



(11) **EP 1 897 638 A1**

(12) **EUROPEAN PATENT APPLICATION**
published in accordance with Art. 153(4) EPC

(43) Date of publication:
12.03.2008 Bulletin 2008/11

(21) Application number: **06766721.2**

(22) Date of filing: **14.06.2006**

(51) Int Cl.:
B22F 1/00 (2006.01) **B21C 23/01** (2006.01)
B22F 3/20 (2006.01) **B22F 9/04** (2006.01)
C22C 1/04 (2006.01) **C22C 23/02** (2006.01)
C22F 1/06 (2006.01)

(86) International application number:
PCT/JP2006/311952

(87) International publication number:
WO 2006/134980 (21.12.2006 Gazette 2006/51)

(84) Designated Contracting States:
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR
HU IE IS IT LI LT LU LV MC NL PL PT RO SE SI
SK TR**

(30) Priority: **16.06.2005 JP 2005176410**

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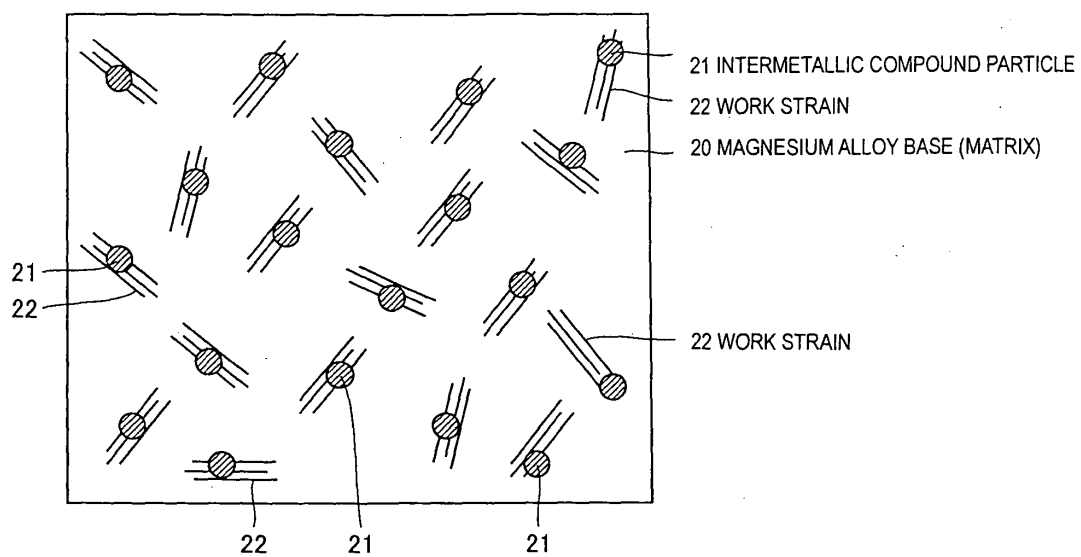
(54) **RAW MAGNESIUM-ALLOY POWDER MATERIAL, MAGNESIUM ALLOY WITH HIGH PROOF STRESS, PROCESS FOR PRODUCING RAW MAGNESIUM-ALLOY POWDER MATERIAL, AND PROCESS FOR PRODUCING MAGNESIUM ALLOY WITH HIGH PROOF STRESS**

(57) A raw magnesium alloy powder material having a relatively small crystal grain diameter is obtained by subjecting a starting material powder having a relatively large crystal grain diameter to a plastic working in which the powder is passed through a pair of rolls to undergo compressive deformation or shear deformation. The starting material powder is a magnesium alloy powder having a fine intermetallic compound (21) precipitated and dispersed in a base (22) by a heat treatment. A work

strain (22) is formed around the precipitated intermetallic compound (21) in the magnesium alloy powder after processed by the plastic working. The magnesium alloy powder after processed by the plastic working has a maximum size of 10mm or less and a minimum size of 0.1mm or more, and the magnesium particle constituting the base (20) has a maximum crystal grain diameter of 20 μm or less.

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FIG. 2



Description

TECHNICAL FIELD

5 **[0001]** The present invention relate to a raw magnesium alloy powder material and a magnesium alloy manufactured from the raw powder material and their manufacturing methods and more particularly, to a magnesium alloy implementing both high proof stress and elongation and its manufacturing method.

BACKGROUND ART

10 **[0002]** A magnesium (referred to as Mg hereinafter) alloy that is lightest among industrial metal materials is widely used for sports products, household electrical goods, aerospace devices and other machine components due to its light characteristics. Meanwhile, when the Mg alloy is applied to a product and a member that require high reliability, such as a car component, it needs to be further strengthened. Especially, improvement in proof stress is strongly required because it is essential in view of design of the component, and at the same time high elongation (toughness) also has to be implemented. In other words, when both high proof stress and high elongation are implemented, it can be replaced with an aluminum alloy that is used as a light material at the present.

15 **[0003]** It has been already known that miniaturization of a crystal grain and high dispersion of a fine intermetallic compound are effective in improving the strength of the Mg alloy. Especially, a manufacturing method in which Mg alloy powder as a starting material is compacted and solidified can form a fine texture as compared with a dissolving/casting method, so that it can be an effective manufacturing process to increase its strength.

20 **[0004]** For example, there is proposed a manufacturing method of a Mg alloy with high strength using a rapid solidification process, but this method is not practical for the following reasons.

25 (A) Although high strength can be provided, its elongation is as low as several %.

(B) Since the grain diameter of the starting material powder is as small as several tens to several hundred of microns, there is a safety problem in a handling process, a problem due to low yield and an economical problem due to high cost because an expensive element is added.

30 **[0005]** Meanwhile, various kinds of methods of manufacturing the Mg alloy in which powder cut from the Mg alloy material as a starting material are compacted and solidified have been studied and proposed. For example, Japanese Unexamined Patent Publication No. 2-182806 discloses a method in which Mg alloy cuttings are solidified by hot pressing and extruded, and Japanese Unexamined Patent Publication No. 5-320715 discloses a method in which Mg alloy cuttings are molded and extruded.

35 **[0006]** In addition, according to a "manufacturing method of magnesium alloy member" disclosed in Japanese Unexamined Patent Publication No. 5-306404, Mg alloy powder containing aluminum that was T6 heat-treated (solution heat treatment + aging heat treatment) is compacted and then extruded. This method disclosed here is characterized in that a Mg alloy member superior in mechanical characteristics can be manufactured by making use of both effects of the T6 heat treatment and the extrusion process at the time of solidification of the Mg alloy cuttings containing appropriate amount of aluminum (Al) are solidified. The effect of the T6 heat treatment is such that fine intermetallic compound $Mg_{17}Al_{12}$ is uniformly dispersed in the base of the extruded Mg alloy, and the effect of the extrusion process is such that the crystal grain constituting the base of the extruded Mg alloy is miniaturized. As a result, it is reported that according to the Mg alloy manufactured in such a manner that an AZ80 magnesium alloy having a composition of Al: 7.8 to 9.2% by weight, Mn (manganese) 0.12 to 0.35% by weight, Zn (Zinc): 0.2 to 0.8% by weight and the balance of Mg as defined by ASTM standard, for example is T6 heat-treated and cut into powder and the powder is molded and extruded, it has a tensile strength of 382MPa and an elongation of 27%, while in the case where the T6 heat treatment is not performed, the tensile strength is 330MPa and the elongation is 15%, so that it can be confirmed that the tensile strength is improved.

45 **[0007]** However, according to the manufacturing method of the Mg alloy member disclosed in the Japanese Unexamined Patent Publication No. 5-306404, it is reported that the proof stress of the extruded material is 196MPa when the T6 heat treatment is performed and it is 200MPa when the T6 heat treatment is not performed, so that it cannot be confirmed that the tensile proof stress is improved. This is attributed to the fact that although the crystal grain is miniaturized due to recrystallization at the time of extruding as compared with the crystal grain (50 to 700 μm) of the Mg alloy manufactured by the conventional dissolving/casting method, the size is still 10 to 20 μm according to the data disclosed at the moment. In order to improve the tensile proof stress, the crystal grain has to be more miniaturized. Based on the disclosed data at the moment, to miniaturize the grain size to 1 to 5 μm , or less than that is effective in improving the proof stress. The Mg alloy having such fine crystal grain diameter cannot be manufactured only by the method in which the T6-treated powder cuttings are compacted and extruded.

50 **[0008]** In addition, although there is proposed another method in which an intermetallic compound $Mg_{17}Al_{12}$ precipitated

and dispersed in the base by the T6 heat treatment is further miniaturized by extruding, the miniaturizing level implemented by plastic deformation through extruding is limited, and in order to improve the proof stress, it is necessary to further miniaturize the intermetallic compound and to miniaturize and uniformly disperse a plurality of intermetallic compounds.

5 DISCLOSURE OF THE INVENTION

[0009] It is an object of the present invention to provide a magnesium alloy implementing both high proof stress and elongation and its manufacturing method.

[0010] It is another object of the present invention to provide a raw magnesium alloy powder material used in manufacturing the above magnesium alloy and its manufacturing method.

[0011] A raw magnesium alloy powder material according to the present invention has a relatively small crystal grain diameter obtained by subjecting a starting material powder having a relatively large crystal grain diameter to a plastic working in which the powder is passed through a pair of rolls to undergo compressive deformation or shear deformation, and it is characterized by the following things. That is, the starting material powder is a magnesium alloy powder having a fine intermetallic compound precipitated and dispersed in a base by a heat treatment. A work strain is formed around the precipitated intermetallic compound in the magnesium alloy powder after processed by the plastic working. The magnesium alloy powder after processed by the plastic working has a maximum size of 10mm or less and a minimum size of 0.1mm or more. A magnesium particle constituting the base of the magnesium alloy powder after processed by the plastic working has a maximum crystal grain diameter of 20 μm or less.

[0012] Preferably, the intermetallic compound is at least one compound selected from a group consisting of $\text{Mg}_{17}\text{Al}_{12}$, Al_2Ca , Mg_2Si , MgZn_2 , Al_3Re (Re: rare-earth element), $\text{Al}_{11}\text{Re}_3$, and Al_6Mn . In addition, the intermetallic compound preferably has a maximum grain diameter of 5 μm or less and more preferably 2 μm or less.

[0013] Preferably, the magnesium particle constituting the base of the magnesium alloy powder has a maximum crystal grain diameter of 10 μm or less.

[0014] A magnesium alloy with high proof stress according to the present invention is provided by compacting and extruding the raw magnesium alloy powder material having the above characteristics, and it is characterized by the following things. That is, a magnesium particle constituting an alloy base has a maximum crystal grain diameter of 10 μm or less, and its tensile proof stress is 250MPa or more at room temperature.

[0015] Preferably, the magnesium particle constituting the magnesium alloy base has a maximum crystal grain diameter of 5 μm or less, and its tensile proof stress is 350MPa or more at room temperature.

[0016] Preferably, at least one intermetallic compound selected from a group comprising $\text{Mg}_{17}\text{Al}_{12}$, Al_2Ca , Mg_2Si , MgZn_2 , Al_3Re (Re: rare-earth element), $\text{Al}_{11}\text{Re}_3$, and Al_6Mn is precipitated and dispersed in the base of the magnesium alloy.

[0017] Preferably, the magnesium alloy contains 0.5% to 4% by weight of a metal element selected from a group consisting of strontium (Sr), zirconium (Zr), scandium (Sc) and titanium (Ti).

[0018] A manufacturing method of a raw magnesium alloy powder material according to the present invention is for miniaturizing a crystal grain diameter of a magnesium particle constituting a base of a starting material powder by performing a plastic working on the starting material powder, and it is characterized by the following things. That is, a magnesium alloy powder having a fine intermetallic compound precipitated and dispersed by a heat treatment in its base is prepared as the starting material powder. The plastic working is performed to provide work strains around the intermetallic compound by passing the starting material powder through a pair of rolls to make it undergo compressive deformation or shear deformation. The plastic working is repeated until the maximum size and minimum size of the powder become 10mm or less and 0.1mm or more, respectively, and the maximum crystal grain diameter of the magnesium particle constituting the base of the powder becomes 20 μm or less.

[0019] According to one embodiment, the step of preparing the magnesium alloy powder as the starting material powder comprises a step of manufacturing a magnesium alloy ingot by casting, a step of precipitating and dispersing fine intermetallic compound in the base of the ingot by performing a solution treatment and then aging heat treatment for the magnesium alloy ingot, and a step of obtaining the magnesium alloy powder from the ingot by machining.

[0020] A manufacturing method of a magnesium alloy with high proof stress according to the present invention comprises a step of obtaining a compact by compacting the raw magnesium alloy powder material having the above characteristics and filled in a die, a step of heating the magnesium alloy powder compact to 150 to 450°C, and a step of manufacturing a magnesium alloy by extruding the magnesium alloy compact immediately after the heating step.

[0021] Preferably, a magnesium particle constituting the base of the magnesium alloy has a maximum crystal grain diameter of 10 μm or less, and its tensile proof stress is 250MPa or more at room temperature. In addition, more preferably, the magnesium alloy compact is heated at 200 to 350°C.

BRIEF DESCRIPTION OF DRAWINGS

[0022]

Fig. 1 is a schematic view showing a roller compactor;

Fig. 2 is a schematic view showing that work strains are provided around an intermetallic compound;

Fig. 3 is structure photographs of an AZ91D ingot, in which (a) shows a structure photograph after casting, (b) shows a structure photograph after a solution heat treatment, and (c) shows a structure photograph after a T6 heat treatment (solution treatment + aging heat treatment).

Fig. 4 is an enlarged photograph of the structure after the T6 heat treatment;

Fig. 5 is structure photographs of AZ91D powder processed by plastic working with rolls, in which (a) shows a structure after the T6 treatment and (b) shows a structure after the solution treatment;

Fig. 6 is a view showing a test result of microhardness (Vickers hardness) of powder;

Fig. 7 is structure photographs of an extruded material taken by an optical microscope, in which (a) shows a structure photograph in a case where the T6 heat-treated AZ91D powder is used and (b) shows a structure photograph in a case where the solution-treated AZ91D powder is used;

Fig. 8 is structure photographs of Mg alloys in their extruded direction, manufactured by extruding and solidifying powder processed by the plastic workings with rolls 3, 10, 20 and 30 times; and

Fig. 9 is pole figures showing a result of evaluation on orientation of the basal plane of magnesium.

BEST MODE FOR CARRYING OUT THE INVENTION

[0023] Embodiments and effects of the present invention will be described hereinafter.

[0024] The present invention was made in order to solve the above conventional problems and it provides a Mg alloy having a high tensile proof stress exceeding 250 to 350MPa, and manufactured in such a manner that a magnesium alloy powder having a fine intermetallic compound precipitated and dispersed in its base by a heat treatment, as a starting material is processed by plastic working in which it is passed through a pair of rolls to undergo compressive deformation or shear deformation to manufacture coarse Mg alloy powder having a fine structure and this Mg alloy powder is compacted and extruded, and its manufacturing method.

(1) Starting material powder and its manufacturing method

[0025] A Mg alloy ingot to which an element that forms an intermetallic compound having Mg as a main component and Al, Mn, Zn, Re (rare-earth element), Ca, Si and the like as other components, and an active metal element selected from a group consisting of strontium (Sr), zirconium (Zr), scandium (Sc) and titanium (Ti) are added is manufactured by casting. By performing a well-known T6 heat treatment (solution heat treatment + aging heat treatment) on the Mg alloy ingot, the fine intermetallic compound formed by the added elements is precipitated and dispersed in the base. The precipitated and dispersed intermetallic compound includes $Mg_{17}Al_{12}$, Al_2Ca , Mg_2Si , $MgZn_2$, Al_3Re (Re: rare-earth element), $Al_{11}Re_3$, and Al_6Mn . Since these intermetallic compounds are uniformly dispersed in the base of the extruded Mg alloy, they contribute to the improvement in proof stress. In addition, the ingot can be further strengthened because it contains 0.5% to 4% by weight of the active metal element such as strontium (Sr), zirconium (Zr), scandium (Sc), and titanium (Ti).

[0026] Since a heat treatment condition is determined by the kind of the added element and its added amount, it is necessary to set an appropriate condition by observing its structure and measuring its hardness (age hardening curve). Then, powder having a size of 0.1 to 10mm is manufactured from the Mg alloy ingot by machining and cutting such as milling, and this becomes the starting material powder of the present invention. In addition, when the grain diameter of the powder is less than 0.1mm, since it is likely to be ignited, the cut powder preferably has a diameter of 0.1mm or more, and more preferably has a diameter of 0.5mm or more in view of safety.

(2) Raw magnesium alloy powder material and its manufacturing process

[0027] The Mg alloy powder that underwent the above T6 heat treatment is used as the starting material powder and poured into a roller compactor shown in Fig. 1.

[0028] The roller compactor shown in Fig. 1 comprises a case 11, a multistage rolling body 12 provided in the case 11, a crusher 13, a powder temperature/supply amount control system 14, and a receiver table 15. The multi-stage rolling body 12 constitutes a plastic working unit for performing the plastic working on the starting material powder and has three pairs of rolls 12a, 12b and 12c for rolling. The starting material powder undergoes compressive deformation and/or shear deformation when it is passed through the pair of rolls.

[0029] The starting material powder is poured into the case 11 after its temperature and amount are adjusted to a predetermined temperature and amount by the powder temperature/supply amount control system 14. Here, the predetermined temperature is to be not more than the temperature of the aging heat treatment as will be described below. An inert gas atmosphere, non-oxygenated gas atmosphere, or vacuum atmosphere is provided in the case 11 to prevent oxidation on a powder surface. In addition, it is to be noted that the surface temperature of the multistage rolling body 12 and the atmosphere temperature in the case 11 are not more than the temperature of the aging heat treatment as will be described below.

[0030] The powder passed through the roller pair 12c is crushed by the crusher 13 and becomes granular powder. This granular powder may be returned to the powder temperature/supply amount control system 14 again to undergo the plastic working by the multistage rolling body 12. The processed granular powder is collected in the receiver table 15.

[0031] The following structures are provided through the plastic working in which the powder is passed through the pair of rolls to undergo the compressive deformation and/or shear deformation.

(a) As shown in Fig. 2, many work strains 22 are provided around an intermetallic compound particle 21 precipitated and dispersed in the base of a magnesium alloy powder 20. These working strains 22 are twin crystals and dislocations provided and accumulated around the intermetallic compound particle 21 through the plastic working, and look like streak lines when observed by a transmission electron microscope (TEM).

(b) The powder that has undergone the plastic working has a maximum size of 10mm or less and a minimum size of 0.1mm or more.

(c) The powder has a crystal grain diameter relatively smaller than that of the magnesium of the starting material powder.

(d) A magnesium particle constituting the base of the powder has a maximum crystal grain diameter of 20 μm or less.

[0032] In addition, according to need, the raw Mg alloy powder material having the fine structure defined by the present invention may be manufactured by repeating the plastic working with the rolls under the same condition again after the raw Mg alloy powder material has undergone the plastic working with the rolls and then the crushing/grinding/granulating process.

[0033] First, regarding (a), when the plastic working is performed by passing the powder through the rolls, although the work strain is provided in the whole powder, since the intermetallic compound particles are precipitated and dispersed in the base, many work strains are provided around the intermetallic compound particle as compared with in the base. Therefore, when the plastic working is repeated, still more work strains are accumulated around the intermetallic compound particle. The inventors of this application have found that when the work strains are increased, more nucleation sites are generated for dynamic recrystallization to be generated at the time of the following extruding process, so that a finer crystal particle grain that could not be manufactured by the conventional manufacturing method of the Mg alloy can be implemented.

[0034] Regarding this new knowledge, similar one is disclosed in Japanese Unexamined Patent Publication No. 5-306404. That is, it discloses a method of manufacturing a Mg alloy member in which Mg alloy powder obtained from a T6-treated Mg alloy member containing A1 through a cutting process is hot-pressed, and then the hot-pressed body is extruded. However, according to the method disclosed here, since the plastic working that is proposed by the present invention is not forcibly performed on the T6-treated cut Mg alloy powder, the above-described nucleation site for the dynamic recrystallization is not formed, so that a fine crystal grain cannot be provided. Thus, the tensile proof stress of the Mg alloy provided from T6 heat-treated AZ80 alloy powder cuttings, for example is as low as about 200MPa. In addition, as described above, since the tensile proof stress of the extruded member using the AZ80 powder that is not T6-treated is also 200MPa, the tensile proof stress is about the same as in the above case where the T6 heat treatment is performed. As a result, the above method is substantially different from the manufacturing method according to the present invention in which the work strains are preferentially accumulated around the precipitated and dispersed particle and they become the nucleation sites for the dynamic recrystallization.

[0035] In addition, according to the present invention, when the plastic working is repeatedly performed by the pair of rolls for the powder, the work strains are provided in a random direction. As a result, the crystal orientation in the extruded Mg alloy is in random order, so that its elongation is improved. That is, according to a conventional extruded member, its elongation deteriorates because the basal plane (0001) that is the slip plane of Mg is aligned along the extruded direction. Meanwhile, according to the extruded Mg alloy powder on which the plastic working with one pair of rolls is performed according to the present invention, non-basal planes such as the prism plane (10-10) and the pyramidal plane (10-11) other than the basal plane (0001) are also aligned along the extruded direction. As a result, the Mg alloy can implement large elongation as well as the high proof stress.

[0036] Meanwhile, when the similar plastic working is performed on the Mg alloy powder that has not been T6 heat-treated by the roller compactor, although it is confirmed that the magnesium crystal grain is miniaturized, the fine crystal like in the case the T6 heat treatment has been performed cannot be provided. Therefore, in order to effectively miniaturize

the crystal grain by the plastic working with the pair of rolls according to the present invention, it is necessary to precipitate and disperse the intermetallic compound in the base of the raw Mg alloy powder material.

[0037] In addition, the size of the intermetallic compound particle has strong relation with the amount of the working strains accumulated around the particle. The smaller the grain diameter of the intermetallic compound is, the more the work strains are accumulated, and as a result, the Mg alloy has the high proof stress. More specifically, when the maximum grain diameter of the intermetallic compound precipitated and dispersed in the base of the raw material powder is 5 μm or less, the magnesium alloy can provide a high proof stress exceeding 250MPa. In addition, when the maximum grain diameter of the intermetallic compound is 2 μm or less, still more work strains can be accumulated with the less number of plastic workings. As a result, there can be provided an economical effect that the Mg alloy having a fine crystal grain and a high proof stress can be manufactured with the less number of plastic workings with one pair of rolls as well as the effect that the high proof stress is provided.

[0038] Therefore, the present invention is characterized by the manufacturing method in which the Mg alloy powder having the fine intermetallic compound particles previously precipitated and dispersed in the base through the T6 heat treatment is passed through the pair of rolls to be subjected to the plastic working, whereby the Mg alloy having the fine crystal grain implement both high proof stress and high toughness.

[0039] Next, regarding (b), the maximum size of the Mg alloy powder processed by the plastic working with the pair of rolls is to be not more than 10mm and the minimum size thereof is to be not less than 0.1mm. When the maximum size of the powder exceeds 10mm, the problem is that the binding property of the powder is lowered at the time of compacting process in the next step or the end of the molded compact is deficient because the powder cannot reach the corner of the die when it is poured into the die. Meanwhile, when the minimum size of the magnesium alloy powder is below 0.1mm, since it is likely to be ignited, the problem is generated in view of the safety in handling it.

[0040] Regarding (c) and (d), the Mg alloy powder has the crystal grain relatively smaller than the crystal grain diameter of the starting material powder due to the plastic working with the pair of rolls. More specifically, the maximum crystal grain diameter of the magnesium particle constituting the base is to be not more than 20 μm in the Mg alloy powder processed by the plastic working with the pair of rolls. When such Mg alloy powder is compacted and extruded, the magnesium alloy can provide the proof stress exceeding 250MPa. When the crystal grain diameter of the magnesium particle of the powder processed by the plastic working with the rolls is beyond 20 μm , it is difficult to implement the high proof stress exceeding 250MPa in the Mg alloy manufactured from such magnesium alloy powder. In addition, in order to obtain a still higher proof stress, that is, to obtain characteristics exceeding 350MPa, for example, the crystal grain diameter in the base of the Mg alloy powder after the plastic working process with the pair of rolls needs to be 10 μm or less.

[0041] In the plastic working with the rolls, the temperature of the starting material powder to be poured and the surface temperature of the roll to be in contact with the powder have to be not more than the temperature of the aging heat treatment at the following step. When the plastic working is performed at the temperature higher than the aging heat treatment temperature, the amount of the work strains to be accumulated around the intermetallic compound particle by an overaging phenomenon is reduced and the dynamic recrystallization at the time of the extruding process does not effectively progressed, and as a result, it is difficult to obtain the Mg alloy having fine crystal grains with high proof stress.

(3) Magnesium alloy and its manufacturing method

[0042] When the raw magnesium alloy powder material processed by the plastic working with the rolls is compacted and warm extruded, a magnesium alloy with high proof stress having the following characteristics is obtained.

(a) The maximum crystal grain diameter of the magnesium particle constituting the base of the obtained Mg alloy is not more than 10 μm .

(b) The tensile proof stress of the alloy is not less than 250MPa at room temperature.

[0043] The inventors of this application have found that especially when the Mg alloy powder having the magnesium particle whose crystal grain diameter is 10 μm or less in the base is used as the raw material, the maximum crystal grain diameter of the magnesium particle constituting the base of the extruded magnesium alloy is 5 μm or less and the tensile strength of the alloy at room temperature is 350MPa or more. In addition, the proof stress of the extruded magnesium alloy can be improved also by the intermetallic compound such as $\text{Mg}_{17}\text{Al}_{12}$, Al_2Ca , Mg_2Si , MgZn_2 , Al_3Re (Re: rare-earth element), $\text{Al}_{11}\text{Re}_3$, and Al_6Mn dispersed in the base of the poured raw material processed by the T6 treatment.

[0044] The raw magnesium alloy powder material processed by the plastic working with the rolls as described above is poured into a die and pressurized, whereby the powder compact is manufactured. The powder compact is heated within a temperature range of 150°C to 450°C and solidified densely through immediate extrusion, whereby a magnesium alloy material is manufactured. When the heating temperature is lower than 150°C, since the dynamic recrystallization is not progressed, a fine magnesium crystal grain cannot be provided. Meanwhile, when the heating temperature is

higher than 450°C, fine recrystalline structure is grown and becomes coarse. In addition, in view of the effect of the heat value at the time of the extruding, the powder compact temperature is preferably at 200°C to 350°C. In addition, in view of densification, an extrusion ratio is to be 10 or more and more preferably 30 or more.

Example 1

[0045] An AZ91D ingot (composition : 9.1% Al, 0.85% Zn, 0.23% Mn, by weight and the balance of Mg) manufactured by casting was subjected to a solution heat treatment (kept at 413°C for 16 hours and then air-cooled) and then subjected to an aging heat treatment (kept at 251°C for 4 hours and then cooled down in an oven of nitrogen gas atmosphere). Powder was manufactured from this ingot by grinding (T6 heat-treated AZ91D powder).

[0046] Meanwhile, as a comparison example, the cast ingot was subjected to only the solution heat treatment and powder was manufactured from the ingot by grinding in the same condition (solution-treated AZ91D powder). The grain diameter of each powder was within a range of 0.5 to 4mm.

[0047] Each AZ91D powder as the starting material was processed by the plastic working in the roller compactor. Here, a roll diameter was 100mm, a roll circumferential velocity was 100mm/second, the clearance between rolls was 0.1mm, and the surface temperature of the roll and the raw material powder temperature were room temperature. The plate-like connected powder processed by the plastic working with the rolls was ground to 1 to 5mm in length by a milling cutter, whereby a predetermined magnesium alloy powder was manufactured (one passed powder). The crystal grain was miniaturized by repeating the above process. Here, powders provided by repeating the above process 20 times and 40 times are referred to as N = 20 and 40, and powder that was not processed by the plastic working is referred to as N = 0.

[0048] Fig. 3 is photographs showing the structures of the AZ91D ingot, in which (a) shows a structure photograph after casting, (b) shows a structure photograph after the solution treatment, and (c) shows a structure photograph after the T6 heat treatment (solution treatment + aging heat treatment). As can be clear from the photographs in Fig. 3, it is confirmed that a coarse $Mg_{17}Al_{12}$ precipitated after casting is dissolved in the magnesium base by the solution heat treatment and the fine intermetallic compound is uniformly dispersed in the base by the aging heat treatment.

[0049] Fig. 4 shows an enlarged structure photograph of the AZ91D after the T6 heat treatment in Fig. 3(c). A granulated compound of 500 to 800nm is uniformly dispersed and the predetermined structure aimed by the present invention as the starting material is formed by the T6 heat treatment.

[0050] Fig. 5 shows structure photographs of the AZ91D powder processed by the plastic working with the rolls, in which (a) shows the structure processed by the T6 heat treatment according to the present invention, and (b) shows the structure of the comparison example processed by only the solution heat treatment. In the case of the AZ91D powder processed by the T6 heat treatment, when the plastic working is performed with rolls 20 times and 40 times, it is confirmed that the magnesium base has the uniform structure and the crystal grain diameter is miniaturized to 2 to 5 microns. Meanwhile, in the case of the AZ91D powder subjected to the solution treatment only shown in 5B, even when the plastic working is performed 40 times, the base has an inhomogeneous mixed structure (white and black regions are mixed in the photograph), and the magnesium base comprises a coarse crystal grain exceeding 20 microns.

[0051] Fig. 6 shows the result of micro hardness (Vickers hardness) test for each powder. Although the hardness of either starting material powder is increased as the number of plastic workings with the rolls is increased, the T6-treated AZ91D powder shows higher hardness value. In addition, the difference in hardness between both powders is increased as the number of workings is increased. That is, it can be confirmed that the work strains generated by the plastic working with the rolls are more effectively accumulated in the base of the T6-treated AZ91D powder.

Example 2

[0052] Each AZ91D powder manufactured in the example 1 was molded by a hydraulic press machine at room temperature, whereby a cylindrical billet for extrusion was manufactured. This billet was heated at 400°C for five minutes in a nitrogen gas atmosphere and immediately subjected to warm extruding (extrusion ratio $r = 37$) to manufacture a dense stick member. A tensile test specimen (parallel part was 20mm) was taken from each extruded magnesium alloy material and a tensile test was performed at a strain rate of 10^{-4} /second at room temperature with respect to each specimen. The measured results of tensile proof stress (0.2% strain), tensile strength and breaking elongation in the test are shown in Table 1.

[Table 1]

TENSILE PROOF STRESS (MPa)	PASSED NUMBER N	0	20	40
	T6 HEAT TREATMENT	210	264	322
	SOLUTION TREATMENT	203	229	246
TENSILE STRENGTH (MPa)	PASSED NUMBER N	0	20	40
	T6 HEAT TREATMENT	332	369	385
	SOLUTION TREATMENT	327	341	354
BREAKING ELONGATION (%)	PASSED NUMBER N	0	20	40
	T6 HEAT TREATMENT	21.2	18.4	18.7
	SOLUTION TREATMENT	18.5	14.1	13.2

[0053] In the case of the T6-treated AZ91D powder according to the present invention, the tensile strength and the 0.2% proof stress of the extruded material through the plastic working with the rolls are considerably increased and especially the tensile proof stress shows a high value of 250 to 300MPa. In addition, regarding the breaking elongation, it maintains a high value of about 18%. Thus, according to the manufacturing method of the present invention, a magnesium alloy having high tensile proof stress and high toughness can be manufactured.

[0054] Meanwhile, according to the comparison example in which the AZ91D powder was subjected to the solution heat treatment only, although its tensile proof stress and tensile strength are increased as the number of plastic workings with the rolls is increased, the values are smaller than those of the T6 heat-treated powder in the present invention example and especially the tensile proof stress does not reach 250MPa.

[0055] Fig. 7 shows an observed result of the structures of the extruded material processed by the plastic working with the rolls 40 times, by an optical microscope. As shown in Fig. 7 (a), in the case of the T6 heat-treated AZ91D powder in the present invention example, when the crystal grain diameter distribution in the magnesium base is measured by image analysis, it has been confirmed that its maximum crystal grain diameter is 4.2 μm and its average crystal grain diameter is 1.5 μm and a fine structure is formed by dynamic recrystallization at the time of the extruding process. Meanwhile, in the case of the solution treated AZ91D powder in the comparison example shown in Fig. 7(b), the maximum crystal grain diameter of the extruded material is 21 μm and the average crystal grain diameter thereof is 9.6 μm and the structure is considerably larger than the case of the T6 heat-treated AZ91D powder shown in Fig. 7(a). That is, when the plastic working with the rolls is performed on the T6 heat-treated Mg alloy powder in the present invention example, many work strains are accumulated around the fine intermetallic compound precipitated and dispersed in the base, and as a result, the dynamic recrystallization can be effectively progressed and a fine crystal grain can be formed.

Example 3

[0056] A ZAXE1713 ingot (composition : 7.1% Al, 0.95% Zn, 0.93% Ca, 2.87% La by weight and the balance of Mg) manufactured by casting was subjected to a solution heat treatment (kept at 420 °C for 16 hours and then air-cooled) and then subjected to an aging heat treatment (kept at 180°C for 36 hours and then cooled down in an oven of nitrogen gas atmosphere). Powder was manufactured from this ingot by grinding (T6 heat-treated ZAXE1713 powder). Meanwhile, as a comparison example, powder was manufactured from a cast ingot that was not subjected to the heat treatment by grinding in the same condition (non heat-treated ZAXE1713 powder). The grain diameter of each powder was within a range of 0.6 to 4mm. Each ZAXE1713 powder as the starting material was processed by the plastic working in the roller compactor.

[0057] Here, a roll diameter was 100mm, a roll circumferential velocity was 100mm/second, the clearance between rolls was 0.1mm, and the surface temperature of the roll and the raw material powder temperature were room temperature similar to the example 1. The plate-like connected powder processed by the plastic working with the rolls was ground to 1 to 5mm in length by a milling cutter, whereby a predetermined magnesium alloy powder was manufactured (one passed powder). The crystal grain was miniaturized by repeating the above process. Here, the repeating number of the plastic workings with the rolls is up to 30 and the case where the plastic working was not performed is referred to as N = 0.

[0058] Each ZAXE1713 powder was molded by a hydraulic press machine at room temperature, whereby a cylindrical billet for extrusion was manufactured. This billet was heated at 400°C for five minutes in a nitrogen gas atmosphere and immediately subjected to warm extruding (extrusion ratio $r = 37$), whereby a dense stick member was manufactured. A tensile test specimen (parallel part was 20mm) was taken from each magnesium alloy extruded material and a tensile test was performed at a strain rate of 5×10^{-4} /second at room temperature with respect to each specimen. The measured

results of the tensile proof stress (0.2% strain), tensile strength and breaking elongation in the test are shown in Table 2.

[Table 2]

5	TENSILE PROOF STRESS (MPa)	PROCESSED NUMBER N	0	3	5	10	15	20	30
		T6 HEAT TREATMENT	201	213	229	249	265	289	318
		NO HEAT TREATMENT	169	173	181	194	203	212	234
10	TENSILE STRENGTH (MPa)	PROCESSED NUMBER N	0	3	5	10	15	20	30
		T6 HEAT TREATMENT	251	309	321	335	343	371	397
		NO HEAT TREATMENT	211	234	251	270	282	299	318
15	BREAKING ELONGATION (%)	PROCESSED NUMBER N	0	3	5	10	15	20	30
		T6 HEAT TREATMENT	20.8	21.6	16.3	18.9	16.9	17.8	18.8
		NO HEAT TREATMENT	22.3	24.3	21.2	19.7	18.7	19.5	18.9

[0059] In the case of the T6-treated ZAXE1713 powder according to the example of the present invention, the tensile strength and the 0.2% proof stress of the extruded material through the plastic working with the rolls were considerably increased and especially its tensile proof stress showed high value of 250 to 300MPa. In addition, regarding the breaking elongation, it maintains a high value of about 16%. Thus, according to the manufacturing method of the present invention, a magnesium alloy having the high tensile proof stress and high toughness can be manufactured.

[0060] Meanwhile, according to the comparison example in which the ZAXE1713 powder that was not subjected to the heat treatment was used, although its tensile proof stress and tensile strength are increased as the number of plastic workings with the rolls is increased, the values are smaller than those of the T6 heat-treated powder in the present invention example and especially the tensile proof stress does not reach 250MPa.

[0061] Fig. 8 shows an observed result of the structures in the extruded direction of the Mg alloy provided by extruding and solidifying the T6 heat-treated ZAXE1713 powder processed by the plastic workings with the rolls 3, 10, 20 and 30 times. The magnesium crystal grain diameter constituting the base becomes small as the number of plastic workings is increased and the maximum crystal grain diameter is 9.2 μm and the average crystal grain diameter is 4.8 μm in the case of 20 times, and the maximum crystal grain diameter is 4.4 μm and the average crystal grain diameter is 1.2 μm in the case of 30 times.

Example 4

[0062] An AZ80A ingot (composition : 8.2% Al, 0.51% Zn, 0.18% Mn by weight and the balance of Mg) manufactured by casting was subjected to a solution heat treatment (kept at 410°C for 6 hours and then air-cooled) and then subjected to an aging heat treatment (kept at 175°C for 26 hours and then cooled down in an oven of nitrogen gas atmosphere). Powder was manufactured from this ingot by grinding (T6 heat-treated AZ80A powder). Meanwhile, as a comparison example, powder was manufactured from an ingot that was not subjected to the heat treatment by grinding in the same condition (non heat-treated AZ80A powder). The grain diameter of each powder was within a range of 0.6 to 4mm.

[0063] Each AZ80A powder as the starting material was processed by the plastic working in the roller compactor. Here, similar to the example 1, a roll diameter was 100mm, a roll circumferential velocity was 100mm/second, the clearance between rolls was 0.1mm, and the surface temperature of the roll and the raw material powder temperature were room temperature. The plate-like connected powder processed by the plastic working with the rolls was ground to 1 to 5mm in length by a milling cutter, whereby a predetermined magnesium alloy powder was manufactured (one passed powder). The crystal grain was miniaturized by repeating the above process. Here, the repeating number of the plastic workings with the rolls was up to 50 and the case where the plastic working was not performed was referred to as N = 0.

[0064] Each AZ80A powder was molded by a hydraulic press machine at room temperature, whereby a cylindrical billet for extrusion was manufactured. This billet was heated at 400 °C for five minutes in a nitrogen gas atmosphere and immediately subjected to warm extruding (extrusion ratio $r = 37$) to manufacture a dense stick member. A tensile test specimen (parallel part was 20mm) was taken from each magnesium alloy extruded material and a tensile test was performed at a strain rate of 5×10^{-4} /second at room temperature with respect to each specimen. The measured result of the tensile proof stress (0.2% strain), tensile strength and breaking elongation in the test are shown in Table 3.

[Table 3]

HEAT TREATMENT OF RAW MATERIAL POWDER	NUMBER OF PLASTIC WORKINGS WITH ROLLS	MECHANICAL CHARACTERISTICS OF EXTRUDED MATERIAL			REFERENCE
		TENSILE STRENGTH (MPa)	TENSILE PROOF STRESS (MPa)	BREAKING ELONGATION (%)	
T6	20	387	262	18.9	PRESENT INVENTION EXAMPLE
T6	30	393	285	18.6	PRESENT INVENTION EXAMPLE
T6	50	409	317	17.9	PRESENT INVENTION EXAMPLE
T6	NOTHING	365	208	16.8	COMPARISON EXAMPLE
NOTHING	20	336	218	17.2	COMPARISON EXAMPLE
NOTHING	NOTHING	318	189	22.4	COMPARISON EXAMPLE

[0065] When the T6-treated AZ80A powder according to the example of the present invention was processed by the plastic working with the rolls, its tensile strength was as high as 262 to 317MPa and its breaking elongation was also as high as 17.9 to 18.9%.

[0066] Meanwhile, according to the comparison example, even when the T6 heat-treated AZ80A powder was used, in the case where the plastic working with the rolls was not performed, the tensile proof stress was as low as 208MPa. Even when the AZ80A powder that was not subjected to the heat treatment was processed by the plastic working with the rolls 20 times, the tensile proof stress was 218MPa, which is considerably low as compared with the present invention example.

Example 5

[0067] A billet for extrusion was manufactured by molding using the T6 heat-treated ZAXE1713 powder manufactured in the example 3 (the number of plastic workings with the rolls was 30). A magnesium alloy extruded material was manufactured under the condition that billet heating temperatures when the billet was warm extruded (extrusion rate $r = 37$) and densely solidified were as shown in Table 4. A tensile test specimen (parallel part was 20mm) was taken from each magnesium alloy extruded material and a tensile test was performed at a strain rate of 5×10^{-4} /second at room temperature with respect to each specimen. The measured result of the tensile proof stress (0.2% strain), tensile strength and breaking elongation in the test are shown in Table 4.

[Table 4]

BILLET TEMPERATURE (°C)	130	210	300	350	390	440	480
TENSILE PROOF STRESS (MPa)	242	327	331	324	318	302	229
TENSILE STRENGTH (MPa)	347	409	418	412	397	383	342
BREAKING ELONGATION (%)	16.8	15.7	15.4	16.7	18.8	19.3	17.5
REFERENCE	COMPARISON EXAMPLE	PRESENT INVENTION EXAMPLE	PRESENT INVENTION EXAMPLE	PRESENT INVENTION EXAMPLE	PRESENT INVENTION EXAMPLE	PRESENT INVENTION EXAMPLE	COMPARISON EXAMPLE

[0068] When the billet temperature is the one defined by the present invention, the tensile proof stress is as high as 300MPa or more. Meanwhile, when the billet temperature is 130 °C in the comparison example, high tensile proof stress is not provided because the recrystallization is not effectively progressed in the extruding process. In addition, when the billet temperature is 480°C in the comparison example, high tensile proof stress is not provided because the fine recrystalline structure is grown and becomes big in the extruding process.

Example 6

[0069] The plastic working with the rolls were performed up to 30 times in the same condition as in the example 1 using the T6 heat-treated ZAXE1713 powder manufactured in the example 3 to miniaturize the powder structure. At that time, the temperature of the roll surface and the powder were at room temperature or 200°C. The obtained Mg alloy powder was molded by a hydraulic press machine at room temperature, whereby a cylindrical billet for extrusion was manufactured. The billet was heated at 400°C for 5 minutes in a nitrogen gas atmosphere, and then immediately the billet was warm extruded (extrusion rate $r = 37$) and densely solidified, whereby a dense stick member was manufactured. A tensile test specimen (parallel part was 20mm) was taken from each magnesium alloy extruded material and a tensile test was performed at a strain rate of 5×10^{-4} /second at room temperature with respect to each specimen. The measured result of the tensile proof stress (0.2% strain), tensile strength and breaking elongation in the test are shown in Table 5.

[Table 5]

TENSILE PROOF STRESS (MPa)	NUMBER OF WORKINGS	0	3	5	10	15	20	30
	ROOM TEMPERATURE	201	213	229	249	265	289	318
	200°C	201	207	213	217	208	215	211
TENSILE STRENGTH (MPa)	NUMBER OF WORKINGS	0	3	5	10	15	20	30
	ROOM TEMPERATURE	251	309	321	335	343	371	397
	200°C	251	267	272	283	289	296	306
BREAKING ELONGATION (%)	NUMBER OF WORKINGS	0	3	5	10	15	20	30
	ROOM TEMPERATURE	20.8	21.6	16.3	18.9	17.9	17.8	18.8
	200°C	20.8	22.1	20.4	18.4	18.9	18.3	17.2

[0070] When the temperatures of the roll surface and the powder are room temperature according to the present invention example, each of the tensile proof stress, tensile strength and breaking elongation of the obtained Mg extruded material shows high value.

[0071] Meanwhile, when the temperatures of the roll surface and the powder are at 200°C higher than the aging treatment temperature (175°C) in the comparison example, the tensile proof stress and the tensile strength are considerably lowered as compared with the present invention example. As for the proof stress especially, even when the working number is increased, it shows almost the constant value. This is because when the Mg alloy powder is heated up to the aging treatment temperature or more and processed by the plastic working with the rolls, the work strains are not sufficiently accumulated around the precipitated particle because of an overaging phenomenon, and as a result, a fine structure is not likely to be formed by the dynamic recrystallization at the time of the extruding process, so that the proof stress is lowered.

Example 7

[0072] Fig. 9 is pole figures showing the result of evaluation of the orientation of the basal plane (0001) of magnesium for the section of the extruded material AZ80A manufactured in the example 4 in its extruded direction. Here, the plastic workings with the rolls were performed 5, 10, 30 and 50 times. Before the working number reaches 10 times, a typical texture of the extruded material in which the plane (0001) is along the extruded direction was formed. However, in the case of the 30 and 50 times, the basal plane orientation is lowered. In other words, the non-basal planes such as the prism plane (10-10) and pyramidal plane (10-11) other than the basal plane are also oriented in the extruded direction.

[0073] Meanwhile, in the case of the Mg alloy powder that was not subjected to the heat treatment, even after the plastic workings were performed 50 times, the basal plane orientation is not eminently lowered.

[0074] As described above, according to the Mg alloy material manufactured such that the T6 heat-treated magnesium alloy powder is processed by the plastic working with the rolls and extruded as defined by the present invention, in

addition to the increase in tensile proof stress due to the miniaturization of the crystal grain because of the dynamic recrystallization, the breaking elongation (toughness) is improved due to disordering of the texture.

Example 8

[0075] A cast magnesium ingot having a composition shown in Table 6 was subjected to a solution heat treatment (kept at 420°C for 16 hours and then air-cooled) and then subjected to an aging heat treatment (kept at 180°C for 36 hours and then cooled down in an oven of nitrogen gas atmosphere).

[Table 6]

SAMPLE NO.	NUMBER OF WORKINGS	ALLOY COMPOSITION (WT%)								
		Zn	Al	Ca	La	Zr	Sr	Sc	Ti	Mg
1	30	0.95	7.11	0.93	2.87	0	0	0	0	THE BALANCE
2	30	1.06	6.94	0.98	3.04	0.85	0	0	0	THE BALANCE
3	30	1.11	7.15	1.08	2.93	1.97	0	0	0	THE BALANCE
4	30	0.99	6.98	1.11	2.89	0	1.93	0	0	THE BALANCE
5	30	1.03	7.09	0.97	2.97	0	2.59	0	0	THE BALANCE
6	30	0.99	6.84	0.95	3.06	0	0	0.89	0	THE BALANCE
7	30	1.05	7.01	1.02	2.98	0	0	0	1.16	THE BALANCE
8	30	1.06	7.07	0.98	2.95	0	0	0	2.07	THE BALANCE
9	30	0.96	7.08	1.11	2.87	0	1.03	0	0.86	THE BALANCE
10	0	0.95	7.11	0.93	2.87	0	0	0	0	THE BALANCE
11	0	1.11	7.15	1.08	2.93	1.97	0	0	0	THE BALANCE
12	0	1.03	7.09	0.97	2.97	0	2.59	0	0	THE BALANCE

[0076] Magnesium alloy powder was manufactured from each ingot by grinding. Each powder had a grain diameter of 0.6 to 4mm. Each powder as the starting material was processed by the plastic working in the roller compactor. Here, similar to the example 1, a roll diameter was 100mm, a roll circumferential velocity was 100mm/second, the clearance between rolls was 0.1mm, and the surface temperature of the roll and the raw material powder temperature were room temperature.

[0077] The plate-like connected powder processed by the plastic working with the rolls was ground to 1 to 5mm in length by a milling cutter, whereby a predetermined magnesium alloy powder was manufactured (one passed powder). The crystal grain was miniaturized by repeating the above process. Here, the repeating number of the plastic workings with the rolls is up to 30 and the case where the plastic working was not performed was referred to as N = 0 for comparison.

[0078] Then, each processed powder was molded by a hydraulic press machine at room temperature to manufacture a cylindrical billet for extrusion. This billet was heated at 400°C for five minutes in a nitrogen gas atmosphere and immediately subjected to warm extruding (extrusion ratio $r = 37$), whereby a dense stick member was manufactured. A tensile test specimen (parallel part was 20mm) was taken from each magnesium alloy extruded material and a tensile test was performed at a strain rate of 5×10^{-4} /second at room temperature with respect to each specimen. The measured

result of the tensile proof stress (0.2% strain), tensile strength and breaking elongation in the test are shown in Table 7.

[Table 7]

SAMPLE NO.	NUMBER OF WORKINGS	TENSILE PROOF STRESS (MPa)	TENSILE STRENGTH (MPa)	BREAKING ELONGATION (%)	REFERENCE
1	30	318	397	18.8	PRESENT INVENTION EXAMPLE
2	30	333	417	18.2	PRESENT INVENTION EXAMPLE
3	30	346	428	17.9	PRESENT INVENTION EXAMPLE
4	30	340	424	18	PRESENT INVENTION EXAMPLE
5	30	352	442	17.4	PRESENT INVENTION EXAMPLE
6	30	335	416	18.4	PRESENT INVENTION EXAMPLE
7	30	336	419	18.1	PRESENT INVENTION EXAMPLE
8	30	345	425	18	PRESENT INVENTION EXAMPLE
9	30	357	449	17.2	PRESENT INVENTION EXAMPLE
10	0	201	251	20.8	COMPARISON EXAMPLE
11	0	205	242	15.2	COMPARISON EXAMPLE
12	0	207	231	12.3	COMPARISON EXAMPLE

[0079] Samples No. 1 to 9 are in the present invention example and samples No. 2 to 9 are extruded materials provided using powder taken from cast magnesium alloy ingots to which active metal elements such as Zr, Sr, Sc and Ti is added to the sample No. 1 within an appropriate range. As compared with the characteristics of the sample No.1, when the active metal element such as Zr, Sr, Sc and Ti is added, the tensile proof stress and the tensile strength can be improved without lowering the elongation (toughness) eminently.

[0080] Meanwhile, according to the samples No. 10 to 12 in the comparison example, unless the plastic working with the rolls is performed, even when the active metal element is added, the tensile proof stress and the tensile strength are not increased and the elongation is lowered.

[0081] Although the embodiments of the present invention have been described with reference to the drawings in the above, the present invention is not limited to the above-illustrated embodiments. Various kinds of modifications and variations may be added to the illustrated embodiments within the same or equal scope of the present invention.

INDUSTRIAL APPLICABILITY

[0082] The present invention can be advantageously applied to provide a magnesium alloy implementing both high proof stress and elongation.

Claims

1. A raw magnesium alloy powder material having a relatively small crystal grain diameter obtained by subjecting a starting material powder having a relatively large crystal grain diameter to a plastic working in which the powder is passed through a pair of rolls to undergo compressive deformation or shear deformation, **characterized in that** said starting material powder is a magnesium alloy powder having a fine intermetallic compound precipitated and dispersed in a base by a heat treatment, a work strain is formed around said precipitated intermetallic compound in said magnesium alloy powder after processed by said plastic working, the magnesium alloy powder after processed by said plastic working has a maximum size of 10mm or less and a minimum size of 0.1mm or more, and a magnesium particle constituting the base of said magnesium alloy powder after processed by the plastic working has a maximum crystal grain diameter of 20 μm or less.
2. The raw magnesium alloy powder material according to claim 1, wherein said intermetallic compound is at least one compound selected from a group consisting of $\text{Mg}_{17}\text{Al}_{12}$, Al_2Ca , Mg_2Si , MgZn_2 , Al_3Re (Re: rare-earth element), $\text{Al}_{11}\text{Re}_3$, and Al_6Mn .
3. The raw magnesium alloy powder material according to claim 1, wherein said intermetallic compound has a maximum grain diameter of 5 μm or less.
4. The raw magnesium alloy powder material according to claim 1, wherein said intermetallic compound has a maximum grain diameter of 2 μm or less.
5. The raw magnesium alloy powder material according to claim 1, wherein said magnesium particle constituting the base of said magnesium alloy powder has a maximum crystal grain diameter of 10 μm or less.
6. A manufacturing method of the raw magnesium alloy powder material according to claim 1, wherein 0.5% to 4% by weight of a metal element selected from a group consisting of strontium (Sr), zirconium (Zr), scandium (Sc) and titanium (Ti) is contained and the balance is magnesium (Mg) substantially.
7. A magnesium alloy with high proof stress provided by compacting and extruding the raw magnesium alloy powder material according to claim 1, **characterized in that** a magnesium particle constituting an alloy base has a maximum crystal grain diameter of 10 μm or less, and its tensile proof stress is 250MPa or more at room temperature.
8. The magnesium alloy with high proof stress according to claim 7, wherein the magnesium particle constituting the magnesium alloy base has a maximum crystal grain diameter of 5 μm or less, and its tensile proof stress is 350MPa or more at room temperature.
9. The magnesium alloy with high proof stress according to claim 7, wherein at least one intermetallic compound selected from a group consisting of $\text{Mg}_{17}\text{Al}_{12}$, Al_2Ca , Mg_2Si , MgZn_2 , Al_3Re (Re: rare-earth element), $\text{Al}_{11}\text{Re}_3$, and Al_6Mn is precipitated and dispersed in the base of said magnesium alloy.
10. A manufacturing method of a raw magnesium alloy powder material for miniaturizing a crystal grain diameter of a magnesium particle constituting a base of a starting material powder by performing a plastic working on the starting material powder, **characterized in that** a magnesium alloy powder having a fine intermetallic compound precipitated and dispersed by a heat treatment in a base is prepared as said starting material powder, said plastic working is performed to provide work strains around said intermetallic compound by passing said starting

material powder through a pair of rolls to make it undergo compressive deformation or shear deformation, and said plastic working is repeated until the maximum size and minimum size of the powder become 10mm or less and 0.1mm or more, respectively, and the maximum crystal grain diameter of the magnesium particle constituting the base of the powder becomes 20 μm or less.

- 5
11. The manufacturing method of the raw magnesium alloy powder material according to claim 10, wherein the step of preparing the magnesium alloy powder as said starting material powder comprises
a step of manufacturing a magnesium alloy ingot by casting,
a step of precipitating and dispersing a fine intermetallic compound in the base of the ingot by performing a solution
10 treatment and then aging heat treatment for said magnesium alloy ingot, and
a step of obtaining the magnesium alloy powder from said ingot by machining.
12. The manufacturing method of the raw magnesium alloy powder material according to claim 11, wherein
15 the temperature of the starting material powder to be poured and the surface temperature of said roll to be in contact
with the starting material powder are to be the temperature of said aging heat treatment or less at the time of said
plastic working.
13. The manufacturing method of the raw magnesium alloy powder material according to claim 11, wherein
20 said magnesium alloy ingot contains 0.5 to 4% by weight of a metal element selected from a group consisting of
strontium (Sr), zirconium (Zr), scandium (Sc) and titanium (Ti) and the balance of magnesium (Mg) substantially.
14. A manufacturing method of a magnesium alloy with high proof stress comprising:
25 a step of obtaining a compact by compacting the raw magnesium alloy powder material according to claim 1
and filled in a die;
a step of heating said magnesium alloy powder compact to 150 to 450°C; and
a step of manufacturing a magnesium alloy by extruding said magnesium alloy compact immediately after said
heating step.
- 30 15. The manufacturing method of the magnesium alloy with high proof stress according to claim 14, wherein
a magnesium particle constituting the base of said magnesium alloy has a maximum crystal grain diameter of 10
 μm or less, and
its tensile proof stress is 250MPa or more at room temperature.
- 35 16. The manufacturing method of the magnesium alloy with high proof stress according to claim 14, wherein
said magnesium alloy compact is heated at 200 to 350°C.

FIG. 1

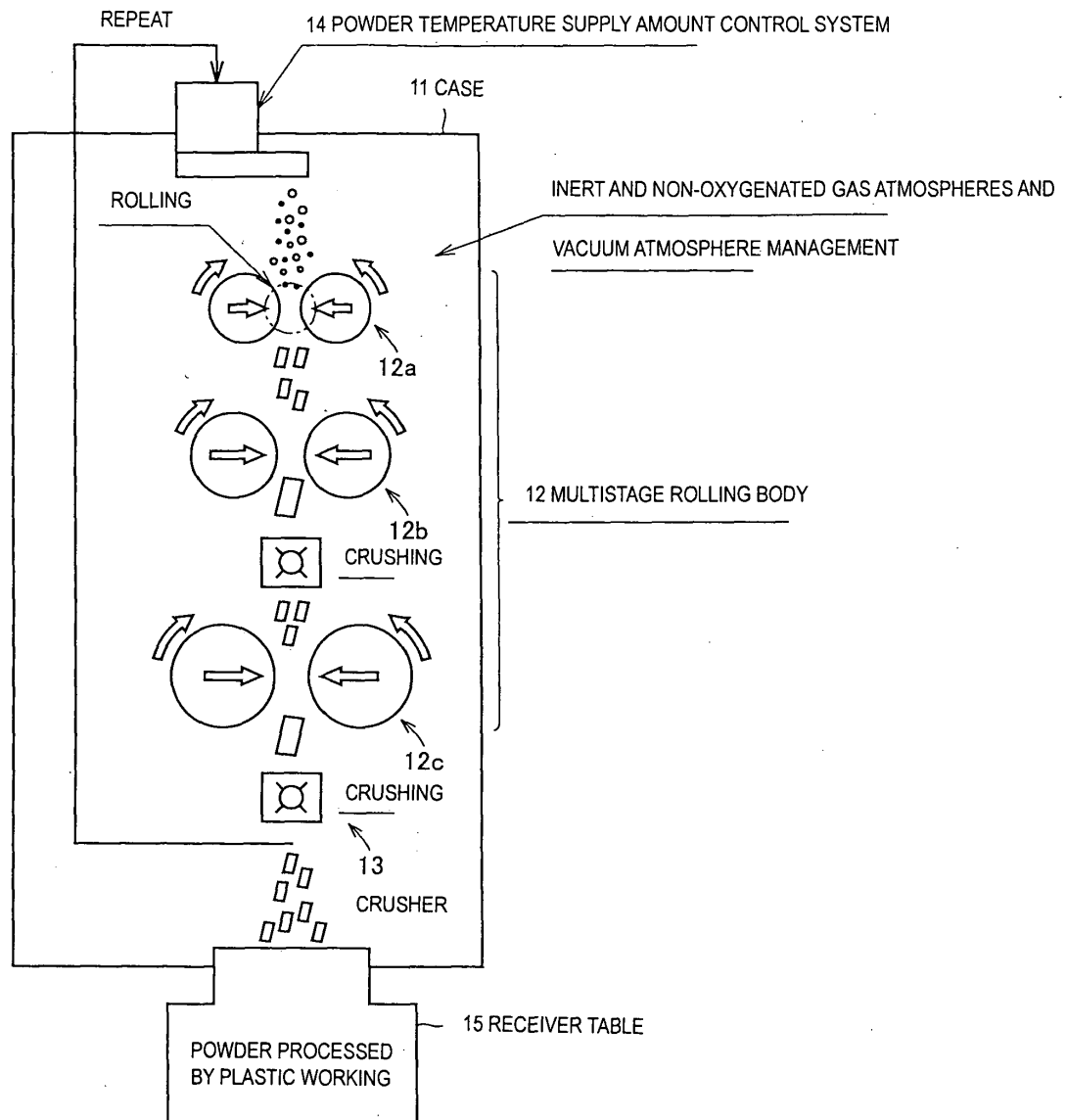


FIG. 2

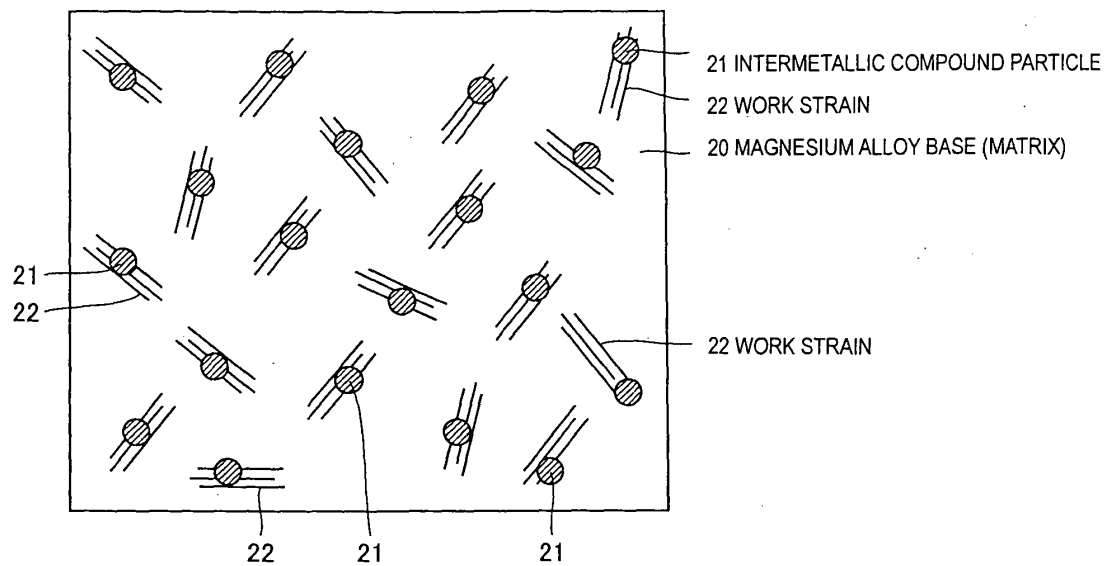


FIG. 3

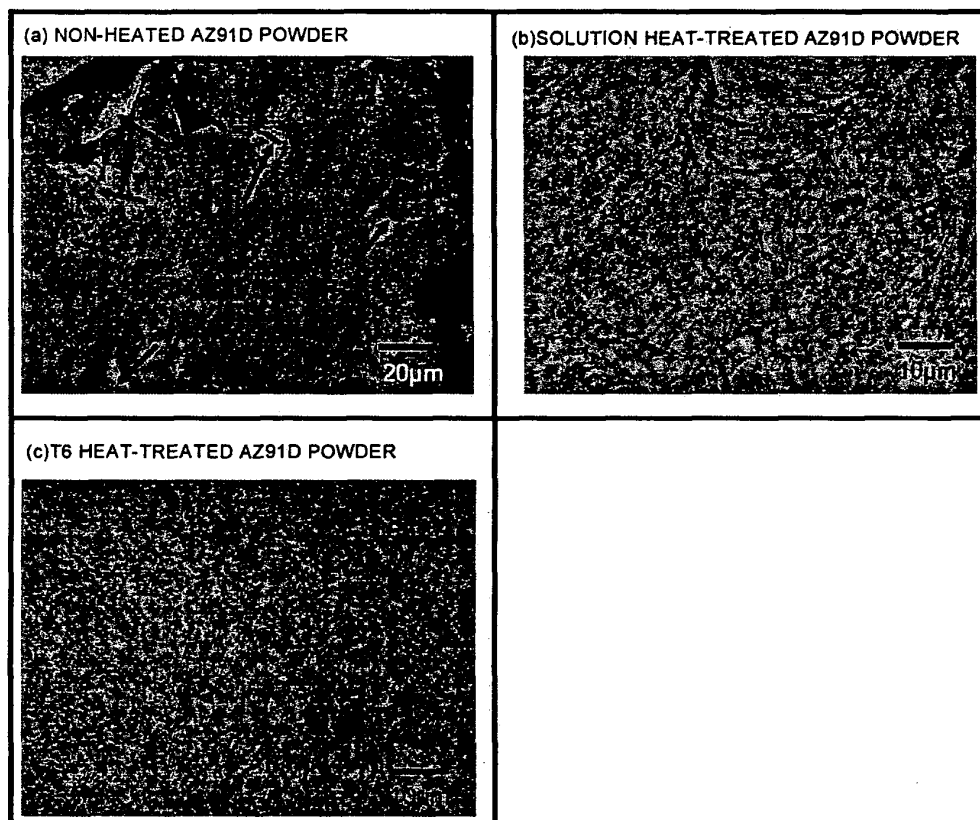
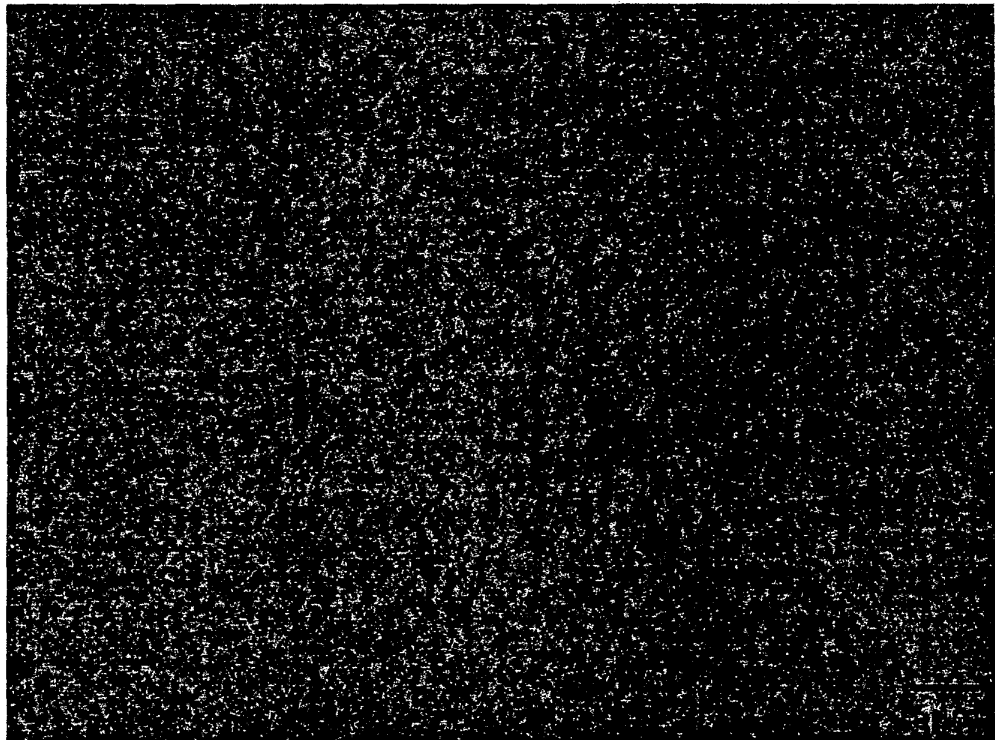
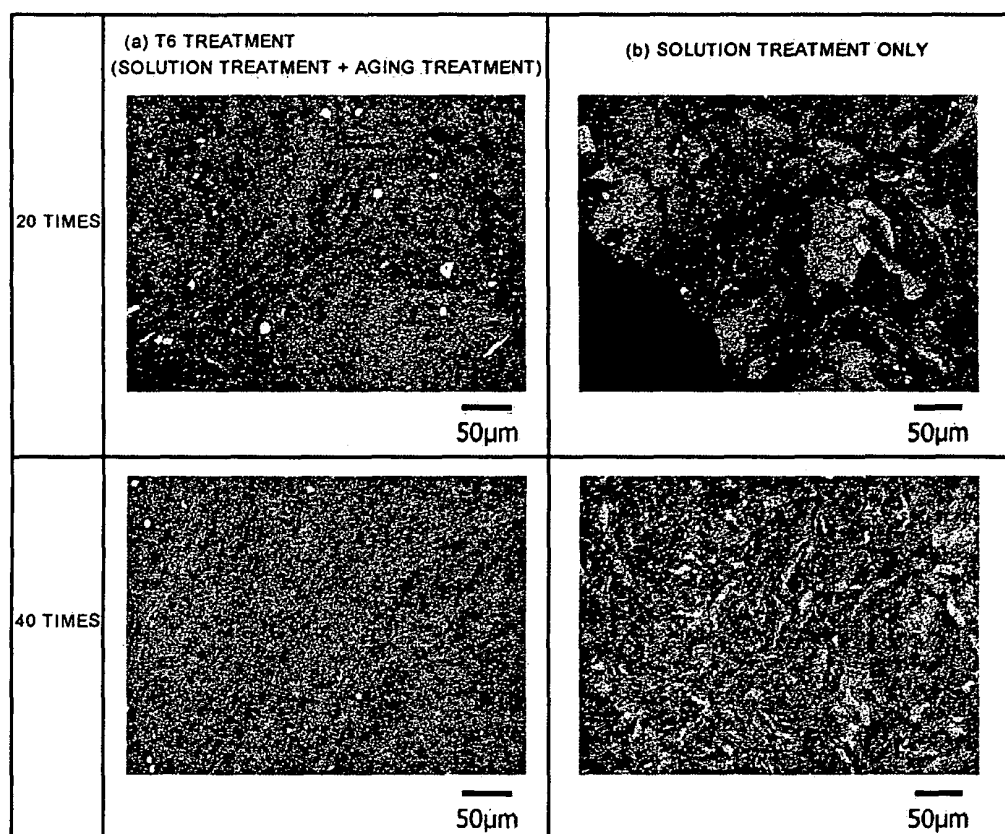


FIG. 4



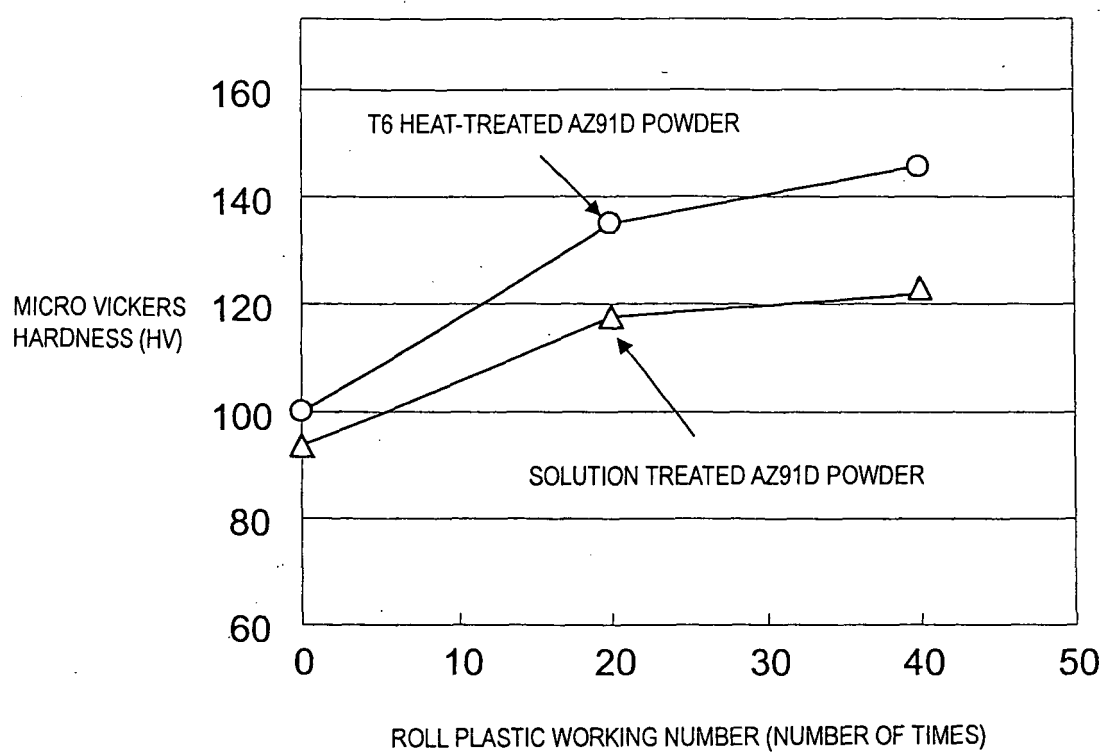
ENLARGED STRUCTURE PHOTOGRAPH OF T6 HEAT-TREATED AZ91D POWDER

FIG. 5



EFFECT OF T6 HEAT TREATMENT IN AZ91D POWDER

FIG. 6



EFFECT OF T6 HEAT TREATMENT IN AZ91D POWDER

FIG. 7

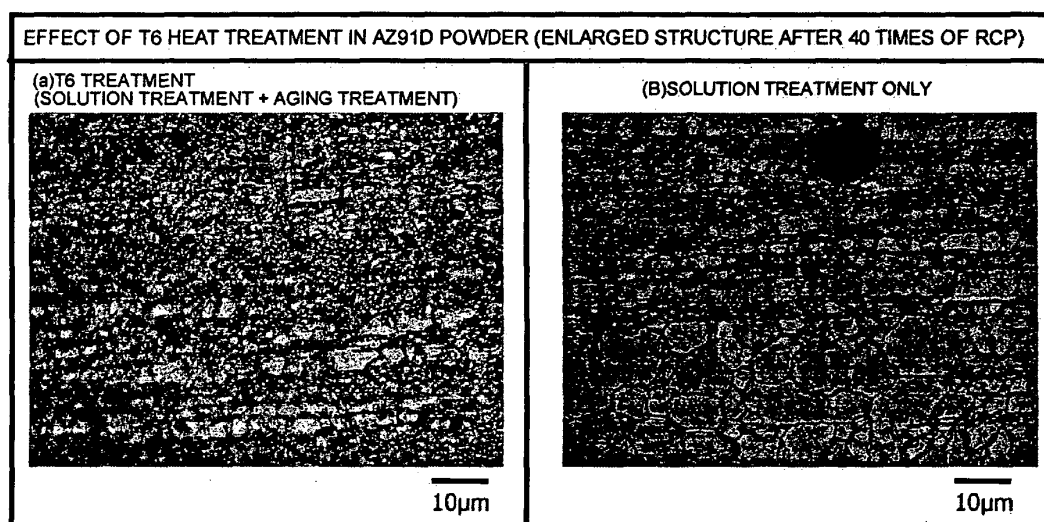


FIG. 8

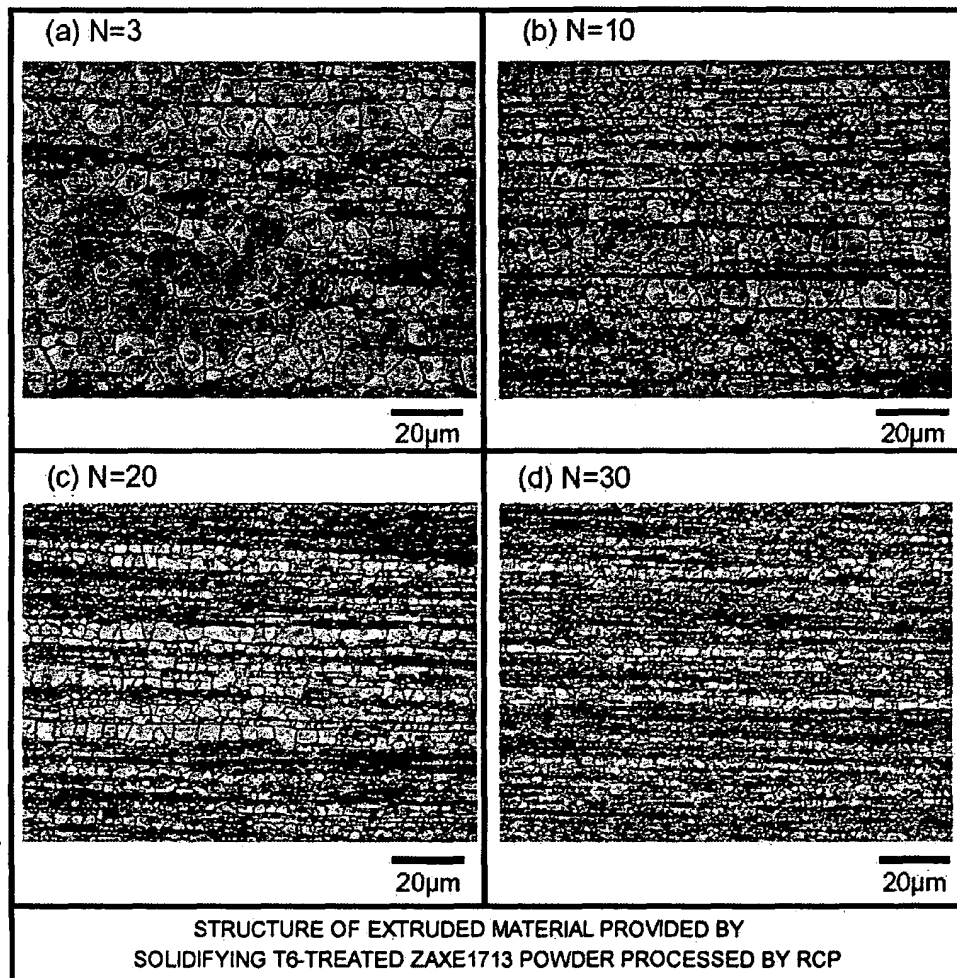
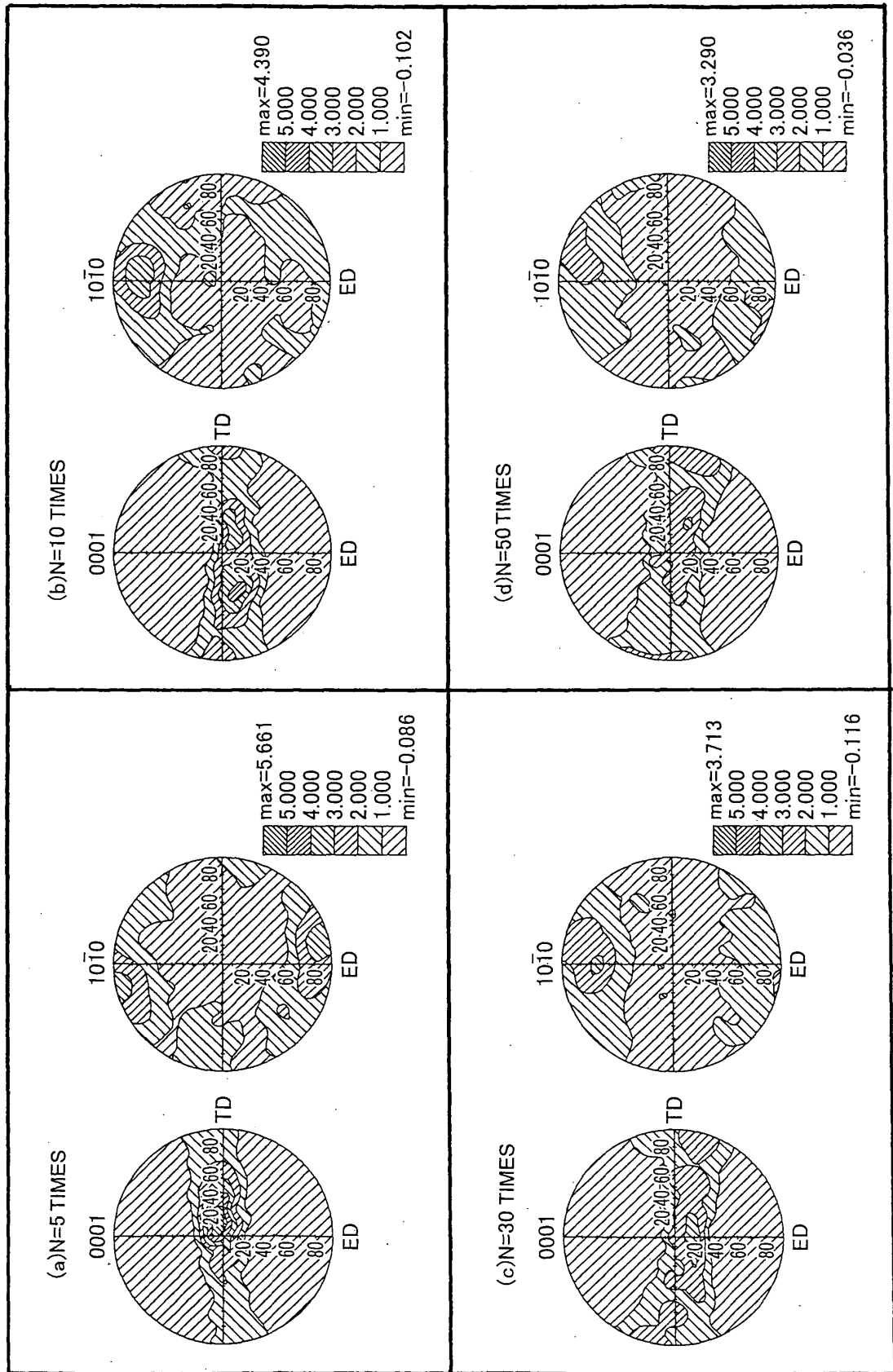


FIG. 9



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2006/311952

A. CLASSIFICATION OF SUBJECT MATTER <i>B22F1/00(2006.01)i, B21C23/01(2006.01)i, B22F3/20(2006.01)i, B22F9/04(2006.01)i, C22C1/04(2006.01)i, C22C23/02(2006.01)i, C22F1/06(2006.01)i</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) <i>B22F1/00, B21C23/01, B22F3/20, B22F9/04, C22C1/04, C22C23/02, C22F1/06</i> Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched <i>Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2006</i> <i>Kokai Jitsuyo Shinan Koho 1971-2006 Toroku Jitsuyo Shinan Koho 1994-2006</i> Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	JP 2006-63411 A (Kurimoto Ltd.), 09 March, 2006 (09.03.06), Claims; Par. Nos. [0030] to [0040], [0045] to [0047], [0059] to [0061]; example 1; table 3; Fig. 3 (Family: none)	1-10, 14-16
A	JP 5-306404 A (Mazda Motor Corp.), 19 November, 1993 (19.11.93), (Family: none)	1-16
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Patent documents cited in the description

- JP 2182806 A [0005]
- JP 5320715 A [0005]
- JP 5306404 A [0006] [0007] [0034]