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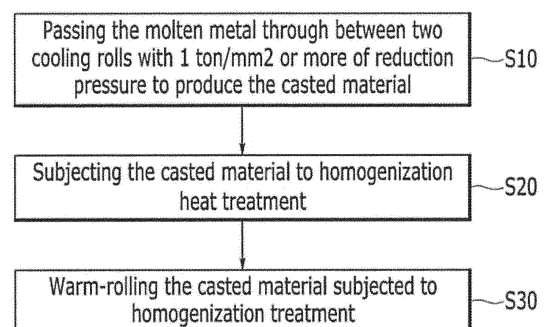
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(54) **MAGNESIUM ALLOY SHEET AND METHOD FOR MANUFACTURING SAME**

(57) A method for manufacturing a magnesium alloy sheet according to an embodiment of the present invention comprises the steps of casting a molten metal containing 2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 0.7 wt% of Ca and 1 wt% or less (Excluding 0 wt%) of Mn and consisting of the balance of Mg and inevitable impurities to produce a casted material (S10), subjecting the casted material to homogenization heat treatment (S20) and warm-rolling the casted material subjected to homogenization treatment (S30).

FIG. 1



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Description**[Technical Field]**

5 **[0001]** The invention relates to a magnesium alloy sheet, and method for manufacturing thereof.

[Background Art]

10 **[0002]** At present, the limitation of carbon dioxide emission and the importance of renewable energy are becoming the conversation topic in the international community and accordingly, lightweight alloys, which are a type of structural materials, are recognized as very attractive research fields.

[0003] In particular, magnesium is the lightest metal with a density of 1.744 g/cm³ such as aluminum and steel and has various advantages such as vibration absorbing ability and electromagnetic shielding capability as compared with other structural materials, therefore Related industry researches are being actively carried out to utilize them.

15 **[0004]** Such an alloy comprising magnesium are currently being applied not only in the field of electronic devices but also in the field of vehicle, but they have fundamental problems in corrosion resistance, flame resistance, and formability, therefore there are limitations in expanding the application range.

[0005] Especially with regard to formability, magnesium is a HCP structure (Hexagonal Closed Packed Structure), and there is not enough slip system at room temperature, making it difficult to progress. That is, a large amount of heat is required in the magnesium processing process, this leads to an increase in the cost of the processing process.

20 **[0006]** On the other hand, among the magnesium alloys, the AZ system magnesium alloys comprise aluminum (Al) and zinc (Zn), which are inexpensive and yet commercially available magnesium alloys, securing adequate strength and physical properties of ductile.

25 **[0007]** However, the physical properties mentioned above mean that it has an appropriate level among the magnesium alloys, and the strength is lower than that of aluminum (Al), which is competitive material.

[0008] Therefore, it is necessary to improve the physical properties such as low formability and strength of the AZ system magnesium alloy, but there is lack of research on this.

[DISCLOSURE]

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[Technical Problem]

[0009] The present invention is intended to provide a method of manufacturing magnesium alloy sheet with improved strength and formability.

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[Technical Solution]

40 **[0010]** A magnesium alloy sheet according to an embodiment of the present invention contains 2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 1 wt% of Ca and 1 wt% or less (Excluding 0 wt%) of Mn, and consists of the balance of Mg and inevitable impurities.

[0011] It may contain 0.3 to 0.8 wt% of Ca.

[0012] The magnesium alloy sheet alloy sheet may comprise Al-Ca secondary phase particles comprising 20 to 25% wt% of Al, 5 to 10% wt% of Ca, 0.1 to 0.5% wt% of Mn, 0.5 to 1% of Zn and the balance of Mg.

45 **[0013]** The average particle size of the Al-Ca secondary phase particles may be 0.01 to 4 μm .
the Al-Ca secondary phase particles may comprise 5 to 15 per 100 μm^2 of the magnesium alloy sheet.
the magnesium alloy sheet may comprise crystal grain and the average particle size of the crystal grain is 5 to 30 μm .
the thickness of the magnesium alloy sheet may be 0.4 to 3 mm.

50 **[0014]** A method for manufacturing a magnesium alloy sheet according to an embodiment of the present invention comprises the steps of casting a molten metal containing 2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 1 wt% of Ca and 1 wt% or less (Excluding 0 wt%) of Mn and consisting of the balance of Mg and inevitable impurities to produce a casted material; subjecting the casted material to homogenization heat treatment; and warm-rolling the casted material subjected to homogenization treatment.

[0015] In the step of producing the casted material; the reduction pressure may be 0.2 ton/mm² or more. More specifically, it may be 1 ton/mm² or more. More specifically, it may be 1 to 1.5 ton/mm² or more.

55 **[0016]** Subjecting the casted material to homogenization heat treatment may be carried out for 1 to 28 hours at a temperature of 350 to 500 °C. More specifically, it may be subjected to homogenization heat treatment for 18 to 28 hours.

[0017] Warm-rolling may be at a temperature of 150 to 350 °C. More specifically, warm-rolling may be at a temperature of 200 to 300 °C.

[0018] Warm-rolling may be performed a plurality of times, and warm-rolling may be at a reduction ratio of 10 % to 30 % per time.

[0019] It may further comprise the step of intermediate annealing in the middle of a plurality of times of warm-rolling.

[0020] The intermediate annealing may be carried out at a temperature of 300 to 500 °C. More specifically, it may be carried out at a temperature of 450 to 500 °C. More specifically, it may be carried out for 1 to 10 hours.

[0021] The step of subjecting to subsequent heat treatment may comprise further after the step of warm-rolling, The step of subjecting to subsequent heat treatment may be carried out for 1 to 10 hours at a temperature of 300 to 500 °C.

[0022] A method for manufacturing a magnesium alloy sheet according to an embodiment of the present invention, may comprise the steps of preparing a master alloy comprising 2.7 wt% or more and 5 wt% or less of Al, 0.75 wt% or more and 1 wt% or less of Zn, 0.1 wt% or more and 1 wt% or less of Ca, more than 0 wt% and 1 wt% or less of Mn and the balance of inevitable impurities and magnesium, for a total of 100 wt%; casting the master alloy to produce a casted material; subjecting the casted material to homogenization heat treatment; warm-rolling the casted material subjected to homogenization heat treatment to produce a rolled material; subjecting the rolled material to subsequent heat treatment; and carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet.

[0023] In the step of carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet; the skin pass may be carried out once, and it may be carried out at a temperature in the range of 250 °C to 350 °C.

[0024] By the step of carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet; the produced magnesium alloy sheet may be rolled at a reduction ratio of 2 to 15 % with respect to the thickness of the rolled material. More specifically, the produced magnesium alloy sheet may be rolled at a reduction ratio of 2 to 6 % with respect to the thickness of the rolled material.

[0025] The step of subjecting the casted material to homogenization heat treatment may comprise a first heat treatment step at a temperature in the range of 300°C to 400°C; and a second heat treatment step at a temperature in the range of 400 °C to 500 °C.

[0026] A first heat treatment step at a temperature in the range of 300 °C to 400 °C may be carried out for 5 to 20 hours.

[0027] A second heat treatment step at a temperature in the range of 400 °C to 500 °C may be carried out for 5 to 20 hours.

[0028] By the step of rolling the casted material subjected to homogenization heat treatment to produce a rolled material, the casted material may be rolled to a thickness of 0.4 to 3 mm.

[0029] By the step of rolling the casted material subjected to homogenization heat treatment to produce a rolled material, the casted material may be rolled 1 to 15 times.

[0030] The step of rolling the casted material subjected to homogenization heat treatment to produce a rolled material may be carried out at 150 °C to 350 °C.

[0031] By the step of subjecting the rolled material to subsequent heat treatment, the rolled material may be annealed at a temperature in the range of 300 °C to 550 °C for 1 to 15 hours.

[0032] The limiting dome height (LDH) of the magnesium alloy sheet may be 7 mm or more. More specifically, the limiting dome height (LDH) of the magnesium alloy sheet may be 8 mm or more.

[0033] A maximum aggregate strength may be 1 to 4 on the basis of the magnesium alloy sheet (0001) surface. In addition, the yield strength of the magnesium alloy sheet may be 170 to 300 MPa.

[Advantageous Effects]

[0034] According to an embodiment of the present invention, it may provide a magnesium alloy sheet that the center segregation which is likely to be generated in the conventional magnesium alloy sheet is removed and the formability is improved.

[0035] In addition, according to an embodiment of the present invention, it may provide a magnesium alloy sheet in which the texture of the magnesium alloy sheet is homogeneously dispersed, and the formability of which is improved.

[0036] In addition, according to other embodiment of the present invention, it may provide a magnesium alloy sheet that the secondary phase particle of Al-Ca system are formed in a magnesium alloy sheet and strength is improved.

[0037] A method for manufacturing a magnesium alloy sheet according to an embodiment of the present invention, it may provide a magnesium alloy sheet which is excellent in strength and formability by controlling the manufacturing process of a commercially available magnesium alloy. Because of this, it may be applied to appliances of vehicle or IT mobile devices in the future.

[Description of the Drawings]

[0038]

FIG. 1 is a schematic flowchart of a method of manufacturing a magnesium alloy sheet according to an embodiment of the present invention.

FIG. 2 is an image of scanning electron microscope (SEM) of the magnesium alloy sheet produced in Example 1.

FIG. 3 is an image of scanning electron microscope of the magnesium alloy sheet produced in Comparative Example 1.

FIG. 4 is an image of a Secondary Electron Microscopy of the magnesium alloy sheet produced in Example 1.

FIG. 5 is an image of a result of measuring the limiting dome height of the magnesium alloy sheet produced in Example 1.

FIG. 6 is a result of analysis of the crystal orientation by the XRD analyzer of the magnesium alloy sheet produced in Example 1.

FIG. 7 is a result of analysis of the crystal orientation by the XRD analyzer of the magnesium alloy sheet produced in Comparative Example 1.

FIG. 8 is an image of Electron Backscatter Diffraction (EBSD) of the magnesium alloy sheet produced in Example 1.

Fig. 9 is a result of EBSD analysis of the surface according to the reduction ratio in the skin pass process.

FIG. 10 shows the aggregate strength of the (0001) surface of Examples and Comparative Examples of the present invention.

[Mode for Invention]

[0039] The first term, second and third term, etc. are used to describe various parts, components, regions, layers and/or sections, but are not limited thereto. These terms are only used to distinguish any part, component, region, layer or section from other part, component, region, layer or section. Therefore, the first part, component, region, layer or section may be referred to as the second part, component, region, layer or section within the scope unless excluded from the scope of the present invention.

[0040] The terminology used herein is only to refer specific embodiments and is not intended to be limiting of the invention. The singular forms used herein comprise plural forms as well unless the phrases clearly indicate the opposite meaning. The meaning of the term "comprise" is to specify a particular feature, region, integer, step, operation, element and/or component, not to exclude presence or addition of other features, regions, integers, steps, operations, elements and/or components.

[0041] Although not defined differently, every term comprising technical and scientific terms used herein have the same meaning as commonly understood by those who is having ordinary knowledge of the technical field to which the present invention belongs. The commonly used predefined terms are further interpreted as having meanings consistent with the relevant technology literature and the present content and are not interpreted as ideal or very formal meanings unless otherwise defined.

[0042] In addition, % means weight% (wt%) unless otherwise referred.

[0043] Hereinafter, embodiments of the present invention will be described in detail with reference to the accompanying drawings so that those people who are having ordinary knowledge will easily carry out. The present invention may, however, be implemented in several different forms and is not limited to the embodiments described herein.

[0044] The magnesium alloy sheet according to an embodiment of the present invention contains 2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 1 wt% of Ca, 1 wt% or less of Mn (excluding 0 wt%) and consist of the balance of Mg and inevitable impurities.

[0045] Hereinafter, the reason for limiting the numerical value of the component content in the embodiment of the present invention will be described.

[0046] First, aluminum (Al) improves the mechanical properties of the magnesium alloy sheet and castability of the molten metal. If Al is added too much, the castability may deteriorate rapidly, and if too little Al is added, the problem that mechanical properties deteriorate may occur. Therefore, the content range of Al may be adjusted within the range mentioned above.

[0047] Zinc (Zn) improves the mechanical properties of the magnesium alloy sheet. If too much Zn is added, a large number of surface defects and center segregation may be generated, leading to a problem of rapidly deteriorated castability, and if too little Zn is added, the problem that mechanical properties of magnesium alloy sheet deteriorate may occur. Therefore, the content range of Zn may be adjusted within the range mentioned above.

[0048] Calcium (Ca) gives flame resistance to the magnesium alloy sheet. If too much Ca is added, the fluidity of the molten metal is reduced, the castability becomes worse, and the center segregation made of the Al-Ca system intermetallic material is generated, which may cause a problem of deteriorating the formability of the magnesium alloy sheet, and if too little is added, the problem that the flame resistance is not sufficiently given may occur. Therefore, the content range of Ca may be adjusted within the range mentioned above. More specifically, Ca may be comprised 0.3 to 0.8 % (wt%).

[0049] Manganese (Mn) improves the mechanical properties of the magnesium alloy sheet material. If too much Mn is added, the problems that the heat emission is reduced and at the same time, uniform distribution control is difficult,

may occur. Therefore, the content range of Mn may be adjusted within the above-mentioned range.

[0050] The magnesium alloy sheet according to embodiment of the present invention may comprise Al-Ca secondary phase particles comprising 20 to 25% wt% of Al, 5 to 10% wt% of Ca, 0.1 to 0.5% wt% of Mn, 0.5 to 1% of Zn and the balance of Mg. In general, when alloying by adding Al and Ca to magnesium, center segregation consisting of Al-Ca intermetallic compound is generated which significantly reduce formability. On the other hand, the magnesium alloy sheet according to an embodiment of the present invention may improve formability by comprising Al-Ca secondary phase particles. The average particle diameter of the Al-Ca secondary phase particles may be 0.01 to 4 μm . The formability may be further improved in the above-mentioned range. In addition, the Al-Ca secondary phase particles may be comprised 5 to 15 particles per 100 μm^2 square of the magnesium alloy sheet material. By comprising Al-Ca secondary phase particles in the above-mentioned scope, the formability of the magnesium alloy sheet may be further improved. In order to obtain the above-mentioned Al-Ca secondary phase particles, the composition ranges of Al, Zn, Mn, and Ca, the temperature and time conditions during the homogenization heat treatment, the temperature and the rolling rate during warm rolling are necessary to be precisely controlled.

[0051] The magnesium alloy sheet comprise crystal grain and the average particle size of the crystal grain is 5 to 30 μm . The formability may be further improved within the range mentioned above. In order to obtain crystal grain of the above-mentioned sizes, the composition scopes of Al, Zn, Mn, and Ca, the temperature and time conditions during the homogenization heat treatment, the temperature and the rolling rate during warm rolling are necessary to be precisely controlled.

[0052] In addition, the limiting dome height of the magnesium alloy sheet according to an embodiment of the present invention may be 7 mm or more. More specifically, it may be 8 mm or more, more specifically 8 to 10 mm.

[0053] In general, the limiting dome height is as an index for evaluating the formability (especially, compressibility) of the material, it means that the formability of the material is improved as such the limiting dome height increases.

[0054] The limited range mentioned above is, significantly higher limiting dome height than magnesium alloy sheet generally known, which caused by an increase in the distribution of crystal grain orientation in magnesium alloy sheet.

[0055] In addition, the thickness of the magnesium alloy sheet according to an embodiment of the present invention may be 0.4 to 3 mm.

[0056] FIG. 1 schematically shows a flow chart of a method manufacturing a magnesium alloy sheet according to an embodiment of the present invention. The flow chart of a method manufacturing a magnesium alloy sheet of FIG. 1 is only to illustrate the present invention and is not limited thereto. Therefore, the method of manufacturing a magnesium alloy sheet may be variously modified.

[0057] A method of manufacturing a magnesium alloy sheet according to an embodiment of the present invention comprises the steps of casting a molten metal containing 2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 1 wt% of Ca and 1 wt% or less (Excluding 0 wt%) of Mn and consisting of the balance of Mg and inevitable impurities to produce a casted material (S10); subjecting the casted material to homogenization heat treatment (S20); and warm-rolling the casted material subjected to homogenization treatment (S30). In addition, if necessary, the manufacturing method of the magnesium alloy sheet may further comprise other steps.

[0058] First, in the step (S10), a molten metal containing 2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 1 wt% of Ca and 1 wt% or less (Excluding 0 wt%) of Mn and consisting of the balance of Mg and inevitable impurities is casted to produce a casted material.

[0059] The reason for limiting the numerical value of the respective components is same as that described, therefore repeated descriptions will be omitted.

[0060] At this time, a method of casting the casted material are die casting, strip casting, centrifugal casting, tilting casting, sand casting, direct chill casting or a combination thereof. More specifically, strip casting may be used. However, the present invention is not limited thereto.

[0061] More specifically, in the step of producing the cast material; the reduction pressure may be 0.2 ton/mm² or more. More specifically, it may be 1 ton/mm² or more. More specifically, it may be 1 to 1.5 ton/mm² or more.

[0062] The casted material may be produced by casting. At this time, when the casted material is coagulated and reduction pressure is simultaneously applied, the formability of the magnesium alloy sheet may be improved by adjusting the reduction pressure to the range mentioned above. In the step (S20), the casted material is subjected to homogenization heat treatment. At this time, the heat treatment condition may be at a temperature of 350 to 500 °C for 1 to 28 hours. More specifically, it may be subjected to homogenization heat treatment for 18 to 28 hours. If the temperature is too low, the homogenization treatment cannot be properly performed, and there may occur a problem that beta phases such as Mg₁₇A₁₂ are not be employed at the base. If the temperature is too high, beta phases condensed the casted material may melt, resulting in a fire or resulting in holes on the magnesium sheet. Therefore, it may be subjected to homogenization heat treatment within the temperature range described above.

[0063] In the step (S30), the homogenized casted material is warm-rolled. At this time, the temperature condition of the warm-rolling may be at a temperature of 150 to 350 °C. If the temperature is too low, there may occur a problem generating large amount of edge crack. If the temperature is too high, there may occur a problem being inappropriate

for mass production. Therefore, warm-rolling may be within the above-mentioned temperature range.

[0064] The step of warm-rolling may be performed a plurality of times, and warm-rolling may be at a reduction ratio of 10 % to 30 % per time. By carrying out warm-rolling a plurality of times, finally, it may be rolled to a thin thickness of 0.4 mm.

[0065] It may further comprise at least one time of the step of intermediate annealing in the middle of a plurality of times of warm-rolling. The formability of the magnesium alloy sheet may be further improved by comprising the step of intermediate annealing. More specifically, the step of intermediate annealing may be carried out at a temperature of 300 to 500 °C for 1 to 10 hours. More specifically, it may be carried out at a temperature of 450 to 500 °C. The formability of the magnesium alloy sheet may be improved within the range mentioned above.

[0066] After the step of warm-rolling (S30), subjecting to the subsequent heat treatment may be further comprised. The formability of the magnesium alloy sheet may be improved by comprising the step of subjecting to the subsequent heat treatment. The step of subjecting to the subsequent heat treatment may be carried out at a temperature of 300 to 500 °C for 1 to 10 hours. The formability of the magnesium alloy sheet may be further improved within the above-mentioned range.

[0067] A method for manufacturing a magnesium alloy sheet according to an embodiment of the present invention may comprise the steps of preparing a master alloy comprising 2.7 wt% or more and 5 wt% or less of Al, 0.75 wt% or more and 1 wt% or less of Zn, 0.1 wt% or more and 0.7 wt% or less of Ca, more than 0 wt% and 1 wt% or less of Mn and the balance of inevitable impurities and magnesium, for a total of 100 wt%; casting the master alloy to produce a casted material; subjecting the casted material to homogenization heat treatment; warm-rolling the casted material subjected to homogenization heat treatment to produce a rolled material; subjecting the rolled material to subsequent heat treatment; and carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet.

[0068] First, in the step of preparing a master alloy comprising 2.7 wt% or more and 5 wt% or less of Al, 0.75 wt% or more and 1 wt% or less of Zn, 0.1 wt% or more and 0.7 wt% or less of Ca, more than 0 wt% and 1 wt% or less of Mn and the balance of inevitable impurities and magnesium, for a total of 100 wt%; the master alloy may be commercially available AZ31 alloy, AL5083 alloy or a combination thereof. However, it is not limited thereto.

[0069] Next, the step of casting the master alloy to produce a casted material; may be carried out.

[0070] More specifically, the master alloy may be dissolved to prepare the molten metal at the temperature range of 650 to 750 °C. Thereafter, the molten metal may be cast to produce a casted material. At this time, the thickness of the casted material may be 3 to 7 mm.

[0071] At this time, a method of casting the casted material are die casting, strip casting, centrifugal casting, tilting casting, sand casting, direct chill casting or a combination thereof. More specifically, strip casting may be used. However, it is not limited thereof.

[0072] In the step of producing the casted material, the reduction pressure may be 0.2 ton/mm² or more. More specifically, it may be 1 ton/mm² or more. More specifically, it may be 1 to 1.5 ton/mm² or more.

[0073] Thereafter, the step of subjecting the casted material to homogenization heat treatment; may be carried out.

[0074] More specifically, the step of subjecting the casted material to homogenization heat treatment may comprise the step of a first heat treatment at a temperature scope of 300 °C to 400°C; and the step of a second heat treatment at a temperature scope of 400 °C to 500 °C. More specifically, the step of first heat treatment at a temperature range of 300 °C to 400 °C may be carried out for 5 to 20 hours. In addition, the step of second heat treatment at a temperature range of 400 °C to 500 °C may be carried out for 5 to 20 hours.

[0075] By carrying out the first heat treatment at the temperature range mentioned above, The Mg-Al-Zn ternary system Pi-phase generated in the casting step may be removed. If the ternary system Pi-phase exists, the subsequent process may be adversely affected. In addition, the stress in the slab may be released by carrying out the second heat treatment step within the above-mentioned temperature range. Furthermore, it may actively induce the formation of recrystallization of the cast structure in the slab.

[0076] Thereafter, the step of warm-rolling the casted material subjected to homogenization heat treatment to produce the rolled material; may be carried out.

[0077] The heat-treated slab may be rolled to a thickness range of 0.4 to 3 mm through 1 to 15 times of rolling. In addition, the rolling may be carried out at a temperature of 150 °C to 350 °C.

[0078] More specifically, if the rolling temperature is less than 150 °C, it may induce a crack on the surface when rolling, and if it exceeds 350 °C, it may not be suitable for actual production facilities. Therefore, it may be rolled at 150 °C to 350 °C.

[0079] Next, the step of intermediate annealing the rolled material may be carried out. When it is rolled several times in the step of rolling, it may be subjected to heat treatment at the temperature range of 300 °C to 550 °C for 1 to 15 hours in the interval between the pass and the pass. For example, it may be subjected to intermediate annealing once after rolling twice, and then rolled to a final target thickness. As another example, it may be rolled to the final target thickness by annealing once after rolling three times. More specifically, in the case where the rolled casted material is

annealed in the above temperature range, the stress generated by rolling may be released. Therefore, it may be rolled several times up to the desired thickness of the casted material. Finally, the step of carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet; may be performed. More specifically, the skin pass is also known as skin pass rolling or temper rolling, which means that it removes a deformation pattern generated in a cold rolled steel sheet after heat treatment, and cold rolling with light pressure to improve the hardness.

[0080] Therefore, in an embodiment of the present invention, a skin pass may be carried out once at a temperature range of 250 °C to 350 °C. More specifically, the magnesium alloy sheet manufactured by carrying out the skin pass may be rolled at a reduction ratio of 2 to 15% with respect to the thickness of the rolled material, more specifically, may be rolled at a reduction ratio of 2 to 6 %. More specifically, in the case of rolling under the conditions of the temperature and the pressure, the development of the texture, which is a weak base surface texture (0001) is reduced, so that formability may be obtained.

[0081] In addition, the magnesium alloy sheet manufactured by the step of carrying out a skin pass to the annealed rolled material to produce a magnesium alloy sheet; may be rolled at a reduction ratio of 2 to 15 % with respect to the thickness of the rolled slab. More specifically, it may be rolled at a reduction ratio of 2 to 6%.

[0082] In the case of rolling with the reduction ratio, the strength may be improved by minimizing the change in the strength of the texture. More specifically, when the reduction ratio is 2 to 6 %, the change in the strength of the texture is the smallest and the yield strength may be 170 to 300 MPa. In addition, the value of the limiting dome height (LDH) may be 8 to 9 mm.

[0083] However, when the reduction ratio is 2 to 15 %, the yield strength may be 250 to 280 MPa, but value of the limiting dome height may be 7 to 8 mm since the texture is somewhat developed. This is because, as disclosed in FIG. 9, when rolling at a reduction ratio of 6 to 15%, a hardening occurs due to the double turning or dislocation. More specifically, when the reduction ratio is 2 to 15 %, the area fraction of the twinned crystal structure may be 5 % or less with respect to 100 % of the total area of the magnesium alloy sheet. More specifically, when the rolling is at the reduction ratio of 6 to 15 %, the area fraction of the twinned crystal structure may be 5 to 20 % with respect to 100 % of the total area of the magnesium alloy sheet. In the image of the structure disclosed in FIG. 9, a black color means twinned crystal structure, and as described above, the strength of the magnesium alloy sheet is maintained and the formability may be improved due to twinned crystal and dislocation.

[0084] Therefore, in the case of rolling with a reduction ratio greater than 15 %, the texture of the (0001) surface may be developed again and the formability may be lowered. This may be the same phenomenon that occurs when the temperature range during rolling is low. Therefore, the skin pass may be carried out under the condition of temperature range and the reduction ratio according to an embodiment of the present invention.

[0085] In addition, The term "Limit Dome Height (LDH)" is an index for evaluating the formability of a sheet, particularly pressability, and may be measured by measuring the deformed height by applying a deformation to the specimen.

[0086] More specifically, the limit dome height (LDH) according to an embodiment of the present invention is measured the distance at which punch moved until the disc-shaped specimen was fractured, that is, the height at which the specimen was deformed, by fixing a peripheral portion of a specimen having a diameter of 50 mm with a force of 10 KN and then applying a deformation at a rate of 5 to 10 mm/min using a spherical punch having a diameter of 20 mm at a room temperature.

[0087] Hereinafter, preferred examples and comparative examples of the present invention will be described. However, the following examples are merely preferred examples of the present invention, and the present invention is not limited thereto.

Example 1

[0088] A molten metal comprising 3.0 wt% of Al, 0.8 wt% of Zn, 0.6 wt% of Ca, 0.5 wt% of Mn and consisting of the balance of Mg and inevitable impurities was passed through between two cooling rolls having a reduction pressure of 1.2 ton/mm² to produce a magnesium casted material.

[0089] The magnesium alloy sheet having a final thickness of 0.7 mm was produced by carrying out subjecting the magnesium casted material to homogenization heat treatment at a temperature of 400 °C for 24 hours, warm-rolling at 250 °C at a reduction ratio of 15 %, intermediate annealing at 450 °C for 1 hour, and warm-rolling again at 250 °C at a reduction ratio of 15 %.

Comparative example 1

[0090] A magnesium alloy sheet was produced in the same manner as in Example 1, except that 3.0 wt% of Al and 0.8 wt% of Zn were comprised.

Test Example 1: Observation of microstructure that composes a magnesium alloy sheet.

[0091] The image of Scanning Electron Microscope (SEM) of the magnesium alloy sheet produced in Example 1 and Comparative Example 1 were shown in FIG. 2 and FIG. 4 respectively.

[0092] In the case of Example 1 (FIG. 2), the central segregation of the magnesium alloy sheet was hardly generated, whereas in the case of Comparative Example 1 (FIG. 3), it may be confirmed that a large amount of central segregation occurred. Such center segregation becomes a factor of being deteriorated the formability of the magnesium alloy sheet.

[0093] The image of Secondary Electron Microscopy of the magnesium alloy sheet produced in Example 1 was shown in FIG. 4.

[0094] The white dot portion in FIG. 4 is Al-Ca secondary phase particles. As a result of analyzing the white dot portion, analysis was made with 65.62 wt% of Mg, 24.61 wt% of Al, 8.75 wt% of Ca, 0.36 wt% of Mn and 0.66 wt% of Zn.

Test Example 2: Measurement of limit dome height of a magnesium alloy sheet.

[0095] The limit dome height was determined by inserting each magnesium alloy sheet of the Examples and the Comparative examples between the upper die and the lower die and fixing the outer periphery of each specimen with a force of 5 kN and the press oil was used as the lubricant. Then, a spherical punch having a diameter of 30 mm was used to deform at a rate of 5 to 10 mm/min, the punch was inserted until each specimen was fractured and then it was performed when occurring such fracture in the manner of measuring of the deformation height of each specimen.

[0096] FIG. 5 is an image of a result of measuring the limit dome height of the magnesium alloy sheet produced in Example 1.

Test Example 3: Analysis of crystal grain orientation.

[0097] The crystal orientation of each of the crystal grains of the magnesium alloy sheet produced in Example 1 and Comparative Example 1 confirmed by an XRD analyzer and was shown in FIG. 6 and FIG. 7 respectively.

[0098] In the case of Example 1 (FIG. 6), contour lines are widely spread, and it may be confirmed that the crystal orientation of the crystal grains in the sheet are presented widely and variously. Therefore, it may be confirmed that the formability of Example 1 is excellent. On the other hand, in the case of Comparative Example 1 (FIG. 7), it may be confirmed that the (0001) peak is concentrated.

[0099] An image of the EBSD of Example 1 was taken and shown in FIG. 8. As shown in , it may be known that the values of misorientation are evenly distributed in each of the crystal grains, and it may be confirmed that each of the crystal grains have various crystal orientations.

Example 2

[0100] A master alloy comprising 3 % of Al, 1 % of Zn, 1 % of Ca, 0.3 % of Mn and the balance of magnesium and inevitable impurities was prepared.

[0101] The master alloy was cast to produce a casted material. The casted material was subjected to a first homogenization heat treatment at a temperature of 350 °C for 10 hours. The casted material subjected to a first homogenization heat treatment was subjected to a secondary homogenization heat treatment at a temperature of 450 °C for 10 hours. The casted material subjected to homogenization heat treatment was rolled to produce a rolled material. Thereafter, the rolled material was subjected to subsequent heat treatment at a temperature of 400 °C for 10 hours.

[0102] Finally, skin pass was carried out to the rolled material subjected to the subsequent heat treatment to produce the magnesium alloy sheet, the temperature and the reduction ratio of execution of the skin pass are shown in Table 1 below.

Test Example 4: Test of Comparison of mechanical properties according to reduction ratio and temperature of skin pass.

[0103]

[Table 1]

	Rolling temperature (°C)	Reduction ratio(%)	Yield strength (MPa)	Maximum tensile strength (MPa)	Elongation (%)	Limit dome height (LDH, mm)
Example 2a	250	5	202	257	22	8.1
Example 2b		9	211	254	22	8.0
Example 2c		15	252	272	16	7.3
Comparative 2a		22	272	289	8.6	7.0
Comparative 2b	300	X	140	233	23	8-9
Example 2d		7	203	253	22	8
Example 2e		12	247	267	18	7.3
Comparative 2c		17	247	272	10	7.3

[0104] As shown in Table 1, the result of carrying out the skin pass to the magnesium alloy sheet having the same component and composition, the yield strength was improved without greatly changing the formability. More specifically, the formability may be compared with the numerical values of the elongation and the limit dome height.

[0105] In addition, the formability was secured by minimizing the change in the aggregate strength, and the aggregate strength is as disclosed in FIG. 10 herein.

[0106] FIG. 10 shows the aggregate strength of (0001) surface of the Examples and Comparative Examples of the present invention.

[0107] As disclosed in FIG. 10, in the case of Comparative Examples 2a and 2c, it may be seen that the yield strength increases significantly as the change in the strength of the texture increases. However, it may be seen that the formability is somewhat reduced as the elongation is rapidly lowered.

[0108] Therefore, as disclosed in Table 1 and FIG. 10. It was confirmed that the present invention minimizes the change in strength of the texture to secure the formability.

Example 3

[0109] As compared with Example 1, a magnesium alloy sheet material was produced under different conditions as disclosed in Table 2 below. As a result, the mechanical properties of the magnesium alloy sheet produced in Example 3 are disclosed in Table 3 below.

[Table 2]

	Al content (wt%)	Ca content (wt%)	Cast roll reduction pressure (mm/ton)	Homogenization annealing time (hr)	Rolling temperature (°C)	Intermediate annealing temperature(°C)
Example 3a	3	0.6	1.2	24	250	450
Example 3b	4	0.6	1.2	24	250	450
Example 3c	5	0.6	1	24	250	450
Example 3d	3	0.6	1.2	24	250	300
Example 3e	3	0.6	1.2	24	250	400
Example 3f	3	0.6	1.2	24	250	500
Example 3g	3	0.7	0.2	24	250	500
Example 3h	3	0.7	1.2	24	250	450
Example 3i	3	0.6	1	1	250	400

(continued)

	Al content (wt%)	Ca content (wt%)	Cast roll reduction pressure (mm/ton)	Homogenization annealing time (hr)	Rolling temperature (°C)	Intermediate annealing temperature(°C)
Comparative 3a	3	0.6	0.8	24	250	None
Comparative 3b	3	0.7	1.2	24	400	250
Comparative 3c	3	0.7	1	48	250	400
Comparative 3d	3	0.7	0.8	24	100	400

[Table 3]

	Crystal grain size (μm)	Sheet thickness (mm)	Yield strength (Mpa)	Limit dome height (LDH, mm)
Example 3a	19	0.7	164	9.4
Example 3b	7	0.6	161	8.2
Example 3c	6	1	166	8.1
Example 3d	13	1	155	7.5
Example 3e	21	1	157	8
Example 3f	25	1	154	9.9
Example 3g	16	0.7	151	9
Example 3h	15	3	155	9.1
Example 3i	17	1	164	9
Comparative 3a	10	0.7	188	2.5
Comparative 3b	11	0.6	155	5
Comparative 3c	40	1.5	145	5.1
Comparative 3d	8	1	166	4.9

[0110] As a result, in the case of Comparative Examples 3a to 3d which did not satisfy the conditions of the homogenization annealing time, the rolling temperature and the intermediate annealing temperature, it was confirmed that the formability was inferior to the example of the present invention. In addition, the yield strength is also inferior to the example herein. In the case of comparative Example 3c, the size of the crystal grain was about $40\mu\text{m}$, which was relatively superior to the other comparative examples but was less than examples of the present invention.

[0111] The present invention is not limited to the above-mentioned examples or embodiments and may be manufactured in various forms, those who have ordinary knowledge of the technical field to which the present invention belongs may understand that it may be carried out in different and concrete forms without changing the technical idea or fundamental feature of the present invention. Therefore, the above-mentioned examples or embodiments are illustrative in all aspects and not limitative.

Claims

1. A magnesium alloy sheet containing 2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 1 wt% of Ca and 1 wt% or less (Excluding 0 wt%) of Mn and consisting of the balance of Mg and inevitable impurities, wherein the area fraction of the twin structure is 5 % or less with respect to 100 % of the area of the magnesium

alloy sheet.

2. A magnesium alloy sheet according to claim 1,
containing 0.3 to 0.8 wt% of Ca.
3. A magnesium alloy sheet according to claim 1,
wherein the magnesium alloy sheet comprises Al-Ca secondary phase particles comprising 20 to 25 wt% of Al, 5
to 10 wt% of Ca, 0.1 to 0.5 wt% of Mn, 0.5 to 1 wt% of Zn and the balance of Mg.
4. A magnesium alloy sheet according to claim 3,
wherein the average particle size of the Al-Ca secondary phase particles is 0.01 to 4 μm .
5. A magnesium alloy sheet according to claim 3,
wherein the Al-Ca secondary phase particles comprises 5 to 15 per 100 μm^2 of the magnesium alloy sheet.
6. A magnesium alloy sheet according to claim 1,
wherein the magnesium alloy sheet comprises grains and the average particle size of the grains is 5 to 30 μm .
7. A magnesium alloy sheet according to claim 1,
wherein the thickness of the magnesium alloy sheet is 0.4 to 2 mm.
8. A method for manufacturing a magnesium alloy sheet comprising the steps of casting a molten metal containing
2.7 to 5 wt% of Al, 0.75 to 1 wt% of Zn, 0.1 to 1 wt% of Ca and 1 wt% or less (Excluding 0 wt%) of Mn and consisting
of the balance of Mg and inevitable impurities to produce a casted material;
subjecting the casted material to homogenization heat treatment; and
warm-rolling the casted material subjected to homogenization treatment.
9. The method for manufacturing a magnesium alloy sheet of claim 8,
wherein the step of producing the casted material,
the reduction pressure is 0.2 ton/mm² or more.
10. The method for manufacturing a magnesium alloy sheet of claim 8,
wherein subjecting the casted material to homogenization heat treatment is carried out for 1 to 28 hours at a
temperature of 350 to 500 °C.
11. The method for manufacturing a magnesium alloy sheet of claim 8,
wherein warm-rolling is at a temperature of 150 to 350 °C.
12. The method for manufacturing a magnesium alloy sheet of claim 8,
wherein warm-rolling is performed a plurality of times and rolling is at a reduction ratio of 10 % to 30 % per time.
13. The method for manufacturing a magnesium alloy sheet of claim 12,
further comprising the step of one or more times of intermediate annealing in the middle of a plurality of times of
warm-rolling.
14. The method for manufacturing a magnesium alloy sheet of claim 13,
wherein the intermediate annealing is carried out for 1 to 10 hours at a temperature of 300 to 500 °C.
15. The method for manufacturing a magnesium alloy sheet of claim 8,
further comprising the step of subjecting to subsequent heat treatment after the step of warm-rolling.
16. The method for manufacturing a magnesium alloy sheet of claim 15,
wherein the step of subjecting to subsequent heat treatment is carried out for 1 to 10 hours at a temperature of 300
to 500 °C.
17. A method for manufacturing a magnesium alloy sheet comprising the steps of preparing a master alloy comprising
2.7 wt% or more and 5 wt% or less of Al, 0.75 wt% or more and 1 wt% or less of Zn, 0.1 wt% or more and 1 wt%
or less of Ca, more than 0 wt% and 1 wt% or less of Mn and the balance of inevitable impurities and magnesium,

for a total of 100 wt%;
 casting the master alloy to produce a casted material;
 subjecting the casted material to homogenization heat treatment;
 warm-rolling the casted material subjected to homogenization heat treatment to produce a rolled material;
 5 subjecting the rolled material to subsequent heat treatment; and
 carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet.

10 **18.** The method for manufacturing a magnesium alloy sheet of claim 17,
 wherein carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet,
 wherein the skin pass is carried out once.

15 **19.** The method for manufacturing a magnesium alloy sheet of claim 18,
 wherein carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet,
 wherein the skin pass is carried out at a temperature in the range of 250 °C to 350 °C.

20 **20.** The method for manufacturing a magnesium alloy sheet of claim 19,
 wherein by the step of carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet,
 the produced magnesium alloy sheet is rolled at a reduction ratio of 2 to 15 % with respect to the thickness of the rolled material.

25 **21.** The method for manufacturing a magnesium alloy sheet of claim 20,
 wherein by the step of carrying out a skin pass to the rolled material subjected to subsequent heat treatment to produce a magnesium alloy sheet,
 the produced magnesium alloy sheet is rolled at a reduction ratio of 2 to 6 % with respect to the thickness of the rolled material.

30 **22.** The method for manufacturing a magnesium alloy sheet of claim 17,
 wherein the step of subjecting the casted material to homogenization heat treatment comprising
 a first heat treatment step at a temperature in the range of 300°C to 400°C; and
 a second heat treatment step at a temperature in the range of 400°C to 500°C

35 **23.** The method for manufacturing a magnesium alloy sheet of claim 22,
 wherein a first heat treatment step at a temperature in the range of 300 °C to 400 °C
 is carried out for 5 to 20 hours.

40 **24.** The method for manufacturing a magnesium alloy sheet of claim 23,
 wherein a second heat treatment step at a temperature in the range of 400°C to 500°C
 is carried out for 5 to 20 hours.

45 **25.** The method for manufacturing a magnesium alloy sheet of claim 17,
 wherein by the step of rolling the casted material subjected to homogenization heat treatment to produce a rolled material,
 wherein the casted material is rolled to a thickness of 0.4 to 3 mm.

50 **26.** The method for manufacturing a magnesium alloy sheet of claim 25,
 wherein by the step of rolling the casted material subjected to homogenization heat treatment to produce a rolled material,
 the casted material is rolled 1 to 15 times.

55 **27.** The method for manufacturing a magnesium alloy sheet of claim 26,
 wherein the step of rolling the casted material subjected to homogenization heat treatment to produce a rolled material
 is carried out at 150 °C to 350 °C.

28. The method for manufacturing a magnesium alloy sheet of claim 17,

wherein by the step of subjecting the rolled material to subsequent heat treatment, the rolled material is annealed at a temperature in the range of 300 °C to 550 °C.

5 **29.** The method for manufacturing a magnesium alloy sheet of claim 28,
wherein by the step of subjecting the rolled material to subsequent heat treatment,
the rolled material is annealed for 1 to 15 hours.

10 **30.** The method for manufacturing a magnesium alloy sheet according to any one of claims 17 to 29,
wherein the limiting dome height(LDH) of the magnesium alloy sheet is 7 mm or more.

15 **31.** The method for manufacturing a magnesium alloy sheet according to any one of claims 17 to 29,
wherein the limiting dome height(LDH) of the magnesium alloy sheet is 8 mm or more.

20 **32.** The method for manufacturing a magnesium alloy sheet according to any one of claims 17 to 29,
wherein a maximum aggregate strength is 1 to 4 on the basis of the magnesium alloy sheet (0001) surface.

25 **33.** The method for manufacturing a magnesium alloy sheet according to any one of claims 17 to 29,
wherein

FIG. 1

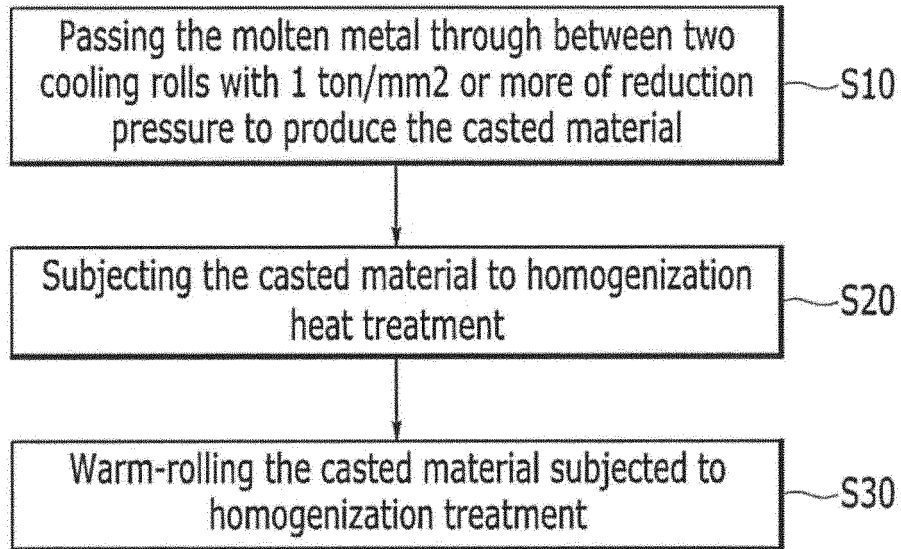


FIG. 2



FIG. 3

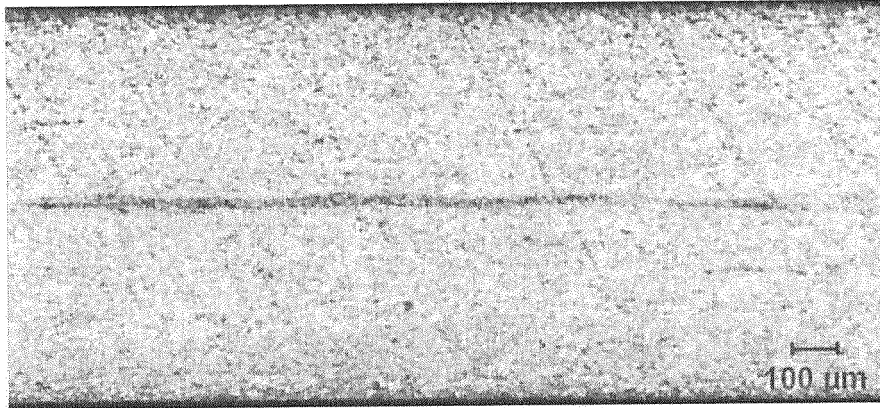


FIG. 4

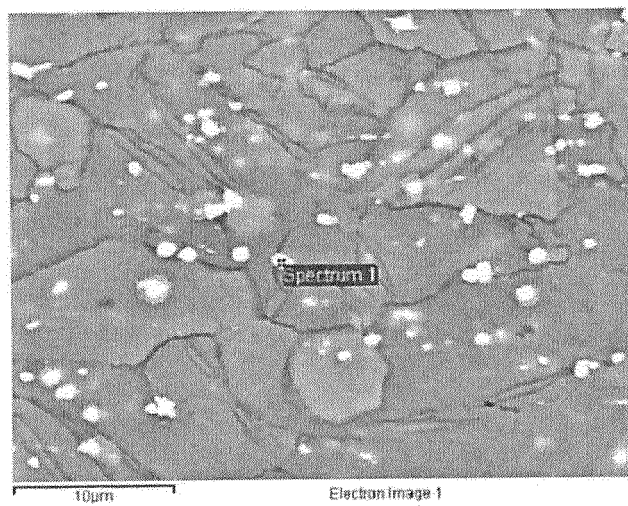


FIG. 5

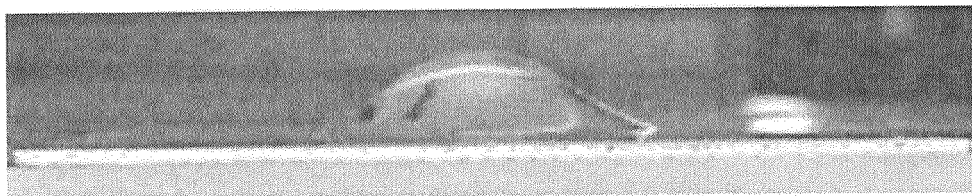


FIG. 6

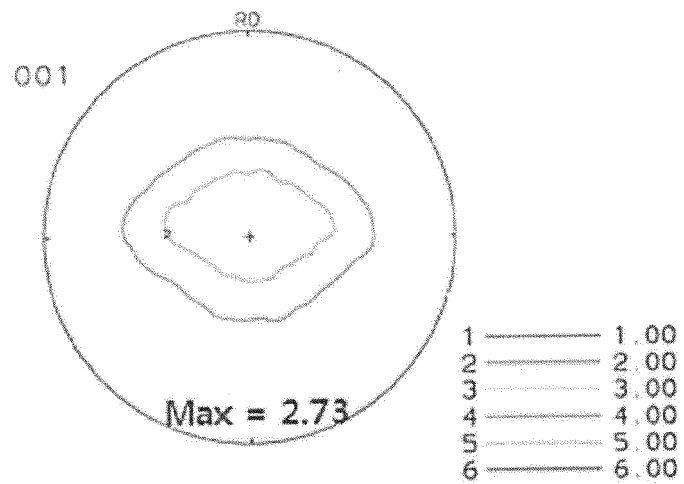


FIG. 7

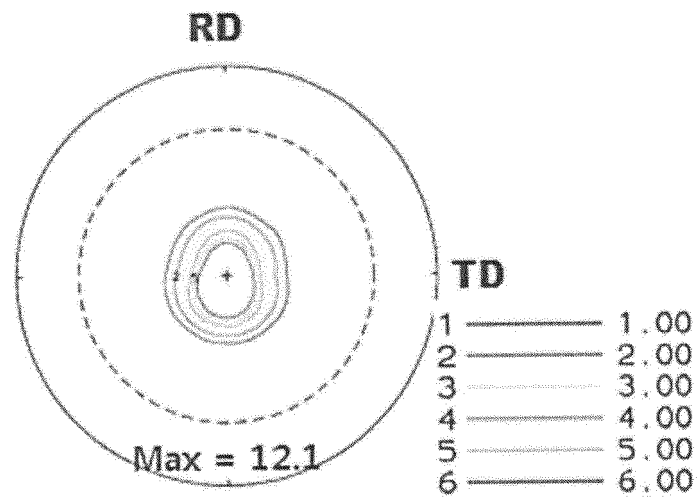
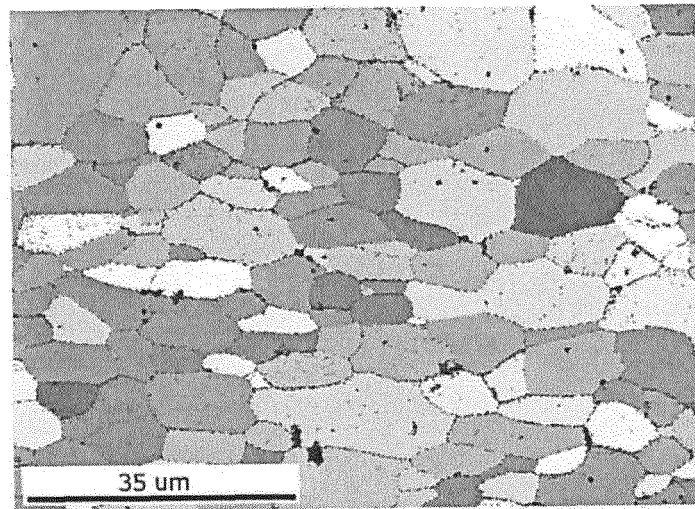


FIG. 8

<a>



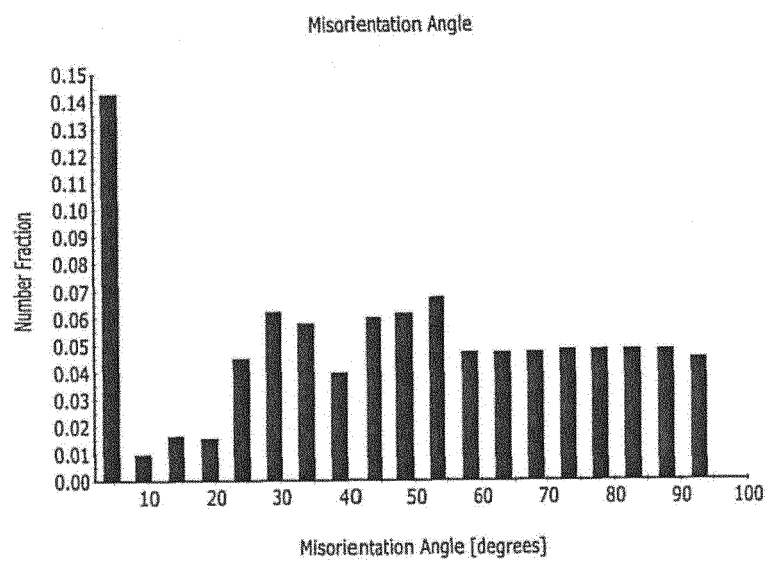


FIG. 9

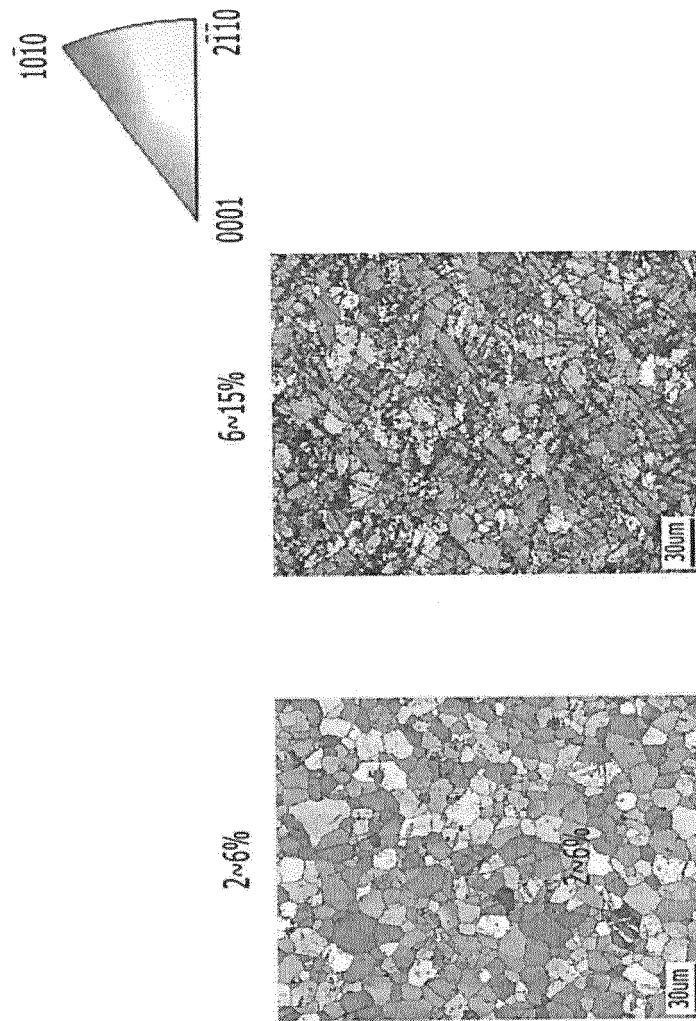
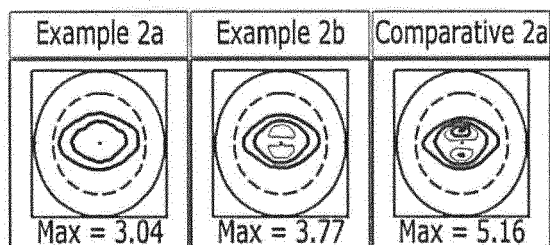
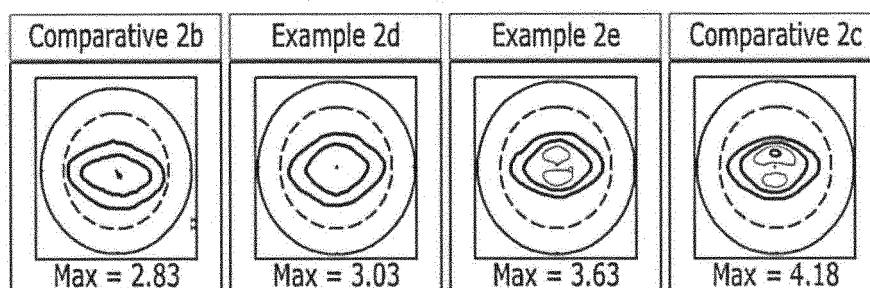


FIG. 10

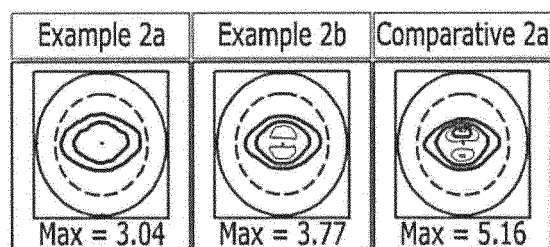
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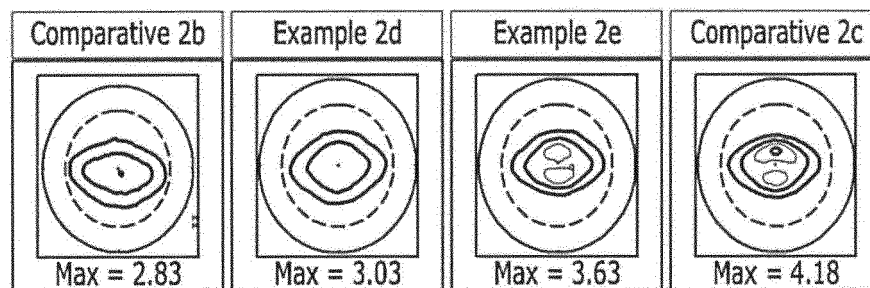
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<Rolling at a temperature of 250°C>



<Rolling at a temperature of 300°C>



INTERNATIONAL SEARCH REPORT

International application No.

PCT/KR2016/015228

A. CLASSIFICATION OF SUBJECT MATTER

B21B 3/00(2006.01)i, B21B 1/46(2006.01)i, B21B 37/16(2006.01)i, B21B 37/74(2006.01)i, B21B 1/42(2006.01)i, C22C 38/06(2006.01)i, C22C 38/04(2006.01)i, C22C 38/00(2006.01)i, C21D 8/02(2006.01)i, C22C 23/00(2006.01)i
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)

B21B 3/00; C22C 23/02; C22F 1/06; B22D 21/04; B21B 1/46; B22D 11/06; B21B 37/16; B21B 37/74; B21B 1/42; C22C 38/06; C22C 38/04; C22C 38/00; C21D 8/02; C22C 23/00

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

Korean Utility models and applications for Utility models: IPC as above

Japanese Utility models and applications for Utility models: IPC as above

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

eKOMPASS (KIPO internal) & Keywords: magnesium alloy, homogenization, warm rolling, skin pass, crystal grain, secondary phase, reduction ratio and annealing

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	KR 10-2015-0065418 A (POSCO) 15 June 2015 See paragraphs [0023], [0055], [0073] and claims 1-13.	8,11-16
Y		1-7,9-10,17-33
Y	JP 2010-229467 A (HITACHI METALS LTD.) 14 October 2010 See paragraphs [0017], [0026] and claims 1-2.	1-7,10,17-33
Y	KR 10-1481909 B1 (POSCO) 13 January 2015 See claim 1 and figure 3.	9
A	KR 10-1078308 B1 (POSTECH ACADEMY-INDUSTRY FOUNDATION) 31 October 2011 See claims 1-3.	1-33
A	JP 2009-007606 A (MITSUBISHI ALUM CO., LTD.) 15 January 2009 See claims 1, 6.	1-33

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

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Date of the actual completion of the international search

30 MARCH 2017 (30.03.2017)

Date of mailing of the international search report

30 MARCH 2017 (30.03.2017)

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/KR2016/015228

Patent document cited in search report	Publication date	Patent family member	Publication date
KR 10-2015-0065418 A	15/06/2015	KR 10-1626820 B1	02/06/2016
JP 2010-229467 A	14/10/2010	NONE	
KR 10-1481909 B1	13/01/2015	NONE	
KR 10-1078308 B1	31/10/2011	KR 10-2009-0120194 A	24/11/2009
JP 2009-007606 A	15/01/2009	JP 05158675 B2	06/03/2013