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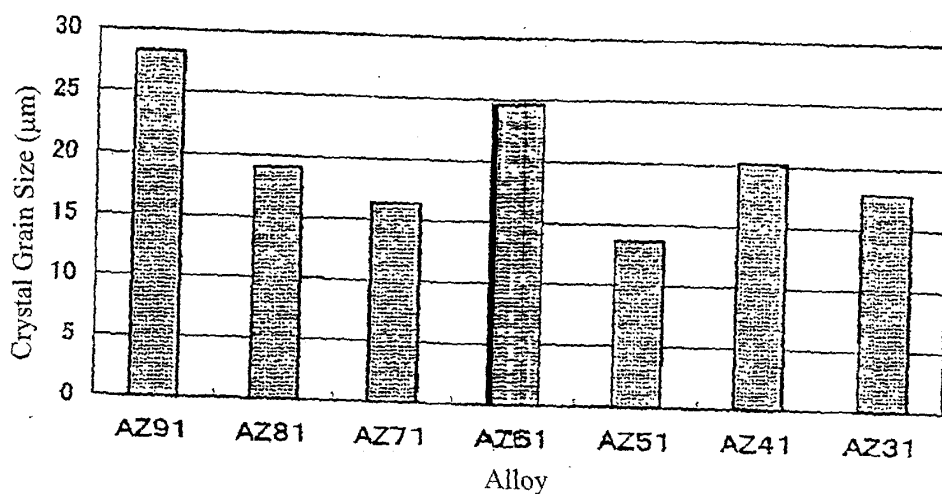
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• **TAKATA CORPORATION****Minato-ku, Tokyo 106-8510 (JP)**• **Higashi, Kenji****Tondabayashi-shi, Osaka 584-0073 (JP)**(54) **Method of manufacturing magnesium alloy products**

(57) In order to achieve the manufacture of products, which have complex and accurate figure and exhibit high reliability of properties and enough corrosion resistance, at sufficiently high yield ratio by employing a combination of casting and forging for forming magnesium alloy of which composition allows casting and which is excellent in forgeability, a magnesium alloy containing 2-10 mass % aluminum is cast to have crystal

grain size not greater than 30 μm . After the cast semi-finished product is subjected to solution treatment, the semifinished product is forged to have crystal grain size not greater than 10 μm and is then further forged to have a desired figure. A magnesium alloy containing 2-10 mass % aluminum is cast to have crystal grain size not greater than 10 μm and the cast semifinished product is forged after solution treatment.

**Fig. 1**

Description

[Industrial Field of the Invention]

[0001] The present invention relates to a method of manufacturing magnesium alloy products comprising casting a magnesium alloy and forging the thus obtained cast semifinished product into a predetermined figure.

[Related Art]

[0002] Since magnesium (Mg) has a specific gravity of 1.8 which is smaller than the specific gravity of 2.7 of aluminum (Al) as a typical light metal, magnesium alloys are extremely lightweight. In addition, magnesium alloys have higher rigidity than the rigidity of aluminum alloys and are excel in heat conductivity. Accordingly, magnesium alloys are widely used as materials for casings and parts of electric equipments and electronics devices.

[0003] Magnesium alloys have poor formability and are therefore hardly formed into desired figures. That is, magnesium alloys have small specific latent heat of solidification and are thus fast in solidification speed. Therefore, it is difficult to cast magnesium alloy so that defects such as porosities and flow lines may be easily created in obtained cast products. Especially on appearance-conscious products, the magnesium alloy has low yield ratio. Further, because putting should be required for such defects, the manufacturing cost should increase. Magnesium alloys have hexagonal close-packed crystal structure and have therefore low ductility. Since the work of pressing or forging a plate or rod material should be conducted at a high temperature from 300 to 500 °C, there are problems such as low processing speed, the increased number of processes, short die's life, and the like.

[0004] To solve the aforementioned problem of poor formability of magnesium alloys, the following method has been proposed in Japanese Unexamined Patent Publication No. H7-224344. That is, in a step forming billets by continuous casting of an AZ-type magnesium alloy containing 6.2 to 7.6 wt.% aluminum, the addition of miniaturization agent and/or the control of cooling speed makes the average grain size of 200 μm or less. The billets are then forged to manufacture large-size parts. Also disclosed in this publication is a method of making the average grain size smaller than 50 μm by a combination of solution treatment and the T6 heat treatment after formed into a final product figure, thereby improving the corrosion resistance.

[0005] On the other hand, the following method is proposed in Japanese Unexamined Patent Publication No. 2001-294966. That is, a magnesium alloy is injected by a die-cast or thixo molding machine to mold a plate. After rolling the plate at a normal temperature and giving a distortion to the plate, the plate is heated at a temperature from 350 to 400 °C to recrystallize the crystals to be miniaturized into small grains from 0.1 to 30 μm, thereby improving the ductility. By pressing or forging the obtained magnesium alloy plate having improved ductility, products formed in an arbitrary figure can be obtained. Further, in Japanese Unexamined Patent Publication Nos. 2001-170734 and 2001-170736, a method of forming a boss part having a thickness of seven times or ten times of that of a main part of a product by forging a magnesium alloy plate and a plurality of rough forging steps and finishing forging steps.

[0006] However, to manufacture parts having complex and accurate figure from magnesium alloy, the method as disclosed in Japanese Unexamined Patent Publication No. 7-224344 is insufficient. Because forming is conducted by forging billet, there are limitations on figure and thickness. On the other hand, the methods disclosed in Japanese Unexamined Patent Publication Nos. 2001-294966, 2001-170734, and 2001-170736 are also insufficient. Because the forming is made from a magnesium alloy plate, it is possible to make thin-walled parts but it is difficult to manufacture a product having complex and accurate figure by pressing and forging the plate.

[0007] In recent years, elucidation of mechanism of superplasticity has been developed with reference to magnesium alloys, as well as aluminum alloys, to the extent that there is now important possibility that magnesium alloys having miniaturized crystal grain size can be processed at high strain rate (see, for example, "Handbook of Magnesium Technology" P119-P125).

[0008] To mold an alloy into a complex and accurate figure, it is generally preferable to employ casting method such as die casting with high injection speed, i.e. high filing speed. Since magnesium alloy is easily solidified as mentioned above, however, flow lines may be easily created when molded by die casting. In addition, in some figures, it is difficult to fill the magnesium alloy in every corner. Accordingly, there are limitations on size and thickness. As the injection speed is increased, air or gas may be easily entrapped in liquid alloy, thereby creating porosities and thus reducing the reliability of properties.

[0009] On the other hand, in case of pressing a plate, it is possible to manufacture products having a size equal to or less than the width of the plate. Since magnesium alloy has low ductility and poor formability, magnesium alloy is hardly formed into a complex figure, for example, it is difficult to form a boss as formed by the casting.

[0010] In viewpoint of alloy composition, the casting property and the elongation property of magnesium alloys are in contracting relationship. Preferably employed as the material to be cast are AZ91, AM50, AM60, and the like containing a larger amount of aluminum so as to have low melting temperature. On the other hand, preferably employed

as the material to be pressed and forged are AZ31 containing a smaller amount of aluminum so as to have high ductility. The larger the aluminum content is, the higher the corrosion resistance is. Accordingly, AZ31 is poor in corrosion resistance as compared to AZ91. The poor corrosion resistance is one of reasons for limited application of AZ31.

[Problems to be resolved by the Invention]

[0011] The present invention was made under the aforementioned conventional circumstances and the object of the present invention is to provide a method of manufacturing magnesium alloy products comprising a combination of casting and forging for forming a magnesium alloy of which composition allows casting and which is excellent in forgeability, thereby achieving the manufacture of products, which have complex and accurate figure and exhibit high reliability of properties and enough corrosion resistance, at sufficiently high yield ratio.

[Means to Solve the Problems]

[0012] According to the present invention, this object is achieved by a method of manufacturing magnesium alloy products as defined in claim 1 or claim 7. The dependent claims define preferred and advantageous embodiments of the invention. A method of manufacturing magnesium alloy products according to a first aspect of the invention, comprises steps of: casting a magnesium alloy containing 2-10 mass % aluminum to obtain a cast semifinished product having crystal grain size not greater than 30 μm , subjecting the cast semifinished product to solution treatment at a temperature between the solid solution temperature and the solidus curve of the composition of the alloy, after that, forging the semifinished product to have a forged semifinished product having crystal grain size not greater than 10 μm , and further forging the forged semifinished product to have a desired figure.

[0013] As the cast semifinished product which is made to have crystal grain size not greater than 30 μm by casting is subjected to solution treatment, the crystal grains are enlarged, but second-phase grains which are formed during the casting and are large and fragile are disappeared, thereby increasing the elongation and thus improving the plastic formability. The cast semifinished product having the improved plastic formability attained by the solution treatment is forged. The dynamic recrystallization according to this forging miniaturizes the crystal grain size to be 10 μm or less, thereby further improving the forgeability. According to the first aspect of the invention, the cast semifinished product which is made to have crystal grain size not greater than 30 μm by casting is subjected to solution treatment, after that, is forged to have crystal grain size not greater than 10 μm , and is further forged into a desired figure.

[0014] In this method, the suitable aluminum content of the magnesium alloy is in a range from 2.5 to 6 mass % and the casting is preferably conducted in the die casting method or the thixo molding method. In addition, the solution treatment is preferably conducted at a temperature between 380 and 440 $^{\circ}\text{C}$ for 1 to 24 hours and the forging for miniaturizing crystal grains after solution treatment and the shaping forging after that are preferably conducted under conditions of a strain rate and temperature which are set to have a Z value in a range from 10^9 to 10^{13} .

[0015] A method of manufacturing magnesium alloy products according to the first aspect of the invention comprises steps of: casting a magnesium alloy containing 2-10 mass % aluminum to obtain a cast semifinished product having crystal grain size not greater than 10 μm , subjecting the cast semifinished product to solution treatment at a temperature between the solid solution temperature and the solidus curve of the composition of the alloy, and after that, forging the semifinished product to have a desired figure.

[0016] As the cast semifinished product which is made to have crystal grain size not greater than 10 μm by casting is subjected to solution treatment, the crystal grains are enlarged, but second-phase grains which are formed during the casting and are large and fragile are disappeared, thereby increasing the elongation and thus improving the plastic formability. By forging the cast semifinished product having the improved plastic formability attained by the solution treatment, the product can be formed into a desired figure. Accordingly, in a method considering a second aspect of the invention, the cast semifinished product which is made to have crystal grain size not greater than 10 μm by casting is subjected to solution treatment, and after that, is further forged into the desired figure.

[0017] In this method, the suitable aluminum content of the magnesium alloy is in a range from 2 to 6 mass % and the casting is preferably conducted in the die casting method. In addition, the solution treatment is preferably conducted at a temperature between 380 and 440 $^{\circ}\text{C}$ for 1 to 24 hours and the forging is preferably conducted under conditions of a strain rate and temperature which are set to have a Z value lower than 10^{13} .

[0018] It should be noted that the Z value is a temperature-compensating strain rate indicating the relation between temperature and strain rate and is so-called Zener-Hollomon Parameter defined by the following equation (I) as the relational expression for the dependence of temperature and strain rate on the flow stress:

$$Z = \dot{\epsilon}' \exp (Q/RT) \quad (I)$$

Wherein ϵ' : strain rate (sec^{-1})

Q : lattice diffusion activation energy

R : gas constant

T : absolute temperature

5 Generally employed as the value of Q is a value of 135kJoule/mol of pure magnesium because no Q value of magnesium alloys is obtained.

[Brief Explanation of the drawings]

10 **[0019]** Fig. 1 is a graph showing crystal grain sizes of thixo molding cast articles (after solution treatment) in Example 1.

[0020] Figs. 2(a), 2(b) are graphs showing results of tensile strength tests of solution-treated articles at 300 °C and $\epsilon' = 1.0 \times 10^{-2} \text{ s}^{-1}$ in Example 1.

15 **[0021]** Fig. 3 is a graph showing crystal grain sizes of die cast articles (before and after solution treatment) in Example 2.

[Embodiments for carrying out the Invention]

20 **[0022]** Hereinafter, embodiments of a method of manufacturing magnesium alloy products according to the present invention will be described.

[0023] First, an embodiment of the method of manufacturing magnesium alloy products of claim 1 will be described.

[0024] In the method of claim 1, first, a magnesium alloy containing 2-10 mass % aluminum is cast to obtain a cast semifinished product having crystal grain size not greater than 30 μm .

25 **[0025]** If the aluminum content of the magnesium alloy is less than 2 mass %, the corrosion resistance should be poor and the melting temperature is high so that it is not suitable for casting. If the aluminum content of the magnesium alloy is more than 10 mass %, it is impossible to obtain enough increase of plastic formability obtained by solution treatment as the following step and it is impossible to obtain products after solution treatment having excellent forge-

30 **[0026]** There is no particular limitation on the method of casting the magnesium alloy. For obtaining a cast product having crystal grain size not greater than 30 μm , preferably employed are die casting method or thixo molding method because their cooling/solidification speed is relatively high and the crystal grains can be miniaturized.

35 **[0027]** In gravity casting method, the obtained cast product has generally large thickness so that the solidification of melt magnesium alloy is slow. Therefore, crystals should grow during the cooling/solidification so as to have large crystal grain size to the extent of 200 μm . On the other hand, in the die casting method and the thixo molding method in which the alloy is injected in a die in the melt state or the semi-melt state, the cooling/solidification speed is fast so that crystal grains are miniaturized to have crystal grain size not greater than 30 μm .

[0028] Smaller crystal grain size of the cast product is better. The crystal grain size not greater than 30 μm is allowed. Generally, the casting is conducted to have crystal grain size from 15 to 30 μm depending on the employed casting method and the composition of used alloy.

40 **[0029]** The thus obtained cast product having crystal grain size not greater than 30 μm is then subjected to solution treatment.

[0030] The temperature of the solution treatment may be in a range between the solid solution temperature and the solidus curve of the composition of the used alloy and the suitable temperature is from 380 to 430 °C. When the temperature of the solution treatment is lower than the solid solution temperature or lower than 380 °C, huge compounds of aluminum and magnesium may be deposited, impairing the plastic formability. When the temperature of the solution treatment exceeds the solidus curve or 430 °C, liquid phase may be generated, thus impairing the plastic formability. The time period of the solution treatment is suitably from 1 to 24 hours. It is preferable to increase the time period when the temperature is low and to decrease the time period when the temperature is high. By the solution treatment, β -phase deposited in crystal grain boundary of α -phase as the parent phase are dissolved in the parent phase, thereby enlarging crystal grains in the parent phase. However, because of decrease in β -phase grains impairing the grain boundary sliding in the plastic forming, effect improving the formability is obtained.

50 **[0031]** After the plastic treatment, the semifinished product is forged to obtain a forged semifinished product having crystal grain size not greater than 10 μm (hereinafter, the forging for miniaturizing crystal grains will be sometimes referred to as "grain-miniaturizing forging"). The forged semifinished product is further forged for shaping the semifinished product into a desired figure, thereby obtaining a product (hereinafter, the forging for shaping a semifinished product into a desired figure will be sometimes referred to as "shaping forging").

[0032] The grain-miniaturizing forging is conducted for miniaturizing crystal grains of cast semifinished product by dynamic recrystallization. The grain-miniaturizing forging and the shaping forging should be conducted under conditions

allowing forging process depending on the composition of magnesium alloy.

[0033] The conditions of the grain-miniaturizing forging depend on the composition of magnesium alloy. However, the conditions of strain rate and temperature for the grain-miniaturizing forging are preferably set to have a Z value in a range from 10^9 to 10^{13} , more preferably, in a range from 10^{10} to 10^{13} .

[0034] The conditions of the shaping forging also depend on the composition of magnesium alloy. However, the conditions of strain rate and temperature for the shaping forging are preferably set to have a Z value of 10^{13} or less, preferably in a range from 10^8 to 10^{13} , more preferably, in a range from 10^9 to 10^{12} .

[0035] In either of the grain-miniaturizing forging and the shaping forging, the forging conditions outside of the range of Z value may create defects such as clacks and chaps, not allowing the forging.

[0036] Normally, the condition for conducting the grain-miniaturizing forging is determined according to the composition of the alloy to have a Z value in the suitable range within a range from 10^{-3} to 10^{-1} sec^{-1} of the strain rate and a range from 200 to 500 °C of the temperature. On the other hand, the condition for conducting the shaping forging is determined according to the composition of the alloy to have a Z value in the suitable range within a range from 10^{-3} to 10^{-2} sec^{-1} of the strain rate and a range from 200 to 400 °C of the temperature.

[0037] The crystal grains are miniaturized to have crystal grain size not greater than 10 μm by grain-miniaturizing forging, thereby improving the plastic formability as an effect of the forging, thus allowing the product to be subjected to the shaping forging. The crystal grain size not greater than 10 μm is allowed. Generally, the range of crystal grain sizes to be obtained by the grain-miniaturizing forging is from 1 to 10 μm .

[0038] Hereinafter, an embodiment of the method of manufacturing magnesium alloy products of claim 7 will be described.

[0039] In the method of claim 7, first, a magnesium alloy containing 2-10 mass % aluminum is cast to obtain a cast semifinished product having crystal grain size not greater than 10 μm .

[0040] If the aluminum content of the magnesium alloy is less than 2 mass %, the corrosion resistance should be poor. If the aluminum content of the magnesium alloy is more than 10 mass %, it is impossible to obtain enough increase of plastic formability to be attained by solution treatment as the following step and it is impossible to obtain products after solution treatment having excellent forgeability. Therefore, the aluminum content of magnesium alloy is from 2 to 10 mass %, preferably, from 2 to 6 mass %.

[0041] The contents of materials other than aluminum in the magnesium alloy to be used are the same as those of the aforementioned method of claim 1.

[0042] Preferably employed are die casting method because its cooling/solidification speed is relatively high and the crystal grains can be significantly miniaturized.

[0043] The crystal grain size of the cast semifinished product is preferably smaller and may be 10 μm or less. Generally, the range of crystal grain size of the semifinished product obtained by the casting is from 5 to 10 μm .

[0044] The thus obtained cast semifinished product having crystal grain size not greater than 10 μm is then subjected to solution treatment at a temperature between the solid solution temperature and the solidus curve of the composition of the used alloy. Because of the same reasons of the solution treatment as the method of claim 1, the suitable temperature is from 380 to 430 °C and the suitable time period of the solution treatment is from 1 to 24 hours. After the solution treatment, the cast semifinished product is forged to obtain a product of a desired figure.

[0045] The forging should be conducted under conditions allowing forging process depending on the composition of magnesium alloy similarly to the forging processes of the method of claim 1.

[0046] The conditions of the forging depend on the composition of magnesium alloy. However, the conditions of strain rate and temperature for the forging are preferably set to have a Z value less than 10^{13} , more preferably in a range from 10^6 to 10^{12} . The Z value of 10^{13} or more may create defects such as clacks and chaps, not allowing the forging.

[0047] In this case, the conditions for conducting the forging is determined according to the composition of the alloy to have a Z value in the suitable range within a range from 10^{-3} to 10^{-1} sec^{-1} of strain rate and a range from 200 to 500 °C of temperature.

[Examples]

[0048] The present invention will be further specifically described with reference to the following examples.

[0049] By using commercially available AZ91 alloy ingots, Mg alloy ingots used for the following examples were prepared by adding magnesium and zinc to the AZ91 alloy ingots and governing the qualities of the ingots. In this manner, Mg alloy ingots having compositions from AZ81 to AZ 21 were prepared. Table 1 shows results of componential analysis of the used AZ 91 alloy ingot and prepared ingots.

[Table 1]

RESULTS OF COMPONENTIAL ANALYSIS OF INGOTS (mass %)								
Ingot	Al	Zn	Mn	Si	Cu	Fe	Ni	Be
AZ91	8.9	0.70	0.21	0.310	0.0400	0.0020	0.0004	0.0008
AZ81	7.6	0.70	0.30	0.025	0.0010	0.0017	tr	0.0034
AZ71	6.9	0.72	0.24	0.024	0.0011	0.0003	tr	0.0019
AZ61	5.7	0.79	0.30	0.024	0.0010	0.0029	tr	0.0026
AZ51	4.8	0.78	0.29	0.018	0.0009	0.0013	tr	0.0022
AZ41	3.6	0.68	0.27	0.013	0.0008	0.0012	tr	0.0014
AZ31	2.6	0.60	0.28	0.010	0.0004	tr	tr	0.0016
AZ21	2.1	0.83	0.28	0.003	0.0052	tr	tr	0.0030

Example 1

(1) Casting and Solution Treatment

[0050] The ingots from AZ91 to AZ21 were ground to make chips for thixo molding. The chips were used in casting process. By using a thixo molding machine JMG-450 manufactured by the Japan Steel Works, Ltd., the injection speed was set at 4 m/sec that is the maximum under the idling condition and the die temperature was set at 250 °C. Under the conditions, cast articles of a box shape of 181 mm length × 255 mm width × 10 mm height having a bottom and no lid and 1.5 mm thickness were obtained. The casting process was conducted with finding a mold-allowing condition by controlling the temperature of a barrel and a nozzle of the molding machine because each ingot has each melting point. Table 2 shows the temperatures of the barrel tips during casting process of the respective alloys.

[Table 2]

TEMPERATURES OF BARREL TIPS DURING THIXO MOLDING CASTING	
ALLOY	TEMPERATURE (°C)
AZ91	620
AZ81	618
AZ71	619
AZ61	624
AZ51	627
AZ41	640
AZ31	638

[0051] As a result, AZ91 through AZ31 were allowed to be cast. However, since the melting point of AZ21 was 645 °C that was over the heating limit of the barrel of the molding machine, AZ21 was not allowed to be cast. Therefore, the limit of AZ alloys allowing the casting by the thixo molding machine is 2.5% aluminum content.

[0052] To measure the crystal grain sizes of the cast articles obtained by the thixo molding casting, specimens were taken from middle portions of the respective articles and were embedded in resin and polished. After that, the specimens were etched by picric or acetic etching agent of which selection depends on the composition of the specimens. Electron micrographs of 500X of the respective specimens were taken. The crystal grain sizes were measured according to the section method of JIS G0522 "Ferritic Grain Size Test of Steel" and were expanded 1.74 times.

[0053] To check the effects attained by the solution treatment, the cast articles were subjected to heat treatment at 430 °C for 1 hour and, after that, the crystal grain size were measured again in the same manner.

[0054] The results are shown in Table 3 and Fig. 1.

[Table 3]

CRYSTAL GRAIN SIZES OF THIXO MOLDING CAST ARTICLES		
ALLOY	CRYSTAL GRAIN SIZE (μm)	
	BEFORE SOLUTION TREATMENT	AFTER SOLUTION TREATMENT
AZ91	13.1	28.3
AZ81	12.3	19.1
AZ71	10.2	16.4
AZ61	13.1	24.6
AZ51	10.1	13.7
AZ41	12.4	20.2
AZ31	10.5	17.9

[0055] As apparent from Table 3 and Fig. 1, while differences in crystal grain size according to the compositions were small before the solution treatment, the crystal grain size was enlarged by the solution treatment. This is attributed to the fact that the solution treatment makes β -phase existing at the grain boundaries to be resolved in α -phase as parent phase so as to enlarge the crystal grains. It is believed that the crystal grain size is smaller as the speed of solidifying liquid alloy by quenching is higher. Therefore, the following results were obtained. That is, in the order from AZ91 to AZ31, the aluminum content decreases so that the melting point increases. Accordingly, the barrel temperature of the barrel tip of molding machine is raised. However, the quenching effect is created by temperature difference between the liquid alloy and the die. Therefore, AZ91 has crystal grain size of 28 μm , i.e. relatively large grain size, because of small temperature difference, while AZ51 has crystal grain size of 14 μm , i.e. relatively small grain size, because of large temperature difference. However, contrariwise, AZ41, ZA31 have grain size from 18 to 20 μm because cooling delay effect is attained in high-temperature liquid alloy.

[0056] To measure the plastic formability of the solution-treated articles, specimens were taken from the respective articles and were subjected to solution treatment at 420 °C for 1 hour. After that, the tensile tests were made at 300 °C at strain rate $1.0 \times 10^{-2} \text{ sec}^{-1}$. The results are shown in Figs. 2(a), 2(b).

[0057] As apparent from Figs. 2(a), 2(b), AZ91 through AZ71 as aluminum-rich alloys have lower elongation from 15 to 24%, while AZ61 through AZ31 have elongation of 40% or more, significantly improving the plastic formability.

[0058] Therefore, the aluminum content of cast article to be forged is equal to or more than 25 mass % in view of castability and equal to or less than 6 mass % in view of plastic formability.

(2) Forging

[0059] In the above (1), the cast articles of AZ61 through AZ31 cast by thixo molding were subjected to solution treatment at 420 °C for 1 hour. After that, samples of 20 mm $\times$ 20 mm were taken out. Each specimen was heated uniformly by an electric furnace and put on a mold which was held at a predetermined forging temperature shown in Table 4. The forging was conducted under a fixed condition of strain rate of $3.3 \times 10^{-2} \text{ sec}^{-1}$. Specimens were cut and taken from the samples after the forging. As for the specimens, the measurement of crystal grain size was carried out in the same manner as the above (1). Z values obtained by substituting the aforementioned strain rates in the aforementioned equation (I) are shown in Table 1. The value for Q in this equation was 135Kjoule/mol. Table 4 also shows crystal grain sizes of the respective samples before forging process (after solution treatment).

[Table 4]

MINIATURIZING OF CRYSTAL GRAINS BY FORGING

		CRYSTAL GRAIN SIZE (μm) AFTER FORGING					CRYSTAL GRAIN SIZE (μm) AFTER SOLUTION TREATMENT (BEFORE FORGING)
FORGING CONDITION	FORGING TEMPERATURE (°C)	150	200	250	300	350	
	STRAIN RATE (sec ⁻¹)	3.3×10 ⁻²	3.3×10 ⁻²	3.3×10 ⁻²	3.3×10 ⁻²	3.3×10 ⁻²	
	Z VALUE	1.5×10 ¹⁵	2.7×10 ¹³	1.0×10 ¹²	6.7×10 ¹⁰	6.9×10 ⁹	
AZ61		□	□	□	6.1	12.9	24.6
AZ51		□	□	3.2	7.3	10.0	13.7
AZ41		□	□	4.0	10.4	18.8	20.2
AZ31		□	1.0	4.6	14.2	15.1	17.9

□ Sample cracked during forging, not allowing the forging.

[0060] The following are apparent from Table 4.

[0061] That is, it is found that, at the same temperature, larger amount of aluminum content of the alloy facilitates the miniaturization of crystals by forging. On the other hand, as for the aluminum-rich alloys, cracks were created during forging when the temperature was low. In the strain rate tests, the forging of AZ61 was allowed at 300 °C or more, while the forging of AZ31 was allowed even at 200 °C, obtaining effect of crystal grain miniaturization.

[0062] As a result, in case of AZ61 through AZ31, the forging conditions for miniaturizing crystal grain size to be 10 μm or less allowing superplasticity forging are Z value ranging from 10⁹ to 10¹³, preferably from 10¹⁰ to 10¹³.

[0063] Samples in which crystal grain miniaturization by the aforementioned forging was sufficient and samples in which crystal grain miniaturization was insufficient were selected. Plate-like specimens of 20 mm × 20 mm × 1.5 mm thickness were cut and taken from these selected samples. Each specimen was inserted into a cavity of 20 mm × 20 mm as a drag of a casting mold. Each specimen was forged under conditions shown in Table 5 until the true strain reaches -1.1 by using a cope having a cylindrical concave of 3 mm diameter and 10 mm height and was formed into a boss shape. The respective forgeabilities during the forging process were evaluated. The evaluation results are shown in Table 5.

[Table 5]

FORGEABILITIES OF THIXO MOLDING CAST ARTICLES

		FORGEABILITY					
FORGING CONDITION	FORGING TEMPERATURE (°C)	150	200	300	350	400	500
	STRAIN RATE (sec ⁻¹)	3.3×10^{-2}	3.3×10^{-2}	3.3×10^{-2}	3.3×10^{-2}	3.3×10^{-2}	3.3×10^{-2}
	Z VALUE	1.5×10^{15}	2.7×10^{13}	6.7×10^{10}	6.9×10^9	9.9×10^8	4.4×10^7
AZ61	6.1 *1	×	□	□	○	○	○
	12.9 *1	×	×	×	×	×	×
AZ51	3.2 *1	×	□	○	○	○	○
	7.3 *1	×	□	○	○	○	○
	10 *1	×	□	□	□	○	○
AZ41	4.0 *1	×	○	○	○	○	○
	18.8 *1	×	×	×	×	×	□
AZ31	1.0 *1	×	○	○	○	○	○
	4.6 *1	×	○	○	○	○	○
	14.2 *1	×	×	×	×	□	○

*1 Crystal grain size after forging for miniaturizing crystal grains (value shown in Table 4)

*2 Evaluation of forgeability

○: No defects were created after forging, i.e. complete formation of boss

□: Fine cracks were created after forging, i.e. incomplete formation of boss

×: Serious cracks were created after forging

[0064] The following are apparent from Table 5.

[0065] Alloys, containing larger amount of aluminum in which β -phase is easily deposited at grain boundaries so as to easily impair the grain boundary sliding, should require higher processing temperature, that is, higher Z value to form boss. On the other hand, even when the crystal grain size exceeds 10 μm , some alloys are allowed to form boss

by setting the temperature high.

[0066] However, from the industrial point of view, the mold temperature of 400 °C or more impairs the durability of mold so that it is not practical. It is possible to improve the heat resistivity of the mold by applying heat resistance material or treating the surface. However, since the cost of the mold is increased, it is not preferable.

[0067] It is found from the results that, in case of AZ61 through AZ31, the forging condition for forming alloy into a desired figure is a Z value of 10^{13} or less, preferably in a range from 10^8 to 10^{13} .

Example 2

(1) Casting and Solution Treatment

[0068] Casting tests were conducting in the die casting method instead of thixo molding of Example 1. Die having the same shape as that for the thixo molding was used. Used alloys were ingots of the same batch as those used for the thixo molding machine, but not processed to be chips. By using a cold chamber die casting machine DC650tCLS available from Toshiba Machine Co., Ltd., articles were sequentially formed by casting under conditions that the temperature of liquid alloy was 700 °C, the injection speed was set at 5.0 m/sec at the highest, and the die temperature was set at 250 °C. The cast articles had the same size and shape as those of

Example 1.

[0069] Even AZ21, impossible to be cast by the thixo molding method, could be cast by the die casting method. This is attributed to the fact that the material was melted by a metal heater provided separately from the casting machine, unlike the thixo molding machine in which material is melted in the barrel provided therein, so that the melting temperature could be raised to 700 °C, thereby melting even AZ21 of which melting temperature was high.

[0070] As for the respective articles, the crystal grain sizes after solution treatment were measured in the same manner as that of Example 1 and the results are shown in Table 6 and Fig. 3. It should be noted that the solution treatment was conducted at 430 °C for 1 hour.

[Table 6]

CRYSTAL GRAIN SIZES OF DIE CAST ARTICLES		
ALLOY	CRYSTAL GRAIN SIZE (μm)	
	BEFORE SOLUTION TREATMENT	AFTER SOLUTION TREATMENT
AZ91	7.3	14.9
AZ81	6.4	13.1
AZ71	7.0	13.8
AZ61	7.8	15.2
AZ51	6.9	10.4
AZ41	6.1	11.3
AZ31	5.7	9.5
AZ21	5.8	9.7

[0071] As apparent from Table 6 and Fig. 3, the crystal grain sizes of the die cast articles were smaller than the crystal grain sizes of the thixo molding cast articles. Even before the solution treatment, the crystal grain sizes were below 10 μm so that the grain-miniaturizing forging is not required. This is attributed to the fact that the cooling effect could be attained because the molding machine was so fast in filling speed.

(2) Forging

[0072] Since the obtained cast articles already had fine crystal grains, casting was conducted under the same conditions as those for grain-miniaturizing forging which had been conducted for the thixo molding articles in Example 1. For giving indication of forgeability of the respective cast articles, it was checked whether cracks were formed or not. As for the samples before the solution treatment, preliminary forging tests were conducted with the result that cracks were easily formed. This may be because the β-phase was thick so that the grain boundary sliding was hardly occurred.

This tendency was enhanced as the aluminum content increased. Therefore, only for the samples after solution treatment, tests were conducted. The results are shown in Table 7. The tests were conducted with plate-like specimens of 20 mm × 20 mm × 1.5 mm thickness which were cut and taken from these samples. Each specimen was forged at a constant strain rate. The true strain for the forging was -1.1.

[Table 7]

FORGEABILITIES OF DIE CAST ARTICLES

		FORGEABILITY *2				
FORGING CONDITION	FORGING TEMPERATURE (°C)	150	200	250	300	350
	STRAIN RATE (sec ⁻¹)	3.3×10^{-2}	3.3×10^{-2}	3.3×10^{-2}	3.3×10^{-2}	3.3×10^{-2}
	Z VALUE	1.5×10^{15}	2.7×10^{13}	1.0×10^{12}	6.9×10^{10}	6.9×10^9
AZ91		×	×	×	×	×
AZ81		×	×	×	□	□
AZ71		×	×	□	□	□
AZ61		×	□	□	○	○
AZ51		×	□	○	○	○
AZ41		×	□	○	○	○
AZ31		×	○	○	○	○
AZ21		×	○	○	○	○

*2 Evaluation of forgeability

○: No defects were created after forging

□: Fine cracks were created after forging

×: Serious cracks were created after forging

[0073] The following are apparent from Table 7.

[0074] That is, similar to the samples before the solution treatment, the higher the aluminum content was, the worse the forgeability was. Under condition of strain rate of 3.3×10^{-2} , AZ91 through AZ71 created serious cracks during forging even when the processing temperature was raised to 350 °C. On the other hand, the lower the aluminum content was, the better the forgeability was. Accordingly, AZ91 created serious cracks at any temperature, AZ81 created no serious cracks but fine cracks when forged at a temperature of 300 °C or more (that is, when Z value was below

6.7×10^{10}), and AZ71 created no serious cracks but created fine cracks when forged at a temperature of 250 °C or more (that is, when Z value was below 1.0×10^{12}).

[0075] With reduced aluminum content, casting was conducted without cracks so that AZ61, AZ51, and AZ41 created no defects when forged at a temperature of 250 °C or more (that is, when Z value was below 1.0×10^{12}), and AZ31 and AZ21 created no defects when forged at a temperature of 200 °C or more (that is, when Z value was below 1.0×10^{13}). In these ranges, excellent forgeabilities were exhibited.

[0076] It is found from the results that composition suitable for forging alloy cast to have crystal grain size not greater than 10 μm is aluminum content ranging from 2 to 6 mass % and the forging condition is a Z value of 1.0×10^{13} or less.

[Effects of the Invention]

[0077] As described in the above, according to the method of manufacturing magnesium alloy products of the present invention, a combination of casting and forging is employed for forming magnesium alloy of which composition allows casting and which is excellent in forgeability, thereby achieving the manufacture of products, which have complex and accurate figure and exhibit high reliability of properties and enough corrosion resistance, at sufficiently high yield ratio.

Claims

1. A method of manufacturing magnesium alloy products comprising steps of:

casting a magnesium alloy containing 2-10 mass % aluminum to obtain a cast semifinished product having crystal grain size not greater than 30 μm ,
subjecting the cast semifinished product to solution treatment at a temperature between the solid solution temperature and the solidus curve of the composition of the magnesium alloy,
after that, forging the semifinished product to have a forged semifinished product having crystal grain size not greater than 10 μm , and
further forging the forged semifinished product to have a desired figure.

2. A method of manufacturing magnesium alloy products as claimed in claim 1, wherein the aluminum content of the magnesium alloy is in a range from 2.5 to 6 mass %.

3. A method of manufacturing magnesium alloy products as claimed in claim 1 or 2, wherein the casting is conducted in a die casting method or a thixo molding method.

4. A method of manufacturing magnesium alloy products as claimed in any one of claims 1 through 3, wherein the solution treatment is conducted at a temperature between 380 and 440 °C for 1 to 24 hours.

5. A method of manufacturing magnesium alloy products as claimed in any one of claims 1 through 4, wherein the forging is conducted under conditions of a strain rate and temperature which are set to have a Z value in a range from 10^9 to 10^{13} , to obtain a crystal grain-miniaturized forged semifinished product having crystal grain size not greater than 10 μm .

6. A method of manufacturing magnesium alloy products as claimed in any one of claims 1 through 5, wherein the crystal grain-miniaturized forged semifinished product is forged under conditions of a strain rate and temperature which are set to have a Z value of 10^{13} or less, to have a desired figure.

7. A method of manufacturing magnesium alloy products comprising steps of:

casting a magnesium alloy containing 2-10 mass % aluminum to obtain a cast semifinished product having crystal grain size not greater than 10 μm ,
subjecting the cast semifinished product to solution treatment at a temperature between the solid solution temperature and the solidus curve of the composition of the magnesium alloy, and
after that, forging the semifinished product to have a desired figure.

8. A method of manufacturing magnesium alloy products as claimed in claim 7, wherein the aluminum content of the magnesium alloy is in a range from 2 to 6 mass %.

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9. A method of manufacturing magnesium alloy products as claimed in claim 7 or 8, wherein the casting is conducted in a die casting method.
- 5 10. A method of manufacturing magnesium alloy products as claimed in any one of claims 7 through 9, wherein the solution treatment is conducted at a temperature between 380 and 440 °C for 1 to 24 hours.
- 10 11. A method of manufacturing magnesium alloy products as claimed in any one of claims 7 through 10, wherein the forging is conducted under conditions of a strain rate and temperature which are set to have a Z value lower than 10^{13} .

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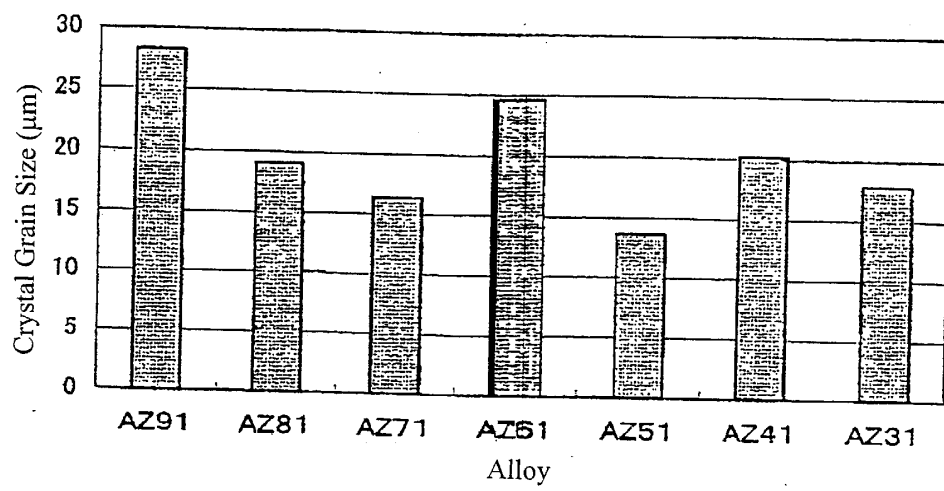


Fig. 1

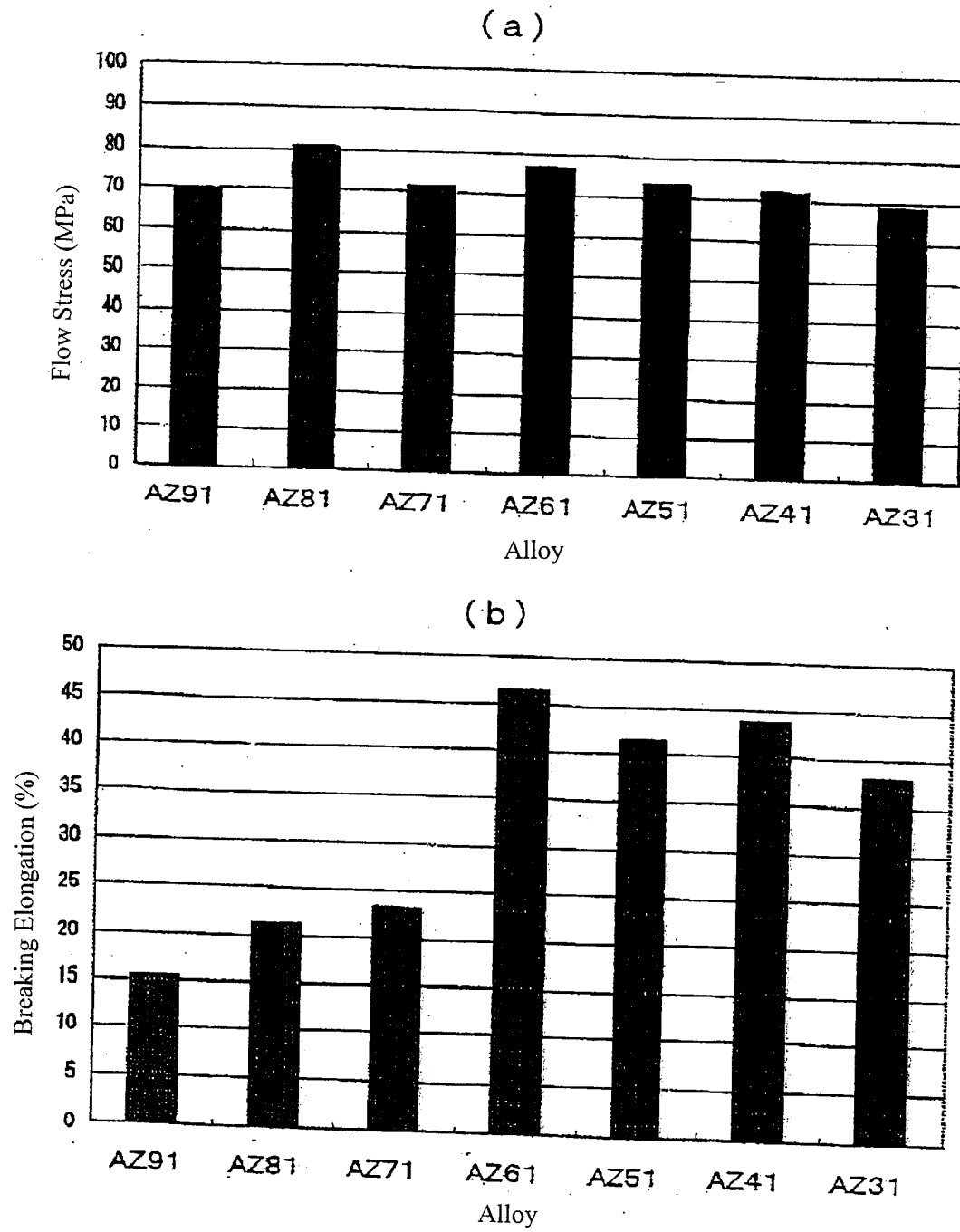
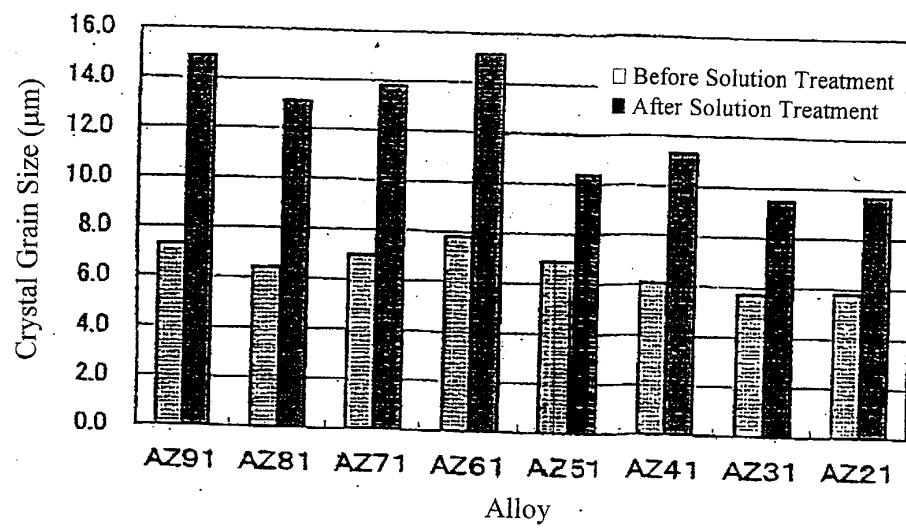


Fig. 2

**Fig. 3**



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EUROPEAN SEARCH REPORT

Application Number
EP 03 00 2095

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Place of search MUNICH		Date of completion of the search 14 April 2003	Examiner Catana, C
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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Place of search MUNICH		Date of completion of the search 14 April 2003	Examiner Catana, C
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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