The 8th TSME International Conference on Mechanical Engineering
12-15 December 2017
Bangkok, Thailand

AMM0021

Changes in the hardness and microstructures of severely deformed β-titanium alloys

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Abstract. Severe plastic deformation processes have been conducted on β-titanium alloys at room temperature using a multi-directional forging process that reduces the likelihood of seizing and galling in the dies, thus, allowing the processing of bulk titanium materials. The purpose of this study is to improve the mechanical properties of metastable β-titanium alloys by applying multi-directional forging and cold rolling. Severe plastic deformations have applied to a β single phase structure and a two phase structure in which the granular α phase precipitated in the matrix β phase as an initial structure before the deformation. After processing, cracks initiate in the coarse grained specimen with a β single phase after 18 cycles of multi-directional forging. The cumulative plastic strain ε is 7.35 after 17 cycles of multi-directional forging and the hardness is 335 HV0.2. After the coarse grained specimen with a β single phase has subjected to six cycles of multi-directional forging and cold rolling with a rolling reduction of 87%, the hardness has increased. Moreover, the α-β two phase structure with a granular α phase precipitated in the matrix β phase has undergone two cycles of multi-directional forging and cold rolling with a rolling reduction of 87%, and the hardness has also increased. The hardness of the specimen subjected to multi-directional forging before cold rolling is larger than the hardness of the specimen subjected to only cold rolling.

1. Introduction

Titanium has excellent characteristics, e.g., high corrosion resistance, biocompatibility, light weight, and high strength. Therefore, it is widely used in medical and transportation applications. By using a β-type titanium alloy, excellent cold workability and high strength after heat treatment can be expected. Equal-channel angular pressing and accumulative roll-bonding have attracted attention as methods for improving the mechanical properties such as tensile strength and hardness of titanium [1, 2]. Severe plastic deformation processes have been considered for titanium; however, difficulties arise as titanium has a relatively high deformation resistance and elastic recovery, and is susceptible to seizing and galling in the dies. For these reasons, severe plastic deformation processes for titanium have not been investigated sufficiently compared with other light metals, e.g., aluminum and magnesium [3, 4].

In this study, severe plastic deformation processes are conducted on β-titanium alloys at room temperature using a multi-directional forging (hereinafter referred to as "MDF") process that reduces the likelihood of seizing and galling in the dies, thus, allowing the processing of bulk titanium materials. The MDF process is performed by changing the loading axes (i.e., x → y → z → x…) from cycle to cycle [5, 6]. By repeating this process, the shape of the specimen is maintained during the severe plastic deformation.

The purpose of this study is to investigate the possibility of improving the mechanical properties of high-strength β-titanium alloys by applying multi-directional forging and cold rolling (hereinafter referred to as "CR”). Severe plastic deformations have applied to a β single phase structure and a two phase structure in which the granular α phase precipitated in the matrix β phase as an initial structure before the deformation.

2. Experiment

2.1. Materials and specimens

Solution-treated plates made from a metastable β-type Ti-15V-3Cr-3Sn-3Al alloy (hereinafter referred to as "Ti-15-3”) with a 5.0 mm thickness have used as the starting material. The plates have cut by a wire-cut electric-discharge machine into rectangular pieces measuring 8.3 mm in the rolling direction, 6.6 mm in width, and 5.0 mm in thickness. The wire-cut electric-discharge machining left an altered layer on the surface of the specimen; this layer has removed by polishing. This rectangular specimen has used as A0 for multi-directional forging. The microstructures of the β single phase specimen (A0) and the two phase specimen (B0), which has annealed at 973 K after three cycles of multi-directional forging and the precipitated granular α phase, are shown in Figure 1.

The average grain size, d, of the recrystallized β single phase of specimen A0 is obtained by $d = 1.128 \times l$ (l is the average intercept length) using the American Society for Testing and Materials (ASTM) intercept method. Specimen A0 is coarse grained (d = 61.7 μm) and the hardness is 285 HV0.2. As determined by an image-processing measurement, the α grain size and the α phase ratio of specimen B0 are 0.66 μm and approximately 20%, respectively. The hardness of specimen B0 is 304 HV0.2.

A list of specimen codes corresponding to the combinations of processing conditions is shown in Table 1. The authors investigated the changes in the mechanical properties of the initial structures after severe plastic deformations. Cold rolling has carried out after multi-directional forging to apply further strain to the multi-directional forged specimens.

Two types of specimens have subjected to severe plastic deformation by multi-directional forging and cold rolling. The initial structures before severe plastic deformation are indicated as follows by (a) β single phase, and (b) α-β two phase with granular α phase precipitated in matrix β phase.

(a) Specimen A1 is a coarse grained β single phase structure (A0) specimen that underwent six cycles of multi-directional forging followed by cold rolling with a rolling reduction of 87%. Specimen ACR is specimen A0 that is cold rolled with a rolling reduction of 87% without multi-directional forging. Specimen ACR has further annealed at 1023 K for 0.6 ks to obtain a fine grained β single phase specimen (AFG) (d = 23.4 μm).

(b) Specimen B0 is an α-β two phase structure that underwent three cycles of multi-directional forging followed by annealing at 973 K for 10.8 ks. Look like voids in Figure 1 (b) are granular α grains. Specimen B1 is an α-β two phase structure (B0) specimen that underwent two cycles of multi-directional forging followed by cold rolling with a rolling reduction of 87%. Specimen BCR is specimen B0 that is cold rolled with a rolling reduction of 87% without multi-directional forging.

All five specimens, A1, ACR, AFG, B1, and BCR, have annealed at 723 K for 18 ks and then precipitation hardened. The same specimens have annealed at 973 K for 1.8 ks and then a granular α phase precipitated in a matrix β phase specimen has fabricated. The effect of the heat treatment process on the hardness of each specimen has investigated. Each cold rolled specimen is water-cooled after every pass to remove the processing heat.

2.2. Evaluation method

The annealing treatment has carried out in an electric furnace in a nitrogen gas-flow atmosphere. A Vickers hardness test has conducted and the microstructure has observed using scanning electron microscope (SEM) for various specimens. For the hardness test, a micro Vickers hardness tester was used. The test conditions are a load of 0.2 kgf and a holding time of 15 s. The specimen was cut in the direction perpendicular to the compressed direction. Subsequently, it was filled in a resin and the surface was mirror-polished. After that, the average of five points near the center of the specimen was used as the hardness data. For the SEM observation of the structure, the specimen surface has mirror-polished and then etched to make it easier to obtain information on the structural state.

2.3. Multi-directional forging process

An outline of the multi-directional forging process is shown in Figure 2. Dies have used to maintain the flat plane of the applied load direction. First, a load has applied to plane A perpendicular to the 8.3 mm lengthwise direction to compress this side from 8.3 mm to 5.2 mm. Plane B, perpendicular to the 6.6 mm width, has restrained by a die to maintain a flat plane; this resulted in a width increase from 6.6 mm to 8.1 mm. This represents one cycle of the multi-directional forging process. After performing one cycle of the multi-directional forging, the specimen has rotated and a load has applied to plane B, which reduced the side from 8.1 mm to 5.2 mm. The load has then similarly applied to plane C. The compression has applied three times from three directions such that each of the three planes experienced MDF from their respective vertical directions.

The specimens that underwent multi-directional forging have cold rolled in the lengthwise direction to reduce their thickness, resulting in a rolling reduction of 87%. Thin-plate specimens have fabricated with severer plastic deformation processes.

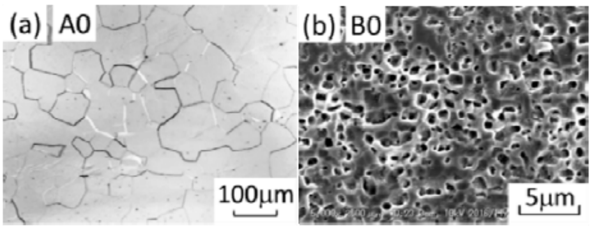

Figure 1. Initial microstructures of specimen before severe plastic deformations in Ti-15-3 alloy. (a) Specimen A0: β single phase, and (b) Specimen B0: α-β two phase with granular α phase precipitated in matrix β phase.

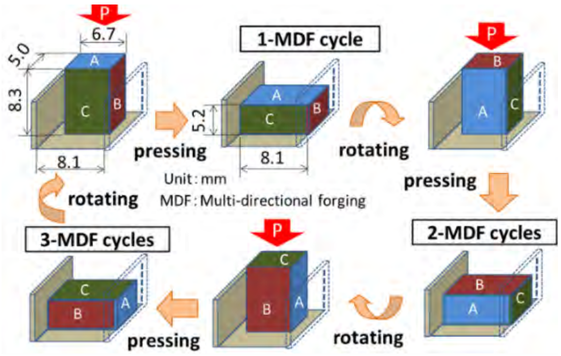

Figure 2. Schematic illustration of multi-directional forging process.

Table 1. Processing conditions of the specimens (a) β single phase, and (b) α-β two phase with granular α phase precipitated in matrix β phase.

(a)

Code	Processing conditions
A0	As received (β single phase, d_β = 61.7 μm)
AFG	A0→Cold rolled to 87%→Recrystallized at 1023 K (d_β = 23.4 μm)
ACR	As cold rolled to 87% (ε = 2.26)
A1	A0→6-MDF→Cold rolled to 87% (ε = 4.93)
A2	A1→Precipitation treated at 723 K
A3	A1→Annealed at 973 K below β transus

(b)

Code	Processing conditions
B0	A0→3-MDF→Fine grained α is precipitated at 973 K (α-β phase, d_α=0.66 μm)
BCR	B0→Cold rolled to 87% (ε=2.52)
B1	B0→2-MDF→Cold rolled to 87% (ε=3.12)
B2	B1→Precipitation treted at 723 K
B3	B1→Annealed at 973 K below β transus

Precipitation treated at 723 K for 18 ks and at 973K for 1.8ks d_α, d_β : grain size of α and β ε : cumulative plastic strain

3. Results and Discussion

3.1. Hardness changes in specimens subjected to severe plastic deformation

The hardness changes with the cumulative plastic strain are shown in Figure 3. The cumulative plastic strain is $\varepsilon = 0.43$ after one forging cycle. In a coarse grained β single phase specimen, cracks have initiated after 18 cycles of multi-directional forging. The crack extends from the top of the specimen towards the center. Although the cracks grow, multi-directional forging is possible up to 24 cycles, which is a very large strain. After 17 cycles of multi-directional forging, $\varepsilon = 7.35$ and the hardness is 335 HV0.2. After 4-MDF cycles ($\varepsilon = 1.73$), the hardness has already reached 328 HV0.2, which is almost the maximum hardness. Since the hardness increase is almost saturated, we attempted to increase the strain imparted by cold rolling. To equalize the hardness distribution, 6-MDF cycles ($\varepsilon = 2.60$) have performed followed by cold rolling with a rolling reduction up to 87%. The cumulative plastic strain, ε, of specimen A1 is 4.93. As a result, the hardness has increased to 365 HV0.2 (see specimen A1 in Figure 3), demonstrating that the hardness could be further increased by cold rolling after multi-directional forging.

The coarse grained β single phase specimen A0 has subjected to 3-MDF cycles. In addition, it has annealed at 973 K for 10.8 ks, and the granular α phase precipitated in the matrix β phase. A specimen made of this granular α phase is called specimen B0, and the hardness is 304 HV0.2. Cracks in specimen B0 initiate at 3-MDF cycles. The hardness at 2-MDF cycles, at which cracks are not observed, is 331 HV0.2 and the cumulative plastic strain, ε, is 0.88. However, cold rolling is possible up to a rolling reduction of 87%, even after 2-MDF cycles. As a result, the hardness has increased to 370 HV0.2 (see specimen B1 in Figure 3). The cumulative plastic strain ε of specimen B1 is 3.12.

The specimens A1 and B1 which were subjected to multi-directional forging before cold rolling have increased in hardness by approximately 11% to approximately 370 HV0.2 compared with the specimen which have only subjected to multi-directional forging. In an ordinary cold rolled specimen (e.g., ACR), the hardness has almost saturated at approximately 345 HV0.2 even when rolling to a reduction of 87%, and it could not be further increased. The specimens A1 and B1 subjected to multi-directional forging before cold rolling have increased in hardness by approximately 7% than the specimen ACR subjected only to cold rolling. The cumulative plastic strains of specimens A1 and B1 are, respectively $\varepsilon = 4.93$ and 3.12. The hardness of specimen B1 has increased when the cumulative plastic strain is smaller than that of specimen A1, which is cold rolled directly after multi-directional forging.

The hardness distributions of the four specimens with various process conditions are shown in Figure 4; (a) specimen A0 with coarse grained β single phase, (b) specimen A0 multi-directionally forged to six cycles, (c) specimen B0 with granular α phase precipitated in matrix β phase, and (d) specimen B0 multi-directionally forged to two cycles. In all the specimens, the hardness difference is only 5 HV0.2 between the average of the five central parts and the average of the four surface parts, and the hardness is almost uniform.

3.2. Hardness change with heat treatments

Various specimens that have subjected to severe plastic deformation also received heat treatment to investigate the change in hardness. The heat-treatment conditions have annealing at 673 K to 973 K for 1.8 ks and annealing at 998 K to 1023 K for 0.6 ks. The results are shown in Figure 5. Specimen B1 is the hardest at room temperature; when the specimens have heated to 723 K for 1.8 ks, the hardness of specimen A1 increased to 513 HV0.2 due to the fine precipitation of the α grains. The hardness of specimen B1 heated to 723 K for 1.8 ks is approximately the same as that of specimen ACR heated to 723 K for 1.8 ks, which has a smaller strain than specimen A1 because the α phase has already precipitated. The hardness of both specimens B1 and ACR heated to 723 K for 1.8 ks is approximately 480 HV0.2. No strain was accumulated in the specimen AFG which is a recrystallized material, and no increase in hardness was observed at any temperature for the 0.6 to 1.8 ks holding times.

Next, various specimens that had been subjected to severe plastic deformations have precipitation hardened. As the test conditions, the annealing temperature is 723 K and the holding times have 0.6 ks, 1.8 ks, 5.4 ks, 18 ks, and 54 ks. The results are shown in Figure 6. Specimen A1 annealed for 5.4 ks increased in hardness to the maximum hardness of 539 HV0.2 due to the fine precipitation of the α phase. No increase in hardness has observed even when it has held longer. The AFG hardness did not increase after a holding time of 18 ks.

3.3. Change in microstructures with heat treatments

The changes in the microstructures of various specimens at various annealing temperatures after subjection to severe plastic deformation are shown in Figure 7. Specimen ACR has fabricated by cold rolling coarse grained specimen A0 to a rolling reduction of 87%. The grain size of specimen A0 is 61.7 µm. Specimen A1 has fabricated by cold rolling coarse grained specimen A0 to a rolling reduction of 87% after six cycles of multi-directional forging. Specimen B1 has fabricated by cold rolling specimen B0 with a granular α phase precipitated in a matrix β phase to a rolling reduction of 87% after two cycles of multi-directional forging.

The grain sizes of α and β, and the α phase ratio of each specimen are shown in Table 2. The grain boundaries of the coarse grained β single phase specimen (d = 61.7 µm) has already collapsed. The deformed zone extending in the rolling direction becomes the texture of specimen ACR and specimen A1 before annealing (Figures 7(a) and (f)). It can be observed that the layer interval of the deformation zone is smaller for specimen A1 to which strain is added. In specimen B1, the granular α phase has precipitated by annealing at 973 K and the structure has extended in the processing direction (Figure 7(k)). By annealing each specimen, the α phase can be seen up to 998 K; however, at 1023 K it is completely solidified. The β-transus temperature of the Ti-15V-3Cr-3Sn-3Al is considered to be present between 998 K and 1023 K.

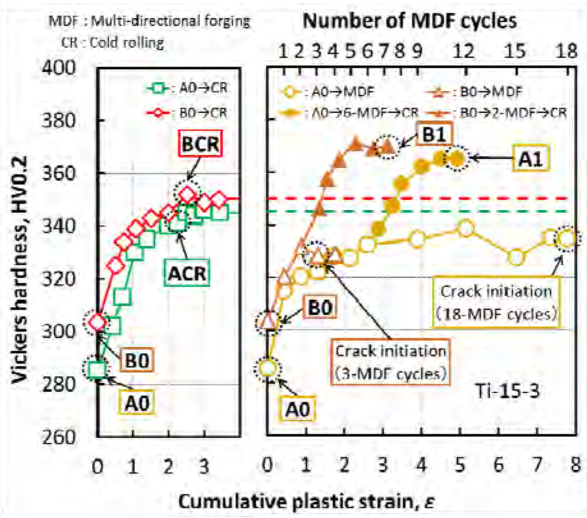

Figure 3. The hardness changes with the cumulative plastic strain in specimens processed by multi-directional forging before cold rolling.

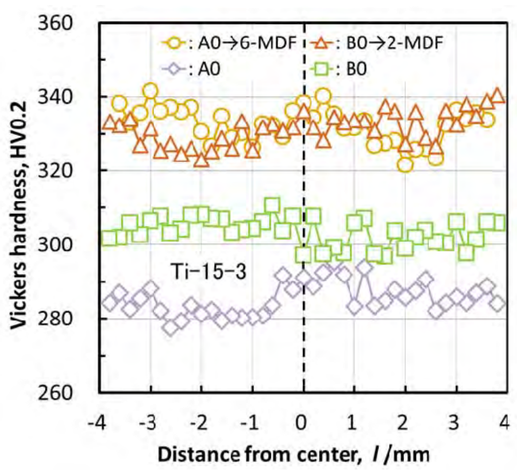

Figure 4. The hardness distributions of specimens processed by multi-directional forging.

In the specimen annealed at 1023 K in which the α phase is not observed, the recrystallization of the matrix β phase has completed in all the specimens and resulted in an equiaxed grain (Figures 7(e), (j), and (o)). In the specimen annealed at 998 K, in which the α grains have slightly observed, specimen ACR has partially recrystallized while the recrystallized grains of the matrix β phase have in the process of recrystallizing. However, specimens A1 and B1, which had a large cumulative plastic strain, have almost recrystallized, and the matrix β phase has finely divided. The grain size at that time is approximately 6 µm (Figures 7(i) and (n)).

In the specimen annealed at 948 K and 973 K, the α phase has precipitated in the form of granules or plates along the deformation zone, and no initiation of recrystallization has observed in ACR (Figures 7(b) and (c)). In specimens A1 and B1, α grains are very uniformly dispersed as compared with the specimen ACR (Figures 7(g), (h), (l), and (m)). Here, the α grains of A1 are extremely fine compared with B1, which already precipitated the α phase before annealing, and the number of α grains is large (Figures 7(g) and (h)).

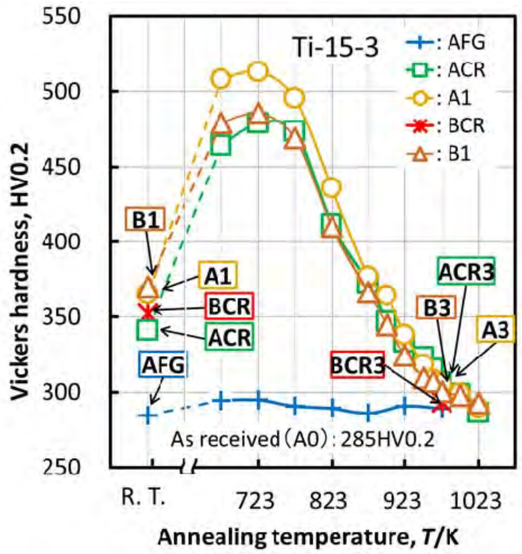

Figure 5. The hardness changes with the annealing temperatures in specimens processed by multi-directional forging before cold rolling.

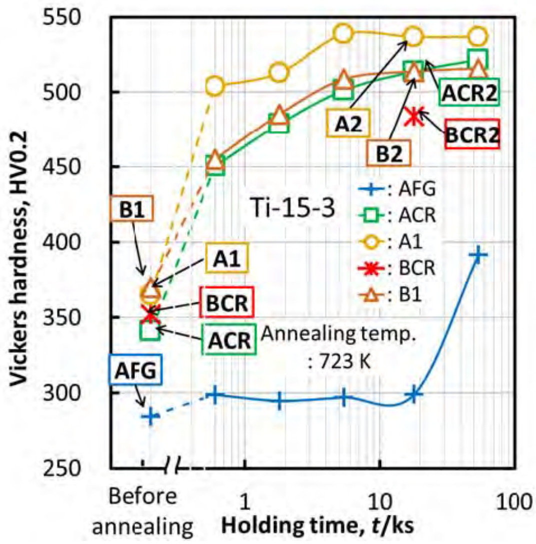

Figure 6. The hardness changes with the holding times in specimens processed by multi-directional forging before cold rolling.

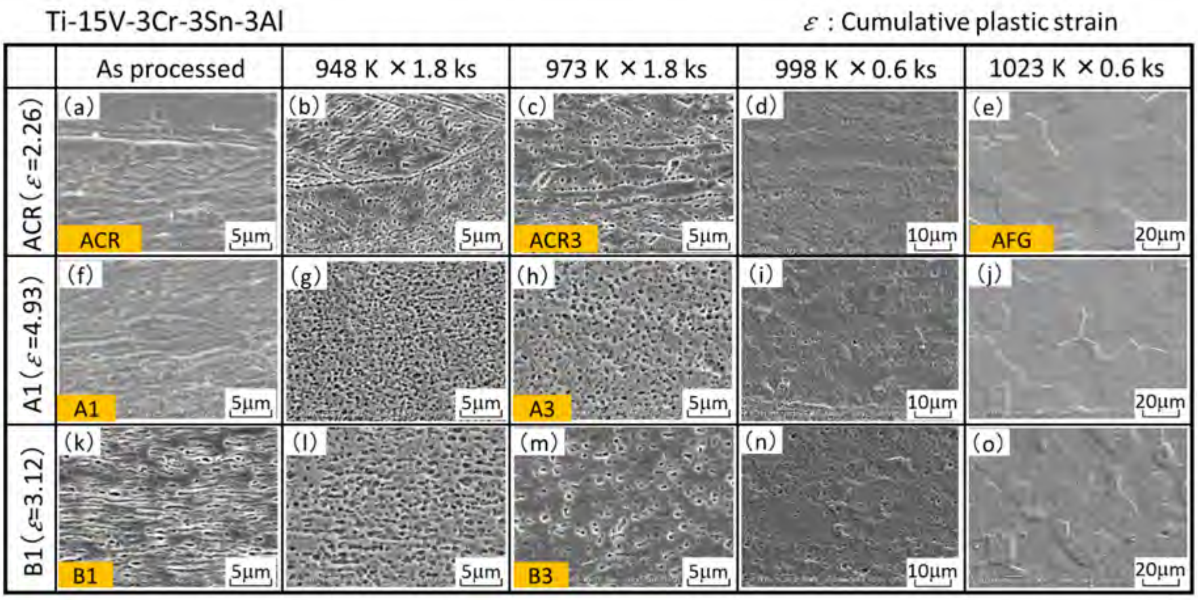

Figure 7. SEM images of specimens, which are subjected to severe plastic deformation processes, annealed at various temperatures.

The α grains of the specimen annealed at 948 K are finer than the α grains of the specimen annealed at 973 K, and the number of α grains has increased. It has found that recrystallization at lower temperatures than ordinary cold rolling is possible. Moreover, severe plastic deformation combined with multi-directional forging and subsequent cold rolling enables a further refinement of the crystal grains. In addition, the α grains can be finely dispersed very uniformly at a temperature just below recrystallization.

Table 2. Grain size and α phase ratio of the specimens, which are subjected to severe plastic deformation processes, annealed at various temperatures.

	ACR (ε = 2.26)				A1 (ε = 4.93)				B1 (ε = 3.12)			
Annealing temp.	948 K	973 K	998 K	1023 K	948 K	973 K	998 K	1023 K	948 K	973 K	998 K	1023 K
d_α (μm)	0.42	0.35	0.50	-	0.35	0.44	0.36	-	0.55	0.58	0.55	-
d_β (μm)	-	-	-	23.4	-	-	6.7	17.6	-	-	5.0	13.3
α (%)	26.3	14.2	3.7	-	26.1	16.3	1.9	-	30.3	18.2	4.0	-

4. Conclusions

The authors carried out severe plastic deformations by multi-directional forging and cold rolling a β-titanium alloy Ti-15V-3Cr-3Sn-3Al. The changes in hardness and microstructure have investigated, leading to the following conclusions.

(1) In specimen A0 with a coarse grained (d = 61.7 μm) β single phase structure, cracking occurred after 18 cycles of multi-directional forging. The cumulative plastic strain ε is 7.35 after 17 cycles of multi-directional forging and the hardness is 335 HV0.2.

(2) Specimen A1 has fabricated by cold rolling to a rolling reduction of 87% after six cycles of multi-directional forging using coarse grained specimen A0. Specimen B0 with a granular α phase precipitated in a matrix β phase has subjected to two cycles of multi-directional forging. Subsequently, the specimen has subjected to cold rolling with a rolling reduction of 87% to fabricate the specimen B1. The cumulative plastic strain, ε, of specimen A1 and B1 have 4.93 and 3.12, respectively. Specimen B1 increased in hardness to 370 HV0.2, although it had a smaller cumulative plastic strain than specimen A1. The hardness of the specimen subjected to multi-directional forging before cold rolling is larger than the hardness of the specimen subjected to only cold rolling. It has found that multi-directional forging before cold rolling is effective for increasing hardness.

(3) Specimen ACR has fabricated by cold-rolling coarse grained specimen A0 to a rolling reduction of 87%. The recrystallization of Al and B1 is almost complete at 998 K annealing where the α grains have slightly observed. The recrystallization in the matrix β phase has more advanced than specimen ACR as it has cold rolled. The grain size at that time is approximately 6 μm. The α grains of specimen A1 annealed at 948 K and 973 K have much finer and more numerous than the α grains of specimen B1 annealed at the same temperatures. The α-phase grains of the specimen annealed at 948 K became finer than the α grains of the specimen annealed at 973 K, and the number of α-phase grains increased.

(4) The hardness of specimen A1 annealed at 723 K for 1.8 ks is approximately 510 HV0.2 or more compared with other specimens under the same conditions. Moreover, the hardness of specimen A1 annealed at 723 K for 5.4 ks increased to approximately 540 HV0.2.

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