

**Synchrotron Radiation X-ray Diffraction Study on
Microstructural and Crystallographic Characteristics
of Deformation-Induced Martensitic Transformation
in SUS304 Austenitic Stainless Steel**

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Martensitic Transformation in SUS304 Austenitic
Stainless Steel**



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Chapter 1: Background and purpose

1.1 Introduction of martensitic transformation in steels

In recent years, steels have been irreplaceable in our society because of their wide utilization possibility in not only construction, but also traffic, medical care, information technology and so on. It is not hard to imagine that how big the influence would be brought to the world if one can improve the properties of steels. Thus, improving mechanical properties of steels has always been an important challenge. As one of the effective methods, deformation-induced martensitic transformation has been utilized in austenitic stainless steels, because deformation-induced martensitic transformation can greatly improve mechanical properties through transformation-induced plasticity (TRIP) [1-5]. When austenite are cooled down slowly from a high temperature, ferrite phase forms. However, when they are quenched under a certain temperature (M_s temperature), a shear-dominant, lattice distortive and diffusionless transformation happens. This thermally induced transformation observed in quenched steels is called martensitic transformation, and the phase is called martensite in honor of the German metallurgist, Professor A. Martens. Later, it is found that martensitic transformation can also occurs even above M_s temperature when apply deformation to austenite. This is the so-called deformation-induced martensitic transformation. Many studies have been carried out on deformation-induced martensitic transformation for decades. However, there are still many unclear parts, such as variant selection etc.

In this section, two typical kinds of thermally induced martensitic transformation in steels are briefly introduced.

1.1.1 $\gamma \rightarrow \alpha'$ martensitic transformation

In $\gamma \rightarrow \alpha'$ martensitic transformation, austenite (γ) which shows a face-centered cubic (FCC) structure transforms into α' martensite which shows a body-centered cubic (BCC) structure. The following orientation relationships are found between austenite and α' martensite:

Nishiyama-Wassermann orientation relationship (N-W relationship)

$$(111)_{\gamma} // (011)_{\alpha'}, \quad [\bar{1}\bar{1}2]_{\gamma} // [0\bar{1}1]_{\alpha'}. \quad (1.1)$$

Kurdjumov-Sachs orientation relationship (K-S relationship)

$$(111)_{\gamma} // (011)_{\alpha'}, \quad [\bar{1}01]_{\gamma} // [\bar{1}\bar{1}1]_{\alpha'}. \quad (1.2)$$

N-W relationship is often observed in Fe-Ni alloys in which lenticular martensite is formed [6,7]. When martensite satisfies the N-W relationship with respect to austenite, twelve different α' crystals can form from a single grain of austenite. Each martensite crystal is called a *variant*. On the other hand, K-S relationship is often observed in low carbon steel, austenitic stainless steel etc., which shows lath martensite [8-10]. In the K-S relationship, twenty-four variants can form from a single grain of austenite.

Bain proposed the first model for FCC-BCC transformation [11]. **Figure 1.1** shows two unit cells of FCC crystals indicated by blue balls. Inside the FCC crystals, there is a body-centered tetragonal (BCT) unit, indicated by red balls. A BCC unit cell can be derived from the BCT unit by contracting $\langle 001 \rangle_{\gamma}$ about 21% and uniformly expanding the other two $\langle 001 \rangle_{\gamma}$ about 12%. This is the so-called “*Bain correspondence*”. The orientation relationship according to Bain correspondence is as follows:

$$\begin{aligned} [001]_{\gamma} & // [001]_{\alpha}, \\ [1\bar{1}0]_{\gamma} & // [100]_{\alpha}, \\ [110]_{\gamma} & // [010]_{\alpha}. \end{aligned} \quad (1.3)$$

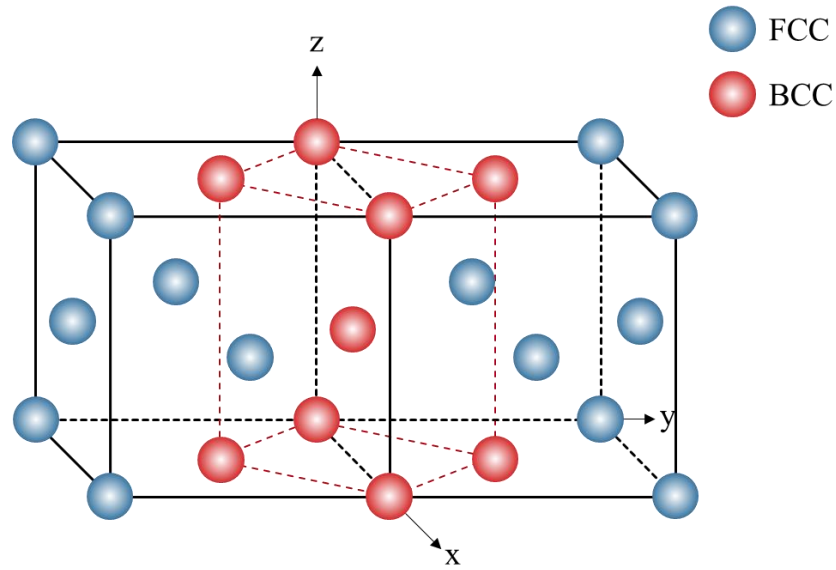


Figure 1.1 Bain correspondence in the transformation of FCC-BCC.

The Bain correspondence is considered to be the most intuitive transformation path for FCC-BCC transformation by giving the austenite a homogeneous lattice-distortive strains, also known as *Bain strain*. **Figure 1.2(a)** shows an undeformed austenite as a sphere with a diameter xy ($= st$) in three dimensions. As a result of Bain strain, the austenite is

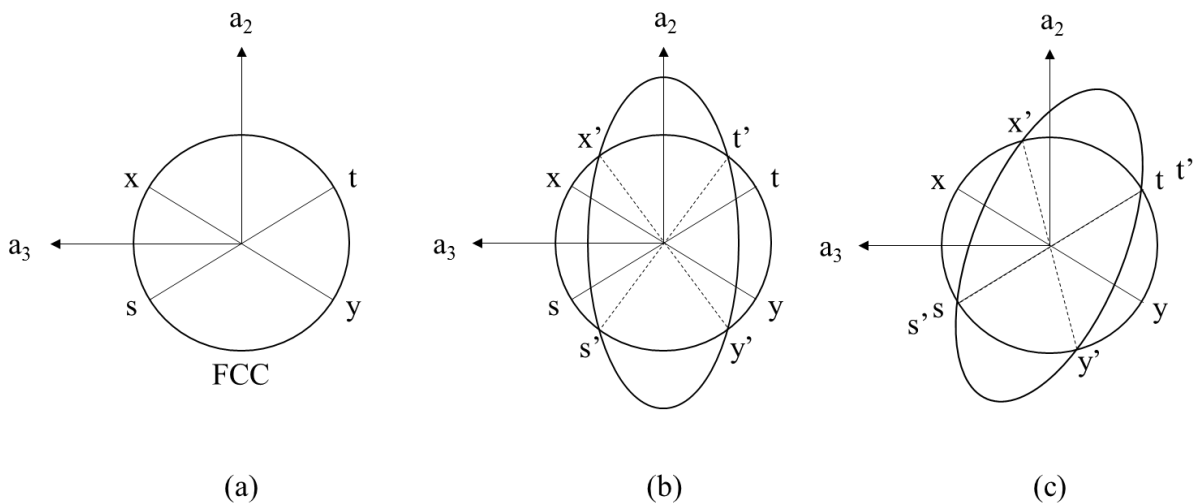


Figure 1.2 (a) Undeformed austenite structure, (b) austenite structure deformed by Bain strain, and (c) austenite structure deformed by Bain strain with a rigid body rotation.

deformed into an ellipsoid shape as shown in **Fig. 1.2(b)**. The lines xy and st are undistorted, but are rotated to the new positions $x'y'$ and $s't'$. This means that there are no lines in the $(100)_\gamma$ plane which are left undistorted or unrotated. However, if a rigid body rotation is added to the Bain strain as shown in **Fig. 1.2(c)**, st can be brought to $s't'$. This indicates that combination of Bain strain and rigid body rotation results in an invariant-line strain. Nevertheless, there is no possible rotation which makes an invariant plane because two non-parallel invariant lines are required. Martensitic transformation has been treated as invariant plane strain, because there is always an undistorted and unrotated plane, i.e. the invariant plane, in the observed shape deformation accompanying the transformation. Therefore, it can be said that FCC-BCC transformation cannot be explained only by Bain correspondence.

The phenomenological theory of martensite crystallography (PTMC) proposed by Wechsler, Lieberman and Read, and Bowles and Mackenzie gives us the solution for obtaining invariant plane in martensitic transformation [12-15]. In the PTMC, the deformation and rotation involved in martensitic transformation is described by 3×3 matrices, so that martensitic transformations can be analyzed numerically by matrix algebra. As described above, the Bain strain $\mathbf{B}$ converts the structure of the parent phase into that of the product phase. When an appropriate rigid body rotation $\mathbf{R}$ is added, the homogeneous lattice deformation $\mathbf{RB}$ only provides an invariant-line strain. However, by adding an adequate lattice-invariant shear $\mathbf{S}$, the shape deformation accompanying martensitic transformation $\mathbf{F}$ can be an invariant plane strain;

$$\mathbf{F} = \mathbf{RBS} \quad (1.4)$$

The crystallography calculated according to **Eq. 1.4** agrees well with experimentally observed results of martensite with habit plane $\{259\}_\gamma$ and $\{3\ 10\ 15\}_\gamma$, but can not explain that of $\{111\}_\gamma$ and $\{557\}_\gamma$. Ross and Croker expanded the original theory from one shear

to two shears as follows [16]:

$$\mathbf{F} = \mathbf{RBS}_1\mathbf{S}_2 \quad (1.5)$$

where $\mathbf{S}_1$ and $\mathbf{S}_2$ are two independent lattice invariant shears. By applying this theory, martensite with habit plane being $\{111\}_\gamma$ and $\{557\}_\gamma$ can be explained. Since $\mathbf{F}$ corresponds to an invariant plane strain, it could also be written as follows:

$$\mathbf{F} = \mathbf{I} + \mathbf{m}\mathbf{d}\mathbf{p}' \quad (1.6)$$

where $\mathbf{I}$ is the unit matrix, $\mathbf{m}$ is the magnitude of the shape deformation, $\mathbf{d}$ is the column vector denoting the shape deformation direction and $\mathbf{p}'$ is the row vector designating the normal to the invariant plane (habit plane).

The PTMC can account for the crystallographic features of martensite in steels. However, it is a purely mathematical approach. For explaining the process of FCC-BCC transformation, another model is proposed by Bogers and Burgers [17]. **Figure 1.3** shows a Bain correspondence between (a) FCC and (b) BCC structure, where two of the close-packed $\langle 110 \rangle_\gamma$ directions on the horizontal plane are indicated by red broken lines and the other four are indicated by green broken lines.

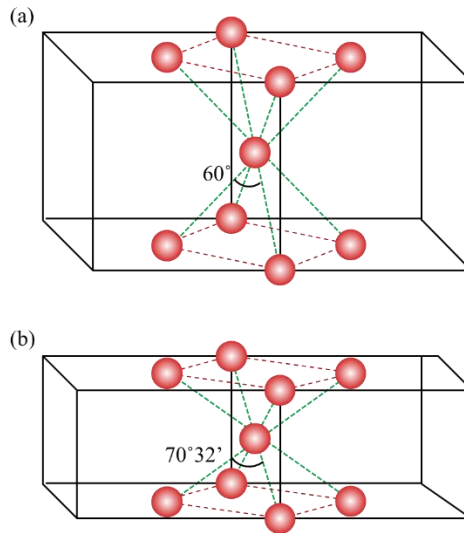


Figure 1.3 Bain correspondence between (a) FCC and (b) BCC structure. Two of the close-packed $\langle 110 \rangle_\gamma$ directions on the horizontal plane are indicated by red broken lines and the other four are indicated by green broken lines.

the other four are indicated by green broken lines. The FCC-BCC transformation can be described as follows: among the six close-packed directions $\langle 110 \rangle_\gamma$, four are retained in the BCC lattice as $\langle 111 \rangle_{\alpha'}$ directions (green broken lines), whereas the other two are elongated (red broken lines). Furthermore, the angle between two adjacent close-packed directions indicated by green broken lines increases from 60° to $70^\circ 32'$ due to the transformation. **Figs. 1.4(a)** shows an FCC tetrahedral arrangement with four $\{111\}_\gamma$ planes represented by a pile of hard spheres. By moving the successive layers parallel to the octahedral plane PVQ over one third of a normal twinning shear with a slip vector of $\frac{a_{fcc}}{18} \langle 112 \rangle$ perpendicular to PV, the pile is transformed into the arrangement shown in **Fig. 1.4(b)**. At the same time, the cross section of the $\{110\}_\gamma$ plane STQ perpendicular to PV shown in **Fig. 1.4(c)** changes into the one shown in **Fig. 1.4(d)**. As can be seen from the black dash line drawn on the top of **Figs. 1.4(c) and (d)**, by applying this shear to the tetrahedral arrangement, the distance between the invariant $\{111\}_\gamma$ planes PVQ and PVS increased slightly. On the other hand, the other two $\{111\}_\gamma$ planes SVQ and SPQ changed their shape. One of the six $\langle 110 \rangle_\gamma$ directions SQ became longer and the others remained the same length. Furthermore, the angles between two adjacent close-packed directions mentioned in **Fig. 1.3** changed from 60° to $70^\circ 32'$ in $S'V'Q'$ and $S'P'Q'$, which means that these two planes have obtained the exactly same shape of $\{110\}_{\alpha'}$ planes in BCC lattice. However, the stacking of these planes, as shown in **Fig. 1.4(e)** is different from that in BCC lattice in **Fig. 1.4(f)**. In order to complete the transition into the BCC lattice, the second shear of $\frac{a_{fcc}}{16} \langle 112 \rangle$, is applied on these planes. The planes parallel to $S'V'Q'$ are moved over each other in the direction perpendicular to $S'Q'$ to attain the correct stacking order. Then the two planes $P'V'Q'$ and $P'V'S'$, which was invariant after the first shear, also changed into the shape of $\{110\}_{\alpha'}$ planes.

Although FCC can successfully transformation into BCC by this sequence, it is only a hypothesis model without solid experimental evidence.

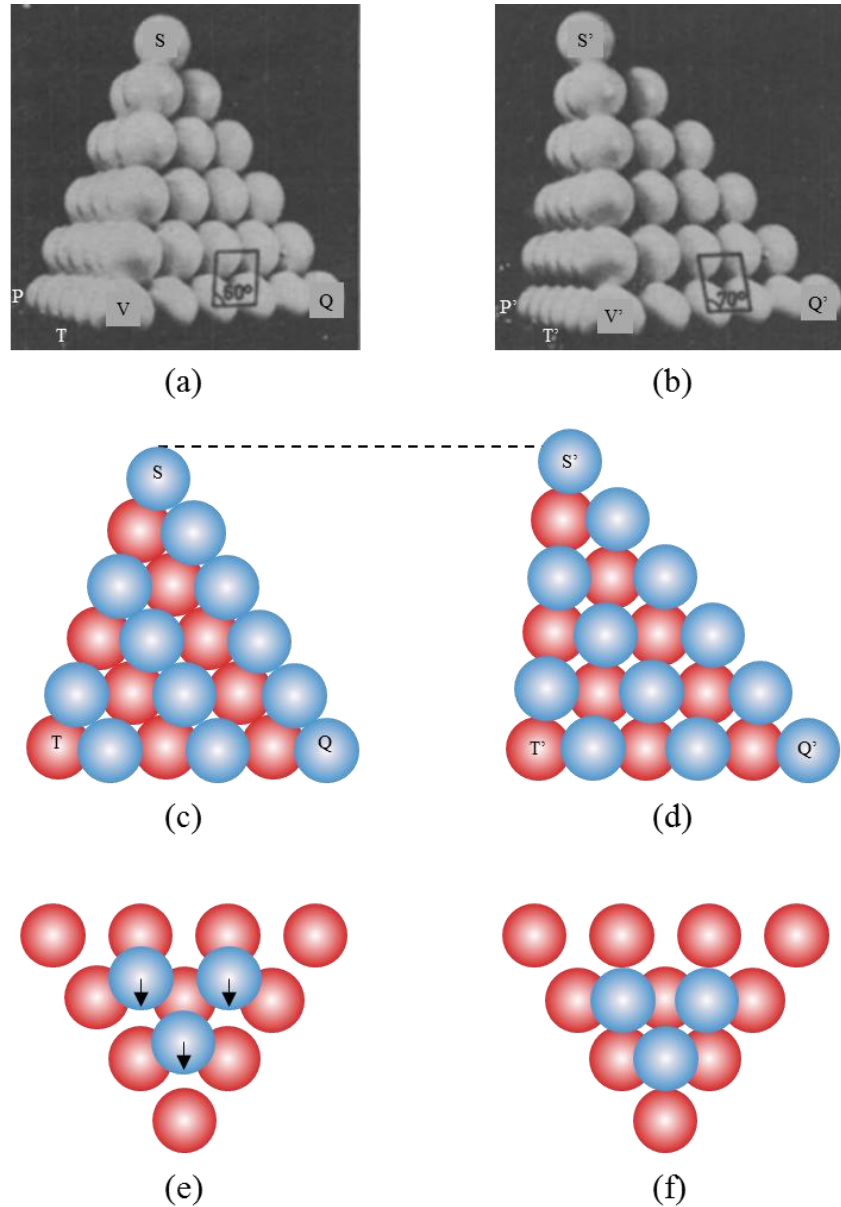


Figure 1.4 (a) An FCC tetrahedral arrangement with four $\{111\}_\gamma$ planes . (b) The intermediate shape of (a) after the first shear of $a_{fcc}/18\langle 112 \rangle$ perpendicular to PV. (c, d) The cross section of the $\{110\}_\gamma$ plane of (a, b), respectively. (e) Stacking of planes parallel to $S'V'Q'$ or $S'P'Q'$. (f) Stacking of correct $\{110\}_{\alpha'}$ planes in BCC lattice.

1.1.2 $\gamma \rightarrow \epsilon$ martensitic transformation

In high Mn steels and stainless steels, austenite sometimes transforms into ϵ martensite with hexagonal closed packed (HCP) structure [18-20]. Because both FCC and HCP structures are close-packed structure, the atomic arrangements of $(111)_\gamma$ and $(0001)_\epsilon$ are identical to each other. Therefore, the lattice correspondence is geometrically simple compared with FCC-BCC transformation.

Figures 1.5(a) and (b) show the basic crystal structures of FCC and HCP, respectively. The atomic arrangement viewed along the direction perpendicular to the close-packed planes is shown in **Figs. 1.5(c) and (d)**. The first, second and third layers are represented by red blue and green colors, respectively. The stacking of FCC structure is ABCABC... While that of HCP structure is ABAB... **Figure 1.6** is a schematic illustration of Thompson tetrahedron in FCC. According to **Fig. 1.6**, the shifts of $\frac{a}{6}[11\bar{2}]_\gamma$, $\frac{a}{6}[1\bar{2}1]_\gamma$ and $\frac{a}{6}[\bar{2}11]_\gamma$ can let the atoms on the third layer go to the same position with the first layer. This means that the FCC-HCP transformation can occur by movement of $\frac{a}{6}[11\bar{2}]_\gamma$, $\frac{a}{6}[1\bar{2}1]_\gamma$ or $\frac{a}{6}[\bar{2}11]_\gamma$ directions on every other $\{111\}_\gamma$ plane. In addition, each of the three shifts on $\{111\}_\gamma$ plane forms same variant. Therefore, four ϵ martensite variants can form from a single grain of austenite.

The reported orientation relationship between austenite and ϵ martensite is:

$$(111)_\gamma // (0001)_\epsilon, \quad [1\bar{1}0]_\gamma // [11\bar{2}0]_\epsilon. \quad (1.7)$$

which is called Shoji-Nishiyama orientation relationship (S-N relationship) [21].

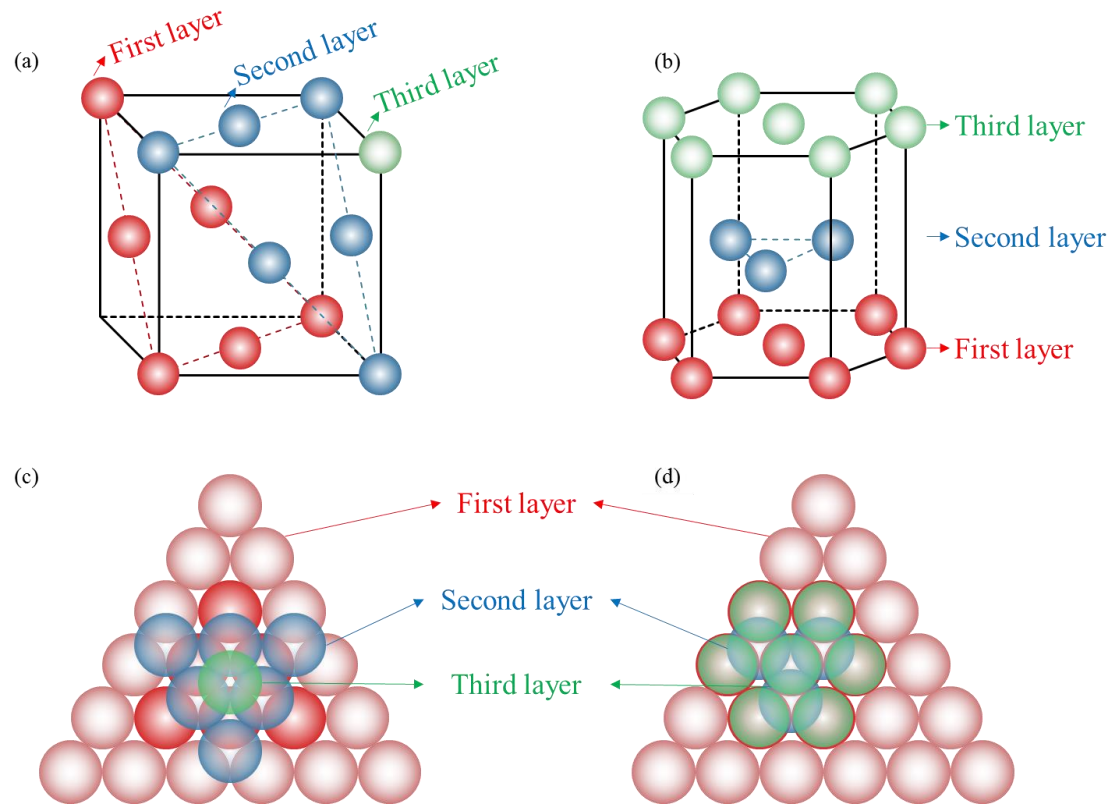


Figure 1.5 Crystal structure of (a) FCC and (b) HCP. Atom arrangement of the close-packed plane of (c) FCC and (b) HCP.

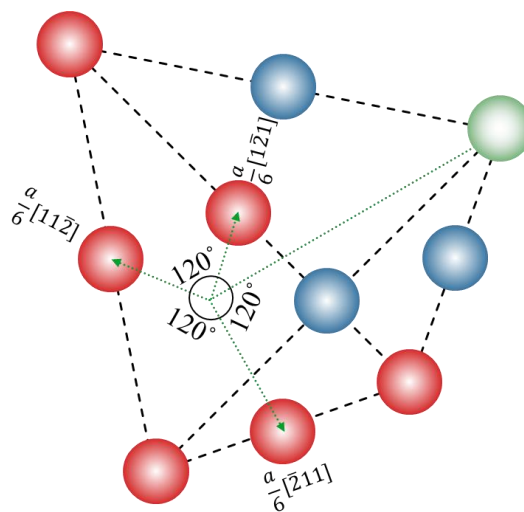


Figure 1.6 Three Shockley partial dislocation on $\{111\}_\gamma$.

1.2 Deformation-induced martensitic transformation in austenitic stainless steel

1.2.1 General introduction of deformation-induced martensitic transformation

As explained in **Section 1.1**, martensitic transformation in steels usually occurs when austenite is rapidly cooled down below a certain temperature, which is called M_s temperature. Meanwhile, martensitic transformation can also happen even above M_s temperature when deformation is applied to austenite. This type of martensitic transformation is termed “deformation-induced martensitic transformation” [3].

Deformation-induced martensitic transformation can be divided into two types. The one occurs before austenite yielding is termed “stress-induced martensitic transformation”, while the one occurs after austenite yielding is termed “strain-induced martensitic transformation”. Stress-induced martensitic transformation is deeply related with superelasticity and shape memory effect. So far, thermally and stress- induced martensitic transformation has not been well studied in austenitic stainless steels because the stability of austenite is very high so that thermally/stress- induced martensitic transformation rarely occurs. On the other hand, strain-induced martensite, which is directly related with transformation-induced plasticity (TRIP) effect, has been extensively studied in austenitic stainless steels because it can improve both strength and ductility simultaneously [1-5].

Although many researches have been carried out on deformation-induced martensitic transformation in austenitic stainless steel, there are many conflicting results among them. For example, Das et al. reported that the $\gamma \rightarrow \epsilon \rightarrow \alpha'$ and $\gamma \rightarrow \epsilon$, $\gamma \rightarrow \alpha'$ transformations were

all observed in a 304LN stainless steel during tensile deformation at different strain rates at room temperature [22]. Shen et al. suggested that the transformation process of stress-induced martensitic transformation was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$, while the strain-induced one occurred via deformation twinning of austenite to martensite in 304L steels [23]. Many other groups agreed that ε martensitic transformation occurs as an intervene transformation between γ and α' in SUS304 steels [24-27]. In contrast, some researches did not observed the ε transformation during deformation of SUS304 steels or other TRIP steels [28-30].

1.2.2 Variant selection in deformation-induced martensitic transformation

As explained previously, several variants of martensite can form from a single grain of austenite phase. Usually, all variants can be observed in thermally induced martensitic transformation. In the case of deformation-induced martensitic transformation, on the other hand, only limited variants are observed. This is the so-called variant selection. Variant selection, known as a key character of deformation-induced martensitic transformation, has been studied a lot. Kato et al. and Higo et al. applied Bogers-Burgers model on analyzing variant selection in single crystals of Fe-Ni-Cr and Fe-Cu alloys by calculating the Schmid factor of the applied stress on two kinds of shear in Bogers-Burgers model. Their results suggested that the variants with positive large Schmid factors on the first shears formed preferentially [31-34]. Other theories/models have also been reported, such as dislocation reaction model proposed by Sum and Jonas [35], the effect of prestrain on variant selection in Fe-Ni single crystal reported by Bokros and Parker, Durlu and Christian [36, 37]. However, their validity have not been sufficiently confirmed. So far, the Patel and Cohen theory seems to have most attention among most models.

Patel and Cohen model, considers the mechanical work (U) by external stress on martensitic transformation [38]. **Figure 1.7** shows the schematic illustration of shape deformation accompanying martensitic transformation. As can be seen in **Fig. 1.7**, the shape deformation can be resolved in the normal component ϵ_0 and the shear stress γ_0 . The mechanical work done by the applied stress for the martensitic transformation can then be calculated by the equation shown below.

$$U = \sigma_n \epsilon_0 + \tau \gamma_0 \quad (1.8)$$

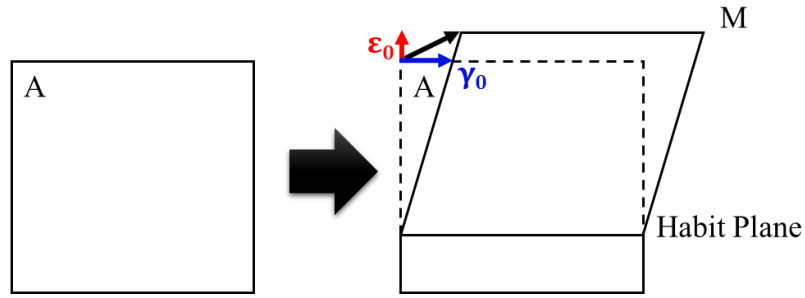


Figure 1.7 Schematic illustration of shape deformation from austenite to martensite.

where σ_n is the resolved normal stress perpendicular to the habit plane, and τ is the resolved shear stress along the shear direction on the habit plane. It is considered that the ones with larger positive mechanical work are expected to form preferentially. Kundu et al. [39, 40] and Viana et al. [41] reported their results were strongly consistent with Patel and Cohen theory. However, in our previous study, neither Patel and Cohen model nor Bogers-Burgers model could explain the variant selection observed in strain-induced martensitic transformation in SUS304 austenitic stainless steel [42]. One of the possible reasons is that the specimen was polycrystalline material. It is easily imagined that local stress of each grains was inhomogeneous and not the same as the macroscopic applied tensile stress used for the analysis.

1.3 Motivation of the dissertation

It is well-known that the strength and ductility of materials are usually in a trade-off relationship with each other. While as explained above, deformation-induced martensitic transformation is one of the promising way to improve both strength and ductility simultaneously via TRIP effect. If we can improve TRIP effect by controlling the formation of deformation-induced martensite, better mechanical properties and wider utilization of austenitic stainless steels can be expected. To make it possible, the nature of deformation-induced martensitic transformation should be clarified. As introduced in the previous sections, some subjects about deformation-induced martensite in austenite stainless steel still remains unclear.

1.4 Purpose and outline of the dissertation

The main purposes of the present dissertation are

(1) to reveal the differences among thermally induced, stress-induced and strain-induced martensitic transformation.

(2) to reveal the transformation sequence of martensitic transformation.

(3) to clarify the variant selection.

In the present dissertation, the microstructure and crystallographic characters of deformation-induced martensitic transformation in SUS304 austenitic stainless steel is studied by means of electron backscattering diffraction (EBSD). At the same time, synchrotron radiation X-ray diffraction is utilized to investigate the relationship between variant selection and local stress field.

The present dissertation is composed of six chapters.

Chapter 1 is “*Background and purpose*”, where the introduction of thermally and deformation- induced martensitic transformation are described. The motivation, purpose and outline of the study are also stated in this chapter.

Chapter 2 is “*Thermally induced martensitic transformation in SUS304 austenitic stainless steel*”, where the microstructure and crystallographic features of thermally induced martensitic transformation are investigated mainly by EBSD analysis.

Chapter 3 is “*Stress-induced martensitic transformation in SUS304 austenitic stainless steel*”, where the microstructure and crystallographic features of stress-induced martensite are investigated by EBSD. Variant selection of stress-induced martensitic transformation is analyzed based on the local stress field measured by synchrotron radiation X-ray diffraction.

Chapter 4 is “*Strain-induced martensitic transformation in SUS304 austenitic stainless steel*”, where the microstructure and crystallographic features of strain-induced martensite are studied by EBSD. Variant selection of stress-induced martensitic transformation is analyzed based on the local stress field measured by synchrotron radiation X-ray diffraction. At last, the comparison among thermally, stress- and strain-induced martensite is carried out.

Chapter 5 is “*Summary and conclusions*”, where the main results and final conclusion and remarks obtained from each chapter are summarized.

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Chapter 2: Thermally induced martensitic transformation in SUS304 austenitic stainless steel

2.1 Introduction

As explained in **Chapter 1**, deformation-induced martensitic transformation, which can improve both strength and ductility through transformation-induced plasticity effect [1-6], could happen when deformation is applied to metastable austenite above M_s temperature. Numerous researchers have studied morphology, nucleate site, and mechanical properties of deformation-induced martensitic transformation [7-11]. To understand the nature of deformation-induced martensitic transformation, the comparison between thermally induced martensitic transformation and deformation-induced martensitic transformation of the same material should be necessary.

The transformation of lath martensite in low carbon steels is characterized as a grain subdivision of a prior austenite grain. As explained in **Chapter 1**, 24 martensite variants with different orientation can form from an austenite grain as shown in **Table 2.1**. According to Marder and Krauss [12], an austenite grain is divided into packets, each of which consists of martensite variants with the same parallel plane relationship in the K-S orientation relationship. As shown in **Table 2.1**, there are four packets in each austenite grain, that is V1 - V6, V7 - V12, V13 - V18, V19 - V24. A packet is further subdivided into blocks, each one which consists of two martensite variants with small misorientation, such as V1-V4, V2-V5, V3-V6.

It has been reported that morphology of martensite in austenitic stainless steels is different from that in low carbon steels. Matsumura et al. reported that the thermally induced carbon-added austenitic stainless steels showed a band morphology composed of

ε and α' martensite [13]. They found that α' martensite grains consisting of six variants aligned on a straight line and ε martensite existed between α' martensite grains. According to their experimental results, they insisted that the transformation sequence was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. Brooks et al. reported, via *in situ* high voltage transmission electron microscopy, that the formation of both thermally and deformation-induced martensite nucleation occurred in association with dislocation motion on $\{111\}_{\gamma}$ slip planes in some stainless steels [14]. In contrast to Matsumura et al., they claimed that both ε and α' were transformed directly from γ . Although several papers investigated thermally induced martensitic transformation in stainless steels, the transformation sequence, morphology, orientation relationship, variant selection, etc. have yet to be understood. Therefore, this chapter aims to reveal the morphology, crystallographic features of thermally induced martensitic transformation in SUS304 austenitic stainless steel.

Table 2.1 24 variants in K-S orientation relationship.

Variant	Close-packed plane	Close-packed direction
No.	(Austenite)//(Martensite)	[Austenite]//[Martensite]
V1	(1 1 1)//(0 1 1)	[-1 0 1]//[-1 -1 1]
V2	(1 1 1)//(0 1 1)	[-1 0 1]//[-1 1 -1]
V3	(1 1 1)//(0 1 1)	[0 1 -1]//[-1 -1 1]
V4	(1 1 1)//(0 1 1)	[0 1 -1]//[-1 1 -1]
V5	(1 1 1)//(0 1 1)	[1 -1 0]//[-1 -1 1]
V6	(1 1 1)//(0 1 1)	[1 -1 0]//[-1 1 -1]
V7	(1 -1 1)//(0 1 1)	[1 0 -1]//[-1 -1 1]
V8	(1 -1 1)//(0 1 1)	[1 0 -1]//[-1 1 -1]
V9	(1 -1 1)//(0 1 1)	[-1 -1 0]//[-1 -1 1]
V10	(1 -1 1)//(0 1 1)	[-1 -1 0]//[-1 1 -1]
V11	(1 -1 1)//(0 1 1)	[0 1 1]//[-1 -1 1]
V12	(1 -1 1)//(0 1 1)	[0 1 1]//[-1 1 -1]
V13	(-1 1 1)//(0 1 1)	[0 -1 1]//[-1 -1 1]
V14	(-1 1 1)//(0 1 1)	[0 -1 1]//[1 -1 1]
V15	(-1 1 1)//(0 1 1)	[-1 0 -1]//[-1 -1 1]
V16	(-1 1 1)//(0 1 1)	[-1 0 -1]//[-1 1 -1]
V17	(-1 1 1)//(0 1 1)	[1 1 0]//[-1 -1 1]
V18	(-1 1 1)//(0 1 1)	[1 1 0]//[-1 1 -1]
V19	(1 1 -1)//(0 1 1)	[-1 1 0]//[-1 -1 1]
V20	(1 1 -1)//(0 1 1)	[-1 1 0]//[-1 1 -1]
V21	(1 1 -1)//(0 1 1)	[0 -1 -1]//[-1 -1 1]
V22	(1 1 -1)//(0 1 1)	[0 -1 -1]//[-1 1 -1]
V23	(1 1 -1)//(0 1 1)	[1 0 1]//[-1 -1 1]
V24	(1 1 -1)//(0 1 1)	[1 0 1]//[-1 1 -1]

2.2 Experimental procedures

An austenitic stainless steel SUS304 was used in the present study. The chemical composition of the material is shown in **Table 2.2**. The as-received material was in sheet form with a thickness of 1 mm. First, the as-received materials were annealed at 1000 °C for 3 hours, and furnace cooled to obtain a microstructure with γ single phase. The annealed sheets were cold rolled by 10% to reduce the thickness to 0.9 mm. Then, the specimens were mechanically polished to reduce the thickness to 0.5 mm. Then again annealed at 1050 °C for 15 hours under argon atmosphere, and furnace cooled. A fully-recrystallized homogeneous microstructure of γ single phase was obtained by this heat treatment. The mean grain size of the specimen after annealing was 190 μm without considering annealing twin. In order to avoid deformation-induced martensitic transformation during mechanical polishing, the specimen was polished electrolytically in a solution of 10% HClO_4 + 90% CH_3COOH . The electrolytic voltage is 12V and the polishing time was 30 ~ 50 minutes. The final thickness of the specimen after the electrolytic polishing was 0.25 mm. The specimens were subzero cooled in liquid nitrogen for 20 min to obtain thermally induced martensite.

Table 2.2 Chemical composition of the studied SUS304 in present research (wt%).

C	Si	Mn	P	S	Ni	Cr	Fe
0.06	0.63	1.02	0.027	0.003	8.07	18.19	Bal.

The microstructure was observed by optical microscopy. The phase identification and orientation analysis were performed by EBSD measurements using a field emission scanning electron microscope (FE-SEM, FEI: XL30Sirion). The EBSD measurements were carried out with a working distance of 15mm and an accelerate voltage of 15V. TSL-

OIM Data Collection (Ver. 5.31) and TSL-OIM analysis (Ver. 5.0) were used to collect and analyze the EBSD data, respectively. The obtained data with a confidence index (CI) value more than 0.1 were used for analysis.

2.3 Results

2.3.1 Microstructure observations

Figure 2.1 shows (a) an optical microscopy image and (b) an EBSD phase map of the annealed specimen. In the EBSD phase map of **Fig. 2.1(b)**, dark red, yellow and green colors represent austenite, ϵ martensite and α' martensite respectively. In addition, high angle grain boundaries with misorientation larger than 15° and annealing twin boundaries are drawn in black and yellow lines. Same coloring is also used in the following EBSD phase maps. As shown in **Fig. 2.1**, the microstructure exhibits fully austenite single phase structure, and ϵ martensite and α' martensite are not observed.

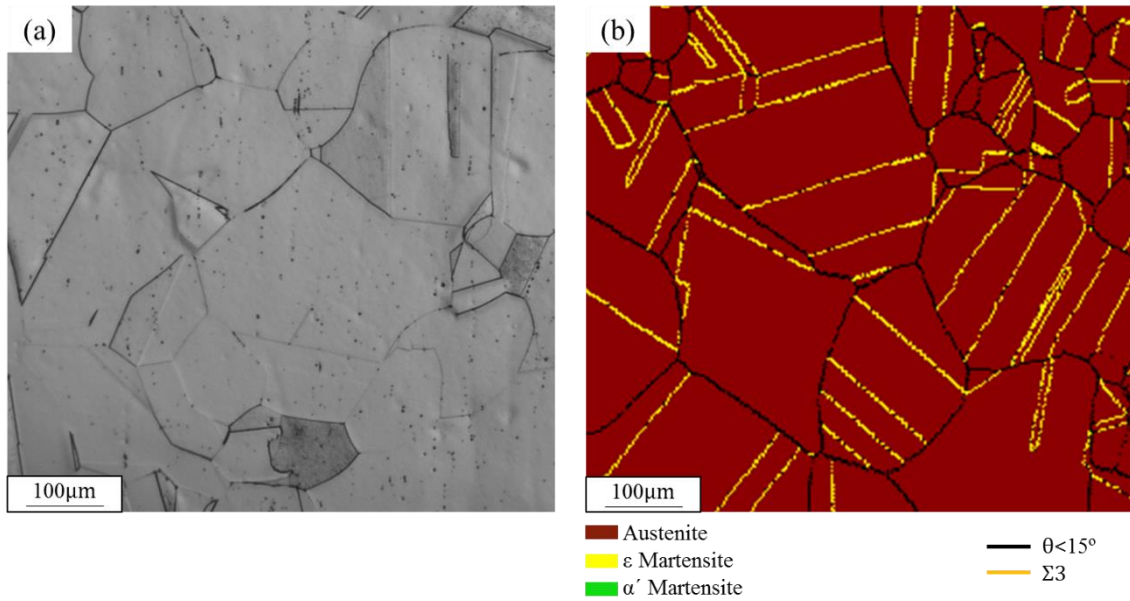


Figure 2.1 (a) Optical microscopy image and (b) EBSD phase map of the annealed SUS304 specimen. Dark red, yellow and green colors represent austenite, ϵ martensite and α' martensite, respectively. High angle grains boundaries with misorientation larger than 15° and annealing twin boundaries are drawn in black and yellow lines, respectively.

The optical microscopy image and SEM image of the specimen after subzero treatment at liquid nitrogen temperature are shown in **Figs. 2.2 and 2.3**. Thermally induced martensite, as pointed out by black arrows, exhibits a straight band morphology with

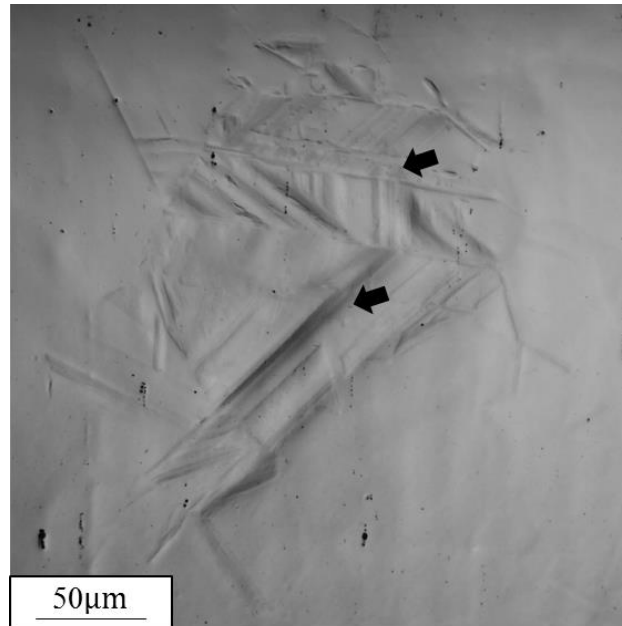


Figure 2.2 Optical microscopy images of the specimen after subzero treatment. Black arrows indicate thermally induced martensite.

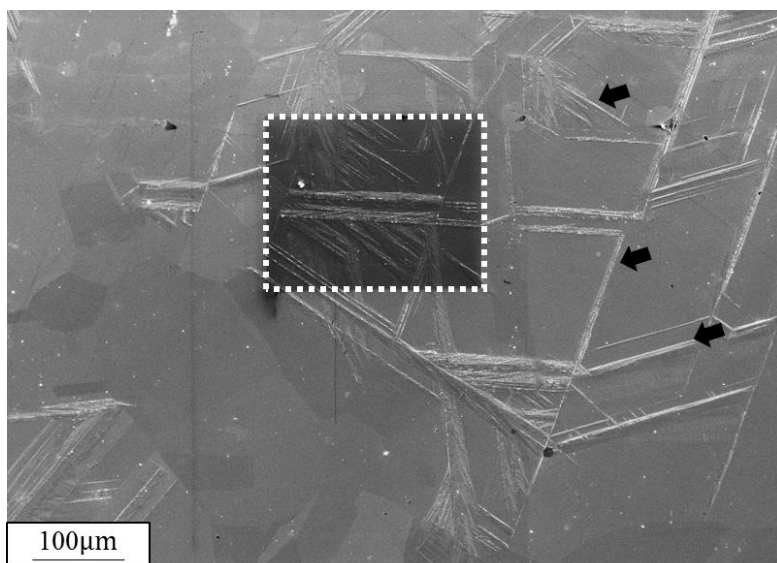


Figure 2.3 SEM image of the specimen after subzero treatment. Black arrows indicate the thermally induced martensite.

different widths and lengths. **Figure 2.4** shows (a) an EBSD orientation map and (b) an EBSD phase map of the area corresponding to the white broken rectangle in **Fig. 2.3**. The color in the orientation map represents the crystallographic orientation parallel to the normal direction of the specimen surface according to the stereo triangles shown below. Although it is hard to observe ε martensite in the phase map clearly due to the large step size, the EBSD measurements detected around 1 % ε martensite in the microstructure. From **Figs. 2.4(a) and (b)**, the straight bands of α' martensite consist of several different variants. In addition, there are four kinds of the straight α' martensite bands whose elongated directions are parallel to one of the four $\{111\}_\gamma$ planes of austenite shown in **Fig. 2.4(c)**. This indicates that the straight α' martensite bands grew along the $\{111\}_\gamma$ planes.

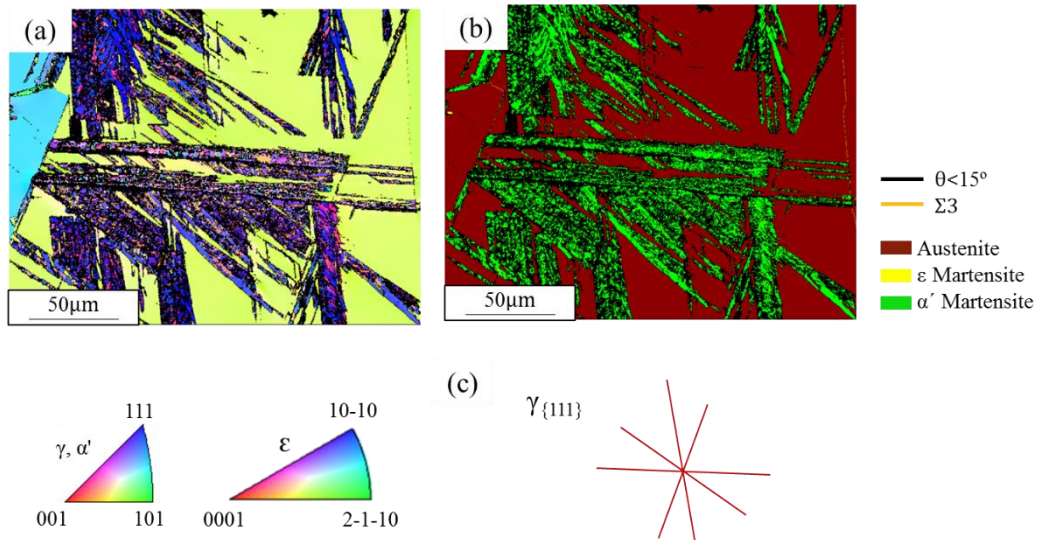


Figure 2.4 (a) EBSD orientation map and (b) EBSD phase map of the area corresponding to the white broken rectangle in **Fig. 2.3**. The color in the orientation color map represents the crystallographic orientation parallel to the normal direction of the specimen surface, according to the stereo triangles. (c) The traces of four $\{111\}_\gamma$ planes.

Figure 2.5 shows (a) an EBSD orientation map and (b) an EBSD phase map of another austenite grain. From the orientation map (**Fig. 2.5(a)**), the phase map (**Fig. 2.5(b)**) and

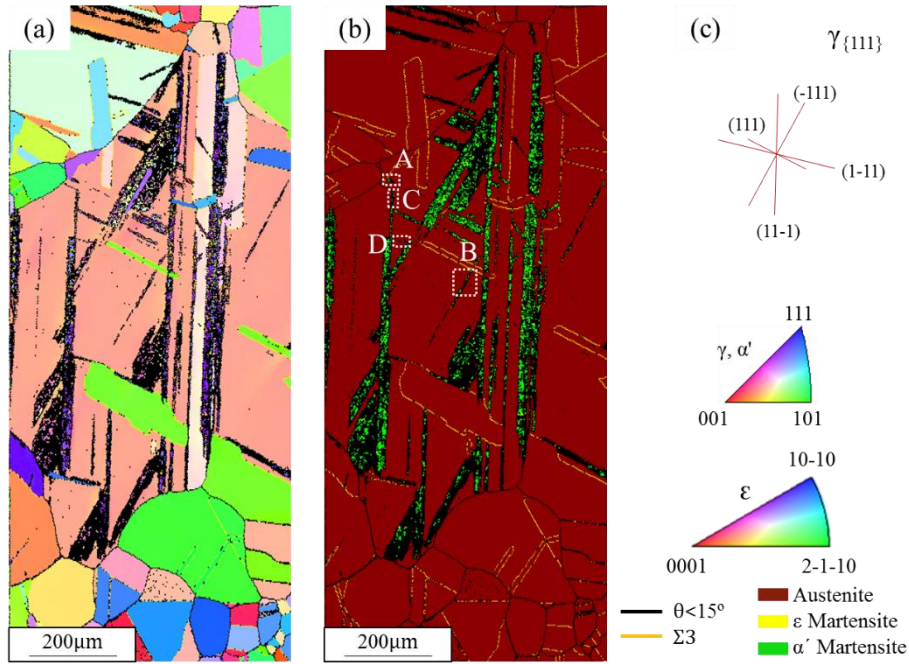


Figure 2.5 (a) EBSD orientation map and (b) EBSD phase map of another austenite grain. The color in the orientation color map represents the crystallographic orientation parallel to the normal direction of the specimen surface, according to the stereo triangles. (c) The traces of four $\{111\}_\gamma$ planes.

the plane trace of $\{111\}_\gamma$ (**Fig. 2.5(c)**), the similar results, that all the α' martensite bands consisting of several different α' variants are elongated along four $\{111\}_\gamma$ planes, are confirmed. **Figures 2.6 – 2.9** show the enlarged microstructures corresponding to the white broken rectangles indicated by A, B, C and D, respectively (EBSD scanning step size is 50 nm). Each of them is parallel to one of the four $\{111\}_\gamma$ planes. As shown in **Figs. 2.6(a) and (b)**, the α' martensite band in the region A (the band A) is parallel to $(111)_\gamma$ planes, and ϵ martensite pointed out by white arrows can be clearly observed. In **Figs. 2.6(c) and (d)**, each α' martensite variant is highlighted in the same colors. It is found that one α' martensite band consists of all six variants. Though the six α' martensite variants elongated in different directions from the $(111)_\gamma$ plane, they managed to form one band which is parallel to the $(111)_\gamma$ plane. In addition, the ϵ martensite crystals are parallel to the $(111)_\gamma$ plane. Same tendency is also confirmed in the α' martensite bands parallel to (-

$111)_\gamma$ and $(11-1)_\gamma$ planes as shown in **Figs. 2.7 and 2.8** (the band B and the band C). Furthermore, in the thin α' martensite band on the right side of **Fig. 2.8(b)**, it is clear that ε martensite plate is partially remained as indicated by white arrows. In contrast, other parts transformed into α' martensite crystals. This clearly suggests that ε martensite plates formed in the γ matrix first, and then α' martensite transformed from ε martensite plates. Such transformation sequence ($\gamma \rightarrow \varepsilon \rightarrow \alpha'$) has been reported not only in SUS304, but also in other Fe alloys [15-20].

As introduced in **Chapter 1**, the orientation relationship between γ and ε martensite is S-N relationship. While between ε and α' martensite, a Burgers orientation relationship is found [21] as follows:

$$\{0001\}_\varepsilon // \{011\}_{\alpha'}, \quad \langle 11\bar{2}0 \rangle_\varepsilon // \langle 111 \rangle_{\alpha'}. \quad (2.1)$$

Maximum six α' variants can form from an ε martensite variant according to the Burgers relationship. Both S-N relationship and Burgers relationship can be described as the close-packed planes and the close-packed directions of two concerned phases are parallel to each other. Thus, under the transformation sequence $\gamma \rightarrow \varepsilon \rightarrow \alpha'$, the K-S relationship could also be obtained between γ and α' martensite, since it also satisfies that the close-packed planes and the close-packed directions of γ and α' martensite are parallel to each other.

As shown in **Fig. 2.9**, however, there are no ε martensite and only two α' martensite variants are observed in the martensite band in the region D (the band D) which is parallel to $(1-11)_\gamma$ plane. One possible reason for this is that γ directly transformed into α' without the occurrence of ε martensite. This transformation sequence ($\gamma \rightarrow \alpha'$) is also proposed in TRIP steels by some groups [22-26]. It is also reported that both the transformation sequences of $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ and $\gamma \rightarrow \alpha'$ can happen simultaneously at the same time [11]. Another possible reason is that the CI index of retained ε martensite was too low for the EBSD measurements to detect.

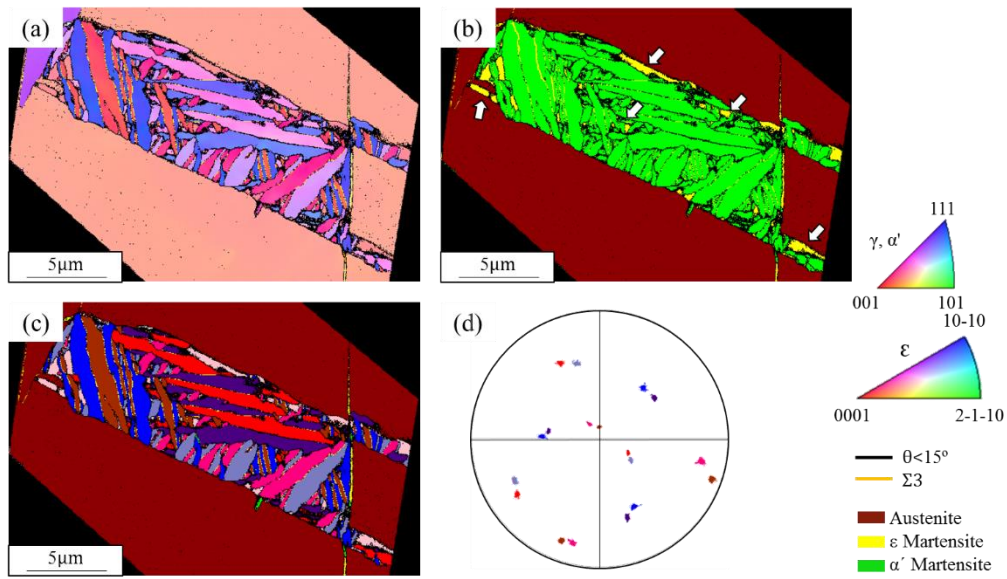


Figure 2.6 Enlarged (a) EBSD orientation map and (b) EBSD phase map of the region A in **Fig. 2.5**, while white arrows indicate ε martensite. (c) EBSD phase map on which all α' martensite bands parallel to $(111)_{\gamma}$ are highlighted in different colors, (d) their $\{001\}$ pole figure in the corresponding colors.

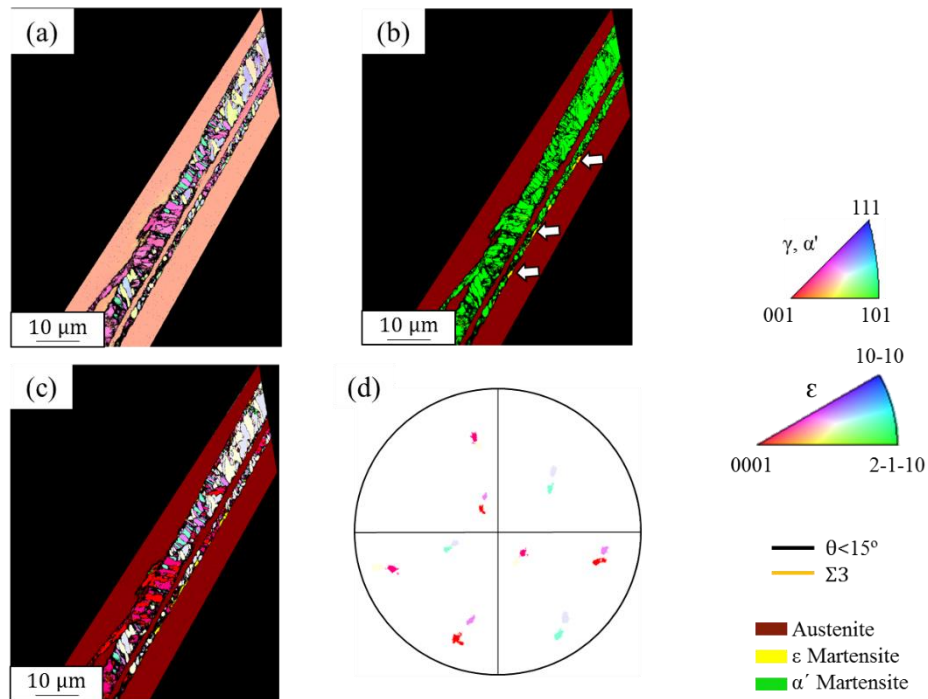


Figure 2.7 Enlarged (a) EBSD orientation map and (b) EBSD phase map of the region B in **Fig. 2.5**, while white arrows indicate ε martensite. (c) EBSD phase map on which all α' martensite bands parallel to $(-111)_{\gamma}$ are highlighted in different colors, (d) their $\{001\}$ pole figure in the corresponding colors.

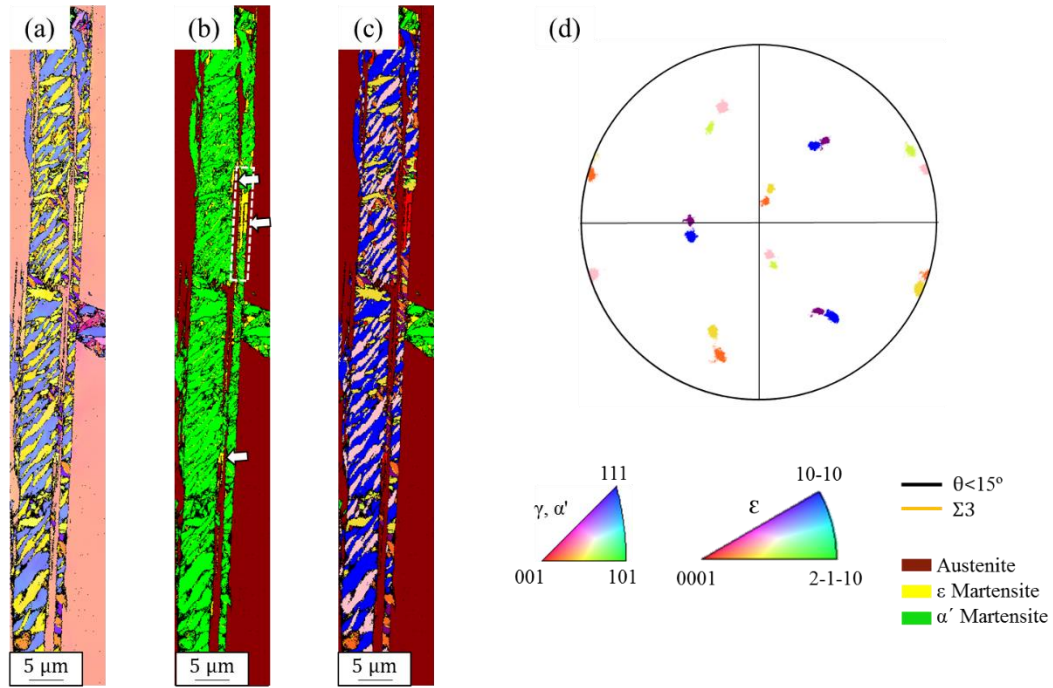


Figure 2.8 Enlarged (a) EBSD orientation map and (b) EBSD phase map of region C in **Fig. 2.5**, while white arrows indicate ϵ martensite. (c) EBSD phase map on which all α' martensite bands parallel to $(11-1)_\gamma$ are highlighted in different colors, (d) their $\{001\}$ pole figure in the corresponding colors.

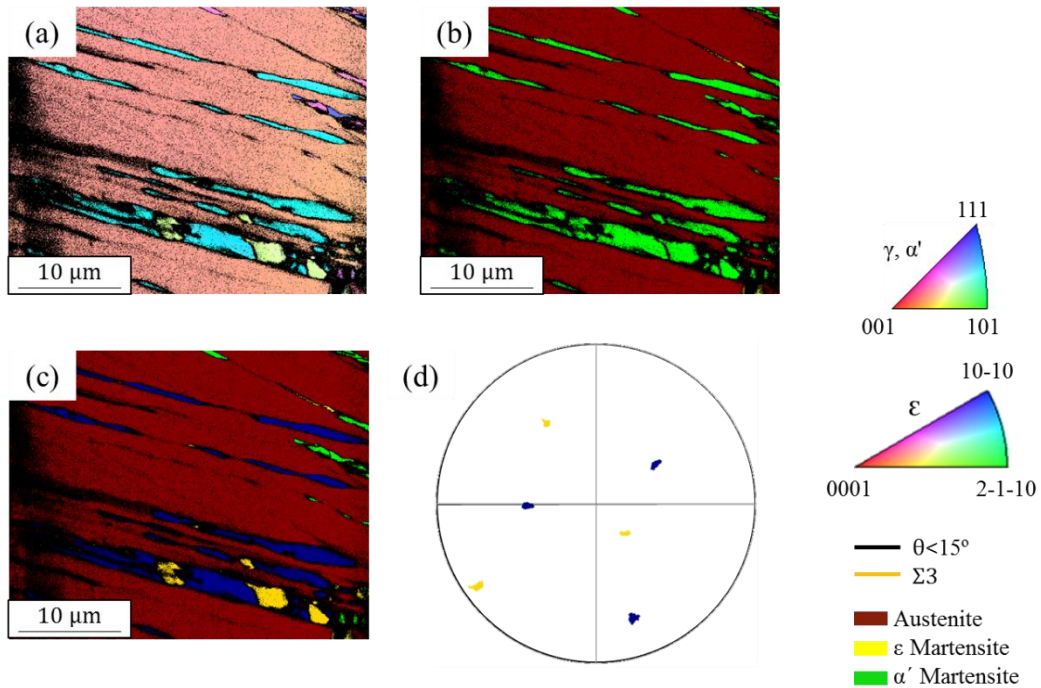


Figure 2.9 Enlarged (a) EBSD orientation map and (b) EBSD phase map of region D in **Fig. 2.5**. (c) EBSD phase map on which all α' martensite bands parallel to $(1-11)_\gamma$ are highlighted in different colors, (d) their $\{001\}$ pole figure in the corresponding colors.

2.3.2 Crystallographic analysis

Figure 2.10 presents pole figures of the close-packed planes ($\{111\}_\gamma$, $\{0001\}_\varepsilon$, $\{110\}_{\alpha'}$) and close-packed directions ($\langle 110 \rangle_\gamma$, $\langle 11\bar{2}0 \rangle_\varepsilon$, $\langle 111 \rangle_{\alpha'}$) of austenite, ε martensite and α' martensite in the band A of **Fig. 2.6**. It is found that both close-packed planes and directions of the three phases are parallel to each other. This suggests the S-N relationship between γ and ε phases (**Figs. 2.10(a), (b), (d), (e)**), the Burgers relationship between ε and α' phases (**Figs. 2.10(b), (c), (e), (f)**), and consequently, the K-S relationship between γ and α' phases (**Figs. 2.10(a), (c), (d), (f)**) are satisfied. Furthermore, as can be seen in **Fig. 2.10(c)**, all the six α' martensite crystals in one martensite band have the common $\{110\}_{\alpha'}$ pole indicated by broken circle. This means that they satisfy the same parallel plane relationship in the Burgers relationship. The same tendency can also be found in **Figs. 2.11 and 2.12**, which are pole figures of the three phases in the band B (**Fig. 2.7**) and the band C (**Fig. 2.8**), respectively. Although ε martensite could not be detected in **Fig. 2.9**, the K-S relationship between austenite and α' martensite can be confirmed in **Fig. 2.13**.

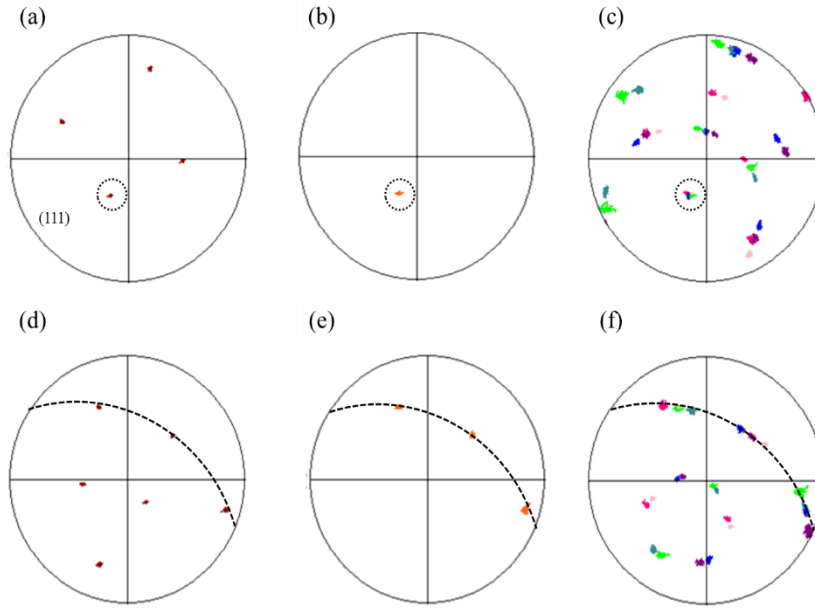


Figure 2.10 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\epsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\epsilon$, (f) $\{111\}_\alpha$ in the band A (**Fig. 2.6**).

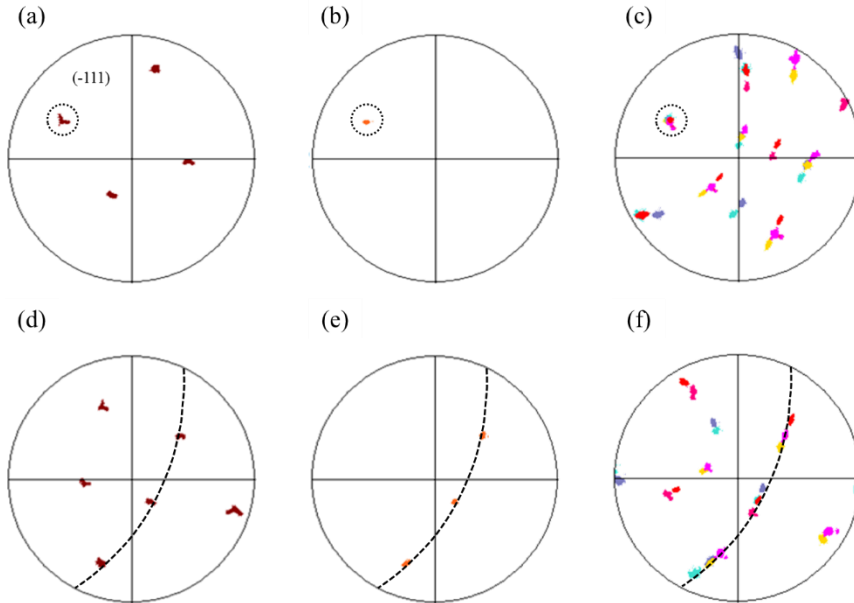


Figure 2.11 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\epsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\epsilon$, (f) $\{111\}_\alpha$ in the band B (**Fig. 2.7**).

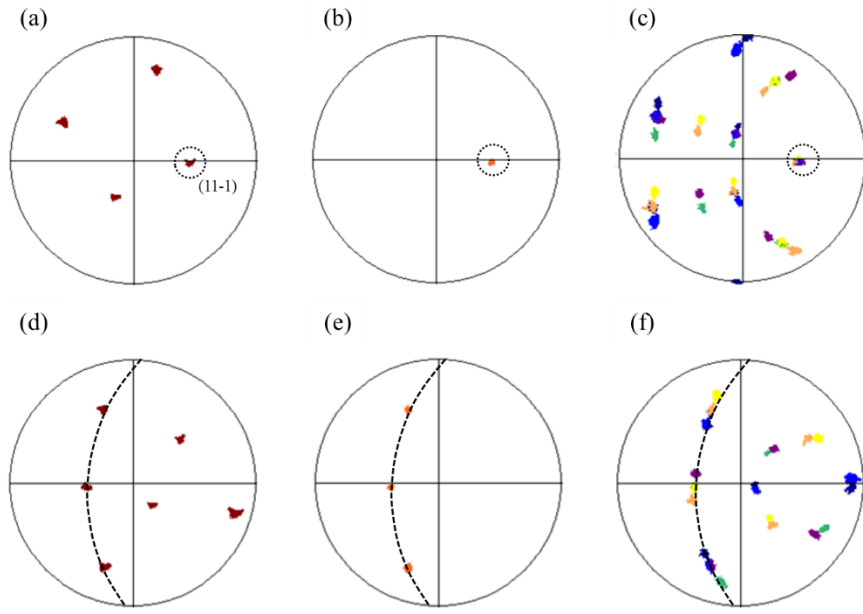


Figure 2.12 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\epsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\epsilon$, (f) $\{111\}_\alpha$ in the band C (**Fig. 2.8**).

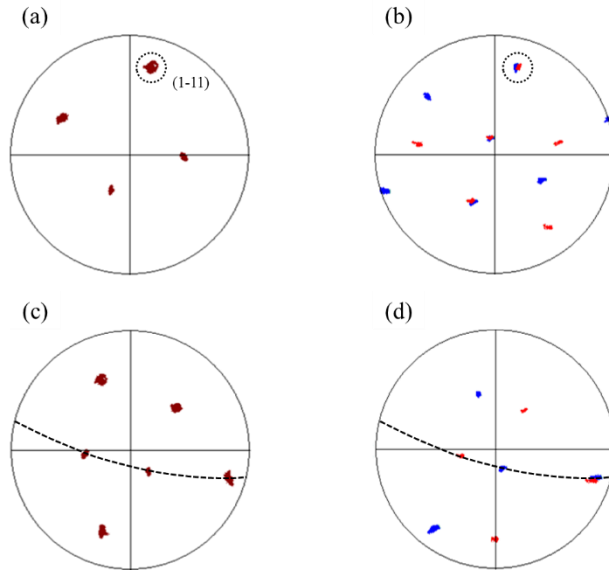


Figure 2.13 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\epsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\epsilon$, (f) $\{111\}_\alpha$ in the band D (**Fig. 2.9**).

In **Fig. 2.14**, different α' martensite variants formed in one martensite band are represented by different colors, where green, dark green, red, dark red, blue and dark blue represent V1, V2, V3, V4, V5 and V6, respectively. It is well known that among all the six variants, V1 and V2, V3 and V4, V5 and V6 have $\Sigma 3$ relationship. As can be seen in **Figs. 2.14(a - c)**, variants with $\Sigma 3$ relationship formed adjacently.

The sequence of thermally induced martensitic transformation is schematically summarized in **Fig. 2.15**, where γ , ε martensite and α' martensite variants are white, yellow and other colors as indicated below the illustration. Thermally induced ε martensite plates parallel to four $\{111\}_{\gamma}$ planes form first from austenite phase. After that, most of the ε martensite plates transform into six α' crystals. The α' crystal with $\Sigma 3$ relationship often form adjacently.

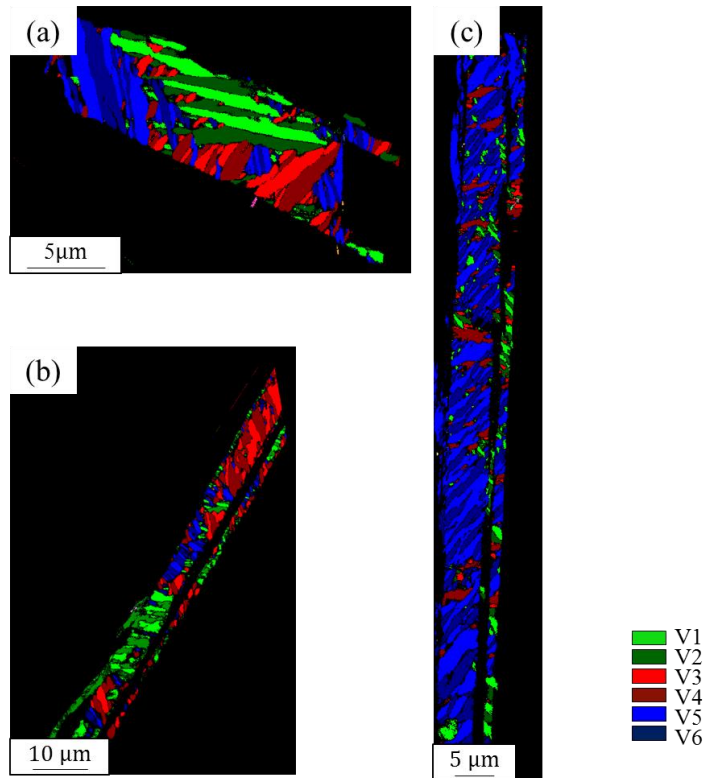


Figure 2.14 α' martensite variants in the martensite band (a) A (**Fig. 2.6**), (b) B (**Fig. 2.7**) and (c) C (**Fig. 2.8**) are presented by different colors, where green, dark green, red, dark red, blue and dark blue represent V1, V2, V3, V4, V5 and V6, respectively.

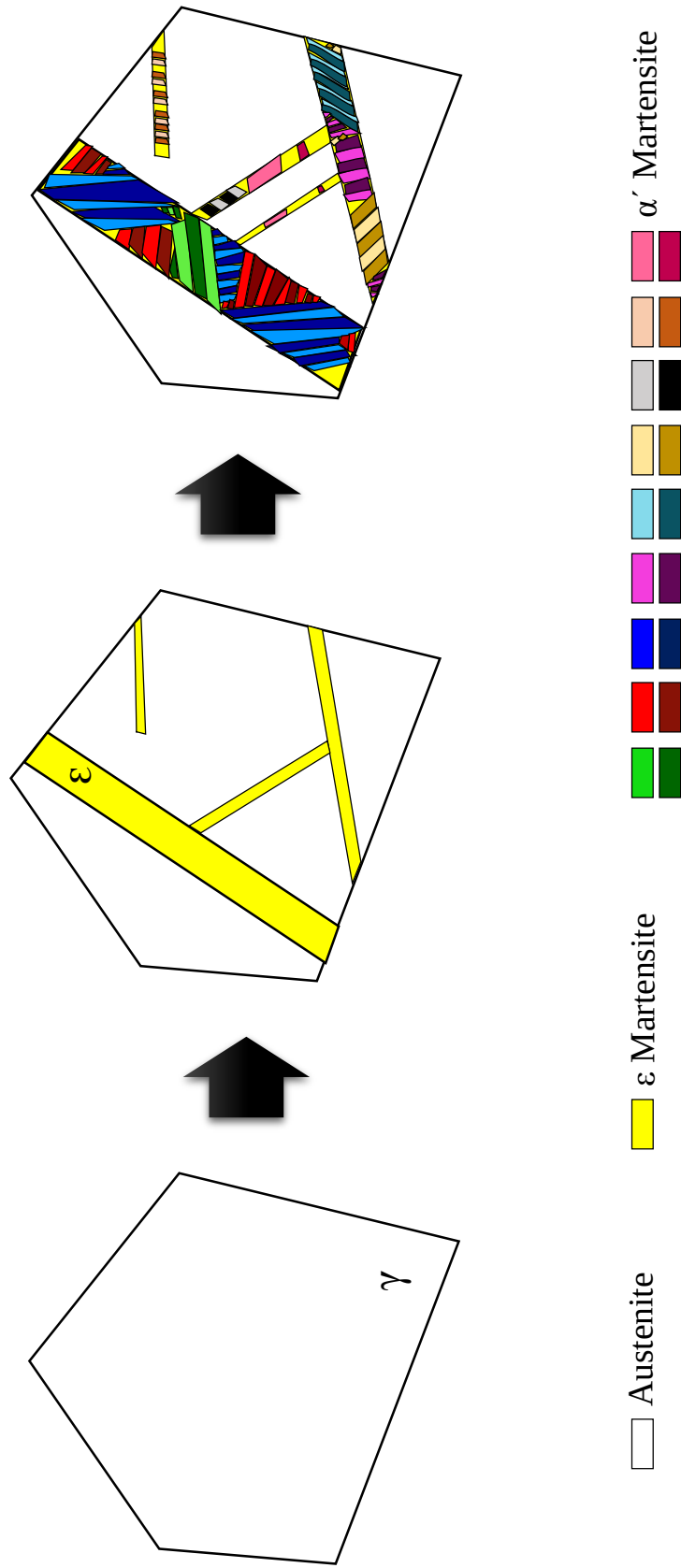


Figure 2.16 Schematic illustration of the formation sequence of thermally induced martensitic transformation in SUS304 stainless steel, where white and yellow represent austenite and ϵ martensite, respectively. The α' martensite is represented by the rest colors, each pair of colors indicates $\Sigma 3$ relationship.

2.4 Discussions

As mentioned in **Section 2.1**, in low carbon steels, one martensite packet consists of six α' martensite variants, whose elongated directions are parallel to each other [12]. In addition, each packet contains three parallel blocks, and each block consists of two specific martensite variants with small misorientation (V1-V4, V2-V5, V3-V6). In the case of SUS304 austenitic stainless steel, however, all the six α' martensite variants observed in one packet were elongated in the different directions as shown in **Figs. 2.6 - 2.8**. Furthermore, as explain in **Fig. 2.14**, instead of the specific K-S variant combination, variants with $\Sigma 3$ relationship formed adjacently in all the 3 martensite bands.

In order to discuss the accommodation of transformation strain by combination of variants, the equivalent strains of total shape deformation produced by various variant combinations were calculated based on phenomenological theory of martensite crystallography (PTMC) [27-31]. As explained in **Chapter 1**, the PTMC is based on the assumption that the martensitic transformation is an invariant plane strain and habit plane corresponds to an invariant plane in which all lines in it are neither distorted nor rotated. The present PMTC calculation used lattice parameters of $a_\gamma = 0.359$ nm, $a_{\alpha'} = 0.285$ nm, and $(101)[-101]_\gamma$ and $(011)[0-11]_\gamma$ as lattice invariant deformations. The shape deformation direction, the habit plane orientation (invariant plane orientation) and the magnitude of the shape deformation calculated by PMTC were $\langle -0.509 \ 0.589 \ -0.627 \rangle$, $\{0.412 \ 0.807 \ 0.423\}$ and 0.138.

Thus, the shape deformation tensor caused by a single variant can be calculated by the following equation:

$$F = I + mdp'$$

$$= \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} + m[d_1 d_2 d_3](p_1 p_2 p_3) \quad (2.2)$$

where $\mathbf{I}$ is the unit matrix, $\mathbf{m}$ is the magnitude of the shape deformation, $\mathbf{d}$ is the column vector representing the shape deformation direction and $\mathbf{p}'$ is the row vector representing the normal direction of the habit plane. The strain tensor produced by the shape deformation can be calculated by the equation shown below:

$$\boldsymbol{\varepsilon} = \frac{1}{2}(\mathbf{F}^t \mathbf{F} - \mathbf{I}) \quad (2.3)$$

where $\mathbf{F}^t$ is the transposed matrix of $\mathbf{F}$.

Since it is difficult to compare the shape strain in tensor form, the equivalent transformation strain was calculated by the following equation:

$$\varepsilon_{eq} = \sqrt{\frac{2}{3}\{\varepsilon_x^2 + \varepsilon_y^2 + \varepsilon_z^2 + \frac{1}{2}(\gamma_{xy}^2 + \gamma_{yz}^2 + \gamma_{zx}^2)\}} \quad (2.4)$$

Assuming that all the variants have equal volume fraction, the total equivalent transformation strains produced by several combinations of α' martensite variants are summarized in **Table 2.3**. Noting that the equivalent transformation strain of single

Table 2.3 Total equivalent transformation strains caused by various variant combinations.

Variant combination	Equivalent strain
V1	0.0718
V1+V2	0.0716
V1+V3	0.0391
V1+V4	0.0426
V1+V5	0.0391
V1+V6	0.0357
V1+V2+V3	0.0425
V1+V3+V5	0.0205
V2+V4+V6	0.0205
V1+V2+V3+V4+V5+V6	0.0205

martensite variant is 0.0718, it is found that the combination of variants with $\Sigma 3$ relationship does not accommodate transformation strain, but the combination of all six martensite variants has the smallest equivalent strain of 0.0205. Because each variant did not form equally as shown in **Fig. 2.16**, the equivalent transformation strains taking into account of area fraction of each variant were calculated. The result summarized in **Table 2.4** indicates that the equivalent transformation strains in the martensite band A and B were considerably small. However, the equivalent transformation strains of the band C, which mainly consisted of V5 and V6, was 0.0556 and not much reduced compared with single martensite variant.

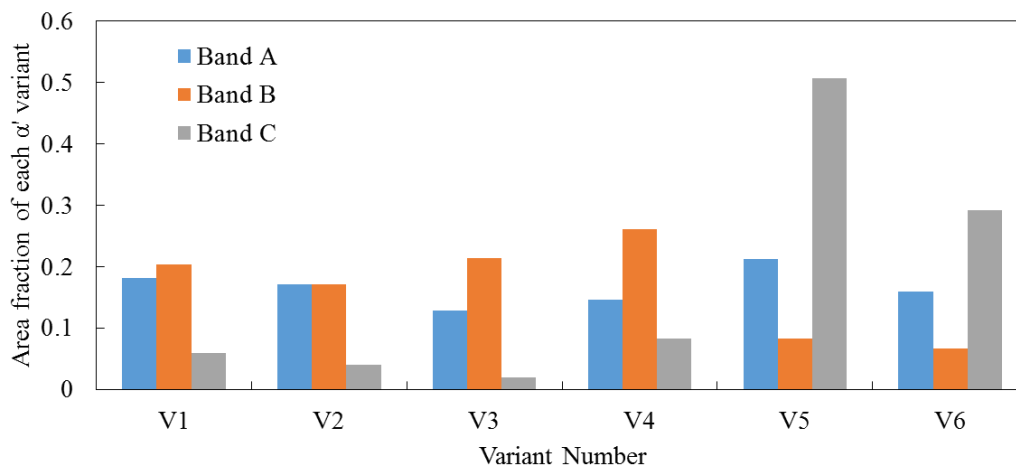


Figure 2.16 Area fractions of α' martensite variants in the bands A, B and C.

Table 2.4 Total equivalent strain caused by various variant

Band Name	Equivalent strain
A	0.0213
B	0.0275
C	0.0556

The above PTMC calculation suggests that six α' martensite variants formed in one martensite band to reduce the total transformation strain. However, the tendency that variants with $\Sigma 3$ relationship formed adjacently could not be explained from the accommodation of transformation strain. The possible reason for the combination of variants with $\Sigma 3$ relationship is to the reduction of boundary energy between martensite variants. However, further discussion is necessary to explain the $\Sigma 3$ variant combination.

2.5 Conclusions

In this chapter, thermally induced martensitic transformation in austenitic stainless steel SUS304 was studied. The main results are summarized as follows:

(1) The transformation sequence of the thermally induced martensitic transformation in the studied SUS304 austenitic stainless steel was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. The thermally induced martensite exhibited a macroscopic straight band morphology, and were parallel to the four $\{111\}_{\gamma}$ planes of austenite phase. The straight bands consisted of little amount of ε martensite and six α' martensite variants that could form from the ε variant.

(2) The S-N relationship between γ and ε phases, the Burgers relationship between ε and α' phases, and resultantly the K-S relationship between γ and α' phases were confirmed from the orientation analysis. There was a tendency that martensite variants with $\Sigma 3$ relationship (V1-V2, V3-V4, and V5-V6) formed adjacently. This is completely different from that the lath martensite formed in low carbon steels.

(3) The PTMC calculation suggested that six α' martensite variants formed in one martensite band to reduce the total transformation strain. However, the combination of variants with $\Sigma 3$ relationship could not be explained from the accommodation of transformation strain.

2.6 References

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Chapter 3: Stress-induced martensitic transformation in SUS304 austenitic stainless steel

3.1 Introduction

As mentioned in **Chapter 1**, deformation-induced martensitic transformation occurred during deformation of austenite in the elastic regime is termed “stress-induced martensitic transformation” and that occurred during plastic deformation of austenite is termed “strain-induced martensitic transformation”. In most researches about austenitic stainless steel, the terminology deformation-induced martensitic transformation is used as strain-induced martensitic transformation [1-5]. Stress-induced martensitic transformation has been mostly studied in Ti-Ni and Cu-based alloys for their superelasticity and shape memory effect [6-11], while only few studies have been carried out in the stainless steel because the high stability of austenite makes it difficult to occur. Fahr reported that austenite directly transformed to α' martensite in the stress-induced martensitic transformation in Fe-Cr-Ni-Mn-C austenitic stainless steel [12]. According to his research, the martensitic transformation occurred before austenite yielding resulted in high work-hardening rate, high tensile strength, but low yield strength. However, the detail of the morphology and crystallographic features of stress-induced martensitic transformation remains unclear.

Usually, variant selection, that some specific variants preferentially form, occurs in deformation-induced martensitic transformation [13-18]. Although several models for explaining the variant selection in deformation-induced martensitic transformation have been proposed (Patel and Cohen model, Bogers-Burgers model, etc.), variant selection rules, especially in polycrystalline materials, have not yet been clearly understood. The

previous studies for variant selection assumed that the stress condition inside a grain was uniform and the same as the externally applied stress even though it is well known that the local stress can be different with the macroscopic applied tensile stress for polycrystalline materials. Therefore, in order to clarify the variant selection rule in deformation-induced martensitic transformation, it is necessary to measure the local stress field of the position where transformation occurred. Several techniques to measure local stress field during tensile deformation have been developed. Digital image correlation technique [19] can evaluate local strain and stress fields by comparing the microstructures on the surface before and after deformation. However, it cannot provide any information of within the specimen. On the other hand, neutron diffraction and X-ray diffraction techniques can measure the local stress field inside the specimen [20]. Recently, a new technique has been developed using synchrotron radiation X-ray diffraction in SPring-8, which is called energy dispersive X-ray diffraction microscopy (EXDM). This technique can evaluate local stress field with spatial resolution down to 10 μm [21, 22], which is much smaller than that of neutron diffraction technique.

Thus, this chapter aims to investigate the morphology, crystallographic features of stress-induced martensitic transformation, and to clarify the correlation between variant selection and local stress field in stress-induced martensitic transformation of austenitic stainless steel by EBSD measurements and EXMD technique.

3.2 Experimental procedures

3.2.1 Local stress field analysis by energy dispersive X-ray diffraction microscopy (EXDM)

The EXDM is a new technique to measure local stress field by the synchrotron radiation white X-ray diffraction at beamline BL28B2 in SPring-8 [21]. **Figure 3.1** is photos of (a) the miniaturized tensile test machine utilized in the EXDM and (b) a miniaturized tensile test machine, a flat panel sensor and a solid state detector at the beamline BL28B2.

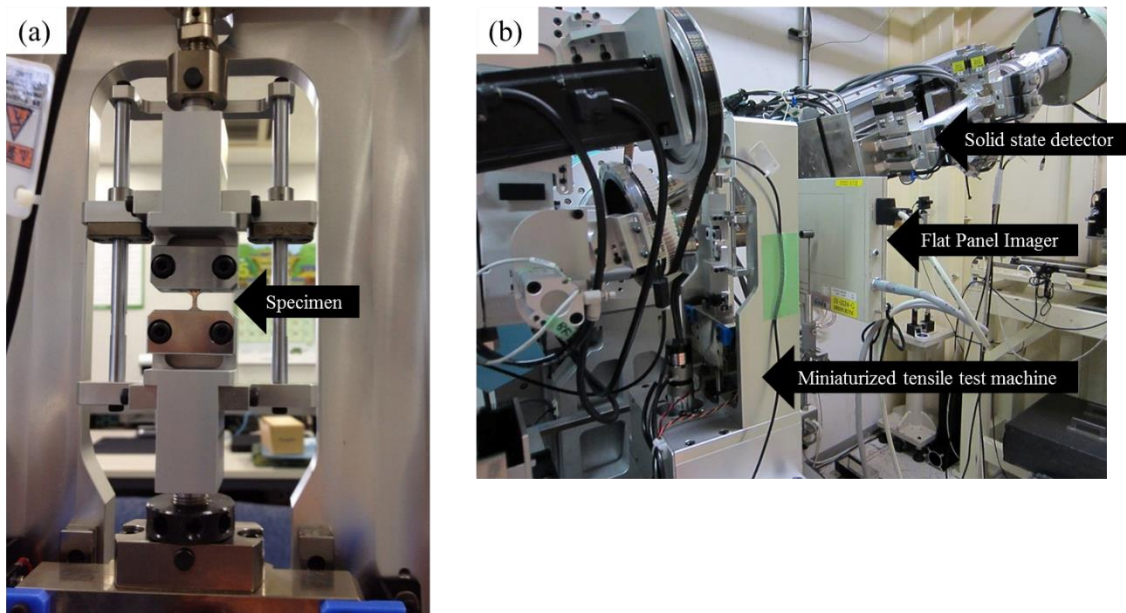


Figure 3.1 Photos of (a) a miniaturized tensile test machine utilized in the EXDM and (b) a miniaturized tensile test machine, a flat panel sensor and a solid state detector at the beamline BL28B2.

Figure 3.2 presents a schematic illustration of the EXDM technique. The EXDM consists of following two steps. First step is to obtain a grain boundary map by analyzing transmission Laue diffraction patterns. As shown in **Fig 3.2(a)**, the synchrotron radiation

white X-ray micro beam with minimum beam size of $10\mu\text{m} \times 10\mu\text{m}$ was scanned in the target area of the specimen by moving the specimen stage in parallel to the specimen surface. Laue diffraction pattern of each position was recorded during the scanning by the flat panel sensor, and a grain boundary map was constructed by comparing the Laue diffraction patterns of neighboring measured position. Second step is to measure energy spectra of each diffraction spot in the Laue diffraction patterns by the solid state detector as shown in **Fig. 3.2(b)**.

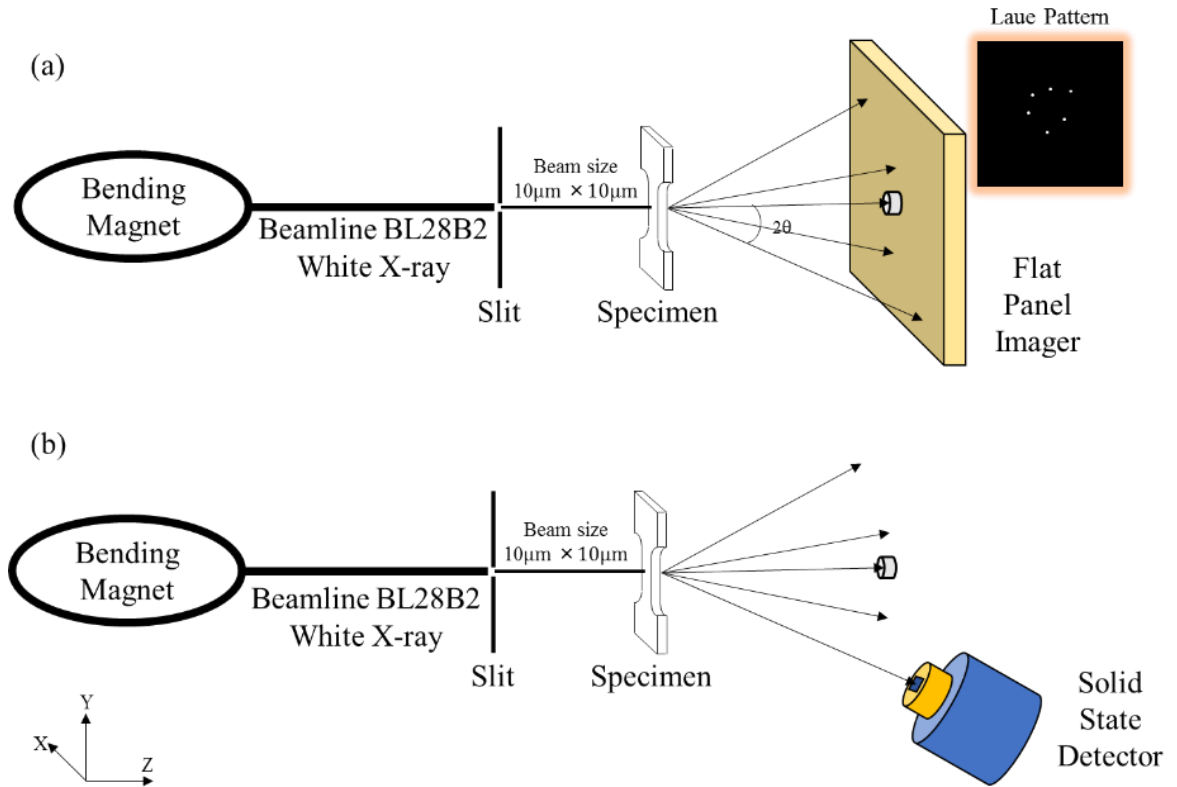


Figure 3.2 Schematic illustrations of the EXDM procedure.

The diffraction angle of each spot was geometrically measured from the distance between the specimen and the flat panel sensor. The wave length was obtained from the energy spectrum of the diffraction spot. Thus, the crystal lattice plane spacing was calculated by Bragg equation shown below:

$$2 d_{(h,k,l)} \sin\theta = \lambda = \frac{hc}{E} \quad (3.1)$$

where $d_{(h,k,l)}$ is the crystal lattice plane spacing of (h k l) plane, θ is the diffraction angle, λ is the wave length, h is Planck's constant, c is the speed of light, and E is the energy measured by the solid state detector. The accuracy of crystal lattice spacing evaluated by this method using Si single crystal was 0.04% [21].

The elastic strain normal to each crystal lattice plane under external stress was evaluated by the following equation:

$$\varepsilon(h, k, l) = \frac{d_{(h,k,l)} - d_{0(h,k,l)}}{d_{0(h,k,l)}} \quad (3.2)$$

where $\varepsilon_{(h,k,l)}$ represents the elastic strain, $d_{0(h,k,l)}$ and $d_{(h,k,l)}$ represent lattice plane spacing of stress-free and stressed specimens, respectively. The $d_{0(h,k,l)}$ was derived from average crystal lattice constant measured under no external stress.

Because the crystal structure of SUS304 is cubic, stress tensor has six individual components. Thus, a system of equations between stress and strain components can be established as follows:

$$\begin{aligned} \varepsilon_1(h_1, k_1, l_1) &= a_1\sigma_{xx} + b_1\tau_{xy} + c_1\sigma_{yy} + d_1\tau_{yz} + e_1\sigma_{zz} + f_1\tau_{zx} \\ \varepsilon_2(h_2, k_2, l_2) &= a_2\sigma_{xx} + b_2\tau_{xy} + c_2\sigma_{yy} + d_2\tau_{yz} + e_2\sigma_{zz} + f_2\tau_{zx} \\ \varepsilon_3(h_3, k_3, l_3) &= a_3\sigma_{xx} + b_3\tau_{xy} + c_3\sigma_{yy} + d_3\tau_{yz} + e_3\sigma_{zz} + f_3\tau_{zx} \\ \varepsilon_4(h_4, k_4, l_4) &= a_4\sigma_{xx} + b_4\tau_{xy} + c_4\sigma_{yy} + d_4\tau_{yz} + e_4\sigma_{zz} + f_4\tau_{zx} \\ \varepsilon_5(h_5, k_5, l_5) &= a_5\sigma_{xx} + b_5\tau_{xy} + c_5\sigma_{yy} + d_5\tau_{yz} + e_5\sigma_{zz} + f_5\tau_{zx} \\ \varepsilon_6(h_6, k_6, l_6) &= a_6\sigma_{xx} + b_6\tau_{xy} + c_6\sigma_{yy} + d_6\tau_{yz} + e_6\sigma_{zz} + f_6\tau_{zx} \end{aligned} \quad (3.3)$$

where σ_{ii} , τ_{ij} ($i, j = x, y, z$, $i \neq j$) are the individual components of local stress, and a_i , b_i , c_i , d_i , e_i , f_i ($i = 1, 2, 3, 4, 5, 6$) are the constants which can be calculated from elastic moduli of C_{11} , C_{12} and C_{44} . The present study used the elastic moduli of SUS304 austenitic

stainless steel measured by Ledbetter [23] ($C_{11} = 209$ GPa, $C_{12} = 133$ GPa , $C_{44} = 121$ GPa). Here, y is the direction of the external tensile stress, and x is the direction perpendicular to y in the plane parallel to specimen surface. The maximum number of the crystal lattice planes that can be measured successfully by the EXDM is three because the direction of the white X-ray beam was fixed perpendicular to the specimen surface. If we assume σ_{zz} , τ_{yz} and τ_{zx} , which are the components perpendicular to the specimen surface, are released in the thin specimen during the deformation, the system of equations in **Eq. 3.3** can be changes into the following forms:

$$\begin{aligned}\varepsilon_1(h_1, k_1, l_1) &= a_1\sigma_{xx} + b_1\tau_{xy} + c_1\sigma_{yy} \\ \varepsilon_2(h_2, k_2, l_2) &= a_2\sigma_{xx} + b_2\tau_{xy} + c_2\sigma_{yy} \\ \varepsilon_3(h_3, k_3, l_3) &= a_3\sigma_{xx} + b_3\tau_{xy} + c_3\sigma_{yy}\end{aligned}\tag{3.4}$$

It is then possible to evaluate the local stress tensor $\mathbf{S}$ by a matrix composed of three component in a coordinate system as follows:

$$\mathbf{S} = \begin{bmatrix} \sigma_{xx} & \tau_{xy} & 0 \\ \tau_{xy} & \sigma_{yy} & 0 \\ 0 & 0 & 0 \end{bmatrix}\tag{3.5}$$

3.2.2 Experimental procedures

When there are several grains in the thickness direction of the specimen, Laue diffraction patterns comprise diffraction spots from several grains. This will make it difficult to construct grain boundary map. In order to make the specimen containing only one grain in the thickness direction, the following treatment was conducted.

The as-received material was homogenized at 1000 °C for 3 hours in vacuum, and then furnace cooled. The annealed sheet were cold rolled by 10% to reduce the thickness to 0.9 mm. Tensile test specimens, whose size is schematically shown in **Fig. 3.3**, were cut from the cold rolled sheet by wire cut machine (Brother, HS-70A) and mechanically polished until the final specimen thickness of 0.5 mm was reached. Then, the specimens were annealed at 1050 °C for 70~85 hours under argon atmosphere followed by furnace cooling. A fully-recrystallized homogeneous microstructure of γ single phase was obtained by this heat treatment. After the heat treatment, only electrolytic polishing was conducted to reduce the thickness of the specimen to avoid deformation-induced martensitic transformation during mechanical polishing. The electrolytic solution was 10% HClO_4 + 90% CH_3COOH . The electrolytic voltage was 12 V and the polishing time was 30 ~ 50 minutes. The microstructures of both surfaces of tensile test specimen were measured by EBSD not only for obtaining the crystallographic information before

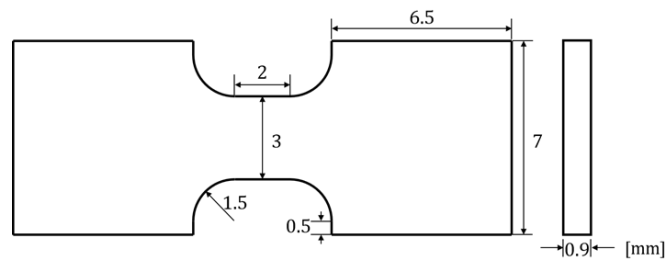


Figure 3.3 Schematic illustration of the tensile specimen.

transformation, but also for finding grains which penetrates the specimens in the thickness direction.

The prepared tensile test specimen was installed at the miniaturized tensile test machine at beamline BL28B2 in SPring-8. The crystal lattice spacing without external stress was measured first. Then the specimen was deformed in tension within the elastic regime at a strain rate of $8.4 \times 10^{-4} \text{ s}^{-1}$ at room temperature. The EXDM was then carried out to the deformed tensile specimen under the external stress.

Because it is reported that martensitic transformation usually starts to occur at the grain boundaries in larger grains [1, 16, 24, 25], the local stress field at the positions near grain boundaries was measured.

One thing needs to be mentioned is that stress-induced martensitic transformation cannot be obtained in the elastic regime at room temperature due to the high stability of austenite. In addition, the EXDM can be conducted only at room temperature. Thus, the specimen was deformed within elastic regime first, and then subzero cooled by liquid nitrogen under external stress to obtain stress-induced martensitic transformation. The flow chart of the main experiment procedures is summarized in **Fig. 3.4**.

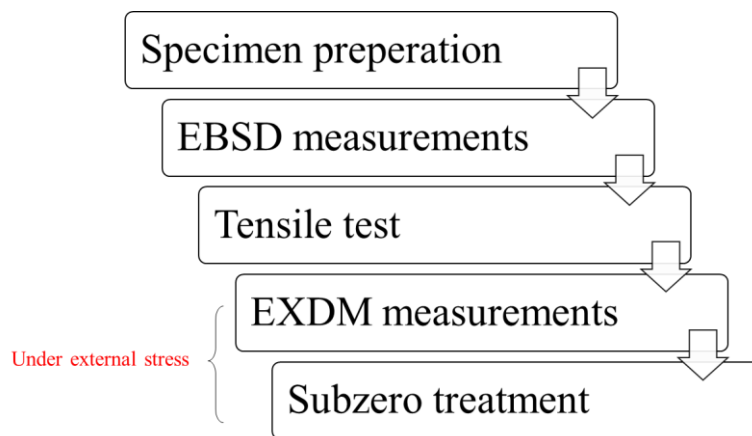


Figure 3.4 Flow chart of main experiment procedures of tensile specimen.

3.3 Results

3.3.1 Local stress distribution within austenite grains under elastic stress

Figure 3.5 presents (a, c) EBSD orientation maps and (b, d) EBSD phase maps of both surfaces of the tensile test specimen; (a, b) and (c, d) correspond to one surface and the other surface, respectively. The colors in the orientation map represent the crystallographic orientation parallel to the tensile direction as shown in the stereo triangle at the right side of the figures. The specimen exhibits a fully recrystallized microstructure with austenite single phase and the mean grain sized is 165.8 μm , which is a little smaller than the specimen thickness. Although, the microstructures of the two surfaces are different to each other, there is a penetrating grain as pointed out by the black arrows. This grain is termed “grain A” hereafter.

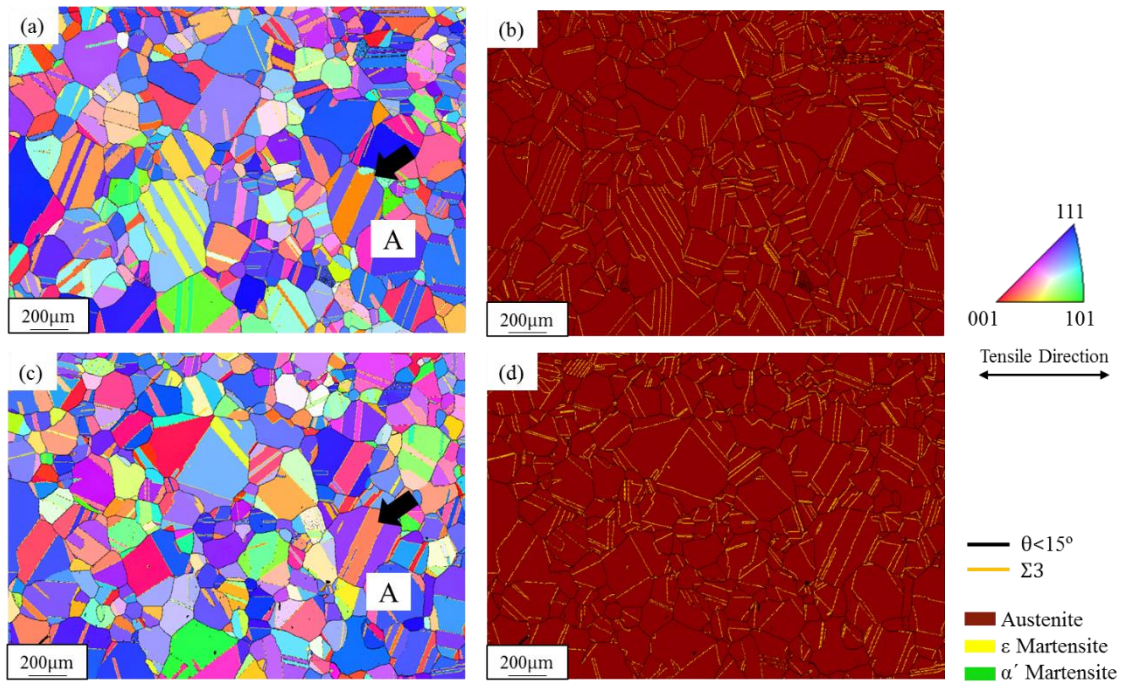


Figure 3.5 (a, c) EBSD orientation maps and (b, d) EBSD phase maps of both surfaces of the annealed tensile specimen. (a, b) and (c, d) correspond to one and the other surfaces, respectively. The black arrows indicate the grain A.

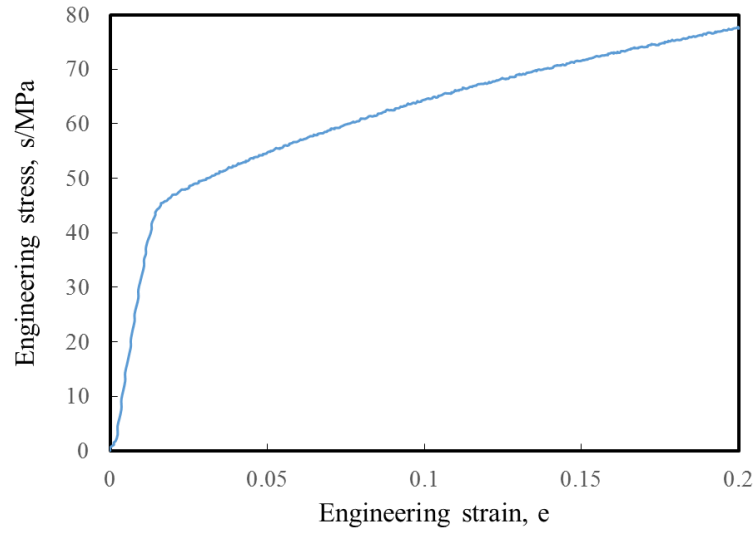


Figure 3.6 Engineering stress-strain curve of the tensile test specimen.

The engineering stress-strain curve of the tensile test is shown in **Fig. 3.6**. Because the yield strength of specimen is 43.7 MPa, the external stress of 40 MPa was applied to the specimen for the EXDM analysis.

Figure 3.7(a) shows an EBSD orientation map of the grain A before tensile deformation. High angle grain boundaries with misorientation larger than 15° and annealing twin boundaries are drawn in black and yellow lines, respectively. **Figure**

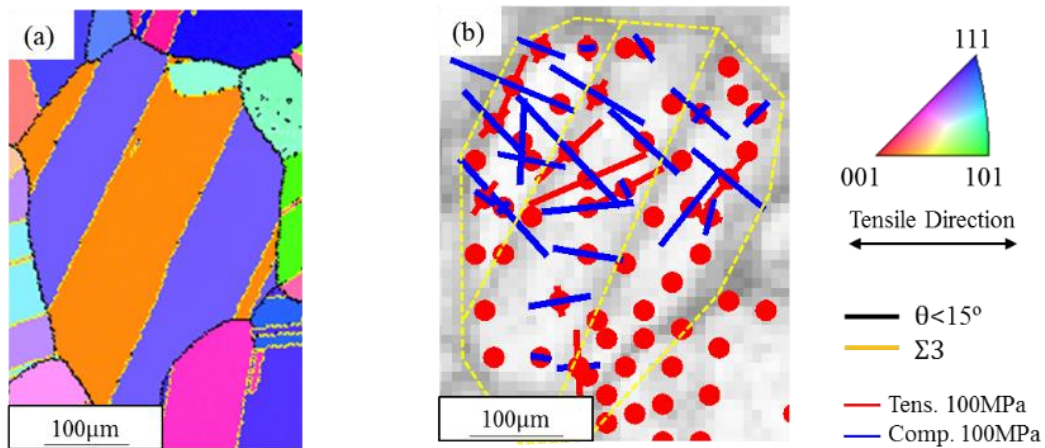


Figure 3.7 (a) EBSD orientation map of the grain A before tensile deformation, (b) EXDM grain boundary map of the grain A under external stress. The measured positions are indicated by red points. The length and direction of the lines correspond to the magnitude and direction of the local principal stress, and the color represent tensile stress (red) or compressive stress (blue).

3.7(b) shows an EXDM grain boundary map of the grain A under external stress. Here, the yellow dash lines are drawn on the grain boundary and annealing twins boundaries to identify the grain A easily. The red points in **Fig. 3.7(b)** indicate the positions where local stress fields were measured. The measured local stress fields are superimposed on the grain boundary map. The length and direction of the lines correspond to the magnitude and direction of the local principal stress, and the color represent tensile stress (red) or compressive stress (blue). Although the external tensile stress direction was horizontal direction as indicated by black arrow, most of the tensile components were substantially different from the external tensile stress direction. In addition, local stress field differed significantly depending on measured position.

Figures 3.8 shows (a) an EBSD orientation map and (b) a corresponding EXDM grain boundary map of another austenite grain in a different specimen. This grain is termed “grain B” hereafter. As well as the case of **Fig. 3.7**, the local stresses were much different from each other. Accordingly, it can be concluded that the local stress field distribution within polycrystalline materials was not uniform.

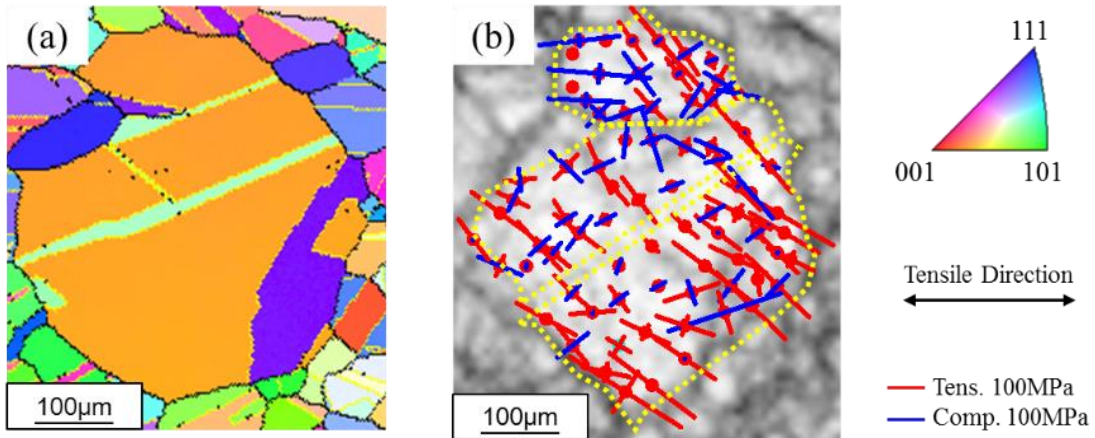


Figure 3.8 (a) EBSD orientation map of the grain B before tensile deformation, (b) EXDM grain boundary map of the grain B under external stress. The measured positions are indicated by red points. The length and direction of the lines correspond to the magnitude and direction of the local principal stress, and the color represent tensile stress (red) or compressive stress (blue).

3.3.2 Microstructure observation and crystallographic features of stress-induced martensitic transformation

Figure 3.9 shows (a) an EBSD orientation map, (b) an EBSD phase map, (c) an optical microscopy image, and (d) the $\{111\}_\gamma$ plane traces of the grain A after subzero cooled by liquid nitrogen under external stress. There are two martensite bands both of which are parallel to $(1-11)_\gamma$ plane. **Figures 3.10(a, b)** show the enlarged microstructure corresponding to the white broken rectangle in **Fig. 3.9(a)**. The martensite band consists

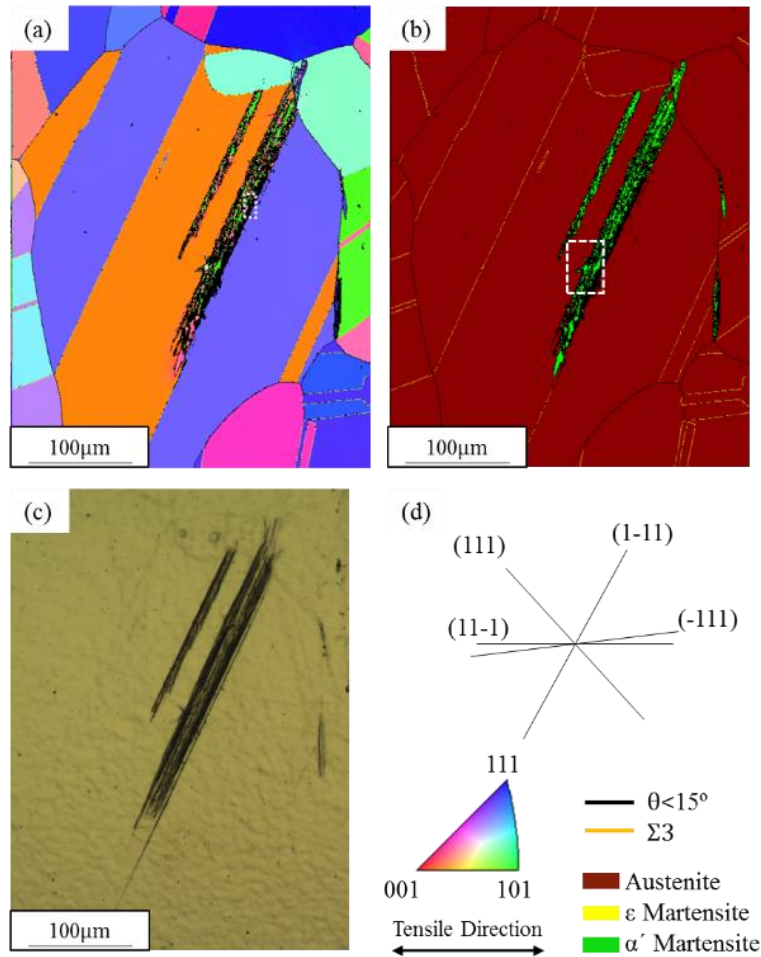


Figure 3.9 (a) EBSD orientation map, (b) EBSD phase map and (c) optical microscopy image, and (d) the $\{111\}_\gamma$ plane traces of the grain A after subzero cooled by liquid nitrogen under external stress.

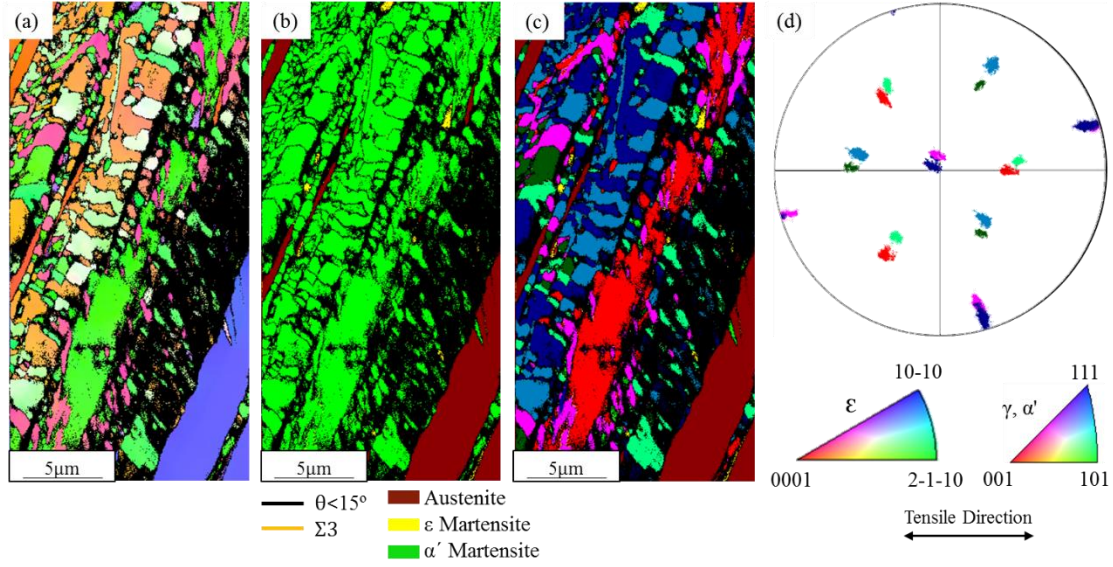


Figure 3.10 Enlarged (a) EBSD orientation map and (b) EBSD phase map corresponding to the white broken rectangle in **Fig. 3.9(a)**. (c) EBSD phase map on which all α' martensite variants are highlighted in different colors, (d) their $\{001\}$ pole figure in the corresponding colors.

of several α' martensite variants with various shapes and directions. Nevertheless, they maintain the macroscopic band shape which was also observed in the thermally induced martensite. In addition, ε martensite can be observed clearly among fine α' martensite variants, indicating that the sequence of the stress-induced martensitic transformation was also $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. Each α' variant and its $\{001\}_{\alpha'}$ pole are highlighted in the same color in **Figs. 3.10(c) and (d)**. These figures revealed that six α' variants formed within the martensite band. **Figure 3.11** presents pole figures of the (a-c) close-packed planes ($\{111\}_{\gamma}$, $\{0001\}_{\varepsilon}$, $\{110\}_{\alpha'}$) and (d-f) close-packed directions ($\langle 110 \rangle_{\gamma}$, $\langle 11-20 \rangle_{\varepsilon}$, $\langle 111 \rangle_{\alpha'}$) of austenite, ε martensite and α' martensite observed in **Fig. 3.10**. It is found that both close-packed planes and directions of all three phases are parallel to each other, and the S-N relationship between γ and ε phases (**Figs. 3.11(a), (b), (d), (e)**), the Burgers relationship between ε and α' phases (**Figs. 3.11(b), (c), (e), (f)**), and the K-S relationship

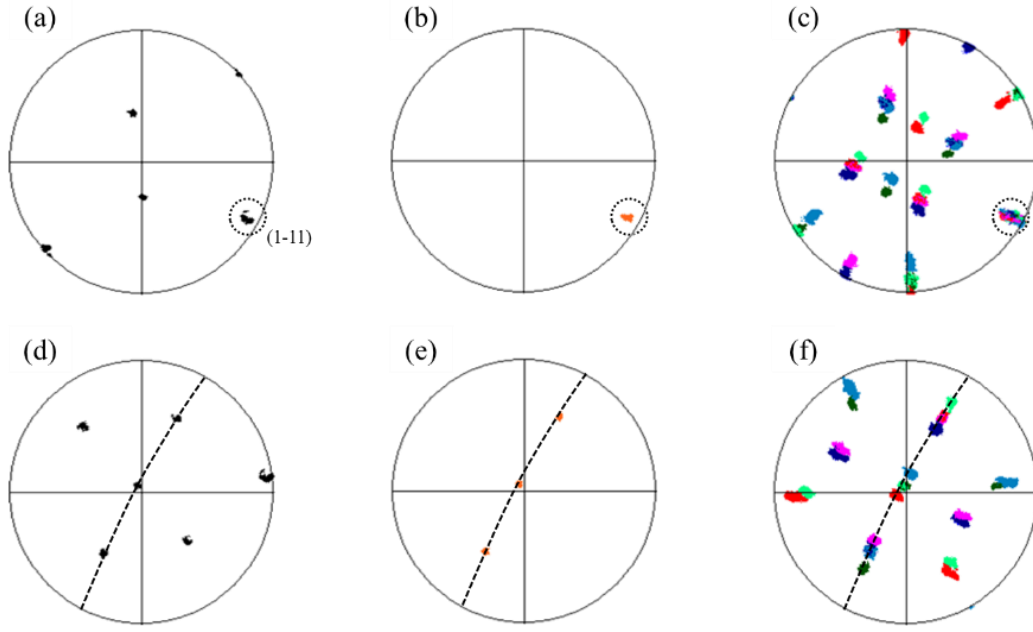


Figure 3.11 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\varepsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\varepsilon$, (f) $\{111\}_\alpha$ in the martensite band observed in **Fig. 3.10**.

between γ and α' phases (**Figs. 3.11(a), (c), (d), (f)**) are satisfied. Furthermore, as can be seen in **Fig. 3.11(c)**, all the six α' martensite crystals have the common $\{110\}_\alpha$ pole, indicating that they satisfied the same parallel plane relationship in the Burgers relationship and the K-S relationship.

Figure 3.12 shows (a) an EBSD orientation map, (b) an EBSD phase map, (c) an optical microscopy image, and (d) the $\{111\}_\gamma$ plane traces of the grain B after subzero treatment under external stress. Three martensite bands which are parallel to $(-111)_\gamma$ are observed. **Figures 3.13(a, b)** show the enlarged figure and corresponding $\{001\}_\alpha$ pole figure of the martensite band indicated by white rectangle in **Fig. 3.12(a)**. It is confirmed from **Fig. 3.13** that the band mainly consists of six variants. **Figure. 3.14** presents pole figures of the close-packed planes ($\{111\}_\gamma$, $\{0001\}_\varepsilon$, $\{110\}_\alpha$) and close-packed directions ($\langle 110 \rangle_\gamma$, $\langle 11-20 \rangle_\varepsilon$, $\langle 111 \rangle_\alpha$) of austenite, ε martensite and α' martensite variants observed within martensite band in **Fig. 3.13**. As well as the case of the grain A (**Fig. 3.11**),

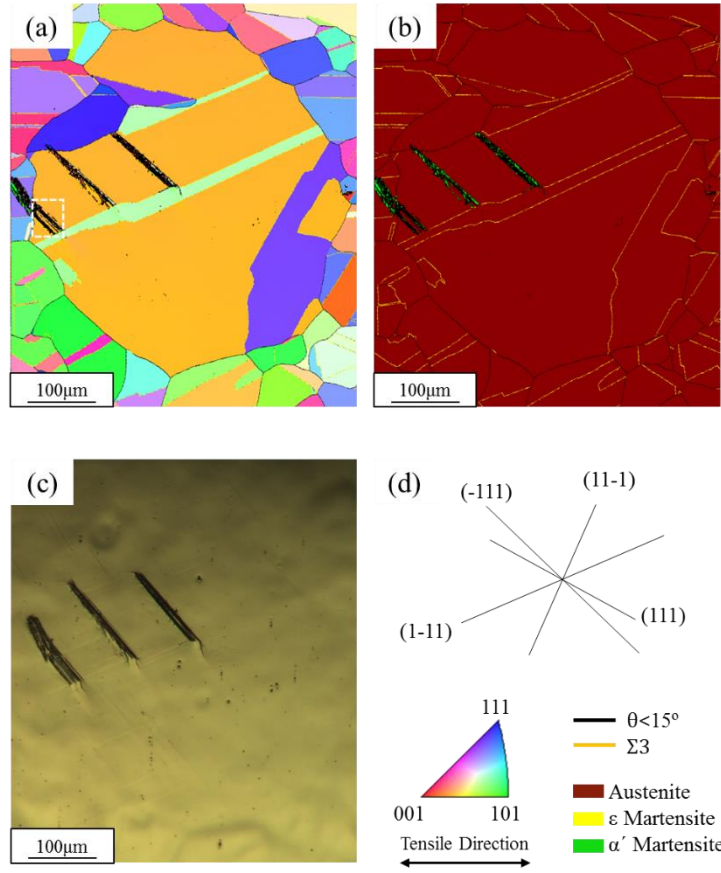


Figure 3.12 (a) EBSD orientation map, (b) EBSD phase map and (c) optical microscopy image, and (d) the $\{111\}_{\gamma}$ plane traces of grain B after subzero cooled by liquid nitrogen under external stress.

austenite/ ϵ martensite, ϵ martensite/ α' martensite, and austenite/ α' martensite satisfy the S-N relationship, the Burgers relationship and the K-S relationship, respectively. In additions, all the six α' variants observed in the martensite band have the same parallel plane relationship in the Burgers relationship and the K-S relationship.

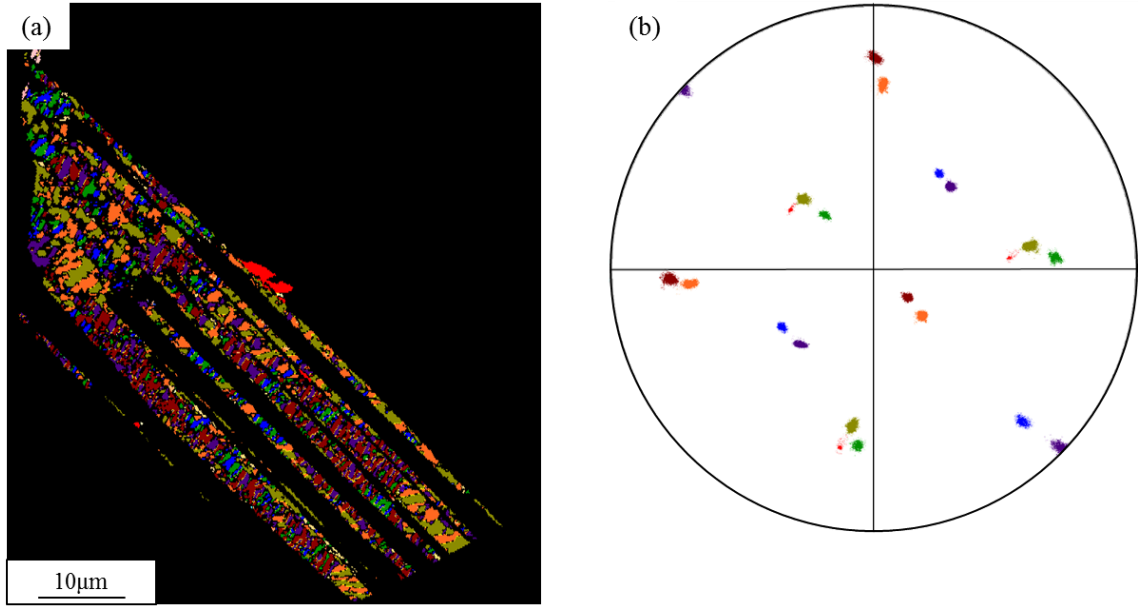


Figure 3.13 (a) Enlarged figure and (b) corresponding $\{001\}$ pole figure of the martensite band indicated by white rectangle in Fig. 3.12(a).

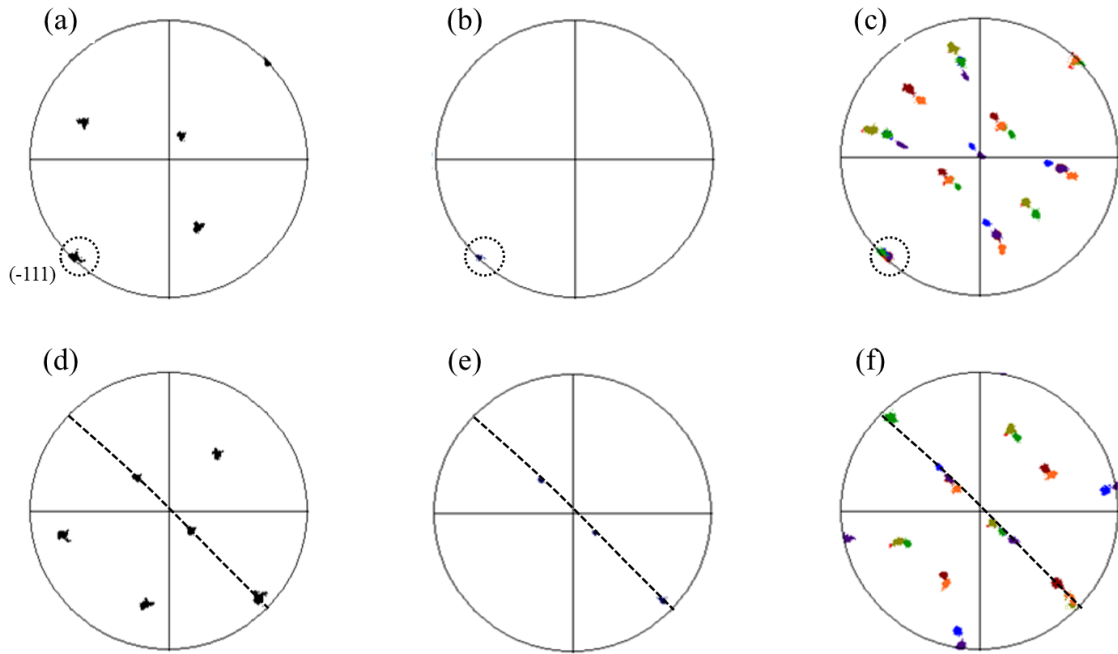


Figure 3.14 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\epsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\epsilon$, (f) $\{111\}_\alpha$ in the martensite band observed in Fig. 3.13.

3.4 Discussions

3.4.1 Comparison between thermally induced martensitic transformation and stress-induced martensitic transformation

The comparison of thermally induced martensite and stress-induced martensite is summarized as follows:

- (1) The sequence of both the thermally induced martensitic transformation and the stress-induced martensitic transformation was a $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. The S-N relationship between γ and ε phases, the Burgers relationship between ε and α' phases, and the K-S relationship between γ and α' phases were satisfied among three phases.
- (2) In both the thermally induced and the stress-induced martensitic transformation, α' martensite formed in a band shape which were parallel to $\{111\}_{\gamma}$. The martensite bands consisted of six α' variants which satisfied same parallel plane relationship in both the Burgers relationship and the K-S relationship.
- (3) The martensite bands formed in a given austenite grain were parallel to all four $\{111\}_{\gamma}$ planes in the thermally induced martensitic transformation. In contrast, all the martensite bands formed in a given austenite grain were parallel to one of the $\{111\}_{\gamma}$ in the case of the stress-induced martensitic transformation. This indicates that variant selection, as will be discussion in the next section, occurred in the stress-induced martensitic transformation.

3.4.2 Variant selection of stress-induced martensitic transformation

In Figs. 3.15(a) and (b), the EXDM grain boundary maps of the grains A and B are superimposed on the EBSD orientation maps. Two local stress fields pointed out by arrows correspond to the region where thick α' martensite band formed. Unfortunately, the local stress field pointed out by white arrow was not successfully obtained. Thus, the local stress field indicated by black arrow was used for variant selection analysis of the grain A. In the grain B, the point indicated by black arrow not only located within the martensite band, but also near the grain boundary. Thus, the local stress field of the position was selected for variant selection analysis of the grain B.

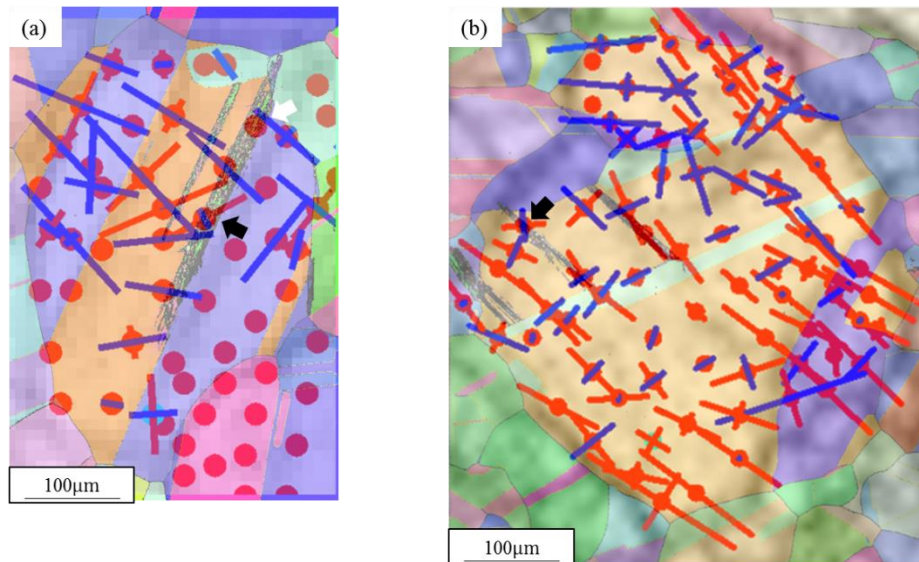


Figure 3.15 EXDM grain boundary maps of (a) the grain A and (b) the grain B superimposed on the EBSD orientation maps.

As mentioned in the previous section, six α' variants which satisfied same parallel plane relationship in the Burgers relationship were observed. That is, all the six α' variants that could form from a ε variant were confirmed. However, all the martensite bands formed in stress-induced martensitic transformation were parallel to one of the $\{111\}_{\gamma}$. This indicates that the variant selection only occurred during austenite to ε martensite

transformation.

As introduced in **Chapter 1**, the FCC to HCP transformation is accomplished by the movements of $\frac{a}{6}\langle 112 \rangle_\gamma$ Shockley partial dislocations. Each of three $\frac{a}{6}\langle 112 \rangle_\gamma$ Shockley partial dislocation motions on a same $\{111\}_\gamma$ plane results in the formation of the same ϵ martensite variant. This means that there are four ϵ martensite variants that could form from a single grain of austenite.

Figure 3.16 shows the shear stresses on twelve Shockley partial dislocations in the grain A, calculated by (a) the uniaxial external stress and (b) the measured local stress field. Because the observed ϵ variants were parallel to $(1-11)_\gamma$, three Shockley partial dislocations on $(1-11)_\gamma$ correspond to the observed ϵ variant in the grain A, which are indicated by red color in **Fig. 3.16**. The blue stars indicate the Shockley partial dislocations on which exerted shear stress was maximum. When the uniaxial external tensile stress was used (**Fig. 3.16(a)**), a Shockley partial dislocation other than the ones corresponding to the observed ϵ variant shows the largest shear stress, has the largest shear stress. In contrast, it can be easily confirmed from **Fig. 3.16(b)** that one of the Shockley partial dislocation corresponding to the observed ϵ variant has the largest shear stress.

The shear stress calculated for the grain B is also shown in **Figure 3.17**. The results indicate that the Shockley partial dislocations corresponding to the observed ϵ variant have the largest shear stress in the calculations using both uniaxial external stress and the measured local stress field. This is because the measured local stress field was similar to the uniaxial external tensile stress as shown in **Fig. 3.16**. Therefore, it can be concluded that the ϵ martensite variant, whose corresponding Shockley partial dislocation has largest shear stress formed preferentially.

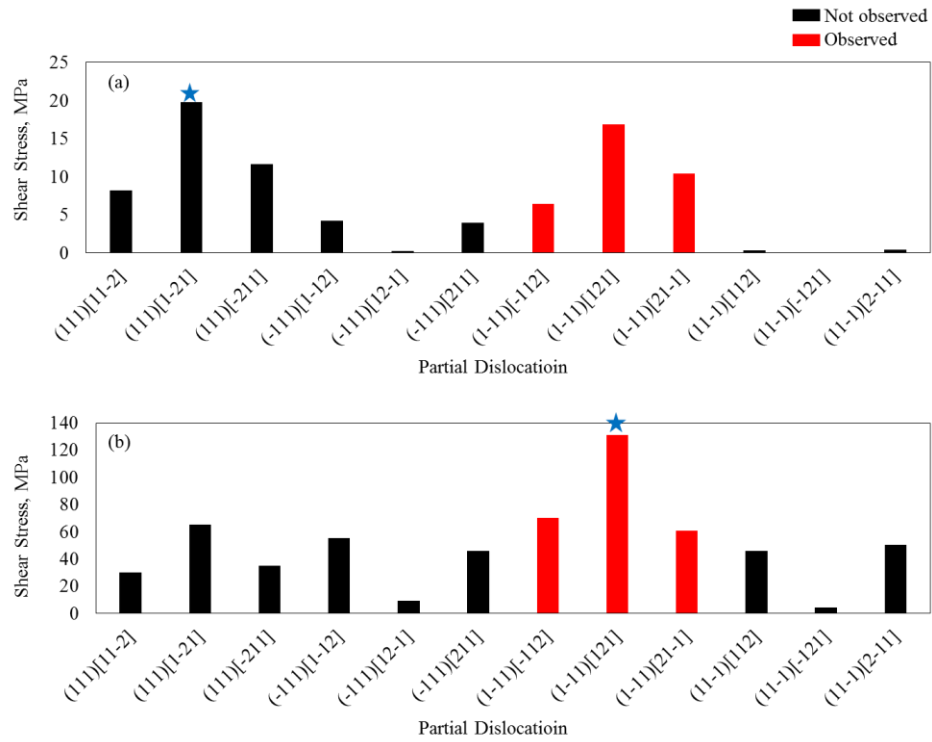


Figure 3.16 The shear stresses on twelve Shockley partial dislocations in the grain A, calculated by (a) the uniaxial external stress and (b) the measured local stress.

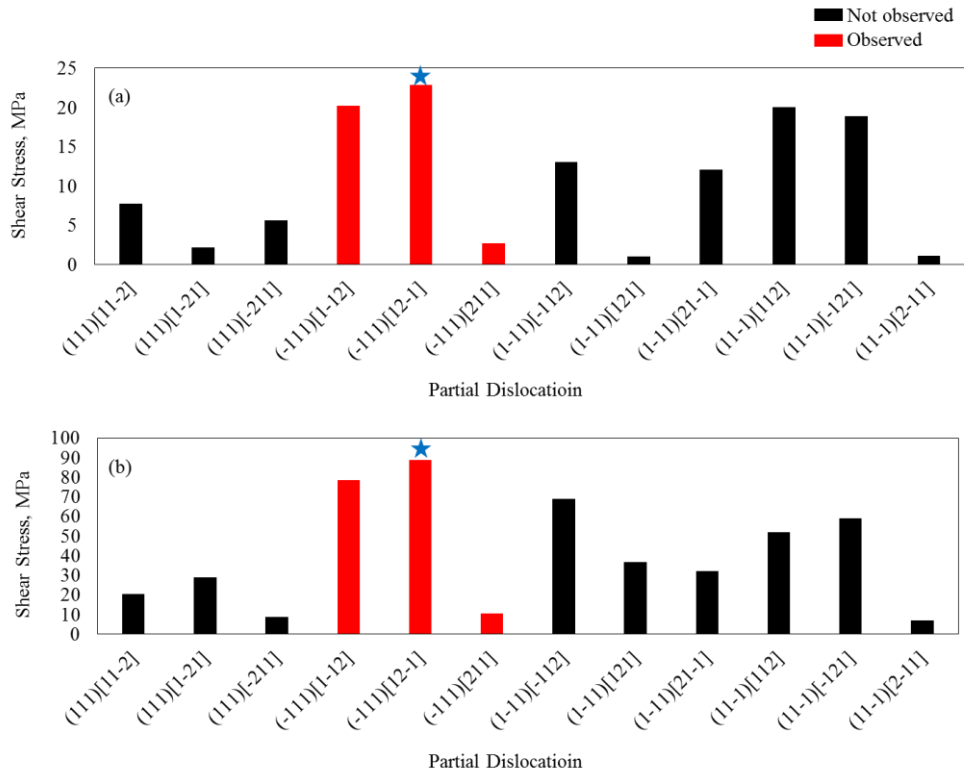


Figure 3.17 The shear stresses on twelve Shockley partial dislocations in the grain B, calculated by (a) the uniaxial external stress and (b) the measured local stress.

3.5 Conclusions

In this chapter, we studied the morphology, crystallographic features of stress-induced martensitic transformation and the correlation between variant selection and local stress field in the stress-induced martensitic transformation by EBSD measurements and EXMD technique. The main conclusions are summarized as follows:

(1) The local stress field was successfully measured by the EXDM technique. The measured local stress fields were different depending on the positions even in the same austenite grain. This suggested that the local stress field distribution in polycrystalline materials was considerably complicated.

(2) The sequence of stress-induced martensitic transformation was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. The S-N relationship between γ and ε phases, the Burgers relationship between ε and α' phases, and the K-S relationship between γ and α' phases were satisfied among three phases. The martensite bands which were parallel to one of $\{111\}_\gamma$ consisted of retained ε martensite and six α' variants.

(3) The transformation mechanism of stress-induced martensitic transformation in SUS304 austenitic stainless steel can be explained as follows: the ε martensite variant whose corresponding Shockley partial dislocation has largest shear stress form preferentially. Then the ε variant transforms into all the six α' variants that could form from one ε martensite variant.

3.6 References

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Chapter 4: Strain-induced martensitic transformation in SUS304 austenitic stainless steel

4.1 Introduction

Strain-induced martensitic transformation occurs during plastic deformation of metastable austenite. Because it can improve both strength and ductility through TRIP effect, many studies on strain-induced martensite in austenitic stainless steels, such as mechanical properties, nucleate sites, transformation behavior, have been studied [1-12]. However, there are still disagreement with their results.

As introduced in **Chapter 1**, a key character of strain-induced martensitic transformation is variant selection [13-25]. Kundu and Bhadeshia [15] and Viana et al. [16] reported that variant selections of austenitic stainless steel and maraging-350 steel could be accounted by Patel and Cohen theory [14]. On the other hand, Kato et al. and Higo et al. suggested that the martensite variants with large Schmid factor on the first shear of Bogers-Burgers model [19] formed preferentially in austenitic stainless steels [20-22]. Several other models for explaining the variant selection in deformation-induced martensitic transformation have also been proposed [23-25]. However, variant selection rules, especially in polycrystalline materials, have not yet been clearly understood.

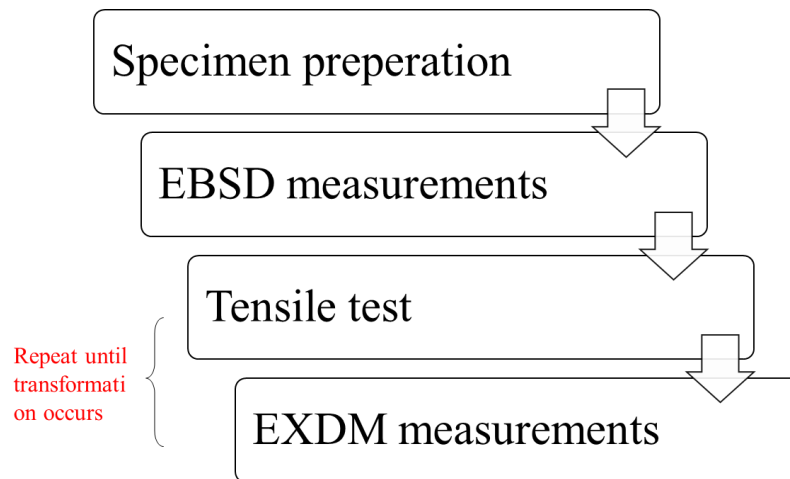
This chapter aims to investigate the morphology, crystallographic features of strain-induced martensitic transformation, and to clarify the correlation between variant selection and local stress field by EXDM technique.

4.2 Experimental procedures

The as-received material was homogenized at 1000 °C for 3 hours in vacuum and then furnace cooled. The annealed sheets were cold rolled by 10% to reduce the thickness to 0.9 mm. Tensile test specimens, whose size is schematically shown in **Fig. 3.3**, were cut from the cold rolled sheet and mechanically polished until the final specimen thickness of 0.5 mm was reached. Then, the specimens were annealed at 1050 °C for 70~85 hours under argon atmosphere followed by furnace cooling. A fully-recrystallized homogeneous microstructure of γ single phase was obtained by this heat treatment. After the heat treatment, only electrolytic polishing was conducted to reduce the thickness of the specimen to avoid deformation-induced martensitic transformation during mechanical polishing. The electrolytic solution was 10% HClO₄ + 90% CH₃COOH. The electrolytic voltage was 12 V and the polishing time was 30 ~ 50 minutes. The microstructures of both surfaces of tensile test specimen were measured by EBSD.

The prepared tensile test specimen was installed at the miniaturized tensile test machine at beamline BL28B2 in SPring-8. The crystal lattice spacing without external stress was measured first. Then the specimen was plastically deformed in tension at a strain rate of $8.4 \times 10^{-4} \text{ s}^{-1}$ at room temperature. The EXDM was then carried out for the deformed tensile test specimen under the external stress. The EXDM technique was repeated at identical positions by increasing the tensile strain until clear martensitic transformation could be identified by optical microscopy observation. The local stress measured just before the transformation was used for the variant selection analysis. As in the case of stress-induced martensitic transformation, the local stress field at the positions near grain boundaries were measured. The detailed procedure of EXDM was described in **Chapter 3**. The flow chart of the main experimental procedures is summarized in **Fig. 4.1**.

Figure 4.1 Flow chart of main experiment procedures of tensile specimen.



4.3 Results

4.3.1 Local stress evolution with increasing the plastic strain

In this section, the changes in local stress field during tensile deformation at 2 positions in different grains, termed as “grain C” and “grain D” in the following, were selected to show the major tendency. **Figure 4.2** shows EBSD orientation maps of the grain C (a – c) before deformation and (d) after tensile deformation to a strain of 4.23%. The colors indicate the orientations parallel to the tensile direction. The measured local stress fields at the tensile strains of 0%, 0.33%, 0.78%, and 4.23% are superimposed on **Fig. 4.2**. The length and direction correspond to the magnitude and direction of the principal stress. In addition, the color of lines represent tensile stress (red) and compressive stress (blue). It was found from **Fig. 4.2(a)** that a large compressive stress approximately perpendicular to the tensile direction was induced only by fixing the specimen to the test machine. With increasing the tensile strain, both magnitude and direction of tensile and compression components of principal stress changed. It was confirmed that deformation-induced martensite did not form after the tensile deformation to the strain of 4.23%.

Figure 4.3 shows EBSD orientation maps of the grain D (a - c) before and (d) after deformed to 4.23% strain. **Figure 4.3(e)** is an EBSD phase map corresponding to (d), where dark red and green colors represent austenite and martensite phases, respectively. The evaluated local stress field at the tensile strains of 0%, 0.33%, 0.78%, and 4.23% are also indicated in **Figs. 4.3(a – d)**. As well as the case of **Fig. 4.2(a)**, compressive stress was induced only by fixing the specimen to the test machine (**Fig. 4.3(a)**). With increasing the tensile strain, both magnitude of compressive and tensile components increased. Moreover, the direction of the principal stress drastically changed when the tensile strain was increased to 4.23%. It can be found from **Figs. 4.3(d) and (e)** that a deformation-

induced martensite appeared at the tensile strain of 4.23%. By observing the microstructures by optical microscopy during the whole deformation process carefully, no martensitic transformation occurred within the grain D until the tensile strain of 0.78%. Accordingly, it can be concluded that the drastic change in the principal stress was due to the formation of deformation-induced martensite.

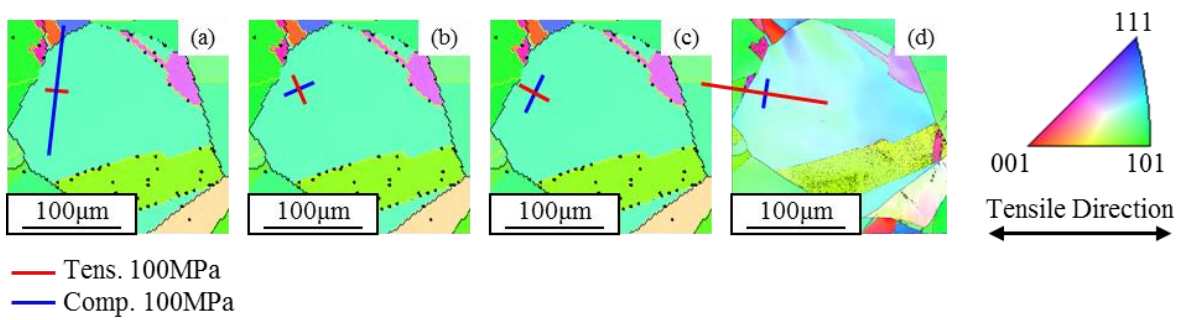


Figure 4.2 EBSD orientation maps in a grain (a) – (c) before and (d) after tensile deformation. The measured local stress fields at the tensile strains of (a) 0%, (b) 0.33%, (c) 0.78% and (d) 4.23% are superimposed. The length and direction correspond to the magnitude and direction of the principal stress. In addition, the color of lines represent tensile stress (red) and compressive stress (blue).. The color of lines represent tensile stress (red) and compressive stress (blue).

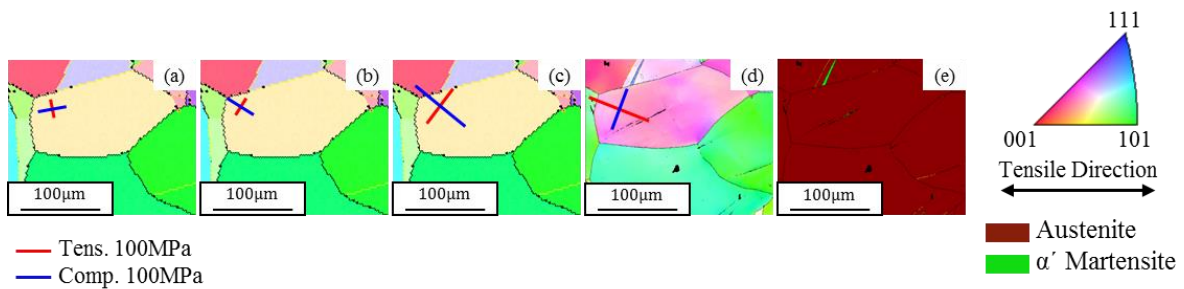


Figure 4.3 EBSD orientation maps in a grain (a) – (c) before and (d) after tensile deformation. The measured local stress fields at the tensile strains of (a) 0%, (b) 0.33%, (c) 0.78% and (d) 4.23% are superimposed. The length and direction correspond to the magnitude and direction of the principal stress. In addition, the color of lines represent tensile stress (red) and compressive stress (blue).. The color of lines represent tensile stress (red) and compressive stress (blue).

4.3.2 Microstructure observation and crystallographic features

In this section, another austenite grain in a different specimen, which is termed “grain E” hereafter, is selected to show the results. The tensile test was stopped at larger strains of 1.74%, 3.52%, 13.96% and 20.00% and EXDM was conducted at the strains of 1.74%, 3.52%, 13.96%. **Figure 4.4** shows (a) an EBSD orientation map and (b) an EBSD phase map of the grain E after the final strain of 20.00%. Several martensite bands formed

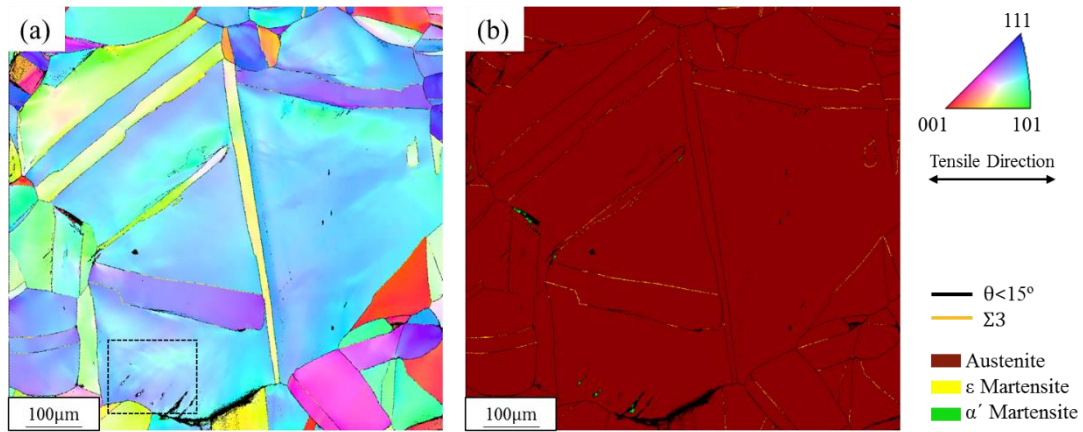


Figure 4.4 (a) EBSD orientation map and (b) EBSD phase map of the grain E after deformation.

around grain boundary as indicated by the black rectangle. **Figure 4.5** shows the enlarged microstructures of the black rectangle area in **Fig. 4.4(a)**. The thicker martensite bands (black arrows) and thinner martensite bands (white arrows) elongating in two different directions can be found. The $\{111\}_{\gamma}$ plane traces of the grain E after deformation to the strain of 20.00% is superimposed on the EBSD phase map (**Fig. 4.5(b)**). As can be seen from **Fig. 4.5(b)**, the elongated direction of thinner martensite bands is slightly deviated from $(-111)_{\gamma}$ plane trace, and that of thicker martensite bands is also deviated from $(111)_{\gamma}$ and $(11-1)_{\gamma}$ plane traces. Most of the strain-induced martensite bands exhibit somewhat irregular compared with that of thermally and stress- induced martensite. In addition, ϵ

martensite, as pointed out by white arrows in **Fig. 4.5(b)**, can be observed clearly among α' martensite, indicating that the sequence of strain-induced martensitic transformation was also $\gamma \rightarrow \varepsilon \rightarrow \alpha'$.

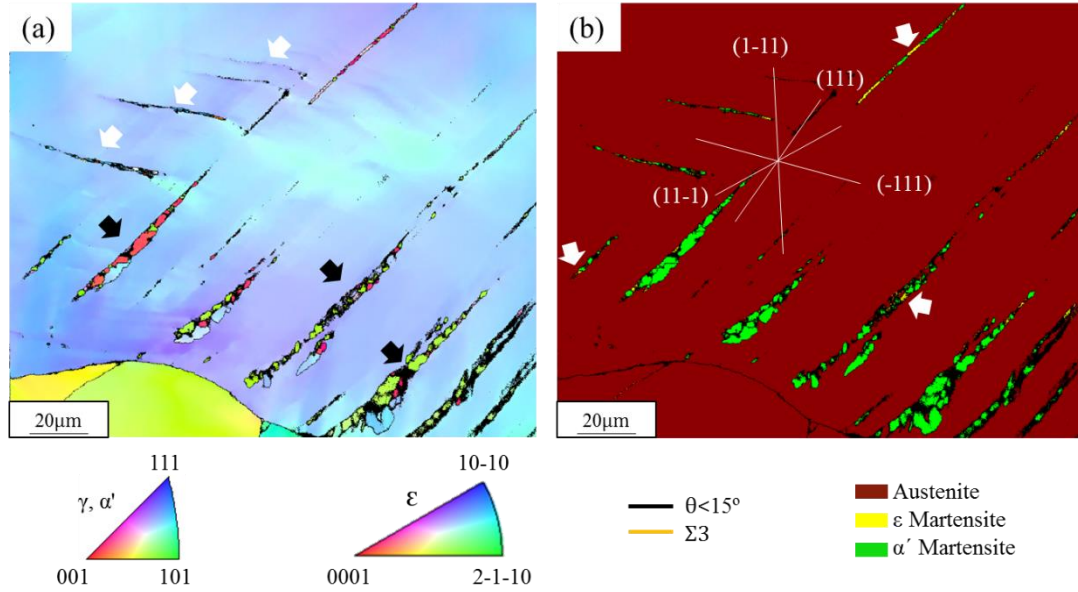


Figure 4.5 Enlarged (a) EBSD orientation map and (b) EBSD phase map of the area corresponding to the black broken rectangle in **Fig. 4.4**.

The crystallographic features of the strain-induced martensite were analyzed in two areas (the area A and the area B) indicated by white broken rectangle in **Fig. 4.6**. In both areas, the austenite, ε martensite and α' martensite variants are highlighted in black, dark red and other colors, respectively. The each martensite band consists of ε martensite and two or three α' martensite variants. **Figures 4.7 and 4.8** present pole figures of the (a - c) close-packed planes ($\{111\}_\gamma$, $\{0001\}_\varepsilon$, $\{110\}_\alpha$) and (d - f) close-packed directions ($\langle 110 \rangle_\gamma$, $\langle 11-20 \rangle_\varepsilon$, $\langle 111 \rangle_\alpha$) of austenite, ε martensite and α' martensite observed in the areas A and B, respectively. The colors in the pole figures are the same as that in **Fig. 4.6**. As can be found from **Fig. 4.7**, both the close-packed planes and directions of all three phases are parallel to each other, and the S-N relationship between γ and ε phases (**Figs. 4.7(a), (b)**,

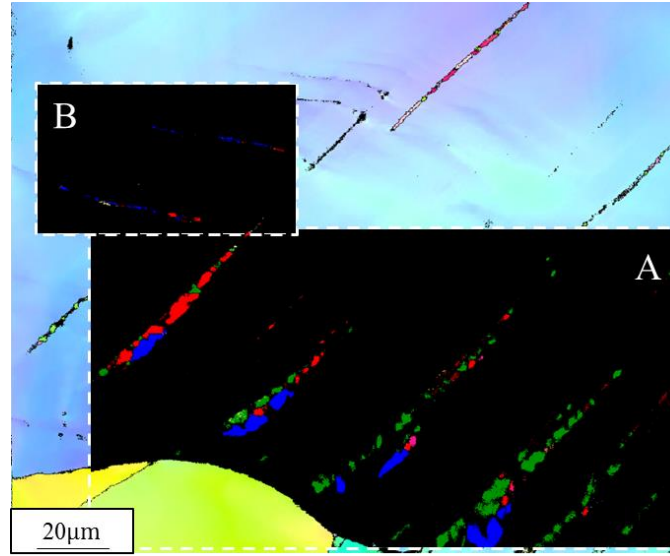


Figure 4.6 Two areas A and B selected for crystallographic analysis in **Figs. 4.7 and 4.8**, respectively.

(d), (e)), the Burgers relationship between ε and α' phases (**Figs. 4.7(b), (c), (e), (f)**), and the K-S relationship between γ and α' phases (**Figs. 4.7(a), (c), (d), (f)**) are satisfied. Furthermore, it was confirmed that three α' martensite variants have the common $\{110\}_{\alpha'}$ pole in the area A. This indicates that they satisfied same parallel plane relationship in the Burgers relationship and the K-S relationship. The martensite bands consist of two α' variants in the area B as shown in **Fig. 4.8(c)**. As well as the area A, both the close-packed planes and directions of all three phases are parallel to each other, and the three orientation relationship are satisfied.

As can be confirmed from the pole figures (**Figs. 4.7(a) and (c), Figs. 4.8(a) and (c)**), the common $\{110\}_{\alpha'}$ planes of α' variants in the areas A and B are parallel to of $(11-1)_{\gamma}$ plane and $(-111)_{\gamma}$ plane of austenite matrix, respectively. However, as mentioned previously, the elongated direction of these bands were deviated from the plane traces (**Fig. 4.5(b)**). This might be attributed to the change in orientation by severe surface relieves due to the large plastic deformation, which can be confirmed by the change in

color of austenite in the orientation map (**Fig. 4.5(a)**) and the spreading of each poles in the pole figures in **Figs. 4.7 and 4.8**.

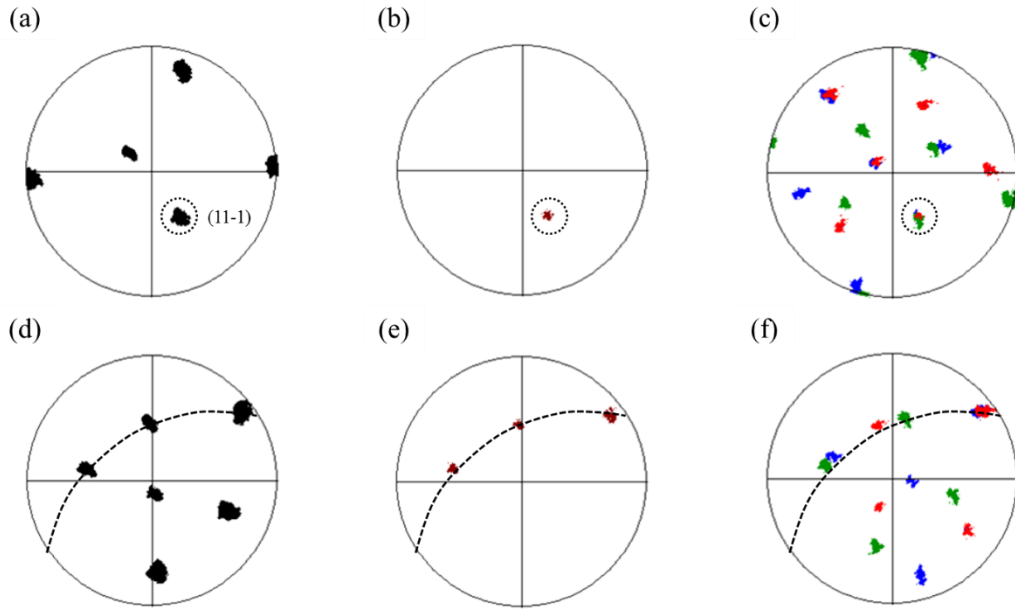


Figure 4.7 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\epsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\epsilon$, (f) $\{111\}_\alpha$ of the martensite bands in the area A.

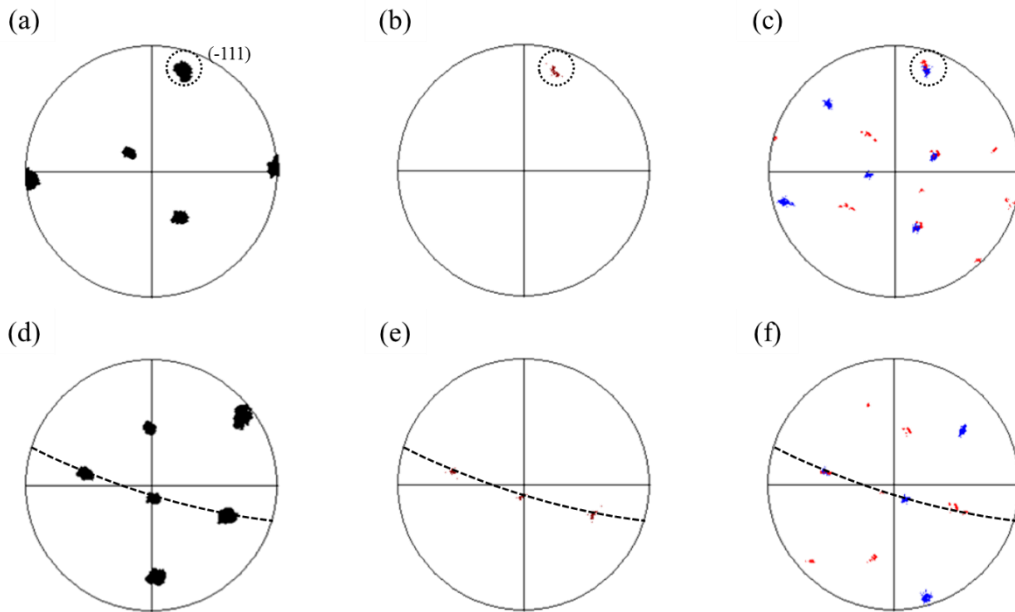


Figure 4.8 Pole figures of (a) $\{111\}_\gamma$, (b) $\{0001\}_\epsilon$, (c) $\{011\}_\alpha$, (d) $\{111\}_\gamma$, (e) $\{11-20\}_\epsilon$, (f) $\{111\}_\alpha$ of the martensite bands in the area B.

4.4 Discussions

4.4.1 Variant selection of strain-induced martensitic transformation

As reported in **Section 4.3.1**, the local stress field changed after the formation of martensite. Thus, for the variant selection analysis, it is necessary to use the local stress field before martensitic transformation occurred. **Figure 4.9** shows optical microscopy images of the grain E deformed to tensile strains of (a, d) 3.52%, (b, e) 13.96% and (c, f) 20.00%. The grain boundary is drawn by white lines. **Figures 4.9(d), (e) and (f)** are the enlarged microstructures of the red broken rectangles in (a), (b) and (c), respectively. The martensite bands indicated by white broken lines can be found in **Figs. 4.9(e) and (f)**, which indicates that strain-induced martensitic transformation had already occurred when the specimen was deformed to strain of 13.96%. While there was no martensite band at the strain of 3.52%. Therefore, the local stress field measured at the tensile strain of 3.52% was used for the variant selection analysis in the grain E.

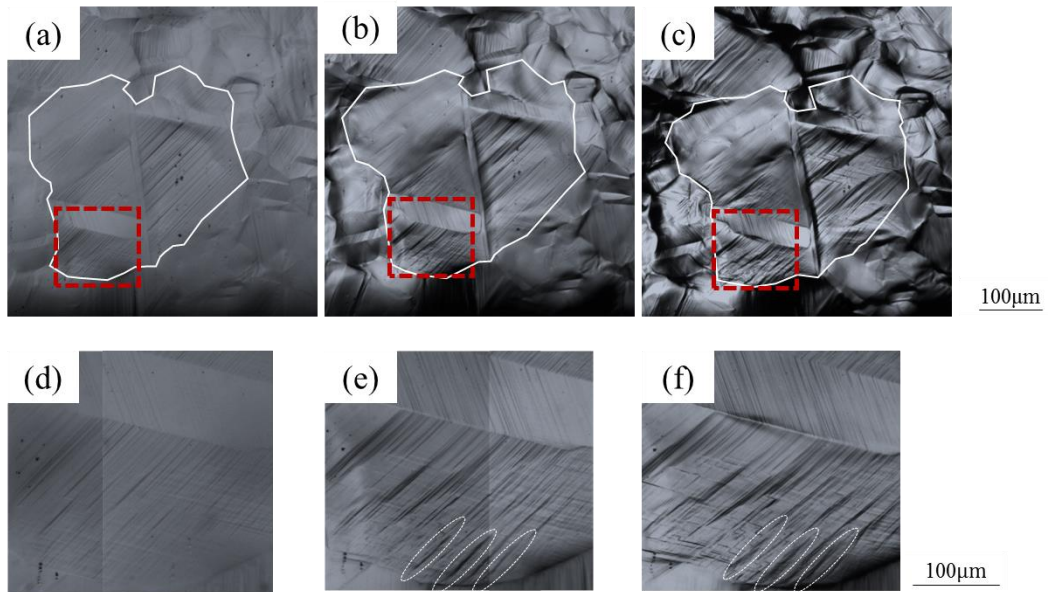


Figure 4.9 Optical microscopy images of the grain E deformed to tensile strains of (a, d) 3.52%, (b, e) 13.96% and (c, f) 20.00%. (d), (e) and (f) are the enlarged microstructures of red broken rectangles in (a), (b) and (c), respectively.

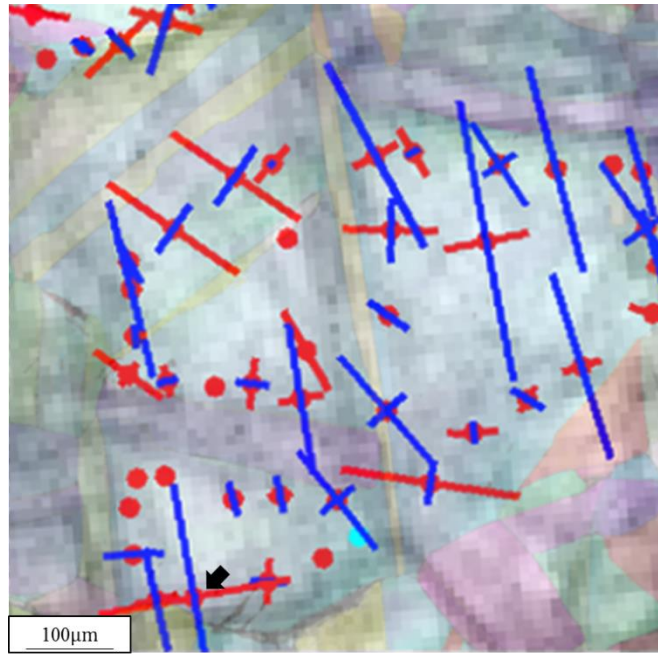


Figure 4.10 EXDM grain boundary maps of the grain E superimposed on the EBSD orientation maps.

As mentioned in the previous sections, according to the S-N and Burgers relationships, four ϵ variants can form from a single grain of austenite, and six α' variants can form from a ϵ variant. According to the observation in **Section 4.3.2**, two ϵ variants formed in the austenite grain, and only two or three α' variants formed in each ϵ variant. This indicates that variant selection occurred during both $\gamma \rightarrow \epsilon$ and $\epsilon \rightarrow \alpha'$ transformations. Because the local stress field around martensite bands in the area B (**Fig. 4.6**) was not measured, the variant selection occurred in the area A was focused in the following. **Figure 4.10** shows an EXDM grain boundary map corresponding to the grain E superimposed on the EBSD orientation map. The local stress field pointed out by black arrow is not only located within the martensite band, but also near the grain boundary. Thus, the local stress field of that position was selected for variant selection analysis in the following.

4.4.1.1 Analysis according to Patel and Cohen model

Humbert et al. analyzed the variant selection of $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ martensitic transformation induced by 10% plastic deformation in 304 stainless steel at -60 °C [13]. Mechanical work due to external stress on each individual austenite grains was calculated for the $\gamma \rightarrow \varepsilon$ and $\varepsilon \rightarrow \alpha'$ transformations. The variant selection was well explained in the $\gamma \rightarrow \varepsilon$ transformation but the $\varepsilon \rightarrow \alpha'$ transformation was more complicate because of the superposition of the applied stress and the internal stresses due to the formation of the ε martensite. They suggested that a sophisticated model should be established to explain the variant selection accurately. Later, Kundu and Bhadeshia also reported the variant selection using Humbert's result. According to their results, the transformation texture could be adequately calculated by considering direct transformation from austenite to α' martensite based on Patel and Cohen model [15].

The orientation relationships among austenite, ε martensite and α' martensite in the present study are summarized as **Table 4.1**. Although the transformation sequence was

Table 4.1 The orientation relationship among austenite, ε martensite and α' martensite in the SUS304 austenitic stainless steel.

Phase	γ	ε	α'
Close-packed plane	$\{111\}$	$\{0001\}$	$\{011\}$
Close-packed direction	$\langle 1-10 \rangle$	$\langle 11-20 \rangle$	$\langle 111 \rangle$
Orientation relationship	$\leftarrow \text{Shoji-Nishiyama} \rightarrow$		
		$\leftarrow \text{Burgers} \rightarrow$	
	$\leftarrow \text{Kurdjumov-Sachs} \rightarrow$		

$\gamma \rightarrow \varepsilon \rightarrow \alpha'$, austenite and α' martensite still satisfied the K-S relationship. Thus, in this section, Patel and Cohen model is used for variant selection analysis assuming that intermediate ε martensitic transformation does not affect the variant selection of α' .

According to Patel and Cohen model [14], the mechanical work (U) done by the applied stress for the martensitic transformation can be calculated by the equation shown below:

$$U = \sigma_n \varepsilon_0 + \tau \gamma_0 \quad (1.8)$$

where σ_n is the resolved normal stress perpendicular to the habit plane, ε_0 is the transformation strain component normal to habit plane, τ is the resolved shear stress along the shear direction on the habit plane, and the γ_0 is the transformation shear strain. In this model, martensite variants with larger positive U are expected to form preferentially. The habit plane orientation $\{-0.412 -0.807 -0.423\}$, shear direction $\langle 0.509 -0.589 0.627 \rangle$, the normal component of the transformation strain 0.022, and transformation shear strain 0.138 obtained from calculation using phenomenological theory of martensite crystallography were used [26-30]. The variant number in the following is based on **Table 2.1**.

Figure 4.11 presents the mechanical works for the 24 martensite variants in the grain E calculated using (a) the uniaxial external stress and (b) the local stress measured by EXDM. The red bars represent the observed variants, while the black ones represent the not observed variants. It is found from **Fig. 4.11** that, the variants which possess largest positive mechanical work did not form whichever calculated by uniaxial external stress and measured local stress field. Accordingly, variant selection in the strain-induced martensitic transformation cannot be accounted by Patel and Cohen model.

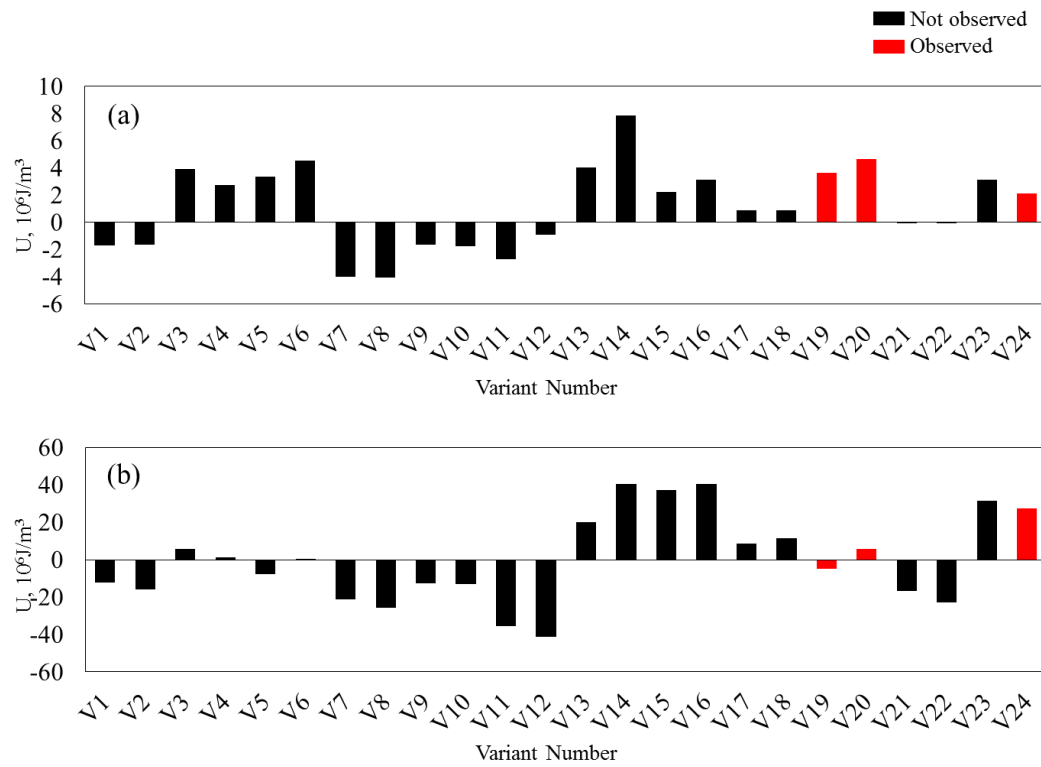


Figure 4.11 The mechanical works for the 24 martensite variants in the grain E calculated using (a) the uniaxial external stress and (b) the local stress measured by EXDM.

4.4.1.2 Analysis according to Bogers-Burgers model

Bogers-Burgers model is that a transformation process from FCC to BCC crystal are accomplished by two kinds of shears [19]. The first shear is $\frac{a_{fcc}}{18} \langle 112 \rangle$ on $\{111\}$ plane to change the atomic configuration on the close-packed plane of FCC, and the second shear is $\frac{a_{fcc}}{16} \langle 112 \rangle$ on $\{111\}$ plane to change the stacking sequence from FCC to BCC. In **Table 4.3**, two shears of Bogers-Burgers model to form 24 variants in the K-S relationship are listed. Because the martensite variants with larger stress on the shears of Bogers-Burgers model are expected to transform preferentially, the shear stresses on first shear and second shear corresponding to 24 variants were calculated.

Figure 4.12 presents the calculated shear stress for the 24 martensite variants in the grain E according to (a, b) the uniaxial external stress and (c, d) the measured local stress field. **Figures 4.12(a, c) and (b, d)** correspond to the shear stresses on the first shear and the second shear, respectively. The red bars represent the observed variants, while the black ones represent the not observed variants. **Figures 4.12** suggests that the shear stresses of the selected variants were both positive and negative values whichever calculated by the uniaxial external stress and the measured local stress field. Meanwhile, many variants possess large positive shear stress did not form. Accordingly, it can be concluded that no special relationship was found between shear stress and variant selection by Bogers-Burgers model.

Table 4.3 Bogers-Burgers shears of 24 variants in the K-S orientation relationship.

Variant	Close-packed plane	Close-packed direction	Bogers-Burgers Model			
No.	$(\gamma) // (\alpha')$	$[\gamma] // [\alpha']$	First shear		Second shear	
V1	$(111) // (011)$	$[-101] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[1-1-2](111)$	$\frac{a_{fcc}}{16}$	$[11-2](111)$
V2	$(111) // (011)$	$[-101] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[-2-11](111)$	$\frac{a_{fcc}}{16}$	$[-211](111)$
V3	$(111) // (011)$	$[01-1] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[-1-21](-111)$	$\frac{a_{fcc}}{16}$	$[1-21](111)$
V4	$(111) // (011)$	$[01-1] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[-11-2](-111)$	$\frac{a_{fcc}}{16}$	$[11-2](111)$
V5	$(111) // (011)$	$[1-10] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[-21-1](11-1)$	$\frac{a_{fcc}}{16}$	$[-211](111)$
V6	$(111) // (011)$	$[1-10] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[1-2-1](11-1)$	$\frac{a_{fcc}}{16}$	$[1-21](111)$
V7	$(1-11) // (011)$	$[10-1] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[-211](111)$	$\frac{a_{fcc}}{16}$	$[-2-11](1-11)$
V8	$(1-11) // (011)$	$[10-1] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[11-2](111)$	$\frac{a_{fcc}}{16}$	$[1-1-2](1-11)$
V9	$(1-11) // (011)$	$[-1-10] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[-1-21](-111)$	$\frac{a_{fcc}}{16}$	$[121](1-11)$
V10	$(1-11) // (011)$	$[-1-10] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[211](-111)$	$\frac{a_{fcc}}{16}$	$[-2-11](1-11)$
V11	$(1-11) // (011)$	$[011] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[112](11-1)$	$\frac{a_{fcc}}{16}$	$[1-1-2](1-11)$
V12	$(1-11) // (011)$	$[011] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[1-2-1](11-1)$	$\frac{a_{fcc}}{16}$	$[121](1-11)$
V13	$(-111) // (011)$	$[0-11] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[11-2](111)$	$\frac{a_{fcc}}{16}$	$[-11-2](-111)$
V14	$(-111) // (011)$	$[0-11] // [1-11]$	$\frac{a_{fcc}}{18}$	$[1-21](111)$	$\frac{a_{fcc}}{16}$	$[-1-21](-111)$
V15	$(-111) // (011)$	$[-10-1] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[-21-1](11-1)$	$\frac{a_{fcc}}{16}$	$[211](-111)$
V16	$(-111) // (011)$	$[-10-1] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[112](11-1)$	$\frac{a_{fcc}}{16}$	$[-11-2](-111)$
V17	$(-111) // (011)$	$[110] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[121](1-11)$	$\frac{a_{fcc}}{16}$	$[-1-21](-111)$
V18	$(-111) // (011)$	$[110] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[-2-11](1-11)$	$\frac{a_{fcc}}{16}$	$[211](-111)$
V19	$(11-1) // (011)$	$[-110] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[1-21](111)$	$\frac{a_{fcc}}{16}$	$[1-2-1](11-1)$
V20	$(11-1) // (011)$	$[-110] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[-211](111)$	$\frac{a_{fcc}}{16}$	$[-2-11](11-1)$
V21	$(11-1) // (011)$	$[0-1-1] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[1-1-2](1-11)$	$\frac{a_{fcc}}{16}$	$[112](11-1)$
V22	$(11-1) // (011)$	$[0-1-1] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[121](1-11)$	$\frac{a_{fcc}}{16}$	$[1-2-1](11-1)$
V23	$(11-1) // (011)$	$[101] // [-1-11]$	$\frac{a_{fcc}}{18}$	$[211](-111)$	$\frac{a_{fcc}}{16}$	$[-21-1](11-1)$
V24	$(11-1) // (011)$	$[101] // [-11-1]$	$\frac{a_{fcc}}{18}$	$[-11-2](-111)$	$\frac{a_{fcc}}{16}$	$[112](11-1)$

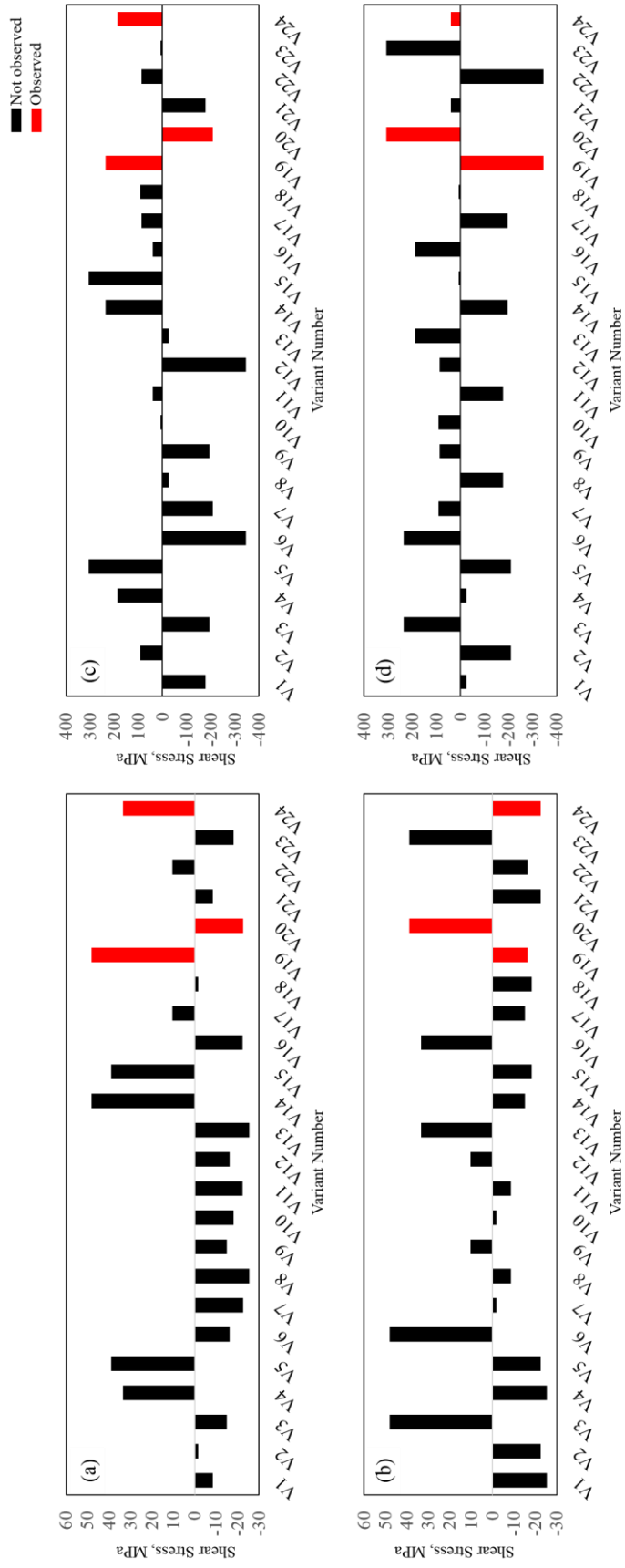


Figure 4.12 The calculated shear stress for the 24 martensite variants in the grain E according to (a, b) the uniaxial external stress and (c, d) the measured local stress field. (a, c) and (b, d) correspond to the shear stress on the first shear and second shear of Burgers-Burgers model, respectively.

4.4.1.3 Analysis according to Shockley partial dislocation

Both Patel and Cohen model and Bogers-Burgers model cannot explain the variant selection in the strain-induced martensitic transformation in SUS304 austenitic stainless steel. One possible reason is that the analysis assumed the direct $\gamma \rightarrow \alpha'$ transformation, despite the transformation sequence was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. Thus, ε martensite is taken into account in this section.

Figure. 4.13 presents the shear stress on twelve Shockley partial dislocations in the grain E, calculated by (a) the uniaxial external stress and (b) the measured local stress field. Because the observed ε variants were parallel to $(1-11)_\gamma$, three Shockley partial dislocations on $(11-1)_\gamma$ correspond to the observed ε variant in the grain E, which are indicated by red color in **Fig. 4.13**. The blue stars indicate the Shockley partial dislocations on which exerted shear stress was maximum. When the uniaxial external tensile stress was used for calculation (**Fig. 4.13(a)**), a Shockley partial dislocation other than the ones corresponding to the observed ε variant shows the largest shear stress. In contrast, one of the Shockley partial dislocations corresponding to the observed ε variant has the largest shear stress when using the measured local stress field, as can be seen in **Fig. 4.13(b)**. Therefore, it can be concluded that the ε martensite variant, whose corresponding Shockley partial dislocation has the largest shear stress, formed preferentially.

The local stress used here should be changed after the $\gamma \rightarrow \varepsilon$ transformation. Because the changed local stress cannot be obtained, it is impossible to analyze variant selection for $\varepsilon \rightarrow \alpha'$ transformation based on the local stress field.

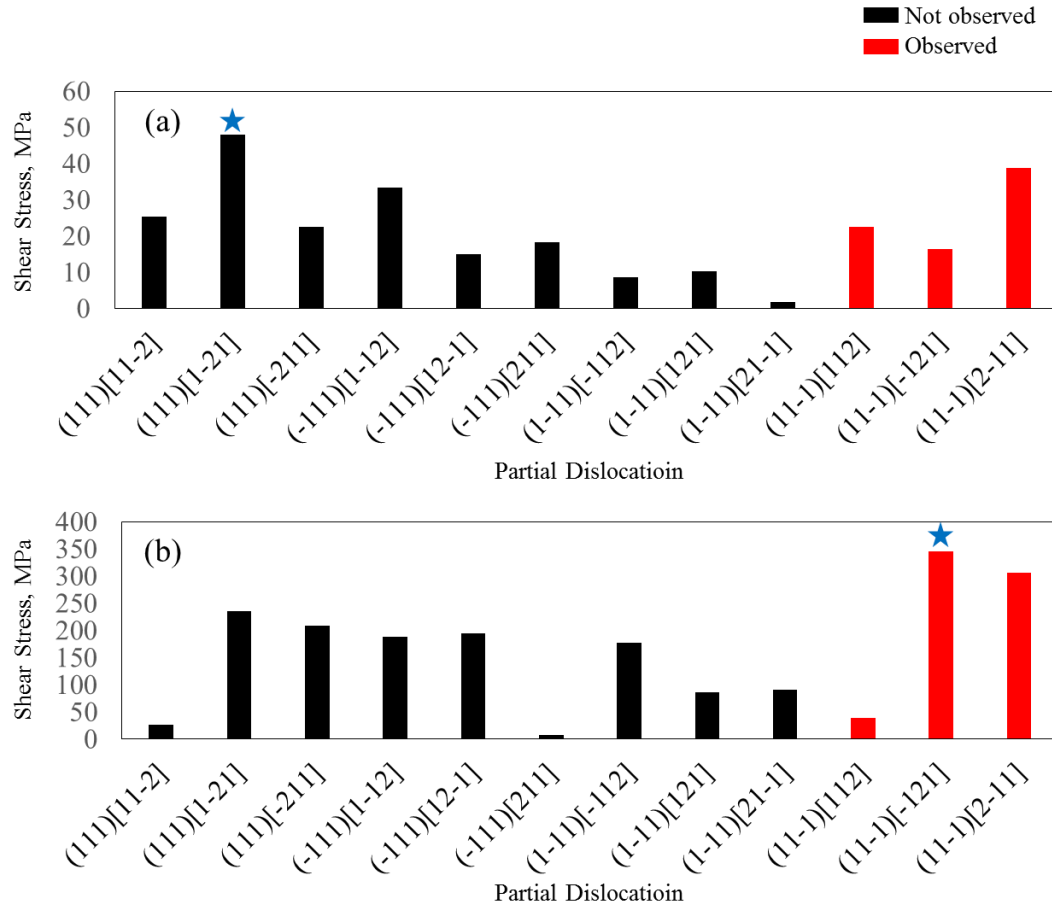


Figure 4.13 The shear stresses on twelve Shockley partial dislocations in the grain E, calculated by (a) the uniaxial external stress and (b) the measured local stress field.

4.4.2 Comparison among thermally induced, stress-induced and strain-induced martensitic transformation

The comparisons among thermally induced, stress-induced and strain-induced martensitic transformation are summarized as follows:

(1) The sequence of the three types of martensitic transformation in the present study was a $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. The S-N relationship between γ and ε phases, the Burgers relationship between ε and α' phases, and the K-S relationship between γ and α' phases were satisfied in all the transformations.

(2) In both the thermally induced martensitic transformation and the stress-induced martensitic transformation, α' martensite formed in a distinct band shape which were parallel to $\{111\}_{\gamma}$. The martensite bands consisted of six α' variants which satisfied same parallel plane relationship in both the Burgers relationship and the K-S relationship. In contrast, strain-induced martensitic transformation exhibited irregular band shape. Although the orientation relationships were satisfied between austenite and martensite, the elongated direction was deviated slightly from the parallel $\{111\}_{\gamma}$ plane trace.

(3) The martensite bands formed in a given austenite grain were parallel to all four $\{111\}_{\gamma}$ planes in the thermally induced martensitic transformation. While, all the martensite bands formed in a given austenite grain were parallel to one of the $\{111\}_{\gamma}$ in the case of stress-induced martensitic transformation. ε martensite plates transformed into all six α' variants in the thermally and stress-induced martensitic transformation. This indicates that variant selection occurred in the stress-induced martensitic transformation at the stage of $\gamma \rightarrow \varepsilon$ transformation. On the other hand, in the case of strain-induced martensitic transformation, the observed martensite bands in a given austenite grain were parallel to two of the $\{111\}_{\gamma}$ planes, and only two or three α' variants were observed. This indicates

that variant selection occurred at both $\gamma \rightarrow \varepsilon$ and $\varepsilon \rightarrow \alpha'$ transformation in the strain-induced martensitic transformation.

4.5 Conclusions

In this chapter, the morphology, crystallographic features and the correlation between variant selection and local stress field in strain-induced martensitic transformation were investigated by EBSD measurements and EXMD technique. The main conclusions are summarized as follows:

(1) Both magnitude and direction of local stress field changed with increasing the tensile strain. In addition, the drastic change in the local stress field was observed after martensitic transformation.

(2) The sequence of strain-induced martensitic transformation was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$. The S-N relationship between γ and ε phases, the Burgers relationship between ε and α' phases, and the K-S relationship between γ and α' phases were satisfied among three phases. The martensite bands exhibited an irregular band shape, and the elongated direction was deviated slightly from the $\{111\}_{\gamma}$ plane trace, which satisfied the orientation relationship with martensite.

(3) The $\gamma \rightarrow \varepsilon$ transformation mechanism of strain-induced martensitic transformation in SUS304 austenitic stainless steel can be explained as follows: the ε martensite variant whose corresponding Shockley partial dislocation has the largest shear stress form preferentially. But the analysis of variant selection in $\varepsilon \rightarrow \alpha'$ transformation is still under discussion.

4.6 References

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Chapter 5 Summary and conclusions

It is well known that the strength and ductility of materials are usually in a trade-off relationship. Deformation-induced martensitic transformation is one of the promising way to improve both strength and ductility simultaneously through transformation-induced plasticity (TRIP). If we can improve TRIP effect by controlling the formation of deformation-induced martensite, better mechanical properties and wider utilization of austenitic stainless steels can be expected. Accordingly, this dissertation aimed to clarify the nature of deformation-induced martensitic transformation in SUS304 austenitic stainless steel. In particular, the microstructure, crystallographic features and correlation between variant selection and local stress field in deformation-induced martensitic transformation were investigated by means of electron backscattering diffraction (EBSD) and energy dispersive X-ray diffraction microscopy using synchrotron radiation X-ray diffraction (EXDM). The main results obtained in this dissertation are summarized below.

In **Chapter 1**, the previous studies of thermally and deformation- induced martensitic transformation were introduced. Then, the purpose and the outline of the present study were described.

In **Chapter 2**, the thermally induced martensitic transformation in SUS304 was studied. The specimens were subzero cooled in liquid nitrogen for 20 min to obtain thermally induced martensite. The thermally induced martensite exhibited a macroscopic straight band morphology, and were parallel to the four $\{111\}_{\gamma}$ planes of austenite phase. The straight bands consisted of little amount of ε martensite and six α' martensite variants. This indicates that the transformation sequence of the thermally induced martensitic

transformation was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$.

The S-N relationship between γ and ε phases, the Burgers relationship between ε and α' phases, and resultantly the K-S relationship between γ and α' phases were confirmed from the orientation analysis. Because at most six α' martensite variants can form from an ε martensite variant according to the Burgers relationship, one martensite bands consisted of all α' martensite variants that could transform from an ε martensite. Moreover, different from the lath martensite formed in low carbon steels, α' variants with $\Sigma 3$ relationship (V1-V2, V3-V4, and V5-V6) formed adjacently. The PTMC calculation suggested that all six α' martensite variants formed in one martensite band to reduce the total transformation strain. However, the combination of variants with $\Sigma 3$ relationship could not be explained from the accommodation of transformation strain.

In **Chapter 3**, the morphology, crystallographic features, and the correlation between variant selection and local stress field in the stress-induced martensitic transformation was studied by EBSD measurements and EXDM technique. The local stress field was successfully measured by the EXDM technique. The measured local stress fields were different depending on the positions even in the same austenite grain, which suggests that the local stress field distribution in polycrystalline materials was considerably complicated.

The stress-induced martensite was obtained by subzero cooling the tensile specimen by liquid nitrogen under external stress. The morphology and transformation sequence of the stress-induced martensite were almost the same as those of thermally induced martensite. In addition, the S-N relationship between γ and ε phases, the Burgers relationship between ε and α' phases, and resultantly the K-S relationship between γ and α' phases were satisfied. On the other hand, variant selection was confirmed in the $\gamma \rightarrow \varepsilon$

transformation because the martensite bands observed in the stress-induced martensite were only parallel to one of $\{111\}_\gamma$ plane. It was confirmed through EXDM technique that the ε martensite variant whose corresponding Shockley partial dislocation had largest shear stress form preferentially. The ε variant transformed into all the six α' variants that could form from one ε martensite variant.

In **Chapter 4**, the morphology, crystallographic features, and the correlation between variant selection and local stress field in the strain-induced martensitic transformation was studied by EBSD measurements and EXMD technique. Both magnitude and direction of local stress field changed with increasing the tensile strain. In addition, the drastic change in local stress field was observed after martensitic transformation.

The strain-induced martensitic transformation was obtained by applying deformation in tension to the plastic regime at room temperature. The transformation sequence and orientation relationship among austenite, ε martensite and α' martensite were almost the same as thermally and stress- induced martensite. However, the martensite bands only consisted of ε martensite and two or three α' martensite. Furthermore, the martensite bands exhibited an irregular band shape, and the elongated direction was deviated slightly from the $\{111\}_\gamma$ plane traces, which satisfied the orientation relationship with martensite.

Variant selection in the strain-induced martensite was confirmed not only in the $\gamma \rightarrow \varepsilon$ transformation, but also in the $\varepsilon \rightarrow \alpha'$ transformation. The ε martensite variant whose corresponding Shockley partial dislocation has the largest shear stress form preferentially. But the analysis of variant selection in $\varepsilon \rightarrow \alpha'$ transformation is still under discussion.

In **Chapter 5**, all the obtained conclusions in each chapter have been summarized.

This dissertation investigated the nature of deformation-induced martensitic transformation of SUS304 austenitic stainless steel by EBSD measurements and EXDM technique. I believe that this dissertation will be helpful to establish the microstructural control methodology for improving mechanical properties of metallic materials through deformation-induced martensitic transformation.

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