Forged aluminium alloy material having excellent high temperature fatigue strength

An Aluminium alloy forged material comprising: Cu: 4.0% - 7.0%; Mg: 0.2% - 0.4%; Ag: 0.05% - 0.7%; and V: 0.05% - 0.15%, wherein a distribution density of an Al-V based precipitate in the forged-material structure is no less than 1.5 piece/µm³. Such constitution may provide an Al alloy forged material having not only high temperature characteristics, such as heat resistance and a high temperature proof stress, but an excellent high temperature fatigue strength.
[0001] The present invention relates to a 2000-series Aluminum alloy forged material (hereinafter, Aluminum referred only to Al), and particularly to an Al alloy forged material having an excellent high temperature fatigue strength, and other excellent high temperature characteristics (heat resistance and high temperature proof stress).

[0002] Al alloy forging materials having excellent high temperature characteristics are used in materials for aviation and space equipment, such as rockets and airplanes; materials for transportation equipment, such as railroad vehicles, cars, and vessels; or materials for machine parts, such as engine parts and compressors; specifically, in parts made from Al alloy used in service conditions of particularly high temperatures exceeding 100°C, such as in rotation rotors, and rotation impellers or pistons. The high temperature characteristics here include a creep resistance and a high temperature proof stress under high temperatures.

[0003] Conventionally, 2000-series Al alloys of AA standard or JIS standard (henceforth referred to 2000-series) have been used for these so-called heat-resistant Al alloy forging materials. This kind of Al alloy includes 2219, 2618, etc. However, prolonged use at high temperatures exceeding 120°C gives remarkable strength reduction in these 2000-series Aluminums alloys. For this reason, in order to improve a creep characteristic and a high temperature proof stress in service conditions at high temperatures exceeding 120°C, in recent years, 2219Al alloy including 0.3% of Mg added, that is, 2519Al alloy (Al / 6.1 Cu; 0.3 Mn; 0.15 Zr; 0.1 V) was developed. In addition, 2519 (Ag) Al alloy in which Ag is added in 2519Al alloy was also developed. And many Al alloys in relation to these 2519Al alloys and 2519(Ag) Al alloys are also proposed (for example, refer to JP-A No. 1987-112748, and U.S. Pat. No.4610733).

[0004] The present inventors also proposed heat-resistant Al alloys enabling guarantee of improved high temperature characteristics with sufficient reproducibility. This includes following contents: an average size of θ′ phase is set as no more than 150 nm, in an average interval between precipitates in θ′ phase is set as no more than 100 nm and an average size of Ω phase is set as no more than 100 nm, and an average interval between precipitates in Ω phase is set as no more than 150 nm, in θ′ phase and/or Ω phase of a heat-resistant Al alloy including Cu: 1.5 - 7.0% and Mg: 0.01 - 2.0%, and furthermore selectively including Ag: 0.05 - 0.7%; (refer to JP-A No. 1999-302764, and 93rd autumn convention lecture outline of Japan Institute of Light Metals (issued Jan, 20, 1997, pp. 233-234.)

[0005] Furthermore, application parts for which high temperature characteristics are required fundamentally have shapes of a thick cylindrical shape, and complicated shape with many blades around. For this reason, when these parts are manufactured by Al alloy, ingots having a shape of bulk of Al alloy (massive) is hot forged (cold forging after hot forging is also included), and then obtained forged material is processed by cutting to give target parts. And since these application parts slide or rotate between narrow space or clearance at high speeds, high accuracy of dimension and high smoothness are severely required for them.

[0006] Therefore, in order to guarantee high temperature characteristics of heat-resistant Al alloy forged materials for high speed motion parts, and machinability in cutting of high speed motion parts, the present inventors proposed: microstructures after solution heat treatment of Al alloy forged materials have θ′ phase and/or Ω phase; and crystal grain diameter is of isometric recrystallized particles of no more than 500 μm (Refer to JP-A No.2000-119786).

[0007] However, even if Al alloy forged materials excellent in high temperature characteristics are designed by such technique in metallurgy, artificial ageing curing processing at high temperatures that is performed after a solution heat treatment and a hardening processing in Al alloy forged materials actually manufactured sometimes may not improve proof stress, and it may decrease proof stress after an artificial ageing curing processing required for this kind of Al alloy forged materials (heat-resistant Al alloy forged materials), and also decrease proof stress at the time of high temperature use. Therefore, the present inventors pay attention to influence of hardening speed after solution heat treatment, and proposed that when a slow (low) hardening speed (low cooling rate) is used, such as in the case where an average cooling rate between 400°C and 290°C is no more than 30000°C / minute, especially, Zr, Cr, and Mn in the Al alloy forged materials are regulated to Zr: no more than 0.09%, Cr: no more than 0.05%, and Mn: no more than 0.6%, respectively (refer to JP-A No. 2001-181771).

[0008] However, even Al alloy forged materials excellent in high temperature characteristics, such as the heat resistances and high temperature proof stress, have furthermore a room for improvement in high temperature fatigue strength. That is, even improved Al alloy forged materials shown in JP-A No. 2000-119786, JP-A No. 2001-181771, etc. have only numbers of fracture repetitions of (3 - 6) x 10⁶ times in a rotating bending fatigue test (under a condition of maximum stress 130 MPa, stress ratio 1, and 150°C) in a fatigue strength under an operating condition of stress load at high temperatures (high temperature fatigue strength). Therefore, further improvement has been required for product application in which a higher high temperature fatigue strength is required.

[0009] The present invention is made paying attention to such a situation, and a purpose thereof is providing Al alloy forged materials having not only high temperature characteristics, such as heat resistance and high temperature proof stress, but an excellent high temperature fatigue strength.

[0010] An important aspect of an Aluminum alloy forged material of the present invention in order to attain the purpose is that the material includes Cu: 4.0 - 7.0%; Mg: 0.2 - 0.4%; Ag: 0.05 - 0.7%; V: 0.05% - 0.15%, Al-V based precipitates
in a forged material structure have a distribution density of no less than 1.5 piece/µm³.

Besides, in Aluminum alloy forged materials of the present invention, in order to set a distribution density of Al-V based precipitates in this forged material structure as no less then 1.5 piece/µm³, an Aluminum alloy cast material including Cu: 4.0 - 7.0%; Mg: 0.2 - 0.4%; Ag: 0.05 - 0.7%; V: 0.05% - 0.15% is preferably hot forged at a temperature of 280 - 430°C after homogenized heat treatment at a temperature of 500 - 535°C for no less than 15 hours, and subsequently solution heat treatment at a temperature of 510 - 545°C is performed to give a hardening processing.

In addition, all of % representations of an alloying element contents represent a mass %. Besides, definition of the distribution density of Al-V based compounds in the above-mentioned forged material structure is specified for thermally refined Aluminum alloy forged materials.

Previously, as an application for Japanese patent application No. 2003-90660, the present inventors applied for a patent as forged material invention which including V as alloying elements, in order to improve high temperature characteristics, such as heat resistance and high temperature proof stress, of Aluminum alloy forged materials. However, the present inventors have found that there might be obtained only a little amount of Al-V based compounds precipitated in forged material structures actually manufactured under some manufacturing conditions, even if substantially sufficient amount of V was included as alloying elements, and accordingly, improvement particularly in high temperature fatigue strength had a limit in high temperature characteristics. In fact, a high temperature fatigue strength indicated by the above-mentioned Japanese patent application No. 2003-90660 shows a number of (3 - 6) x 10^6 times [represented also as (3 -6)x10^6] of fracture repetitions in rotating bending fatigue test (under a conditions of maximum stress 130 MPa and a stress ratio of 1, and 150°C).

On the other hand, in the present invention, an Al-V based compound is precipitated in a forged material structure so that V included satisfies a sufficient amount (number) in order to increase high temperature fatigue strength. As a result, a high temperature fatigue strength is remarkably improved as compared in a forged material including comparatively small amount of Al-V based compound precipitated in a forged material structure in spite of similar content of V.

Observation using a transmission electron microscope (TEM) having 10,000 times of magnification for the forged material structure after thermal refining processing (heat treatment) mentioned later may give the distribution density of Al-V based precipitate. Namely, the above-mentioned observation for a plurality of regions over whole of structures of each region of the forged material or at least in a forged material region that requires a high temperature fatigue strength.

In order to secure the high temperature fatigue strength of an Aluminum alloy forged material, it is preferable that the distribution density definition of such Al-V based precipitate may be satisfied over whole of the forged material structure, or at least in a forged material region that requires a high temperature fatigue strength.

Observation using a transmission electron microscope (TEM) having 10,000 times of magnification for the forged material structure after thermal refining processing (heat treatment) mentioned later may give the distribution density of Al-V based precipitate. Namely, the above-mentioned observation for a plurality of regions over whole of structures of each region of the forged material or for the forged material region that at least requires high temperature fatigue strength may give a number of Al-V based precipitates within a microscopic field (dispersed grain), which may be converted into a number per µm³. Although distribution density measurement of the Al-V based precipitate may be performed to one point of the forged material region that particularly requires a high temperature fatigue strength, in order to satisfy reproducibility, measurement in two or more points is preferable. In measurement in two or more points, the distribution density of Al-V based precipitate are of course expressed by an average value of measured values of a plurality of measurement points.

In addition, visual observation based on a shape-characteristic of dispersed grains in a structure (precipitate) etc. will enable differentiation between an Al-V based precipitate and other precipitate in observation using the above-mentioned transmission electron microscope having 10,000 times of magnification. However, elements and the amounts of elements (in the case of Al-V based precipitate, element of V) constituting precipitates in the forged material structure may be identified to be discriminated from other precipitates using EPMA (X-ray microanalysis) for further accuracy.

In order to precipitate V that is included as alloying elements in a forged material structure like the present invention so that no less than 1.5 piece/µm³ of distribution density of Al-V based precipitate may be obtained, prolonged homogenized heat treatment is required to be given for cast materials comprising a component composition of the present invention including V. That is, a prolonged homogenized heat treatment at temperatures of 500 - 535°C, for
no less than 15 hours is required.

[0020] Usually, a homogenized heat treatment is performed to this kind of cast material at temperatures of 500 - 535 °C, for less than 15 hours at the maximum and in many cases for a processing time of about 8 hours. Even a homogenized heat treatment on such a short period enable homogenization of the cast material itself. However, V has a very slow diffusion rate as compared with other elements. Therefore, under such short-time homogenized heat treatment conditions, V included as alloying elements keeps a state of solid solution during the homogenized heat treatment, which disables precipitation to give an actual mass sufficient to remarkably improve high temperature fatigue strength as an Al-V based compound, that is, to give no less than 1.5 piece/µm³ of distribution density in the forged material structure of an Al-V based precipitate.

[0021] A description about manufacturing method of the present invention forged material will, hereinafter, be given. Manufacturing conditions and manufacturing method of an Al alloy forged material in the present invention are fundamentally same as conventional methods except for a period of the above-mentioned homogenized heat treatment. In other words, it is also an advantage of the present invention to avoid large modification of manufacturing conditions or manufacturing methods of an Al alloy forged material.

[0022] In casting, an Al alloy molten metal is melted, adjusted within a component range of the present invention, and is cast to manufacture ingots using usual melting casting methods selected suitably, such as a continuous casting rolling method and a semicontinuous casting method (direct chill casting process).

[0023] At temperatures of 500 - 535 °C, hot forging of the ingots is carried out after the above-mentioned prolonged homogenized heat treatment to manufacture an Al alloy forged material. In addition, as materials for forging, extruding or rolling processed ingots, that is, extruded materials and rolled materials may be used. Temperatures of less than 500 °C in the above-mentioned homogenized heat treating may not give a solid solution of crystallized object of an ingot here, but provide an inadequate homogenization. On the other hand, temperatures exceeding 535 °C of the above-mentioned homogenized heat treatment increases a possibility of generation of burning. Therefore, a temperature of the above-mentioned homogenized heat treatment is set in a range of 500 - 535 °C.

[0024] Temperature conditions of hot forging are important for manufacturing an Al alloy forged material with sufficient reproducibility according to designed high temperature characteristics. Conventionally, well-known forging methods, such as free forging and die forging (stretching forging), have been suitably adopted independently or in combination, and a hot-forging temperature has been set about 380 - 430 °C in order to obtain a microstructure having an equiaxial crystal grain after solution treatment of the Al alloy forged material, because there has been a recognition that a low hot-forging temperature tends to provide a locally mixed particle in the structure of the Al alloy forged material, leading to deterioration in high temperature characteristics.

[0025] In this point, in the present invention, hot-forging temperatures are preferably set in a range of 280 - 430 °C, that is, below recrystallization temperatures. Hot-forging temperatures exceeding 430 °C easily form coarse grains in Al alloy forged materials within a range of components of the present invention, which deteriorates the high temperature characteristics of the Al alloy forged materials, and disables manufacturing of Al alloy forged materials having excellent high temperature characteristics. On the other hand, hot-forging temperatures of less than 280 °C tends to give a crack at the time of hot forging, and make the forging processing itself difficult.

[0026] Even if the temperatures of hot forging is set in a range of 280 - 430 °C in the present invention, in an Al alloy forged material in range of components of the present invention, suitable solution-treatment and hardening processing give equiaxial crystal grain to microstructures after thermal refining of the Al alloy forged material, and do not give it mixed grains.

[0027] In addition, the microstructures of the Al alloy forged materials are influenced by a forging ratio in the hot forging. Therefore, in order to obtain equiaxial crystal grain in the microstructures, preferably, a proper forging ratio for the hot forging is set preferably no less than 1.5 in the Al alloy forging materials. Forging ratios less than 1.5 easily provide mixed grains to structures of the Al alloy forged materials. More preferably, forging is performed not only in one direction but in at least two different directions, and forging ratios in each directions are set no less than 1.5.

[0028] Next, solution treatment and hardening processing will be described. Processing is preferably performed within conditions specified in JIS H 4140, AMS-H -6088, etc. in order to transform soluble intermetallic compounds to solid solutions again and to suppress re-precipitation during cooling as much as possible in this solution treatment and hardening processing. However, even if the heat treatment is performed based on standards of AMS-H-6088 etc., when solution treatment temperatures are excessively high, burning will arise, which will markedly deteriorate mechanical properties. And solution treatment temperatures no more than minimum temperatures may not provide a proof stress of no less than 400 MPa at room temperatures after artificial ageing curing processing, and also make the solution treatment itself difficult. Therefore, a maximum of the solution treatment temperature is set 545 °C, and a minimum is set 510 °C.
[0029] In applications, such as small parts, pistons having diameters to about 100 mm, and in products in which a comparatively large remaining stress does not cause problems in processing of cutting etc., artificial ageing curing processing is preferably performed after a solution treatment and a hardening processing to obtain thermally refined T6 materials. In this case, in order to obtain high strength properties and high temperature characteristics even if remaining stress becomes comparatively larger, it is desirable for hardening temperatures to be no more than 40°C. If the hardening temperatures are high, it is difficult to set the proof stress at room temperatures after artificial ageing curing processing as no less than 400 MPa.

[0030] On the other hand, in large-sized products, such as rotors, since cooling rates in a product surface and in a central area have largely different values from each other during a hardening processing, a high remaining stress exceeding 10 kgf/mm² in the product surface is generated. Generation of such a high remaining stress provides large distortion at the time of cutting of the product, and makes precise cutting very difficult, and at the worst, breakage by cracks caused by the remaining stress etc. is sometimes generated during cutting processing. Even if breakage by cracks etc. does not arise in cutting processing, in use over a long period of time of the product, cracks easily spread and grow from intermetallic compounds, such as crystallized matter remaining in the material, or from very small surface cracks generated during transportation of the product as starting points, possibility leading to resulting final fracture. Therefore, products having possible remaining stress, such as rotors, needs to remove remaining stress and to preferably decrease to no more than 3.0 kgf/mm². To do this, it is preferable that water hardening temperatures after solution treatment is set comparatively high temperatures, such as no less than 90°C, and subsequently, artificial ageing curing processing is performed to obtain a thermal refined T61 materials.

[0031] Moreover, remaining stress needs to be severely managed depending on applications and products regardless of size of products. In such products, in order to make remaining stress as small as possible, cold pressing or cold work is added, and the remaining stress is preferably removed or reduced to no more than 3 kgf/mm², and subsequently, artificial ageing curing processing is performed to obtain a thermal refined T652 materials. In these products, in order to remove or reduce remaining stress to preferably no more than 3 kgf/mm² and to obtain high strength properties and high temperature characteristics, hardening temperatures are preferably no more than 40°C. When this hardening temperature is high, it will become difficult for proof stress at room temperature after artificial ageing curing processing to be set no less than 400 MPa. When an amount of cold pressing (processing) of the cold pressing or cold work is small, sufficient reduction effect of the remaining stress may not be obtained. On the other hand, since a large amount of the cold pressing makes an amount of precipitation of 0° phase increase, during processings for artificial ageing curing or under high temperatures, it easily deteriorates proof stress. Therefore, in the cold pressing (processing), it is preferable that a rate of compression (processing) is set to 1 - 5%.

[0032] Subsequently, these Al alloy forged materials are processed to make application parts. Of course, after processing of Al alloy forged material to obtain the application products, solution treatment, hardening processing, cold pressing, artificial ageing curing processing, etc. may suitably be performed.

[0033] As furnaces used for thermal refining (heat treatment), such as solution treatment and hardening processing, a batch type furnace, a continuous annealing furnace, a molten salt bath furnace, an oil furnace are suitably usable. As cooling methods for hardening, methods, such as water immersion, warm water immersion, boiled water immersion, water injection, and air injection, may suitably be selected.

[0034] An average grain diameter of crystals of an Al alloy forged material of the present invention obtained in this way is no more than 1 mm, preferably in a range of 10 - 500 µm, and more preferably in a range of 50 - 300 µm, and the crystals are minute recrystallized grains (equiaxial recrystallized grain) having an almost fixed size. The Al alloy forged material of the present invention obtained in this way has high temperature characteristics and machinability, such as excellent creep characteristics, and does not have groups obtained by aggregation of minute recrystallized grains (or subgrains) having grain diameters of no more than 1 µm as found in the above-mentioned mixed grain structure, coarse recrystallized grains having grain diameters of about several mm - several cm, or remaining ingots structures.

[0035] However, a structure of preferable equiaxial recrystallized grain in the present invention does not only necessarily represent a structure including 100% of equiaxial recrystallized grain having a fixed size, and allows intermixing of cast structures or mixed grain structures within a range in which high temperature characteristics, such as machinability, creep rupture strength, are not adversely affected. For example, existence in a dispersed state of single crystal grains of minute recrystallized grains (or subgrains) having grain diameters of no more than 1 µm does not deteriorate high temperature characteristics, such as the machinability, creep rupture strength. However, in the case where these crystal grains aggregate or group in a state of closely gathered mutually, machinability and high temperature characteristics are deteriorated. Therefore, in view of this point, a rate of area of aggregate of minute recrystallized grains having no more than 1 µm of diameter in the microstructure after solution treatment is preferably set no more than 10%.

[0036] Besides, in specification of equiaxial recrystallized grains in the present invention and identification of existence of mix grain structures, sample is treated by a micro etching processing, such as electrolytic etching, and may be observed or measured with an optical microscope having 50 to 400 times of magnification.
Next, in order to further increase high temperature characteristics, such as high temperature proof stress and creep rupture strength, in an Al alloy forged material structure of the present invention, preferably, $\theta'$ phase is precipitated in a plane (100) of Al alloy, and $\Omega$ phase in a plane (111) under conditions selected from a range of 7 - 60 hours at 160 - 190°C in an artificial ageing curing processing after a solution treatment and a hardening processing. Missing of these precipitation by artificial ageing curing processing lowers a high temperature proof stress at temperatures of about 180°C, even when the artificial ageing curing processing is provided.

In addition, identification of a precipitation state of $\theta'$ phase and $\Omega$ phase in the Al alloy forged-material structure may be enabled by a structure observation using a transmission electron microscope (TEM) having 50000 times of magnification and, if necessary, using the above-mentioned EPMA.

(Chemical component composition in the Al alloy forged material)

Next, description will be given about chemical component composition in the Al alloy forged material of the present invention. Although a chemical component composition of the Al alloy of the present invention has fundamentally a component standard of Al alloys, such as 2519 or 2618, and 2519 (Ag) based Al alloy in which Ag is added into 2519, it may suitably be selected from a component composition range described below. First, elements positively included will be described.

(Cu: 4.0 - 7.0%)

Cu is a fundamental component of the present invention Al alloy forged material, and it demonstrates both functions of solid solution strengthening and precipitation strengthening, and furthermore it is indispensable in order to secure creep characteristics at normal temperatures and high temperatures, and a high temperature proof stress, and further a high temperature fatigue strength that is required mainly in applications of the Al alloy forged material of the present invention. More specifically, as mentioned above, Cu precipitates $\theta'$ phases and $\Omega$ phases in a plane (100) and a plane (111) of the Al alloy in a minute state with high density during a hot artificial ageing curing processing, improving a strength of the Al alloy forged material after the artificial ageing curing processing. This effect is demonstrated by no less than 4.0% of content, and less than 4.0% of the content of Cu gives small above-mentioned effect, and does not give sufficient creep characteristics and a high temperature proof stress at normal temperatures and high temperatures of the Al alloy forged material. On the other hand, a content exceeding 7.0% of Cu gives an excessive high strength, and deteriorates forgeability of the Al alloy forged material. Therefore, a content of Cu is set in a range of 4.0 - 7.0%.

(Mg: 0.2 - 0.4%)

Mg as well as Cu demonstrates both function of solid solution strengthening and precipitation strengthening, and is indispensable in order to mainly secure sufficient creep characteristics at normal temperatures and high temperatures, and a high temperature proof stress, and also a high temperature fatigue strength of the Al alloy forged material. More specifically, Mg as well as Cu precipitates $\theta'$ phases and $\Omega$ phases in a plane (100) and a plane (111) of the Al alloy in a minute state and with high density during a hot artificial ageing curing processing, improving a strength of the Al alloy forged material after the artificial ageing curing processing. This effect is demonstrated with no less than 0.2% of content, and less than 0.2% of the content of Cu gives small above-mentioned effect, and does not give sufficient creep characteristics and a high temperature proof stress at normal temperatures and high temperatures of the Al alloy forged material. On the other hand, a content of Mg exceeding 0.4% gives an excessive high strength, and generates cracks called burning at the time of solution treatment, or increases a possibility of deteriorating forgeability. Therefore, a content of Mg is set in a range of 0.2 - 0.4%.

(Ag: 0.05 - 0.7%)

While Ag forms minute and uniform $\Omega$ phases in an Al alloy forged material, it is also indispensable, in order to form a zone without existence of precipitate phase (PFZ; solute-depleted precipitate free zone) with very narrow width and to improve a strength at normal temperatures and high temperatures of the Al alloy forged material. A content of less than 0.05% of Ag does not demonstrate this effect, and on the other side, a content exceeding 0.7% of Ag saturates the effect. Therefore, a content of Ag is set in a range of 0.05 - 0.7%.

(V: 0.05% - 0.15%)

V precipitates in forged material structures as Al-V based compounds and is indispensable element in order
to improve a high temperature fatigue strength. During a homogenized heat treatment, V precipitates Al-V based dispersed grains that are thermally stable compounds in the Al alloy forged material structures. This precipitate has a function for disturbing grain boundary migration after recrystallization, and thus may demonstrate an effect of prevention of coarsening, that is, refining a diameter of average crystal grain in a range of no more than 500 μm. As a result, it forms fiber structures of microstructures of the Al alloy forged material, which improves a strength at normal temperatures, and a strength at high temperatures and particularly a high temperature fatigue strength. And V has comparatively small function for precipitating stable and coarse phase as compared with Zr, Cr, and Mn.

In order to demonstrate this effect, no less than 0.05% of content is required. A content of less than 0.05% of V is inadequate. When content of V is less than 0.05%, even the above-mentioned prolonged homogenized heat treatment of no less than 15 hours may not precipitate the Al-V based precipitate in the forged material structure by no less than 1.5 piece/μm³ of distribution density. On the other hand, a content exceeding 0.15% of V tends to form coarse insoluble intermetallic compounds at the time of melting casting, leading to defect of molding and breakage. Therefore, V is included in a range of 0.05% - 0.15%.

Hereinafter, description about element to be preferably regulated will be given. Zr, Cr, and Mn precipitate Al-Zr based, Al-Cr based, and Al-Mn based dispersed grains that are thermally stable compounds, respectively, in Al alloy forged material structures like the above-mentioned V at the time of homogenized heat-treatment. And this dispersed grains form fiber structures with microstructures of the Al alloy forged material, and has an effect for improving a strength at normal temperatures and a strength at high temperatures.

However, when an average cooling rate between 400°C and 290°C is set late below to 3000°C / minute in a hardening processing after a solution treatment, a content of these Zr, Cr, and Mn coarsely precipitates stable phases, such as of AlCu₂ around the Al-Cr based, Al-Zr based, and Al-Mn based dispersed grains in a process of hardening processing after solution treatment. As a result, even if the hot artificial ageing curing processing is performed in a next step, a proof stress at high temperatures, such as no less than 310 MPa after use of 100 hours at a temperature of 120°C, may not be obtained. Therefore, in order to lower a hardening sensitivity of the Al alloy forged material, regulations for Zr: no more than 0.09%, Cr: no more than 0.05%, and Mn: no more than 0.8%, respectively, are preferable.

Fe is preferably regulated to no more than 0.15%. However, since contamination from scraps etc. is unavoidable and it is effective in improving high temperature characteristics of the Al alloy forged material, content of Fe up to 0.15% may be allowed. A content exceeding 0.15% forms insoluble intermetallic compounds, and tends to become defects of molding and of breakage.

Si combines with Mg to form Mg₂Si and Al-Fe-Si based crystallized matter in the Al alloy forged material structure. Therefore, this Si precipitates θ’ phases and Ω phases at the time of artificial ageing curing processing at high temperatures, and consumes Mg required for improving a strength of the Al alloy forged material after artificial ageing curing processing, which deteriorates a strength of the Al alloy after artificial ageing curing processing. Since Mg is originally included fewer as compared with Cu, this influence induced by the Si is significant. In addition, although most of the crystallized matter enters into solid solution state by the solution treatment, excessive formation of Mg₂Si remains also in solution treatment, and forms starting points of fracture to deteriorate moldability. Therefore, Si is preferably regulated no more than 0.1%.

In addition, although Ti makes minute crystal grains at the time of casting, excessive addition will form coarse intermetallic compounds to form starting points of fracture at the time of fabrication, leading to decrease in moldability. Therefore, no more than 0.1% of content of Ti may be allowed.

Therefore, following elements in the Al alloy forged material are preferably regulated: Si: no more than 0.1%; Fe: no more than 0.15%; Zr: no more than 0.09%; Cr: no more than 0.05%; Mn: no more than 0.8%; and Ti: no more than 0.1%, respectively, in order to prevent deterioration of a proof stress after artificial ageing curing processing and a proof stress in use at high temperatures of an Al alloy forged material. Since Mg is originally included fewer as compared with Cu, this influence induced by the Si is significant. In addition, although most of the crystallized matter enters into solid solution state by the solution treatment, excessive formation of Mg₂Si remains also in solution treatment, and forms starting points of fracture to deteriorate moldability. Therefore, Si is preferably regulated no more than 0.1%.

Besides, as for Zn, Ni, and B other than the above-mentioned elements, content may be allowed in a range that does not deteriorate high temperature characteristics or other characteristics of the Al alloy forged material concerning the present invention, or content about a maximum standard for 2000-series Aluminum.

Descriptions about Examples of the present invention will, hereinafter, be given. A relationship was investigated between a distribution density of an Al-V based precipitate and a high temperature fatigue strength in an Aluminum alloy forged material structure including V, and influence of homogenized heat treatment time to a distribution density of the Al-V based precipitate.

That is, Al alloy ingots (500 mm x 2000 mm) were ingoted that have mainly different V content as shown in Table 1, and that have chemical component compositions of A - C within a range of the present invention, and chemical component compositions of D and E outside of a range of the present invention, respectively. Subsequently, only
processing periods were varied, as shown in Table 2, and homogenized heat treatment (air furnace) was performed at a temperature of 510°C.

[0054] In each example, ingots after the homogenized heat treatment were processed so that no less than 1.5 of forging ratios in all directions may be given to obtain square bars of 150 mm per side (thickness), and square bars of 80 mm per side (thickness). Those square bars were cut by a length of 300 mm, and Al alloy forged materials were manufactured. In each example, the Al alloy forged materials were heated by a heating rate of 200°C / hr with air furnace, and after a solution treatment of 528°C x 6 hr, water hardening was performed at various hardening temperatures, respectively, shown in Table 2 (average cooling rate between 400°C and 290°C, more than 30000°C /minute), and then they were taken out after maintenance underwater for 10 minutes.

[0055] Based on an assumption that applications, such as small parts and pistons, in which a comparatively large remaining stress is allowed, a water hardening processing at low temperature of 30°C was performed after solution treatment to the Al alloy forged materials of these samples (example 5) having a thickness of 80 mm, and then artificial ageing curing processing at 175°C x 18 hr was performed to obtain a refined T6 material.

[0056] Moreover, based on an assumption that applications in which a residual stress induces a problem, 91°C warm water hardening was performed after solution treatment to the Al alloy forged material having a thickness of 150 mm to reduce a residual stress, and artificial-aging curing processing of 175°C x 18 hr was performed without cold compression to obtain a refined T61 material.

[0057] In addition, based on an assumption that applications in which a residual stress induces a problem, 30°C water hardening processing was performed after solution treatment to an example 6 among samples having 150 mm of thickness, then a cold pressing processing was performed at 0.8% of rate of cold pressing to reduce a remaining stress, and subsequently artificial ageing curing processing of 175°C x 18 hr was performed to obtain a refined T652 material.

[0058] Samples were obtained from these refined Al alloy forged materials, and each fatigue strength at room temperature (maximum stress 190 MPa, stress ratio 1) and at high temperature of 150°C (maximum stress 130 MPa and stress ratio 1) was obtained. Rotating bending fatigue test was performed in this fatigue strength evaluation. The above-mentioned stress was repeatedly given to a round bar-shaped sample bar having a 8 mm diameter of parallel portion diameter and a 20 mm of length of parallel portion, and finished with emery paper of #1000, and a number of times of repetition which results in breakage was investigated. Table 2 shows test results. In addition, in Table 2, a number of repetition times of fracture is shown as 1.2 x 10^7 for 1.2 x 10^7 times and as 9.0 x 10^6 times.

[0059] Following items were measured as tensile characteristics: mechanical properties at room temperature (breaking load σB: MPa; 0.2% proof stress σ0.2: MPa; elongation δ: %); mechanical properties at the temperature in case of sample being exposed to a high temperature of 180°C x 100 hr as high temperature characteristics (σB; σ0.2; elongation); and 1000 hr creep rupture strength at 204°C, Charpy impact value (J/cm²). Sample bar having the shape of the round bar has a parallel portion of 10 mm x 28 mm. Table 2 shows these test results.

[0060] Three positions having interval of 100mm in longitudinal direction of a sample were measured for a distribution density [piece/μm³] of Al-V based precipitate in a structure using the above-mentioned method, and average value was obtained. Table 2 shows average distribution density measurement results of the Al-V based precipitates.

[0061] Furthermore, in each Example, and in Comparative example except 12, microstructure observation for structures of the three above-mentioned positions were conducted under each above-mentioned conditions, and Al alloy structures proved to have an equiaxial structure and a average crystal grain diameter of fixed size in a range of 50 - 500 μm, and furthermore precipitation of θ' phases on (100) plane and Ω phases on (111) plane were confirmed, respectively.

[0062] Descriptions about items which Table 1 and Table 2 shows clearly will, hereinafter, be given.

[0063] Homogenized heat treatment of no less than 15 hours is performed to Examples 1 - 6 having chemical component compositions within the range of A-C including V of the present invention, and the samples have a distribution density of 1.5 piece/μm³ of Al-V based precipitate in a forged material structure. As a result, a fatigue strength at room temperature and a fatigue strength at high temperature of no less than 8.0e6 (8.0 x 10^6) are shown, and it is clear that these are excellent in these physical properties.

[0064] However, in homogenized heat treatment time in a same alloy, when Examples giving a long time of 20 hours and Examples giving a comparatively short hour of 15 hours are compared with each other, Example 1 gives a comparatively higher distribution density of Al-V based precipitate in a forged material structure as compared with Example 2, as a result, it shows a comparatively excellent fatigue strength at high temperatures.

[0065] On the other hand, even if Comparative examples 7-10 in which short homogenized heat treatment time of 8 hours or 12 hours are adopted and alloys within a range of A-C shown in Table 1 of the present invention are used, they show notably lower distribution densities of less than 1.5 piece/μm³ of Al-V based precipitate in the forged material structure as compared with the above-mentioned Examples, which shows that a fatigue strength particularly at high temperatures is markedly inferior.

[0066] Furthermore, Comparative example 11 in which an alloy example D having a V content lower than a limit is
used shows a distribution density of less than 1.5 piece/µm³, notably lower than in the above-mentioned Example, of Al-V based precipitate in the forged material structure in spite of 20 hours of long homogenized heat treatment period, which shows a markedly inferior fatigue strength particularly at high temperatures.

[0067] Besides, in Comparative example 12 using an alloy example E having a V content higher than a limit, structure observation of the above-mentioned forged material showed coarse intermetallic compounds that was not found in other examples. Therefore, it was clear that this gave inferior mechanical characteristics, and detailed measurements and identification was not further performed.

[0068] Therefore, these results will guarantee a critical meaning in definition of distribution density for an Al-V based precipitate in a forged-material structure to a high temperature fatigue strength, and a meaning of a homogenized heat-treating period to improvement in distribution density of an Al-V based precipitate.

[Table 1]

<table>
<thead>
<tr>
<th>Chemical composition of Al alloy forged material (mass %)</th>
<th>Class.</th>
<th>Comp. Example</th>
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<tr>
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<td>0.05</td>
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<tr>
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<td>0.1</td>
</tr>
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</tr>
<tr>
<td>E</td>
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Claims

1. An Aluminum alloy forged material comprising: Cu: 4.0% - 7.0%, Mg: 0.2% - 0.4%, Ag: 0.05% - 0.7%, and V: 0.05% - 0.15%, wherein a distribution density of an Al-V based precipitate in the forged-material structure is no less than 1.5 piece/µm³.

2. An Aluminum alloy forged material obtainable by a method comprising the following steps:
   - homogenized heat treatment at temperatures of 500°C - 535°C for no less than 15-hour is performed to an Aluminum alloy cast material including Cu: 4.0% - 7.0%, Mg: 0.2% - 0.4%, Ag: 0.05% - 0.7% and V: 0.05% - 0.15%;
   - the Aluminum alloy material after the homogenized heat treatment is hot forged at temperatures of 280°C - 430°C;
   - the hot forged Aluminum alloy material is solution treated at temperatures of 510°C - 545°C; and
   - the Aluminum alloy material after the solution treatment is hardened.

Amended claims in accordance with Rule 86(2) EPC.

1. An Aluminum alloy forged material comprising: Cu: 4.0% - 7.0%, Mg: 0.2% - 0.4%, Ag: 0.05% - 0.7%, and V: 0.05% - 0.15%, and optionally: Si: no more than 0.1%, Fe: no more than 0.15%, Zr: no more than 0.09%, Cr: no more than 0.05%, Mn: no more than 0.8%, and Ti: no more than 0.1%, with the balance being Al, wherein the Aluminum alloy forged material is obtainable by a method comprising the following steps:
   - homogenized heat treatment at temperatures of 500°C - 535°C for no less than 15-hour is performed to the Aluminum alloy cast material;
   - the Aluminum alloy material after the homogenized heat treatment is hot forged at temperatures of 280°C - 430°C and at a forging ratio no less than 1.5;
   - the hot forged Aluminum alloy material is solution treated at temperatures of 510°C - 545°C; and
   - the Aluminum alloy material after the solution treatment is hardened,

wherein a distribution density of an Al-V based precipitate in the forged-material structure is no less than 1.5 piece/µm³,
and wherein an average grain diameter of the Aluminum alloy forged material is in a range of 10 to 500 µm.
### DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
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<tr>
<th>Category</th>
<th>Citation of document with indication, where appropriate, of relevant passages</th>
<th>Relevant to claim</th>
<th>CLASSIFICATION OF THE APPLICATION (Int.Cl.7)</th>
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<tr>
<td>X</td>
<td>US 5 512 112 A (CASSADA III WILLIAM A) 30 April 1996 (1996-04-30) * column 6, line 44 - line 65 * * tables 1, AND, 2 *</td>
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<td>A</td>
<td>US 4 772 342 A (POLMEAR IAN J) 20 September 1988 (1988-09-20) * column 2 - column 6 * * column 3, line 15 - line 32 *</td>
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<td>US 5 211 910 A (KRAME LAWRENCE S ET AL) 18 May 1993 (1993-05-18) See Abstract * column 10, line 34 - line 43 * * table 1 * * column 12, line 59 - line 65 * * column 13, line 8-10 *</td>
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The present search report has been drawn up for all claims.

### Place of search

MUNICH

### Date of completion of the search

26 January 2004

### Examiner

Brown, A

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**Letters and Symbols:**

- **T:** theory or principle underlying the invention
- **E:** earlier patent document, but published on, or after the filing date
- **D:** document cited in the application
- **L:** document cited for other reasons
- **A:** member of the same patent family, corresponding document

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**Categories:**

- **X:** particularly relevant if taken alone
- **Y:** particularly relevant if combined with another document of the same category
- **A:** technological background
- **O:** non-written disclosure
- **P:** intermediate document

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**Note:**

This search report is intended to provide a comprehensive overview of relevant documents. Further analysis and evaluation may be required for a thorough understanding of the context and implications of each document cited.
### Annex to the European Search Report

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on 26-01-2004.

The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

**EP 1 522 600 A1**

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For more details about this annex: see Official Journal of the European Patent Office, No. 12/82

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