



Equiaxed α microstructure evolution in wrought Ti-10Al-1Zr-1Mo-1Nb alloy during annealing

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ABSTRACT

Evolution of α microstructure on the thermomechanical treated Ti-10Al-1Zr-1Mo-1Nb alloy during annealing was studied. The solution-treated materials were groove-rolled or uniaxially compressed in the $\alpha+\beta$ region and annealed at 1173 K. The flow softening behavior and crystal rotation in α platelets revealed an evolution of deformation texture. The volume fraction of equiaxed α grains was increased during annealing. Especially in the material compressed at a strain rate of 1 s^{-1} , the equiaxed α grains developed within a shorter annealing duration than 1.8 ks. The deformation with higher strain rates promoted the division and fragmentation of α platelets during annealing. Transmission electron microscopy and X-ray diffraction analyses were employed to characterize dislocation components and structure, where the installed screw dislocations provided a fine substructure and high energy α/α boundaries in α platelets. The triple junction consisting of α/β boundaries and α/α boundaries may provide a site for thermal grooving, which induces the division and fragmentation of α platelets. Therefore, deformation at a higher strain rate is necessary in $\alpha+\beta$ processing to develop a fine equiaxed α microstructure for the Ti-10Al-1Zr-1Mo-1Nb alloy during annealing.

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1. Introduction

Titanium alloys may be utilized in aerospace industry owing to their high strength-to-weight ratio and excellent corrosion resistance [1]. In particular, $\alpha+\beta$ titanium alloys facilitate the design of balanced mechanical properties since their characteristics strongly depend on the volume ratio between the α (hcp) and β (bcc) phases and their morphology. Alloying elements such as Al, Sn, Zr, Mo, Nb, V, and Si are chosen to determine the α/β volume ratio and to enhance heat resistance by solid solutions in near α -type titanium alloys [2–4]. Following oxidation at high temperatures, the Sn content decreases and of Nb content increases in Ti-15Al-5(Sn-Nb) (mol%), enhancing resistance [5]. The simultaneous addition of Nb and Zr promotes strong adhesion to the oxide layers [6]. A new Sn-

free Ti-Al-Nb-Zr alloy has been proposed to increase the service temperature of near α -type titanium alloy, and the creep properties related to its microstructures have been investigated [7–9]. Influence of temperature and compression strain rate in the β -single-phase region for the microstructural evolution of the Ti-10Al-1Zr-1Mo-1Nb alloy was evaluated. The flow behavior of the alloy during high-temperature compressive deformation was evaluated by the Arrhenius-type constitutive model and discussed on the basis of microstructural evolution [10].

The mechanical properties of near α -type titanium alloys are typically controlled by thermomechanical treatment [11,12]. Process parameters such as temperature, applied strain, and strain rate play a significantly important role in the development of their microstructure [13–15]. A lamellar and/or acicular α microstructure was formed following various thermomechanical processes in the β -single-region. It forms during cooling and exhibits moderate strength and fatigue crack propagation resistance but low ductility [16]. In contrast, the equiaxed α microstructure exhibits a superior balance of strength and ductility and high fatigue crack initiation

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resistance [17]. Therefore, a kind of bi-modal microstructure consisting of equiaxed α grains and α platelets is desirable for various applications.

The evolution of the equiaxed α microstructure in $\alpha+\beta$ type titanium alloys such as Ti-6Al-4V (in mass%) alloy has been investigated [18–21]. Two mechanisms have been proposed for developing equiaxed α microstructure during annealing, i.e. (i) division and fragmentation of deformed α platelets, and (ii) migration termination of α platelets or coarsening of α grains. The former focuses on the triple junctions of α/β interphase boundaries and α/α interface in α platelets. The α/α interfaces may result from recovered substructure, dynamic recrystallization, or localized shear bands. The latter is a type of diffusional coarsening process and is quantitatively evaluated by the Gibbs-Thompson equation. Few studies, however, have been done on the evolution of equiaxed α microstructure in Ti-10Al-1Zr-1Mo-1Nb alloy and its dominant mechanism.

In the present study, the influence of process conditions on the evolution of equiaxed α microstructure in Ti-10Al-1Zr-1Mo-1Nb alloy during annealing was investigated. The alloy was groove-rolled or uniaxially compressed in the $\alpha+\beta$ region at different temperatures and strain rates, followed by annealing. Microstructural analyses were carried out to characterize the morphology of α phase and the substructure in α platelets.

2. Experimental procedure

2.1. Material and thermomechanical treatment

Ti-10Al-1Zr-1Mo-1Nb, in mol% (Ti-5.7Al-1.9Zr-2.0Mo-2.0Nb, in mass%) alloy was examined. The β -transus temperature of the alloy was approximately 1253 K. The 1.1 kg ingot alloy was melted by a cold crucible levitation method. The ingot was then cut into three pieces and solution-treated at 1323 K for 10.8 ks, followed by air cooling.

A cylindrical sample with a diameter of 75 mm was cut from the solution-treated material. Fig. 1a illustrates the thermomechanical treatment of the sample in the $\alpha+\beta$ region. The sample was heated in a furnace at 1223 K over three cycles, each characterized by heating at 1223 K for 300 s and forging, followed by air cooling. The sample resulted in a 40-mm-square rectangular bar with a 63% reduction in the cross-section area, over a total processing time of approximately 1.2 ks. Furthermore, the rectangular bar was formed into a 14.3-mm-square bar following multi-steps groove-rolling through three cycles of heating at 1223 K for 300 s, followed by air cooling. An 87% reduction in the cross-section area was realized (the equivalent strain in the total reduction was approximately 2.4), and the processing time was approximately 1.2 ks. The strain rate during groove-rolling was estimated to be about 0.002 s^{-1} .

High-temperature uniaxial compressive tests were conducted in the $\alpha+\beta$ region at 1173 K with different constant true strain rates using a thermomechanical simulator (THERMECMASTOR-MD, Fuji Electronic Industrial Co., Ltd.). Fig. 1b illustrates the uniaxial compression in the $\alpha+\beta$ region. Cylindrical samples with a diameter of 8 mm and a height of 12 mm were cut from the solution-treated material and mechanically polished their surface. The test temperature was measured using a thermocouple that was welded onto the surface of the sample and was controlled by induction heating in vacuum and He gas cooling. The samples were heated to the test temperature at 10 K/s; the temperature was maintained at 1173 K for 300 s to homogenize its distribution, and isothermally compressed along the cylindrical axis for a reduction of up to 75% at the constant true strain rates of 0.01 s^{-1} , 0.1 s^{-1} or 1 s^{-1} , followed by He gas cooling to 500 K at 50 K/s. The temperature gradient in a sample and

the friction between a sample and the anvils should be eliminated; therefore, mica sheets were inserted between each sample and the anvils to facilitate heat insulation and lubrication. True strain and true stress values were calculated from load-stroke measurements by assuming uniform, constant volume-deformation.

Both the groove-rolled material and the uniaxially compressed ones were annealed in a muffle-type furnace at 1173 K for 0.3 ks, 1.8 ks and 10.8 ks, followed by water quenching.

2.2. Microstructural characterization

Equiaxed α grain formation during annealing in the $\alpha+\beta$ region was evaluated by field-emission scanning electron microscopy (SEM). SEM samples were taken from the central position in the section of groove-rolled material parallel to the rolling direction (RD) and that of uniaxially compressed material parallel to the compression axis as illustrated in Fig. 2a and c. Those surfaces were prepared by grinding with SiC papers and by vibratory polishing with Al_2O_3 and SiO_2 suspensions. To quantitatively evaluate the morphology of the $\alpha+\beta$ microstructure, the boundaries between α platelets, equiaxed α and β grains were manually demarcated. The micrographs were examined using automated image analysis (ImageJ software package, U.S. National Institutes of Health [22]) to determine the volume fraction, grain size and number density of equiaxed α grains. Here, an equiaxed α grain was defined as an approximately elliptical grain with an aspect ratio of less than 2:1. On average, 500 to 600 α grains were taken for each analysis to provide statistically reliable data.

Microstructures of α platelets, especially substructures developed were characterized by transmission electron microscopy (TEM) and X-ray diffraction (XRD). A JEOL JEM-2100F TEM was employed at 200 keV to identify substructure in α platelets. A Rigaku Smart Lab XRD with a Cu-target sealed tube and a 1D semiconductor detector was employed at 40 keV and 45 mA to determine the average characteristics of the substructure, i.e. dislocation density, crystallite size and so on.

TEM disks of about 600 μm thick were sectioned from the center of the uniaxially compressed materials parallel to compression axis (Fig. 2c). They were reduced to about 100 μm in thickness by abrasion with no. 600 SiC paper. XRD samples were taken from the cross sections of groove-rolled material perpendicular to RD and that of uniaxially compressed materials perpendicular to the compression axis as illustrated in Fig. 2b and d. Those surfaces were grinded with SiC paper. Both TEM foils and XRD samples were prepared by a conventional electrical jet polishing technique using a stirred solution of 59% CH_4O -35% $\text{C}_4\text{H}_{10}\text{O}$ -6% HClO_4 at 243 K.

X-ray line profile analyses were performed based on the modified Williamson-Hall and modified Warren-Averbach procedure [23,24]. The line profiles were collected for diffraction angles ranging from 30 to 80° with a nominal step interval of 0.01°. A fixed-angle divergence slit of 0.25° and a couple of soller slits with an open-angle of 5.0° were used. Ni foil of 0.05 mm in thickness was used as a $\text{K}\beta$ filter. Instrument broadening of the measured X-ray line profiles was corrected by scale-transformation and deconvolution-convolution treatment [25–27] optimized for the above optical system.

3. Results

3.1. Microstructure

The microstructure of the test material after solution treatment at 1323 K for 10.8 ks in the β phase region followed by air cooling is represented in Fig. 3. The backscatter electron image shown in Fig. 3a represents a basket-weave microstructure that consists of

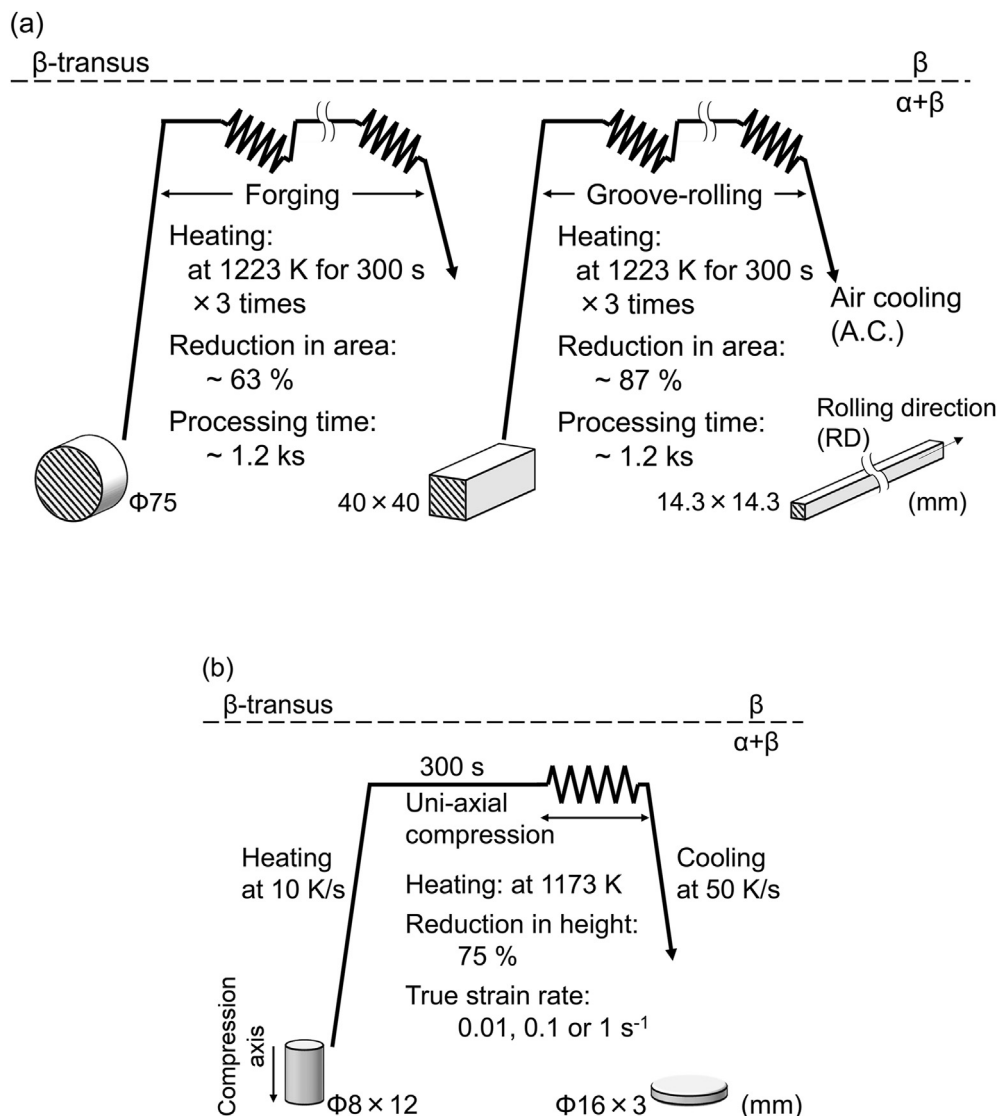


Fig. 1. Schematic diagram of thermomechanical treatment in the $\alpha+\beta$ region: (a) forging and groove-rolling steps, and (b) uniaxial compression.

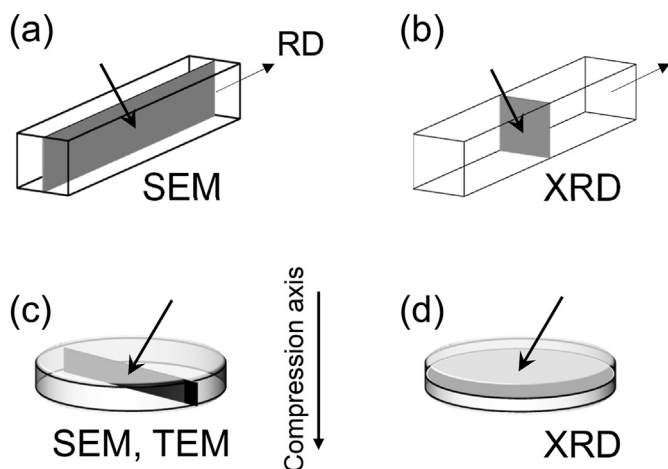


Fig. 2. Analyses sections (dark) for SEM (a, c), XRD (b, d) and TEM (c) in the groove-rolled material (a, b) and the uniaxially compressed (c, d) materials. Arrows show the analyses area from the central position in each section.

transformed acicular α (α_{AC}) and retained β in a prior- β grain, in which the dark and bright areas correspond to the α and β phases, respectively. Individual α_{AC} platelets were fitted with the prior- β matrix to a variant that satisfies the Burgers orientation relationship [28]. These α_{AC} platelets of approximately 1 μm in thickness were predominantly developed in a prior- β grain. Lower volumes of the grain boundary α (α_{GB}) and side-plate α (α_{SP}) were detected (Fig. 3b). The α_{GB} is formed due to heterogeneous nucleation at the prior- β grain boundaries, and the α_{SP} grows from the α_{GB} into β grains.

Fig. 4 represents the microstructure of the test materials annealed at 1223 K and 1173 K in the $\alpha+\beta$ two-phase region for 300 s followed by water quenching, i.e. the microstructures were heated at the temperatures of groove-rolling and uniaxial compression, respectively. These microstructures consisted of α_{AC} and β phase with similar as solution-treated in β phase region and air-cooled materials as shown in Fig. 4. The volume fraction of the β phase was increased to 61% at 1223 K and 38% at 1173 K due to heating in the $\alpha+\beta$ two-phase region, while the thickness of their α_{AC} platelets was kept almost the same as that of the solution-treated material at approximately 1 μm .

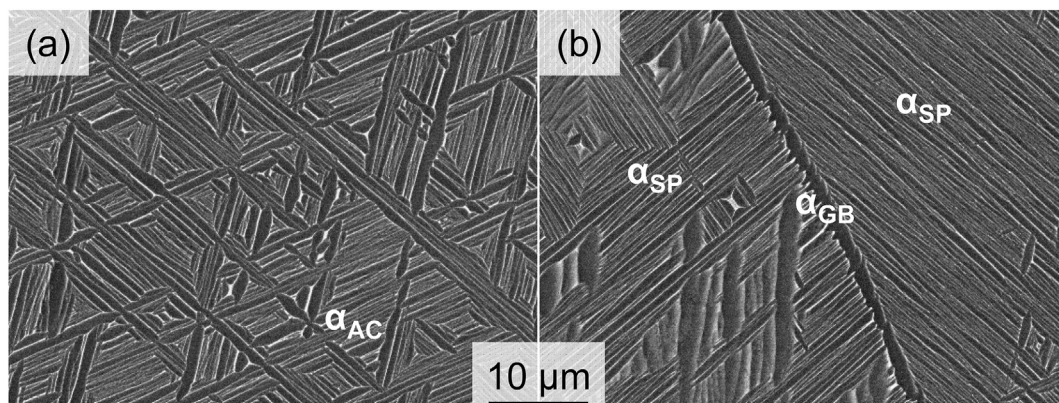


Fig. 3. Backscatter electron images in a prior-β grain (a) and in the vicinity of a prior-β grain boundary (b) of the material that was solution-treated at 1323 K for 10.8 ks followed by air cooling. Acicular α (α_{AC}) platelets are dominant in the prior-β grain.

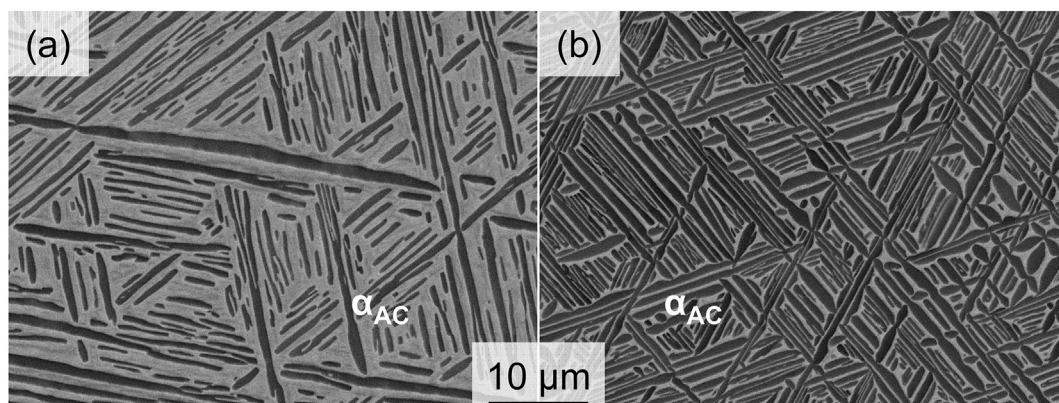


Fig. 4. Backscatter electron images in a prior-β grain of the material that was heat-treated at 1223 K (a) and 1173 K (b) for 300 s followed by water quenching. Those volume fractions of β phase (white) are 61% (a) and 38% (b), respectively.

3.2. Flow curves

In the uniaxial compressive tests, the flow stresses of the material increased when the strain rate was increased from 0.01 s^{-1} to 1 s^{-1} . Fig. 5 represents the true stress – true strain (flow) curves of the materials compressed at 1173 K. All flow curves exhibit a peak flow stress at relatively low strains (true strain of approximately 0.03) followed by extensive flow softening. The two possible explanations of flow softening during hot deformation are adiabatic temperature increase associated with plastic deformation, and deformation texture evolution. Although the surface temperature of the samples compressed at strain rates of 0.01 s^{-1} and 0.1 s^{-1} momentarily decreased by approximately 10 K due to contact with the anvils at the start of the deformation, the surface temperature can be immediately controlled to the test temperature. Therefore, the deformation texture evolution may be responsible for the flow softening behavior. In contrast, the surface temperature increased by approximately 20 K with a strain rate of 1 s^{-1} , due to deformation-induced temperature rise at the start of the compression, and the temperature was not sufficiently controlled because the strain rate was so high. For α+β titanium alloys, e.g. Ti-6Al-4V, with a lamellar and/or acicular α microstructure, flow softening in uniaxial compression and tension can be ascribed to the rotation of α platelets toward softer orientations with high Schmid factors and/or slip transfer across the α/β interfaces [19,21,29,30].

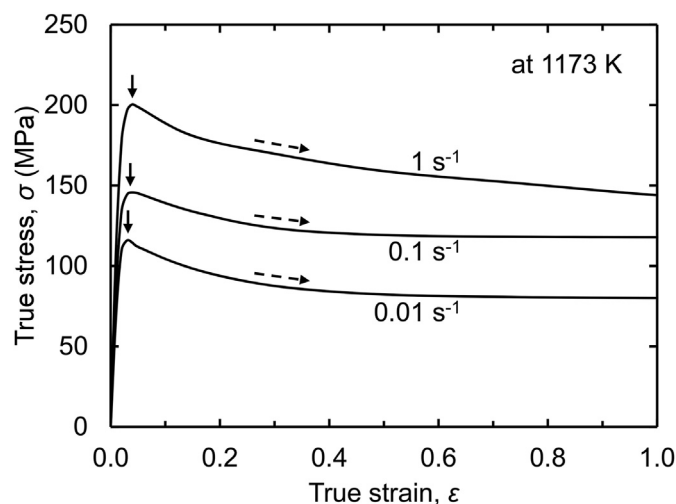


Fig. 5. True stress–true strain (flow) curves of the materials compressed at 1173 K at strain rates of 0.01, 0.1 and 1 s^{-1} . Solid and dashed arrows represent the peak flow stress and the flow softening, respectively.

3.3. Thermomechanically treated microstructure

The microstructure of groove-rolled material consisted of primary α (α_p) and secondary α (α_s) platelets as shown in Fig. 6a.

Compared with the annealed structure shown in Fig. 4a, it can be determined that the α_p is developed through the division and/or fragmentation of α_{AC} platelets during groove-rolling and that the α_s is transformed from the β phase during air cooling. The α_p was coarsened with a thickness of approximately 2 μm , due to the multi-steps groove-rolling through three heating-deformation cycles. On the other hand, the thickness of the α_s was approximately 500 nm and finer than that of the retained β .

The orientation spread is defined as the angle of misorientation between all the points in a grain, while the grain orientation spread (GOS) is the average value of orientation spread in a grain. The difference in GOS value between the α_p and α_s provides an effective way of partitioning the deformed and transformed platelets. In fact, the average lattice rotation in the individual α_p platelets was larger than that in the individual α_s platelets. The maximum rotation angle in α_p platelets was approximately 10° as shown in Fig. 6b, whereas the lattice rotation in the α_s platelets was less than several degrees. Since the deformation in the $\alpha+\beta$ processing is also responsible for giving heterogeneous nucleation sites for the transformation from β to α , the lattice rotation in fine α_s platelets is suggested to derive from the lattice rotation in the deformed β , in which the transformation to α_s follows nucleation and growth according to the Burgers orientation relationship [31].

For the material that was uniaxially compressed at 1173 K, few α_s platelets transformed from deformed β platelets were detected as shown in Fig. 7, and the distorted and/or kinked α_p microstructure was observed due to lower volume fraction of the β phase during deformation as shown in Fig. 4b. Regardless of the strain rate, the fine equiaxed α (α_{eq}) grains distributed as a necklace shape were aligned on the shear bands developed by the heavy deformation. Since the uniaxial compression was done with no reheating and under high strain rate deformation, no coarsening of α_p platelets was detected. The thickness of the deformed α_p platelets was maintained at approximately 1 μm .

The traces of the α_p platelets in the microstructures deformed by groove-rolling and uniaxial compression were rotated and aligned to the direction of metal flow to some extent, respectively. In alignment with softening in the flow curves of the uniaxial compression test, the rotation of α_p platelets may correspond to deformation texture evolution rather than to deformable orientation [19,32].

3.4. Microstructure evolution during annealing

Fig. 8 shows microstructures of the materials that were thermomechanically treated and annealed at 1173 K for 1.8 ks. For the

groove-rolled material, the equiaxed α (α_{eq}) grains were developed mainly in the α_p platelets during annealing, in which large lattice rotations were introduced (Fig. 8a). The average diameter of the α_{eq} grains was approximately 2.5 μm as shown in Fig. 8b. On the contrary, no morphological change in the α_s platelets was observed (Fig. 8a and b), and the volume fraction and aspect ratio of α_s platelets decreased slightly. For the uniaxially compressed materials with strain rates of 0.01 s^{-1} and 0.1 s^{-1} , the α_{eq} grains were partially introduced around shear bands where the distorted and/or kinked α_p platelets had been developed in advance (Fig. 8c and e). On the other hand, a fully equiaxed α (α_{eq}) microstructure was developed in the case of the material compressed with a strain rate of 1 s^{-1} as shown in Fig. 8g. The average grain diameter of the α_{eq} grains was approximately 1.5 μm regardless of the strain rates (Fig. 8d, f and 8h). The longer annealing time results in an equiaxed microstructure but coarsens α_{eq} grains, making it difficult to develop a fine equiaxed microstructure.

Fig. 9 represents the microstructural evolution during annealing at 1173 K. Particularly, the volume fraction, average diameter, and number density of α_{eq} grains are evaluated as a function of annealing time. Here, the volume fraction and number density of α_{eq} are defined as the volume ratio of the α_{eq} grains to the entire α phase, and the number of α_{eq} grains per unit area (μm^{-2}), respectively. The volume fraction of α_{eq} grains increased with annealing time as shown in Fig. 9a. In particular, the uniaxially compressed material with the strain rate of 1 s^{-1} exhibited rapid globularization behavior, resulting in the highest fraction of α_{eq} with a shorter annealing time of 1.8 ks? On the other hand, globularization behaviors of other uniaxial compressed materials were similar and proceeded slowly, taking an annealing time of 10.8 ks. The factors increasing the volume fraction of α_{eq} grains include (i) formation of new α_{eq} grains by division and/or fragmentation of α platelets and (ii) coarsening of α_{eq} grains [20].

To understand the rapid globularization behavior in the uniaxial compressed material with the strain rate of 1 s^{-1} , the coarsening of α_{eq} grains during annealing was evaluated. An exponential increase in the diameter of α_{eq} grains was exhibited with the increase in annealing time as shown in Fig. 9b. The number density of α_{eq} grains also converged to about 0.1, according to a power law function as shown in Fig. 9c. The uniaxially compressed materials revealed almost the same coarsening behavior regardless of the strain rates (Fig. 9b). Although the diameter of α_{eq} grains in the groove-rolled material was more than twice as large as that in the uniaxially compressed ones at the initial stage of annealing, it was almost similar to the diameters of other materials. The number density of the α_{eq} grains in the groove-rolled material and the

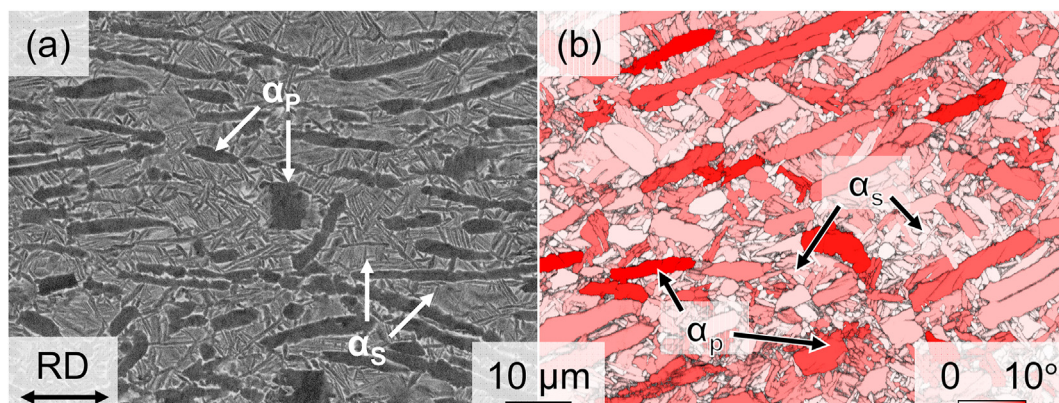


Fig. 6. Backscatter electron image (a) and grain orientation spread map (b) of the groove-rolled material. Primary α (α_p) deformed during groove-rolling and secondary α (α_s) platelets transformed from deformed prior- β grain were observed.

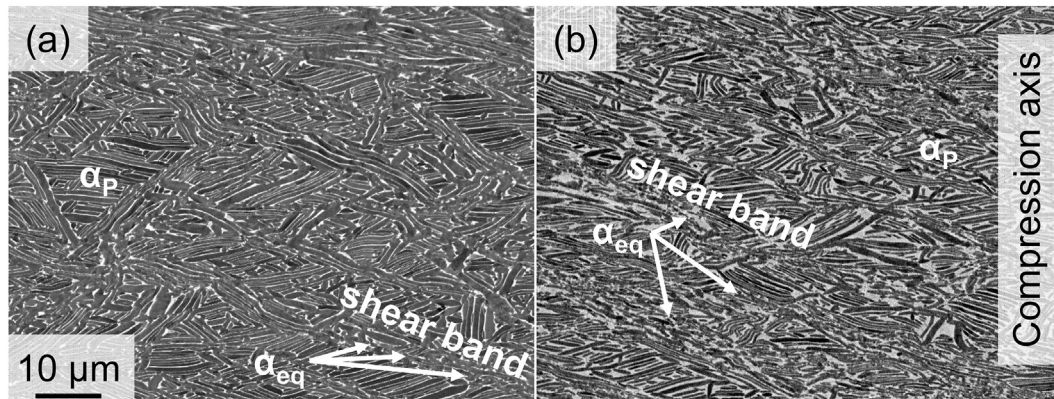


Fig. 7. Backscatter electron images of the uniaxially compressed materials at 1173 K at strain rates of 0.01 s⁻¹ (a) and 1 s⁻¹ (b). Distorted and/or kinked primary α (α_p) platelets and fine equiaxed α (α_{eq}) grains distributed in a necklace shape were observed.

uniaxially compressed materials with the strain rate of 0.1 s⁻¹ and 0.01 s⁻¹ decreased with annealing time, respectively (Fig. 9c). On the other hand, the number density of the α_{eq} grains increased during annealing up to 1.8 ks in the material compressed with the strain rate of 1 s⁻¹, while their volume fraction increased as shown in Fig. 9a.

4. Discussion

4.1. Equiaxed α microstructure evolution

Growth and coarsening behavior of α_{eq} grains in the $\alpha+\beta$ titanium alloys during annealing can be estimated by the Lifshitz–Slyosov–Wagner (LSW) theory model [33–35].

$$r^n - r_0^n = K_{LSW}(t - t_0) \quad (1)$$

where t_0 is the initial time, r and r_0 are average radii of the α_{eq} grains at times of t and t_0 , $t - t_0$ is the annealing time, n is the coarsening exponent (integer), K_{LSW} is the rate constant of the coarsening behavior depending on the composition of alloying elements and temperature. The coarsening exponent n and rate constant K_{LSW} in Equation (1) are obtained by optimizing the measurements in term of $r^n - r_0^n$ vs $t - t_0$ for the groove-rolled or uniaxially compressed materials as shown in Table 1. The dotted (groove-rolled) and dashed (uniaxially compressed) curves in Fig. 9b are the calculated diameters as functions of annealing time based on equation (1) and with the constants shown in Table 1. According to Fig. 9b and Table 1, the constants K_{LSW} and n representing the coarsening rate of α_{eq} grains, are comparable between the groove-rolled and the uniaxially compressed materials; whereas the diameters of α_{eq} grains before annealing were different, i.e. $r_0 \sim 1.1 \mu\text{m}$ in groove-rolled and $r_0 \sim 0.3 \mu\text{m}$ in uniaxially compressed materials. Since the coarsening rate of $K_{LSW} \sim 0.15 \mu\text{m}^3/\text{ks}$ and the coarsening exponent of $n \sim 3$ were identically determined for all the materials, the coarsening behavior of α_{eq} grains in them is the same regardless of the thermomechanical processing conditions such as strain rate. Namely, the coarsening of α_{eq} grains may not be responsible for their rapid globularization behavior in the material compressed under a strain rate of 1 s⁻¹.

The number density of the α_{eq} grains, (i) increases with the division or fragmentation of the α platelets, and (ii) decreases with the coarsening of α_{eq} grains. The number density of the α_{eq} grains increased by the annealing up to 1.8 ks only in the material compressed with a strain rate of 1 s⁻¹ as shown in Fig. 9c. This increase in the number density implies that the division or fragmentation of

the α platelets was dominant in the material compressed under a strain rate of 1 s⁻¹ during annealing up to 1.8 ks. The constant number density of α_{eq} grains suggests that the α_{eq} grains were preferentially coarsened, and that the fine α_s platelets in the vicinity of α_{eq} grains were absorbed by the α_{eq} grains. From the above, the division and fragmentation of the α platelets govern the equiaxed behavior during annealing if strain is introduced to the material at high strain rates, so that it is assumed to be possible to obtain a fine α_{eq} microstructure through relatively short annealing. Therefore, deformation with higher strain rate in $\alpha+\beta$ processing due to the installation of plastic strain in α_p platelets is necessary for developing a fine equiaxed α microstructure without coarsening the α_{eq} grains. The substructure that promotes division and fragmentation in the α platelets will be discussed in section 4.2.

4.2. Substructure in α platelets

“Thermal grooving” at the triple point of the α/β interphase boundary and the α/α interface is known as a mechanism for the division and fragmentation of α platelets during annealing in $\alpha+\beta$ titanium alloys [18,20]. In this section, the substructure in the α platelets that affects the formation of thermal grooving is investigated with TEM observations and XRD line profile analyses. Then, the influence of thermomechanical treatments such as groove-rolling and uniaxial compression on the substructure formation, and division and fragmentation of α platelets during annealing is discussed.

4.2.1. TEM observation

Fig. 10 represents the TEM bright-field images of the α platelets in the material that was uniaxially compressed with strain rates of 0.01 s⁻¹ and 1 s⁻¹, respectively. In the material compressed with a strain rate of 0.01 s⁻¹, the dislocation density in the α platelets was low, and interfaces such as dislocation walls and sub-grain boundaries were observed (Fig. 10a). These interfaces appeared to be formed by the dynamic recovery of dislocations during compression. In contrast, in the material that was uniaxially compressed with the higher strain rates of 1 s⁻¹, higher-density dislocations and the interfaces formed by their aggregation were partly observed in the α platelets as shown in Fig. 10b. These dislocations introduced to the α platelets are expected to recover and rearrange statically during subsequent annealing and to form the α/α interface. Thermal grooving occurs by the protrusion of the α/β interphase boundary into the α platelets, due to the balance of the interfacial energies between the α/β interphase boundary and the α/α interface. The division and fragmentation of the α platelets by

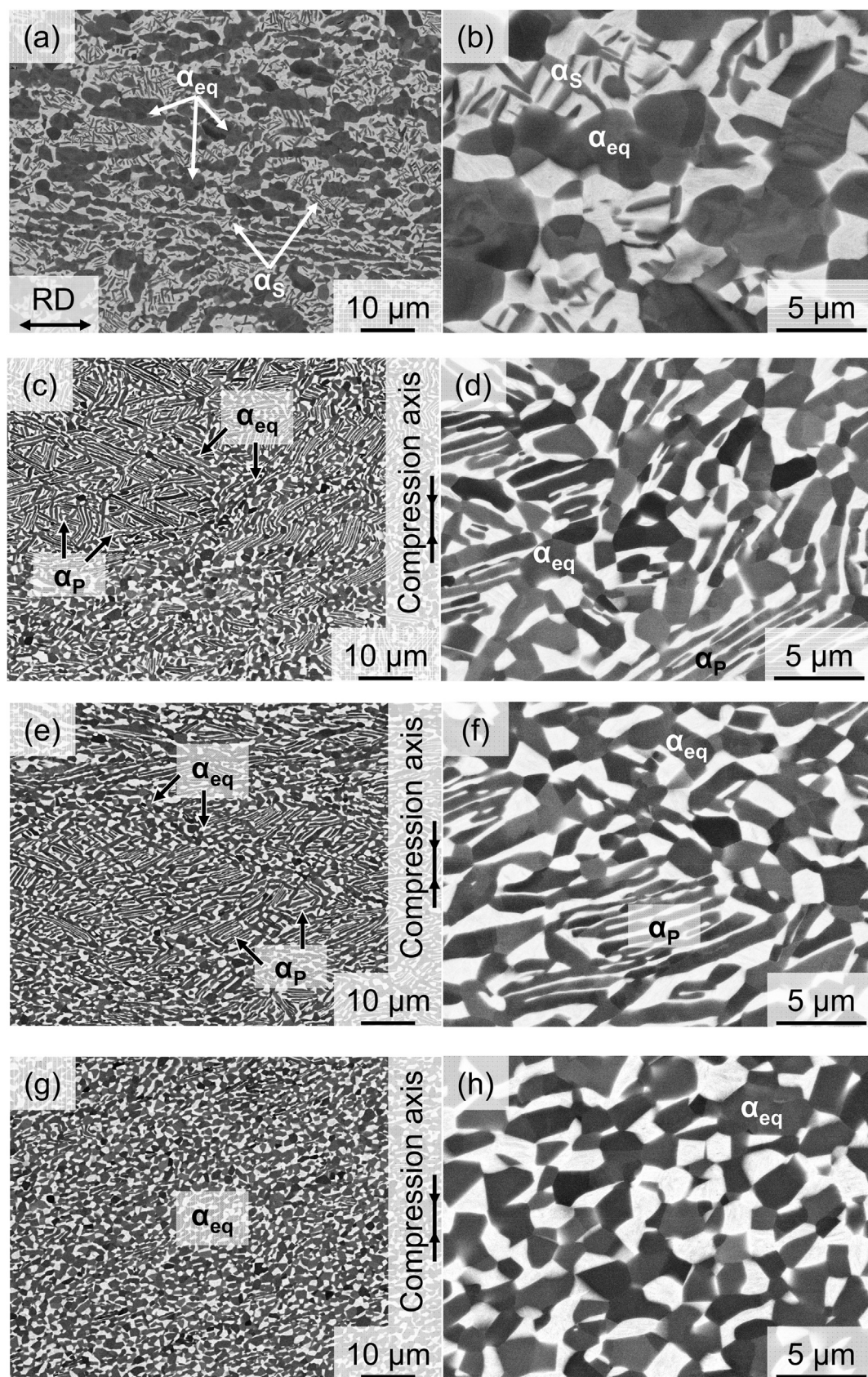


Fig. 8. Backscatter electron images of the samples that were thermomechanically treated and annealed at 1173 K for 1.8 ks followed by water quenching. The samples were groove-rolled (a),(b), and uniaxial compressed with the strain rates of 0.01 s⁻¹ (c),(d), 0.1 s⁻¹ (e),(f), and 1 s⁻¹ (g),(h). Photographs (b), (d), (f) and (h) are magnified images of photographs (a), (c), (e) and (g), respectively.

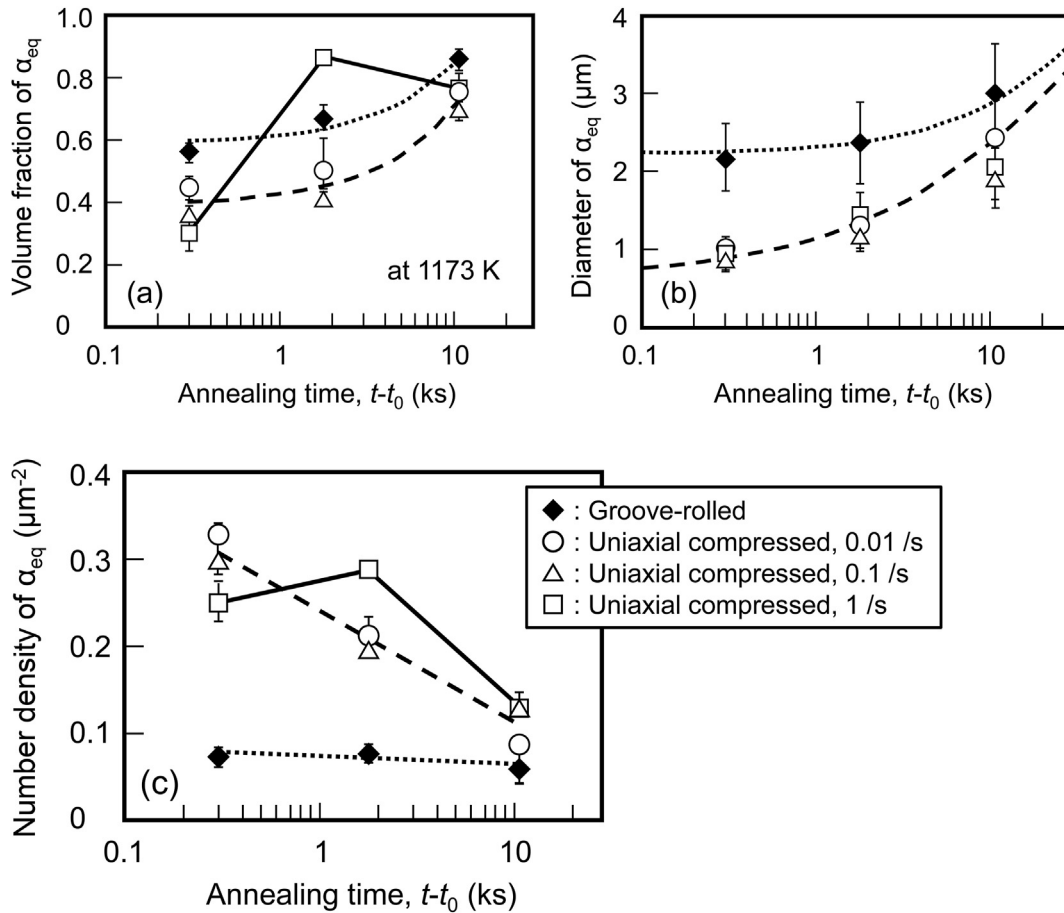


Fig. 9. vol fraction (a), average diameter (b) and number density (c) of equiaxed α_{eq} grains during annealing at 1173 K. The grain coarsening curves given by Equation (1) in (b) represent the optimized radii with the annealing time under the LSW theory model.

Table 1

Calculated values of the initial radii of the equiaxed α grains r_0 , coarsening exponent n and rate constant K_{LSW} under the LSW theory model following optimization by Equation (1) and data plots in Fig. 9(b).

Processing	r_0 (μm)	n	K_{LSW} ($\mu\text{m}^3/\text{ks}$)
Groove-rolling	1.1	3	0.15
Uniaxial compression	0.3		

thermal grooving occurs easily with a large groove angle. The groove angle δ satisfies the equilibrium equation for the surface energies of the α/β interphase boundary ($\gamma_{\alpha\beta}$) and the α/α interface ($\gamma_{\alpha\alpha}$) [18].

$$\gamma_{\alpha\alpha} = 2\gamma_{\alpha\beta} \sin \delta \quad (2)$$

Equation (2) indicates that an increase in the α/α interfacial energy $\gamma_{\alpha\alpha}$ is required to increase in groove angle δ . The α/α interfacial

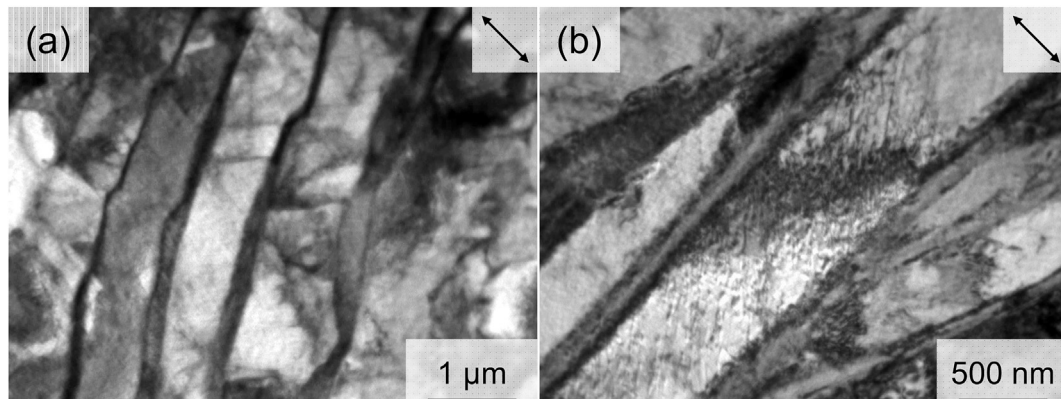


Fig. 10. Bright-field images represent dislocation structures in the α platelets developed by uniaxial compression under strain rates of 0.01 s^{-1} (a) and 1 s^{-1} (b). Each arrow in photographs denotes the compression axis.

energy, $\gamma_{\alpha/\alpha}$, increases in the interfaces consisting of fine substructures [36]. Here, the α/α interface involves not only low-angle boundary but also high-angle one. Therefore, by introducing the α/α interface with higher interfacial energy into the α platelets during rolling and compression, it is expected that the division and fragmentation of the α platelets are promoted and the equiaxed materials are obtained within a short annealing time.

4.2.2. XRD line profile analysis

The modified Williamson-Hall and modified Warren-Averbach procedures [23,24] were employed to quantify and characterize the substructure in α platelets, and the influence of the condition of thermomechanical treatment on the substructure formation were discussed. The detailed procedure of the XRD line profile analysis is summarized in the Appendix. Fig. 11 represents the dislocation density and crystallite size in the α phase of the groove-rolled or uniaxially compressed materials. The edge component of dislocations in the α phase for each material was 80% or more, and the

density of edge dislocation showed no significant difference or tendency. In contrast, focusing on the screw component of the dislocations, the density of screw dislocations was higher in the material compressed with higher strain rates as shown in Fig. 11a. Compared to edge dislocations, screw dislocations move faster and were easy to recover and annihilate by cross-slips during high-temperature deformation. The trends in the screw dislocation density shown in Fig. 11a suggest that the recovery and annihilation of the screw dislocations were suppressed with the increase of the strain rate. On the other hand, the crystallite size of the α phase monotonically decreased with the increase of the strain rate during either groove-rolling or uniaxial compression as shown in Fig. 11b. The crystallite is interpreted as the region without lattice defects such as dislocations. Therefore, the refinement of the crystallite corresponds to the densification of the substructure introduced to the α phase. Substructures consisting of dislocations rearrange and form α/α interfaces in α platelets during subsequent annealing. In this process, the formation of higher energy α/α interfaces

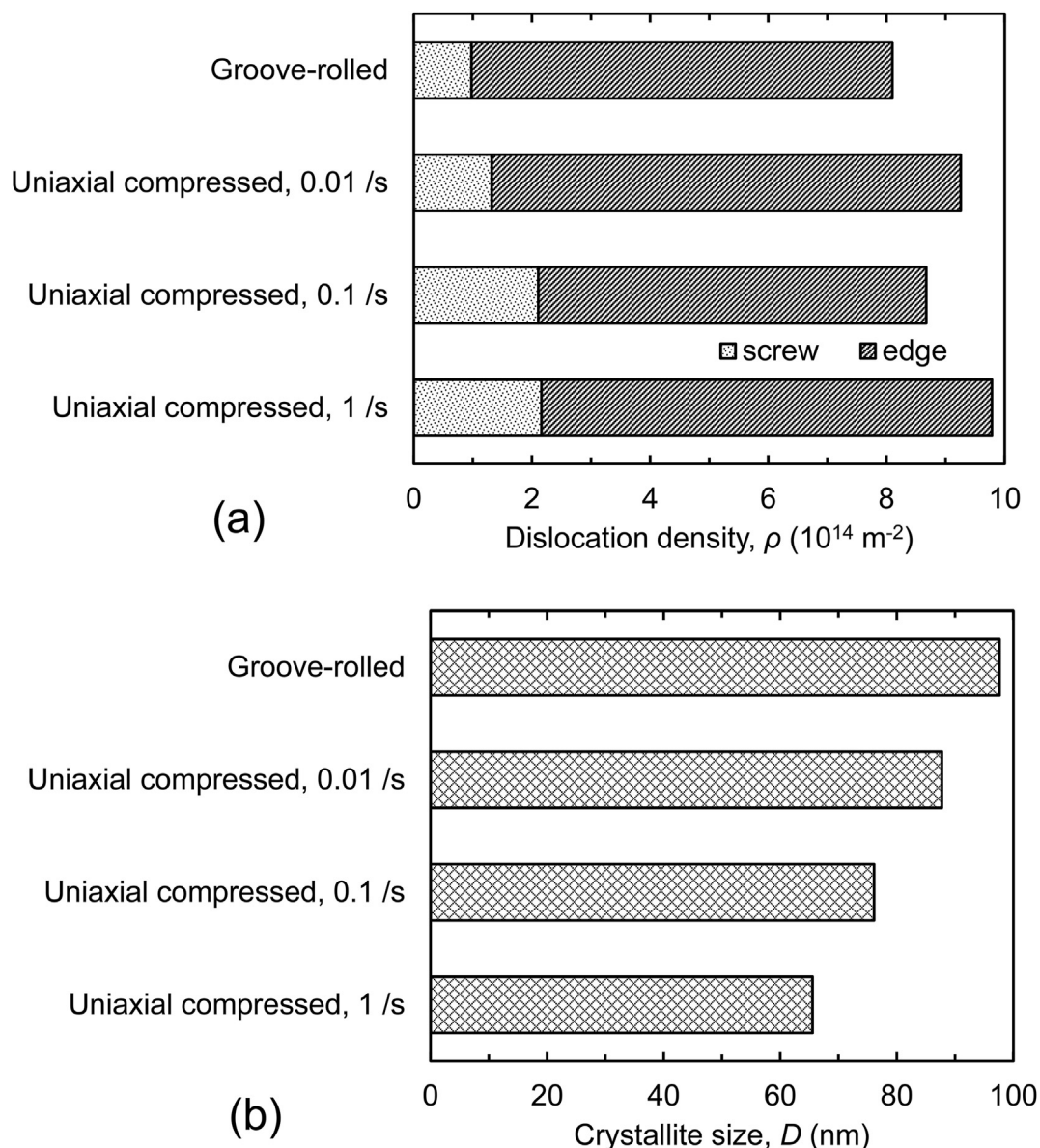


Fig. 11. Dislocation density (a) and crystallite size (b) of the groove-rolled material and the uniaxially compressed materials. The screw dislocation density increased, and the crystallite size decreased with the increase of strain rate.

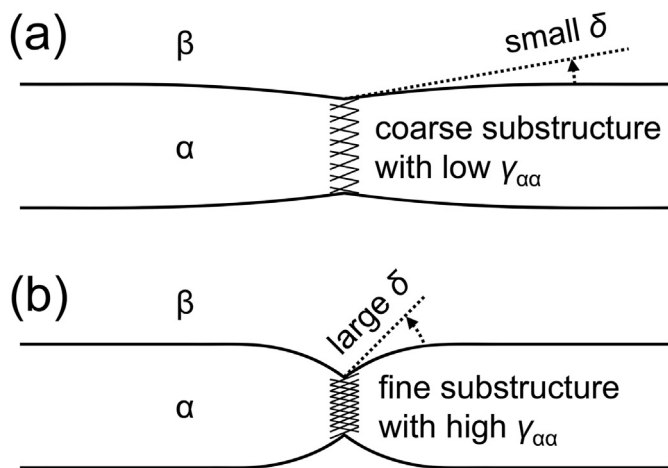


Fig. 12. Schematic illustration of thermal grooving. The groove angle δ is greater as finer substructure with higher α/α interfacial energy $\gamma_{\alpha\alpha}$, which promotes the division and fragmentation of α platelets.

consisting of higher-density dislocations is expected in the case of finer substructures existing in α platelets before annealing. From the above, it is suggested that groove-rolling and uniaxial compression with higher strain rates promote the formation of higher energy α/α interfaces during annealing.

4.3. Influence of the substructure of α platelets on equiaxed α microstructure evolution

In the material that was uniaxially compressed with the relatively high strain rate of 1 s^{-1} , the equiaxed α platelets were

completed following annealing over a short time of 1.8 ks as shown in Fig. 9a. Based on the variation in the number density of α_{eq} grains during annealing as shown in Fig. 9c, the equiaxing process of wrought materials is mainly classified into two stages: i.e. (i) formation of new α_{eq} grains by the division and fragmentation of the deformed α platelets, and (ii) extinction of the α platelets by coarsening of the α_{eq} grains. Stage (i) generally proceeds at the beginning of annealing since it requires a driving force provided by the strain energy. In this work, the investigated wrought materials exhibited almost the same coarsening rates of the α_{eq} grains, as shown in Fig. 9b. Thus, it is presumed that stage (ii) has little influence on the difference in the increasing rate of the α_{eq} volume fraction. In contrast, stage (i) dominated the increase in the volume fraction of the α_{eq} grains in the material compressed at a strain rate of 1 s^{-1} , and that had been equiaxed over a short annealing time as shown in Fig. 9c. TEM observation (Fig. 10) and XRD line profile analyses (Fig. 11) revealed that compression with higher strain rates promotes the retention of the screw dislocation introduced during deformation and the formation of finer substructures in the α platelets. The fine substructure introduced during the deformation is expected to form the α/α interface having high surface energy during subsequent annealing. From Equation (2), these high energy α/α interfaces increase the groove angle δ in thermal grooving as shown in Fig. 12. Therefore, it is presumed that large quantities of α/α interfaces, where thermal grooving occurs easily in the α platelets, were formed, and the frequency of the division and fragmentation of the α platelets increased in the material compressed at a higher strain rate of 1 s^{-1} . From the above, it is suggested that materials should be deformed with higher strain rates to introduce fine substructures in the α platelets in order to obtain fine equiaxed microstructures within a short annealing time without coarsening the α_{eq} grains.

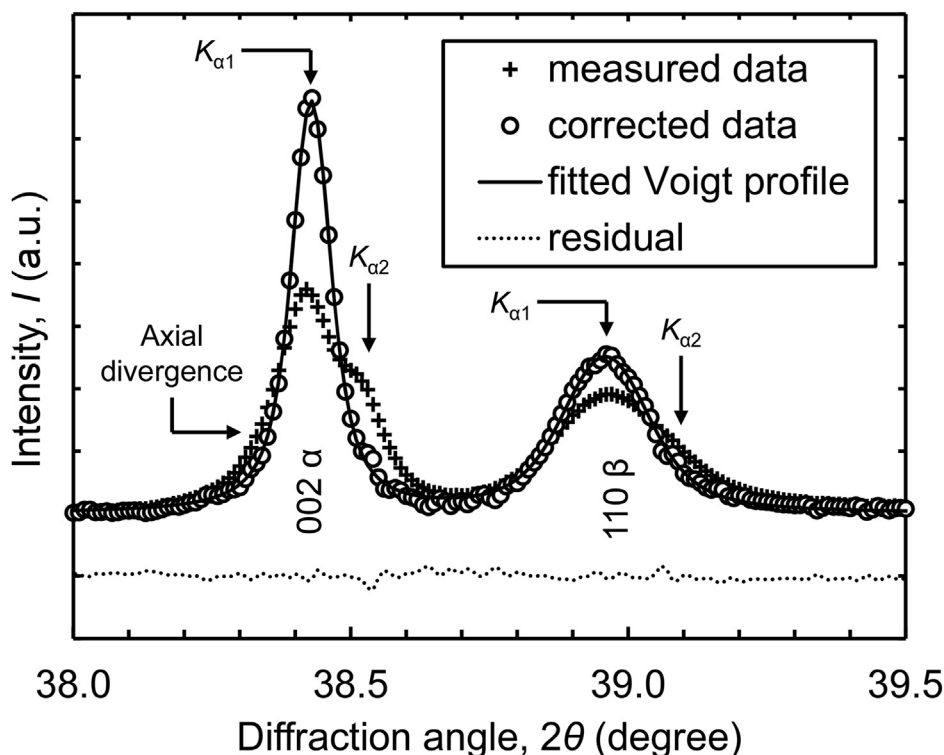


Fig. 13. XRD line profile of the uniaxially compressed material at a strain rate of 0.01 s^{-1} . Measured data are corrected by the deconvolution–convolution treatment and fitted by superimposed Voigt profile.

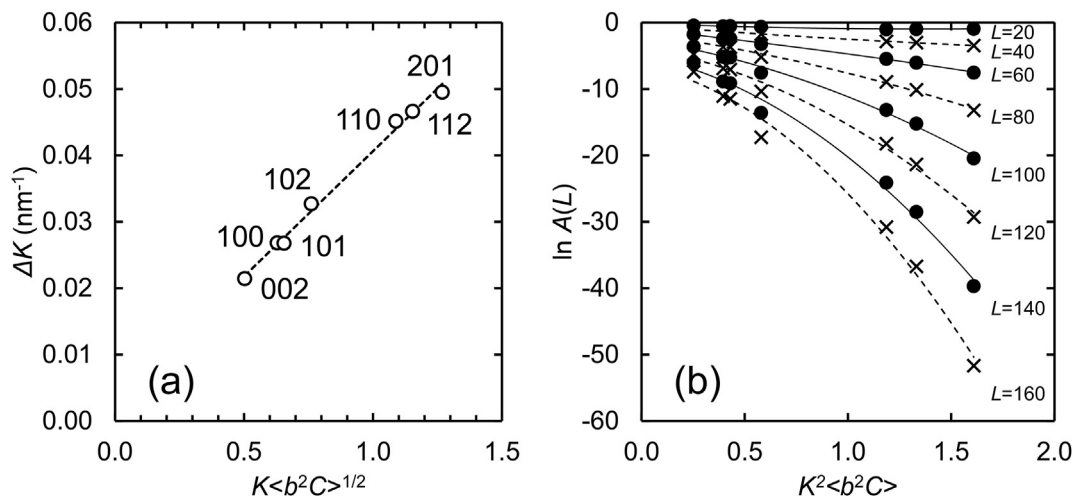


Fig. 14. Modified Williamson-Hall (a) and modified Warren-Averbach (b) plots of the uniaxially compressed material at a strain rate of 0.01 s^{-1} . Average contrast factor b^2C is optimized by the generic algorithm.

5. Conclusions

The influence of high-temperature deformation such as groove-rolling and uniaxial compression in the $\alpha+\beta$ region on the equiaxed α microstructure evolution during annealing for Ti-10Al-1Zr-1Mo-1Nb alloy was investigated. A summary of the main conclusions are as follows:

- (1) The compressive flow behavior of the alloy at 1173 K (in $\alpha+\beta$ region) exhibited a peak flow stress at relatively low strains (the true strain of approximately 0.03) followed by flow softening, regardless of the strain rates.
- (2) The microstructure developed in the groove-rolled material consisted of deformed α_p platelets and transformed α_s platelets due to repeated deformation and reheating. In contrast, the uniaxially compressed materials showed heavily deformed α microstructure such as distorted and kinked α_p platelets and fine α_{eq} grains distributed in a necklace shape around shear bands. The rotation of α_p platelets in the direction of metal flow revealed the evolution of the deformation texture to be characterized by a rather deformable orientation with higher Schmid factor during groove-rolling and uniaxial compression, and corresponded to the softening of the uniaxial compression flow curves.
- (3) The process increasing the volume fraction of α_{eq} during annealing was classified into two steps, i.e. (i) formation of new α_{eq} grains by the division and fragmentation of α platelets, and (ii) extinction of α platelets by coarsening of α_{eq} grains. In the material that was uniaxially compressed with higher strain rate of 1 s^{-1} , the equiaxed α grains were developed within a shorter annealing compared to the others, where the division and fragmentation of α platelets in the material was predominant and an advance to coarsening α grains.
- (4) The installed screw dislocations under higher strain rate deformation provided a fine substructure and high energy α/α boundaries in α platelets. The fine substructure promotes the division and fragmentation of the α platelets due to thermal grooving during annealing. Therefore, deformation with higher strain rates for the alloy is necessary for developing a fine equiaxed α microstructure during the annealing process without its coarsening.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix. Procedure of XRD line profile analysis

The estimation of the substructure characteristics such as dislocation density and crystallite size is based on following equation [37].

$$\ln A(L) = \ln A^S(L) - 2\pi^2 L^2 g^2 \epsilon_g(L)^2 \quad (3)$$

where $A(L)$ is the real part of Fourier coefficients of the structural profile, $A^S(L)$ is the size component of Fourier coefficients, g is the absolute value of the diffraction vector, $\epsilon_{g,L}^2$ is the mean square strain, and L is the Fourier length. In dislocated crystals, $\epsilon_{g,L}^2$ is defined as follows [38].

$$\epsilon_g(L)^2 \approx \frac{\rho b^2 C}{4\pi} f\left(\frac{L}{R_e}\right) \quad (4)$$

in which,

$$\overline{b^2 C} = \sum_{i=1}^N f_i b_i^2 \overline{C}^{(i)} \quad (5)$$

where ρ is the dislocation density, b is the modulus of the Burgers vector, $\overline{C}$ and $\overline{C}^{(i)}$ are the average dislocation contrast factor, $f(L/R_e)$

is the Wilkens function, and R_e is the effective outer cutoff radius of dislocations, respectively. Furthermore, f_i ($N = 11$ for hexagonal crystals) is the fraction of the activated slip plane families. The dislocation contrast factors are averaged over the permutations of the corresponding hkl indices. The average contrast factors for a particular slip plane family in a hexagonal crystal are given as follows.

$$\bar{C}_{hkl}^{(i)} = \bar{C}_{hk0}^{(i)} [1 + q_1^{(i)}x + q_2^{(i)}x^2] \quad (6)$$

Here $x = (2/3)(l/ga)^2$, $q_1^{(i)}$ and $q_2^{(i)}$ are parameters depending on the i th slip plane family and the elastic properties of the crystal, $\bar{C}_{hk0}^{(i)}$ are the average contrast factors for the i th slip plane family corresponding to the $hk0$ type reflections, and a is the lattice constant in the basal plane. In the present work, the $q_1^{(i)}$ and $q_2^{(i)}$ parameters and the values of $\bar{C}_{hk0}^{(i)}$ of pure titanium are employed [24].

Fig. 13 represents 0002 α and 110 β diffraction peaks in the XRD line profile of the material that was uniaxially compressed at a strain rate of 0.01 s⁻¹. A measured line profile is usually the convolution of a physical line profile from a sample and an instrumental line profile from an optical system of XRD. In fact, the 0002 α diffraction peak shown in Fig. 13 exhibits an asymmetrical profile due to the instrumental aberrations, i.e. the axial-divergence aberration in the lower angle and the CuK α_2 spectrum in the higher angle of the peak. In the present work, therefore, the measured line profiles were corrected by the deconvolution–convolution treatment [25–27]. In the modified Warren-Averbach procedure, each diffraction peak profile is separated and analyzed by Fourier transformation. Since the diffraction peak tails may overlap each other, the whole line profile was fitted and separated by superimposing Voigt profiles in the following K abscissa, and the fitted Voigt profiles were analyzed.

$$K = 2 \sin \theta / \lambda \quad (7)$$

Here, 2θ and λ are diffraction angle and wavelength of CuK α_1 spectrum, respectively. The centered Voigt profile ($V(K; A, \sigma, \gamma)$), for which the peak at zero is expressed as a convolution of the following centered Gaussian ($G(K; \sigma)$) and Lorentzian ($L(K; \gamma)$) profiles:

$$G(K; \sigma) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left(-\frac{K^2}{2\sigma^2}\right) \quad (8)$$

$$L(K; \gamma) = \frac{1}{\pi\gamma} \left(1 + \frac{K^2}{\gamma^2}\right)^{-1} \quad (9)$$

$$V(K; A, \sigma, \gamma) = A \int_{-\infty}^{\infty} G(K'; \sigma) L(K - K'; \gamma) dK' \quad (10)$$

where $f_G = 2\sigma\sqrt{2\ln 2}$ and $f_L = 2\gamma$ are the full width at half maximum of the Gaussian and Lorentzian profiles, and A is the amplitude of the Voigt profile. A better approximation for the full width at half maximum of the Voigt profile (ΔK) is given as follows [39].

$$\Delta K \approx \sqrt{f_G^2 + 0.2166f_L^2} + 0.5346f_L \quad (11)$$

The normalized Fourier coefficient ($A(L)$) of the Voigt profile is analytically derived as follows:

$$A(L) = \exp\left(-2\pi^2\sigma^2L^2 - 2\pi\gamma L\right) \quad (12)$$

and these Fourier coefficients were analyzed by Equation (3).

To determine the average contrast factor by Equations (5) and (6), the fractions of the activated slip plane families are required. The fractions that optimize the modified Williamson-Hall plot [23] are adopted in the present work. The relationship between the diffraction peak position (K) and the full width at half maximum (ΔK) in crystal introduced dislocations is expressed as follows:

$$\Delta K \approx \frac{0.9}{D} + \left(\frac{\pi B^2 \rho}{2}\right)^{\frac{1}{2}} \cdot (K^2 \bar{b}^2 C)^{\frac{1}{2}} + O(K^2 \bar{b}^2 C) \quad (13)$$

where B denotes the variable depending on the effective outer cutoff radius of dislocations (R_e). By applying the average contrast factor calculated by Equation (5) from the optimal fractions of slip plane families (f_i), ΔK exhibits a quadratic correlation with $(K^2 \bar{b}^2 C)^{1/2}$. Conversely, it is possible to derive the effective average contrast factor by optimizing the fractions f_i that give a better quadratic correlation between ΔK and $(K^2 \bar{b}^2 C)^{1/2}$. Here, the optimal fractions f_i that maximize the quadratic correlation coefficient are calculated with the genetic algorithm. Fig. 14a represents the modified Williamson-Hall plot optimized for the material compressed at a strain rate of 0.01 s⁻¹. The plots of ΔK and $(K^2 \bar{b}^2 C)^{1/2}$ show a good correspondence with the dashed line given by Equation (13). Then the relationship between the Fourier coefficients $A(L)$ and the optimized values of $K^2 \bar{b}^2 C$ for each Fourier length L can be described as shown in Fig. 14b. Specifically, the dislocation density and crystallite size in the material can be evaluated by comparing the quadratic fitting coefficients of the plots shown in Fig. 14b with Equations (3) and (4).

References

- [1] R.R. Boyer, An overview on the use of titanium in the aerospace industry, *Mater. Sci. Eng., A* 213 (1996) 103–114, [https://doi.org/10.1016/0921-5093\(96\)10233-1](https://doi.org/10.1016/0921-5093(96)10233-1).
- [2] D. Eylon, S. Fujishiro, P.J. Postans, F.H. Froes, High-temperature titanium alloys—a review, *JOM* 36 (1984) 55–62, <https://doi.org/10.1007/BF03338617>.
- [3] H. Onodera, K. Ohno, T. Yamagata, M. Yamazaki, The effect of β -stabilizer content on tensile properties of α - β titanium alloys, *Tetsu-To-Hagane* 72 (1986) 284–291, https://doi.org/10.2355/tetsutohagane1955.72.2_284.
- [4] K. Yamada, T. Noda, T. Iikubo, The influence of alloying elements on the mechanical behavior of near- α titanium alloys, *Denki-Seiko (Electr. Furn. Steel)* 64 (1993) 93–98, <https://doi.org/10.4262/denkiseiko.64.93>.
- [5] Y. Yamabe-Mitarai, R. Zempo, T. Kitashima, S. Emura, H. Murakami, *Proc. 1st Int. Conf. (2015) 335*, 123HiMAT.
- [6] S. Matsunaga, A. Serizawa, Y. Yamabe-Mitarai, Effect of Zr on microstructure and oxidation behavior of α and $\alpha + \alpha_2$ Ti-Al-Nb alloys, *Mater. Trans.* 57 (2016) 1902–1907, <https://doi.org/10.2320/matertrans.MAW201603>.
- [7] K. Shimagami, S. Matsunaga, A. Yumoto, T. Ito, Y. Yamabe-Mitarai, Solid solution hardening and precipitation hardening of α_2 -Ti $_3$ Al in Ti-Al-Nb alloys, *Mater. Trans.* 58 (2017) 1404–1410, <https://doi.org/10.2320/matertrans.MAW201707>.
- [8] K. Shimagami, T. Ito, Y. Toda, A. Yumoto, Y. Yamabe-Mitarai, Effects of Zr and Si addition on high-temperature mechanical properties and microstructure in Ti-10Al-2Nb-based alloys, *Mater. Sci. Eng., A* 756 (2019) 46–53, <https://doi.org/10.1016/j.msea.2019.04.031>.
- [9] M. Masuyama, K. Shimagami, Y. Toda, M. Tetsuya, T. Ito, M. Shimojo, Y. Yamabe-Mitarai, Microstructure evolution and creep behavior of near- α Ti alloy produced by thermomechanical processing, *Mater. Trans.* 60 (2019) 2336–2345, <https://doi.org/10.2320/matertrans.MT-MAW2019010>.
- [10] S. Tani, O. Umezawa, Y. Yamabe-Mitarai, Microstructural evolution in Ti-10Al-12r-1Mo-1Nb alloy under thermomechanical treatment in β -region, *Mater. Sci. Eng. A* 796 (2020) 140149, <https://doi.org/10.1016/j.msea.2020.140149>.
- [11] W.J. Evans, Optimising mechanical properties in alpha+beta titanium alloys, *Mater. Sci. Eng., A* 243 (1998) 89–96, [https://doi.org/10.1016/S0921-5093\(97\)00784-3](https://doi.org/10.1016/S0921-5093(97)00784-3).
- [12] S. Roy, S. Suwas, Orientation dependent spheroidization response and macrozone formation during sub β -transus processing of Ti-6Al-4V alloy, *Acta*

- Mater. 134 (2017) 283–301, <https://doi.org/10.1016/j.actamat.2017.04.071>.
- [13] G. Lüttjering, Influence of processing on microstructure and mechanical properties of (α + β) titanium alloys, Mater. Sci. Eng., A 243 (2002) 32–45, [https://doi.org/10.1016/S0921-5093\(97\)00778-8](https://doi.org/10.1016/S0921-5093(97)00778-8).
- [14] H. Imai, G. Yamane, H. Matsumoto, V. Vidal, V. Velay, Superplasticity of metastable ultrafine-grained Ti6242S alloy: mechanical flow behavior and microstructural evolution, Mater. Sci. Eng., A 754 (2019) 569–580, <https://doi.org/10.1016/j.msea.2019.03.085>.
- [15] R. Hosseini, M. Morakabati, S.M. Abbasi, A. Hajari, Development of a trimodal microstructure with superior combined strength, ductility and creep-rupture properties in a near alpha titanium alloy, Mater. Sci. Eng., A 696 (2017) 155–165, <https://doi.org/10.1016/j.msea.2017.04.068>.
- [16] H. Ghonem, Microstructure and fatigue crack growth mechanisms in high temperature titanium alloys, Int. J. Fatig. 32 (2010) 1448–1460, <https://doi.org/10.1016/j.jfatigue.2010.02.001>.
- [17] M. Peters, A. Gysler, G. Lüttjering, Influence of texture on fatigue properties of Ti-6Al-4V, Metall. Trans. A 15 (1984) 1597–1605, <https://doi.org/10.1007/BF02657799>.
- [18] W. Mullins, Theory of thermal grooving, J. Appl. Phys. 28 (1957) 333–339, <https://doi.org/10.1063/1.1722742>.
- [19] S.L. Semiatin, V. Seetharaman, I. Weiss, Flow behavior and globularization kinetics during hot working of Ti-6Al-4V with a colony alpha microstructure, Mater. Sci. Eng., A 263 (1999) 257–271, [https://doi.org/10.1016/S0921-5093\(98\)01156-3](https://doi.org/10.1016/S0921-5093(98)01156-3).
- [20] N. Stefansson, S.L. Semiatin, Mechanisms of globularization of Ti-6Al-4V during static heat treatment, Metall. Mater. Trans. A 34 (2003) 691–698, <https://doi.org/10.1007/s11661-003-0103-3>.
- [21] S. Zherebtsov, M. Murzinova, G. Salishchev, S.L. Semiatin, Spheroidization of the lamellar microstructure in Ti-6Al-4V alloy during warm deformation and annealing, Acta Mater. 59 (2011) 4138–4150, <https://doi.org/10.1016/j.actamat.2011.03.037>.
- [22] W.S. Rasband, U. S. Natl. Institutes Heal. Bethesda, Maryland, USA, Retrieved from, <https://imagej.nih.gov/ij/>.
- [23] T. Ungár, A. Borbély, The effect of dislocation contrast on x-ray line broadening: a new approach to line profile analysis, Appl. Phys. Lett. 69 (1996) 3173–3175, <https://doi.org/10.1063/1.117951>.
- [24] I.C. Dragomir, T. Ungár, Contrast factors of dislocations in the hexagonal crystal system, J. Appl. Crystallogr. 35 (2002) 556–564, <https://doi.org/10.1107/S0021889802009536>.
- [25] T. Ida, H. Toraya, Deconvolution of the instrumental functions in powder X-ray diffractometry, J. Appl. Crystallogr. 35 (2002) 58–68, <https://doi.org/10.1107/S0021889801018945>.
- [26] T. Ida, S. Ono, D. Hattan, T. Yoshida, Y. Takatsu, K. Nomura, Deconvolution–convolution treatment on powder diffraction data collected with CuK α X-ray and NiK β filter Powder Diff. <https://doi.org/10.1017/S0885715618000258>, 2018, 33,80–87.
- [27] T. Ida, S. Ono, D. Hattan, T. Yoshida, Y. Takatsu, K. Nomura, Improvement of deconvolution–convolution treatment of axial-divergence aberration in Bragg–Brentano geometry, Powder Diff. 33 (2018) 121–133, <https://doi.org/10.1017/S0885715618000349>.
- [28] W.G. Burgers, On the process of transition of the cubic-body-centered modification into the hexagonal-close-packed modification of zirconium, Physica 1 (1934) 561–586, [https://doi.org/10.1016/S0031-8914\(34\)80244-3](https://doi.org/10.1016/S0031-8914(34)80244-3).
- [29] R.M. Miller, T.R. Bieler, S.L. Semiatin, Flow Softening during hot working of Ti-6Al-4V with a lamellar colony microstructure, Scripta Mater. 40 (1999) 1387–1393, [https://doi.org/10.1016/S1359-6462\(99\)00061-5](https://doi.org/10.1016/S1359-6462(99)00061-5).
- [30] S.L. Semiatin, T.R. Bieler, Effect of texture changes on flow softening during hot working of Ti-6Al-4V, Metall. Mater. Trans. A 32 (2001) 1871–1875, <https://doi.org/10.1007/s11661-001-0166-y>.
- [31] J.L.W. Warwick, N.G. Jones, I. Bantounas, M. Preuss, D. Dye, In situ observation of texture and microstructure evolution during rolling and globularization of Ti-6Al-4V, Acta Mater. 61 (2013) 1603–1615, <https://doi.org/10.1016/j.actamat.2012.11.037>.
- [32] J.L.W. Warwick, N.G. Jones, I. Bantounas, M. Preuss, D. Dye, In situ observation of texture and microstructure evolution during rolling and globularization of Ti-6Al-4V, Acta Mater. 61 (2013) 1603–1615, <https://doi.org/10.1016/j.actamat.2012.11.037>.
- [33] I.M. Lifshitz, V.V. Slyozov, The kinetics of precipitation from supersaturated solid solutions, J. Phys. Chem. Solid. 19 (1961) 35–50, [https://doi.org/10.1016/0022-3697\(61\)90054-3](https://doi.org/10.1016/0022-3697(61)90054-3).
- [34] S.L. Semiatin, B.C. Kirby, G.A. Salishchev, Coarsening behavior of an alpha-beta titanium alloy, Metall. Mater. Trans. A 35 (2004) 2809–2819, <https://doi.org/10.1007/s11661-004-0228-z>.
- [35] J. Xu, W. Zeng, Z. Jia, X. Sun, J. Zhou, Microstructure coarsening behavior of Ti-17 alloy with equiaxed alpha during heat treatment, J. Alloys Compd. 618 (2015) 343–348, <https://doi.org/10.1016/j.jallcom.2014.08.223>.
- [36] W.T. Read, W. Shockley, Dislocation models of crystal grain boundaries, Phys. Rev. 78 (1950) 275–289, <https://doi.org/10.1103/PhysRev.78.275>.
- [37] B.E. Warren, X-ray studies of deformed metals, Prog. Met. Phys. 8 (1959) 147–202, [https://doi.org/10.1016/0502-8205\(59\)90015-2](https://doi.org/10.1016/0502-8205(59)90015-2).
- [38] M. Wilkens, In J. Simmons, R. DeWit, & R. Bullough (Eds.), Proc. Conf. Fundam. Asp. Dislocation Theory, (pp. 1195–1221). Washington DC: U.S. National Bureau of Standards.
- [39] J.J. Olivero, R.L. Longbothum, Empirical fits to the Voigt line width: a brief review, J. Quant. Spectrosc. Radiat. Transfer 17 (1977) 233–236, [https://doi.org/10.1016/0022-4073\(77\)90161-3](https://doi.org/10.1016/0022-4073(77)90161-3).