



(11)

EP 2 628 813 A1

(12)

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

(43) Date of publication:

21.08.2013 Bulletin 2013/34

(51) Int Cl.:

C22C 23/06 (2006.01)
B22F 3/20 (2006.01)

B22F 3/14 (2006.01)
C22C 23/00 (2006.01)

(21) Application number: **11832618.0**

(86) International application number:

PCT/JP2011/073649

(22) Date of filing: **14.10.2011**

(87) International publication number:

WO 2012/050188 (19.04.2012 Gazette 2012/16)

(84) Designated Contracting States:

**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR**

(30) Priority: **15.10.2010 JP 2010232364**

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(54) **HIGH-STRENGTH MAGNESIUM ALLOY WIRE AND METHOD FOR MANUFACTURING SAME, HIGH-STRENGTH MAGNESIUM ALLOY PRODUCT, AND HIGH-STRENGTH MAGNESIUM ALLOY SPRING**

(57) A high-strength magnesium alloy wire rod suitable for products in which at least one of bending stress and twisting stress primarily acts is provided. The wire rod has required elongation and 0.2 % proof stress, whereby strength and formability are superior, and has higher strength in the vicinity of the surface. In the wire

rod, the surface portion has the highest hardness in a cross section of the wire rod, the highest hardness is 170 HV or more, and the inner portion has a 0.2 % proof stress of 550 MPa or more and an elongation of 5% or more.

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Description

Technical Field

5 **[0001]** The present invention relates to a high-strength magnesium alloy wire rod, production method therefor, high-strength magnesium alloy part, and high-strength magnesium alloy spring, which are suitable for products in which at least one of bending stress and twisting stress primarily acts.

Background Art

10 **[0002]** In various fields, such as aerospace, vehicles (automobiles, motor cycles, trains), medical instruments, welfare devices, and robots, low weights of parts are desired for improvement of function, improvement of performance, and improvement of operability. Specifically, in the field of vehicles such as automobiles, emission amounts of carbon dioxide has been desired to be reduced in view of the environment. Therefore, requirements for lightweight for improvement of fuel consumption has become increasingly stringent every year.

15 **[0003]** Development of lightweight parts has been active primarily in the field of vehicles, and great strengthening of steels by improvements in composition, surface modification, and combination thereof in steels has been primarily researched. For example, high-tension steels have been primarily used for springs, which are typical strong parts, and fatigue strength thereof is further improved by applying surface modification such as nitriding and shot peening, thereby yielding lightweight springs. However, great strengthening of steels by conventional improvements in composition is nearing a limit, and great reductions in weight in the future cannot be anticipated.

20 **[0004]** Therefore, lightweight alloys, typically having low specific gravity, such as titanium alloys, aluminum alloys, and magnesium alloys are desired for further reduction in weight. Magnesium alloys have the lowest specific gravity in the practical metals, which is about 1/4 of that of steels, about 1/2.5 of that of titanium alloys, and about 1/1.5 of that of aluminum alloys. Therefore, magnesium alloys have great advantages in being low in weight and as a resource, and they are expected to be widely used in the market.

25 **[0005]** However, conventional magnesium alloys are limited in use as products. The main reason of this is that the strengths of the conventional magnesium alloys are low. Therefore, in order to obtain strength for parts, it is necessary to increase size of parts compared to that of the conventional steel parts. That is, the conventional magnesium alloys have not been accepted as strong parts in the market since low weight and compact size are incompatible.

30 **[0006]** Under such circumstances, research in high-strength magnesium alloys for use as strong parts has been actively made. For example, Japanese Patent Unexamined Publication No. 3-90530 discloses a technique in which a molten Mg-Al-Zn-Mn-Ca-RE (rare earth) alloy is subjected to wheel casting, thereby forming a solid member, which is drawn and densified, thereby obtaining a magnesium alloy member having a 0.2 % proof stress of 565 MPa.

35 **[0007]** Japanese Patent Unexamined Publication No. 3-10041 discloses a technique in which a molten Mg-X-Ln (X is one or more of Cu, Ni, Sn, and Zn, Ln is one or more of Y, La, Ce, Nd, Sm) alloy is rapidly cooled and solidified, thereby obtaining an amorphous foil strip composed of a magnesium alloy foil strip having a hardness of 200 HV or more.

40 **[0008]** Japanese Patent Unexamined Publication No. 2003-293069 discloses a technique in which a cast material or an extruded material composed of a Mg-Al-Mn alloy is drawn, thereby obtaining a magnesium alloy wire having a tensile strength of 250 MPa or more and an elongation of 6 % or more.

45 **[0009]** The techniques disclosed in the publications are effective for greatly strengthening magnesium alloys. However, in the magnesium alloy disclosed in Japanese Patent Unexamined Publication No. 3-90530, mechanical properties for satisfying requirements of the market as strong parts are not sufficient. For example, when it is assumed that the alloy is applied to a spring in which at least one of bending stress and twisting stress primarily acts, according to estimates by the inventors, the magnesium alloy wire rod must have a 0.2 % proof stress of 550 MPa or more in an inner portion of the wire rod and a 0.2 % proof stress of 650 MPa or more in the vicinity of the surface of the wire rod if the size of the wire rod is the same as that of existing steel springs and light weight can be achieved. Furthermore, in order to form a coiled spring, at least an elongation of 5 % or more in an inner portion is required. However, in the invention product disclosed in Japanese Patent Unexamined Publication No. 3-90530, which has the highest 0.2 % proof stress of 565 MPa, the ductility is low and the elongation is merely 1.6 %. On the other hand, in the invention product disclosed in Japanese Patent Unexamined Publication No. 3-90530, which has the highest ductility and an elongation of 4.7 %, the elongation is close to the value that is required in the present invention. However, the strength is low in a 0.2 % proof stress of 535 MPa, and the requirement is not satisfied.

50 **[0010]** In the magnesium alloy disclosed in Japanese Patent Unexamined Publication No. 3-10041, a hardness of 170 HV or more is obtained. The hardness corresponds to 0.2 % proof stress of 650 MPa or more according to estimates by the inventors. However, in Japanese Patent Unexamined Publication No. 3-10041, properties related to ductility are not disclosed. The magnesium alloy disclosed in this publication contains a large amount of rare earth elements and 50 % of amorphous phase, whereby the ductility is extremely low, and it is easily assumed that the elongation that is required

in the present invention is not obtained. Furthermore, amorphous phases show poor thermal stability and easily crystallize by external causes such as environmental temperature. Since a mix-phase alloy of amorphous phase and crystal phase greatly varies the properties according to the proportion of the phases, it is difficult to stably produce products having uniform properties, and it is not suitable for applying to industrial products because of difficulty of quality guaranty and safety guaranty in the market.

[0011] In the magnesium alloy disclosed in Japanese Patent Unexamined Publication No. 2003-293069, the elongation is 6 % or more and shows sufficient ductility. However, the tensile strength is 479 MPa at most, and the above-mentioned 0.2 % proof stress of 550 MPa or more in the inner portion of the wire rod is not obtained.

Disclosure of the Invention

[0012] Thus, the conventional magnesium alloys do not satisfy 0.2 % proof stress and elongation for strong parts (for example, springs) to which at least one of bending stress and twisting stress primarily acts. Therefore, an object of the present invention is to provide a high-strength magnesium alloy wire rod, a high-strength magnesium alloy part, and production method therefor, in which 0.2 % proof stress and elongation, which are in a trade-off, are both satisfied, whereby strength and formability (ductility required for bending and coiling) are improved, and higher surface strength is provided, thereby being suitable for products in which at least one of bending stress and twisting stress primarily acts.

[0013] The present invention provides a high-strength magnesium alloy wire rod used for members in which at least one of bending stress and twisting stress primarily acts, the wire rod comprising: a surface portion having the highest hardness in a cross section of the wire rod, the highest hardness being 170 HV or more, and an inner portion having a 0.2 % proof stress of 550 MPa or more and an elongation of 5 % or more.

[0014] The vicinity of a surface is defined as a range from the surface of the wire rod to a depth of about $d/10$ (d is the diameter of the wire rod). Since the wire rod has a surface portion having the highest hardness in the cross section of the wire rod and the highest hardness is 170 HV or more, 0.2 % proof stress of 650 MPa or more in the vicinity of the surface of the wire rod can be achieved, as mentioned as above. In the present invention, although strength (hardness) gradually decreases from in the vicinity of surface to the center of the wire rod, the inner portion has a 0.2 % proof stress of 550 MPa or more and an elongation of 5 % or more. That is, the present invention is a high-strength magnesium alloy having strength and formability suitable for products in which at least one of bending stress and twisting stress primarily acts.

[0015] Thus, since the present invention has a high-strength and high-ductile inner portion and a higher-strength portion in the vicinity of the surface, 0.2 % proof stress and elongation which are in relation of trade-off can be satisfied for products in which at least one of bending stress and twisting stress primarily acts by providing suitable distribution of mechanical properties. In this case, the outermost surface can be reformed by providing compressive residual stress by shot peening, whereby fatigue properties can further be improved for parts in which at least one of bending stress and twisting stress primarily acts.

[0016] Next, the present invention provides a production method for a high-strength magnesium alloy wire rod, the method comprising: a step for yielding a raw material in a form of foil strips, foil pieces, or fibers of a magnesium alloy by rapid solidification method, a sintering step for forming a billet by bonding, compressing, and sintering the raw material, a step for plastic forming the billet, thereby obtaining the above-mentioned wire rod.

[0017] In the present invention, a raw material having below-mentioned compositions in a form of foil strips, foil pieces, or fibers of a magnesium alloy by rapid solidification method is preferably used. Therefore, special steps disclosed in Japanese Patent Unexamined Publication No. 3-90530, in which a raw material is charged in a container in a moment after forming or a raw material is subjected to canning, are not needed, although such steps are required for a powder having large specific surface area or an alloy having active composition.

[0018] The present invention provides another production method for a high-strength magnesium alloy wire rod, the method comprising: a step for forming fibers by molten metal extraction method, a sintering step for forming a billet by bonding, compressing, and sintering the fibers, an extruding step for directly charging the billet into a container of a press machine and extruding the billet, thereby obtaining the above-mentioned wire rod.

[0019] In the present invention, a billet that is not subjected to canning is directly extruded, whereby a high-strength and high-ductile inner portion and a higher-strength portion in the vicinity of the surface can be obtained. The high-strength and high-ductile inner portion and the higher-strength portion in the vicinity of the surface are gradually connected and do not have a clear boundary of mechanical properties. This is greatly preferable for fatigue in which cyclic stresses act. If the portions have a clear boundary, the boundary may be an initiation of a crack due to difference of hardness or elastic strain. Therefore, since the portions do not have a clear boundary and are gradually connected, there is no risk that a boundary will be an initiation of a crack. In the present invention, since a billet is directly charged into a container of a press machine, the number of steps can be reduced and production cost can be lowered compared to the case in which canning is performed.

Effects of the Present Invention

[0020] According to the present invention, a high-strength magnesium alloy wire rod has high-surface strength and high formability. Therefore, by applying the invention to formed parts in which at least one of bending stress and twisting stress primarily acts, great reduction in weight of parts can be achieved without increasing size of parts compared to conventional steel parts. Specifically, the present invention has strength and formability that are sufficient for, for example, automobile parts such as seat frames which have higher proportion of weight and springs (suspension springs, valve springs, clutch torsion springs, torsion bars, stabilizers) which are required to have high strength.

Brief Description of the Drawings

[0021] Figs. 1A and 1B show a production apparatus for metallic fiber used in an embodiment of the present invention.

[0022] Fig. 2 shows an extrusion apparatus used in an embodiment of the present invention.

[0023] Fig. 3 shows a graph showing a relationship between distance from the center in a cross section and hardness of a wire rod in each extruding temperature in the example of the present invention.

[0024] Fig. 4 shows a graph showing a relationship between distance from the center in a cross section and hardness of a wire rod in each composition of a material in the example of the present invention.

[0025] Fig. 5 shows a graph showing a relationship between distance from the center in a cross section and hardness of a wire rod in each inner diameter of a container and each extruding rate.

Embodiment of the Invention

1. Composition of Materials

[0026] Zn has been conventionally added in magnesium alloys as a primary additional element for improving strength and ductility. However, Zn is not sufficient for compatibility of high strength and ductility which are required in the present invention. Therefore, Ni is preferably added in magnesium alloys as a primary additional element. Ni has great function for improving high strength and high ductility compared to Zn.

[0027] However, high strength, which is required in the present invention, is not easy merely by adding Ni, which greatly contributes to improving high strength and high ductility. Therefore, Y is preferably added as a secondary additional element. A high-strength compound phase of Mg-Ni-Y type is formed by adding Y. Y has high solubility with respect to Mg and is effective for solid-solution strengthening in an α -Mg phase. Furthermore, by combining with yielding a raw material by a rapid solidification method, as mentioned below, greater strengthening can be achieved. The magnesium alloy in the present invention is not limited to compositions composed of three elements of Mg, Ni, and Y. The main elements are Mg, Ni, and Y, and a third element such as Zr and Al can be added for refinement of crystal grain and improvement of corrosion resistance.

[0028] When a magnesium alloy in which Ni and Y are added to Mg as a main element is used, the alloy preferably consists of, by atomic %, Ni: 2 to 5 %, Y: 2 to 5 %, and the balance of Mg and inevitable impurities. If Ni is less than 2 atomic % and Y is less than 2 atomic %, the highest hardness in the vicinity of the surface is not the hardness required in the present invention and the strength is not sufficient for strength parts in which at least one of bending stress and twisting stress primarily acts. On the other hand, if Ni is more than 5 atomic % and Y is more than 5 atomic %, formability is extremely deteriorated and breakage occurs in extruding. In this case, amount of high-hardness compounds formed by Ni and Y increases and the compounds become coarse, whereby the deformation resistance of the alloy increases and the toughness of the alloy is decreased, and thereby the alloy breaks.

2. Production of Raw Materials

[0029] A raw material of a magnesium alloy having the above composition is produced. A rapid solidification method such as a single roller method, a molten metal spinning method, and a molten metal extraction method was used, and a raw material in a form of foil strips, foil pieces, or fibers was produced. The amounts of additional elements contained by solid solution in an α -Mg phase of foil strips, foil pieces, or fibers which is yielded by rapid solidification method is large compared to common casting methods in which solidification rate is low. Therefore, even though amounts of additional elements are the same as in the casting method, the alloy is greatly strengthened by solid solution strengthening. The crystal grain is fine in a rapid solidification method. Fine crystal grain improves strength and elongation, and combined with solid solution strengthening, all of the mechanical properties are improved.

[0030] It should be noted that rapid solidification powders such as atomized powder that is yielded by rapid solidification of a raw material is not suitable for the present invention. Since Mg is active, an extremely thin oxide film is easily formed on a surface of the powder when Mg is exposed in air. In a powder having large specific surface area, the total area of

the oxide film is greatly large compared to that of foil strips, foil pieces, or fibers. If obtained powder is exposed in air and subjected to sintering, the oxide film prevents bonding at the contacting surface of the particles. Even though particles are bonded, oxides or oxygen generated by resolution of the oxides is largely taken in the particles. Thus, in powders having large specific surface area, poor bonding and embrittlement caused by contamination of oxygen and oxides easily occur, whereby the properties may be reduced compared to the case in which foil strips, foil pieces, or fibers are used. In order to avoid such disadvantages, powders must be subjected to canning in a moment after forming the powder. As a result, high strengthening in the vicinity of a surface of a wire rod after plastic forming (for example, extruding) is difficult, as mentioned below.

[0031] In a condition of a powder, there may be a concern that a dust explosion may occur. Therefore, active magnesium alloy powder cannot be handled in air. Specifically, if powder is used, powder that is yielded in a vacuum or in an inert atmosphere must not be exposed to air, and is charged into a metallic capsule such as copper capsule in a sequential apparatus having a vacuum or an inert atmosphere. When an inert atmosphere is used, the metallic capsule is degassed and sealed. Thus, if powder is used, the above-mentioned canning in a vacuum process or an inert atmosphere process is required. In an apparatus for performing canning in a vacuum or in an inert atmosphere, the sizes of products are limited. Therefore, it is difficult to realize sequential processes composed of a vacuum process or an inert atmosphere process using powder in industrial mass-production with respect to parts having such sizes as springs for automobiles (suspension springs, valve springs, clutch torsion springs, torsion bars, stabilizers) and seat frames.

[0032] Figs. 1A and 1B show schematic structures of a production apparatus for metallic fiber 100 (hereinafter referred to simply as "apparatus 100") for performing a step for forming a fiber in an embodiment of the present invention, Fig. 1A shows a cross sectional view of the entire apparatus 100 and Fig. 1B shows a cross sectional view of a circumferential portion 141a of a rotating disk 141. Fig. 1B is a side sectional view in a direction perpendicular to the plane of the paper.

[0033] The apparatus 100 is a production apparatus for metallic fiber using a molten metal extraction method. In the apparatus 100 using a molten metal extraction method, an upper end portion of a rod-shaped raw material M is melted, and a molten metal Ma contacts the circumferential portion 141a of the rotating disk 141, a portion of the molten metal Ma is extracted toward the direction of the substantially tangential line of the circumference of the disk 141, and is rapidly cooled, thereby forming a magnesium alloy fiber F. For example, a magnesium alloy such as Mg-Ni-Y type is used as a raw material M, and a magnesium alloy fiber F having a diameter 200 μ m or less is produced. The diameter of the magnesium alloy fiber F is not limited, and the diameter is selected according to production efficiency and handling facility in a later process. When diameter is 200 μ m or less, sufficient amounts of additional elements can be contained in α -Mg phase by solid solution, and the structure can be fine.

[0034] As shown in Fig. 1A, the apparatus 100 includes a chamber 101 which can be sealed. A raw material feeding portion 110, a raw material holding portion 120, a heating portion 130, a metallic-fiber forming portion 140, a temperature measuring portion 150, a high-frequency generating portion 160, and a metallic fiber receiving portion 170 are provided in the chamber 101.

[0035] An inert gas such as argon gas is provided in the chamber 101 as an atmosphere gas, thereby inhibiting reaction of impurities such as oxygen included in an atmosphere gas and a molten material Ma. The raw material feeding portion 110 is located at the bottom of the chamber 101, feeds the raw material M toward the direction of the arrow B at predetermined speed, and provides the raw material M to the raw material holding portion 120. The raw material holding portion 120 prevents movement of the molten material Ma toward a radial direction thereof and guides the raw material M toward a suitable position of the fiber forming portion 140.

[0036] The raw material holding portion 120 is a tubular member and is located between the raw material feeding portion 110 and the metallic fiber-forming portion 140 and below the disk 141. The heating portion 130 is a high-frequency induction coil which generates magnetic flux for melting the upper portion of the raw material M and forming the molten material Ma. As a material for the raw material holding portion 120, a material which does not react with the molten material Ma is preferable. Graphite is preferable as a material for the raw material holding portion 120 for practical use.

[0037] The fiber forming portion 140 produces a magnesium alloy fiber F from the molten material Ma by the disk 141 which rotates around a rotating shaft 142. The disk 141 is made from copper or a copper alloy having high thermal conductivity. As shown in Fig. 1B, a V-shaped circumference 141a is formed on the circumferential portion of the disk 141.

[0038] The temperature measuring portion 150 measures the temperature of the molten material Ma. The high-frequency generating portion 160 provides high-frequency current to the heating portion 130. The power of the high-frequency generating portion 160 is controlled based on the temperature of the molten material Ma, which is measured by the temperature measuring portion 150, and the temperature of the molten material Ma is maintained to be constant. The metallic fiber receiving portion 170 receives the metallic fiber F which is formed by the metallic fiber forming portion 140.

[0039] In the above apparatus, the raw material feeding portion 110 continually feeds the raw material M in a direction of the arrow B, thereby supplying it to the raw material holding portion 120. The heating portion 130 melts the upper portion of the raw material M by induction heating, thereby forming the molten material Ma. Then, the molten material Ma is continually fed to the circumference 141a of the disk 141 rotating in the direction of the arrow A, the molten material

Ma contacts the circumference 141a of the disk 141, a part thereof is extracted toward a direction of an approximate tangential line of the circle of the disk 141 and is rapidly cooled, thereby forming a magnesium alloy fiber F. The formed magnesium alloy fiber F extends toward the direction of an approximate tangential line of the circle of the disk 141 and received by the metallic fiber receiving portion 170 which is located in the direction in which the fiber F extends.

3. Sintering

[0040] The yielded raw material is formed to a billet for plastic working by sintering. Sintering is performed by atmosphere sintering, vacuum sintering, or discharge plasma sintering in a non-pressurized or a pressurized condition. Properties and quality of the billet after sintering affect properties and quality of products after plastic working. Therefore, in order to form a billet in which the cleanliness is high, the structure is uniform, and number of pores is small, sintering is preferably performed by a vacuum hot press (HP) apparatus which has a compressing mechanism and enables sintering in a vacuum or an inert gas atmosphere. By compressing heating in vacuum or an inert gas atmosphere, a billet which has few pores can be obtained.

[0041] In an HP apparatus, a heating chamber is disposed in a vacuum vessel, a mold is disposed in the heating chamber, a cylinder is disposed in the upper portion of the vacuum vessel, a press ram projected from the cylinder is vertically movable in the heating chamber, and an upper punch installed at the press ram is inserted into the mold. A magnesium alloy fiber F as a raw material is charged into the mold of the HP apparatus constructed as above, the vacuum vessel is evacuated or purged with an inert gas, and the heating chamber is heated to a predetermined sintering temperature. Then, the magnesium alloy fiber F is compressed by the upper punch inserted into the mold, and is sintered.

[0042] The sintering is preferably performed at a temperature of 250 to 500 °C for 10 minutes or more at a pressure of 25 MPa or more. By such conditions, a billet in which sintering is sufficiently promoted at contacting points of the magnesium alloy fibers can be obtained. More preferably, sintering is performed at a temperature of 350 to 500 °C for 30 minutes or more at a pressure of 40 MPa or more. By such conditions, a densified billet in which sintering is sufficiently promoted at contacting points of the magnesium alloy fibers and the porosity thereof is less than 10 % can be obtained. It should be noted that if the heating temperature is less than 250 °C, sintering is not sufficiently promoted at contacting points of the magnesium alloy fibers and large numbers of pores are remained. In the products after plastic working, contacting points which are not sufficiently sintered and boundaries of magnesium alloy fibers which are not sintered are remained, whereby the strength is lowered. Therefore, the heating temperature is preferably 250 °C or more. If the heating temperature is more than 500 °C, sintering is sufficiently promoted at contacting points of fibers and pores are few. However, in this condition, the structure is coarse and products after plastic working do not have required fine structure. As a result, a magnesium alloy wire rod having desired strength cannot be obtained. Therefore, the heating temperature is preferably 500 °C or less.

[0043] If the raw material is a powder, sintering must be performed before sealing in canning. However, a big sequential apparatus for providing a vacuum or an inert atmosphere is required, and it is difficult to uniformly charge a powder into a mold or a metallic capsule in a closed apparatus. As a result, it is difficult to produce a densified compact. That is, if a powder is used, canning must be performed before the powder is exposed to air, and sintering of the particles in the compact in the capsule is insufficient. Furthermore, the compact has large numbers of pores and density thereof is not uniform. Since the compact has pores communicated with the surface thereof, the inner portion thereof is exposed to air after the metallic capsule is removed. Therefore, the metallic capsule cannot be removed in a condition of a billet, whereby next process of plastic working must be performed in a condition of canning.

4. Plastic Working

[0044] Working from a billet to a wire rod is performed by warm plastic working such as drawing, rolling, extruding, or forging. Plastic working performed at a suitable temperature and a working ratio (reduction ratio of cross section) generates refinement of the structure caused by dynamic recrystallization and work hardening, and is effective for high strengthening of the magnesium alloy. In these plastic working, drawing and extruding are preferable for wire rods in which at least one of bending stress and twisting stress primarily acts. In the plastic working, a uniform cross section, which is indispensable for a wire rod can be obtained and greater strain can be introduced in the surface area of the wire rod compared to the inner portion thereof. As a result, the structure in the surface area is further refined and strengthened compared to the inner portion.

[0045] Naturally, strength and elongation are in a trade-off. Magnesium alloys in which the structure is refined and highly strengthened have been researched by using powders. Although the magnesium alloys had high-strength structure, they did not have sufficient elongation and was not able to be formed to a shape of part. Since the powder was charged into a metallic capsule and worked, strain generated by the working is preferentially introduced to the metallic capsule that was the outermost portion. Therefore, high-strengthening of the portion in the vicinity of the surface as obtained by the present invention could not be obtained.

[0046] In the case in which a billet is produced by casting, high strengthening cannot be obtained even if the magnesium alloy has the same composition as in the present invention. The reason for this is that the crystal grain of an α -Mg phase in a cast metal is naturally coarse and precipitated compounds are also coarse, deformation resistance is large, and accumulation of strain is large, whereby the metal is shear fractured before obtaining required fine structure. Furthermore, the amounts of additional elements contained in the α -Mg phase by solid solution is small, whereby high strengthening of the α -Mg phase by solid solution is poor. In contrast, in the billet produced from foil strips, foil pieces, or fibers having fine structure, by sintering at a suitable temperature, the working resistance is small since the structure after sintering is fine. Therefore, since the billet has superior deformability, large strain can be introduced at a lower temperature in plastic working, and large internal energy, which is a driving force for recrystallization can be accumulated, whereby further fine structure can be obtained. Furthermore, since the amount of additional elements contained in the α -Mg phase by solid solution is large, high strengthening is achieved as a joint result of large effects of solid-solution strengthening and the fine structure.

[0047] Fig. 2 shows an extruding apparatus 200 used when extruding is applied as plastic working. In Fig. 2, reference numeral 205 is an outer mold, reference numeral 210 is a container installed in the outer mold. The container 210 has a tubular shape. A lower mold 220 is coaxially disposed at an end surface of the container 210. A die 230 is disposed between the container 210 and the lower mold 220. A punch 240 is slidably inserted in the container 210. A heater 260 is disposed in the outer circumference of the container 210.

[0048] In the extruding apparatus 200, a billet B which is heated is charged into the container 210, the punch 240 moves downward, thereby compressing the billet B. The diameter of the compressed billet B is reduced by the die 230 and the billet B is extruded to the space of the lower mold 220, thereby forming a wire rod.

[0049] The extrusion in the extruding apparatus is preferably performed at a temperature of the billet B of 315 to 335 °C, at an extrusion ratio of 5 to 13, and at a forwarding speed of the punch 240 of 0.01 to 2.5 mm/second. By such conditions, refinement of structure caused by dynamic recrystallization and work hardening caused by introduction of strain are sufficient. Therefore, a high-strength magnesium alloy wire rod in which the inner portion thereof has high strength and high ductility and the portion in the vicinity of the surface has higher strength, is formed. Specifically, the portion in the vicinity of the surface has the highest hardness of 170 HV or more and the inner portion has a 0.2 % proof stress of 550 MPa or more and an elongation of 5 % or more, whereby a magnesium alloy wire rod suitable for a strength part in which at least one of bending stress and twisting stress primarily acts is obtained.

[0050] It should be noted that if the heating temperature is less than 315 °C, extruding is difficult since the deformation resistance is large, thereby resulting breakage in extruding and rough surface and cracking in the surface of the wire rod. Even though a wire rod is formed, the hardness of the wire rod is too high and elongation is deteriorated, whereby elongation of 5 % or more which is required to be formed cannot be obtained. On the other hand, if the heating temperature is more than 335 °C, refinement of structure caused by dynamic recrystallization and work hardening caused by introduction of strain are not sufficient. As a result, required hardness in the vicinity of the surface cannot be obtained, whereby the wire rod cannot be applied to parts in which at least one of bending stress and twisting stress primarily acts.

[0051] The conditions in the extruding are not limited to the abovementioned range and below-mentioned examples, and should be decided in focusing on obtaining high strength and high elongation in the inner portion and higher strength in the vicinity of the surface. That is, introduction of strain and inducement of dynamic recrystallization are affected by complex relationship of composition of the material, working ratio, working temperature, and so on, whereby the conditions should be suitably decided based on theory, experience, and experimentation.

[0052] The average crystal grain diameter of α -Mg phase in the portion having the highest hardness in the vicinity of the surface of the high-strength magnesium alloy wire rod produced in the above is preferably 1 μ m or less measured by an EBSD method. It is well known that refinement of crystal grain greatly contributes to high strengthening as well as the theory of Hall-Petch. Refinement of crystal grain is effective for inhibiting generation of initial crack on a surface of a fatigue part to which repeated stress acts. In the below-mentioned practical examples of the present invention having the highest hardness in the vicinity of the surface and the highest hardness is 170 HV or more, the average crystal grain diameter is greatly fine at 1 μ m or less, whereby the examples are suitable for static strength and fatigue strength.

Examples

[0053] The present invention will be explained in detail by way of specific examples. Raw materials of each element for casting were weighed such that required composition of a magnesium alloy and required size of a cast metal were obtained, and raw materials were melted in a vacuum and were cast. The compositions of the cast metals are shown in Table 1. In the melting, a crucible made from graphite and a die made from a copper alloy were used. Fibers were produced using the apparatus shown in Fig. 1 according to a molten metal extraction method. In production of fibers according to the molten metal extraction method, a raw material holding portion made from graphite and a disk made from a copper alloy were used, and fibers having an average diameter of 60 μ m were produced in an argon gas substituted inert atmosphere.

[0054]

Table 1

No.	Composition(at%)			Form of billet	Sintering temperature (°C)	Inner diameter of container (mm)	Extruding ratio	Extruding temperature (°C)	Extruding speed (mm/second)	Result
	Mg	Ni	Y							
Practical Example 1	93.5	3.0	3.5	Fiber sintered body	400	35	10	300	0.05	Bad
Practical Example2	↑	↑	↑	↑	↑	↑	↑	305	↑	Bad
Practical Example3	↑	↑	↑	↑	↑	↑	↑	310	↑	Notgood
Practical Example4	↑	↑	↑	↑	↑	↑	↑	315	↑	Good
Practical Example5	↑	↑	↑	↑	↑	↑	↑	320	↑	Good
Practical Example6	↑	↑	↑	↑	↑	↑	↑	325	↑	Good
Practical Example7	↑	↑	↑	↑	↑	↑	↑	330	↑	Good
Practical Example8	↑	↑	↑	↑	↑	↑	↑	335	↑	Good
Practical Example9	↑	↑	↑	↑	↑	↑	↑	340	↑	Good
Practical Example10	↑	↑	↑	↑	↑	↑	↑	350	↑	Good
Practical Example11	↑	↑	↑	↑	↑	↑	↑	375	↑	Good
Practical Example12	↑	↑	↑	↑	↑	↑	↑	400	↑	Good
Practical Example13	98.0	1.0	1.0	↑	↑	↑	↑	325	↑	Good

(continued)

No.	Composition(at%)			Form of billet	Sintering temperature (°C)	Inner diameter of container (mm)	Extruding ratio	Extruding temperature (°C)	Extruding speed (mm/second)	Result
	Mg	Ni	Y							
Practical Example14	96.0	2.0	2.0	↑	↑	↑	↑	↑	↑	Good
Practical Example15	90.0	5.0	5.0	↑	↑	↑	↑	↑	↑	Good
Practical Example16	88.0	6.0	6.0	↑	↑	↑	↑	↑	↑	Bad
Practical Example17	93.5	3.0	3.5	↑	300	↑	↑	↑	↑	Good
Practical Example18	↑	↑	↑	↑	350	↑	↑	↑	↑	Good
Practical Example19	↑	↑	↑	↑	450	↑	↑	↑	↑	Good
Practical Example20	↑	↑	↑	↑	500	↑	↑	↑	↑	Good
Practical Example21	↑	↑	↑	↑	525	↑	↑	↑	↑	Good
Practical Example22	↑	↑	↑	↑	400	16	↑	325	↑	Good
Practical Example23	↑	↑	↑	↑	↑	↑	↑	350	↑	Good
Practical Example24	↑	↑	↑	↑	↑	35	3	325	↑	Good
Practical Example25	↑	↑	↑	↑	↑	↑	5	↑	↑	Good
Practical Example26	↑	↑	↑	↑	↑	↑	13	↑	↑	Good
Practical Example27	↑	↑	↑	↑	↑	↑	15	↑	↑	Bad

(continued)

No.	Composition(at%)			Form of billet	Sintering temperature (°C)	Inner diameter of container (mm)	Extruding ratio	Extruding temperature (°C)	Extruding speed (mm/second)	Result
	Mg	Ni	Y							
Practical Example28	↑	↑	↑	↑	↑	↑	10	↑	0.01	Good
Practical Example29	↑	↑	↑	↑	↑	↑	↑	↑	0.5	Good
Practical Example30	↑	↑	↑	↑	↑	↑	↑	↑	2.5	Good
Practical Example31	↑	↑	↑	↑	↑	↑	↑	↑	5	Not good
Comparative Example1	93.5	3.0	3.5	Cast metal	-	↑	↑	375	0.05	Bad
Comparative Example2	↑	↑	↑	↑	-	↑	↑	400	↑	Notgood
Comparative Example3	↑	↑	↑	↑	-	↑	↑	425	↑	Good

[0055] The produced fibers were directly charges into a sintering die made from graphite without canning, and sintered by HP method, thereby obtaining a billet having a diameter of 15 mm and a length of 50 mm and a billet having a diameter of 33 mm and a length of 50 mm. The sintering according to the HP method was performed at a temperature of 300 to 525 °C and at a pressure of 50 MPa in an argon gas substituted inert atmosphere (atmosphere pressure of 0.08 MPa).

[0056] Next, the billet was formed to a wire rod using the extruding apparatus shown in Fig. 2. Specifically, a graphite type lubricant (provided by Japan Acheson, OILDAG-E) was used, the extruding speed (forward speed of the punch 240) was 0.01 to 5 mm/minute, and the extruding temperature was 300 to 425 °C as shown in Table 1. The billet having a diameter of 15 mm was extruded using a container 210 having an inner diameter of 16 mm and a die 230 having a bore diameter of 5 mm (extruding ratio of 10), thereby obtaining a wire rod. The billet having a diameter of 33 mm was extruded using a container 210 having an inner diameter of 35 mm, a die 230 having a bore diameter of 20 mm (extruding ratio of 3), a die 230 having a bore diameter of 15.5 mm (extruding ratio of 5), a die 230 having a bore diameter of 11 mm (extruding ratio of 10), a die 230 having a bore diameter of 9.7 mm (extruding ratio of 13), a die 230 having a bore diameter of 9 mm (extruding ratio of 15), thereby obtaining a wire rod. A cast billet was extruded for comparison.

[0057] Tensile test of the produced wire rod was performed. In the tensile test, a test piece having a 1.6 mm thick parallel portion was machined from the wire rod having a diameter of 5 mm, and a test piece having a 3 mm thick parallel portion was machined from the wire rod having a diameter of 9 mm or more. The test pieces were subjected to tensile test at room temperature using a universal testing machine (provided by Instron, No. 5586) at a test speed of 0.5 mm/minute. The results of the tensile test are shown in Table 2.

[0058]

Table 2

No.	0.2% Proof stress (MPa)	Elongation (%)	Hardness (HV)	
			Center	Highest value in the vicinity of surface
Practical Example3	670	4.0	169	168
Practical Example4	663	5.0	159	180
Practical Example5	643	5.2	158	182
Practical Example6	620	5.3	160	181
Practical Example7	613	5.9	154	178
Practical Example8	580	6.2	152	170
Practical Example9	563	6.4	146	168
Practical Example10	532	6.7	141	156
Practical Example11	510	10.3	138	155
Practical Example12	493	13.3	137	140
Practical Example13	540	7.2	144	159
Practical Example14	582	6.0	154	173
Practical Example15	660	5.1	165	183
Practical Example17	422	3.8	164	165
Practical Example18	579	5.2	163	173
Practical Example19	623	5.5	161	183
Practical Example20	601	6.1	159	174
Practical Example21	483	8.2	143	152
Practical Example22	645	5.0	158	175
Practical Example23	633	5.1	144	159
Practical Example24	483	7.7	136	142
Practical Example25	551	7.0	153	172

(continued)

No.	0.2% Proof stress (MPa)	Elongation (%)	Hardness (HV)	
			Center	Highest value in the vicinity of surface
Practical Example26	655	5.0	159	177
Practical Example28	615	5.4	161	182
Practical Example29	622	5.2	158	176
Practical Example30	625	5.2	159	179
Practical Example31	600	5.1	158	155
Comparative Example2	408	10.6	109	129
Comparative Example3	399	10.0	102	130

[0059] In Table 1, the section specified by "Form of billet" shows a production method of a billet before extruding, "Fiber sintered body" shows a billet obtained by sintering fibers, and "Cast metal" shows a billet as cast. In Table 1, "Bad" shows the case in which breakage occurred in extruding and a wire rod could not be obtained, "Not good" shows the case in which rough surface and cracking in a surface of a wire rod was confirmed by visual contact, although a wire rod was obtained, and "Good" shows the case in which a good wire rod without rough surface and cracking was obtained. The tensile test was performed to the test piece in which the result of extruding was "Not good" and "Good".

[0060] Hardness was measured with respect to the wire rod in which the result of extruding was "Not good" and "Good". The test piece for measuring hardness was embedded in a resin so that the cross section of the extruded wire rod is exposed and mirror finished by mechanical polishing. Distribution of hardness of the cross section of the extruded wire rod was measured using a Vickers hardness testing machine (provided by Future-Tech, No. FM-600) at a testing load of 25 gf. The result of the measuring hardness is shown in Table 2 and Figs 3 to 5.

[0061] In Table 2 and Figs. 3 to 5, the test pieces in which the highest hardness in the vicinity of the surface of the wire rod was 170 HV or more and 0.2 % proof stress of 550 MPa or more and elongation was 5.0 % or more in the inner portion were practical examples of the present invention (Practical Examples Nos. 4 to 8, 14, 15, 18 to 20, 22, 25, 26, and 28 to 30). The strength in the practical examples was very high compared to the Comparative Examples No. 2 and 3, which were produced from the cast billets. The inner portion of the wire rod had a high strength and high ductility portion in which the 0.2 % proof stress was 563 MPa or more and the elongation was 5 % or more was. In these practical examples, since the highest hardness in the vicinity of the surface was 170 HV or more, a higher strengthened portion in which the 0.2 % proof stress was 650 MPa or more was provided. The high strength and high ductility portion in the inner portion and the higher strengthened portion in the vicinity of the surface were gradually connected and did not have clear boundary, whereby the whole wire rod had superior strength and toughness and sufficient formability.

[0062] As shown in Table 1, in Practical Examples Nos. 1 and 2, deformation resistance was large since the extruding temperature (heating temperature of the billet) was low, breakage occurred in extruding and a wire rod was not obtained. In Practical Example No. 3, rough surface and cracks were generated in the surface layer, although a wire was obtained, and the inner portion was deteriorated in ductility as strengthening was promoted, whereby elongation of 5 % or more which was required for formability was not obtained.

[0063] In Practical Examples Nos. 9 to 12 and 23, since the extruding temperature was greater than 335 °C, refinement of structure caused by dynamic recrystallization and work hardening caused by introduction of strain were not sufficient. As a result, the highest hardness in the vicinity of the surface was less than 170 HV. Therefore, the hardness in the vicinity of the surface was insufficient for applying the wire rod to strong parts in which at least one of bending stress and twisting stress primarily acts. In Practical Example No. 13, since amounts of Ni and Y were small at 1.0 atomic %, solid solution strengthening in the α -Mg phase and amount of precipitated high strength Mg-Ni-Y type compound were small, the highest strength of 170 HV or more in the vicinity of the surface was not obtained. In contrast, in Practical Example No. 16, since amounts of Ni and Y were large at 6.0 atomic %, high-strength Mg-Ni-Y type compounds were greatly precipitated and coarse. As a result, deformation resistance was large and toughness was low, whereby breakage occurred in extruding.

[0064] In Practical Example No. 27, since the extruding ratio was more than 13, the wire rod was greatly strengthened and toughness was low, and breakage occurred in extruding. In Practical Example No. 21, since the sintering temperature was more than 500 °C, phase effective for high strengthening was decomposed and the crystal grain was coarse, whereby the hardness in the vicinity of the surface was less than 170 HV. In Practical Example No. 17, since the sintering temperature was less than 350 °C, a densified billet was not obtained. In the billet, a large amount of unbonded boundaries

of fibers, which was difficult to eliminate by the next process of plastic working and was a defect of a wire rod after extruding, were present, and bonding strength at contacting points of the magnesium alloy fibers was insufficient. As a result, sufficient 0.2 % proof stress and elongation were not obtained, although the hardness was improved. In Practical Example No. 31, since the extruding speed was more than 2.5 mm/second, lubrication was insufficient, whereby rough surfaces such as scuffing in the surface of the wire rod were formed. Deformation strain was released by such a rough surface, the hardness in the vicinity of the surface was less than 170 HV although a 0.2 % proof stress of 600 MPa and an elongation of 5.1 % in the inner portion were obtained. In Comparative Examples Nos. 1 and 2, since the billet was a cast metal, the α -Mg phase was coarse and the precipitated compound phases were also coarse. As a result, the deformation resistance and accumulation of strain were large. Therefore, in Practical Example No. 1, breakage occurred in extruding, and in Practical Example No. 2, rough surfaces and cracks were formed in extruding. In practical Example No. 3, required properties were not obtained, although breakage did not occur since the extruding temperature was high.

[0065] Next, the relationship between the average crystal grain diameter and the hardness in the α -Mg phase in the vicinity of the surface in the practical examples of the present invention and Comparative Example No. 3 was evaluated. The results are shown in Table 3. Measurement of the average crystal grain diameter of the α -Mg was performed on the test piece that was subjected to the measurement of the hardness using an EBSD method (electron beam back-scattering diffraction apparatus, provided by TSL) utilizing an FE-SEM (electrolysis emission type scanning electron microscope, provided by JEOL, No. JSM-7000F) and. The measurement was performed at the position in the vicinity of the surface at which the highest hardness was obtained for practical examples at analysis magnification of 10,000 times and for Comparative Example No. 3 at analysis magnification of 2,000 times. In Table 3, the highest hardness in the vicinity of the surface is shown together.

[0066]

Table 3

No.	Highest hardness in the vicinity of surface (HV)	Average crystal diameter of α -Mg phase (μm)
Practical Example4	180	0.21
Practical Example5	182	0.26
Practical Example6	181	0.23
Practical Example7	178	0.59
Practical Example8	170	0.35
Practical Example14	173	0.27
Practical Example15	183	0.20
Practical Example18	173	0.36
Practical Example19	183	0.62
Practical Example20	174	0.76
Practical Example22	175	0.33
Practical Example25	172	0.69
Practical Example26	177	0.33
Practical Example28	182	0.30
Practical Example29	176	0.19
Practical Example30	179	0.53
Comparative Example3	130	6.76

[0067] As shown in Table 3, the average crystal grain diameter of the α -Mg was very fine at 0.19 to 0.76 μm compared to 6.76 μm of Comparative Example No. 3. It is apparent that the fine crystal grain contributes to improvement of the hardness.

Industrial Applicability

[0068] The magnesium alloy wire rod of the present invention is suitable for a high-strength part in which at least one of bending stress and twisting stress primarily acts. By using the magnesium alloy wire rod of the present invention, great weight reduction can be achieved without increase in size of parts compared to conventional steel parts. The weight reduction is very effective for, for example, automobile parts such as seat frames, which have a higher proportion of weight, and springs (suspension springs, valve springs, clutch torsion springs, torsion bars, stabilizers) which are required to have high strength.

Claims

1. A high-strength magnesium alloy wire rod used for members in which at least one of bending stress and twisting stress primarily acts, the wire rod comprising:
 - a surface portion having the highest hardness in a cross section of the wire rod, the highest hardness being 170 HV or more;
 - an inner portion having a 0.2 % proof stress of 550 MPa or more and an elongation of 5 % or more.
2. The high-strength magnesium alloy wire rod according to claim 1, wherein the magnesium contains Mg as a main element and Ni and Y.
3. The high-strength magnesium alloy wire rod according to claim 2, wherein the magnesium consists of 2 to 5 atomic % of Ni, 2 to 5 atomic % of Y, and the balance of Mg and inevitable impurities.
4. The high-strength magnesium alloy wire rod according to one of claims 1 to 3, wherein the portion having the highest hardness in the vicinity of the surface has an average grain diameter of 1 μm measured by an EBSD method.
5. A production method for a high-strength magnesium alloy wire rod, the method comprising:
 - a step for yielding a raw material in a form of foil strips, foil pieces, or fibers of a magnesium alloy by a rapid solidification method,
 - a sintering step for forming a billet by bonding, compressing, and sintering the raw material,
 - a step for plastic forming the billet, thereby obtaining the wire rod according to one of claims 1 to 4.
6. A production method for a high-strength magnesium alloy wire rod, the method comprising:
 - a step for forming fibers by a molten metal extraction method,
 - a sintering step for forming a billet by bonding, compressing, and sintering the fibers,
 - an extruding step for directly charging the billet into a container of a press machine and extruding the billet, thereby obtaining the wire rod according to one of claims 1 to 4.
7. The production method for a high-strength magnesium alloy wire according to claim 5 or 6, wherein the sintering step is performed in a temperature of 350 to 500 $^{\circ}\text{C}$ for 10 minutes or more at a pressure of 25 MPa or more.
8. The production method for a high-strength magnesium alloy wire according to claim 6 or 7, wherein the extruding step is performed in a temperature of 315 to 335 $^{\circ}\text{C}$ at an extruding rate of 5 to 13 with a speed of 2.5 mm/second or less of a press ram.
9. A high-strength magnesium alloy part produced from the high-strength magnesium alloy wire according to one of claims 1 to 4.
10. A high-strength magnesium alloy spring produced from the high-strength magnesium alloy wire according to one of claims 1 to 4.

Fig. 1A

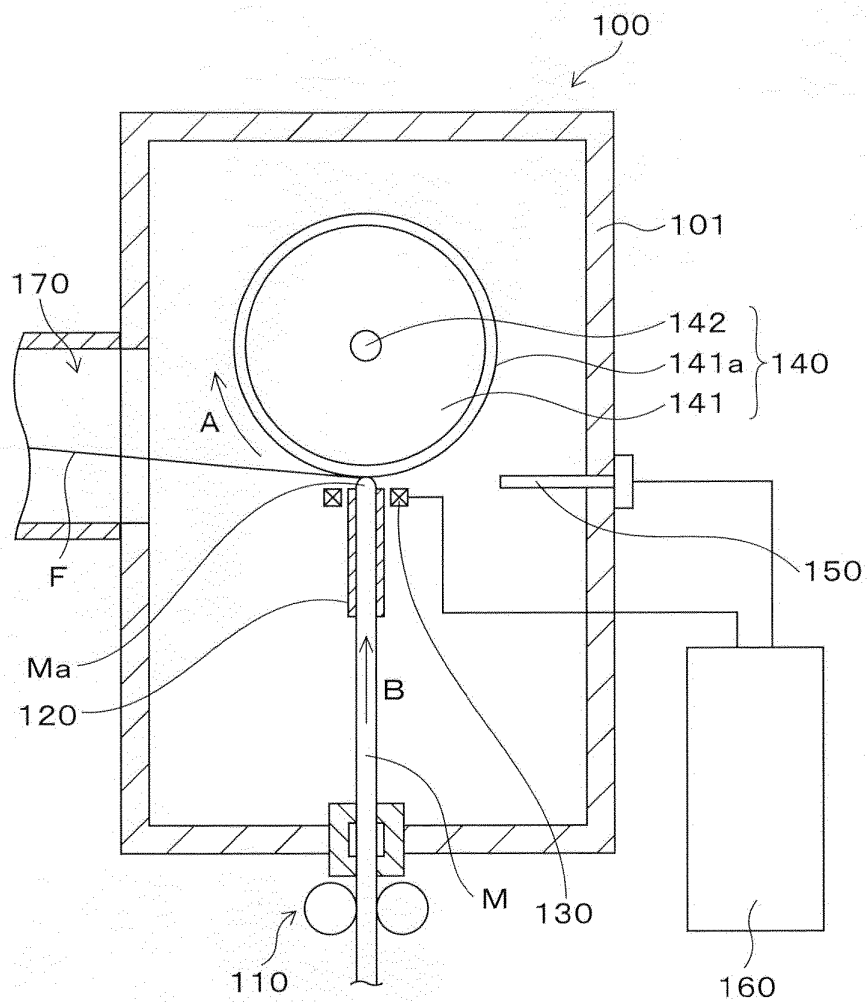


Fig. 1B

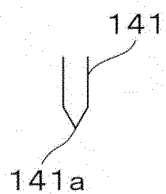


Fig. 2

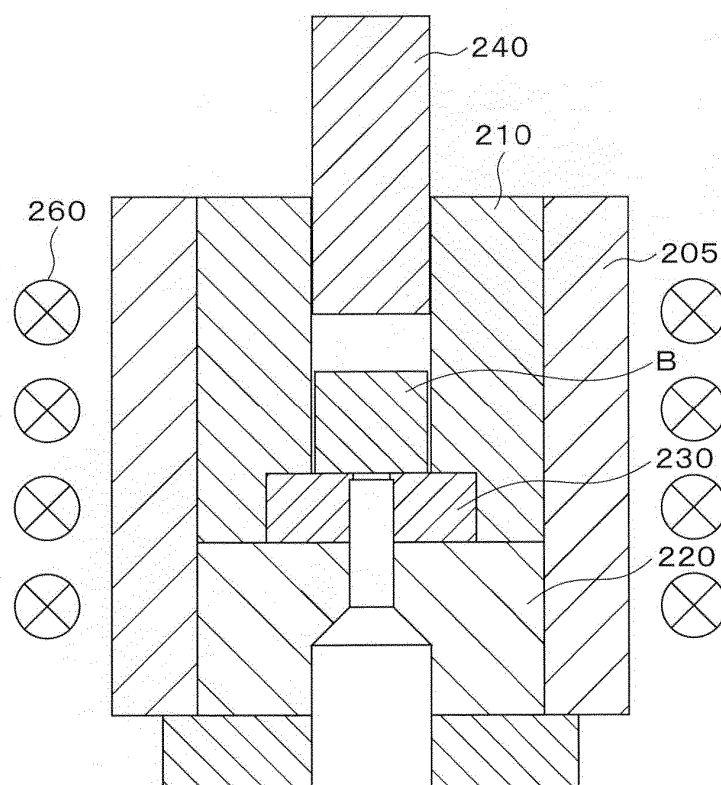


Fig. 3

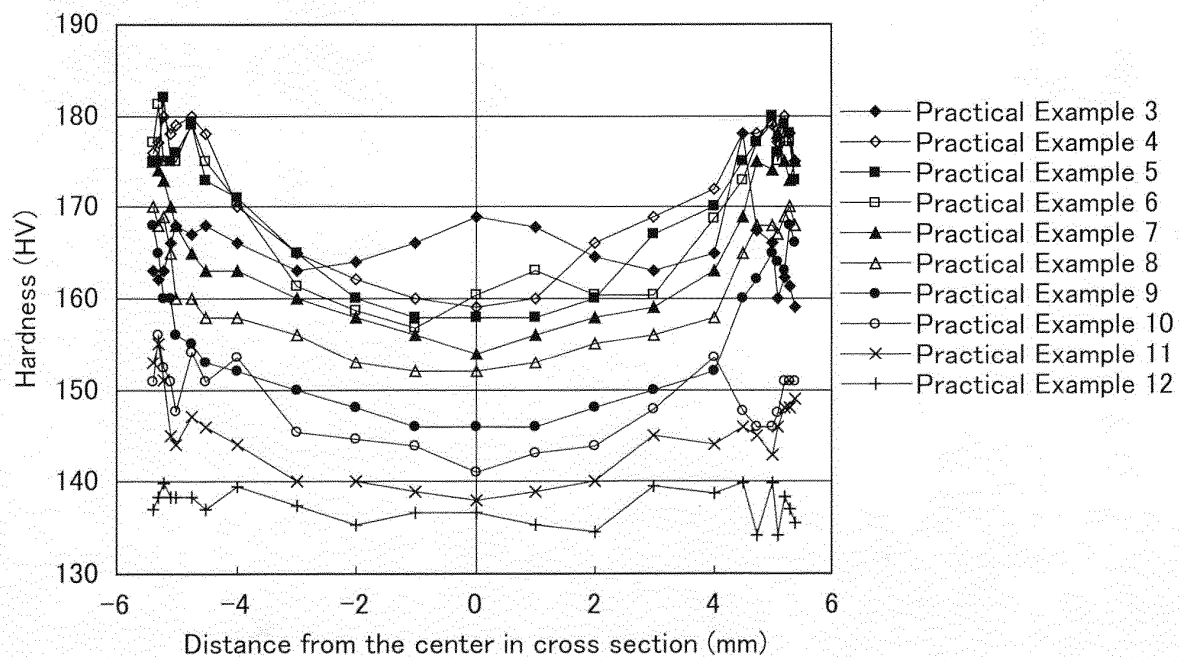


Fig. 4

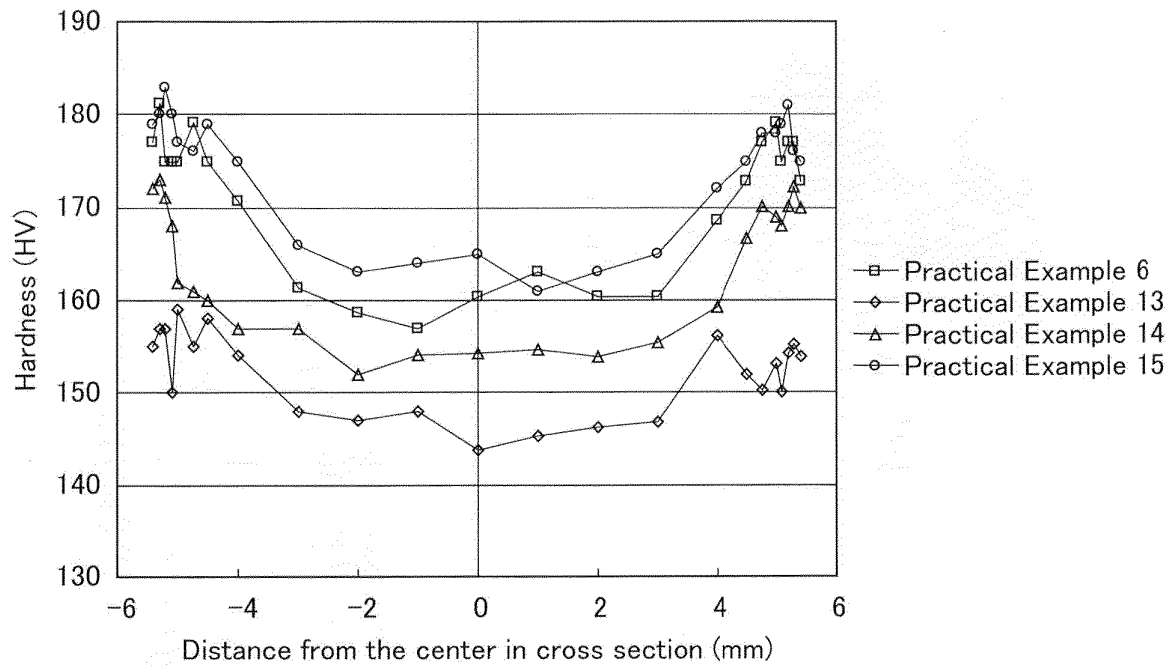
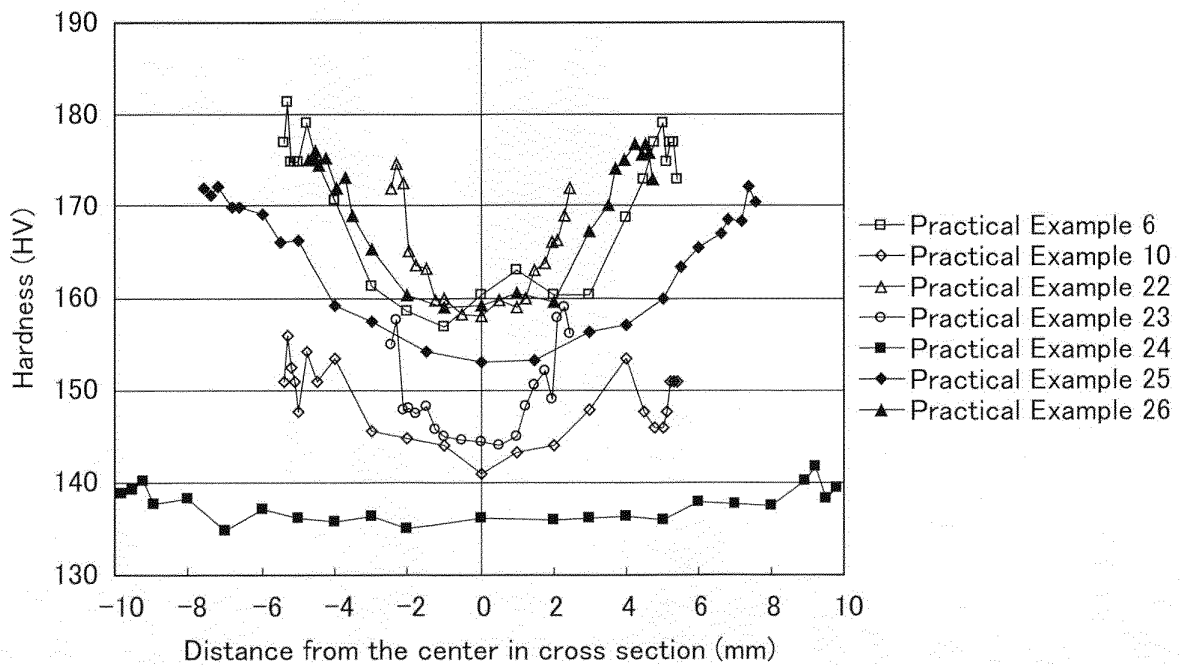


Fig. 5



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2011/073649

A. CLASSIFICATION OF SUBJECT MATTER

C22C23/06(2006.01)i, B22F3/14(2006.01)i, B22F3/20(2006.01)i, C22C23/00(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)

C22C23/06, B22F3/14, B22F3/20, C22C23/00

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

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2012

Kokai Jitsuyo Shinan Koho 1971-2012 Toroku Jitsuyo Shinan Koho 1994-2012

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2007/111342 A1 (Kumamoto University, National University Corporation Chiba University), 04 October 2007 (04.10.2007), entire text; all drawings & US 2009/0035171 A1 & DE 112007000673 T5 & CN 101405417 A	1-10
A	JP 3-47941 A (Yoshida Kogyo Co., Ltd.), 28 February 1991 (28.02.1991), entire text; all drawings & US 5304260 A & EP 0407964 A2 & DE 69028009 T2 & CA 2020484 C	1-10

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

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Date of the actual completion of the international search

04 January, 2012 (04.01.12)

Date of mailing of the international search report

17 January, 2012 (17.01.12)

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INTERNATIONAL SEARCH REPORT

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 5-320715 A (Mazda Motor Corp.), 03 December 1993 (03.12.1993), entire text; all drawings (Family: none)	1-10

Form PCT/ISA/210 (continuation of second sheet) (July 2009)

REFERENCES CITED IN THE DESCRIPTION

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- JP 3090530 A [0006] [0009] [0017]
- JP 3010041 A [0007] [0010]
- JP 2003293069 A [0008] [0011]