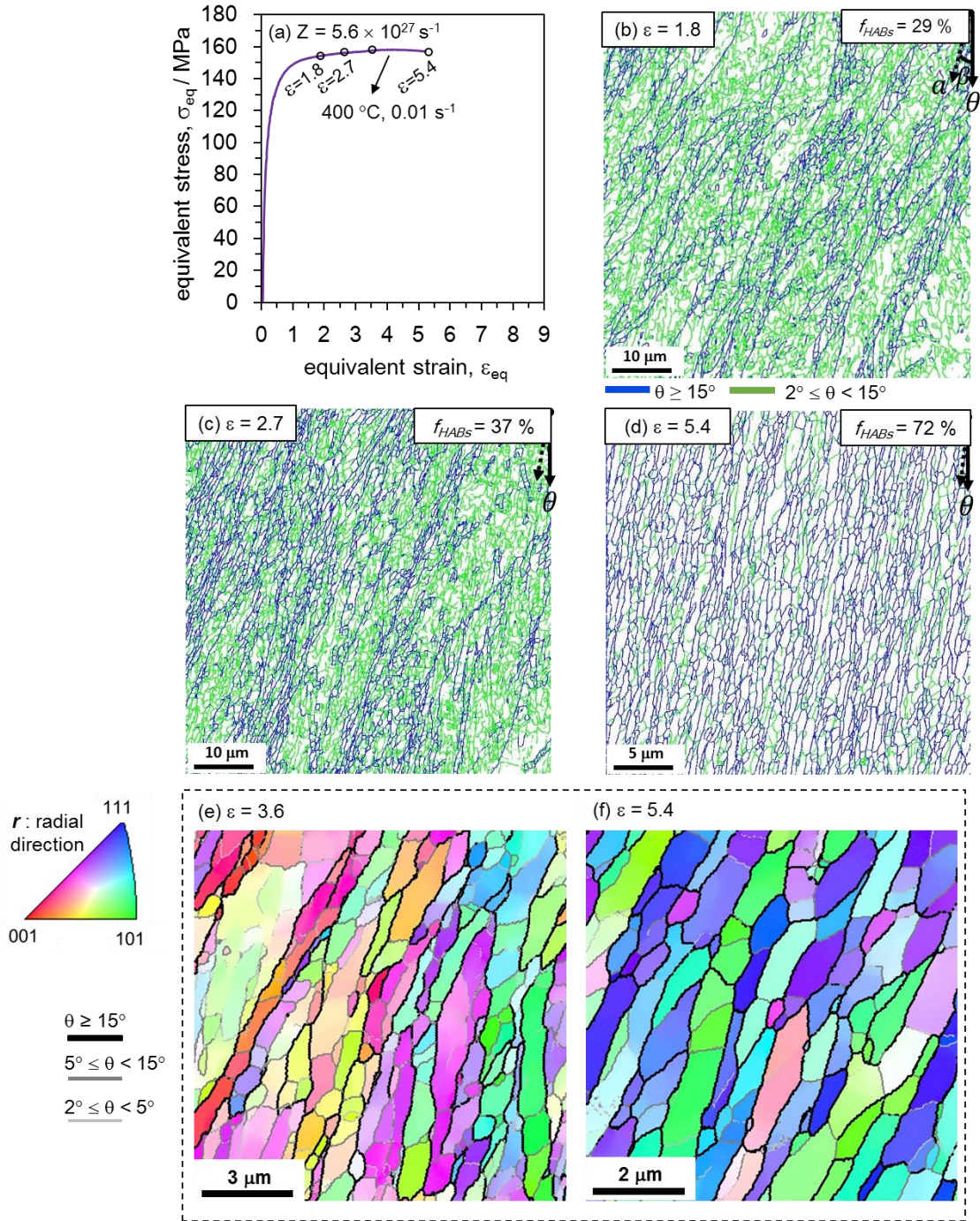


Figure 3.11. (a) Flow stress-strain diagram under the deformation condition of $Z = 5.6 \times 10^{27} \text{ s}^{-1}$ (400 °C/ 0.01 s⁻¹). Microstructure observations were conducted at different strains indicated by the open circle. (b-d) Representative torsion-deformed microstructures (EBSD boundary maps) at the strains of (b) $\varepsilon = 1.8$, (c) $\varepsilon = 2.7$, (d) $\varepsilon = 5.4$. HABs (with misorientation $\theta \geq 15^\circ$) are drawn by blue lines while LABs ($2^\circ \leq \theta < 15^\circ$) drawn by green lines. β indicates the angle between the shear direction (θ) and the major axis (a) of original grains after the corresponding shear deformation. (e, f) EBSD inverse pole figure (IPF) maps at the strains of (e) $\varepsilon = 3.6$ and (f) $\varepsilon = 5.4$. In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines.

Figures 3.11e and 3.11f display the IPF maps at the strain of 3.6 and 5.4, respectively. Applying high strains, the deformation-induced HABs were progressively generated while their spacing decreased. Eventually the HAB-spacing seemed to reach a saturation condition, under which their average distance was comparable to the size of subgrains. Comparing to the medium Z conditions explained in section 3.3.2, the lamellar structure seemed to be more stable even at high strains.

The lamellar boundaries were barely corrugated, even containing many short and interconnecting HABs and LABs. A few equiaxed grains (like those formed under medium Z conditions) could be also found, for example at triple junctions or along the thin lamellae. Some researchers [18,19](#) have tried to explain such a microstructure evolution happening during high strain deformation in terms of Y-junction motions [18](#), stress-driven boundary migration [19](#), etc. Under any scenarios, the size of equiaxed grains was not larger than the spacing of the lamellae, indicating that the grain growth or boundary migration was highly restricted.

3.3.4. Microstructure evolution as a function of strain in deformation under low Z condition: $Z < 10^{19} \text{ s}^{-1}$

The same approach for the microstructural investigations was taken for the deformation under the low Z conditions. Here, another method based on the flow stress analysis is introduced first. Under the low Z condition, it is well-known that as a restoration mechanism DRX may initiate at a critical strain (ϵ_{cr}) before the maximum stress (ϵ_m). Since finding the critical point (ϵ_{cr}) by microstructure examinations is often time-consuming, the flow stress is often used. Such a method has been proposed [20,21](#) based on the fact that the initiation of DRX corresponds to a deviation in the change of the work hardening rate ($\theta = \partial\sigma/\partial\epsilon$). To exactly determine the inflection point in the work hardening rate, a second derivative method [21](#) can be applied by plotting the work hardening rate ($\partial\theta/\partial\sigma$) as a function of the flow stress (σ). The minimum point on the plot (= inflection point) is considered as the critical stress that can be used to determine the corresponding critical strain. The same method was used in this study for the IF steel even though such an analysis is normally used for studying conventional DRX metals.

• 3.3.4.1. $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ (850 °C/ 0.01 s^{-1})

The deformation condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ (850 °C/ 0.01 s^{-1}) was the lowest Z (or σ) condition tried in this study on the IF steel. **Figure 3.12** shows the associated flow stress-strain curve (**Fig. 3.12a**) and the plots for the second derivative method (**Figs. 3.12b, 3.12c**) used to determine the critical strain for DRX under this condition. As a result, the critical strain was estimated as $\varepsilon \approx 0.3$ while the peak strain ≈ 0.5 . The microstructures of the present IF steel at this critical strain are shown by different types of EBSD maps (**Figs. 3.12d, e**): i.e., image quality (IQ) map (**Fig. 3.12d**) and inverse pole figure (IPF) map (**Fig. 3.12e**), of which HABs are illustrated by the blue lines and

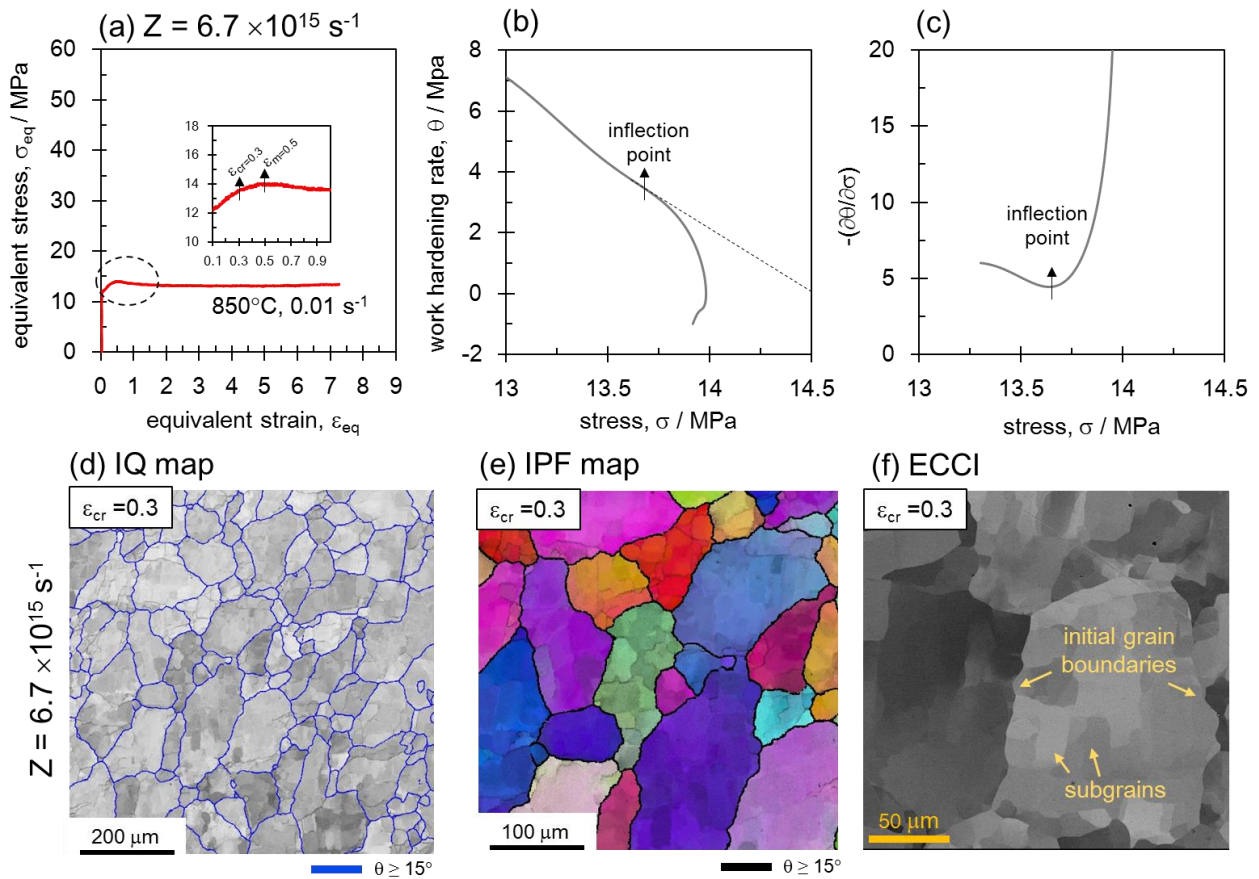


Figure 3.12. (a) Flow stress-strain curve under the deformation condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ (850 °C/ 0.01 s^{-1}), (b) work hardening rate ($\theta = \partial\sigma/\partial\varepsilon$) as a function of stress, and (c) $-(\partial\theta/\partial\sigma)$ as a function of stress to determine the critical strain (ε_{cr}) for the initiation of dynamic restoration mechanisms. EBSD IQ map (d) and IPF map (e) of the IF steel deformed by the critical strain ($\varepsilon_{cr} = 0.3$). HABs ($\theta \geq 15^\circ$) in the EBSD maps are illustrated by blue lines in (d) and black lines in (e). (f) ECC image of the substructure at $\varepsilon_{cr} = 0.3$.

the black lines, respectively. Additionally, electron channeling contrast (ECC) image in **Fig. 3.12f** represents the substructure of the IF steel at the critical strain of 0.3. The EBSD maps (**Figs. 3.12d, e**) barely showed any significant changes showing the appearance of DRX grains or noticeable bulging of initial boundaries. Instead, well-defined equiaxed subgrains in the ECC image (**Fig. 3.12f**) were found to develop inside the initial grains.

Compared to the band-like substructures formed under the medium Z and high Z conditions (e.g. **Fig. 3.2b**), equiaxed subgrains could easily form even at a very low strain (0.3) in **Fig. 3.12f**. This indicates that the recovery events such as dislocation climb and annihilation could happen so efficiently as restoration mechanism to form the subgrains. Local migration of some initial boundaries (or strain-induced boundary migration: SIBM) could be found at this stage, which might be partly responsible for softening. Nonetheless, it was clear that the occurrence of conventional DRX was difficult in microstructures of the easy-recovery ferrite.

Figure 3.13 shows the effect of the increasing strain on the microstructure evolution under the same condition ($Z = 6.7 \times 10^{15} \text{ s}^{-1}$). The deformation was stopped at various strains displayed on the flow stress-strain curve (marked by open circles) in **Fig. 3.13a**. Accordingly, the microstructural changes (**Figure 3.13b-f**) were as follows: original grain boundaries (shown by white arrows) in **Fig. 3.13b** were serrated at $\varepsilon = 0.8$. However, only a few fine grains (pointed out by a red arrow) could be found, even at this post-peak stage. The fine grains mainly formed along original grain boundaries or at triple junctions.

With further deformation by $\varepsilon = 1.3$ (**Fig. 3.13c**) and $\varepsilon = 2.1$ (**Fig. 3.13d**), more new grains could be observed, although their number was not copious. Some regions (outlined by rectangular frames) were selected for the detailed microstructure study which will be discussed later (in **Fig. 3.14**). Some new grains could grow while fragmentation and thinning of original grains occurred simultaneously. Therefore, sometimes it was difficult to distinguish the new grains from the old ones at this stage, because their size differences were not significant. The additional highlighted map inserted in **Fig. 3.13g** (corresponding to $\varepsilon = 2.1$) illustrates the grains in three different categories using their GOS values. In the TSL OIM software, the grain orientation spread or GOS can express the extension of deformation inside a grain. For example, the freshly generated grains with the green highlight in **Fig. 3.13g** had GOS values below 2° . On the other hand, GOS values in the old grains with no highlight in **Fig.**

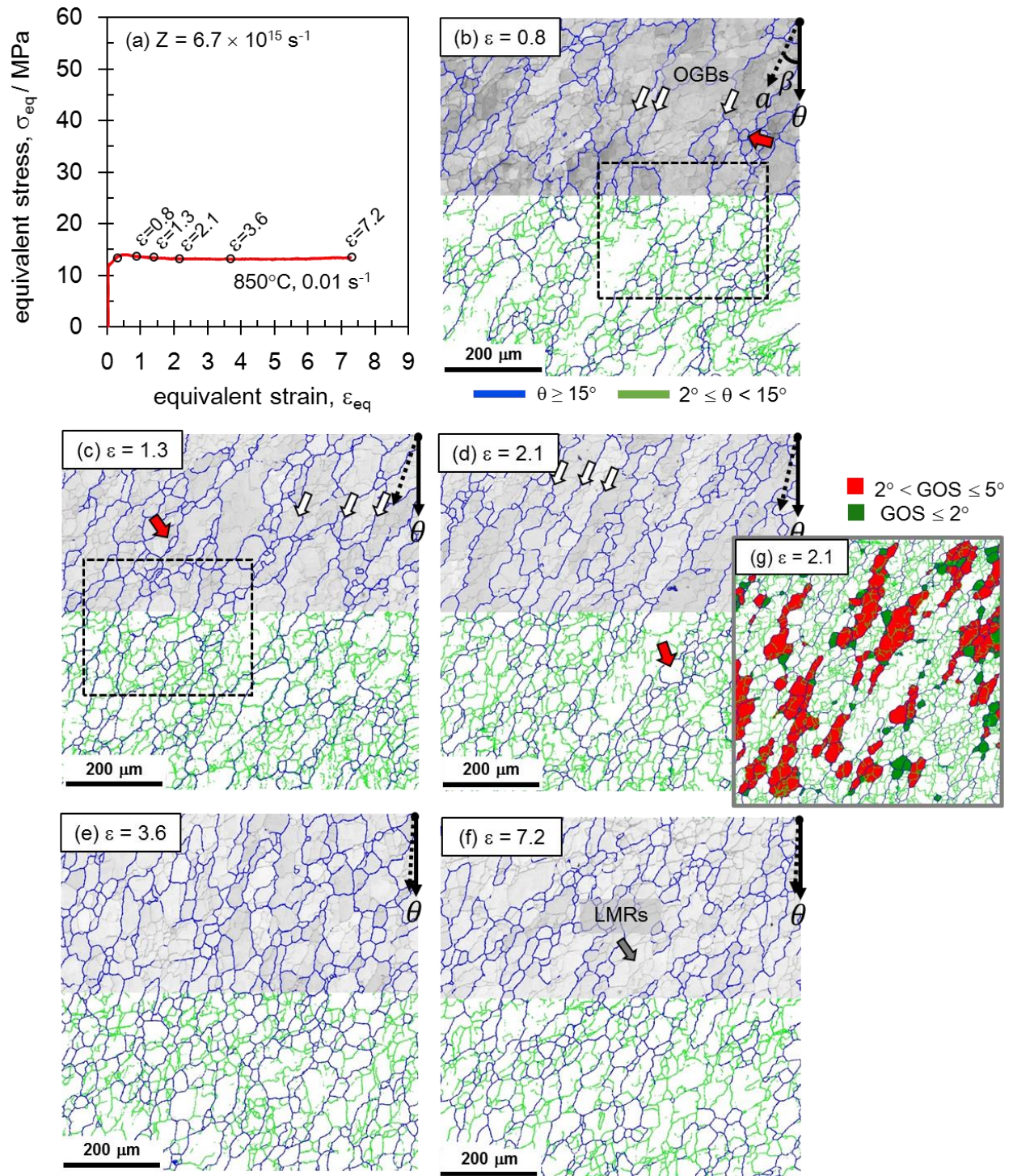


Figure 3.13. (a) Flow stress-strain curves under the deformation condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ ($850^\circ\text{C} / 0.01 \text{ s}^{-1}$). Microstructural observations were conducted at different strains indicated by the open circle. Representative torsion-deformed microstructures at the strains of (b) $\epsilon = 0.8$, (c) $\epsilon = 1.3$, and (d) $\epsilon = 2.1$, (e) $\epsilon = 3.6$, and (f) $\epsilon = 7.2$. In each EBSD map (b-f), the upper half shows HABs (with misorientation $\theta \geq 15^\circ$) drawn by blue lines superimposed on the image quality (IQ) map, while the lower half includes LABs ($2^\circ \leq \theta < 15^\circ$) drawn by green lines in addition to HABs. Moreover, β indicates the angle between the shear direction (θ) and the major axis (a) of original grains after the corresponding shear deformation. Abbreviations: OGBs as original grain boundaries (pointed out by white arrows in (b), (c), and (d)), NGs as new grains (red arrows in (b), (c), and (d)), LMRs as low misorientation regions (gray arrow in (f)). (g) The additional highlighted map at $\epsilon = 2.1$, categorizing grains having different values of GOS (grain orientation spread).

3.13g was generally above 5°. Additionally, there were some grains with the red highlight, in which GOS values were in a range of 2° ~ 5°. The red highlighted grains, of which the sizes were larger than those of freshly generated grains with the green highlight, could be the early-formed new grains which grew to some extent and were deformed again. They could also be some grain fragments separated from the old grains.

It was interesting that at $\varepsilon = 2.1$, the flow stress reached a steady-state. However, old grains could be noticed in the microstructure still (**Figs. 3.13d** and **3.13g**). By further deformation, initial grain boundaries (pointed out by white arrows) became closer to each other and mixed microstructures of fragmented old grains and new equiaxed grains were observed at higher strains (**Figs. 3.13e, 3.13f**). The fraction of HABs in the microstructures at high strains did not increase significantly owing to the formation of LMRs.

It was found that the new grains could not quickly replace the deformed matrix, unlike conventional DRX. Instead, a small fraction of new grains formed and distributed inhomogeneously within the matrix ⁵. This can be considered as a characteristic behavior of the hot-deformed ferrite, where enhanced DRV suppresses DRX to a significant extent, although it does not completely rule out the DRX events under the low Z condition.

Figure 3.14 shows IPF and IQ maps of some regions selected from **Fig. 3.13** in post-peak conditions: i.e., $\varepsilon = 0.8$ (**Figs. 3.13a, 3.13b**) and $\varepsilon = 1.3$ (**Figs. 3.13c, 3.13d**). The microstructure at $\varepsilon = 0.8$ was basically consisted of original (old) grains with serrated boundaries. However, the serrated boundary segments were hardly found to bulge into the deformed grains. Bulging could be considered as an initiation of conventional DRX and probably a necessary step to copiously generate DRX grains at subsequent stages. However, this process seemed to be restricted in the present ferritic matrix of the IF steel and only a few equiaxed grains (pointed out by red arrows) formed at this strain (**Fig. 3.14a**) or the later stage (**Fig. 3.14c**).

Decoration of few DRX grains along initial grain boundaries can be noticed in **Fig. 3.14c**. Subgrain rotation might also contribute to separate those equiaxed grains from parent grains. Such an event could be accelerated in grain boundary affected zones due to enhanced the difference of slip patterns to accommodate the incompatibility of deformation between neighboring grains.

Due to the low-density of newly formed equiaxed grains, a noticeable amount of

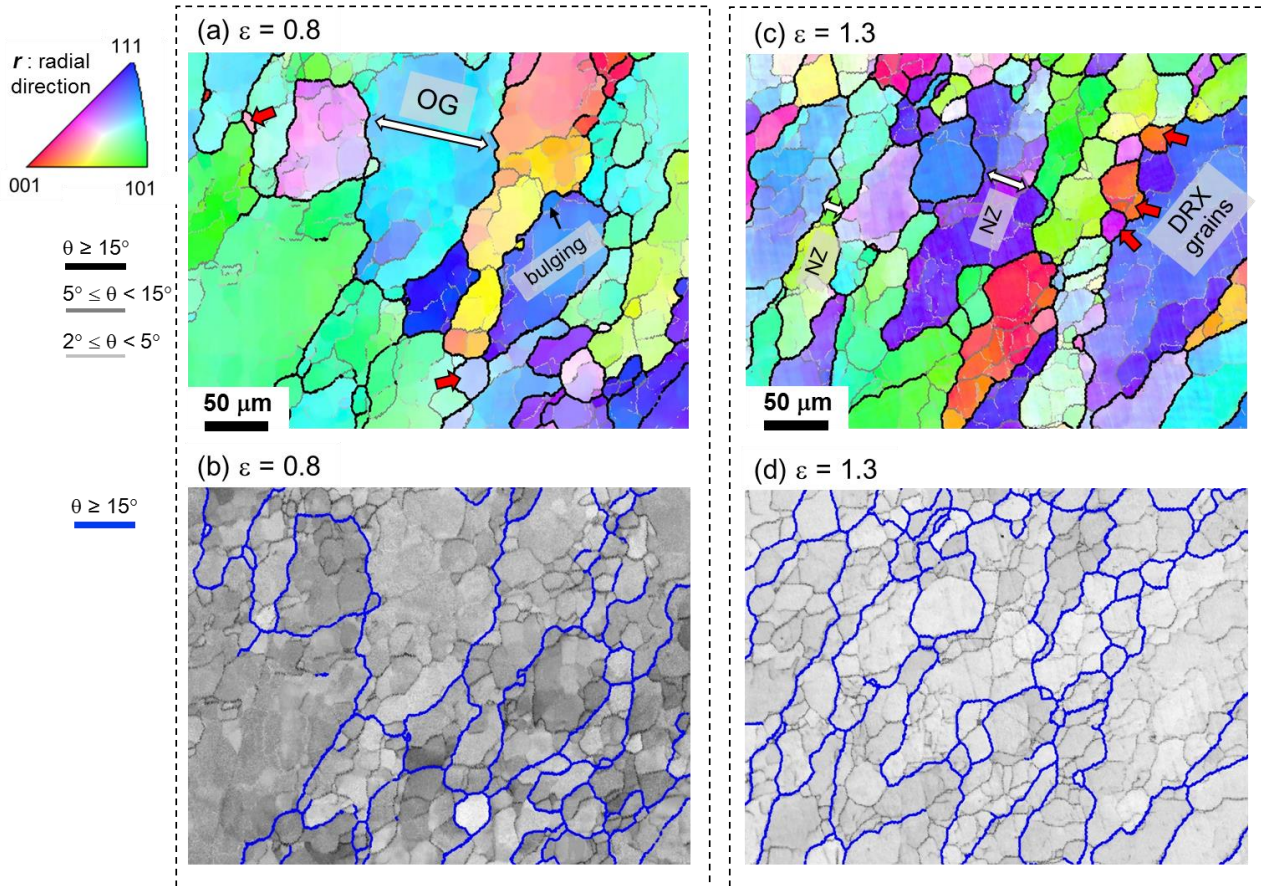


Figure 3.14. (a, c) EBSD inverse pole figure (IPF) maps and (b, d) image quality (IQ) maps of some regions selected in Fig. 3.13. The IF steel specimen was deformed by $Z = 6.7 \times 10^{15} \text{ s}^{-1}$. (a, b) $\varepsilon = 1.8$ and (c, d) $\varepsilon = 2.7$. In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. In the IQ maps, the blue lines show HABs ($\theta \geq 15^\circ$). Red arrows point out newly formed equiaxed grains, OG the original grain, and NZ the narrow zone.

HABs were initial boundaries at $\varepsilon = 1.3$ (Fig. 3.14c) or even higher strains, making elongated and narrow regions remained. Consequently, GDRX could more likely happen under the low Z condition.

Similar to the medium Z conditions explained in the section 3.3.2, the evolution of microstructure and micro-texture was analyzed under the deformation condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$. As an example, Fig. 3.15 represents f_{HABs} as a function of equivalent strain (Fig. 3.15a), together with misorientation distributions at different strains (Fig. 3.15b-d). In Fig. 3.15a, f_{HABs} slightly increased after the initial drop, however it did not increased significantly in following stages due to the low density of newly formed HABs. It should be noted that the main fraction of HABs (l_{HABs}) came from initial

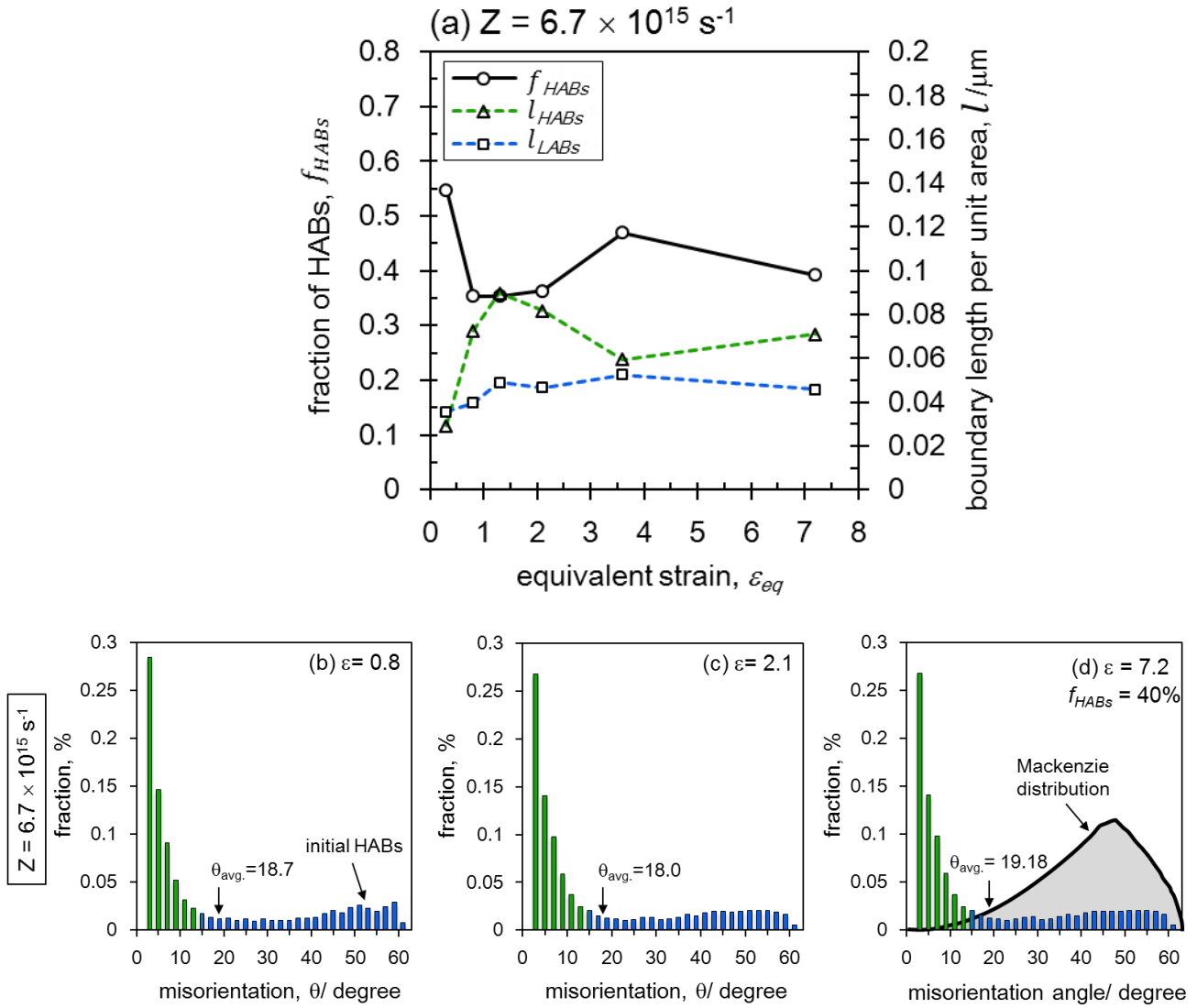


Figure 3.15. (a) Fraction of HABs (f_{HABs}) together with the density (length per unite area, μm^{-1}) of LABs (l_{LABs}) and HABs (l_{HABs}) as a function strain under the deformation condition of $Z = 6.7 \times 10^{15} s^{-1}$. (b-d) Distribution of misorientation angles of boundaries at various strains of (b) $\epsilon = 0.8$, (c) $\epsilon = 2.1$, and (d) $\epsilon = 7.2$. The Mackenzie-type ²³ distribution of misorientation angles of boundaries (for a polycrystalline material with a random texture) is also illustrated in (d).

boundaries. The same trend could be seen in the misorientation distribution charts where, unlike the medium Z conditions, increasing strain did not remarkably shift the average misorientation towards higher values.

It should be mentioned that in some literatures ^{2,22}, misorientation profiles similar to the Mackenzie-type distribution ²³ (for a polycrystalline material with a random texture)

have been reported in low- Z deformed (hot-deformed) microstructures. This was often accompanied with a large f_{HABs} estimated around 70% ~ 80%. These results were claimed to be caused by the occurrence of typical DRX. However, this was not the case in the present study. **Figure 3.15d** showed a smooth distribution of boundary misorientation with a low average value below 20° (19.18°) and a small fraction of HABs ($f_{HABs} = 40\%$).

Figure 3.16 demonstrates local orientation maps of the IF steel specimens torsion-deformed under the condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$, by different strains of **Figs. 3.13d** ($\varepsilon = 2.1$), **3.13e** ($\varepsilon = 3.6$), and **3.13f** ($\varepsilon = 7.2$). Some typical orientation components are expressed in different colors in the microstructures. Under this low Z condition, the orientation maps once again verified the dominance of the D2 orientation component having a low Taylor factor, which was different from the conventional DRX metals where initiation and propagation of DRX grains were considered to randomize the texture or to lead to textures different from as-deformed ones.

• 3.3.4.2. $Z \sim 10^{17} \text{ s}^{-1}$ ($850^\circ\text{C}/1.0 \text{ s}^{-1}$ and $750^\circ\text{C}/0.01 \text{ s}^{-1}$)

In this section, other low- Z conditions are also discussed, whose Z values were slightly higher than $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ explained in the previous section. **Figure 3.17** demonstrates flow stress-strain curves (**3.17a**) and some microstructural examples

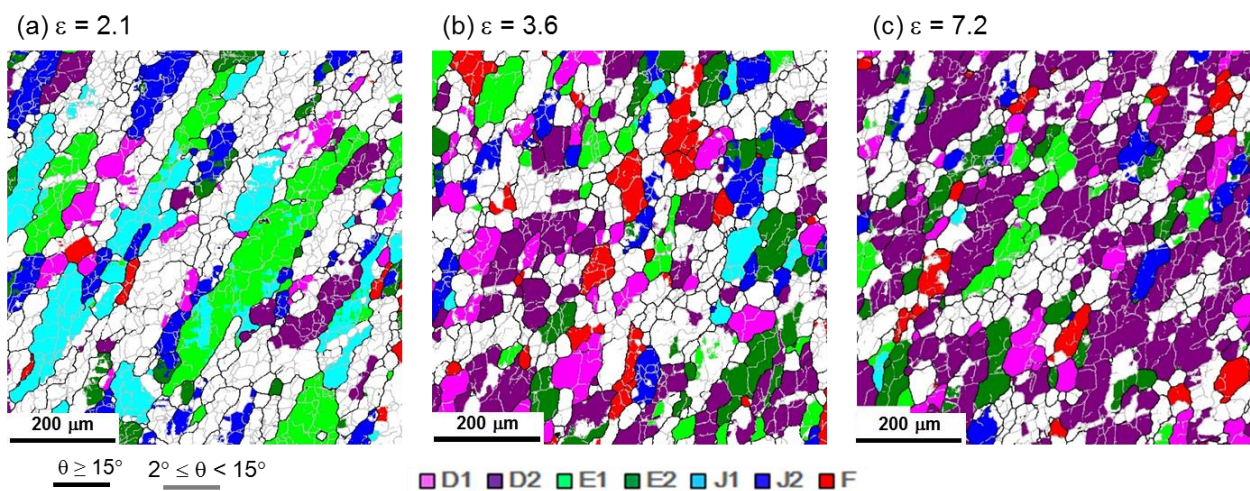


Figure 3.16. (a-c) EBSD orientation maps (with various highlighting colors illustrating the distribution of different crystallographic orientations) associated to the deformation conditions of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$, whose microstructures were previously shown in Figs. 3.13d ($\varepsilon = 2.1$), 3.13e ($\varepsilon = 3.6$), and 3.13f ($\varepsilon = 7.2$). Here, HABs and LABs are shown by black and gray lines, respectively (details regarding the Miller indexes of the orientations provided in Fig. 3.9).

under the deformation conditions of $Z = 6.7 \times 10^{17} \text{ s}^{-1}$: 850 °C/ 1.0 s^{-1} (**Figs. 3.17b-d**) and $Z = 3.7 \times 10^{17} \text{ s}^{-1}$: 750 °C/ 0.01 s^{-1} (**Figs. 3.17e-g**). The stress-strain curve under the condition of $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ discussed in the former section is also indicated in a thin line (**3.17a**).

In metals showing conventional DRX, a noticeable fraction of DRX grains are normally expected around the peak strain. In the easy-recovery ferrite of the present IF steel, however, the microstructures at strains around 2 (**Figs. 3.17b, 3.17e**) were essentially characterized by the elongated initial (old) grains of which boundaries are shown by white arrows, plus a low density of fine equiaxed grains (shown by red arrows) and HAB segments scattering within the matrix. It should be noted that the flow stress-strain curves (**Fig. 3.17a**) were about to reach steady-state conditions at strains around 2. Even the highly deformed ($\varepsilon = 7.2$) ferrite (**Figs. 3.17c, 3.17f**) contained only a small fraction of fine equiaxed grains heterogeneously distributed. As discussed before, the trace of initial grains could not be recognized at this high-strain stage (**Figs. 3.17c, 3.17f**) since some microstructural events such as bulging, triple-junction motion (and GDRX) could happen earlier.

Figures 3.17d and 3.17g display IPF maps at early stages of the deformation ($\varepsilon \sim 2$), . Again typical DRX structures were not observed under these two low- Z conditions. Compared to the condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ described in the previous section (**Fig. 3.14**), the old grains were more fragmented, as expected to happen with increasing Z slightly. Some dislocation boundaries with misorientation around $5^\circ \sim 15^\circ$ (drawn by thick gray lines) or HAB segments were scattered within the matrix. However, their density was not still comparable to that observed under the medium Z conditions (see **Figs. 3.4a, 3.6a, and 3.6d**), where initial grains were highly subdivided by deformation induced HABs.

In **Figs 3.17d and 3.17g**, a low density of new equiaxed grains surrounded by HABs (shown by red arrows) could be found again. They partly formed near serrated boundaries, and their formation is similar to those under the condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ in the previous section. There are also some new equiaxed grains isolated inside the old grains. Formation (or nucleation) of the newly formed grains is still an argumentative issue. It is uncertain whether they have a separate nucleation stage (as is discussed by the concept of conventional DRX) or they basically form owing to the progress of grain subdivision inside the deformed grains. More detailed study is necessary in future.

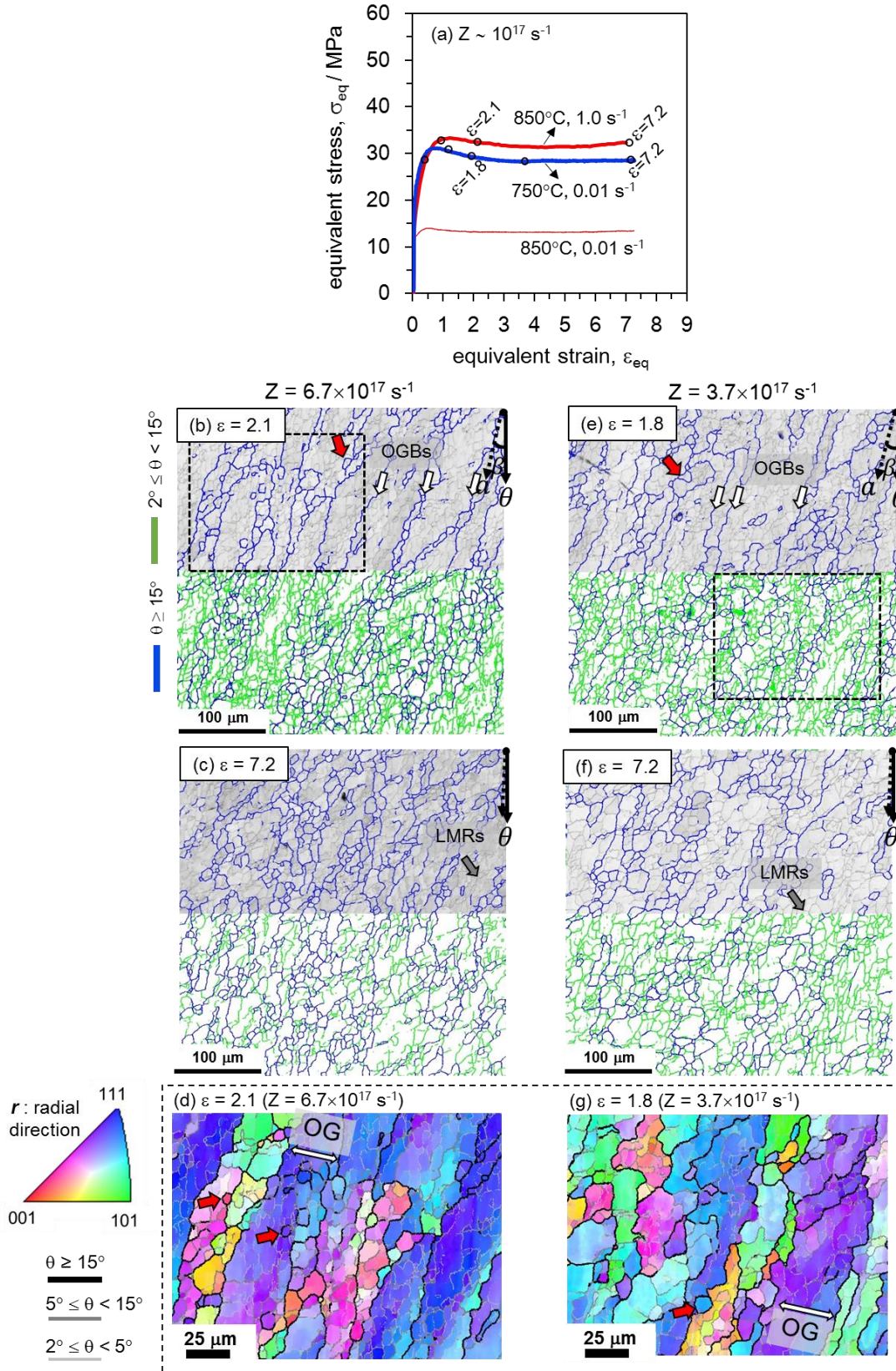


Figure 3.17. (a) Flow stress-strain curves under the deformation condition of $Z = 6.7 \times 10^{17} \text{ s}^{-1}$ ($850^\circ\text{C}/1.0 \text{ s}^{-1}$) and $Z = 3.7 \times 10^{17} \text{ s}^{-1}$ ($750^\circ\text{C}/0.01 \text{ s}^{-1}$). The curve under the condition of $Z = 6.7 \times 10^{15} \text{ s}^{-1}$ ($650^\circ\text{C}/0.01 \text{ s}^{-1}$) discussed in Fig. 3.13, is also indicated in thin line. Microstructure observations were conducted at different strains (open circle). Representative microstructures under the conditions of (b)-(c) $Z = 6.7 \times 10^{17} \text{ s}^{-1}$ and (e)-(g) $Z = 3.7 \times 10^{17} \text{ s}^{-1}$ at the strains of (b, e) $\epsilon \sim 2$ and (c, f) $\epsilon = 7.2$. In each EBSD boundary map (b-c, e-f), the upper half shows HABs (with misorientation $\theta \geq 15^\circ$) drawn by blue lines superimposed on the IQ map, while the lower half includes LABs ($2^\circ \leq \theta < 15^\circ$) drawn by green lines in addition to HABs. Moreover, β indicates the angle between the shear direction (θ) and the major axis (a) of original grains after the corresponding shear deformation. (d, g) IPF maps of some regions selected in (b) and (e). In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. Abbreviations: OGBs as original grain boundaries (pointed out by white arrows in (b) and (e)), NGs as new grains (red arrows in (b) and (e)), LMRs as low misorientation regions (gray arrows in (d) and (g)).

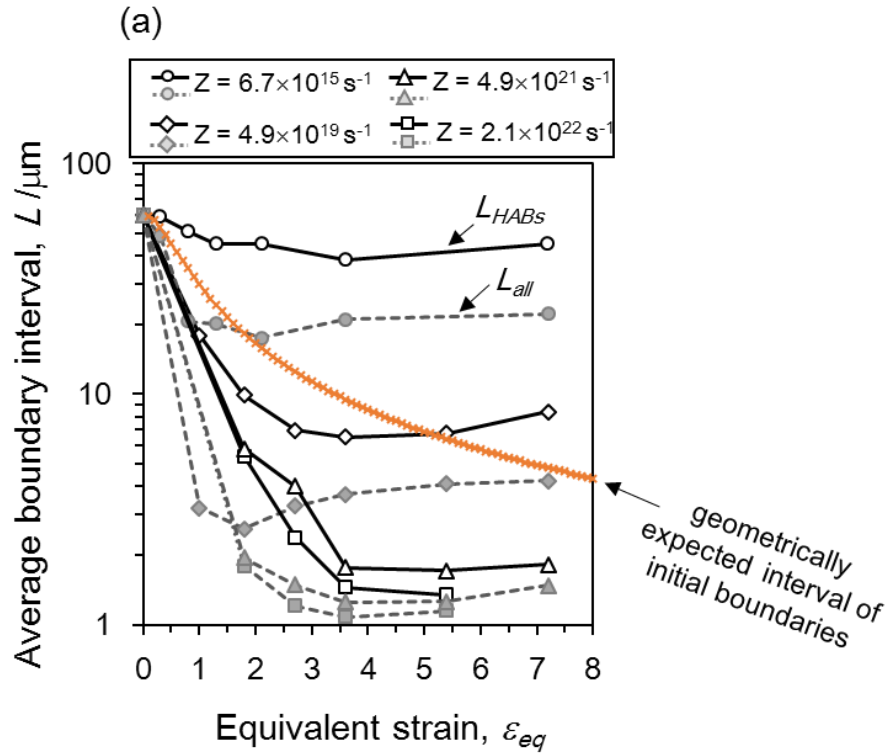
3.3.5. Grain refinement vs. coarsening

Fig. 3.18a shows average intervals between neighboring HABs ($L_{HABs} : \theta \geq 15^\circ$) or between all boundaries ($L_{all} : \theta \geq 2^\circ$) plotted as a function of the equivalent strain under some Z conditions. The boundary intervals firstly decreased, mainly due to the development of many deformation-induced boundaries. This was accelerated under the higher Z conditions. However, the boundary intervals eventually approached the apparent saturation state where microstructure coarsening events cancelled out grain refinement processes. The strain required to reach the steady boundary spacing clearly increased with increasing Z .

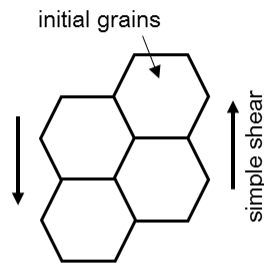
The curve indicated by the cross marks in **Fig. 3.18a** illustrates the geometrically expected interval of initial boundaries at different strains, which was calculated under the assumption that initial grains are deformed in the same way as the global shape of the specimen. **Figures 3.18b** and **3.18c** schematically illustrate the grain structures before and after a high amount of deformation, respectively. Even without any microstructural reactions, the geometrical refinement occurs due to the change in the grain shape. As shown by the dashed lines in **Fig. 3.18c**, the initial grains must become elongated and thinner. It is considered that the actual boundary interval should be smaller than the geometrically expected interval of initial boundaries as a result of extra refinement of microstructures by grain subdivision, DRX and so on. However, it was interestingly found that the actual boundary interval under the condition of $Z = 6.7 \times 10^{16} \text{ s}^{-1}$ and $Z = 4.9 \times 10^{19} \text{ s}^{-1}$ (**Fig. 3.18a**), became larger than the geometrically expected one at high strains. Even under the other conditions with of higher Z , the actual boundary interval saturated at high strains and was about to reach the geometrically expected one. This observation is simply shown by the illustration in **Fig. 3.18b**, where the size of new grains at a high strain is larger than the geometrically expected interval of initial boundaries. This corresponds with the so-called grain size saturation often observed in severe plastic deformation processes ²⁴. The result indicates that certain microstructure coarsening events happen during high-strain deformations.

As a matter of fact, the diagram shown in **Fig. 3.18** can be used as a simple indicator of the coarsening effect. It should be noted that it is almost impossible to inspect this effect solely by examining dynamically evolved microstructures, because continuous deformation of original and newly formed grains masks the effect of boundary migration. However, **Fig. 3.18** can simply suggest a migration ability of

boundaries particularly under the lower Z conditions, where coarsening may balance with microstructural and geometrical refinement.



(b) Before deformation



(c) After deformation to a high strain

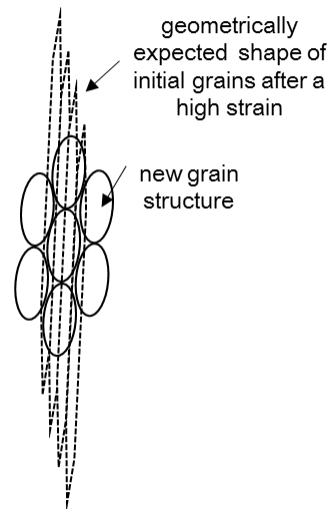


Figure 3.18. (a) Average Interval (spacing) of HABs (L_{HABs}) with misorientation, $\theta \geq 15^\circ$ and all boundaries (L_{all}) with $\theta > 2^\circ$ as a function of strain, under some Z conditions. The geometrically expended interval of initial grain boundaries¹⁰ during a simple shear deformation is shown as orange cross marks. (b, c) Schematic illustration demonstrating initial grains (a) and new grain structure (c) after a high strain deformation, respectively. The geometrically expected shape of the initial grains due to the simple shear deformation is illustrated by dashed lines in (c).

3.4. Discussion

In Chapter 2, the effect of the Z parameter on the formation of fine or ultrafine grains in the IF steel highly (torsion) deformed was studied. Based on the $\sigma - D$ plot (**Fig. 2.13**), three regions or deformation conditions were distinguished; i.e., high Z , medium or transition Z , and low Z conditions. In this Chapter, the effect of increasing strain on the evolution of new grain structures under high Z , medium Z , and low Z conditions was investigated. According to the results explained in this chapter as well as those in Chapter 2, the evolution of new grain structures could be categorized in three different types for an easier discussion.

High Z condition is one extreme case, where the lamellar structures with $f_{HABs} > 70\%$ and boundary spacing $< 1 \mu\text{m}$ developed. The microstructure evolution at different strains (**Fig. 3.11**) indicated that the formation of new ultrafine structures was basically governed by the typical grain-subdivision events without large-scale boundary migration. As explained in the Chapter 2, N in the relation, $\sigma = D^{-N}$, took a value around $1 \sim 1.15$ in this grain subdivision region.

Low Z (with N : $0.78 \sim 0.88$) is the other extreme case, in which grain growth was dominant. It was interestingly found that under the low Z conditions, only a small fraction of equiaxed grains surrounded by HABs ($f_{HABs} < 45\%$) distributed heterogeneously within the matrix. The remains of the microstructures were dominated by low misorientation regions (LMRs). The microstructure examination at low and moderate strains (**Figs. 3.12, 3.14**) proved that typical DRX events, e.g. frequent bulging or copious formation of DRX grains decorating along prior grain boundaries, were greatly restricted due to the enhanced DRV in bcc ferrite of the IF steel which reduced the driving force for recrystallization, and is therefore partly responsible for the formation of new grains. In fact, a large fraction of initial grains remained even at moderate strains (**Fig. 3.14c**, and **Fig. 3.17d, g**), indicating their resistance to the occurrence of DRX. Finally, the initial grains became highly elongated (and thinned) after a sufficient amount of strain, so that geometrical DRX might partly happen and the low misorientation regions (LMRs) dominated the microstructure at high strains.

Medium Z is the transition condition (with N : $0.3 \sim 0.5$), which could be found between the two extremes (high Z and low Z conditions), where rather equiaxed microstructures indicated an ability of boundary migration (like under the condition of low Z). The fraction of fine equiaxed grains surrounded by HABs was noticeably raised with increasing Z (**Fig. 3.7a**).

Under the medium Z , the initial grains were firstly subdivided by many deformation-induced boundaries and deformation bands (like under the condition of high Z). However, the subdivided microstructures were not stable after applying some moderate strains around $2 \sim 3$. Original boundaries started to serrate and fine equiaxed grains appeared particularly within highly fragmented regions. Eventually, the subdivided microstructures were replaced by equiaxed grains at high strains (see **Figs. 3.3-3.6**). The increase in Z could provide more misoriented areas and therefore generated more equiaxed grains.

In brief, it seemed that the microstructure evolution under the condition of medium Z , was governed by (1) the grain subdivision and the progressive increase in the misorientation of deformation-induced boundaries and (2) the formation of equiaxed grains with the assistance of some boundary interactions and rearrangement, caused by triple-junction motion and short-range boundary migration. The driving force for the process (2) was to reduce the boundary energy, particularly within highly misoriented regions.

Based on the aim of this research introduced in Chapter 1 (**Sec. 1.6.**), the IF steel was highly deformed in a series of torsion tests carried out under various Z conditions, and the formation processes of new fine/ultrafine grains were studied. **Figure 3.19**, schematically summarizes representative microstructures formed under different conditions shown on the ε - Z diagram. The ε - Z diagram is divided into three regions: i.e. low Z , medium Z , and high Z regions. In each region, two representative microstructures (EBSD boundary maps) are provided: one at a moderate strain ($\varepsilon = 2 \sim 3$) and another at a high strain ($\varepsilon = 5 \sim 7$). The red cross marks in the ε - Z diagram denote all (torsion) test conditions (various temperatures, strain rates, and strains) applied to deform the IF steel in the present study. Additionally, the data associated to the peak strain (ε_p), steady-state strain (ε_s), and critical strain (ε_{cr}) are added to the lower Z region of the ε - Z diagram. The peak strain (ε_p) and the steady-state strain (ε_s) correspond to the strains at which flow stress-stain curves (Chapter 2, **Fig. 2.5**) reached the maximum stress and the steady-state (after a stress drop), respectively. The critical strain (ε_{cr}) for the initiation of dynamic restoration mechanisms (e.g. DRX) was determined by the second derivative method introduced before (**Fig. 3.12**). Two characteristic lines (denoted by ε_{cr} and ε_s) were fitted on the data points associated to ε_{cr} and ε_s , inside the low Z region in **Fig. 3.19**. It should be noted that in conventional DRX-type metals and alloys, DRX initiates at ε_{cr} and finishes at ε_s . In other words, at a

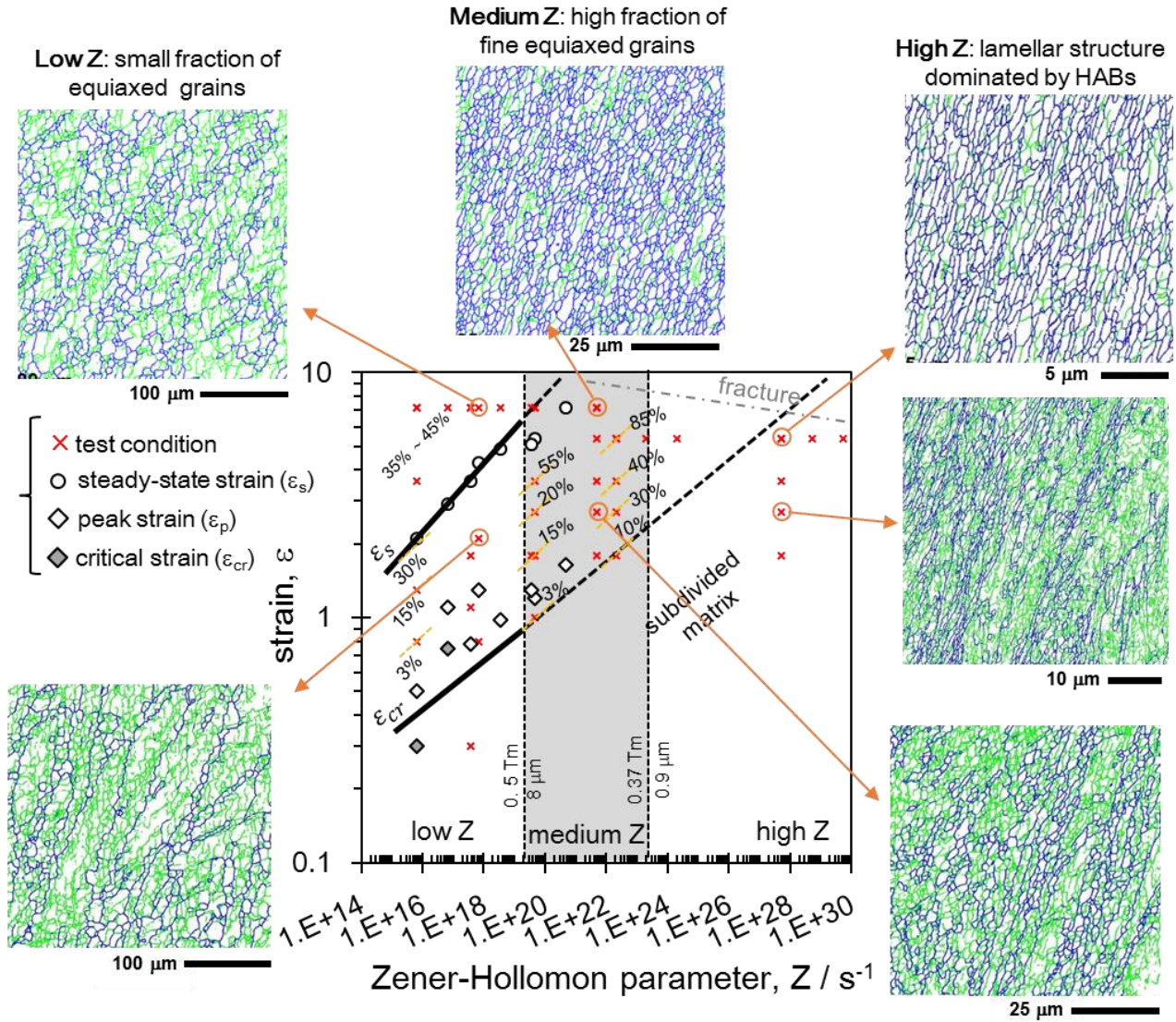


Figure 3.19. ε – Z diagram illustrating the various high Z , medium Z , and low Z regions in the torsion-deformed IF steel in the present study. In each region, two representative microstructures (EBSD boundary maps) are provided at a moderate strain ($\varepsilon = 2 \sim 3$) and a high strain ($\varepsilon = 5 \sim 7$). Red cross marks denotes various (test) conditions applied for the IF steel. Additionally, fractions of fine/equiaxed grains in the microstructures are shown for some deformation conditions. The data points associated to ε_{cr} (the critical strain to initiate dynamic restoration mechanisms such as DRX), ε_p (the peak strain on flow stress-strain curve), and ε_s (the strain at which the flow stress reaches a steady-state) are also inserted in the lower Z region of the diagram. Two characteristic lines are fitted on the data points associated to ε_{cr} and ε_s .

strain above the ε_s line in **Fig. 3.19**, a fully recrystallized microstructure is normally expected to form in DRX-type metals.

In **Fig. 3.19**, in the high- Z region ($> 10^{23} s^{-1}$), the grain subdivision is dominant leading to the ultrafine lamellar structures, in which HABs dominated at high strains.

Shifting toward the medium Z region ($10^{19} \text{ s}^{-1} \sim 10^{23} \text{ s}^{-1}$), boundary migration and grain growth (due to enhanced diffusion) happened. As a result, the subdivided microstructures were replaced by equiaxed grains at sufficiently high strains. With further decrease in Z , the grain growth became dominant especially under the low Z region ($< 10^{19} \text{ s}^{-1}$), resulting in the formation of coarse equiaxed grains. Sometimes the microstructure evolution under the low Z is discussed by considering the DRX phenomenon. However, conventional DRX was not observed in the easy-recovery ferrite of the present IF steel. The number of equiaxed (DRX) grains surrounded by HABs was small. Even at a strain much above the ε_s line in the low Z region, the fraction of equiaxed grains was only around 35% ~ 45% and low misorientation regions (LMRs) dominated the microstructure.

From the microstructural point of view, the condition of medium Z could be considered as an optimum condition for obtaining the uniform distribution of fine equiaxed grains. In other words, further increase in Z significantly restricted the boundary mobility, leading to lamellar structures rather than equiaxed structures. On the other hand, further decrease in Z limited the degree of subdivision (in-grain fragmentation). At the same time, the formation of DRX grains was restricted in the bcc ferrite. Both facts led to a small fraction of newly formed equiaxed grains with heterogeneous distribution, which could be considered as the characteristic behavior in high-strain deformation under the low- Z conditions in the bcc ferrite.

3.5. Conclusions

An ultra-low carbon IF steel was deformed under certain Z conditions, all within the single-phase **bcc-ferrite** region ($< 900\text{ }^{\circ}\text{C}$). The microstructure evolution and formation of new grain structures were investigated at different **low to very high strains**. The followings are the main results and new findings obtained:

High Z ($> 10^{23}\text{ s}^{-1}$) conditions: With applying strain, the deformation-induced boundaries resulted from the **grain subdivision** were generated. Increasing the strain increased the fraction of HABs and decreased their spacing. At sufficiently high strains ($\varepsilon > 5$), the grain subdivision led to the **UFG lamellae** dominated by elongated HABs ($f_{\text{HABs}} > 70\%$). The boundary migration was highly constrained to maintain the lamellae morphology.

Medium Z ($10^{19}\text{ s}^{-1} \sim 10^{23}\text{ s}^{-1}$) conditions: Microstructure evolution at early stages of deformation was similar to that under the high Z conditions. Initial grains were firstly elongated and subdivided through the generation of deformation-induced boundaries. After some moderate strains ($\varepsilon = 2 \sim 3$), the subdivided microstructures became not stable; that is, a noticeable fraction of fine grains with rather equiaxed shapes appeared within the highly misoriented regions such as narrow bands. Finally, the microstructure at high strains ($\varepsilon > 4 \sim 5$) was characterized as a **high fraction of fine equiaxed grains** with uniform distribution. It was suggested that the microstructure evolution was basically governed by the grain subdivision and progressive increase in the misorientation of boundaries. This was accompanied with **triple-junction motion** and **local boundary migration** to make the morphology of microstructure equiaxed.

Low Z ($< 10^{19}\text{ s}^{-1}$) conditions: **Boundary migration** and **grain growth (i.e. diffusion processes)** were expected to be enhanced. At early stages of deformation, initial grains became elongated. The degree of in-grain subdivision, however, was limited significantly. Only **small fraction of (coarse) equiaxed grains** along initial grain boundaries or isolated inside initial grains formed. Due to the enhanced recovery in bcc-ferrite, typical DRX events (e.g. copious formation of DRX grains decorated along initial grain boundaries) were not observed. With increasing the strain, the initial grains became more and more elongated although the fraction of equiaxed grains did not increase significantly. Such a microstructure evolution could lead to the formation of low misorientation regions (LMRs) at high strains, as well as some boundary interactions similar to GDRX.

3.6. References

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Chapter 4: Formation of new grain structures in SUS 304 stainless steel during high-strain torsion deformation at various temperatures and strain rates

4.1. Introduction

In previous Chapters 2 and 3, the formation of fine and ultrafine grains in an easy-recovery metal (i.e. bcc-ferrite in the IF steel) has been investigated and discussed. For comparison, this chapter is dedicated to an fcc-metal (austenitic stainless steel), in which dynamic recovery is known to be suppressed and dynamic recrystallization is easy to occur.

The same high-strain torsion test was carried out under various deformation conditions, using a SUS 304 austenitic stainless. The results are explained in terms of the flow stress-strain curve, microstructure evolution as a function of the Zener-Hollomon parameter and strain, etc. Similar to the IF steel, the microstructure evolution in the 304 steel under various conditions of low Z to high Z is discussed. Regarding the formation of new grain structures, the differences and similarities between different kinds of metals are also clarified.

4.2. Experimental procedures

4.2.1. Material

A commercial SUS 304 austenitic stainless steel was used in this study, as a candidate fcc metal. The austenitic phase in the 304 stainless steel is stabilized due to a high content of nickel (Ni) in chemical composition. The stable austenitic phase, makes it possible to deform the 304 stainless steel in a wide range of temperature without any phase transformation. **Table 4.1** shows the chemical composition of the as-received 304 austenitic stainless steel, which has 8% Ni and 18% Cr as main

alloying elements. The torsion specimens were cut off from the as-received 304 plate (solution-heat treated at 1140 °C and water quenched) with 15mm thickness.

The as-received microstructure is shown as an EBSD boundary map (**Fig. 4.1a**) and an IPF map (**Fig. 4.1b**). The SUS 304 specimen had a fully recrystallized microstructure with a rather randomized texture, of which the average grain size was around 20 μm , including the annealing twin boundaries. **Figure 4.1c** shows the distribution of misorientation angles of boundaries in the microstructure presented in **Fig. 4.1a**. As can be seen, the fraction of twin boundaries in the initial microstructure was high (50%), as expected in the 304 stainless steel which is an fcc-metal with low stacking fault energy.

4.2.2. Methods

Figure 4.2a shows the thermo-mechanical processing schedules used to deform the 304 stainless steel. The specimen was heated up to 1200 °C at a heating rate of 5 °C/s and held at this temperature for 3 minutes. Subsequently, the specimen was cooled down (at 3 °C s⁻¹) to the deformation temperature at 1100 °C (0.76 T_m), 1000 °C (0.7 T_m), 900 °C (0.65 T_m), 800 °C (0.59 T_m), and 700 °C (0.54 T_m), where T_m is the melting point of iron in Kelvin (1811 K). The specimen was held at the temperature for 2 minutes for temperature homogenization, and then deformed at a strain rate of 0.01 s⁻¹ or 0.1 s⁻¹. In most cases, the specimens were deformed up to an equivalent strain of 3.6 (corresponding to 1 rotation of torsion) and in a few cases up to 5. In general, the fracture strain in the 304 stainless steel was smaller than that in the IF steel. It should be noted that the deformation at temperatures below 700 °C was difficult because of the high load above the capacity of the torsion machine, owing to high strength of the 304 steel. Immediately after the deformation, the specimen was water quenched within 1 second. More details regarding the torsion specimen size and the torsion test have been described in Chapter 2 (section. 2.2).

Table 4.1. Chemical composition (wt%) of the as-received SUS 304 austenitic stainless steel (balance: Fe)

C	Si	Mn	P	S	Ni	Cr	Co
0.04	0.57	0.92	0.035	0.004	8.05	18.14	0.20

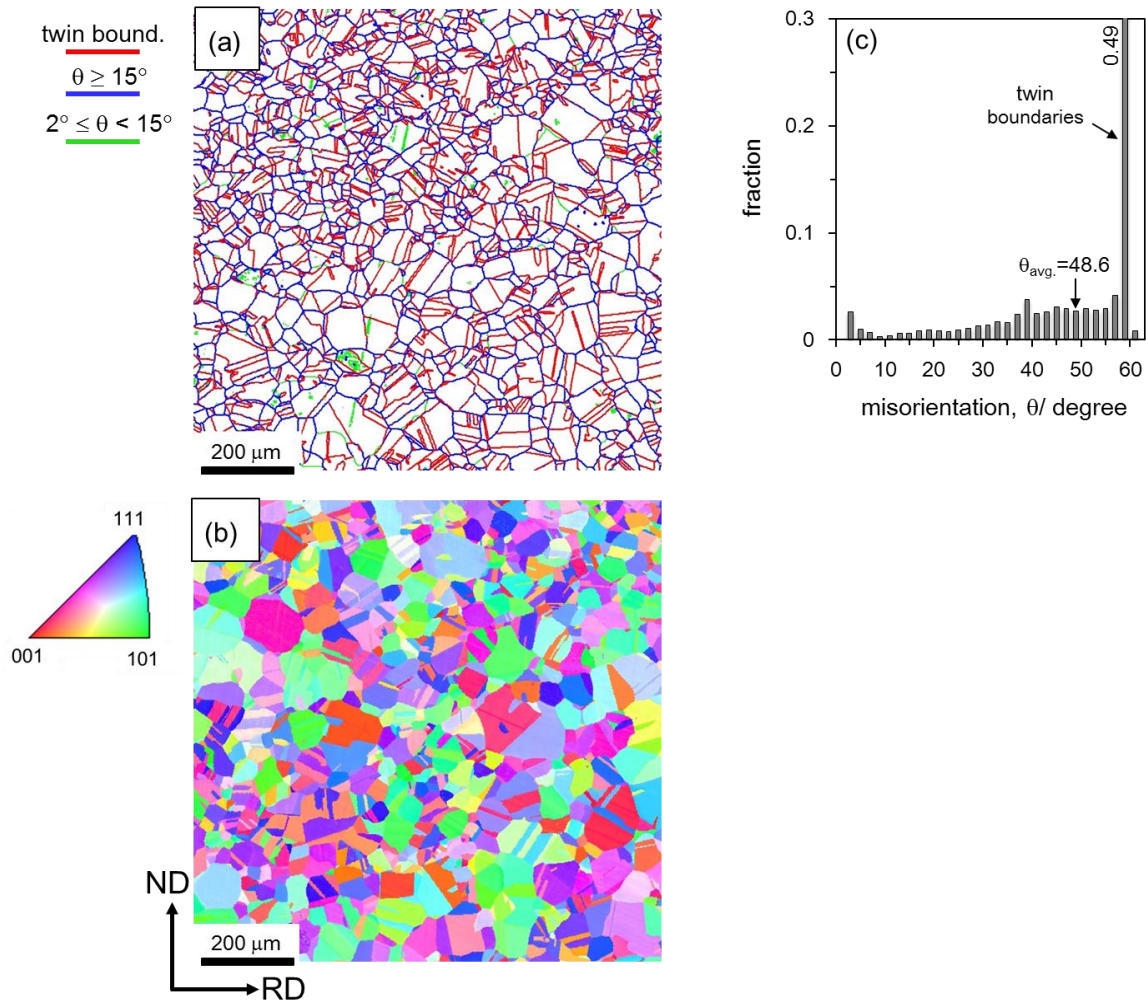


Figure 4.1. As-received microstructure of the SUS 304 austenitic stainless steel. (a) EBSD boundary map, (b) EBSD inverse pole figure (IPF map), and (c) distribution of misorientation angles of boundaries in the microstructure presented in (a).

Microstructures of the torsion deformed 304 steel were observed by Electron Back-scattering Diffraction (EBSD) method and Electron Channeling Contrast Imaging (ECCI) technique, on the surface of the gauge part of the torsion specimen (**Fig. 4.2b**). In **Fig. 4.2b**, r is the radial direction perpendicular to the observed section, Z and θ are the shear plain normal direction and the shear direction, respectively. For the EBSD analysis and ECCI technique, the surface was prepared for the observation by the mechanical polishing and subsequent final polishing by a 0.04 and 0.02 μm colloidal silica suspension. EBSD measurements and ECCI technique were carried out in field emission scanning electron microscopes (FE-SEM; JEOL JSM 7100F) and (FE/SEM-JEOL JSM 7800F), respectively. EBSD data were analyzed using Orientation Imaging Microscopy (TSL OIM, version 7.2.1) software.

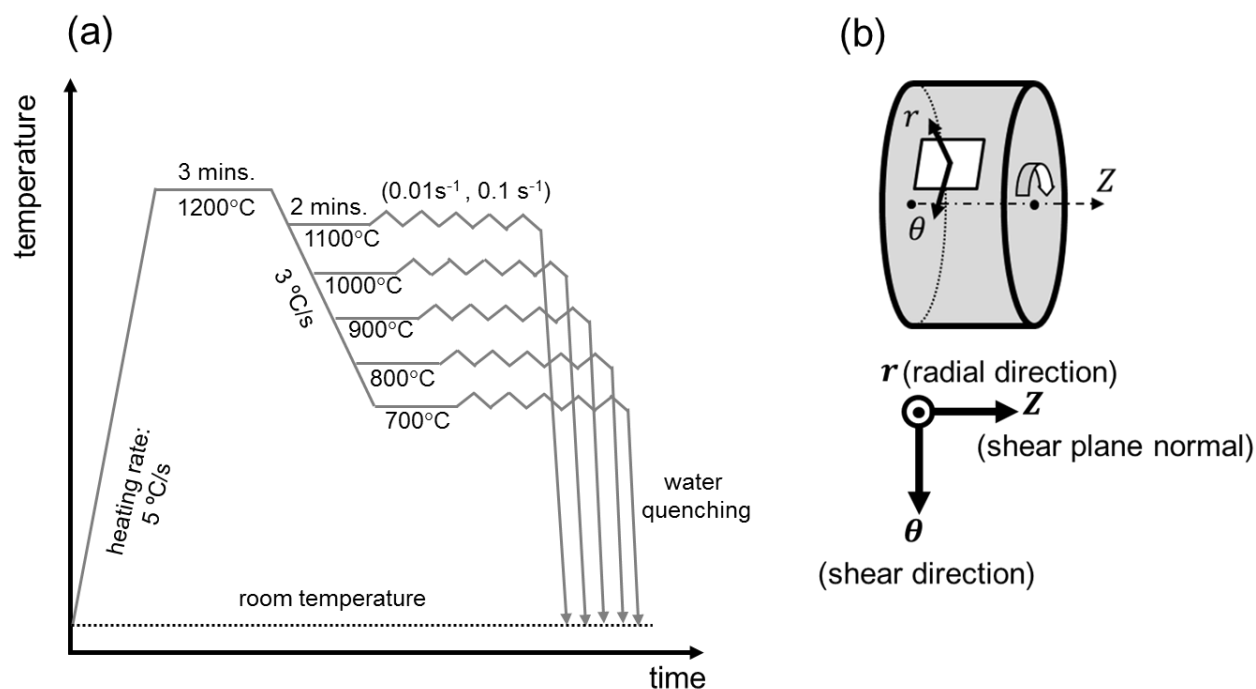


Figure 4.2. (a) Thermo-mechanical processing schedules used for the 304 austenitic stainless steel together with (b) configuration of the torsion specimen; a tangential section (white rectangle) on the gauge part of the specimen used for the microstructure observation; r is the radial direction perpendicular to the observed section, Z and θ are the shear plane normal direction and the shear direction, respectively.

4.3. Results

4.3.1. Flow stress-strain curves

Torque-rotation data obtained during the torsion test were converted into shear stress – shear strain data by employing Fields and Backofen analysis ¹ (Equations 2.1 and 2.2). Subsequently, von Mises criteria was used to attain equivalent stress (σ) and equivalent strain (ϵ) (Equations 2.3 and 2.4).

Figure 4.3 shows the flow stress-strain curves of the 304 austenitic stainless steel torsion-deformed to a strain of 3.6, at a wide range of deformation temperatures from 1200 °C to 700 °C at different strain rates; i.e. 0.01 s^{-1} (**Fig. 4.3a**) and 0.1 s^{-1} (**Fig. 4.3b**). With decreasing the temperature or increasing the strain rate (increasing Z),

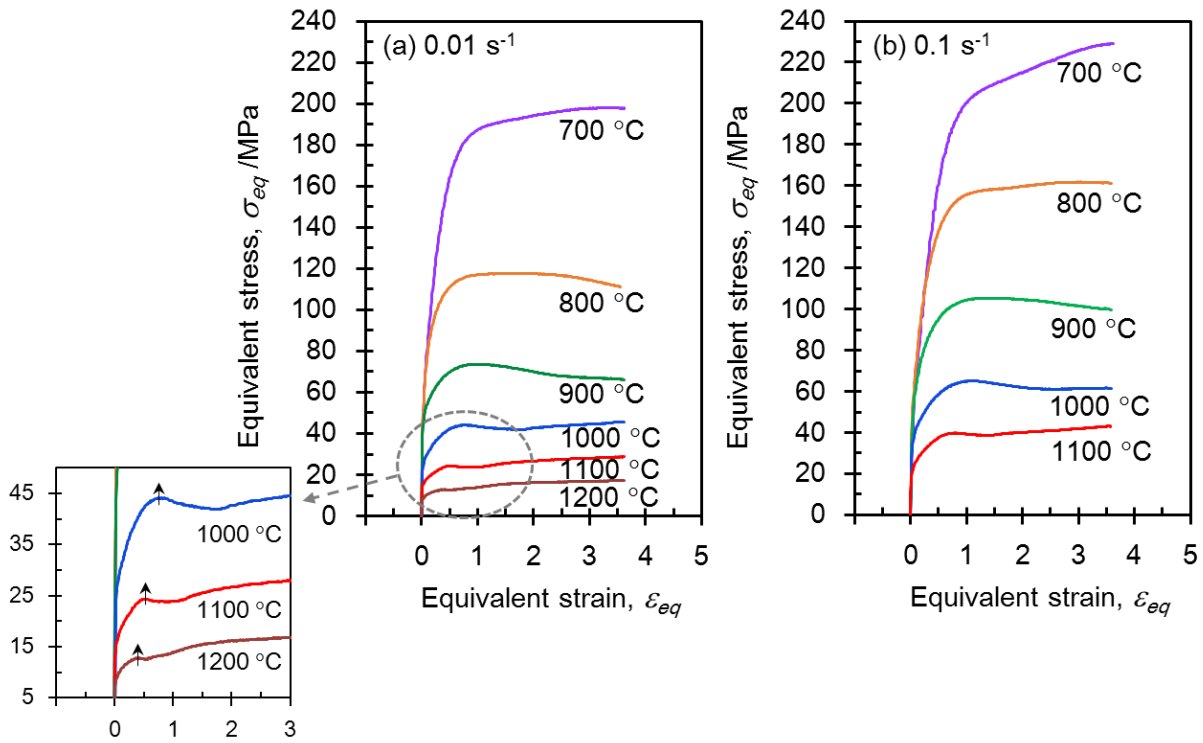


Figure 4.3. Flow stress-strain curves (von Mises equivalent stress vs. equivalent strain) of the 304 stainless steel torsion-deformed to a strain of $\varepsilon \approx 3.6$ at various temperatures (700 °C - 1200 °C) shown at two strain rates of (a) 0.01 s⁻¹ and (b) 1.0 s⁻¹.

the flow stress level increased. Similar to the IF steel, shapes of the flow stress-strain curves changed with Z ; the flow stress under low Z conditions (1000 °C - 1200 °C) contained a distinctive peak stress shown by arrows in the magnified stress-strain curves in **Fig. 4.3a**, and then a softening part to a steady-state condition. The peak strain increased with increasing Z , leading to a stress-strain curve with a broader peak (e.g. at 900 °C). On the other hand, a plateau or work-hardening curve could be observed under large Z conditions (e.g. at 700 °C - 800 °C).

It should be noted that the low- Z curves in the 304 steel showed an increasing part after reaching the steady-state condition (often at higher strains). This was believed to be attributed to an increasing friction caused by the dimensional change of the torsion specimens (decreasing length of the gauge part), and not caused by any microstructural events in the material. Sometimes such a rise in the flow stress may mask the stress drop after the peak (e.g. see curve at 1200 °C/ 0.01 s⁻¹). Nonetheless, the peak stress was distinguishable.

The peak or maximum stress (σ_m) and the final stress (σ^*) were measured from the

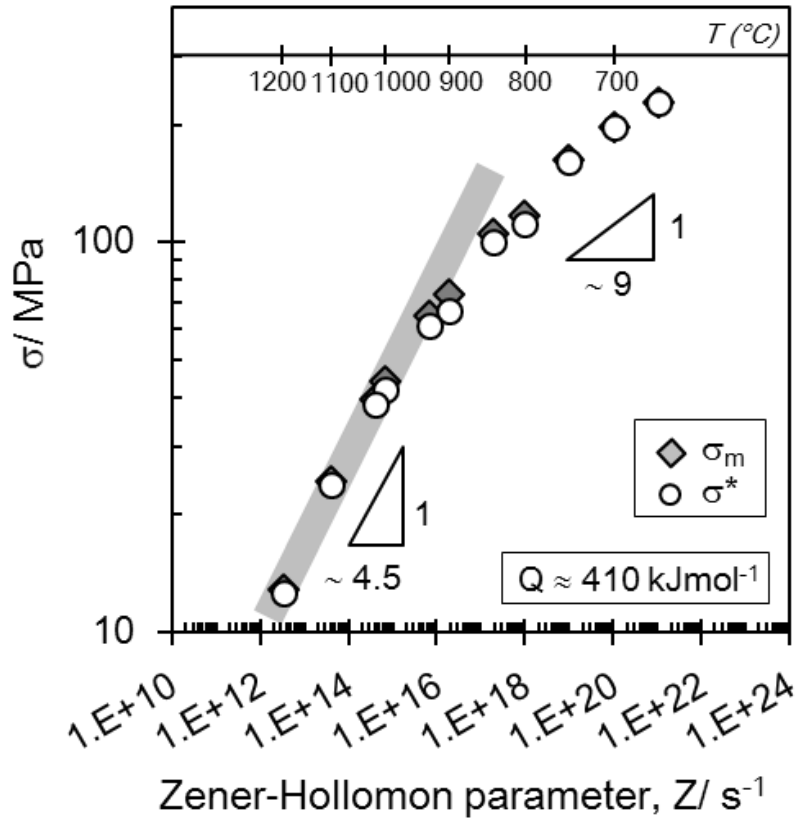


Figure 4.4. Flow stress (σ_m : the maximum stress and σ^* : the stress at the final strain) as a function of the Z parameter. The gray bay indicates a power-law relationship. Corresponding deformation temperatures at a constant strain rate (0.01 s^{-1}) are indicated on the upper line.

flow stress-strain curves. **Figure 4.4** displays these stress values as a function of the Zener-Hollomon parameter. Here, the activation energy was simply taken from the literature ²; i.e. $Q \approx 410 \text{ kJmol}^{-1}$ as an average value for hot-deformation in several 304-type alloys. As expected, with increasing Z , the stress data deviated from the linearity (or power-law break down) at a certain point of Z (corresponding to $\sim 900 \text{ }^\circ\text{C}$). The $\dot{n}$ value in $Z = \dot{A} \sigma^{\dot{n}}$ relationship in the low Z region was estimated ~ 4.5 , which was in agreement with the value reported by McQueen et al. ² for hot deformation of 304-type alloys. However, $\dot{n}$ changed and increased to ~ 9 in the larger Z region. Similar to the IF steel, this deviation was accompanied with the change in the shape of the flow stress-strain curves below $1000 \text{ }^\circ\text{C}$; i.e. stress-strain curves with obvious peaks into those with broader peaks or without distinct peaks.

4.3.2. Microstructure vs. Z parameter

Fig. 4.5 exhibits EBSD microstructures on the θ -Z planes of the 304 stainless steel deformed up to $\varepsilon \approx 3.6$, at 1100°C (**Figs. 4.5a, 4.5b**), 1000°C (**Figs. 4.5c, 4.5d**), 900°C (**Figs. 4.5e, 4.5f**), 800°C (**Figs. 4.5g, 4.5h**), and 700°C (**Figs. 4.5g, 4.5h**), at two different strain rates: 0.01 s⁻¹ (upper maps) and 0.1 s⁻¹ (lower maps). The equivalent Z value for each condition is also provided. In the EBSD boundary maps, the blue and the red lines represent high angle boundaries (HABs; $\theta \geq 15^\circ$) and twin boundaries ($\Sigma 3$ type), respectively.

It can be seen that the grain refinement was greatly enhanced by increasing the Z parameter. As expected from previously reported results in conventional DRX metals, DRX grains under low Z conditions occupied the entire matrix, which is an essential difference from low-Z microstructures in the IF steel (e.g. **Fig. 2.9a, b**). DRX grains had various sizes and shapes, representing DRX grains at different stages; that is, newly generated ones (small and equiaxed), growing ones (bigger and rather equiaxed), and ones being deformed again (large and rather elongated). The feature also indicates that the DRX repeatedly happens during deformation, as has been reported.

On the other hand, under high Z conditions, the applied strain (3.6) was not enough to reach a steady-state condition from a microstructural point of view (e.g. **Figs. 4.5f-j**). For those cases, recrystallized (refined) regions were firstly separated from the remnants of old grains (based on their GOS values) so that only the size of newly generated grain structure could be measured. Grain orientation spread (or GOS) in TSL OIM software expresses the extension of deformation inside a grain. For example, GOS values in newly formed grains (e.g. DRX grains) are smaller than those in deformed and elongated grains. In the 304 stainless steel, values of GOS < 5° were found to be appropriate to extract refined regions from old (deformed) ones.

The average spacing of HABs (L_{HABs}) in recrystallized (or refined) microstructures was measured and reported as the grain size (D). The grain size was estimated within a range of 27 μm ~ 0.4 μm , i.e. a wide range of fine or ultrafine grains were obtained. Despite the fact that the submicron grain size was achieved, the new grains' morphology seemed to stay rather equiaxed over the entire range of Z. **Figure 4.6** demonstrates the variation of the grain size as a function of the Z parameter. Additionally, the subgrain size (d_{SG}) measured in ECC imaging technique is provided in **Fig. 4.6** for some deformation conditions. A single power-law relationship ($D \propto$

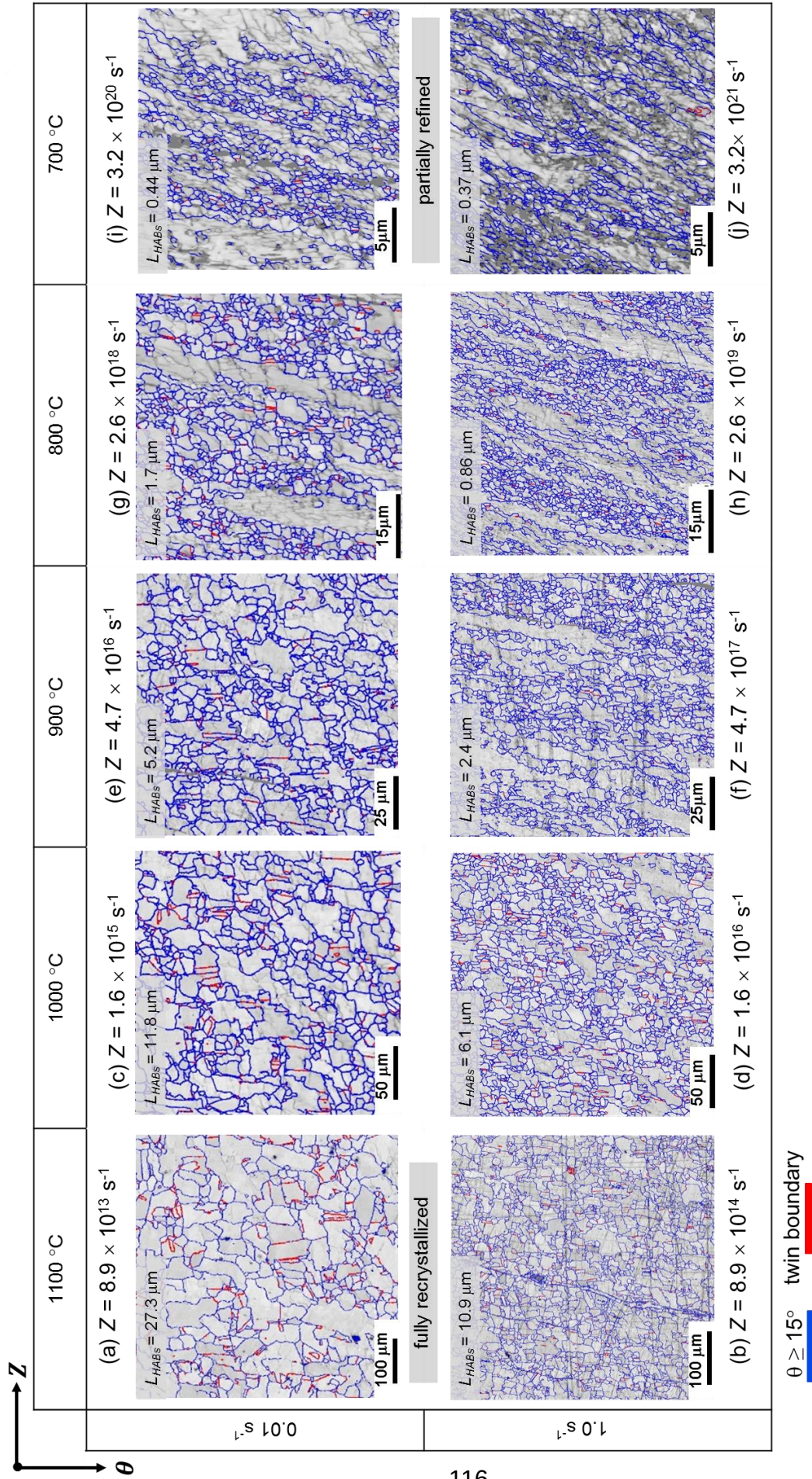


Figure 4.5. EBSD microstructures of the 304 stainless steel deformed up to $\varepsilon \approx 3.6$ at a strain rate of 0.01 s^{-1} (upper maps) or 0.1 s^{-1} (lower maps) at (a, b) 1100°C, (c, d) 1000°C, (e, f) 900°C, (g, h) 800°C, and (i, j) 700°C. EBSD map descriptions: HABs ($\theta \geq 15^\circ$) as blue lines and $\Sigma 3$ twin boundaries as red lines. In addition, corresponding Z values for various conditions are provided.

$Z^{-0.26}$) could be fitted on all the data, without showing any significant deviation.

Power-law break down was not observed in the 304 steel, unlike what found in the IF steel (**Fig. 2.11a**), which the grain size saturated under conditions of very high Z . However, the grain size in **Fig. 4.6** was approaching to the subgrain size with increasing Z . It seemed that the grain size associated to the largest Z in **Fig. 4.6** somewhat deviated (dash line) from the linear fit. This might correspond to the onset of saturation, like that found in the IF steel. Unfortunately, however, tests at lower deformation temperatures for confirming the saturation were not possible in the 304 stainless steel due to the load limitation.

The variations of stress (**Fig. 4.4**) and grain size (**Fig. 4.6**) as a function of the Z parameter has been explained. A direct relationship between the stress and the grain size, both of which are the material's properties, is considered in this section. The same

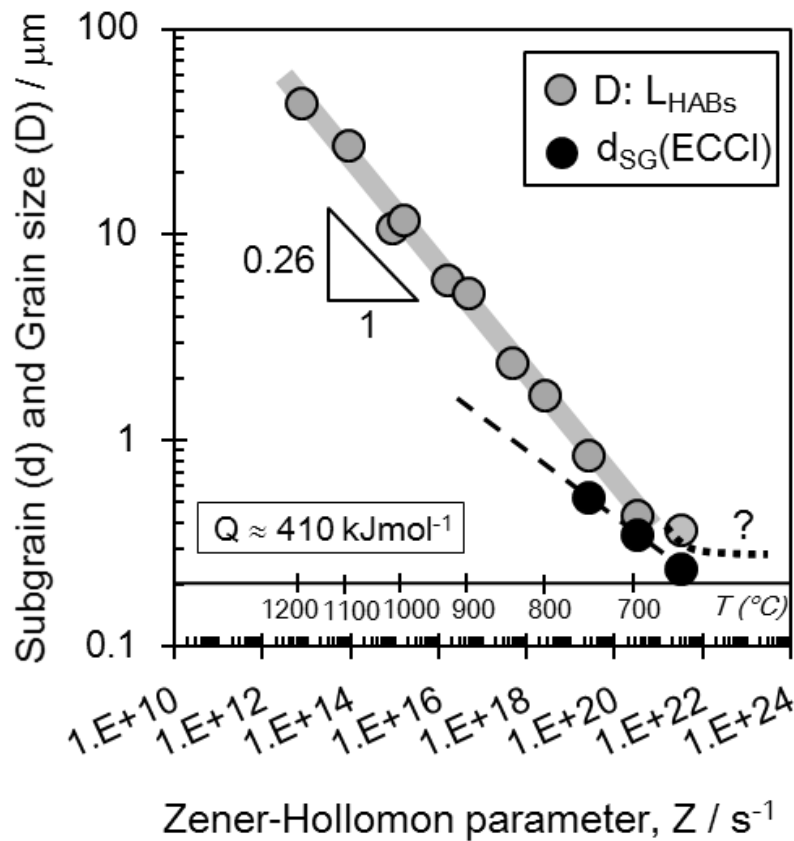


Figure 4.6. Mean grain sizes of DRX grains in the 304 stainless steel deformed at 700 °C - 1200 °C and 0.01 s⁻¹ or 0.1 s⁻¹, plotted as a function of the Z parameter. $D: L_{HABs}$ indicates the average spacing of all HABs. The subgrain size: d_{SG} measured in ECCI technique is added (filled circles) for some deformation conditions. Corresponding temperatures for a constant strain rate (0.01 s⁻¹) are also shown.

analysis was carried out in the IF steel (**Fig 2.13**) which has been explained in Chapter 2 (section 2.3.3). **Figure 4.7** shows the $\sigma - D$ relationship ($\sigma = D^{-N}$) in the 304 stainless steel. Based on the $\sigma - D$ plot, there seemed to be mainly two regions with different N values of ~ 0.46 and ~ 0.77 , respectively. These values were closed to the N values (0.42, 0.72) reported for a 304-type stainless steel in previous literature ^{3–5}. The three stages in the $\sigma - D$ relationship like that found in the IF steel might be hypothetically the case for the 304 steel under higher Z conditions. Accordingly, the region with $N \sim 0.46$ could be referred as a transition (medium Z or medium σ) condition since the grain sizes were approaching to the subgrain size. On the other hand, the low Z or low stress region with larger N (0.77) indicated that the stress was sensitive

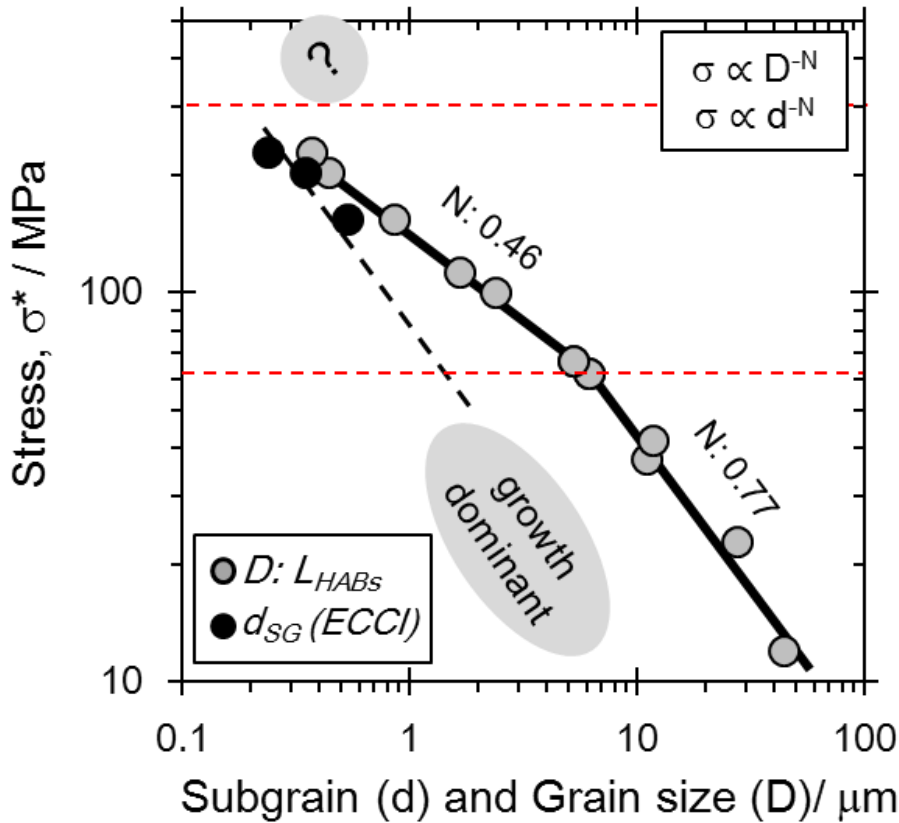


Figure 4.7. (a) Stress (σ^*) as a function of grain size ($D: L_{HABs}$). $D: L_{HABs}$ indicates the average spacing of all HABs. The subgrain size: d_{SG} measured in ECCI technique is added (filled circles) for some deformation conditions. The red dashed lines divide the graph into low Z (or low stress) and medium Z (or medium stress), of which N value ($\sigma \propto D^{-N}$) differs. The question mark indicates that there might be a third region as high Z (or high stress) region although it cannot be confirmed in the present study.

to the grain size. In other words, extra softening (accelerated stress drop) could happen in spite of shifting towards the larger grain size. Since the grain size was larger than the subgrain size, grain growth was considered as a dominant under the low Z condition.

Based on to the $\sigma - D$ plot shown in **Fig. 4.7**, all Z conditions under which the 304 steel was deformed in the present study could be categorized in two regions. The summary of such a categorization is represented in **Table 4.2**, in which the deformation conditions are associated to the medium Z region and the low Z region highlighted by different colors. In the 304 stainless steel, it was realized the medium Z region was expanded in a range of σ values estimated as 60 Mpa ~ 230 Mpa. However, in the IF steel (**Table 2.5**), the medium Z corresponded to a narrow range of σ values estimated as 45 Mpa ~ 90 Mpa. This difference could be also realized by comparing the $\sigma - D$ plots associated to the 304 steel (**Fig. 4.7**) and the IF steel (**2.13**). As a result, it could be said that the high Z condition in the 304 steel must be shifted towards a higher range of σ , which could not be achieved in the current study due to the load capacity in the torsion machine.

The microtexture analysis was also carried out in the torsion-deformed 304 steel, of which result is presented in **Fig. 4.8**. The schematic ODF plots for $\varphi_2 = 0^\circ$ and $\varphi_2 = 45^\circ$ sections shown in **Fig. 4.8a**, illustrate important ideal orientations in torsion texture of fcc metals ^{7,8}. The notations for different orientations and their Miller indexes are presented in the figure, with respect to the shear plane normal direction (Z) and the shear direction (θ). **Figures 4.8b-e** display the ODF plots associated to some deformation conditions whose microstructures were previously shown in **Figs. 4.5a** ($Z = 8.9 \times 10^{13} \text{ s}^{-1}$), **4.5c** ($Z = 1.6 \times 10^{15} \text{ s}^{-1}$), **4.5e** ($Z = 4.7 \times 10^{16} \text{ s}^{-1}$), and **4.5g** ($Z = 2.6 \times 10^{18} \text{ s}^{-1}$).

Table 4.2. Summary of deformation conditions (temperature and strain rate) applied for the 304 stainless steel in the present study. All conditions are categorized into the medium Z (σ) and the low Z (σ) regions with the associated N values ($\sigma = D^{-N}$).

strain rate, $\dot{\epsilon} / \text{s}^{-1}$	Zener-Hollomon parameter, Z / s^{-1}						Z / s^{-1}	σ / MPa	N ($\sigma = D^{-N}$)
	700 °C	800 °C	900 °C	1000 °C	1100 °C	1200 °C			
10^{-1}	3.2×10^{21}	2.6×10^{19}	4.7×10^{17}	1.6×10^{16}	8.9×10^{14}	-	Medium Z $10^{16} \sim 10^{21}$	Medium σ 60 ~ 230	0.46
10^{-2}	3.2×10^{20}	2.6×10^{18}	4.7×10^{16}	1.6×10^{15}	8.9×10^{13}	7.3×10^{12}	Low Z $< 10^{16}$	Low σ < 60	0.77

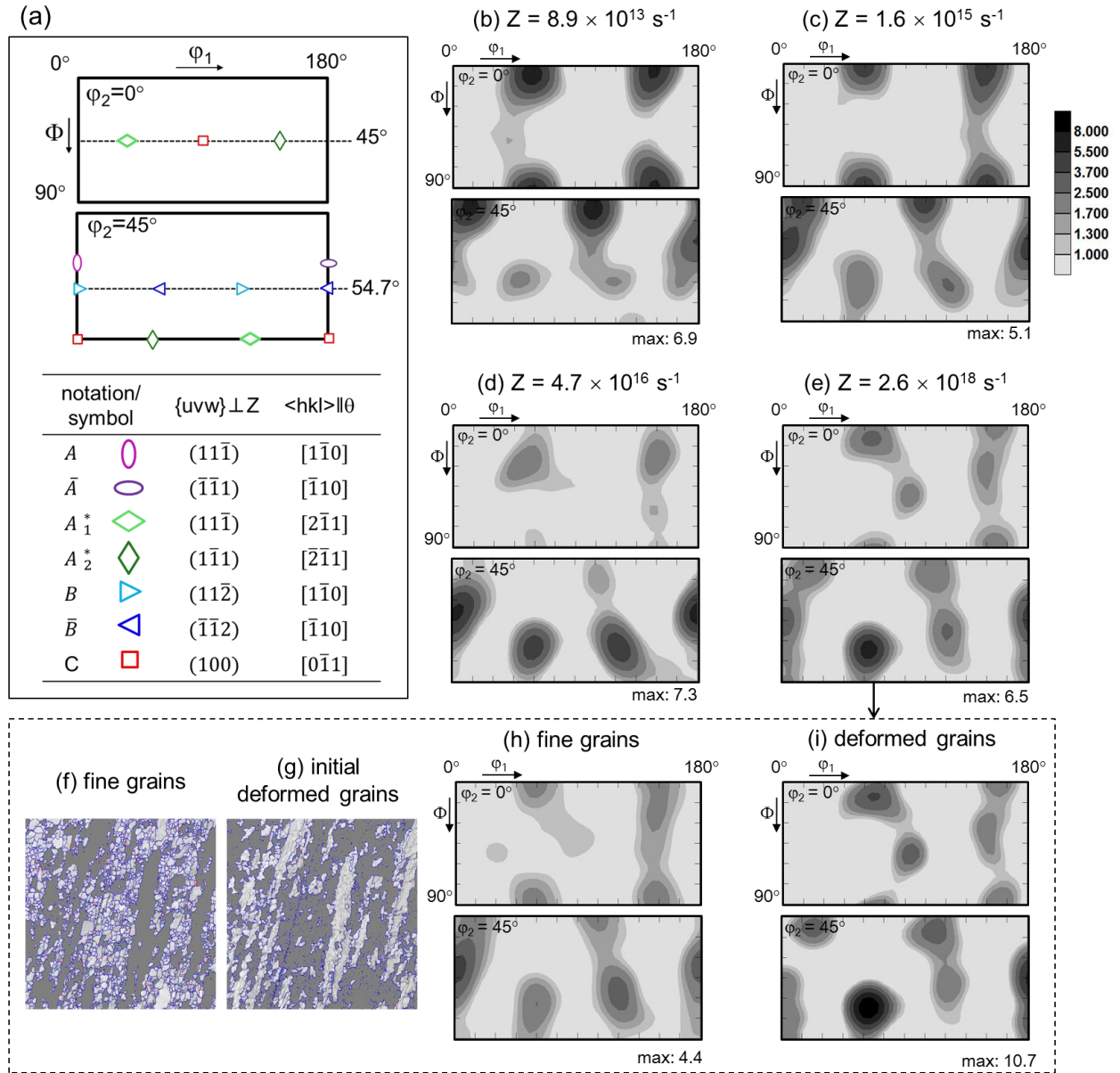


Figure 4.8. (a) Schematic ODF (orientation distribution function) plots at constant ϕ_2 sections (0° , 45°) including the ideal crystallographic orientations in fcc-torsion [7,8](#) together with the notation and the symbol related to each orientation. Z and θ are the shear plain normal direction and the shear direction, respectively. (b-d) ODF plots associated to some deformation conditions whose microstructures were previously shown in Figs. 4.5a ($Z = 8.9 \times 10^{13} \text{ s}^{-1}$), 4.5c ($Z = 1.6 \times 10^{15} \text{ s}^{-1}$), 4.5e ($Z = 4.7 \times 10^{16} \text{ s}^{-1}$), and 4.5g ($Z = 2.6 \times 10^{18} \text{ s}^{-1}$). Microstructure under the condition of $Z = 2.6 \times 10^{18} \text{ s}^{-1}$ was not at the steady-state, composed of fine grains (f) and initial deformed grains (g). Therefore, separate ODF plots associated to fine grains (h) and initial deformed grains (i) were also provided. In fine grains, Grain Orientation Spread (GOS) values were below 5° and could be used to separate them from initial deformed grains which had higher GOS values.

The microtexture under the condition of $Z = 8.9 \times 10^{13} \text{ s}^{-1}$ (**Fig. 4.8b**) did not show a strong texture around major main ideal orientations in fcc-torsion. This indicates that DRX under the low Z condition could quickly reset deformation textures and led to a different texture. The same result was found under the condition of $Z = 1.6 \times 10^{15} \text{ s}^{-1}$ (**Fig. 4.8c**) although $A/\bar{A}$ components seemed to have some intensity. With increasing Z (**Figs. 4.8d, 4.8e**), the microtexture gradually changed and some orientations such as $B/\bar{B}$ components developed. However in all cases, the intensities of the texture components were not high. The microstructure under the condition of $Z = 2.6 \times 10^{18} \text{ s}^{-1}$ was not in the steady-state, composed of fine grains (**Fig. 4.8f**) and initial deformed grains (**Fig. 4.8g**). Therefore, separate ODF plots associated to fine grains (**Fig. 4.8h**) and initial deformed grains (**Fig. 4.8i**) were also provided. As explained before, GOS values in new fine grains were below 5° and could be used to extract fine grains from deformed grains. It can be seen that the texture intensity of the new grain structure (**Fig. 4.8h**) was low indicating a level of randomization in texture, while the texture intensity of the remnants of old grains (**Fig. 4.8i**) was high, indicating development of deformation texture inside initial deformed grains.

In general, it can be said that DRX events became slower (or DRX cycle longer) with increasing Z . Therefore, large fraction of grains are in the work-hardened stage under higher Z conditions. In other words, the deformation effect became stronger at higher Z , which might favor the development of some ideal orientations in deformation texture. Such microtexture results were different from those found in the IF steel, where a high degree of texture strengthening associated to some ideal orientations (D2) was developed. Here, the key point is whether the conventional DRX happens or not.

The flow stress behavior and the microstructure evolution under various Z conditions have been studied. For further investigation, some Z conditions are chosen to examine how the new grain structures evolve during the deformation. In the next section results of such an analysis using Z conditions selected from the low Z region ($Z < 10^{16} \text{ s}^{-1}$) and from the medium Z region ($Z > 10^{16} \text{ s}^{-1}$) are explained.

4.3.3. Microstructure evolution during deformation

• 4.3.3.1. Medium Z conditions ($Z > 10^{16} \text{ s}^{-1}$)

As an example, the microstructure evolution under the condition of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$ (800 °C/ 0.1 s^{-1}) is shown in **Fig. 4.9**. The deformation was stopped at various strains displayed on the flow stress-strain curve (marked by open circles) in **Fig. 4.9a**, and the quenched specimens were analyzed by EBSD. **Figures 4.9b** through **4.9f** demonstrate representative microstructures at different strains. Here, two types of EBSD maps are displayed. The upper half of **Figs. 4.9a-f** shows high angle boundaries (HABs; with misorientation $\theta \geq 15^\circ$) as blue lines and $\Sigma 3$ twin boundaries as red lines, while the lower half contains low angle boundaries (LABs; $2^\circ \leq \theta < 15^\circ$) as green lines as well as HABs and $\Sigma 3$ twin boundaries. The latter displays distribution and density of all boundaries with misorientation above 2° . In **Fig. 4.9**, the vertical direction coincides with the shear direction (θ) in the torsion deformation, and the axis (**a**) shown by dotted arrows indicates the direction of the major axis of original grains after the corresponding shear deformation, assuming that the initial shape of the grains is a circle, they are deformed into elliptical shapes by shear and the axis (**a**) corresponds to the major axis of the ellipses. Based on the theory of simple shear deformation, the axis (**a**) can be determined by measuring β ($\tan 2\beta = 2/\sqrt{3}\varepsilon$ ¹⁰), i.e., the angle between the shear direction (θ) and the axis (**a**).

Under the condition of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$, the flow stress analysis (the double differentiation method ⁹) did not verify any critical strain for the initiation of DRX. In fact, the flow stress-strain curve in **Fig. 4.9a** showed no distinctive peak after the work-hardening part. A stress drop could be observed at high strains ($\varepsilon > 4$) which was, however, believed to be due to the initiation of surface cracking. Even though the stress-strain curve was a plateau type, the initial microstructure was progressively replaced by increasing fraction of fine and equiaxed grains (f_{NG}), which are pointed out by red arrows). They looked like DRX grains, although their generation was very slow and started at strains above 1.8 (**Fig. 4.9c**). Even at a high strain of ~ 5 (**Fig. 4.9f**), the equiaxed grains could occupied only about 70% of the matrix. They could formed either along initial grain boundaries or inside original grains.

Figure 4.10 demonstrates more details of the development of new grain structures under the condition of the condition of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$. At $\varepsilon = 1.8$, initial grains were elongated as shown by the IPF map in **Fig. 4.10a**, while the fraction of new grains pointed out by a red arrow was negligible ($< 1\%$). Bulging of initial grain boundaries

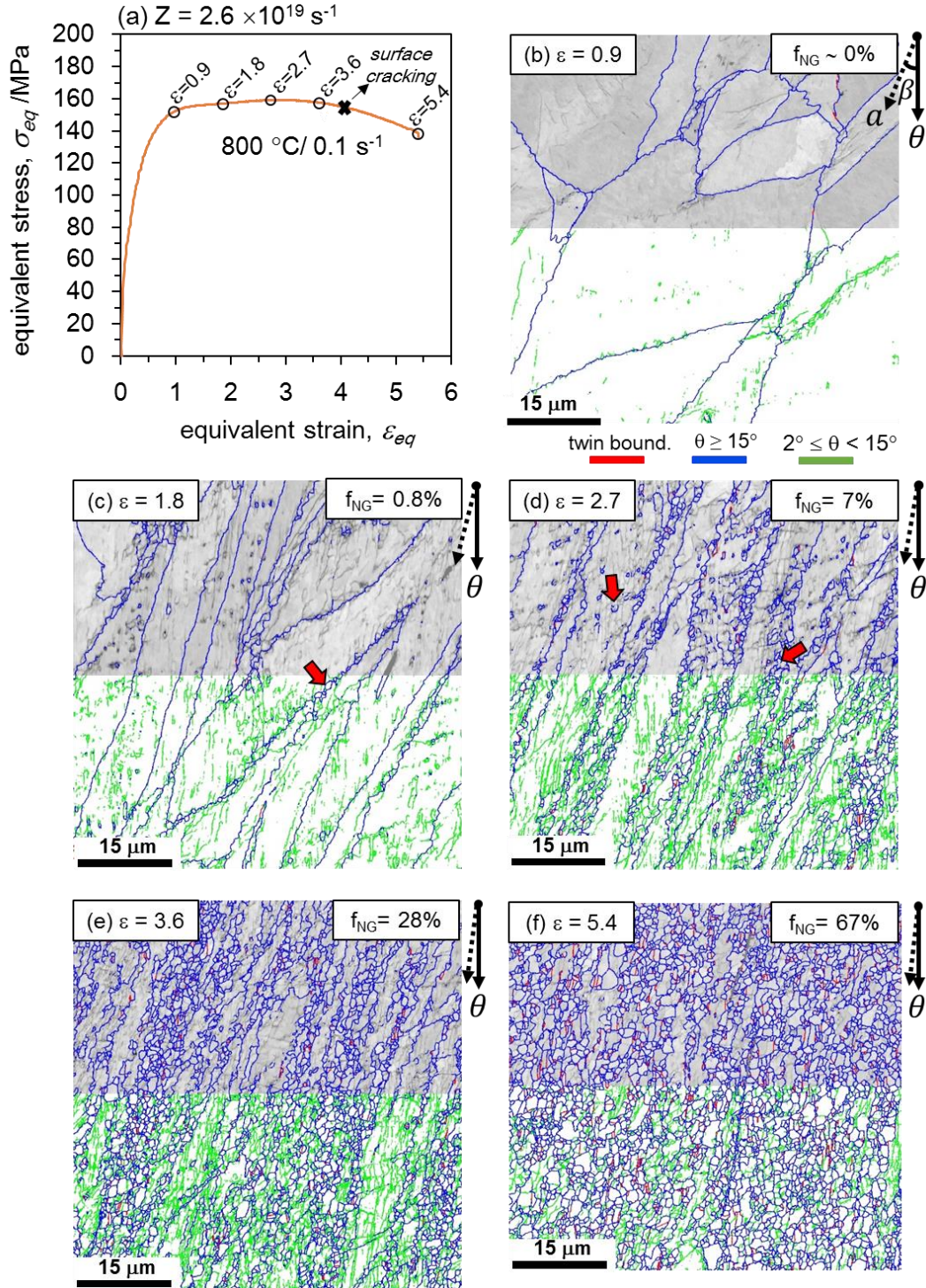


Figure 4.9. (a) Flow stress-strain diagram under the deformation condition of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$ ($800^\circ\text{C} / 0.1 \text{ s}^{-1}$). Microstructure observations were conducted at different strains indicated by the open circle. (b-f) Representative torsion-deformed microstructures at the strains of (b) $\epsilon = 0.9$, (c) $\epsilon = 1.8$, (d) $\epsilon = 2.7$, (e) $\epsilon = 3.6$, and (f) $\epsilon = 5.4$. In each EBSD map (b-f), the upper half shows HABs (with misorientation $\theta \geq 15^\circ$) drawn by blue lines and $\Sigma 3$ -twin boundaries drawn by red lines superimposed on the image quality (IQ) map, while the lower half includes LABs ($2^\circ \leq \theta < 15^\circ$) drawn by green lines in addition to HABs and twin boundaries. Small-angle boundaries with misorientation below 2° are excluded here for removing the inaccuracy of EBSD measurement and analysis. Moreover, β indicates the angle between the shear direction (θ) and the major axis (α) of original grains after the corresponding shear deformation ($\tan 2\beta = 2/\sqrt{3}\epsilon$ ¹⁰). f_{NG} as a fraction of new fine grains which are pointed out by red arrows.

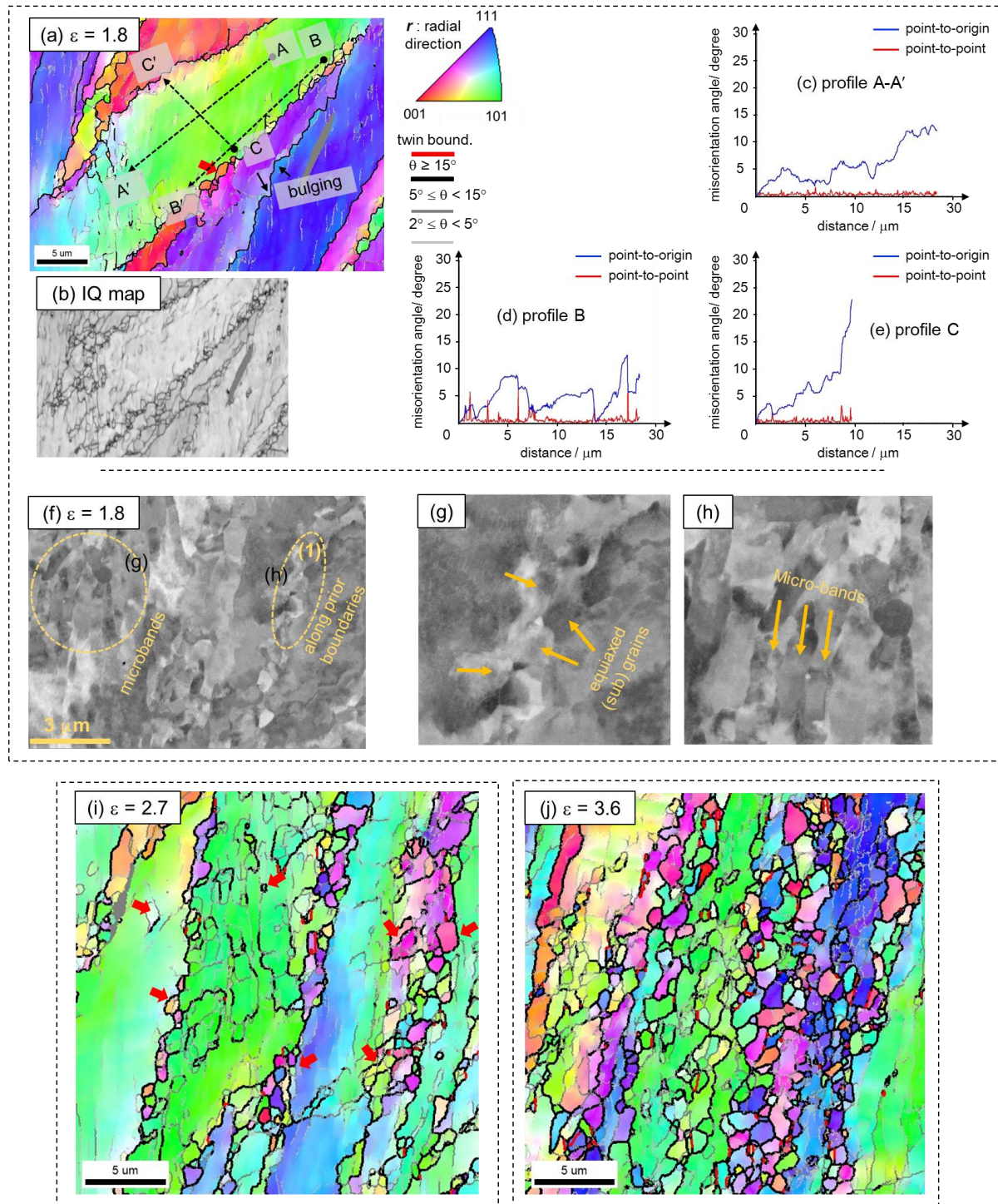


Figure 4.10. EBSD inverse pole figure (IPF) maps of some regions selected in Fig. 4.9. The 304 steel was deformed under the condition of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$ (800 $^\circ\text{C}$ / 0.1 s^{-1}). (a-h) $\varepsilon = 1.8$, (i) $\varepsilon = 2.7$, and (j) 3.6. In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) and the red lines show $\Sigma 3$ twin boundaries while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. Red arrows point out newly formed equiaxed grains. Misorientation profiles along the lines A-A', B-B', and C-C' marked in (a) are also indicated in (c), (d) and (e). (f-h) ECC Images of some regions at $\varepsilon = 1.8$.

could happen even under this medium Z conditions. It seemed that the grain boundary zones were more misoriented than the grain interiors as suggested by the corresponding IQ map (**Fig. 4.10b**). Misorientation gradients along some lines marked in **Fig. 4.10a** are represented in **Figs. 4.10c, d, e**. It can be seen that the point-to-point profile along the line A-A', which is parallel to the direction of grain elongation, exhibits a smooth and accumulative misorientation gradient. The profile B-B' along the similar direction was somehow different, showing a formation of boundaries with misorientation of $5^\circ < \theta < 10^\circ$ adjacent to grain boundaries (see the point-to-point data). The profile C-C' also showed a type of accumulated misorientation, particularly the misorientation arose near grain boundaries.

Further investigation using the ECCI technique (**Figs. 4.10f, g, h**) revealed that various types of substructures appeared at this stage. While some equiaxed (sub) grains formed along initial grain boundaries (**Fig. 4.10g**), band-type substructures (normally referred as micro-bands) were observed inside the deformed grains (**Fig. 4.10h**). Grain interiors became more and more misoriented (fragmented) with increasing the applied strains shown by the IPF maps in **Figs. 4.10i** ($\varepsilon = 2.7$) and **4.10j** ($\varepsilon = 3.6$). In fact, there existed many HAB segments isolated or connected to grain boundaries as well as aggregate of crystallites (shown by red arrows in **Fig. 4.10i**) surrounded partly by HABs and partly by LABs. They seemed to be generated due to the progressive increase in the boundary misorientation. Nonetheless, some grain-like morphologies inside the refined regions looked like that they formed through typical DRX events such as bulging of HABs and grain growth. In other words, the microstructure evolution seemed to be governed by a combination (mixture) of progressive grain subdivision and DRX events.

Figure 4.11 shows some representative microstructures under other medium Z conditions such as $Z = 3.2 \times 10^{20} \text{ s}^{-1}$ (700 °C/ 0.01 s^{-1}) and $Z = 3.2 \times 10^{21} \text{ s}^{-1}$ (700 °C/ 0.1 s^{-1}), which had increased Z values compared to the condition of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$. The IPF maps shown in **Figs. 4.11a** and **4.11c**, are associated to highly deformed microstructures ($\varepsilon = 3.6 \sim 5.1$). The microstructures were basically characterized as subdivided original grains with a few fractions of fine grains bounded by HABs in part. As can be seen, much higher strains were necessary for reaching a steady-state microstructure, although the steady-state was not achieved due to fracture of the specimen. Additionally, substructures under the similar deformation conditions was investigated by ECC imaging technique. The ECC images in **Figs. 4.11b** and **4.11d**

revealed that the substructure contains many band-type features (micro-bands). On the other hands, bulging of some boundaries as a sign of DRX could be observed in **Figs. 4.11b** and **4.11d**, indicating that some boundaries could migrate and replace the deformed (strain-hardened) matrix.

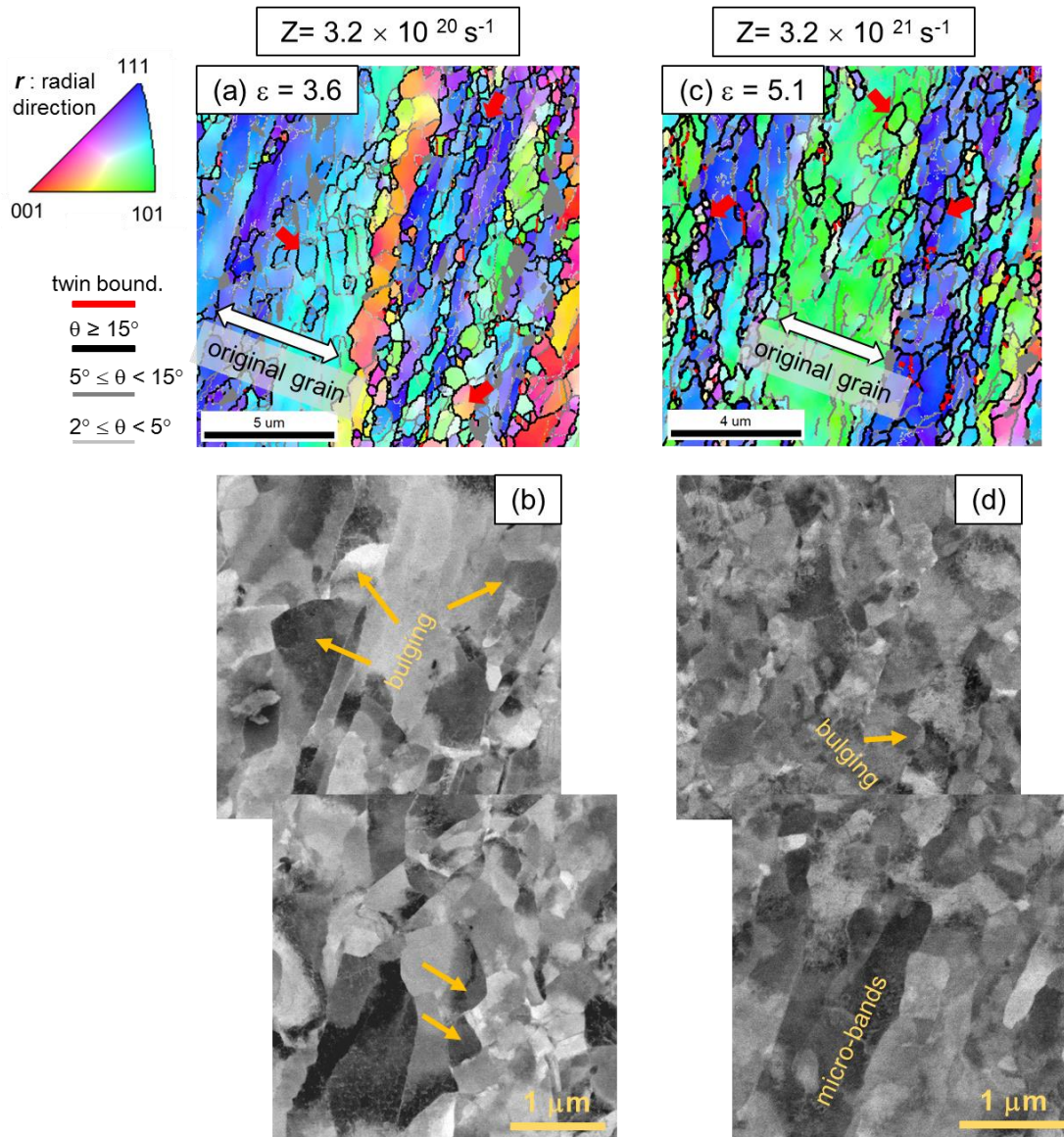


Figure 4.11. (a, c) EBSD inverse pole figure (IPF) maps and (b, d) ECC images, shown as representative microstructures in the 304 steel deformed under the conditions of (a, b) $Z = 3.2 \times 10^{20} \text{ s}^{-1}$ (700 °C / 0.01 s^{-1}) and (c, d) $Z = 3.2 \times 10^{21} \text{ s}^{-1}$ (700 °C / 0.1 s^{-1}). In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) and the red lines show $\Sigma 3$ twin boundaries while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. Red arrows point out newly formed equiaxed grains.

- *Low Z conditions ($Z < 10^{16} \text{ s}^{-1}$)*

To study the low Z conditions in the 304 steel, the flow stress analysis was firstly used to determine the critical strain (ϵ_{cr}) to initiate DRX. **Figure 4.12** shows the flow stress-strain curve under the condition of $Z = 1.6 \times 10^{15} \text{ s}^{-1}$ (1000 °C/ 0.01 s^{-1}) (**Fig. 3.12a**) and the plots for the second derivative method (**Figs. 4.12b, 4.12c**) used to determine the critical strain for DRX under this condition. As a result, the critical strain was estimated as $\epsilon_{cr} \approx 0.35$. The strain at which maximum stress on the flow stress was realized was around 0.8 (ϵ_m). The microstructures of the present 304 steel at this critical strain are shown by different types of EBSD maps (**Figs. 4.12d, e**): i.e., image quality (IQ) map (**Fig. 4.12d**) and inverse pole figure (IPF) map (**Fig. 4.12e**), of which

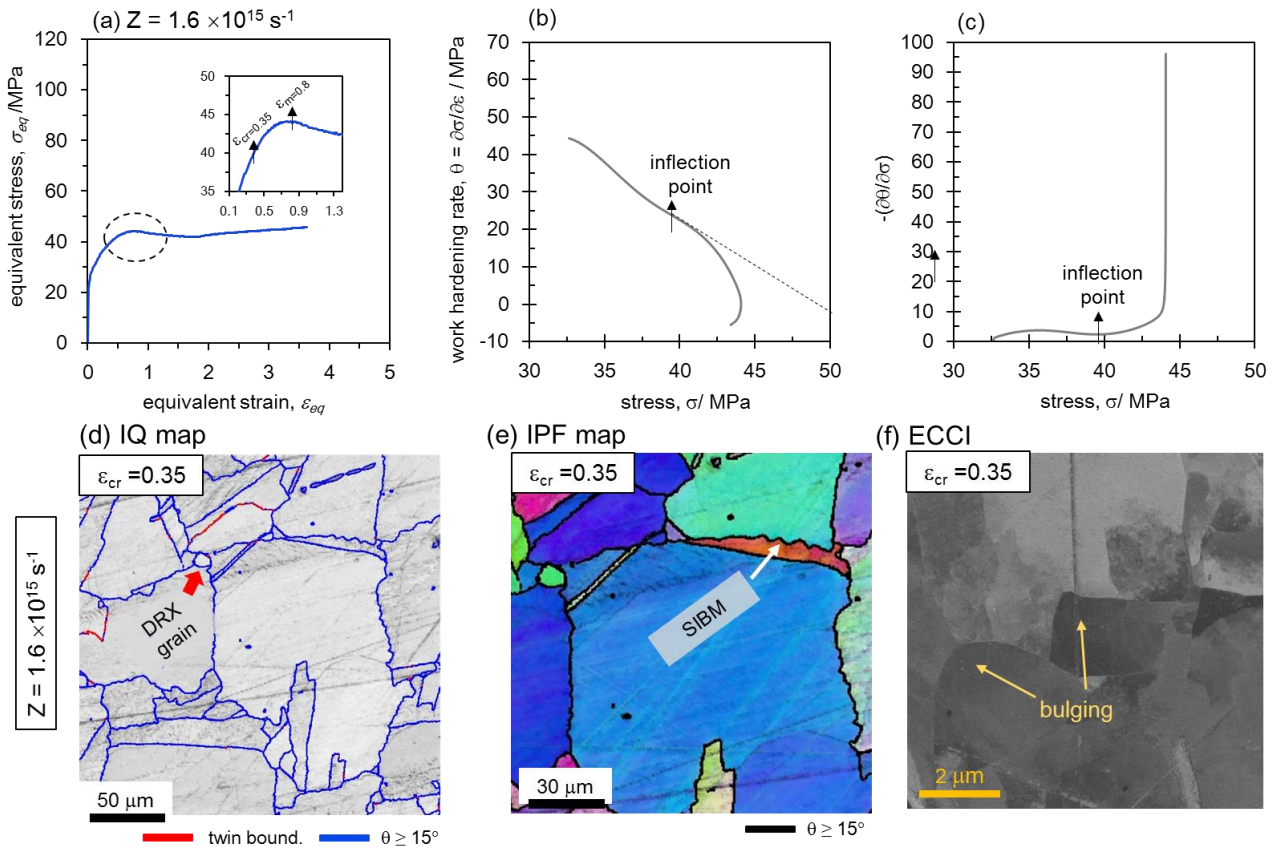


Figure 4.12. (a) Flow stress-strain curve under the deformation condition of $Z = 1.6 \times 10^{15} \text{ s}^{-1}$ (1000 °C/ 0.01 s^{-1}), (b) work hardening rate ($\theta = \partial\sigma/\partial\epsilon$) as a function of stress, and (c) $-(\partial\theta/\partial\sigma)$ as a function of stress to determine the critical strain (ϵ_{cr}) for the initiation of dynamic restoration mechanisms. EBSD IQ map (d) and IPF map (e) of the 304 steel deformed by the critical strain ($\epsilon_{cr} = 0.35$). HABs ($\theta \geq 15^\circ$) in the EBSD maps are illustrated by blue lines in (d) and black lines in (e). (f) ECC image of the substructure at $\epsilon_{cr} = 0.35$.

HABs are illustrated by the blue lines the black lines, respectively. Additionally, electron channeling contrast (ECC) image in **Fig. 4.12f** represents the substructure of the 304 steel at the critical strain of 0.35.

Unlike the IF steel, the microstructure of the 304 steel at this critical strain contained few fraction of DRX grains (**Figs. 4.12-d**), accompanied with the migration of some prior boundaries (strain induced boundary migration: SIBM) as an initiation sign of DRX (**Figs. 4.12-e**). Additionally, the ECC Image (**Fig. 4.12f**) clearly demonstrates bulging of grain boundaries towards interior of another grain.

Figure 4.13 shows the effect of the increasing strain on the microstructure evolution under the same condition ($Z = 1.6 \times 10^{15} \text{ s}^{-1}$). The deformation was stopped at various strains displayed on the flow stress-strain curve (marked by open circles) in **Fig. 4.13a**. Accordingly, the microstructural changes were as follows; at $\varepsilon = 0.6$ before the peak (**Fig. 4.13b**), frequent bulging of initial grain boundaries was found and the fraction of DRX grains (pointed out by a red arrow) was estimated around $f_{NG} \sim 4\%$. It is also noteworthy that $\Sigma 3$ -twin boundaries, of which initial fraction was high in the as-received 304 steel (**Fig. 4.1**), lost their characteristic boundary definition after a strain of 0.6 and became normal HABs. The fraction of DRX grains quickly increased to 25% at $\varepsilon = 0.9$ (**Fig. 4.13c**) and to 66% at $\varepsilon = 1.3$ (**Fig. 4.13d**), resulting in the stress drop on the flow stress. Unlike the low- Z deformed ferrite in the IF steel, the austenitic matrix showed copious nucleation sites normally along initial grain boundaries or at triple junctions. DRX region could quickly expand into deformed (old) grains. Such a process was completed (for one cycle) at $\varepsilon = 1.8$ (**Fig. 4.13e**), where the flow stress reached the steady condition. In other words, the stress-strain curves could well reflect the microstructural changes happening during the deformation.

A more detailed examination of the microstructure evolution under the same deformation condition ($Z = 1.6 \times 10^{15} \text{ s}^{-1}$) is shown in **Fig. 4.14**. IPF maps and corresponding IQ maps illustrate the progress of DRX at various strains. DRX grains formed along the initial grain boundaries at a low strain of 0.6 (**Figs. 4.14a, d**) and expand quickly into grain interiors with increasing the strain (**Figs. 4.14b,e and 4.14c,f**). Bulging of boundaries towards grain interiors could be frequently observed, which is known to driven by dislocation density gradients within the matrix.

Finally, **Fig. 4.15** compares the kinetic of the formation of new grain structures under the deformation conditions of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$ (800 °C/ 0.1 s^{-1}) and $Z = 1.6 \times 10^{15} \text{ s}^{-1}$ (1000 °C/ 0.01 s^{-1}), of which the microstructure evolution was

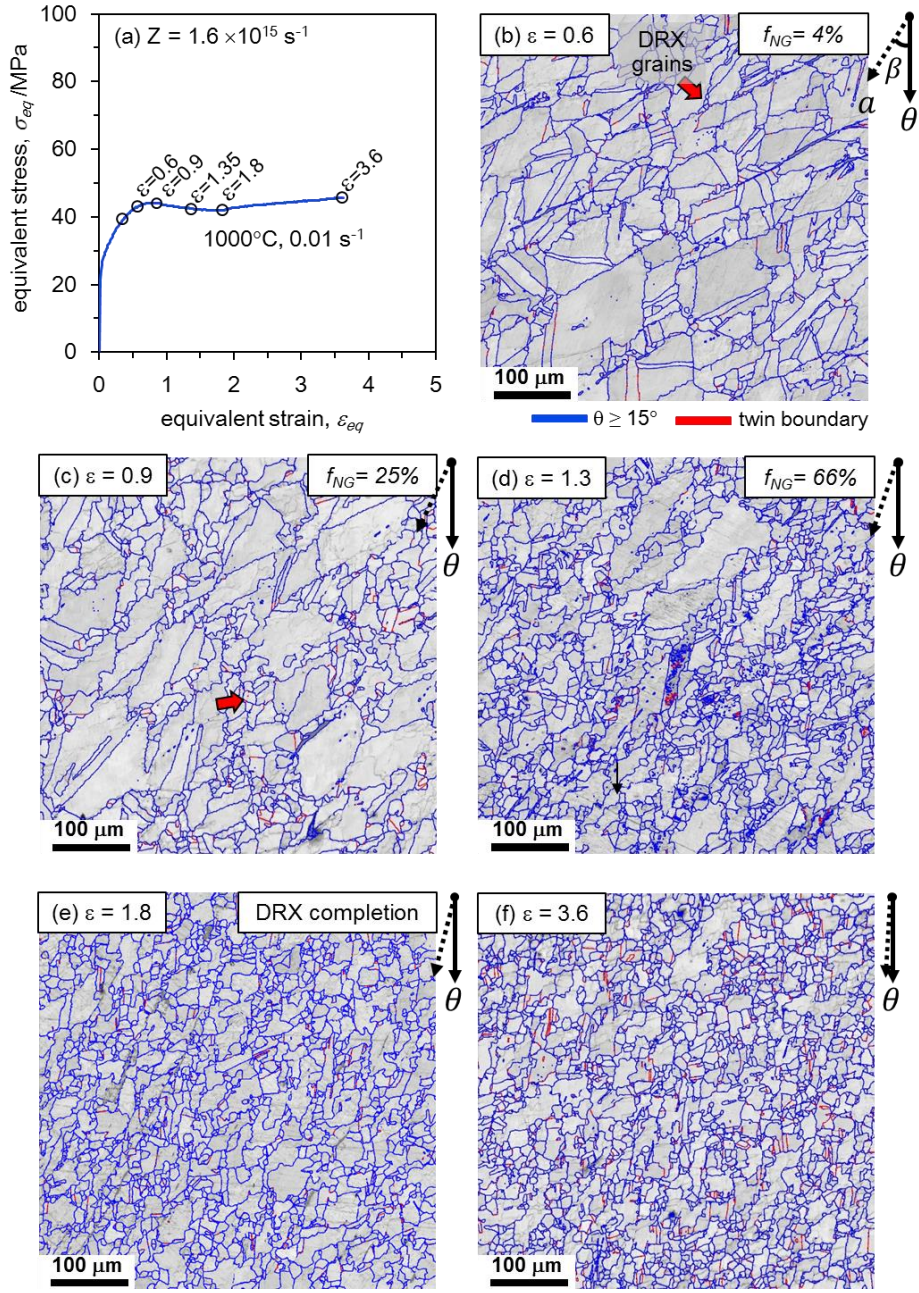


Figure 4.13. (a) Flow stress-strain diagram under the deformation condition of $Z = 1.6 \times 10^{15} \text{ s}^{-1}$ ($1000^\circ\text{C} / 0.01 \text{ s}^{-1}$). Microstructure observations were conducted at different strains indicated by the open circle. (b-f) Representative torsion-deformed microstructures at the strains of (b) $\epsilon = 0.6$, (c) $\epsilon = 1.9$, (d) $\epsilon = 1.3$, (e) $\epsilon = 1.8$, and (f) $\epsilon = 3.6$. In each EBSD map (b-f), HABs (with misorientation $\theta \geq 15^\circ$) drawn by blue lines and $\Sigma 3$ -twin boundaries drawn by red lines superimposed on the image quality (IQ) map. Moreover, β indicates the angle between the shear direction (θ) and the major axis (a) of original grains after the corresponding shear deformation ($\tan 2\beta = 2/\sqrt{3}\epsilon$ ¹⁰). f_{NG} as a fraction of new fine grains which are pointed out by red arrows.

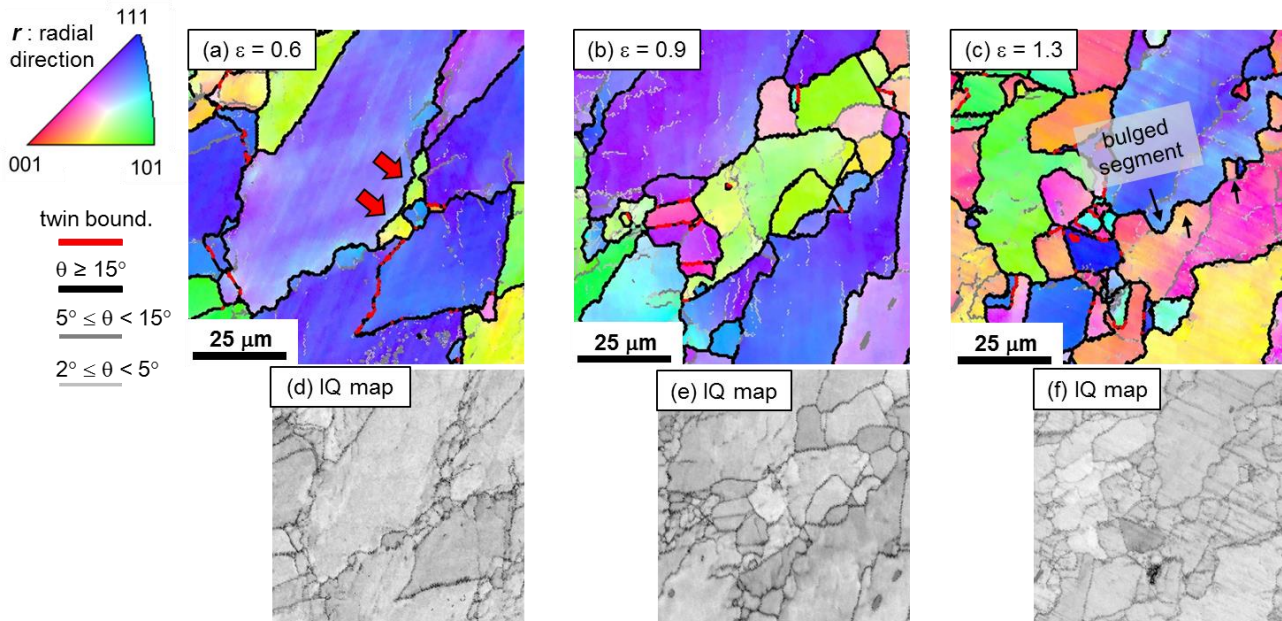


Figure 4.14. Progress of DRX at various strains of (a) $\varepsilon=0.6$, (b) $\varepsilon=0.9$, (c) $\varepsilon=1.3$. (a-c) IPF maps and corresponding (d-f) IQ maps shown as representative microstructures of the 304 steel deformed under the deformation condition of $Z = 1.6 \times 10^{15} \text{ s}^{-1}$ ($1000^\circ\text{C} / 0.01 \text{ s}^{-1}$). In the IPF maps, the black lines show HABs ($\theta \geq 15^\circ$) and the red lines show $\Sigma 3$ twin boundaries while LABs are illustrated by thick ($5^\circ \leq \theta < 15^\circ$) and thin ($2^\circ \leq \theta < 5^\circ$) gray lines. Red arrows point out newly formed equiaxed grains.

presented as the medium Z and low Z conditions, respectively. The fraction of new grain (f_{NG}) or DRX grains is shown as a function of the applied strain. The kinetic showed an Avrami-type behavior [11,12](#), where the formation rate of new grains accelerated after a critical strain and then slowed down. Under the low Z condition, only small amount of strain ($\varepsilon < 2$) was enough to achieve the fully DRX microstructure. At a fix amount of strain, f_{NG} decreased with increasing Z . Under the medium Z , the formation rate of new fine grains was slow so that the new grain structure even at a high strain ~ 5 could not fully develop.

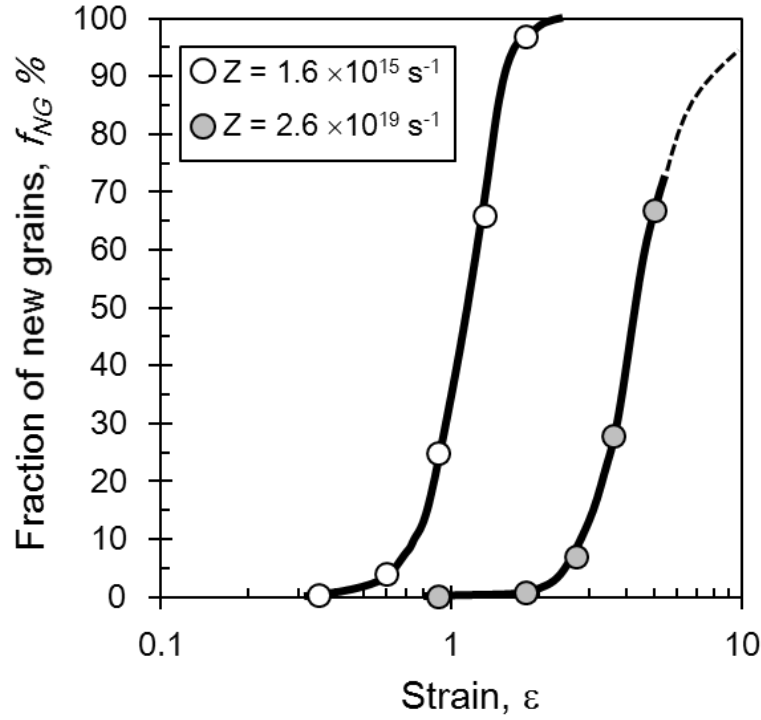


Figure 4.15. Fraction of new grains (f_{NG}) as a function of the applied strain. The 304 steel was deformed under the conditions of $Z = 2.6 \times 10^{19} \text{ s}^{-1}$ (800 °C/ 0.1 s^{-1}) and $Z = 1.6 \times 10^{15} \text{ s}^{-1}$ (1000 °C/ 0.01 s^{-1}), considered as the medium Z condition and the low Z condition, respectively.

4.4. Discussion

4.4.1. Formation of new grains

The SUS 304 austenitic stainless steel was highly (torsion-) deformed under various Z conditions. The materials' behavior was studied in terms of flow stress-strain curve and microstructure evolution. The stress (σ) increased with increasing Z , i.e., with decreasing the temperature and increasing the strain rate (**Fig. 4.3**). This was, however, accompanied with a power-law break down in the $Z = \dot{A} \sigma^{\dot{n}}$ relationship (**Fig. 4.4**). In other words, $\dot{n}$ took different values ($\dot{n}$: 4.5 $\rightarrow$ 9) when shifting from low Z to high Z conditions or vice versa. On the other hand, the grain size was refined with increasing Z , leading to the formation of fine and ultrafine grains (**Fig. 4.5**) with the grain size in a range of 27 μm $\sim$ 0.4 μm . The relationship between the grain size

(D) and the Z parameter, however, could be expressed by an identical power-law relationship; that is, $D \propto Z^{-0.26}$ (**Fig. 4.6**). The grain size was larger than the subgrain size owing to the migration of HABs and grain growth. The power-law break down or saturation in the grain size was not found under the Z conditions applied for the 304 stainless steel in the present study, although it seemed that the grain size started to saturate under the high Z conditions where the grain size approached to the subgrain size (**Fig. 4.6**).

Similar to the IF steel, the σ - D plot (**Fig. 4.7**) was applied as a more straightforward way to understand the microstructure evolution in the 304 stainless steel. With respect to the $\sigma = D^{-N}$ relationship, it could be said that the formation of fine and ultra-fine grains could be categorized in two extreme regions (low Z and high Z) with a transition region (medium Z) in between. However, most of the deformation conditions tried for the 304 steel were found to fall under the conditions of low Z and medium Z .

Under the condition of low Z (or low stress), DRX occurred. The critical strain to initiate DRX, of which the value was low ($\varepsilon_{cr} < 0.5$), could be predicted from the flow stress curve by using the double differentiation method (**Fig. 4.12**). The fully recrystallized structure could be developed at a strain of 1.0 ~ 2.0 (**Fig. 4.13**). This was associated to the onset of steady-state on the flow stress-strain curve. DRX under the low Z conditions could occur very fast and quickly consume the strain-hardened matrix.

Under the condition of medium Z , however, the new grain structure needed very high strains to fully develop (**Fig. 4.9**). The flow stress-strain curve did not show a distinctive peak stress and subsequent stress drop like that found under the condition of low Z . It can be said that the kinetic of DRX as an essential restoration mechanism in the 304 steel became very slow with increasing Z . In other words, the DRX cycle became longer and it could not quickly restore (reset) the strain-hardened matrix. Under such a circumstance, the grain subdivision which is generally a slow process, is expected to have reasonably enough time to cause the fragmentation of the grain interior. As a result, the deformation structure became more and more prominence with increasing the Z parameter. Under the medium Z conditions, the substructures (**Figs. 4.10, 4.11**) showed many geometrically necessary boundaries such as microbands, resulted from the process of grain subdivision. Simultaneously, DRX events could happen; e.g. in the form of boundary bulging into micro-bands (see **Fig.**

4.11b). In other words, a competition between progressive grain subdivision and DRX events seemed to govern the microstructure evolution under the medium Z conditions. Such a mixed microstructure existed until the new grain structure replaced the old grains at high strains (> 5). It might be possible that the grain subdivision itself accelerate the DRX events particularly at higher strains. In other words, the grain subdivision could provide many HAB segments which then could bulge and migrate into the neighboring regions.

Unfortunately, there was a limitation in the present study to deform the 304 stainless steel under higher Z conditions, due to difficulties such as materials' fracture as well as a load limit in the torsion machine. It is of interest to find out how far DRX or related events can be activated in the high- Z plastic deformation. This requires to examine the deformed microstructure at much higher Z (lower temperature) and sufficiently high strains where a steady-state condition could be achieved in the microstructure. In some literature¹⁵⁻¹⁷, DRX-like grains (free of subgrain) were reported even under low-temperature SPD processes such as (interrupted) multidirectional forging of pure copper at 195-273 K^{15,16} and pure titanium at 77K¹⁷. However, the nature of those grains is still a controversial topic.

Based on the aim of this chapter, the 304 austenitic stainless steel was highly deformed in a series of torsion tests carried out under various Z conditions, and the formation processes of new fine/ultrafine grains were studied. **Figure 4.16** schematically summarizes representative microstructures formed under different conditions shown on the ε - Z diagram. The ε - Z diagram is divided into three low Z , medium Z , and high Z regions, although the high Z region could not be confirmed in the present study due to the test difficulties as mentioned before. In the low and the medium Z regions, some representative microstructures (EBSD IPF maps) are provided at low to very high strains. The red cross marks in the ε - Z diagram denote all (torsion) test conditions (various temperature, strain rate, and strain) applied to deform the 304 steel in the present study. Additionally, data associated to the peak strain (ε_p), steady-state strain (ε_s), and critical strain (ε_{cr}) are added to the lower Z region of the ε - Z diagram. Peak strain (ε_p) and steady-state strain (ε_s) correspond to the strains at which flow stress-strain curves (**Fig. 4.3**) reached the maximum stress and the steady-state (after stress drop), respectively. Critical strain (ε_{cr}) for the initiation of dynamic restoration mechanisms (e.g. DRX) was determined by the second derivative method introduced before (**Fig. 4.12**). Two characteristic lines (denoted by

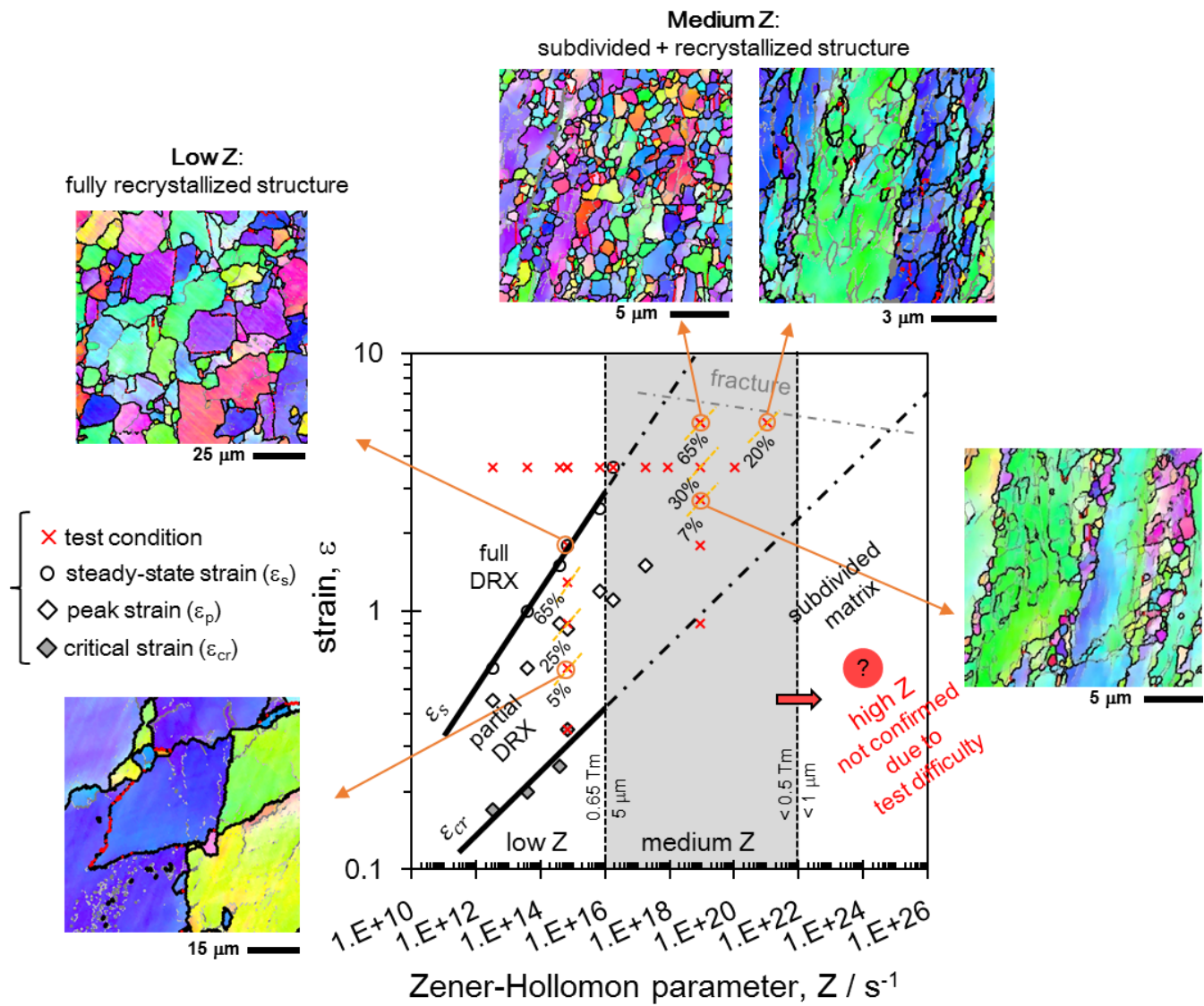


Figure 4.16. $\epsilon - Z$ diagram illustrating the various low Z, medium Z, and high Z regions in the torsion-deformed 304 stainless steel in the present study, although the high Z region could not be confirmed due to the test difficulties. In the low Z and the medium Z regions, some representative microstructures (EBSD IPF maps) are provided at different low to very high strains. Red cross marks denotes various (test) conditions applied for the 304 stainless steel. Additionally, fractions of DRX (new fine) grains in the microstructures are shown for some deformation conditions. The data points associated to ϵ_{cr} (critical strain to initiate DRX), ϵ_p (peak strain on the flow stress-strain curve), and ϵ_s (strain at which the flow stress-strain reaches the steady-state) are also inserted in the lower Z region of the diagram. Two characteristic lines are fitted on the data points associated to ϵ_{cr} and ϵ_s . These two line are also extrapolated into the regions with higher Z.

ϵ_{cr} and ϵ_s) were fitted on the data points associated to ϵ_{cr} and ϵ_s , inside the low Z region in **Fig. 4.16**. As a result, the area bounded between these two characteristic lines indicates the initiation and completion of DRX process, as can be realized from the

representative microstructures in the low Z region. Therefore, at a strain above the ε_s line in **Fig. 4.16**, a fully recrystallized microstructure exists. The characteristic lines were also extrapolated to the other regions having higher Z, even though ε_{cr} and ε_s could not be exactly determined as the shape of the stress-strain curve changed into a work-hardening type. Schematically, it can be seen that the extrapolation of the DRX lines (ε_{cr} and ε_s) took high strain values while entering into the medium Z region, indicating that DRX became a slow process. Therefore, initial grains could be subdivided concurrently, as shown by the representative microstructures in the medium Z region, in which many deformation-induced HABs were generated. A microstructure associated to the high Z region was not available although it can be assumed that the microstructure evolution in the high Z region is essentially governed by the grain subdivision.

4.4.2. Dissimilarities with bcc-ferrite

Metals such as 304 austenitic stainless steel have a low stacking fault energy (SFE) compared to that in ferritic steel. Dynamic recovery in the low SFE metals is known to be slower, causing high dislocation gradients in deformed grains as a driving force for initiation of DRX. On the other hand, DRV is very efficient in the high SFE metals such as bcc-ferritic steel. Additionally, a higher diffusion coefficient associated to the bcc structure can also enhance DRV. As a result, it is generally assumed that DRX is suppressed in the easy-recovery ferrite except in case of higher purity.

Such a difference in dynamic restoration mechanisms (DRV and DRX) was also confirmed in the current study under the low Z conditions, which is summarized in **Table 4.3**. Under the low Z conditions, the stress-strain curve associated to the IF steel did not have a sharp peak as a sign of DRX. Typical DRX microstructure was not found (**Figs. 3.13, 3.14, and 3.17**), as the number of newly formed (DRX) grains was low. The initial (old) grains remained until high strains, became highly elongated so that GDRX could possibly happen. This was also accompanied with a type of texture strengthening in favor of orientation with low Taylor factor (**Fig. 3.16**). On the other hand, the stress-strain associated to the 304 stainless had a sharp peak followed by a stress drop and steady-state. DRX grains were found at a low strain before the peak strain (**Figs. 4.12, 4.13**). DRX microstructure was fully develop at a strain of 1 ~ 2, which corresponded to the steady-state on the strain-strain. The texture in the 304 specimen was weak showing a level of texture randomization as a result of DRX (**Fig.**

4.8).

Table 4.3. Some dissimilarities between the 304 stainless steel and the IF steel during the deformation under the low Z conditions.

types	bcc-ferrite in IF steel	SUS 304 stainless steel
stress-strain curve	Flat type (weak single peak)	sharp single peak
new grain structure	restricted DRX, low density of equiaxed (DRX) grains, possibility of GDRX	copious DRX grains accompanied with bulging along the initial grain boundaries, short DRX cycle
microtexture	texture strengthening in favor of orientations with low Taylor factor	comparatively weak texture

The dissimilarities in the types of microstructure evolution under the low Z conditions could also be realized to some extent under the medium Z conditions. In the IF steel deformed under the medium Z conditions, the deformed grains were subdivided by many deformation-induced boundaries (e.g. deformation bands) (**Fig. 3.6**). Subsequently, the fine equiaxed grains formed as a result of the boundary migration and growth inside the subdivided region. In the 304 steel deformed under the medium Z conditions, similar phenomena; i.e., the grain subdivision accompanied with the boundary migration could happen. Additionally, however, the bulging of HABs as a sign of DRX could happened to some extent, providing some discontinuities in the deformed microstructure (**Figs. 4.11b, d**).

The occurrence of DRX in the 304 steel under the medium Z conditions might also explain the difference in the $\sigma - D$ plots associated to the IF steel (**Fig. 2.13**) and the 304 steel (**Fig. 4.7**). In the IF steel, the medium Z conditions was rather narrow corresponded to a range of stress (σ) around 45 MPa ~ 90 MPa (see **Table 2.5**). Above this range (> 90 MPa), the high Z conditions were found under which the UFG lamellae formed. In the 304 steel, however, the medium Z conditions were expanded within a range of stress around 60 MPa ~ > 230 MPa (see **Table 4.2**). The deformation under the medium Z conditions led to a mixed/complex microstructure evolution, of which it was somehow difficult to determine whether new grains were generated by the progressive increase in the misorientation of deformation-induced boundaries or formed through typical DRX events. In the 304 steel, the high Z

conditions must be shifted towards a higher range of stress, which could not be achieved in the current study due to the limitation of load capacity in the torsion machine.

4.5. Conclusions

The formation of **fine/ultrafine grains** in the high-strain (torsion) deformation under various Z (temperature and strain rate) conditions was studied in a 304 austenitic stainless steel. The followings are the main results and new findings obtained:

A wide range of fine and ultrafine grains with various grain sizes from $\sim 27 \mu\text{m}$ to $\sim 0.4 \mu\text{m}$ were achieved in the highly (torsion-) deformed 304 stainless steel. Under the higher Z conditions, however, the largest strain possible in the torsion deformation (~ 5.4) was not enough to reach the steady-state condition in the microstructure.

With increasing Z ; i.e., decreasing the temperature and increasing the strain rate, material's properties such as **flow stress** (σ) and **grain size** (D) increased and decreased, respectively. With respect to the σ - D relationship ($\sigma = D^{-N}$), the entire Z conditions could be divided into two regions; i.e., (1) the **low Z** or **low σ** region ($N : 0.77$) under which the grain size was larger than the subgrain size indicating the dominance of grain growth (and DRX) in this region, and (2) the **medium Z** or **medium σ** region ($N : 0.46$), under which the grain size was approaching to the subgrain size. The latter is called the medium Z region since it is believed that there must be another region as the **high Z** region, under which the sizes of grain and subgrain should be nearly the same, like that found in the IF steel (Chapter 2 and 3).

During the deformation under the low Z conditions ($< 10^{16} \text{ s}^{-1}$), DRX grains were found along the initial grain boundaries at very low strains ($\varepsilon < 0.5$). The DRX grains could be quickly generated and grow so that a fully recrystallized microstructure was obtained at strains below $1 \sim 2$. The low Z conditions can be considered as the **growth dominant** or **DRX dominant** region.

Under the medium Z conditions ($> 10^{16} \text{ s}^{-1}$), the formation of new grain structure was complicated and required very high strains ($\varepsilon > 5$) to completely replace the initial grains. The newly formed grains were found not only along the initial grain boundaries but inside the initial (deformed) grains. With increasing Z , DRX was delayed so that the grain subdivision could progress and subdivide the grain interiors. In fact, the microstructure evolution could be characterized as **a combination of the grain subdivision and the DRX**.

4.6. References

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Chapter 5: Summary and conclusions

In the present dissertation, ***“formation mechanisms of fine and ultrafine grains in different types of metals highly deformed by torsion at various temperature and strain rate”*** were studied. An interstitial free (IF) steel in the single-phase ferrite region and a SUS 304 austenitic stainless steel were highly deformed in torsion under various Zener-Hollomon ($Z = \dot{\epsilon} \exp(Q / RT)$) conditions; i.e. various temperature and strain rate. Materials' behaviors were investigated in terms of flow stress-strain curves, microstructure evolution (grain size, morphology, etc.), possible mechanisms to generate new fine and ultrafine grained microstructures, and texture. It was found that the formation of various types of microstructures could be categorized under three different deformation conditions of high Z , medium Z , and low Z . Under the conditions of high Z , ultrafine lamellae ($< 1\mu\text{m}$) formed, and the grain subdivision was believed to be the dominant mechanism. Under the conditions of low Z , equiaxed grains ($> 10\mu\text{m}$) formed and the grain growth (and the DRX) became dominant. From high Z to low Z or vice versa, there was the transition of which the microstructure could be considered as a mixture under the medium Z conditions.

The contents of the present dissertation are presented in 5 Chapters:

In **Chapter 1**, different ways of grain refinement in plastic deformation of metals such as low-temperature SPD and hot deformation were reviewed. It was pointed out that the grain refinement under various deformation conditions and related mechanisms, such as grain subdivision in SPD and DRX in hot deformation, have been studied separately in literatures. It was, therefore, concluded that it is necessary to get an universal understanding of the formation of new grain structures under various deformation conditions. Therefore, the present study aims to investigate the formation of fine and ultrafine grains in metals fabricated by the high-strain deformation at different temperatures and strain rates, i.e. under various Z conditions. The current study was carried out in different types of metals; that is, the IF steel in the bcc-ferrite region which is known as an easy-recovery type metal and the SUS 304 stainless steel having fcc-austenitic structure, which is known as a DRX-type metal. A set of high-strain torsion tests were systematically designed to

deform those metals in wide ranges of Z conditions; i.e. $6.8 \times 10^{15} \text{ s}^{-1} \sim 5.7 \times 10^{29} \text{ s}^{-1}$ in the IF steel and $7.3 \times 10^{12} \text{ s}^{-1} \sim 5.7 \times 10^{21} \text{ s}^{-1}$ in the 304 stainless steel. The data associated to the IF steel are presented in two chapters; that is, **Chapter 2** categorizing different types of fine and ultrafine grained ferrite achieved by high strain deformation under various Z conditions and **Chapter 3** explaining how those microstructures categorized in the former chapter gradually evolve with increasing the strain. DRX as one of the mechanisms to generate new fine grains was found to be restricted in the easy-recovery ferrite (IF steel). For comparison, the same analysis was carried out in the 304 austenitic stainless steel, in which DRX is known to occur easily. The results associated to the 304 stainless steel are presented in **Chapter 4**. The main results and new findings in all Chapters 2, 3 and 4 are summarized in this chapter (**Chapter 5**) which are described in the following parts.

Table 5.1 represents a summary of deformation conditions and results (e.g. stress and grain size) found in the IF steel and the 304 stainless steel. In both types of metals, the grain refinement was greatly enhanced by increasing the Z parameter; i.e., decreasing the temperature and increasing the strain rate. Various fine and ultrafine grain structures, of which the grain sizes (D) were in ranges of $46 \mu\text{m} \sim 0.6 \mu\text{m}$ (for the IF steel) and $27 \mu\text{m} \sim 0.4 \mu\text{m}$ (for the 304 stainless steel) were obtained. However, in the 304 stainless steel, the evolution of new grain structures after high strain deformation could not be confirmed under some conditions of higher Z due to the specimen fracture. In general, the fracture strain in the 304 stainless steel was lower than that in the IF steel.

Flow stress–strain curves were obtained using torque-rotation data recorded during the torsion test. Increasing the Z parameter rose the flow stress levels, and gradually changed the shape of the stress-strain curves from a peak-containing curves under low Z to a plateau/work-hardening curves under high Z . Constitutive equations were used to describe the variation of the flow stress (σ) as a function of the Z parameter. However, the relationship could not be expressed by a single function over the wide range of Z in the present study. The stress exponent or n in the $Z \propto \sigma^n$ relationship increased significantly with increasing Z , showing a power-law break down. In other words, the stress apparently became less sensitive to the Z parameter under high Z conditions.

Constitutive equations were also used to describe the variation of the grain size

(D) as a function of the Z parameter. As a result, $D - Z$ relationships could be described as $D \propto Z^{-0.26}$ (-0.35) (for the IF steel) and $D \propto Z^{-0.26}$ (for the 304 steel). However, under very high Z conditions, an apparent saturation in the grain refinement (a power-law break down) was observed in the IF steel. This was concurrent with the change in microstructure morphologies from equiaxed grains to rather lamellar (elongated) structures. The saturation of the grain size, however, could not be confirmed in the 304 steel under the Z conditions conducted in the present study for this metal.

Table 5.1. A summary of the results found in the IF steel and the 304 stainless steel deformed in the high-strain torsion deformation under various conditions.

types	IF steel in the ferrite region	304 austenitic stainless steel
conditions	400°C (0.37T _m) ~ 850°C (0.62T _m) Z: 6.8×10 ¹⁵ ~ 5.7×10 ²⁹ (Q:383 kJmol ⁻¹)	700°C (0.54T _m) ~ 1200°C (0.81T _m) Z: 7.3×10 ¹² ~ 3.2×10 ²¹ (Q:410 kJmol ⁻¹)
flow stress vs. Z	$Z \propto \sigma^{\dot{n}}$	$Z \propto \sigma^{\dot{n}}$
Low Z	$\dot{n} \sim 6$	$\dot{n} \sim 4.5$
Medium Z	$\dot{n} > 6$	$\dot{n} > 4.5$
high Z	$\dot{n} \sim 25 - 30$	Not confirmed.
grain size vs. Z	46 μm ~ 0.6 μm	27 μm ~ 0.4 μm
Low Z	$D \propto Z^{-0.26 \sim -0.35}$	$D \propto Z^{-0.26}$
Medium Z	$D \propto Z^{-0.26 \sim -0.35}$	$D \propto Z^{-0.26}$
high Z	saturation (power-law break down)	Not confirmed.
stress vs. grain size	$\sigma \propto D^{-N}$	$\sigma \propto D^{-N}$
Low Z	N: 0.78 ~ 0.88	N ~ 0.77
Medium Z	N: 0.3 ~ 0.5	N ~ 0.46
high Z	N: 1.0 ~ 1.15	Not confirmed.

A direct relationship between the flow stress (σ) and the grain size (D), both of which are the material's properties, was considered then. Such a relationship might reveal more straightforward view in the microstructure evolution during high strain (torsion) deformation under various conditions. The stress (σ) - grain size (D) plot ($\sigma \propto D^{-N}$) was found to be valuable to determine the different regions (deformation

conditions) under which various types of fine and ultrafine grained microstructures could be obtained. In the IF steel, three conditions of high Z (high σ), medium Z (medium σ), and low Z (low σ) with different N values were found. The medium Z condition could be considered as a transition between the high Z and the low Z conditions. This seemed to be also the case in the 304 steel although the medium Z (medium σ) condition was more expanded in this metal, compared to that in the IF steel. In other words, the boundary between medium Z and high Z conditions shifted towards much higher σ in the SUS 304. Since there was a limitation of load capacity in the torsion machine the high Z condition could not be confirmed in the 304 steel.

Under the high Z condition ($N = 1.0 \sim 1.15$) which was confirmed only in the IF steel, the grain size was found to be nearly the same as the subgrain size, indicating the lack of mobility of HABs and grain growth. With decreasing Z , the grain size became larger than the subgrain size gradually. Therefore, the medium Z condition ($N = 0.3 \sim 0.5$) were realized in both metals. Finally, the low Z condition with ($N = 0.7 \sim 0.9$) were stabilized, under which the boundary migration and grain growth became dominant.

In the IF steel deformed under high Z conditions ($Z > 10^{23} \text{ s}^{-1}$: $\sigma > 90 \text{ MPa}$), deformation-induced boundaries resulted from the grain subdivision mechanism were generated. Increasing the strain increased the fraction of HABs and at sufficiently high strains ($\varepsilon > 5$), the UFG lamellae dominated by elongated HABs formed. The boundary migration was highly constrained within the lamellar microstructures. Under medium Z conditions ($10^{19} \text{ s}^{-1} \sim 10^{23} \text{ s}^{-1}$: $\sigma = 45 \text{ MPa} \sim 90 \text{ MPa}$), initial grains were firstly elongated and subdivided. After some moderate strains ($\varepsilon = 2 \sim 3$), the subdivided structures were replaced by the grains with rather equiaxed shapes, due to local boundary migration of finely fragmented regions. As a result, microstructures at high strains ($\varepsilon > 4 \sim 5$) showed high fractions of fine equiaxed grains uniformly distributed. Under low Z conditions ($Z < 10^{19} \text{ s}^{-1}$: $\sigma < 45 \text{ MPa}$), the degree of grain subdivision was reduced significantly. On the other hand, the DRX seemed to be (not completely but greatly) suppressed due to the enhanced DRV in the bcc-ferrite of the IF steel. Therefore only a small fraction of new equiaxed grains were generated. With increasing the strain, the initial grains became more and more elongated, leading to the formation of low misorientation regions (LMRs) at high strains, accompanying with other phenomena such as GDRX and texture strengthening.

In the 304 stainless steel, high Z conditions and corresponding microstructure

evolution could not be investigated owing to the limitations of the deformability of the material and the test machine. Under medium Z conditions ($10^{16} \text{ s}^{-1} \sim 10^{21} \text{ s}^{-1}$; $\sigma = 60 \text{ MPa} \sim 230 \text{ MPa}$), new grains seems to be generated essentially by the progressive increase in the boundary misorientation caused by grain subdivision, similar to that happened in the IF steel. However, bulging as a sign of DRX was also confirmed under medium Z conditions. The microstructure evolution could be characterized as a combination of the grain subdivision and the DRX mechanisms. On the other hand, the DRX under low Z conditions ($< 10^{16} \text{ s}^{-1}$; $\sigma < 60 \text{ MPa}$) quickly happened in the 304 steel, different from the IF steel. Copious DRX grains along original grain boundaries could be found at very low strains ($\varepsilon < 0.5$). Fully recrystallized microstructures could be obtained at $\varepsilon < 1 \sim 2$, leading to a type of texture randomization. The low Z conditions could be considered as the growth dominant or DRX dominant region.

In this dissertation it was found that the 3-stage variations in the σ - D relationship could be considered as an universal behavior of metals in high-strain plastic deformation under various conditions, regardless of the types of metals. Most importantly, the σ - D relationship can effectively describe how the transition occurs between an ultrafine microstructure resulted from the grain subdivision under high Z conditions and fine equiaxed grains resulted from the dynamic restoration mechanism (DRV, DRX, and grain growth) under low Z conditions.

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