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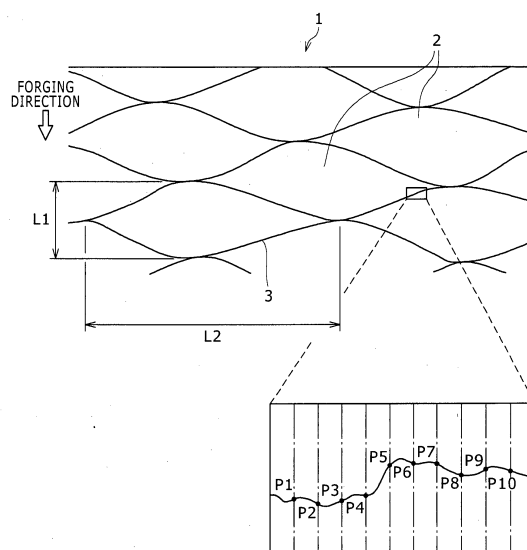
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(54) **TITANIUM-ALLOY FORGING MATERIAL AND METHOD FOR MANUFACTURING SAME**

(57) Provided is a titanium-alloy forging material in which fatigue-strength characteristics are improved without worsening ultrasonic flaw detection. A β -forged titanium-alloy forging material (1) is characterized in that the area ratio of non-flat grains, which are prior β -grains (2) having an aspect ratio of 3 or less and a diameter in the forging direction of at least 20 μm , and an α -phase ratio at the crystal grain boundary (3) of at least 80%, is less than 10%, and the area ratio of flat grains, which are prior β -grains having an aspect ratio greater than 3 and a diameter in the forging direction of 20-700 μm , and an α -phase ratio at the crystal grain boundary (3) of at least 80%, is 85% or greater, and the average orientation difference of the α -phase crystal orientation deposited at the crystal grain boundary (3) of the flat grains is at least 6°.

FIG. 1



Description

Technical Field

5 **[0001]** The present invention relates to a forged titanium alloy material to be used for engine components of aircraft and the like and a method of manufacturing the forged material.

Background Art

10 **[0002]** Since $\alpha+\beta$ titanium alloys typified by Ti-6Al-4V alloy are excellent in various properties such as weldability, superplasticity, and diffusion bonding property as well as light weight, high strength, and high corrosion resistance, the $\alpha+\beta$ titanium alloys have been used widely in the aircraft industry as engine components and the like.

15 **[0003]** The $\alpha+\beta$ titanium alloys stably have both an α phase which is a main phase composed of close-packed hexagonal crystals (hct structure) and a β phase composed of body-centered cubic crystals (bcc structure) at room temperature and they have a β single phase in a temperature range of a β -transus temperature (T_β) or higher.

20 **[0004]** Forged materials of the $\alpha+\beta$ titanium alloy include those ($\alpha+\beta$ forged materials) obtained by $\alpha+\beta$ forging in which the titanium alloy is forged by heating it to a temperature range ($\alpha+\beta$ two phase region) less than T_β so as to prevent it from reaching T_β or higher and those (β forged materials) obtained by β forging in which the alloy material is forged by heating the alloy material to a temperature range (β single phase region) of T_β or higher. It is known that the $\alpha+\beta$ forged materials and the β forged materials are completely different in the microstructure of a material to be formed and therefore, different in material properties.

25 **[0005]** Forged titanium alloy materials manufactured by the former $\alpha+\beta$ forging have a granular α microstructure. FIG. 5 shows it. In FIG. 5, the microstructure shown in white is an α phase.

30 **[0006]** Forged titanium alloy materials manufactured by the latter β forging, on the other hand, have an acicular α phase microstructure. Described specifically, the microstructure is formed as follows. First, the titanium alloy has a β single phase in a temperature range of T_β or higher and an equiaxed β phase (β grains) is formed. The β grains thus formed are crushed into flattered grains by forging. When the resulting flattered grains are cooled to a temperature range less than T_β and are retained in this temperature, an α phase in film form is precipitated along the crystal grain boundary of the β grains, which is followed by the precipitation of the α phase in acicular form in the crystal grains of the β grains. FIG. 6 shows precipitation of the α phase in acicular form. The α phase is shown in white in FIG. 6.

35 **[0007]** It is to be noted that β forging includes forging which is completed in a β single phase region, forging which is continued after a temperature drop to a range outside the β single phase ($\alpha+\beta$ two phase region), and forging which is started after a temperature drops to the $\alpha+\beta$ two phase region.

40 **[0008]** The β forged materials may change in the morphology or diameter of the α phase on the crystal grain boundary of prior β grains (the above-mentioned equiaxed β grains) or length or diameter of the acicular α phase in the grains, depending on the forging conditions or subsequent cooling conditions. Further, some may have no film-like α phase on the grain boundary.

45 **[0009]** In general, with regard to fracture toughness of $\alpha+\beta$ forged titanium alloy materials, β forged materials are superior to $\alpha+\beta$ forged materials, while with regard to a fatigue strength property, $\alpha+\beta$ forged materials are superior to β forged materials.

50 **[0010]** Components (engine components) used for engines of aircraft are required to have a high fatigue strength property. In order to satisfy such a request, $\alpha+\beta$ forged titanium alloy materials are frequently used for engine components.

55 **[0011]** In addition, engine components are required to have high reliability. In order to satisfy such a request, ultrasonic inspection of engine components is performed to check for defects. In this ultrasonic inspection, defects inside an inspection object are checked by causing a probe to transmit (send) ultrasonic waves to make them incident into the inspection object from the surface thereof and to receive waves reflected from the defects such as flaws.

60 **[0012]** Since the $\alpha+\beta$ forged titanium alloy materials have both an α phase and a β phase irrespective of whether they are $\alpha+\beta$ forged materials or β forged materials, they have a high noise due to the microstructure of the material. Such a high noise reduces defect inspection accuracy or causes misunderstanding of a noise derived from the microstructure of the material as a defect. For engine components and the like made of an $\alpha+\beta$ titanium alloy, reduction in noise at the time of ultrasonic inspection and improvement in ultrasonic flow inspection property are demanded.

65 **[0013]** It is known that in the $\alpha+\beta$ forged titanium alloy materials, as the α phase (grain boundary α phase) precipitated along the prior β grain boundary is more continuous, fatigue cracks are likely to occur or grow. It is said to be recommendable to cut the continuity of the grain boundary α phase in order to prevent occurrence or growth of such fatigue cracks. Increase in strain during forging is effective for cutting the continuity of the grain boundary α phase, but it simultaneously deteriorates the ultrasonic inspectability.

70 **[0014]** To satisfy the demand for improvement of ultrasonic inspectability, for example, Patent Literature 1 describes a method of manufacturing an $\alpha+\beta$ titanium alloy plate, comprising cooling an $\alpha+\beta$ titanium alloy slab obtained by crude

forging under heated state or slabbing at a cooling rate of 0.5°C/s or more from the β single phase region, heating the resulting slab to an $\alpha+\beta$ temperature range of from a [β -transus temperature] to a

[0015] [β -transus temperature - 200°C] to subject it to hot forging at a height ratio of 10% or more, and then successively performing hot rolling in the $\alpha+\beta$ temperature range and heat treatment in the $\alpha+\beta$ temperature range.

[0016] According to Patent Literature 1, such an invention makes it possible to form an equiaxed and minute α grain microstructure and thereby reduce ultrasonic noise to an extent not to disturb inspection of minute defects.

[0017] As described above, engine components of aircraft need a high fatigue strength property. To satisfy such a request, Patent Literature 2 describes a near- β titanium alloy excellent in low cycle fatigue property. This near β titanium alloy is characterized in that a Mo equivalent determined from the following formula: $[\text{Mo}]+[\text{Ta}]/5+[\text{Nb}]/3.5+[\text{W}]/2.5+[\text{V}]/1.5+1.25[\text{Cr}]+1.25[\text{Ni}]+1.7[\text{Mn}]+1.7[\text{Co}]+2.5[\text{Fe}]$ is from 5 to 10%; an average aspect ratio of a primary α phase in its metal microstructure is from 40 to 52%; the primary α phase has an average aspect ratio of from 3.3 to 5.0; and an average maximum long diameter is from 25 to 40 μm (in the above formula, however, numerals in the parentheses are the contents (mass%) of elements, respectively).

[0018] According to Patent Literature 2, by adjusting the average area fraction of the acicular primary α phase having an average aspect ratio of from 3.3 to 5.0 to from 40 to 52%, a near β titanium alloy excellent in tensile strength and elongation and having a long low cycle fatigue life and therefore having an excellent low cycle fatigue property can be obtained.

Citation List

Patent Literature

[0019]

Patent Literature 1: Japanese Patent No. 2988269

Patent Literature 2: Japanese Patent Laid-Open No. 2011-102414

Summary of Invention

Technical Problem

[0020] The invention described in Patent Literature 1 can improve the ultrasonic inspectability and the invention described in Patent Literature 2 can improve the fatigue strength property. The ultrasonic inspectability and fatigue strength property are however in a trade-off relationship and it is extremely difficult to strike a balance between them at a high level. In the industry, there is an eager demand for the development of forged titanium alloy materials that have struck a balance between fatigue strength property and ultrasonic inspectability at a high level in order to realize engine components with higher reliability.

[0021] In view of such a situation, the present invention has been made. An object of the invention is to provide a forged titanium alloy material having an improved fatigue strength property without deteriorating ultrasonic inspectability and a method of manufacturing the forged material.

Solution to Problem

[0022] The forged titanium alloy material of the present invention provided to overcome the above-mentioned problem is a β forged titanium alloy material. It is characterized in that an area fraction of non-flattered grains of prior β -grains having 80% or more its grain boundaries decorated alpha phase, which ratio is 3 or less and which diameter in a forging direction is 20 μm or more, is less than 10%; an area fraction of flattered grains of prior β -grains having 80% or more its grain boundaries decorated alpha phase, which aspect ratio is greater than 3 and which diameter in the forging direction is 20 μm or more but not more than 700 μm , is 85% or more; and an average misorientation of an α -phase precipitated along the grain boundary of the flattered grains is 6° or more.

[0023] By adjusting the area fraction of non-flattered grains to less than 10%, deterioration in fatigue strength can be prevented and by adjusting the area fraction of flattered grains to 85% or more, high fracture toughness and fatigue strength can be achieved. By adjusting the average misorientation of an α phase precipitated along the grain boundary of flattered grains to 6° or more, continuity of the α phase is broken to prevent occurrence or growth of fatigue cracks. Using such a method prevents deterioration of ultrasonic inspectability. In other words, it prevents formation of ultrasonic noise.

[0024] The forged titanium alloy material of the present invention is made of preferably a titanium alloy having a Mo equivalent $[\text{Mo}]_{\text{eq}}$ represented by the following formula (1) more than 2.7 but less than 15.

$$[\text{Mo}]_{\text{eq}} = [\text{Mo}] + [\text{Ta}]/5 + [\text{Nb}]/3.6 + [\text{W}]/2.5 + [\text{V}]/1.5 + 1.25[\text{Cr}] + 1.25[\text{Ni}] + 1.7[\text{Mn}] + 1.7[\text{Co}] + 2.5[\text{Fe}] \dots (1)$$

[0025] In the above formula, each element symbol in the parentheses on the right side represents a content (mass%) of each element in the titanium alloy.

[0026] When such a titanium alloy is used, flattered grains, which are prior β -grains, are more effective for improving the fracture toughness and fatigue strength.

[0027] The forged titanium alloy material of the present invention has preferably a thickness, at the thinnest portion, of 30 mm or more and an average thickness of 70 mm or more.

[0028] Since the thickness is defined as described above, a large forged material can be provided.

[0029] The method of manufacturing a forged titanium alloy material according to the present invention is a method of manufacturing the above-mentioned forged titanium alloy material by β forging. The β forging includes a heating step in which a titanium alloy material is heated to $(T_{\beta} + 10)^{\circ}\text{C}$ or more wherein T_{β} represents β -transus temperature and kept until a β crystal grain diameter falls within a range of 300 μm or greater but not greater than 1000 μm ; a forging step in which the titanium alloy material is forged at forging temperature, T_F [$^{\circ}\text{C}$] which satisfies the following formula (2) and under conditions in which the forging temperature T_F [$^{\circ}\text{C}$] satisfy the formulas (3) and (4), respectively to produce a forged titanium alloy material; and a cooling step in which the forged titanium alloy material obtained by the above-mentioned forging is cooled to temperature lower than $(T_{\beta} - 150)^{\circ}\text{C}$;

$$T_{\beta} - 150 \leq T_F \leq T_{\beta} + 100 \dots (2)$$

$$\text{Ln}(S_R) + 22800/(T_F + 273) \cdot 18.6 \leq 0 \dots (3)$$

$$\text{Ln}(S_R) + 22800/(T_F + 273) \cdot 13.2 \geq 0 \dots (4).$$

[0030] In the formulas (2) to (4), T_{β} represents the β -transus temperature [$^{\circ}\text{C}$], T_F represents the forging temperature [$^{\circ}\text{C}$], and S_R represents a strain rate [s^{-1}] upon forging.

[0031] Control of the forging temperature and the strain rate to fall within a predetermined range enables α sub-grain microstructure to grow upon forging. As a result, an α phase microstructure precipitated in the crystal grain boundary of flattered grains can be obtained in a desired form and a forged titanium alloy material excellent in fatigue strength and ultrasonic inspectability can be manufactured.

[0032] The method of manufacturing a forged titanium alloy material according to the present invention includes a billet forging step in which an ingot made of a titanium alloy is forged into a corresponding billet. The method preferably has, between the billet forging step and the heating step, an $\alpha + \beta$ forging step in which the billet obtained from the titanium alloy is forged in an $\alpha + \beta$ two phase region.

[0033] By using such a manufacturing method, the P crystal grain diameter can be stably controlled to fall within a desired range in the heating step.

[0034] In the method of manufacturing a forged titanium alloy material according to the present invention, the billet obtained from the titanium alloy may have an acicular microstructure.

[0035] Even when a titanium alloy has an acicular microstructure, the β crystal grain diameter can be stably controlled to fall within a desired range in the heating step by carrying out the above-mentioned billet forging step.

[0036] The method of manufacturing a forged titanium alloy material according to the present invention preferably includes a ultrasonic inspection step in which after the cooling step, ultrasonic waves are irradiated in a direction parallel to a direction of the largest reduction in the β forging to inspect flaws of the forged titanium alloy material.

[0037] The method including such a step can provide a forged titanium alloy material free of defects and the like.

[0038] In the method of manufacturing a forged titanium alloy material according to the present invention, the forged titanium alloy material is preferably a material to be used in the manufacture of engine components of aircraft.

[0039] The method using such a material can realize engines for aircraft using engine components free of defects and the like. Advantageous Effects of Invention

[0040] The forged titanium alloy material according to the present invention can have an improved fatigue strength property without deteriorating an ultrasonic inspectability.

[0041] The method of manufacturing a forged titanium alloy material according to the present invention can manufacture a forged titanium alloy material having an improved fatigue strength property without deteriorating an ultrasonic inspect-

ability.

Brief Description of Drawings

[0042]

[FIG. 1] FIG. 1 is a schematic cross-sectional view for describing the metal microstructure of a forged titanium alloy material according to one embodiment of the present invention.

[FIG. 2] FIG. 2 is a flow chart for describing a method of manufacturing the forged titanium alloy material according to the one embodiment of the present invention.

[FIG. 3] FIG. 3 is an SEM/EBSD image of Test specimen No. 2 in which the scale bar indicates 10 μm .

[FIG. 4] FIG. 4 is an SEM/EBSD image of Test specimen No. 3 in which the scale bar indicates 10 μm .

[FIG. 5] FIG. 5 is an electron micrograph of a conventional $\alpha+\beta$ forged material, in which the scale bar indicates 100 μm .

[FIG. 6] FIG. 6 is an electron micrograph of a conventional β forged material, in which the scale bar indicates 100 μm .

Description of Embodiments

[0043] Modes (embodiments) for providing and carrying out the forged titanium alloy material and manufacturing method thereof according to the present invention, respectively, will next be described in detail with reference to drawings as needed. First, one embodiment of the forged titanium alloy material according to the present invention will be described.

[Forged titanium alloy material]

[0044] The forged titanium alloy material according to the present embodiment is a β forged titanium alloy material. Described specifically, the forged titanium alloy material according to the present embodiment is composed of an $\alpha+\beta$ titanium alloy (which will hereinafter be called "titanium alloy" simply) and similar to conventional β forged materials, it has an α phase (grain boundary α phase (refer to FIG. 3)) precipitated in the crystal grain boundary of prior β grains and an α phase (refer to FIG. 3) precipitated in acicular form in the prior β grains.

[0045] In β forging, when a titanium alloy material is heated and retained in a temperature range (β single phase region) of a β -transus temperature (T_β) or higher and therefore has a β single phase state, equiaxed β phase crystal grains (β crystal grains, β grains) having an aspect ratio close to 1 are formed and grow. As shown in FIG. 1, the β crystal grains thus obtained are crushed by forging, spread perpendicularly in a forging direction (press direction) to become flat, and have a pancake shape. The resulting β grains (prior β grains) (refer to Reference numeral 2 in FIG. 1). These grains will hereinafter be called "prior β grains 2", if necessary) are stacked one after another to have a microstructure having a polycrystalline structure. When by cooling after forging, the temperature drops to a sufficiently low temperature range ($\alpha+\beta$ two phase region) lower than T_β , an α phase is precipitated on the grain boundary 3 of the prior β grains 2 or in the grains. In the β forged material, therefore, the prior β grains 2 tend to have the smallest diameter in the forging direction (refer to Diameter L1 in FIG. 1). In the forged titanium alloy material 1, equiaxed β grains are formed newly and grow when after forging, a cooling rate is slow and retention time within the β single phase region is long.

[0046] Area fractions of non-flattered grains and flattered grains in the forged titanium alloy material according to the present embodiment are less than 10% and 85% or more, respectively. At the same time, an average misorientation of the crystal orientation of an α phase precipitated in the crystal grain boundary of the flattered grains (which may also be called "grain boundary α phase of flattered grains", hereinafter) is 6° or more.

[0047] The term "non-flattered grains" as used herein means prior β grains having an aspect ratio of 3 or less, a forging-direction diameter of 20 μm or more, and the proportion of an α phase in the crystal grain boundary of 80% or more.

[0048] The term "flattered grains" as used herein means prior β grains having an aspect ratio exceeding 3, a forging-direction diameter of 20 μm or more but not more than 700 μm , and the proportion of the α phase in the crystal grain boundary of 80% or more.

[0049] In the definition of non-flattered grains and flattered grains, the proportion of the α phase in the crystal grain boundary is specified to 80% or more in order to specify the area fraction while omitting sub-grains and paying attention only to recrystallized β grains. The grain boundary of the recrystallized β grains is occupied by a linear grain boundary α phase.

[0050] The non-flattered grains and flattered grains containing too many fine crystal grains disturb the measurement so that the minimum size is specified.

[0051] The upper limit of the diameter of the flattered grains is specified in order to omit flattered grains many of which have a diameter greater than the upper limit. In the measurement of the average misorientation of the crystal orientation, which will be described later, the flattered grains must be limited to a certain size. When most of the flattered grains are

occupied by large flattered grains, the grain boundary is reduced, making it impossible to achieve a desired effect even if an average misorientation of the crystal orientation of a very slight grain boundary α phase is specified. An upper limit of the diameter of the flattered grains is therefore specified in order to omit such a titanium alloy. Another reason for placing an upper limit in the diameter of the flattered grains is that diameters exceeding the upper limit deteriorate the fatigue strength.

[0052] The term "aspect ratio" means, in prior β grains 2, a ratio of L2 to L1, in which L1 means the diameter of crystal grains in a forging direction and L2 means the diameter of crystal grains in a direction perpendicular to the forging direction. With reference to FIG. 1, the aspect ratio is a ratio of horizontal-direction Diameter L2 to perpendicular-direction Diameter L1.

(Area fraction of non-flattered grains is less than 10%)

[0053] A continuous grain boundary α phase is easily formed in the grain boundary of non-flattered grains so that fatigue strength deteriorates. When the area fraction of non-flattered grains is less than 10%, a formation amount of the continuous grain boundary α phase decreases and deterioration in fatigue strength can be prevented. When the area fraction of non-flattered grains becomes 10% or more, on the other hand, a formation amount of the continuous grain boundary α phase increases and fatigue strength deteriorates.

[0054] The area fraction of non-flattered grains is preferably less than 8%, more preferably less than 6%.

(Area fraction of flattered grains is 85% or more)

[0055] Similar to conventional β forged materials, the forged titanium alloy material according to the present embodiment can have high fracture toughness and fatigue strength because of a polycrystal structure of β crystal grains (prior β grains) in flattered form. In the forged titanium alloy material, prior β grains equiaxed and having a small aspect ratio (close to 1) before forging have an increased aspect ratio (become more flat) with an increase in strain applied upon forging and greatly contribute to improvement in fatigue strength. The fatigue strength can be improved certainly by adjusting the area fraction of flattered grains to 85% or more. On the other hand, sufficient fatigue strength cannot be attained when the area fraction of flattered grains becomes below 85%. The area fraction of the flattered grains is preferably set at 90% or greater.

(Average misorientation of the crystal orientation of the grain boundary α phase of flattered grains is 6° or more)

[0056] A small average misorientation of the crystal orientation of the crystal boundary α phase of flattered grains means that the grain boundary α phase having almost the same crystal orientation is present along the prior β grain boundary over a long distance, in short, the grain boundary α phase is continuous. A small average misorientation may therefore cause deterioration in fatigue strength. Average misorientation of 6° or more do not cause deterioration in fatigue strength, while those less than 6° may cause considerable deterioration in fatigue strength. The average misorientation is preferably 10° or more, more preferably 15° or more, still more preferably 25° or more. No particular limitation is imposed on the upper limit of the average misorientation, but it does not exceed 90° crystallographically and 70° is a practical upper limit.

(Measurement method, etc.)

[0057] The aspect ratio and diameter of the prior β grains, the area fraction of the non-flattered β grains (non-flattered grains), and the area fraction of the flattered β grains (flattered grains), each of the forged titanium alloy material according to the present invention can be determined from one or more visual fields in a cross-section parallel to the forging direction of the forged titanium alloy material. Described specifically, the aspect ratio and the like of the prior β grains can be determined by cutting the forged titanium alloy material into pieces along a plane parallel to the forging direction (refer to FIG. 1), subjecting the cross-section of each of the pieces to polish (mechanical polish, electrolytic polish) finishing, corroding the resulting pieces, selecting one or more visual fields of about one to several mm square from the cross-section, observing the microstructure on the cross section by an optical microscope, and calculating an average value.

[0058] The lengths (diameter) of the prior β grains in the forging direction and a direction perpendicular thereto in the cross-section are measured, respectively, and an aspect ratio is calculated. Then, based on the resulting diameters and aspect ratio, non-flattered grains can be identified.

[0059] The average misorientation of the crystal orientation of the grain boundary α phase of the flattered grains of the forged titanium alloy material according to the present embodiment can be determined based on the measurement results, in a plurality of visual fields, of a cross-section parallel to the forging direction of the forged titanium alloy material.

[0060] Described specifically, the forged titanium alloy material is cut along a plane parallel to the forging direction (refer to FIG. 1). After mechanically polishing the cross-section and finishing it by electrolytic polishing, a visual field of, for example, about 100 μm square is selected from the cross-section so that the grain boundary α phase of the flattered grains comes to the vicinity of the center and the crystal orientation of the microstructure on the cross section is measured by SEM/EBSD (Scanning Electron Microscope/Electron Back Scatter Diffraction) method.

[0061] With regards to the measurement result thus obtained, a straight line (dashed line in FIG. 1) parallel to the forging direction is drawn at intervals of 10 μm and the crystal orientation difference between grain boundary α phases adjacent to each other, among the grain boundary α phases intersecting these straight lines (points of intersection: P1, P2 ...P10), is measured. The orientation differences between all the points of intersection adjacent to each other can be averaged to determine an average misorientation.

[0062] When an average misorientation of the crystal orientation of the grain boundary α phase of the flattered grains is determined, a visual field in which the prior β grain boundary does not branch (a plurality of prior β grains is absent in a direction perpendicular to the forging direction) is selected desirably. If it includes a plurality of prior β grains, the crystal orientation difference is determined as follows. Described specifically, when one of them is a non-flattered grain, the crystal orientation difference is calculated along the grain boundary of the flattered grain. When the flattered grains are present on both sides with the prior β grain boundary therebetween, the crystal orientation difference of the grain boundary α phase of both branches is calculated.

[0063] The thickness of the grain boundary α phase precipitated in the grain boundary of the flattered grains and the thickness of the grain boundary α phase precipitated in the grain boundary of the non-flattered grains are each preferably 3 μm or less on the average of the entirety of the β forged material. When cooling is conducted under inappropriate conditions after β forging, the thickness of the grain boundary α phase may exceed 3 μm , leading to deterioration in fatigue strength. When the thickness of the grain boundary α phase is 3 μm or less on an average in a certain visual field, it is presumed to be 3 μm on the average of the entirety of the β forged material.

[0064] An $\alpha+\beta$ titanium alloy can be used for the forged titanium alloy material of the present embodiment described above, but it has preferably a Mo equivalent [Mo]eq, which is represented by the following formula (1), exceeding 2.7 but less than 15.

(Mo equivalent [Mo]eq: exceeding 2.7 but less than 15)

[0065] With an increase in the Mo equivalent of a titanium alloy, the volume content of the α phase decreases and the shape of the prior β grain boundary has an enhanced influence. As a result, the above-mentioned effect of the flattered grains of the prior β grains for improving fracture toughness and fatigue strength becomes more effective. The Mo equivalent [Mo]eq represented by the following formula (1) is preferably 4.5 or more, more preferably 6.5 or more. On the other hand, a titanium alloy is likely to cause segregation of an alloy element with an increase in the Mo equivalent [Mo]eq represented by the following formula (1), which may lead to variation in microstructure. The Mo equivalent [Mo]eq represented by the following formula (1) is less than 15. The Mo equivalent [Mo]eq represented by the following formula (1) is preferably 12 or less.

$$[\text{Mo}]_{\text{eq}} = [\text{Mo}] + [\text{Ta}]/5 + [\text{Nb}]/3.6 + [\text{W}]/2.5 + [\text{V}]/1.5 + 1.25[\text{Cr}] + 1.25[\text{Ni}] + 1.7[\text{Mn}] + 1.7[\text{Co}] + 2.5[\text{Fe}] \dots (1)$$

[0066] In the above formula, each element symbol in the parentheses on the right side of the formula (1) means a content (mass%) of each element contained in the titanium alloy.

[0067] Specific examples of such a titanium alloy include titanium alloys specified by AMS4981 or AMS4995.

[0068] The titanium alloy (Ti-6Al-2Sn-4Zr-6Mo alloy, Ti-6246 alloy) defined by AMS4981 contains from 5.50 to 6.50 mass% Al, from 1.75 to 2.25 mass% Sn, from 3.50 to 4.50 mass% Zr, from 5.50 to 6.50 mass% Mo and the balance Ti and inevitable impurities. The Mo equivalent calculated from an average of each element is 6.0. The titanium alloy roughly contains, as the above-mentioned inevitable impurities, 0.04 mass% N, 0.08 mass% C, 0.015 mass% H, 0.15 mass% Fe, and 0.15 mass% O.

[0069] The titanium alloy (Ti-5Al-2Sn-2Zr-4Cr-4Mo alloy, Ti-17 alloy) defined by AMS4995 contains from 4.5 to 5.5 mass% Al, from 1.5 to 2.5 mass% Sn, from 1.5 to 2.5 mass% Zr, from 3.5 to 4.5 mass% Cr, from 3.5 to 4.5 mass% Mo, and from 0.08 to 0.12 mass% O, and the balance Ti and inevitable impurities. The Mo equivalent calculated from an average of each element is 9.5. The titanium alloy roughly contains, as the above-mentioned inevitable impurities, 0.03 mass% Fe, 0.05 mass% C, 0.04 mass% N, and 0.0125 mass% H.

[0070] The forged titanium alloy material of the present embodiment is suited as a material to be used for the manufacture of engine components of aircraft. It is particularly suited as a material requiring inspection of defects therein by ultrasonic inspection. For example, it can be used for a large forged titanium alloy material (large forged material) such

as disks and shafts to be used for engine components of aircraft. The term "large forged material" means a forged material having a thickness at the thinnest portion thereof 30 mm or more and an average thickness of 70 mm or more. Although the upper limit of the thickness of the large forged material is not particularly limited, it is, for example, 350 mm.

5 (Control of area fraction of non-flattered or flattered grains and average misorientation of crystal orientation of grain boundary α phase of flattered grains)

[0071] Control of the above-mentioned area fraction of non-flattered grains, area fraction of flattered grains, and average misorientation of crystal orientation of grain boundary α phase of flattered grains can be carried out by a method of manufacturing a forged titanium alloy material which will be described later. Details of it will be described later.

10 **[0072]** As described above, the forged titanium alloy material according to the present embodiment can have an improved fatigue strength property without deteriorating an ultrasonic inspectability. The forged titanium alloy material according to the present embodiment enables inspection of defects with high accuracy by ultrasonic inspection so that it improves reliability of products such as engine components of aircraft. In addition, excellent fatigue strength of the material enables reduction in thickness and weight of engine components. Reduction in weight leads to improvement in fuel efficiency of aircraft and the like. Further, excellent fatigue strength of the forged material enables operation of an engine under severer conditions.

[Manufacturing method of forged titanium alloy material]

20 **[0073]** Next, referring to FIG. 2, one embodiment of the manufacturing method of a forged titanium alloy material according to the present invention will next be described.

[0074] In the manufacturing method of a forged titanium alloy material according to the present embodiment, a forged titanium alloy material (product) in a desired shape is manufactured by forging an ingot composed of an $\alpha+\beta$ titanium alloy, more preferably, an ingot composed of a titanium alloy defined by AMS4981 or AMS4995 into a billet under known conditions (billet forging step S11), machining the billet as needed, and then carrying out β forging under specific conditions which will be described later specifically.

(Billet forging step)

30 **[0075]** The billet forging step S11 is performed, for example, in the following order: β forging, $\alpha+\beta$ forging, β heat treatment, stress relief heat treatment, $\alpha+\beta$ forging, and heat treatment.

[0076] In $\alpha+\beta$ forging, the ingot is heated to a temperature range lower than a β -transus temperature (which will be abbreviated as T_β as needed) by from about 10 to 200°C and in β forging, the ingot is heated to a temperature range higher than a β -transus temperature by from about 10 to 150°C and forging is performed at a predetermined forging ratio (a ratio of an area after forging to an area before forging in a cross-section perpendicular to a stretching direction, for example, 1.5), followed by cooling to room temperature.

[0077] Whether $\alpha+\beta$ forging or β forging is used as forging in the billet forging step S11 is determined, depending on the properties which a product is required to have. The frequency of forging may also be determined, depending on the desired diameter of the billet. Heat treatment to be performed twice may be performed as needed. For example, the second heat treatment is performed to facilitate subsequent machining and to facilitate ultrasonic inspectability.

[0078] By machining of the billet obtained by the billet forging step S11, an oxide film, wrinkle, and burr are removed from the surface and the billet can have controlled surface roughness. This machining facilitates subsequent forging (β forging in the manufacture of a forged titanium alloy material).

45 **[0079]** In order to manufacture the forged titanium alloy material according to the present invention, a titanium alloy billet is subjected to β forging in the following manner. Prior to β forging, the titanium alloy billet may be subjected to preform forging in the $\alpha+\beta$ two phase region to finish it into a desired shape.

[0080] A billet ($\alpha+\beta$ billet) finished by $\alpha+\beta$ forging has conventionally been used as described above. No particular attention has been paid to whether the $\alpha+\beta$ billet is subjected to preform forging or not.

50 **[0081]** As forged titanium alloy materials have recently been required to have advanced properties, billets are also required to have high level of properties. Particularly in ultrasonic inspection, inspection of defects smaller than before is required so that using a billet (β billet) having a β microstructure as the final microstructure by carrying out heat treatment in the range after the $\alpha+\beta$ forging for final finishing or by increasing the temperature range of the final finishing from the conventional $\alpha+\beta$ range to the β range is under investigation. The β microstructure, different from the $\alpha+\beta$ microstructure, is rough and has an α phase in acicular form so that using the β billet manufactured by a method similar to a conventional method sometimes prevents exhibition of desirable properties.

[0082] When the β billet is used, an $\alpha+\beta$ forging step S12 (refer to FIG. 2) for forging the β billet (titanium alloy) in the $\alpha+\beta$ two phase region is preferably provided as a step before β forging, more specifically, between the billet forging step

S11 and a heating step S1 which will be described later. When such forging is performed, a strain of 0.05 or more, more preferably a strain of 0.10 or more is applied to the position in the billet which will be a product. This makes it possible to stably control the β crystal grain diameter to fall within a desired range in the subsequent heating step S1.

[0083] In the method of manufacturing a forged titanium alloy material according to the present embodiment, β forging is performed under the conditions described below by using the billet manufactured by the known billet forging step S11 as described above. The β forging includes, as shown in FIG. 2, the heating step S1, a forging step S2, and a cooling step S3. These steps are carried out in order of mention, but another step may be included before the heating step S1, between these steps, and after the cooling step S3. The heating step S1, the forging step S2, and the cooling step S3 are preferably performed successively.

[0084] For example, a preform forging step (not shown) in which the above-mentioned preform forging is performed can be mentioned as the another step to be performed prior to the heating step S1.

[0085] The step to be performed between the heating step S1 and the forging step S2 is, for example, an air cooling step (not shown) in which the billet heated excessively in the heating step S1 is allowed to cool to reduce the billet temperature to a predetermined temperature or a lubricant application step (not shown) in which, upon forging, a lubricant is applied to the surface of the billet if necessary.

[0086] The step to be performed between the forging step S2 and the cooling step S3 is, for example, a retention step (not shown) in which the forged titanium alloy material after forging is retained under predetermined conditions for refining.

[0087] The another step to be performed after the cooling step S3 is, for example, a refining heat treatment step (not shown) or a machining step (not shown) which will be described later. An ultrasonic inspection step S4, which will be described later, also follows the cooling step.

[0088] In the following description, a material before β forging, in the manufacture of a forged titanium alloy material, is called "titanium alloy raw material" and the description will be made using, as an example, the case where the billet manufactured in the billet forging step S11 is used as the titanium alloy raw material. In addition, the heating step S1, the forging step S2, and the cooling step S3 are performed successively in the following example.

(Heating step)

[0089] The heating step S1 is a step of heating the billet to $(T_{\beta}+10)^{\circ}\text{C}$ or higher and retain it until the β crystal grain diameter (average grain diameter) falls within a range of $300\text{ }\mu\text{m}$ or more but not more than $1000\text{ }\mu\text{m}$. In Claims, the billet in this step is called "titanium alloy".

[0090] Heating of the billet to $(T_{\beta}+10)^{\circ}\text{C}$ or higher before forging is, similar to conventional β forging, performed to heat the billet to a β single phase region to form a β phase single phase.

[0091] The term " β single phase region" means a temperature range of a β -transus temperature (T_{β}) or higher and the term " T_{β} " means the lowest temperature at which the entirety (100%) of the titanium alloy material has a β phase. The T_{β} varies depending on the composition of a titanium alloy constituting the titanium alloy raw material. For example, a titanium alloy (Ti-6246 alloy) defined by AMS4981 has a T_{β} of about 960°C and a titanium alloy (Ti-17 alloy) defined by AMS4995 has a T_{β} of about 890°C .

[0092] With an increase in the temperature of the billet in the β single phase region, crystal grains of the β phase show a higher growth rate, which makes it difficult to control the crystal grain diameter. At the temperature of the billet exceeding $(T_{\beta}+250)^{\circ}\text{C}$, a thick oxide scale is likely to appear on the surface and it needs to be removed after forging. The heating temperature of the billet in the heating step S1 is therefore preferably $(T_{\beta}+250)^{\circ}\text{C}$ or lower.

[0093] After heating the billet to the β single phase region, it was maintained within the temperature range for a predetermined time before forging to grow the β crystal grains to an adequate size, more specifically, to grow the β crystal grains to a diameter size of $300\text{ }\mu\text{m}$ or more but not more than $1000\text{ }\mu\text{m}$. The retention time differs depending on the kind of the titanium alloy or retention temperature of the billet, but the billet may be retained, for example, at 960°C for from about 30 to 600 minutes. Although after formation of a desired β crystal grain microstructure, the temperature of the billet may drop to less than $(T_{\beta}+10)^{\circ}\text{C}$ before forging is started, it is preferred to retain it within a temperature range of $(T_{\beta}-150)^{\circ}\text{C}$ or higher until completion of forging as will be described later.

[0094] When the grain diameter of the β crystal grains falls within the above-mentioned range, a desired fatigue strength property can be achieved and this facilitates manufacture. On the other hand, grain diameters of the β crystal grains less than $300\text{ }\mu\text{m}$ make difficult the manufacture, while grain diameters of the β crystal grains exceeding $1000\text{ }\mu\text{m}$ are likely to deteriorate the fatigue strength property. The grain diameter of the β crystal grains is adjusted preferably to $800\text{ }\mu\text{m}$ or less.

(Forging step)

[0095] The forging step S2 is a step of forging the billet under conditions under which a forging temperature T_F [$^{\circ}\text{C}$] satisfies the following formula (2) and respective left side values of the following formulas (3) and (4) indicating the

relationship with the forging temperature T_F satisfy the formulas (3) and (4), respectively, to obtain a forged titanium alloy material.

$$T_\beta - 150 \leq T_F \leq T_\beta + 100 \dots (2)$$

$$\ln(S_R) + 22800/(T_F + 273) - 18.6 \leq 0 \dots (3)$$

$$\ln(S_R) + 22800/(T_F + 273) - 13.2 \geq 0 \dots (4)$$

In the formulas (2) to (4), T_β represents a β -transus temperature [$^{\circ}\text{C}$], T_F represents a forging temperature [$^{\circ}\text{C}$], and S_R represents a strain rate [s^{-1}] upon forging. The term "Ln" as used herein means a natural logarithm.

[0096] The formula (3) is preferably $\ln(S_R) + 22800/(T_F + 273) - 17.1 \leq 0$.

[0097] In the forging step S2, forging within a temperature range of $T_\beta - 150 \leq T_F \leq T_\beta + 100^{\circ}\text{C}$ of the formula (2) makes it difficult to precipitate an α phase on the grain boundary of the β crystal grains or therein. This makes it difficult to deteriorate the fracture toughness and fatigue strength.

[0098] At forging temperatures T_F less than $(T_\beta - 150)^{\circ}\text{C}$, precipitation of α phase starts on the grain boundary of β crystal grains and therein. When such an α phase is formed before completion of forging, the fracture toughness may deteriorate. The forging temperature T_F (more specifically, the temperature at the time of completion of billet forging) is therefore set at $(T_\beta - 150)^{\circ}\text{C}$ or higher. The forging temperature T_F is preferably $(T_\beta - 110)^{\circ}\text{C}$ or higher. A die to be used for forging is heated to preferably 400° or higher, more preferably the forging temperature T_F (temperature of the billet). By using the heated die as described above, it is possible to prevent the surface of the billet to be forged from being cooled too early compared with the inside of the billet and to complete forging while retaining the temperature in the vicinity of the surface at $(T_\beta - 150)^{\circ}\text{C}$ or higher. Only a product portion of the forged titanium alloy material needs to be retained in the temperature range of $(T_\beta - 150)^{\circ}\text{C}$ or higher until completion of forging. The temperature of an excess thickness portion (except the product portion) such as surface layer to be removed after forging (after cooling) is not limited to it.

[0099] When the temperature upon forging is excessively high, on the other hand, it takes time, after completion of forging, to cool the forged material to a temperature lower than $(T_\beta - 150)^{\circ}\text{C}$ in the cooling step S3 which will be described later. If it takes time, growth of new β grains or precipitation of a wide (thick) α phase on the prior β grain boundary may occur and the forged titanium alloy material may have deteriorated fatigue strength. The forging temperature T_F (more specifically, temperature from the start of forging to completion thereof) is set at $(T_\beta + 100)^{\circ}\text{C}$ or less. The forging temperature T_F is more preferably $(T_\beta + 50)^{\circ}\text{C}$ or lower.

[0100] In the forging step S2, a strain rate upon forging is controlled precisely to grow a sub-grain microstructure upon forging. The strain rate upon forging can be controlled by a move speed of a forging die, that is, a speed during which the forging die brought into contact with a material to be forged processes the material. By growing a sub-grain microstructure as described above, a desired grain boundary α phase microstructure can be formed and a forged titanium alloy material having an improved fatigue strength property can be manufactured without deteriorating the ultrasonic inspectability.

[0101] In the forging step S2, the average misorientation of the grain boundary α phase is increased to obtain desired fatigue strength by forging under the condition under which the left side value of the formula (3) satisfies the formula (3). When the left side value of the formula (3) does not satisfy the formula (3), the β grain boundary after processing extends linearly and the average misorientation of the grain boundary α phase is small so that desired fatigue strength cannot be obtained.

[0102] Further, in the forging step S2, formation of non-flattered grains during processing is suppressed to prevent deterioration in fatigue strength by carrying out forging under the condition under which the left side value of the formula (4) satisfies the formula (4). When the left side value of the formula (4) does not satisfy the formula (4), non-flattered grains are likely to be formed during processing, which facilitates deterioration in fatigue strength.

[0103] Here, derivation of the formulas (3) and (4) will be described. With regard to the formation of a hot forging microstructure, it is generally known that there is a following correlation between a forging temperature and a strain rate.

$$\ln(S_R) = A \cdot B/T_F$$

[0104] In the above formula, T_F represents a forging temperature [$^{\circ}\text{C}$], S_R represents a strain rate [s^{-1}], and A and B

represent coefficients determined by a test for specifying respective ranges of the forging temperature and the strain rate within which a desired hot forging microstructure is formed. The coefficient A and coefficient B are determined by forming, by way of trial, a plurality of β forged materials while changing the condition of the forging temperature and the strain rate in a test, evaluating their microstructures, and revealing an area of the forging temperature and the strain rate in which a predetermined microstructure is formed and indicating the boundary of the range.

[0105] The above formulas (3) and (4) can be derived respectively by substituting the coefficients thus determined by the test in the above formula, transposing the right side in the left side to obtain inequalities defining the respective conditions.

(Cooling step)

[0106] The cooling step S3 is a step of cooling the forged titanium alloy material obtained by forging to a temperature lower than $(T_{\beta}-150)^{\circ}\text{C}$. After completion of forging of the billet, it was cooled to a temperature lower than $(T_{\beta}-150)^{\circ}\text{C}$, that is, a temperature range outside the β single phase region ($\alpha+\beta$ two phase region), in the cooling step S3 to terminate the growth of new β grains. Further, this cooling step suppresses precipitation of a wide (thick) α phase on the prior β grain boundary and prevents deterioration of the fatigue strength of the forged titanium alloy material thus obtained. Cooling is therefore started as immediately as possible after completion of forging. More specifically, it is preferred to decrease the temperature to below $(T_{\beta}-150)^{\circ}\text{C}$ within 1200 seconds after completion of forging. A cooling rate after completion of forging is therefore preferably $10^{\circ}\text{C}/\text{min}$ or more, more preferably $20^{\circ}\text{C}/\text{min}$ or more. Although an upper limit of the cooling rate is not specified, $500^{\circ}\text{C}/\text{min}$ or less is practical. In order to lengthen the acicular α phase in the grains and improve the fracture toughness, the upper limit of the cooling rate is preferably $500^{\circ}\text{C}/\text{min}$ or less. Cooling may be carried out by a known method such as air cooling, air sending, water cooling, hot-water cooling, or oil cooling. The forged titanium alloy material is cooled to room temperature in the cooling step S3. However, no particular limitation is imposed on a cooling rate in a temperature range lower than $(T_{\beta}-150)^{\circ}\text{C}$ and it may be set depending on another property required.

[0107] The forged titanium alloy material thus manufactured can be provided as a product after subjecting to a refining heat treatment step and/or machining step as needed and then an ultrasonic inspection step S4 described later.

[0108] The refining heat treatment step is a step of carrying out refining heat treatment by solution treatment and aging treatment. The refining heat treatment step can be carried out in a known manner.

[0109] The machining step is a step of removing an oxide film or excessively thick portion by machining. This machining step can also be carried out in a known manner.

[0110] These steps can be performed, for example, by removing at least 1 mm from the surface of the forged titanium alloy material after completion of forging, planarizing it to a surface roughness of 6.3S or more, and then subjecting the planarized material to ultrasonic inspection. After re-machining if necessary, the resulting forged titanium alloy material can be provided as products, for example, engine components such as disks and shafts.

(Ultrasonic inspection step)

[0111] The ultrasonic inspection step S4 shown in FIG. 2 is a step of ultrasonically inspecting flaws of the forged titanium alloy material which has finished the refining heat treatment step and/or machining step (each not shown in FIG. 2) if necessary after the cooling step S3. In the ultrasonic inspection step S4, flaw inspection of the forged titanium alloy material is carried out by irradiating it with ultrasonic waves in a direction in which the amount of forging in β forging is the largest, that is, a direction parallel to the forging direction (refer to FIG. 1).

[0112] The direction in which the amount of forging in forging is the largest is a direction in which a size reduction ratio between before forging and after forging (that is, between the titanium alloy raw material and the forged titanium alloy material) is the largest. It is the forging direction shown in FIG. 1. The forging direction can also be presumed from the shape of the prior β grains in the microstructure after forging (forged titanium alloy material). The ultrasonic inspection direction is a traveling direction of transmission waves (a direction of transmission waves passing through the inside of the forged titanium alloy material). In the forged titanium alloy material, the direction in which the amount of forging in forging is largest tends to be a direction with the largest noise. The forged titanium alloy material according to the present embodiment however has sufficiently less noise even when inspected in such a direction and therefore can be inspected with high precision. The forged titanium alloy material according to the present embodiment can be inspected easily because an area of the surface perpendicular to a probe scanning direction is often wide.

[0113] Ultrasonic inspection can be performed in a known manner, but the following mode is recommended for achieving reliable ultrasonic flaw inspection. For example, a probe is selected from those having a probe diameter ranging from 5 mm to 30 mm and as ultrasonic waves (transmission waves), those having a frequency ranging from 1 to 20MHz are preferably used. The probe diameter and the frequency of ultrasonic waves are preferably 10 mm or more and 15 MHz or less, respectively. Water immersion method is preferably used for inspection because it has high inspection resolution

in the vicinity of the surface layer of defective forged products. Depending on the shape of the forged titanium alloy material of the present embodiment, not only flaw inspection in one direction but also inspection twice or more in total in varied directions is preferred. Further, transmission waves may be made incident from a direction contrary to the conventional direction, depending on the thickness (length in the traveling direction of the transmission waves) of the forged titanium alloy material.

[0114] The manufacturing method of a forged titanium alloy material as described above facilitates manufacture of the forged titanium alloy material of the present embodiment. In addition, the manufacturing method of a forged titanium alloy material according to the present embodiment can provide, as a product, a forged titanium alloy material that has finished high-precision ultrasonic inspection.

[Examples]

[0115] Next, examples by which the advantage of the present invention has been confirmed will be described.

[Example 1: Preparation of test specimen using $\alpha+\beta$ billet]

[0116] As a titanium alloy raw material, an $\alpha+\beta$ billet composed of a Ti-17 alloy (T_{β} : 890°C, Mo equivalent: the Mo equivalent calculated from an average of elements contained therein was 9.5) defined by AMS4995 was used. The press ratio upon forging was set at 67% and the thickness of a forged titanium alloy material (after β forging) was set at 45 mm.

(β Forging)

[0117] The billet was retained in a furnace at 850°C for 2 hours so as to have a uniform temperature distribution inside thereof. Then the resulting billet was heated to 980°C and retained at that temperature until the β grains before forging had an average particle diameter of from 400 to 600 μm . Then, the resulting billet was taken out from the furnace, air-cooled to the forging temperature shown in Nos. 1 to 9 in Table 1A, and forged using a die heated to the forging temperature in advance by low-frequency heating equipment. Forging was performed by using a pair of dies having a flat surface shape, moving the dies at a (average) strain rate described in Table 1A, and deforming in a direction of the axis of the billet (press direction). The underlined number in Table 1A shows that it does not satisfy the requirement of the present invention.

[0118] After completion of forging, the billet was taken out from the dies immediately (within 15 seconds) and cooled to room temperature to obtain a forged titanium alloy material. During heating, retention and forging, the forging temperature and the like of the billet was controlled by measuring the temperature by a thermocouple at the 1/2H position and the 1/4D position (H: thickness of the forged material, D: diameter of the forged material), that is, at the intermediate position of the forged material in the thickness direction and the radius direction thereof. In control of the forging temperature and the like, a cooling rate (28°C/min) after forging was determined by a preliminary test. Described specifically, a cooling curve was obtained by preparing a titanium alloy raw material having a shape equal to that of the forged titanium alloy material, inserting a thermocouple in the 1/2H position and the 1/4D position of the raw material, heating and retaining the material at 1000°C, and cooling it in a manner similar to that of the above-mentioned forging. Then, a cooling rate was calculated assuming that the cooling rate was uniform from a time point when the temperature reached 900°C to a time point when the temperature reached 750°C.

(Refining)

[0119] The forged titanium alloy material cooled to room temperature was heated to 805°C, that is, a temperature lower than T_{β} ($\alpha+\beta$ two phase region) and retained thereat for 4 hours. After solubilizing treatment for cooling it at 150°C/min, the resulting material was retained at 610°C for 8 hours, followed by aging treatment for cooling it at 60°C/min to room temperature to manufacture Nos. 1 to 9 test specimens. With regard to the Nos. 1 to 9 test specimens thus manufactured, the microstructure of the material was observed and the average misorientation of crystal orientation of α phase (grain boundary α phase) precipitated in the crystal grain boundary of flattered grains, fatigue property as mechanical property, and ultrasonic inspectability were analyzed. The results thus obtained are shown in Table 1B as an area fraction (%) of flattered grains, average misorientation (°) of grain boundary α phase, area fraction (%) of non-flattered grains, fatigue strength, and ultrasonic inspectability. They were analyzed as follows. The underlined number in Table 1B shows that it does not satisfy the requirement of the present invention.

[Observation of microstructure of material]

(Aspect ratio and diameter of prior β grain, angle of prior β grains boundary, and area fraction of non-flattered β grains)

- 5 **[0120]** From the test specimen, a cubic small sample 15 mm on a side and including the 1/2H and 1/4D positions of it were cut out. From this small sample, cross-sections parallel to the forging direction and the radial direction of the test specimen were cut out, respectively. The cross-sections were mechanically polished with emery paper and finish-polished with diamond abrasive grains, corroded with a nitrohydrofluoric solution and, then provided for microstructure observation.
- 10 **[0121]** The microstructure was observed using an optical microscope and a visual field of 3200 μm x 2000 μm was observed panoramically at a magnification of 100. The diameter in the forging direction (axis direction) and aspect ratio of the prior β grains were determined and an average of them of all the prior β grains in the visual field was calculated. Based on the aspect ratio and diameter, non-flattered β grains (non-flattered grains) and flattered β grains (flattered grains) were detected and an area fraction (%) in the visual field was determined. The aspect ratio of the non-flattered grains was set at 3 or less and the diameter of the non-flattered grains in the forging direction was set at 20 μm or more.
- 15 The aspect ratio of the flattered grains was set at more than 3 and the diameter of the fat grains in the forging direction was set at 20 μm or more but not more than 700 μm .

(Average misorientation)

- 20 **[0122]** The test specimens were electrolytically polished and the crystal orientation of the microstructure on the cross section was measured by SEM/EBSD method (the sampling position and observation surface of the test specimens were similar to those of the above-mentioned optical microscopic observation). The visual field measured had a size of 60 μm in the forging direction and 100 μm in a direction perpendicular thereto and five visual fields were measured. Examples of the results are shown in FIGS. 3 and 4. FIG. 3 shows the measurement result of the crystal orientation in the microstructure on the cross section of Test specimen No. 2 and FIG. 4 shows the measurement result of the crystal orientation in the microstructure on the cross section of Test specimen No. 3.
- 25 **[0123]** On those measurement results, straight lines parallel to the forging direction were drawn at an interval of 10 μm . With regard to each of the grain boundary α phases which intersect with the straight lines, the crystal orientation difference between the grain boundary α phases adjacent to each other was measured. An average misorientation was determined by calculating the crystal orientation difference at all the intersections and averaging them. The results are shown in Table 1B. The average misorientation of 6° or more was regarded to satisfy the requirement.
- 30

(Mechanical property)

- 35 **[0124]** As a mechanical property of the forged titanium alloy material, fatigue strength (fatigue property) was evaluated. From the 1/2H and 1/4D positions of a test specimen, test pieces were cut out so that the circumferential direction (tangential direction) of the test specimen became parallel to a load axis. One of them was provided for evaluating the mechanical property and the other one was provided for ultrasonic inspection described later.
- 40 **[0125]** The mechanical property was evaluated by carrying out a low cycle fatigue test at room temperature in conformity to ASTM E466. The low cycle fatigue test was strain-controlled test under the conditions of the maximum strain of 0.9, a strain ratio of 1.0, and triangle wave until the fracture of the test piece. As the number of cycles to failure, a standardized value with Test specimen No. 1 as a standard (1.0) was calculated and it was shown in Table 1B as a ratio of the number of cycles to failure. A ratio of the number of cycles to failure not less than 1.0 was regarded to satisfy the requirement.

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(Ultrasonic inspectability)

- [0126]** A cubic test piece having a thickness of 41 mm was cut from the test specimen and was subjected to ultrasonic inspection by a water immersion method. In the inspection, a probe having a probe diameter of 19.05 mm and a focal distance of 152.4 mm was used, ultrasonic waves having a frequency of 5 MHz were used as transmission waves, and a water distance (distance from the probe to the surface of the test piece) was set at 140 mm. With the standardized test piece, sensitivity adjustment was performed as to set the reflection intensity from a flat-bottomed hole having a diameter of 0.79 mm at 80%. Then, ultrasonic inspection was performed in a direction parallel to the forging direction (the axial direction of the test specimen) while moving the probe to scan a 50 mm x 50 mm area at the center of the surface of the test piece (surface perpendicular to the forging direction) as an area to be inspected to obtain a C scope.
- 50 **[0127]** The C scope is a two-dimensional image showing flaw inspection results obtained by moving, for scanning, a probe along the surface of an inspection object with a predetermined water distance and extracting the maximum noise intensity within a flaw inspection depth range inspected by the probe at every surface scanning point. The maximum
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noise inspected by the probe thus moved to scan each test piece is shown in Table 1B as a reference.

[Table 1A]

Test specimen No.	Forging conditions				
	β particle size before forging (μm)	Forging temperature ($^{\circ}\text{C}$)	Average strain rate (s^{-1})	Left side value of Formula (3)	Left side value of Formula (4)
1	600	930	0.350	-0.64	4.76
2	600	930	0.035	-2.95	2.45
3	600	870	0.350	<u>0.35</u>	5.75
4	600	930	0.010	-4.24	1.16
5	600	980	0.002	-6.57	<u>-1.17</u>
6	400	930	0.350	-0.64	4.76
7	400	930	0.035	-2.95	2.45
8	600	910	0.005	-4.57	0.83
9	600	<u>1000</u>	0.350	-1.69	3.71

[Table 1B]

Test specimen No.	β crystal grains			Fatigue property	Ultrasonic inspectability	Remarks
	Area fraction of flattered grain (%)	Average misorientation Area fraction of grain boundary α phase ($^{\circ}$)	of non-flattered grain (%)			
1	90	8	8	1.0	35	Example
2	98	29	2	1.3	33	Example
3	100	<u>4</u>	0	0.8	40	Comp. Ex.
4	99	28	1	1.2	33	Example
5	86	15	<u>12</u>	0.6	24	Comp. Ex.
6	98	10	2	1.3	21	Example
7	99	35	1	4.2	19	Example
8	92	11	5	1.0	32	Example
9	<u>75</u>	10	<u>20</u>	0.6	25	Comp. Ex.

[0128] As shown in Table 1A and Table 1B, Test specimens Nos. 1, 2, 4, 6, 7, and 8 that satisfy the requirements of the present invention show an excellent fatigue strength property. Although they are each superior to Test specimen No. 3 corresponding to Comparative Example in fatigue strength property, they show neither an increase in the maximum noise nor deterioration in ultrasonic inspectability (each, Example)

[0129] Test specimen No. 3, on the other hand, shows a high strain rate in spite of a low forging temperature. This means that neither the forging temperature nor the strain rate satisfy the formula (3). Due to a decrease in the curve of the grain boundary of the flattered grains and formation of a linear grain boundary α phase, Test specimen No. 3 therefore has deteriorated fatigue strength (Comparative Example).

[0130] Test specimen No. 5 shows a low strain rate in spite of a high forging temperature. This means that a strain

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rate does not satisfy the formula (4). Due to formation of non-flattered grains during forging or immediately after forging and an increase in the area fraction of the non-flattered grains, Test specimen No. 5 has deteriorated fatigue strength (Comparative Example).

[0131] Test specimen No. 9 has a high forging temperature and does not satisfy the formula (2). Due to recrystallization which has therefore been caused immediately after forging and an increase in the area fraction of non-flattered grains, the test specimen has deteriorated fatigue strength (Comparative Example).

[0132] Test specimens Nos. 1, 5, 8, and 9 contained almost 2%, 2%, 3%, and 5% of crystal grains that did not apply to the above-mentioned definition of non-flattered grains and flattered grains, respectively.

[Example 2: Preparation of test specimen using β billet]

[0133] Similar to Example 1, a β billet composed of a Ti-17 alloy (T_{β} : 890°C, Mo equivalent: the Mo equivalent calculated from an average of elements contained therein is 9.5) specified by AMS4995 were used as a titanium alloy raw material.

[0134] The billet was subjected to heat treatment in which it was air cooled after heated to a β single phase region to obtain a β billet (which will hereinafter be called "former β billet").

[0135] Under conditions similar to those employed in Example 1 except that a titanium alloy raw material was prepared by preform forging ($\alpha+\beta$ forging) the billet into a desired shape in the $\alpha+\beta$ two phase region and the forging temperature was set at that shown in Nos. 10 to 15 of Table 2A, β forging was performed to obtain a β billet (which will hereinafter be called "latter billet"). The strain (preform strain) applied by preform forging is as shown in Nos. 10 to 15 in Table 2A.

[0136] The latter β billet thus obtained was defined under conditions similar to those described in Example 1 and the microstructure of the material was observed. The former β billet not subjected to preform forging in the $\alpha+\beta$ two phase region did not have a desired size because the β crystal grain diameter before forging became coarse by heating at 980°C. Subsequent observation of the microstructure of the material was therefore not performed.

[0137] Table 2A and Table 2B show the β forging conditions of latter P billet and observation results of the microstructure of the material.

[Table 2A]

Test specimen No.	Preform strain	Forging conditions				
		β particle size before forging (μm)	Forging temperature ($^{\circ}\text{C}$)	Average strain rate (s^{-1})	Left side value of Formula (3)	Left side value of Formula (4)
10	0.1	650	920	0.010	-4.04	1.36
11	0.1	650	915	0.100	-1.66	3.74
12	0.1	650	930	0.900	<u>0.30</u>	5.70
13	0.25	450	920	0.025	-3.12	2.28
14	0.25	450	915	0.250	-0.74	4.66
15	0.25	450	940	0.001	-6.66	<u>-1.26</u>

[Table 2B]

Test specimen No.	β crystal grains			Fatigue property	Ultrasonic inspectability	Remarks
	Area fraction of flattered grain (%)	Average misorientation of grain boundary α phase ($^{\circ}$)	Area fraction of non-flattered grain (%)			
10	96	17	2	1.2	35	Example
11	98	9	1	1.1	36	Example
12	88	<u>4</u>	9	0.8	28	Comp. Ex.
13	95	30	3	2.8	24	Example

(continued)

Test specimen No.	β crystal grains			Fatigue property	Ultrasonic inspectability	Remarks
	Area fraction of flattered grain (%)	Average misorientation of grain boundary α phase (°)	Area fraction of non-flattered grain (%)	Ratio of the number of cycles to failure	Maximum noise (%)	
14	97	12	<u>2</u>	1.3	26	Example
15	<u>80</u>	11	<u>15</u>	0.8	20	Comp. Ex.

[0138] As shown in Table 2A and Table 2B, Test specimens Nos. 10, 11, 13, and 14 satisfying the requirements of the present invention has an excellent fatigue strength property (each, Example).

[0139] Test specimen No. 12, on the other hand, shows a high strain rate in spite of a low forging temperature. This means that neither the forging temperature nor the strain rate satisfies the formula (3). Test specimen No. 12 therefore has deteriorated fatigue strength due to a decrease in the curve of the grain boundary of the flattered grains and formation of a linear grain boundary α phase (Comparative Example).

[0140] Test specimen No. 15 shows a low strain rate in spite of a high forging temperature. This means that the strain rate does not satisfy the formula (4). Test specimen No. 15 therefore has deteriorated strength due to formation of non-flattered grains during or immediately after forging and an increase in an area fraction of non-flattered grains (Comparative Example).

[0141] The forged titanium alloy material and the manufacturing method thereof according to the present invention have been described specifically by the modes for carrying out the invention and examples. The gist of the present invention is however not limited by this description. On the other hand, it shall be interpreted widely based on the description of Claims. Various changes, modifications, and the like made based on such a description are also embraced in the technical scope of the present invention.

[Description of the Reference Numerals and Signs]

[0142]

1. Forged titanium alloy material
2. Prior β grains
3. Grain boundary
 - S1. Heating step
 - S2. Forging step
 - S3. Cooling step
 - S4. Ultrasonic inspection step
 - S11. Billets forging step
 - S12. $\alpha+\beta$ Forging step

Claims

1. A β forged titanium alloy materials,
 wherein an area fraction of non-flattered grains of prior β grains having 80% or more its grain boundaries decorated alpha phase, which aspect ratio is 3 or less, and which diameter in a forging direction is 20 μm or more, is less than 10%;
 an area fraction of flattered grains of prior β grains having 80% or more its grain boundaries decorated alpha phase, which aspect ratio exceeding 3, and which diameter in the forging direction is 20 μm or more but not more than 700 μm , is 85% or more; and
 an average misorientation of crystal orientation of an α -phase precipitated along the grain boundary of the flattered grains is 6° or more.

2. The β forged titanium alloy materials according to Claim 1, comprising a titanium alloy having a Mo equivalent

[Mo]_{eq}, which are represented by the following formula (1), more than 2.7 but less than 15:

$$[\text{Mo}]_{\text{eq}} = [\text{Mo}] + [\text{Ta}]/5 + [\text{Nb}]/3.6 + [\text{W}]/2.5 + [\text{V}]/1.5 + 1.25[\text{Cr}] + 1.25[\text{Ni}] + 1.7[\text{Mn}] + 1.7[\text{Co}] + 2.5[\text{Fe}] \dots (1)$$

(wherein, each element symbol in the parentheses on the right side of the formula (1) represents a content [mass%] of each element in the titanium alloy).

3. The β forged titanium alloy materials according to Claim 1 or Claim 2, having a thickness of 30 mm or more at the thinnest portion thereof and 70 mm on average.

4. A method of manufacturing the β forged titanium alloy materials as claimed in Claim 1, wherein the β forging comprises:

a heating step in which a titanium alloy material is heated to $(T_{\beta}+10)^{\circ}\text{C}$ or higher wherein T_{β} represents β -transus temperature and kept until a β crystal grain diameter falls within a range of 300 μm or more but not more than 1000 μm ,

a forging step in which the titanium alloy material is forged at forging temperature, T_F [$^{\circ}\text{C}$] which satisfies the following formula (2) and under conditions in which the forging temperature T_F [$^{\circ}\text{C}$] satisfy the formulas (3) and (4), respectively to produce a forged titanium alloy material; and

a cooling step in which the forged titanium alloy material obtained by the above-mentioned forging is cooled to temperature lower than $(T_{\beta}-150)^{\circ}\text{C}$;

$$T_{\beta}-150 \leq T_F \leq T_{\beta}+100 \dots (2)$$

$$\ln(S_R) + 22800/(T_F+273) - 18.6 \leq 0 \dots (3)$$

$$\ln(S_R) + 22800/(T_F+273) - 13.2 \geq 0 \dots (4)$$

(wherein, in the formulas (2) to (4), T_{β} represents the β -transus temperature [$^{\circ}\text{C}$], T_F represents the forging temperature [$^{\circ}\text{C}$], and S_R represents a strain rate [s^{-1}] upon forging).

5. The method of manufacturing the forged titanium alloy materials according to Claim 4, further comprising:

a billet forging step in which an ingot composed of a titanium alloy is forged into a corresponding billet; and an $\alpha+\beta$ forging step, between the billet forging step and the heating step, in which the billet obtained from the titanium alloy is forged in an $\alpha+\beta$ two phase region.

6. The method of manufacturing the forged titanium alloy materials according to Claim 5, wherein the billet obtained from the titanium alloy has an acicular microstructure.

7. The method of manufacturing the β forged titanium alloy materials according to Claim 4, further comprising:

an ultrasonic inspection step in which after the cooling step, ultrasonic waves are irradiated in a direction parallel to a direction in which an amount of forging in the β forging is the largest to inspect a flaw of the forged titanium-alloy material.

8. The method of manufacturing the forged titanium alloy materials according to Claim 5, further comprising:

an ultrasonic inspection step in which after the cooling step, ultrasonic waves are irradiated in a direction parallel to a direction in which an amount of forging in the β forging is the largest to inspect a flaw of the forged titanium-alloy material.

9. The method of manufacturing the forged titanium alloy materials according to Claim 6, further comprising:

an ultrasonic inspection step in which after the cooling step, ultrasonic waves are irradiated in a direction parallel to a direction in which an amount of forging in the β forging is the largest to inspect a flaw of the forged titanium alloy material.

10. The method of manufacturing the forged titanium alloy materials according to any one of Claims 4 to 9, wherein the forged titanium alloy material is a material to be used for manufacture of engine components of aircraft.

FIG. 1

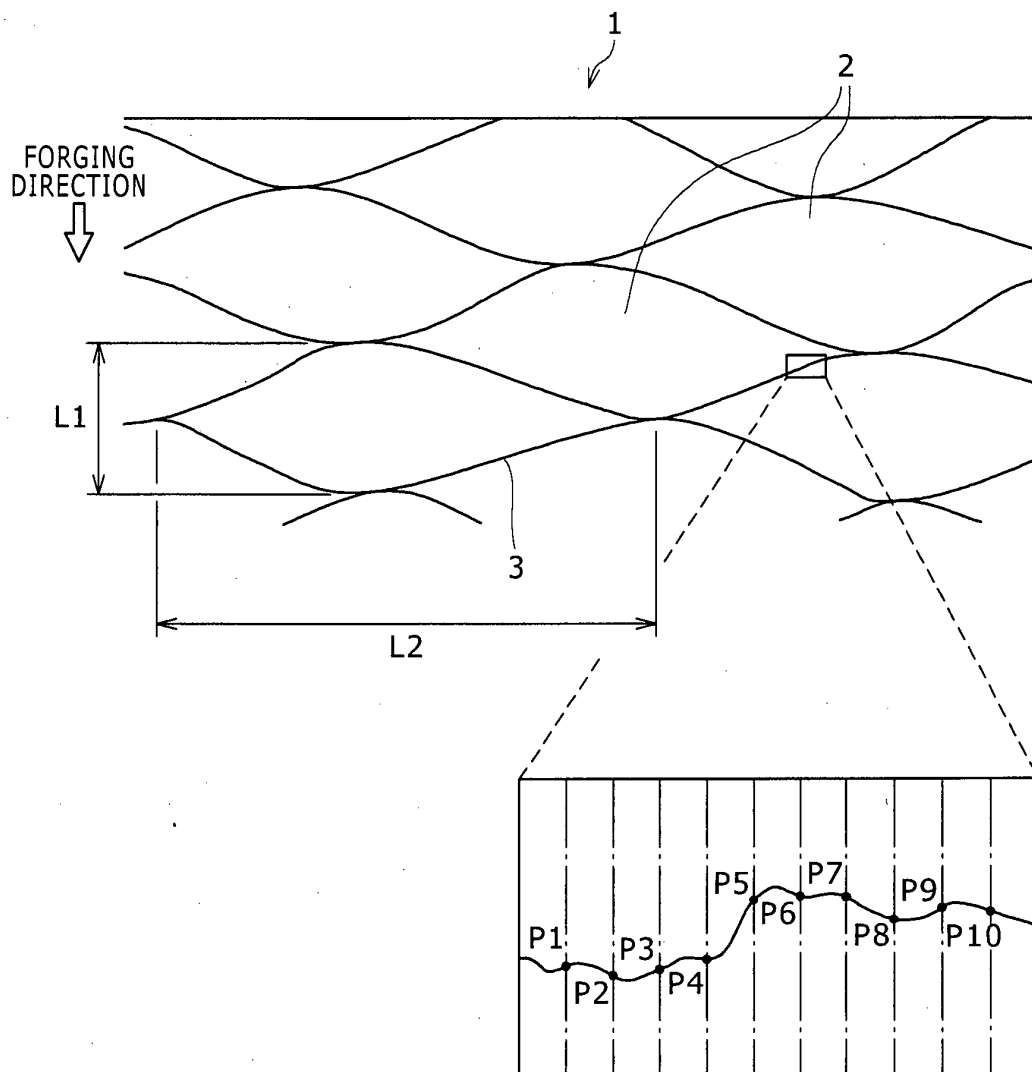


FIG. 2

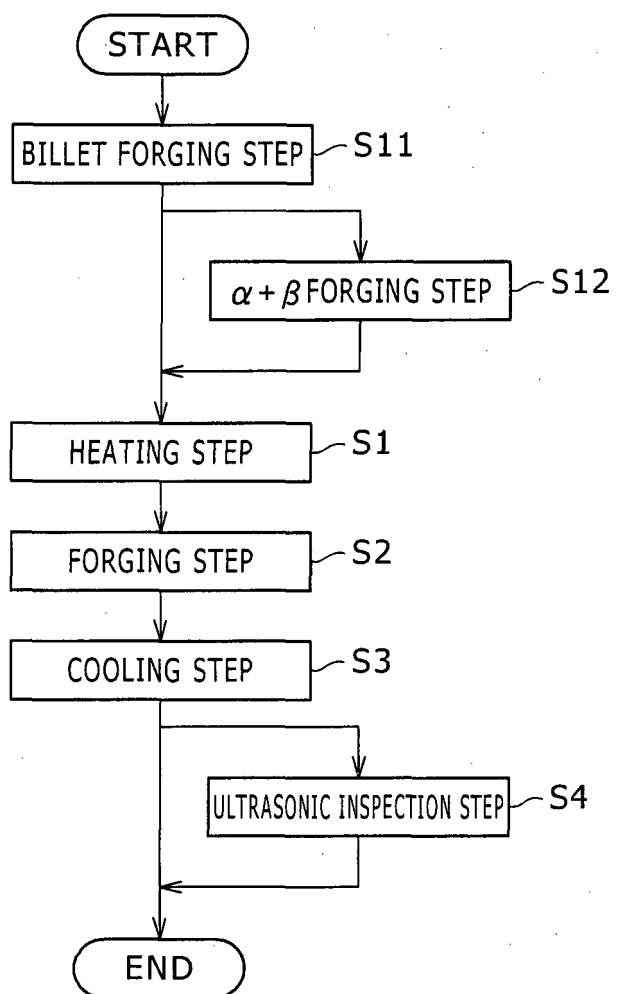


FIG. 3

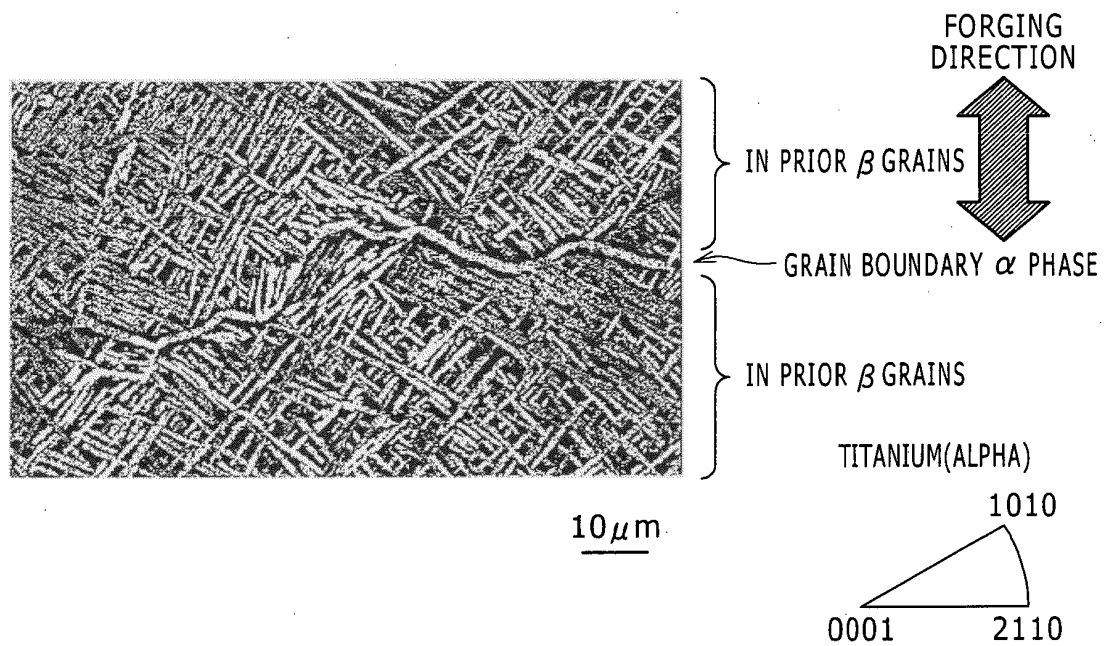


FIG. 4

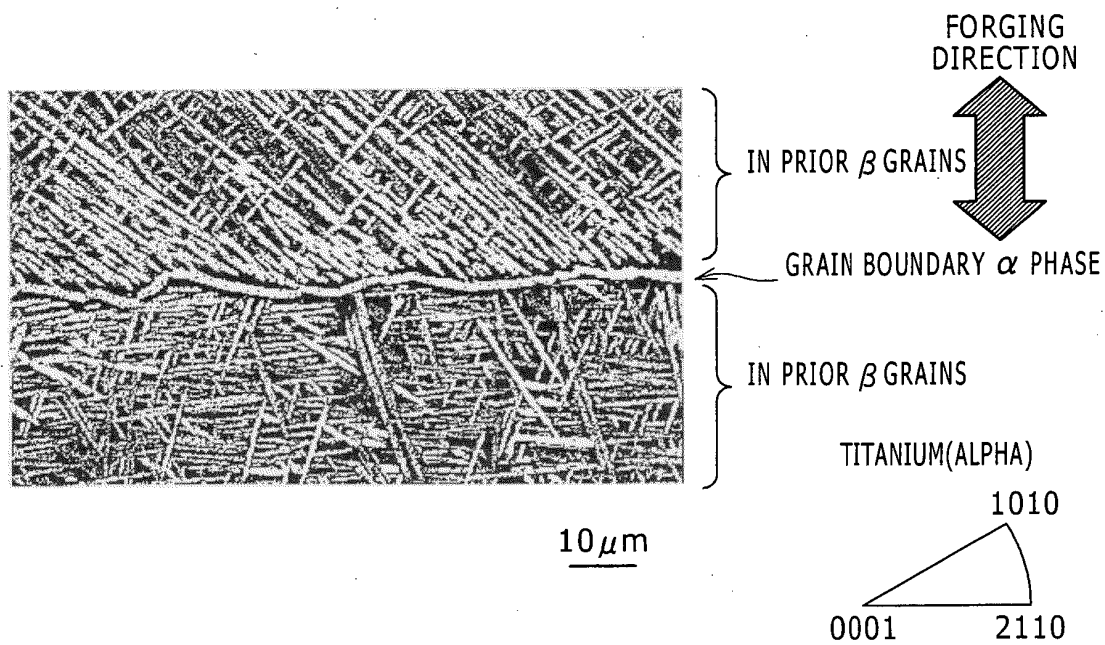


FIG. 5

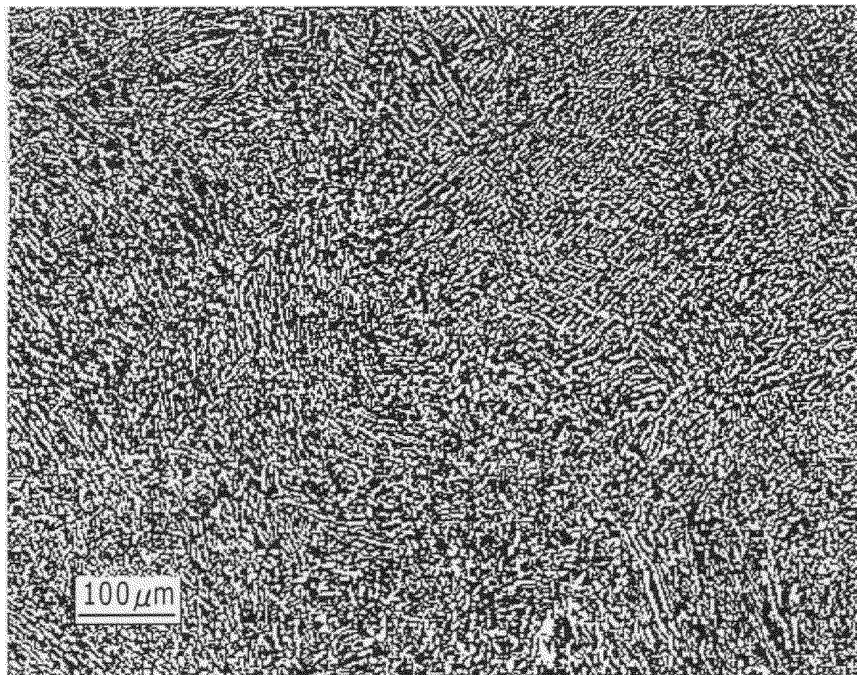
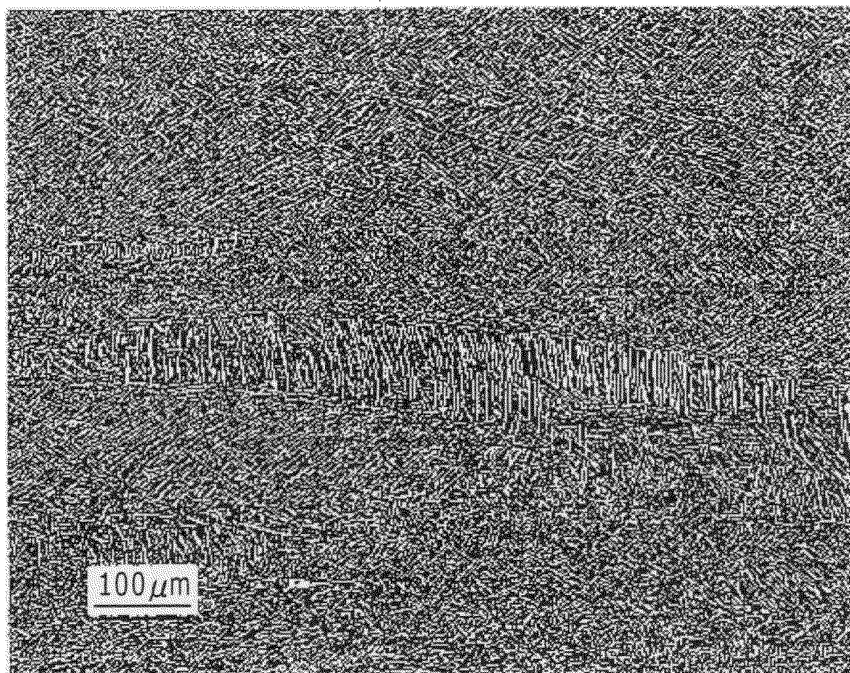


FIG. 6



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2014/051119

A. CLASSIFICATION OF SUBJECT MATTER

C22C14/00(2006.01)i, C22F1/18(2006.01)i, C22F1/00(2006.01)n

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C22C14/00, C22F1/18, C22F1/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2014

Kokai Jitsuyo Shinan Koho 1971-2014 Toroku Jitsuyo Shinan Koho 1994-2014

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2002-146499 A (NKK Corp.), 22 May 2002 (22.05.2002), entire text & US 2004/0244887 A1 & EP 1500715 A1 & WO 2003/091468 A1	1-10
A	JP 10-17961 A (Kobe Steel, Ltd., Citizen Watch Co., Ltd.), 20 January 1998 (20.01.1998), entire text & US 5885375 A & EP 834586 A1 & WO 1997/037049 A1 & CN 1194671 A	1-10

☒ Further documents are listed in the continuation of Box C.☐ See patent family annex.

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Date of the actual completion of the international search
26 March, 2014 (26.03.14)Date of mailing of the international search report
08 April, 2014 (08.04.14)Name and mailing address of the ISA/
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INTERNATIONAL SEARCH REPORT

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5

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 03/091468 A1 (JFE Steel Corp.), 06 November 2003 (06.11.2003), entire text & JP 2002-146499 A & US 2004/0244887 A1 & EP 1500715 A1	1-10

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REFERENCES CITED IN THE DESCRIPTION

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- JP 2011102414 A [0019]