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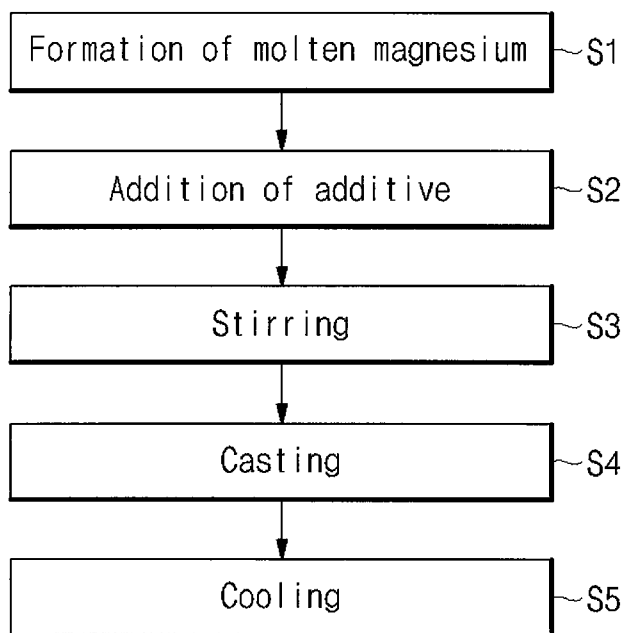
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(54) Title: MAGNESIUM ALLOY AND MANUFACTURING METHOD THEREOF

Fig. 1



(57) Abstract: A magnesium alloy with improved resistance to oxidation and ignition is provided. The magnesium alloy comprises magnesium grains and an additive compound present at grain boundaries defined by the magnesium grains but absent in the magnesium grains. Further provided is a method for producing the magnesium alloy. The method comprises melting magnesium in a crucible at 600 to 800°C under a protective gas atmosphere to form molten magnesium, adding an additive to the molten magnesium, stirring the molten mixture for 1 to 400 minutes, casting the molten mixture in a mold at 100 to 300°C, and cooling the cast product. According to the method, the amount of the protective gas used can be reduced.



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MAGNESIUM ALLOY AND MANUFACTURING METHOD THEREOF

【Technical Field】

5 The present invention relates to a magnesium alloy and a method for producing the same.

【Background Art】

10 In general, magnesium is the lightest metal in practical use at present. Magnesium and its alloys (hereafter collectively referred to as 'magnesium alloys') are of growing interest as lightweight structural materials due to their high specific strength and specific 'stiffness. In addition, magnesium alloys are expected to be useful as replacements for resinous materials. Magnesium alloys are currently produced by various
15 processes, such as die casting and thixocasting, and are widely used as casing materials for electronic devices and electric home appliances where high strength is not critical. Other practical applications of magnesium alloys are automobile parts, for example, handles and cylinder head covers, for the purpose of weight reduction.

20 Magnesium alloy solutions (*i.e.* molten magnesium alloys) prepared by heating are liable to ignite. Various kinds of protective gases are used to prevent molten magnesium alloys from being ignited. Representative protective gases include SF₆, SO₂, CO₂, HFC-134a, Novec™612, inert gases, and mixtures thereof.

However, most of the protective gases are harmful to humans and cause metallic equipment to corrode. Particularly, many protective gases are categorized as greenhouse gases, and thus regulations regarding their use are becoming more stringent. For example, SF₆ has a global warming
5 potential 23,900 times that of CO₂. In advanced countries, regulatory guidelines restrict the use of SF₆ and much research is actively underway to replace SF₆.

On the other hand, the addition of calcium (Ca) to magnesium alloys is known to effectively inhibit ignition and oxidation of the
10 magnesium alloys at high temperatures, but it causes the disadvantages of poor flowability, hot cracking, mold sticking, etc. The market price of calcium per kg amounts to about 200 dollars, which is responsible for the high production cost of magnesium alloys. Further, calcium varies the original alloy specification of magnesium alloys to reduce the availability of
15 the magnesium alloys. Moreover, calcium is completely removed during recycling of magnesium alloys, thus considerably deteriorating the recyclability of the magnesium alloys. That is, the recycling of magnesium alloys needs continuous addition of calcium, which becomes a cause of increased production cost of the magnesium alloys.

20

【Disclosure】**【Technical Problem】**

The present invention has been made in an effort to solve the above-mentioned problems of the prior art, and it is an object of the

present invention to provide a magnesium alloy produced using a protective gas in a reduced amount or without the use of any protective gas, and a method for producing the magnesium alloy.

A further object of the present invention is to provide a magnesium
5 alloy produced using an additive capable of improving the cleanness of molten magnesium in a melting furnace or during transfer or feeding, and a method for producing the magnesium alloy.

Another object of the present invention is to provide a magnesium alloy that does not encounter the problems of poor flowability, hot cracking,
10 mold sticking, etc. during production to achieve improved casting, forming, welding and powder metallurgy (PM) performance characteristics, and a method for producing the magnesium alloy.

Another object of the present invention is to provide a magnesium alloy produced using a cheap additive and a protective gas in a reduced
15 amount or without the use of any protective gas, thus lowering the production cost, and a method for producing the magnesium alloy.

Another object of the present invention is to provide a magnesium alloy that uses an additive causing no change in original alloy specification to execute the inherent application of the alloy, and a method for producing
20 the magnesium alloy.

Another object of the present invention is to provide a magnesium alloy whose mechanical properties are improved by grain refinement and internal soundness, and a method for producing the magnesium alloy.

Another object of the present invention is to provide a magnesium

alloy with improved welding and joint performance characteristics, and a method for producing the magnesium alloy.

Another object of the present invention is to provide a magnesium alloy with improved resistance to oxidation and ignition that can be safely
5 used in a variety of applications, and a method for producing the magnesium alloy.

Still another object of the present invention is to provide a magnesium alloy with improved recyclability depending on the type of a flux and temperature control during recycling, and a method for producing
10 the magnesium alloy.

【Technical Solution】

In accordance with an aspect of the present invention, the above and other objects can be accomplished by the provision of a magnesium
15 alloy comprising magnesium grains and an additive compound present at grain boundaries defined by the magnesium grains but absent in the magnesium grains.

In an embodiment, the additive compound may be selected from: a compound of magnesium and an alkali metal; a compound of magnesium
20 and an alkaline earth metal; a compound of a constituent material of the magnesium alloy other than magnesium and an alkali metal; and a compound of a constituent material of the magnesium alloy other than magnesium and an alkaline earth metal.

In an embodiment, the magnesium grains may be pure magnesium

grains or magnesium alloy grains.

In an embodiment, the additive compound may be present in an amount of 0.0001 to 30 parts by weight, based on 100 parts by weight of the magnesium alloy.

5 In an embodiment, the additive compound may have a size of 0.1 to 500 μm .

In an embodiment, the magnesium alloy may have an ignition temperature of 500 to 1,500°C.

In an embodiment, the magnesium alloy may be selected from the
10 group consisting of a casting alloy, a wrought alloy, a creep alloy, a damping alloy, a biodegradable alloy, a powder metallurgy alloy, and mixtures thereof.

In accordance with another aspect of the present invention, there is provided a method for producing a magnesium alloy, the method
15 comprising melting magnesium in a crucible at 600 to 800°C under a protective gas atmosphere to form molten magnesium, adding an additive to the molten magnesium, stirring the molten mixture for 1 to 400 minutes, casting the molten mixture in a mold at 100 to 300°C, and cooling the cast product.

20 In an embodiment, the magnesium may be pure magnesium or a magnesium alloy.

In an embodiment, the additive may be selected from an alkali metal, an alkali metal oxide, an alkali metal compound, an alkaline earth metal, an alkaline earth metal oxide, an alkaline earth metal compound,

and mixtures thereof.

In an embodiment, the alkali metal oxide may be selected from sodium oxide, potassium oxide, and a mixture thereof.

In an embodiment, the alkaline earth metal oxide may be selected
5 from beryllium oxide, magnesium oxide, calcium oxide, strontium oxide, and mixtures thereof.

In an embodiment, the alkaline earth metal compound may be selected from calcium carbide (CaC_2), calcium cyanamide (CaCN_2), calcium carbonate (CaCO_3), calcium sulfate hemihydrate (CaSO_4), and
10 mixtures thereof.

In an embodiment, the additive may be added in an amount of 0.0001 to 30 parts by weight, based on 100 parts by weight of the magnesium alloy.

In an embodiment, the additive may have a size of 0.1 to 500 μm .

15 In an embodiment, the additive may be added to increase the ignition temperature and hardness of the magnesium alloy and to reduce the required amount of the protective gas.

【Advantageous Effects】

20 The present invention has the following advantageous effects.

The use of the additive increases the ignition temperature of the magnesium alloy and inhibits the oxidation of the magnesium alloy, so that the protective gas categorized as a greenhouse gas can be used in a reduced amount or the need for the use of the protective gas can be

eliminated.

Further, the use of the additive can improve the cleanness of the molten magnesium in a melting furnace or during transfer or feeding.

Further, problems such as poor flowability, hot cracking and mold
5 sticking are not encountered in the production of the magnesium alloy so that the casting, forming, welding and powder metallurgy (PM) performance characteristics of the magnesium alloy can be improved.

Further, the use of the cheap additive and the use of the protective gas in a reduced amount or no use of the protective gas can contribute to a
10 reduction in the production cost of the magnesium alloy.

Further, the additive causes no change in the original alloy specification of the magnesium alloy so that the inherent application of the magnesium alloy can be executed.

Further, the use of the additive leads to grain refinement and
15 internal soundness of the magnesium alloy, resulting in an improvement in the mechanical properties of the magnesium alloy.

Further, the use of the additive inhibits the oxidation of the magnesium alloy and increases the ignition temperature of the magnesium alloy, so that the magnesium alloy can be safely used in a variety of
20 applications.

Further, the additive in the form of a compound present in the grain boundaries but absent in the magnesium grains does not respond to a flux supplied and temperature changes during recycling of the magnesium alloy, thus ensuring good recyclability of the magnesium alloy. That is,

recycling of the magnesium alloy requires no additional processing for the addition of the additive.

【Description of Drawings】

5 In the figures:

FIG. 1 is a flow chart illustrating a method for producing a magnesium alloy according to the present invention;

FIG. 2 is an image showing the structure of a magnesium alloy produced by a method of the present invention;

10 FIGS. 3 through 8 are images showing the results of electron probe micro analysis (EPMA) for a magnesium alloy produced by a method of the present invention;

FIGS. 9 through 12 are images showing the results of transmission electron microscopy (TEM) for a magnesium alloy produced by a method
15 of the present invention;

FIGS. 13 and 14 are graphs showing the results of protective gas reduction experiments of magnesium alloys produced by a method of the present invention;

FIG. 15 is a graph showing the results of thermogravimetric
20 analysis (TGA) for a magnesium alloy produced by a method of the present invention;

FIGS. 16 and 17 are graphs showing the results of Auger emission spectroscopy (AES) for an AZ91D magnesium alloy and a magnesium alloy produced by a method of the present invention;

FIG. 18 is a graph showing the hardness values of magnesium alloys produced by a method of the present invention at room temperature and high temperatures;

FIG. 19 is a graph showing the ignition temperatures of magnesium alloys produced by a method of the present invention under an ambient atmosphere and under a nitrogen atmosphere;

FIG. 20 is a graph showing the hardness of magnesium alloys produced by a method of the present invention at room temperature;

FIG. 21 is a graph showing the ignition temperatures of magnesium alloys produced by a method of the present invention under an ambient atmosphere and under a nitrogen atmosphere;

FIG. 22 shows surface images of billets of magnesium alloys produced by a method of the present invention;

FIG. 23 is a graph showing the ignition temperatures of magnesium alloys produced by a method of the present invention;

FIG. 24 is a graph showing the hardness of magnesium alloys produced by a method of the present invention;

FIG. 25 shows images of the structures of an AZ91D magnesium alloy and a magnesium alloy produced by a method of the present invention;

FIG. 26 is a table showing the hardness data of magnesium alloys produced by a method of the present invention;

FIG. 27 is a graph showing the ignition temperature of a magnesium alloy produced by a method of the present invention;

FIG. 28 shows images of the structures of an AZ91D magnesium alloy and a magnesium alloy produced by a method of the present invention;

FIG. 29 is a table showing the hardness data of magnesium alloys
5 produced by a method of the present invention;

FIG. 30 is a graph showing the ignition temperature of a magnesium alloy produced by a method of the present invention;

FIG. 31 is a graph showing the ignition temperatures of magnesium alloys produced by a method of the present invention;

10 FIG. 32 is a graph showing the hardness of magnesium alloys produced by a method of the present invention;

FIGS. 33 and 34 are graphs showing the results of ignition experiments of magnesium alloys produced by a method of the present invention;

15 FIGS. 35 and 36 are graphs showing the results of protective gas reduction experiments of magnesium alloys produced by a method of the present invention;

FIGS. 37 and 38 are graphs showing the results of ignition experiments of magnesium alloys produced by a method of the present
20 invention; and

FIGS. 39 and 40 are surface images of magnesium alloys produced in the absence (FIG. 39) and presence (FIG. 40) of a protective gas, and FIG. 41 is a surface image of a magnesium alloy produced by a method of the present invention.

【Best Mode】

Preferred embodiments of the present invention will now be described in detail with reference to the accompanying drawings, such that those skilled in the art can easily practice the present invention.

5 FIG. 1 is a flow chart illustrating a method for producing a magnesium alloy according to the present invention.

As illustrated in FIG. 1, the method of the present invention comprises the following steps: formation of molten magnesium (S1), addition of an additive (S2), stirring (S3), casting (S4) and cooling (S5).

10 In step S1, magnesium is put into a crucible and heated to 600 to 800°C under a protective gas atmosphere. As a result, the magnesium is melted to form molten magnesium.

A temperature below 600°C is insufficient to form molten magnesium. Meanwhile, a temperature above 800°C can increase the risk of ignition of the molten magnesium. The protective gas serves to inhibit the ignition of the magnesium. The protective gas is selected from SF₆, SO₂, CO₂, HFC-134a, Novec™612, inert gases, and equivalents thereof. These protective gases may be used alone or as a mixture of two or more thereof.

20 In step S1, the magnesium may be selected from pure magnesium, magnesium alloys, and equivalents thereof. Examples of the magnesium alloys include, but are not limited to, AZ91D, AM20, AM30, AM50, AM60, AZ31, AS41, AS31, AS21X, AE42, AE44, AX51, AX52, AJ50X, AJ52X, AJ62X, MRI153, MRI230, AM-HP2, Mg-Al, Mg-Al-Re, Mg-Al-Sn, Mg-Zn-

Sn, Mg-Si, Mg-Zn-Y, and equivalents thereof.

In step S2, the additive added to the molten magnesium is in the form of a powder.

As the additive, there can be used an alkali metal, an alkali metal
5 oxide, an alkali metal compound, an alkaline earth metal, an alkaline earth metal oxide, an alkaline earth metal compound, or an equivalent thereof. A mixture of these additives may also be used.

The alkali metal oxide may be selected from sodium oxide, potassium oxide and equivalents thereof. These alkali metal oxides may be
10 used alone or as a mixture of two or more thereof. The alkaline earth metal oxide may be selected from beryllium oxide, magnesium oxide, calcium oxide, strontium oxide, and mixtures thereof. The alkaline earth metal compound may be selected from calcium carbide (CaC_2), calcium cyanamide (CaCN_2), calcium carbonate (CaCO_3), calcium sulfate
15 hemihydrate (CaSO_4), and mixtures thereof. However, there is no restriction on the kinds of the alkali metal oxide, alkaline earth metal oxide and the alkaline earth metal compound. That is, any material may be used as the additive so long as it can increase the ignition temperature of the magnesium alloy, reduce the oxidation of the magnesium alloy or decrease
20 the required amount of the protective gas.

In step S2, the additive may be added in an amount of 0.0001 to 30 parts by weight, based on 100 parts by weight of the magnesium alloy. If the amount of the additive added is less than 0.0001 parts by weight, the intended effects (*i.e.* increased ignition temperature, reduced oxidation and

decreased amount of the protective gas) of the additive are negligible. Meanwhile, if the amount of the additive added is more than 30 parts by weight, the inherent characteristics of the magnesium or magnesium alloy are not exhibited. The additive may have a size of 1 to 500 μm . It is
5 practically difficult and economically undesirable to prepare the additive in a size smaller than 1 μm . Meanwhile, the additive larger than 500 μm may not react with the molten magnesium.

In step S3, the molten mixture is stirred for 1 to 400 minutes. A stirring time of less than 1 minute does not ensure sufficient mixing of the
10 additive with the molten magnesium, while a stirring time of more than 400 is unnecessary because further improvement of stirring effects is not attained.

In step S3, the additive is present in the form of an intermetallic compound outside the magnesium or magnesium alloy grains, *i.e.* at grain
15 boundaries defined by the magnesium grains or magnesium alloy grains, rather than inside the magnesium or magnesium alloy grains. That is, the additive is in the form of an additive compound. Specifically, the additive is in the form of: a compound of magnesium and an alkali metal; a compound of magnesium and an alkaline earth metal; a compound of a constituent
20 material of the magnesium alloy other than magnesium and an alkali metal; or a compound of a constituent material of the magnesium alloy other than magnesium and an alkaline earth metal. The other constituent elements (*e.g.*, oxygen, carbon, nitrogen, etc.) of the additive float on the surface of the molten magnesium and are removed by manual or automatic means.

In step S4, the molten mixture is cast in a mold at 100 to 300°C.

The mold may be selected from metal molds, ceramic molds, graphite molds and equivalents thereof. The casting may be performed by gravity casting, continuous casting and equivalent processes thereof.

5 However, there is no restriction on the materials of the mold and the casting processes.

In step S5, the cast product is cooled to room temperature, and then the final magnesium alloy (e.g., magnesium alloy ingot) is taken out of the mold.

10 The magnesium alloy produced by the method of the present invention is composed of magnesium grains and an additive compound present at grain boundaries defined by the magnesium grains but absent in the magnesium grains, as will be explained later.

The additive added during production of the magnesium alloy is distinguished from the additive compound present in the final magnesium alloy. The additive compound present in the magnesium alloy is in the form of an intermetallic compound.

FIG. 2 is an image showing the structure of a magnesium alloy 1 produced by the method of the present invention.

20 As shown in FIG. 2, the magnesium alloy 1 comprises magnesium grains 2 and an additive compound 3.

The additive compound 3 is present at grain boundaries defined by the magnesium grains 2 but is absent in the magnesium grains 2. The additive compound 3 is in the form of an intermetallic compound.

The magnesium grains 2 may be selected from pure magnesium grains, magnesium alloy grains, and equivalents thereof.

The additive compound 3 may be selected from: a compound of magnesium and an alkali metal; a compound of magnesium and an alkaline earth metal; a compound of a constituent material of the magnesium alloy other than magnesium and an alkali metal; and a compound of a constituent material of the magnesium alloy other than magnesium and an alkaline earth metal.

The additive compound 3 may be present in an amount of 0.0001 to 30 parts by weight, based on 100 parts by weight of the magnesium alloy. The additive compound may have a size of 0.1 to 500 μm . The critical meanings of the numerical limitations in the content and size of the additive compound 3 have been already explained above.

The magnesium alloy 1 of the present invention may have a hardness (HRF) of 40 to 80. However, the hardness of the magnesium alloy 1 may vary depending on various conditions such as processing and annealing. The magnesium alloy 1 has an ignition temperature of 500 to 1,500°C, indicating improved ignition resistance. Further, the amount of a protective gas necessary for the production of the magnesium alloy 1 is considerably reduced, as will be explained below.

On the other hand, the magnesium alloy may be selected from the group consisting of a casting alloy, a wrought alloy, a creep alloy, a damping alloy, a biodegradable alloy, a powder metallurgy alloy, and mixtures thereof.

There is no restriction on the kind of the casting alloy. For example, the casting alloy may be produced from a mixture of AZ91D, AM20, AM50 or AM60 and CaX (e.g., CaO, CaC₂ or CaCN₂).

The wrought alloy may be produced from a mixture of AZ31 or
5 AM30 and CaX.

The creep alloy may be produced from a mixture of Mg-Al or Mg-Al-Re and CaX or SrO or a mixture of Mg-Al-Sn or Mg-Zn-Sn and CaX.

The damping alloy may be produced from a mixture of pure Mg, Mg-Si or SiCp/Mg and CaO.

10 The biodegradable alloy may be produced from a mixture of pure Mg and CaO.

The powder metallurgy alloy may be produced from a mixture of Mg-Zn-(Y) and CaX.

Hereinafter, the present invention will be explained with reference
15 to experimental results in the following examples.

EXAMPLES

(Example 1)

Three AZ91D magnesium alloys (each 3 kg) were heated to 680°C
20 to form molten magnesium alloys. Calcium oxide (CaO) powders having a size of < 100 μm, 100-200 μm and 200-500 μm were added to the respective molten magnesium alloys. Each of the calcium oxide powders was used in an amount of one part by weight (30 g). Thereafter, each of the molten mixtures was stirred for 10 minutes, poured into a metal mold,

followed by gravity casting. The cast product was cooled. The elemental analysis of the cast product was conducted by an inductively coupled plasma (ICP) method. The powder sizes, target compositions, elemental analysis results by ICP and yields are shown in Table 1.

5 TABLE 1

Powder size (μm)	< 100	100~200	200~500
Target composition (Part by weight of CaO)	1	1	1
ICP elemental analysis (Parts by weight of CaO)	0.45	0.0078	0.0042
Yield (%)	45	0.78	0.42

As can be seen from the results in Table 1, the yield of the calcium oxide having a size smaller than 100 μm was 45%. That is, 0.45 parts by weight of the calcium oxide was melted in the corresponding molten
10 magnesium. In contrast, the yields of the calcium oxide having a size of 100-200 μm and the calcium oxide having a size of 200-500 μm were drastically lowered to 0.78% and 0.42%, respectively.

FIGS. 3 through 8 are images showing the results of electron probe micro analysis (EPMA) for the magnesium alloy produced by the method of
15 the present invention.

FIG. 3 is a back scattering electron image of the magnesium alloy. As shown in FIG. 3, the magnesium alloy was substantially composed of grains and grain boundaries defined by the grains. FIG. 4 is an image for magnesium, which is indicated by the red areas, and clearly shows that the
20 magnesium was composed of a number of grains and grain boundaries defined by the grains. In FIG. 4, the blue areas indicate the absence of

magnesium. FIG. 5 shows the presence of a small amount of aluminum (red areas) in the grain boundaries. FIG. 6 shows the presence of zinc (red areas) in a much smaller amount than aluminum. FIG. 7 shows the presence of a small amount of calcium (red areas) in the grain boundaries.

5 FIG. 8 shows the substantial absence of oxygen.

These results lead to the conclusion that magnesium made up the largest portion of the magnesium alloy and that aluminum, zinc and calcium were present in the grain boundaries. The minor metal elements were substantially in the form of intermetallic compounds.

10 That is, the calcium oxide added to the AZ91D magnesium alloy was reduced and was present in the form of an aluminum-calcium compound (Al_2Ca) in the grain boundaries. Although calcium oxide is more thermodynamically stable than magnesium, the mechanism of the reduction of calcium oxide has not been clearly established.

15 FIGS. 9 through 12 are images showing the results of transmission electron microscopy (TEM) for a magnesium alloy produced by the method of the present invention.

FIG. 9 is a magnified TEM image. The central black area indicates Al_2Ca as an aluminum-calcium compound. FIG. 10 is an image of
20 magnesium present in areas other than the area of the aluminum-calcium compound. FIGS. 11 and 12 are images of aluminum and calcium, respectively. These results demonstrate that aluminum and calcium were present in the form of an intermetallic compound in the magnesium grain boundaries.

FIGS. 13 and 14 are graphs showing the results of protective gas reduction experiments of magnesium alloys produced by the method of the present invention.

The graphs of FIGS. 13 and 14 were obtained from experiments at
5 680°C as a molten metal temperature for die casting and 720°C indicating an overheated state, respectively.

In FIGS. 13 and 14, "sealed" refers to a state in which ambient air was not introduced, and "unsealed" refers to a state in which ambient air was introduced.

10 As shown in FIG. 13, 1,000 ppm SF_6 was necessary to inhibit ignition in the production of AZ91D, AZ91D-0.04wt%CaO and AZ91D-0.13wt%CaO in the unsealed state at 680°C. In contrast, 500 ppm SF_6 was necessary to inhibit ignition in the production of AZ91D and AZ91D-0.04wt%CaO, and about 300 ppm SF_6 was necessary to inhibit ignition in
15 the production of AZ91D-0.13wt%CaO in the sealed state. Consequently, the amount of the protective gas necessary to inhibit ignition was decreased with increasing content of the additive at the operation temperature for die casting in the sealed state.

Next, as shown in FIG. 14, 3,200 ppm, 2,000 ppm and 1,000 ppm
20 SF_6 were necessary to inhibit ignition in the production of AZ91D, AZ91D-0.04wt%CaO and AZ91D-0.13wt%CaO, respectively, in the unsealed state at 720°C. In contrast, about 500 ppm SF_6 was necessary to inhibit ignition in the production of AZ91D and AZ91D-0.04wt%CaO in the sealed state, and 300 ppm SF_6 was necessary to inhibit ignition in the production of

AZ91D-0.13wt%CaO in the sealed state. These results show that the amount of the protective gas necessary to inhibit ignition was decreased with increasing content of the additive in the sealed state even at the overheating temperature.

5 FIG. 15 is a graph showing the results of thermogravimetric analysis (TGA) for a magnesium alloy produced by the method of the present invention.

 In FIG. 15, the x axis shows time and the y axis shows an increment in weight (%). The weight of AZ91D sharply increased with the
10 passage of time, indicating rapid oxidation of the magnesium alloy. In contrast, the weight of AZ91D-0.14wt%CaO gradually increased with the passage of time, indicating slow oxidation of the magnesium alloy. For example, after 400 minutes, the weight of AZ91D and AZ91D-0.14wt%CaO increased to 113% and 101%, respectively.

15 These results demonstrate that the addition of the additive inhibited the oxidation of the magnesium alloy.

 FIGS. 16 and 17 are graphs showing the results of Auger emission spectroscopy (AES) for an AZ91D magnesium alloy and a magnesium alloy produced by the method of the present invention.

20 In FIG. 16, the x axis shows sputtering time and the y axis shows the atomic contents ration of oxygen detected. About 40 atomic contents ration of oxygen was detected in AZ91D until about 9 minutes after sputtering, and thereafter, the amount of oxygen detected decreased, indicating that a relatively thick oxide film was formed in AZ91D.

As shown in FIG. 17, about 26 atomic contents ratio of oxygen was detected in AZ91D-0.7wt%CaO until about one minute after sputtering, and thereafter, the amount of oxygen detected decreased, indicating that a relatively thin oxide film was formed in AZ91D-
5 0.7wt%CaO.

These results demonstrate that the addition of the additive inhibited the oxidation of the magnesium alloy.

FIG. 18 is a graph showing the hardness values of AZ91D magnesium alloys produced by the method of the present invention at
10 room temperature and high temperatures.

As shown in FIG. 18, the hardness of the AZ91D magnesium alloys containing 0.007-0.41% by weight of calcium oxide increased with increasing calcium oxide content. Specifically, the hardness of the AZ91D magnesium alloy containing no calcium oxide (CaO) was about 57, and the
15 hardness of the AZ91D magnesium alloys containing small amounts of calcium oxide increased to 68 at room temperature (25°C). The hardness values of the AZ91D magnesium alloys at 100°C and 150°C were slightly lower than those of the alloys at room temperature because of poor thermal stability of β phase ($\text{Mg}_{17}\text{Al}_{12}$) at the high temperatures.

20 FIG. 19 is a graph showing the ignition temperature of magnesium alloys produced by the method of the present invention.

As shown in FIG. 19, the ignition temperature of the magnesium alloys increased with increasing content of calcium oxide. Specifically, the ignition temperature of the AZ91D magnesium alloy under an ambient

atmosphere was about 490°C and that of the AZ91D magnesium alloy was about 510°C under a nitrogen atmosphere, whereas the ignition temperatures of the magnesium alloys containing calcium oxide were increased by at least 100°C in proportion to the content of the additive.

5

(Example 2)

FIG. 20 is a graph showing the hardness of AZ91D magnesium alloys produced by the method of the present invention at room temperature.

10 As shown in FIG. 20, the hardness values of the AZ91D magnesium alloys containing different strontium oxide amounts (0.02-0.48 wt%) were increased with increasing strontium oxide content. Specifically, the AZ91D magnesium alloy containing no strontium oxide had a hardness of about 57 and the AZ91D magnesium alloy containing a small amount of
15 strontium oxide had a hardness of about 65 at room temperature.

FIG. 21 is a graph showing the ignition temperatures of AZ91D magnesium alloys produced by the method of the present invention under an ambient atmosphere and under a nitrogen atmosphere.

As shown in FIG. 21, the ignition temperatures of the AZ91D
20 magnesium alloy containing no strontium oxide were about 490°C and 510°C under an ambient atmosphere and under a nitrogen atmosphere, respectively, while the ignition temperatures of the AZ91D magnesium alloys containing different amounts of strontium oxide were increased by at least 100°C.

(Example 3)

FIG. 22 shows surface images of billets of AZ91D magnesium alloys for die casting produced by the method of the present invention.

5 As shown in FIG. 22, the surface of the billet of the AZ91D magnesium alloy containing 0.0001 wt% of beryllium oxide was clean without being oxidized or ignited despite the presence of a very small amount of the alkaline earth metal oxide. Further, the surface of the billet of the AZ91D magnesium alloy containing 0.5 wt% of magnesium oxide was
10 clean without being oxidized or ignited despite the presence of a small amount of the alkaline earth metal oxide.

FIG. 23 is a graph showing the ignition temperatures of AZ91D magnesium alloys for die casting produced by the method of the present invention.

15 The ignition temperatures of the AZ91D magnesium alloy containing 0.0001 wt% of beryllium oxide and the AZ91D magnesium alloy containing 0.5 wt% of magnesium oxide were increased due to the presence of the alkaline earth metal oxides, leading to an improvement in mechanical properties and oxidation resistance.

20 FIG. 24 is a graph showing the hardness of AZ91D magnesium alloys for die casting produced by the method of the present invention.

As shown in FIG. 24, the hardness values of the AZ91D magnesium alloy containing 0.0001 wt% of beryllium oxide and the AZ91D magnesium alloy containing 0.5 wt% of magnesium oxide were increased

due to the presence of the alkaline earth metal oxides.

(Example 4)

FIG. 25 shows images of the structures of an AZ91D magnesium
5 alloy containing no additive and an AZ91D magnesium alloy produced by
the method of the present invention.

As shown in FIG. 25, there was no significant difference in grain
size between the AZ91D magnesium alloy containing no additive and the
AZ91D magnesium alloy containing 0.007 wt% of calcium cyanamide
10 (CaCN_2) as a calcium-based additive. It was confirmed that the presence
of the small amount of the additive did not affect the characteristics of the
AZ91D magnesium alloy.

FIG. 26 is a table showing the hardness data of AZ91D magnesium
alloys produced by the method of the present invention.

15 As shown in FIG. 26, the AZ91D magnesium alloys containing
calcium cyanamide as a calcium compound were measured to have an
average hardness of about 66, which is about 4 higher than the hardness
(about 62) of an AZ91D magnesium alloy containing no additive.

FIG. 27 is a graph showing the ignition temperature of an AZ91D
20 magnesium alloy containing no additive and an AZ91D magnesium alloy
produced by the method of the present invention.

As shown in FIG. 27, the ignition temperature of the AZ91D
magnesium alloy containing calcium cyanamide as a calcium compound
reached about 540°C under a nitrogen atmosphere, which is about 30°C

higher than the ignition temperature (510°C) of the AZ91D magnesium alloy containing no additive.

(Example 5)

5 FIG. 28 shows images of the structures of an AZ91D magnesium alloy containing no additive and an AZ91D magnesium alloy produced by the method of the present invention.

As shown in FIG. 28, there was no significant difference in grain size between the AZ91D magnesium alloy containing no additive and the
10 AZ91D magnesium alloy containing 0.03 wt% of calcium carbide (CaC_2) as a calcium-based additive.

FIG. 29 is a table showing the hardness data of AZ91D magnesium alloys produced by the method of the present invention.

As shown in FIG. 29, the AZ91D magnesium alloys containing
15 calcium carbide as a calcium compound were measured to have an average hardness of about 65.3, which is about 3 higher than the hardness (about 62) of an AZ91D magnesium alloy containing no additive.

FIG. 30 is a graph showing the ignition temperatures of an AZ91D magnesium alloy containing no additive and an AZ91D magnesium alloy
20 produced by the method of the present invention.

As shown in FIG. 30, the ignition temperature of the AZ91D magnesium alloy containing calcium carbide as a calcium compound reached about 540°C under a nitrogen atmosphere, which is about 30°C higher than the ignition temperature (510°C) of the AZ91D magnesium

alloy containing no additive.

FIG. 31 is a graph showing the ignition temperatures of magnesium alloys produced by the method of the present invention.

As shown in FIG. 31, the ignition temperatures of the magnesium alloy AZ91D-0.007wt%CaCN₂ containing calcium cyanamide (CaCN₂) and the magnesium alloy AZ91D-0.03wt%CaC₂ containing calcium cyanamide (CaCN₂) were much higher than the ignition temperature of a pure magnesium alloy AZ91D, indicating that the magnesium alloys produced by the method of the present invention had better heat resistance. These results lead to the conclusion that the ignition temperatures of the magnesium alloys were dramatically increased due to the presence of the calcium compound.

(Example 6)

FIG. 32 is a graph showing the hardness of AM50 magnesium alloys produced by the method of the present invention.

As shown in FIG. 32, the AM50 magnesium alloy containing no calcium oxide, the AM50 magnesium alloy containing about 0.05% by weight of calcium oxide and the AM50 magnesium alloy containing about 0.15% by weight of calcium oxide were found to have hardness values of about 50, indicating that little influence was given to the microstructure and hardness of the magnesium alloys due to the addition of a small amount of calcium oxide.

FIGS. 33 and 34 are graphs showing the results of ignition

experiments of AM50 magnesium alloys produced by the method of the present invention.

As shown in FIG. 33, the AM50 magnesium alloy containing no calcium oxide had an ignition temperature of about 570°C, the AM50
5 magnesium alloy containing 0.05% by weight of calcium oxide had an ignition temperature of about 590°C, and the AM50 magnesium alloy containing 0.15% by weight of calcium oxide had an ignition temperature of about 610°C under an ambient atmosphere.

As shown in FIG. 34, the AM50 magnesium alloy containing no
10 calcium oxide had an ignition temperature of about 550°C, the AM50 magnesium alloy containing 0.05% by weight of calcium oxide had an ignition temperature of about 570°C, and the AM50 magnesium alloy containing 0.15% by weight of calcium oxide had an ignition temperature of about 640°C under a nitrogen atmosphere. From these results, it can be
15 seen that the presence of the additive increased the ignition temperature of the magnesium alloys.

(Example 7)

FIGS. 35 and 36 are graphs showing the results of protective gas
reduction experiments of magnesium alloys produced by the method of the
20 present invention.

The graphs of FIGS. 35 and 36 were obtained from experiments at 680°C as a molten metal temperature for die casting and 720°C indicating an overheated state, respectively.

As shown in FIG. 35, 500 ppm SF₆ was necessary to inhibit ignition

in the production of AZ31, AZ31-0.05wt%CaO and AZ31-0.32wt%CaO in the unsealed state at 680°C. In contrast, about 100 ppm SF₆ was necessary to inhibit ignition in the production of AZ31 and AZ31-0.05wt%CaO, and about 40 ppm SF₆ was necessary to inhibit ignition in the production of AZ31-0.32wt%CaO in the sealed state. Consequently, the amount of the protective gas necessary to inhibit ignition was decreased with increasing content of the additive at the operation temperature for die casting in the sealed state.

Next, as shown in FIG. 36, 1,000 ppm SF₆ was necessary to inhibit ignition in the production of AZ31, AZ31-0.05wt%CaO and AZ31-0.32wt%CaO in the unsealed state at 720°C. In contrast, about 200 ppm, 100 ppm and 40 ppm SF₆ were necessary to inhibit ignition in the production of AZ31, AZ31-0.05wt%CaO and AZ31-0.32wt%CaO, respectively, in the sealed state. These results show that the amount of the protective gas necessary to inhibit ignition was decreased with increasing content of the additive in the sealed state even at the overheating temperature.

FIGS. 37 and 38 are graphs showing the results of ignition experiments of AZ31 magnesium alloy containing no calcium oxide and AZ31 magnesium alloys produced by the method of the present invention.

As shown in FIG. 37, the AZ31 magnesium alloy containing no calcium oxide had an ignition temperature of about 570°C under an ambient atmosphere, while the AZ31 magnesium alloy containing 0.3% by weight of calcium oxide had an ignition temperature of about 610°C under

an ambient atmosphere. That is, the presence of the additive increased the ignition temperature of the AZ31 magnesium alloy.

As shown in FIG. 38, the AZ31 magnesium alloy containing no calcium oxide had an ignition temperature of about 640°C under a nitrogen atmosphere, while the AZ31 magnesium alloy containing 0.3% by weight of calcium oxide had an ignition temperature of about 690°C under a nitrogen atmosphere. That is, the presence of the additive increased the ignition temperature of the AZ31 magnesium alloy.

FIGS. 39 and 40 are surface images of AZ31 magnesium alloys produced in the absence of (FIG. 39) and presence (FIG. 40) of a protective gas, and FIG. 41 is a surface image of a cast sheet of a magnesium alloy produced by the method of the present invention.

As shown in FIG. 39, the AZ31 magnesium alloy produced without the use of any protective gas was ignited and its surface turned black.

As shown in FIG. 40, a metal oxide was incorporated into the AZ31 magnesium alloy produced by spraying a protective gas, leaving cracks on the surface of the magnesium alloy.

In contrast, no ignition or cracking occurred in the AZ31 magnesium alloy produced using calcium oxide, as shown in FIG. 41.

Although the foregoing embodiments have been described to practice the magnesium alloy and the production method thereof according to the present invention, these embodiments are set forth for illustrative purposes and do not serve to limit the invention. Those skilled in the art will readily appreciate that many modifications and variations can be made,

without departing from the spirit and scope of the invention as defined in the appended claims, and such modifications and variations are encompassed within the scope and spirit of the present invention.

【CLAIMS】**【Claim 1】**

A magnesium alloy comprising
magnesium grains, and
5 an additive compound present at grain boundaries defined by the
magnesium grains but absent in the magnesium grains.

【Claim 2】

The magnesium alloy according to claim 1, wherein the additive
10 compound is selected from: a compound of magnesium and an alkali
metal; a compound of magnesium and an alkaline earth metal; a
compound of a constituent material of the magnesium alloy other than
magnesium and an alkali metal; and a compound of a constituent material
of the magnesium alloy other than magnesium and an alkaline earth metal.

15

【Claim 3】

The magnesium alloy according to claim 1, wherein the magnesium
grains are pure magnesium grains or magnesium alloy grains.

20 **【Claim 4】**

The magnesium alloy according to claim 1, wherein the additive
compound is present in an amount of 0.0001 to 30 parts by weight, based
on 100 parts by weight of the magnesium alloy.

【Claim 5】

The magnesium alloy according to claim 1, wherein the additive compound has a size of 0.1 to 500 μm .

5 **【Claim 6】**

The magnesium alloy according to claim 1, wherein the magnesium alloy has an ignition temperature of 500 to 1,500°C.

【Claim 7】

10 The magnesium alloy according to claim 1, wherein the magnesium alloy is selected from the group consisting of a casting alloy, a wrought alloy, a creep alloy, a damping alloy, a biodegradable alloy, a powder metallurgy alloy, and mixtures thereof.

15 **【Claim 8】**

A method for producing a magnesium alloy, the method comprising
melting magnesium in a crucible at 600 to 800°C under a protective
gas atmosphere to form molten magnesium,
adding an additive to the molten magnesium,
20 stirring the molten mixture for 1 to 400 minutes,
casting the molten mixture in a mold at 100 to 300°C, and
cooling the cast product.

【Claim 9】

The method according to claim 8, wherein the magnesium is pure magnesium or a magnesium alloy.

【Claim 10】

5 The method according to claim 8, wherein the additive is selected from an alkali metal, an alkali metal oxide, an alkali metal compound, an alkaline earth metal, an alkaline earth metal oxide, an alkaline earth metal compound, and mixtures thereof.

10 **【Claim 11】**

 The method according to claim 8, wherein the alkali metal oxide is selected from sodium oxide, potassium oxide, and a mixture thereof.

【Claim 12】

15 The method according to claim 8, wherein the alkaline earth metal oxide is selected from beryllium oxide, magnesium oxide, calcium oxide, strontium oxide, and mixtures thereof.

【Claim 13】

20 The method according to claim 10, wherein the alkaline earth metal compound is selected from calcium carbide (CaC_2), calcium cyanamide (CaCN_2), calcium carbonate (CaCO_3), calcium sulfate hemihydrate (CaSO_4), and mixtures thereof.

【Claim 14】

The method according to claim 8, wherein the additive is added in an amount of 0.0001 to 30 parts by weight, based on 100 parts by weight of the magnesium alloy.

5

【Claim 15】

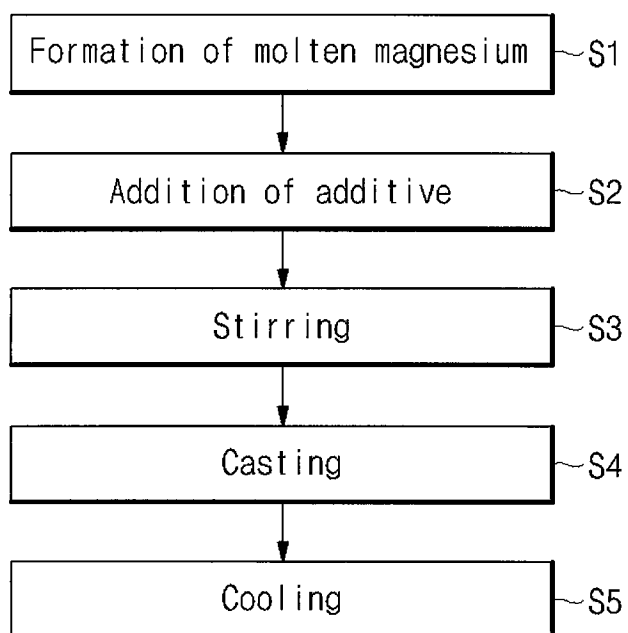
The method according to claim 8, wherein the additive has a size of 0.1 to 500 μm .

10 **【Claim 16】**

The method according to claim 8, wherein the additive is added to increase the ignition temperature and hardness of the magnesium alloy and to reduce the required amount of the protective gas.

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Fig. 1



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Fig. 2

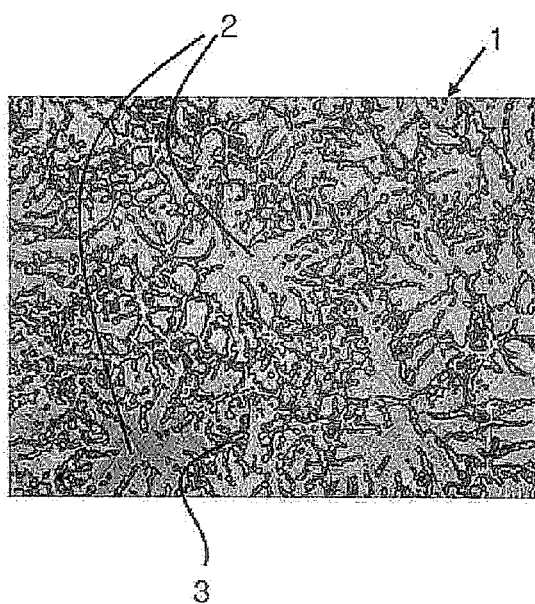
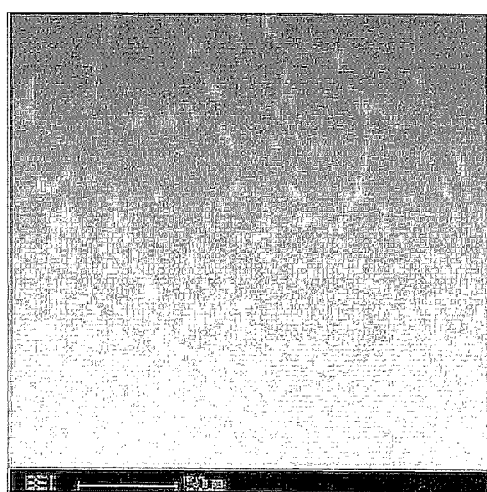


Fig. 3



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Fig. 4

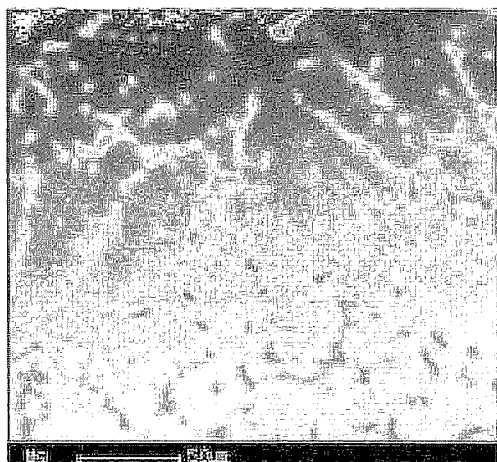
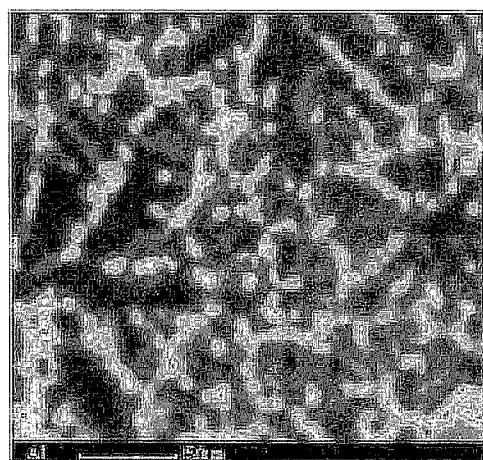


Fig. 5



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Fig. 6

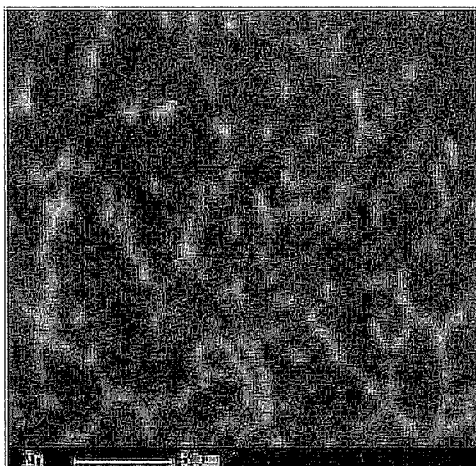
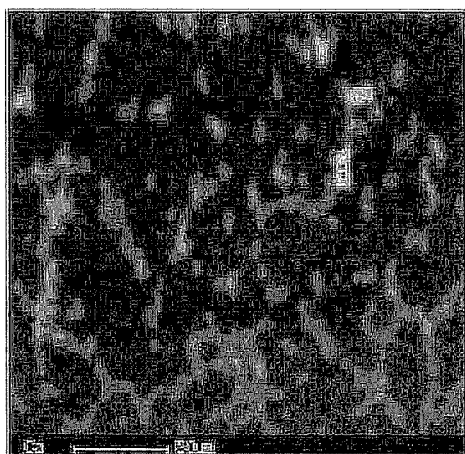


Fig. 7



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Fig. 8

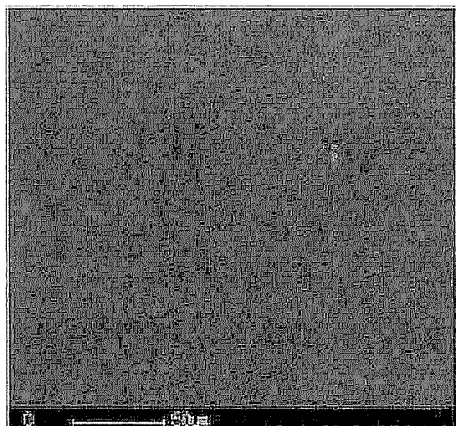


Fig. 9



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Fig. 10

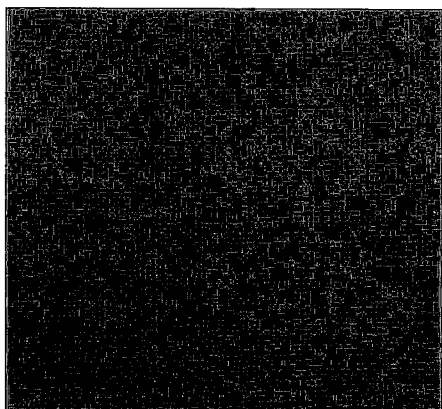
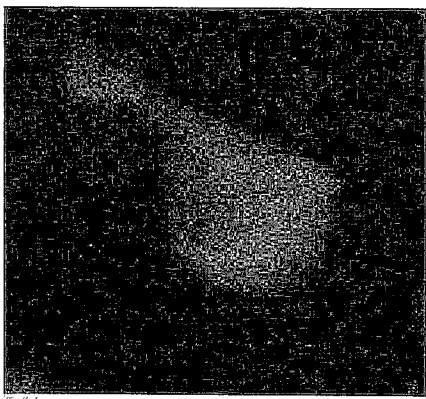


Fig. 11



Fig. 12



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Fig. 13

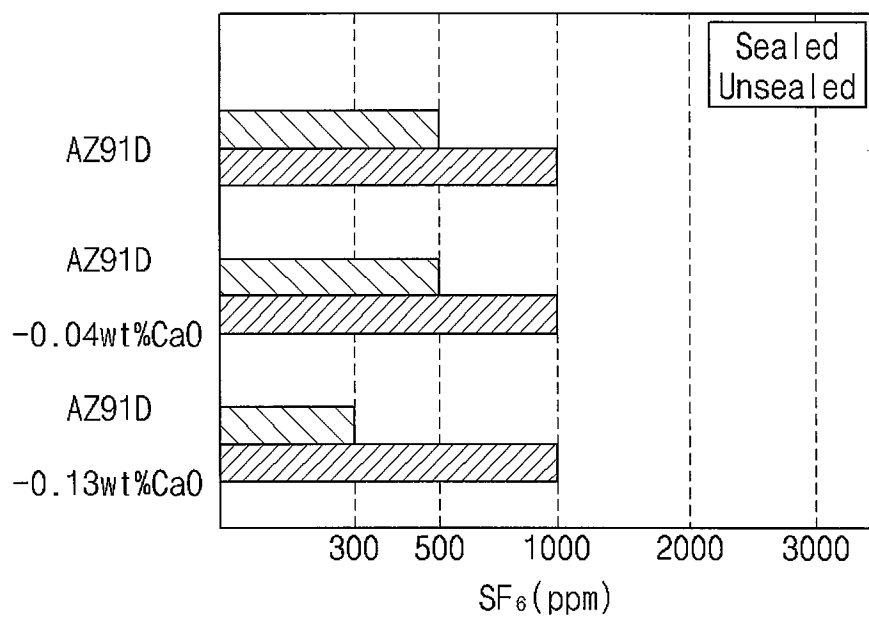
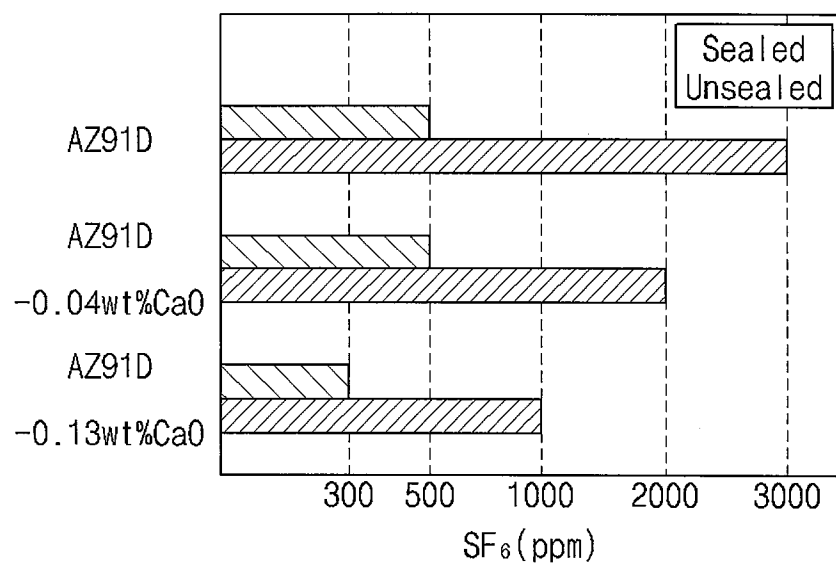


Fig. 14



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Fig. 15

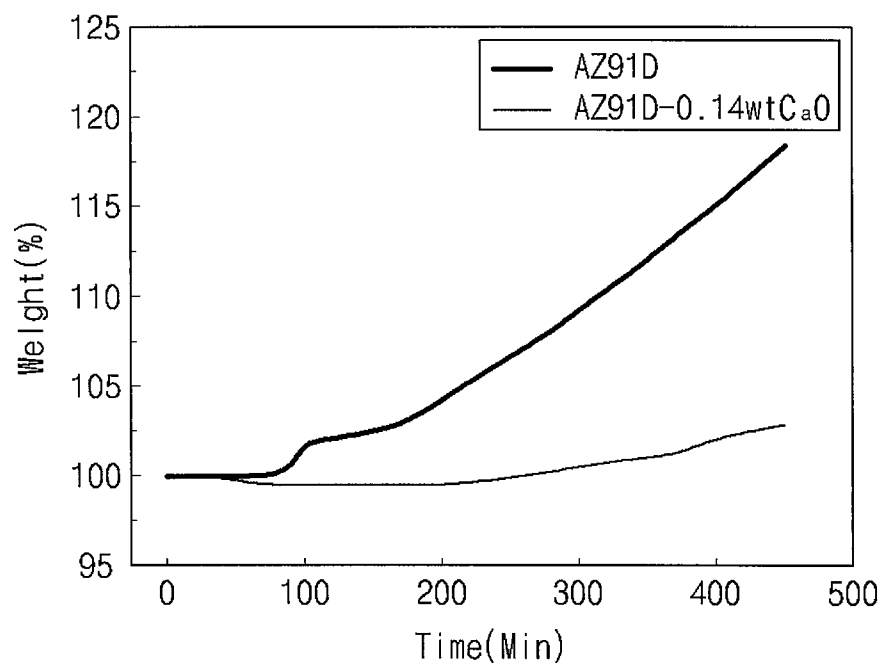
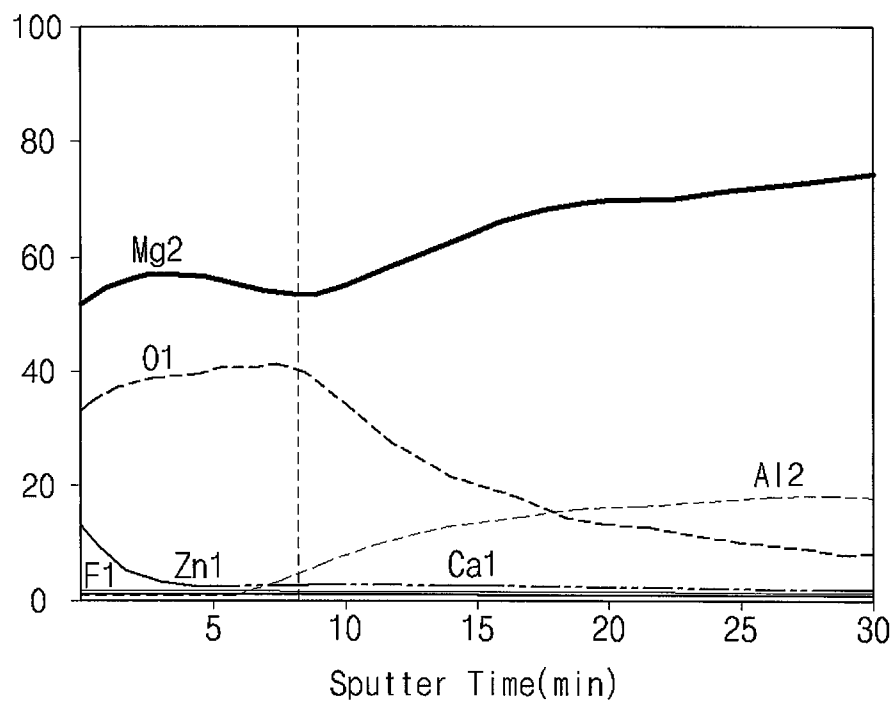
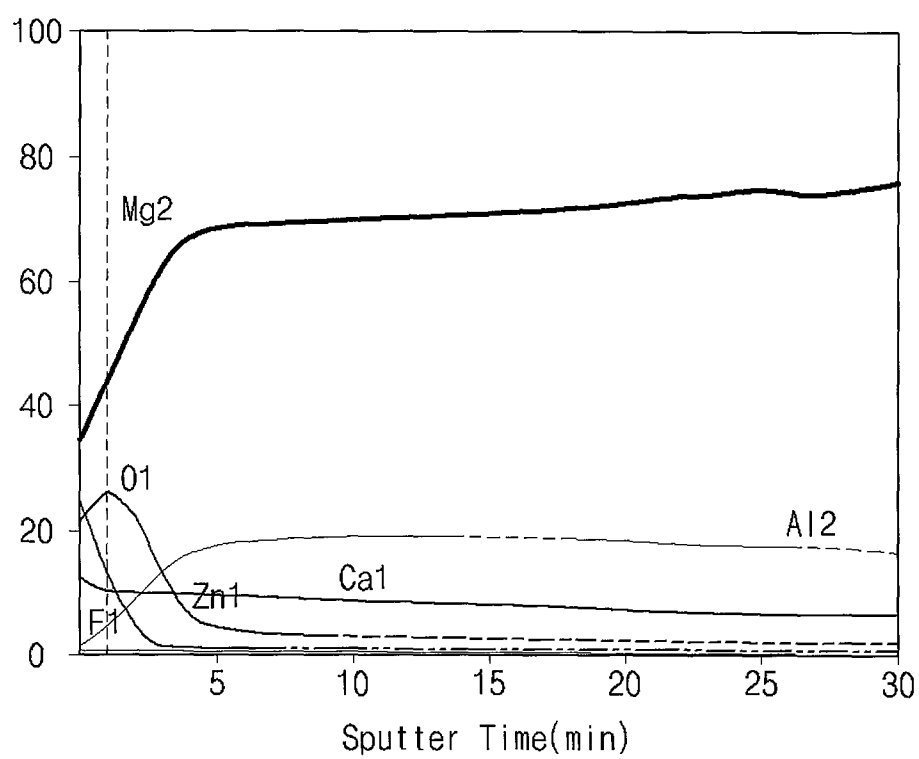


Fig. 16



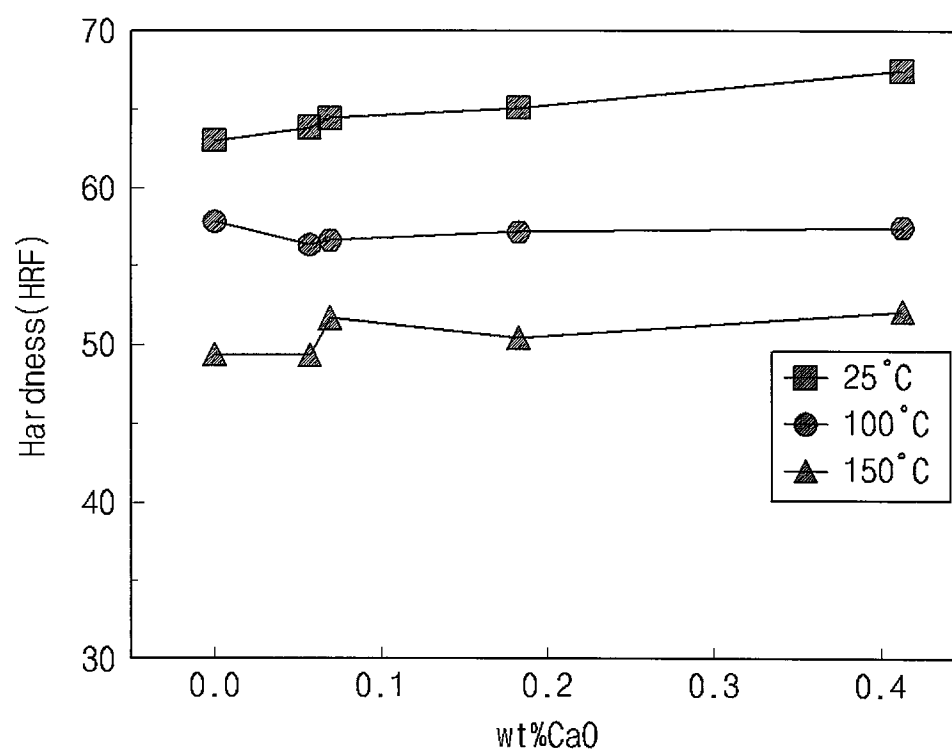
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Fig. 17



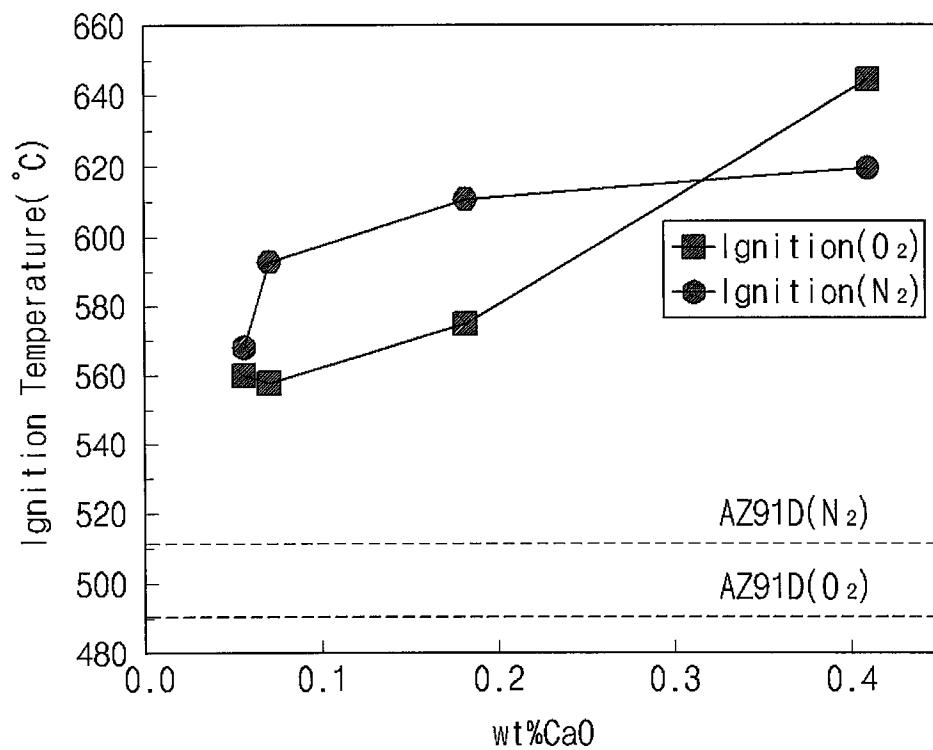
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Fig. 18



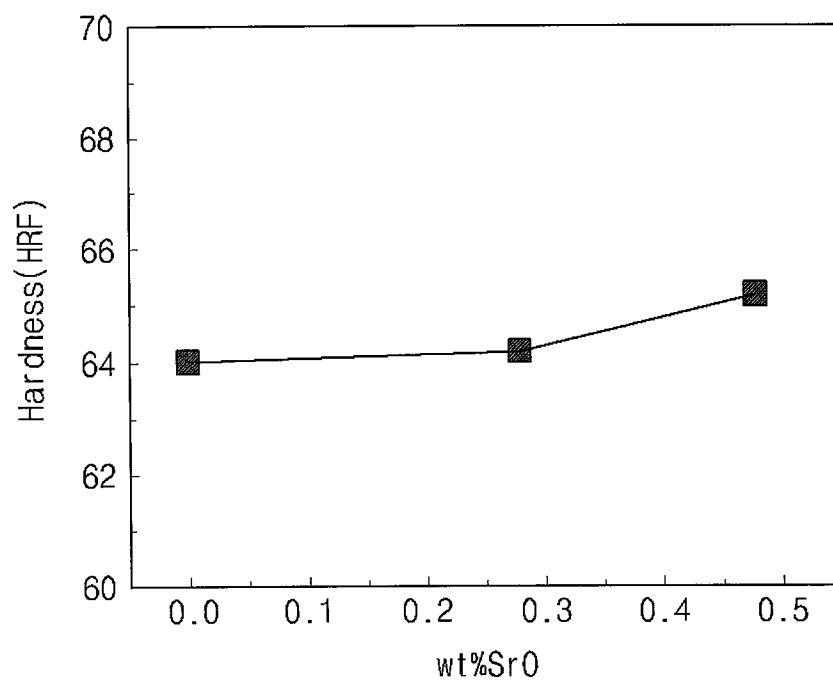
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Fig. 19



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Fig. 20



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Fig. 21

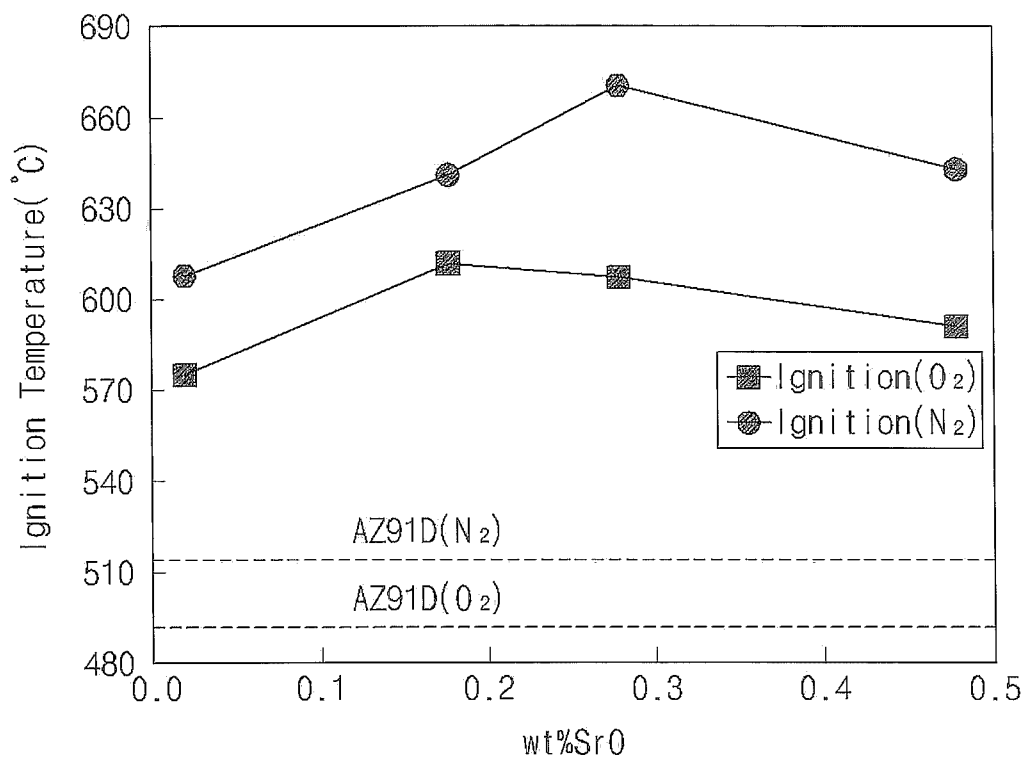
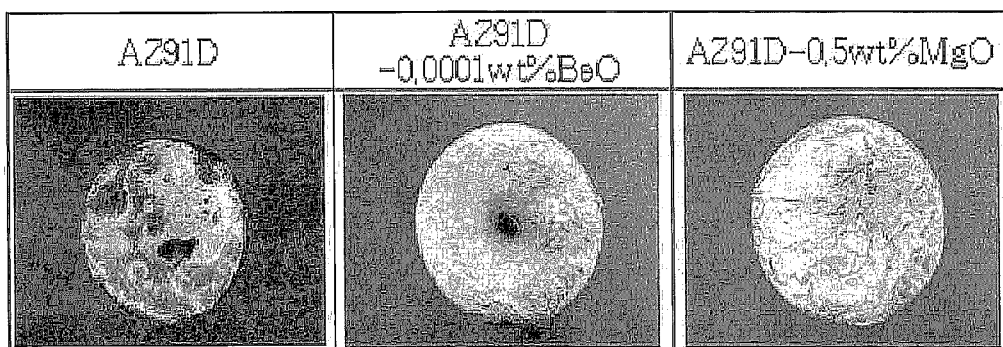
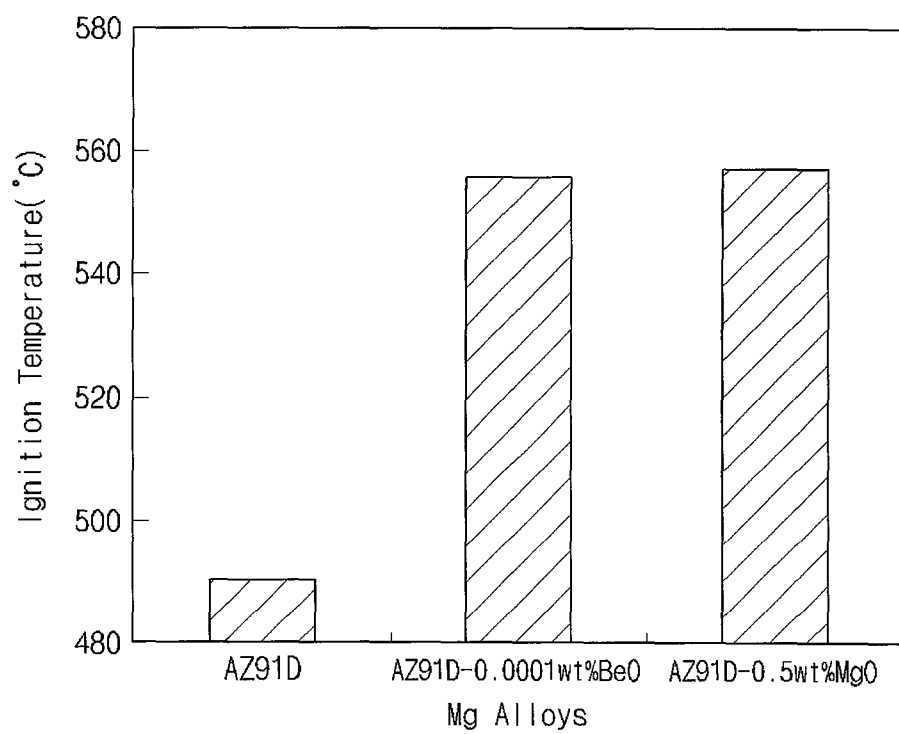


Fig. 22



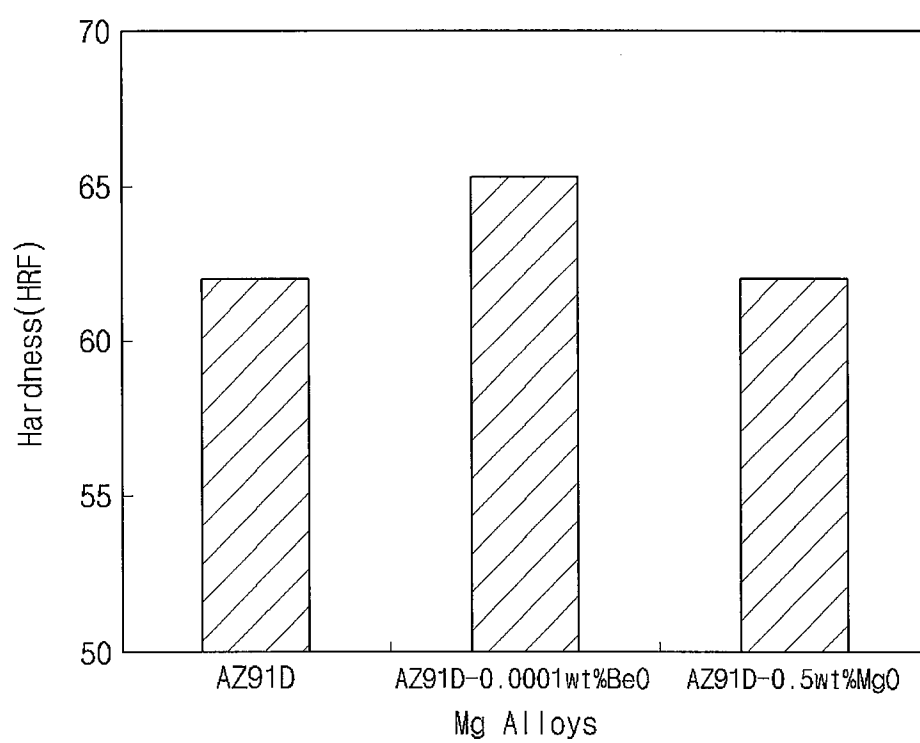
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Fig. 23



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Fig. 24



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Fig. 25

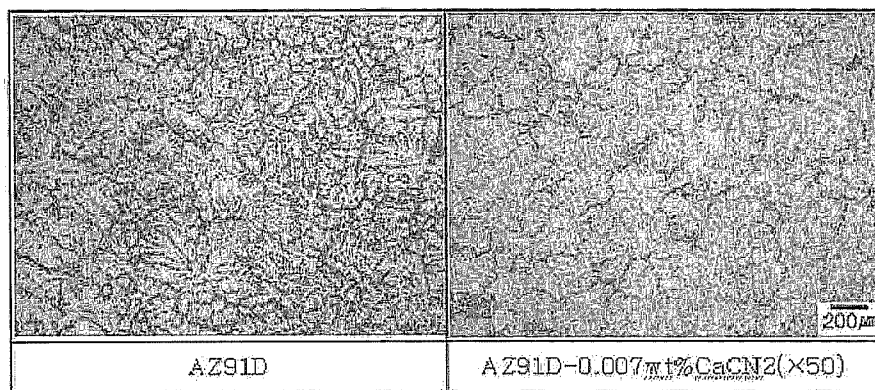
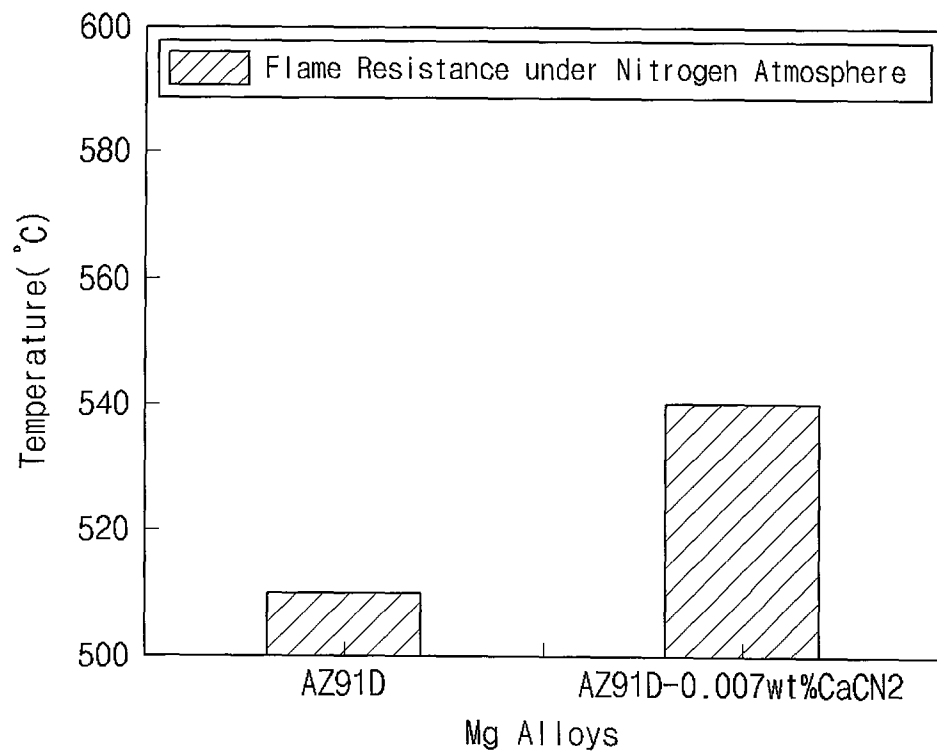


Fig. 26

	1	2	3	4	5	Average
AZ91D-0.007wt%CaCN ₆	65	64	67	67	66	66

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Fig. 27



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Fig. 28

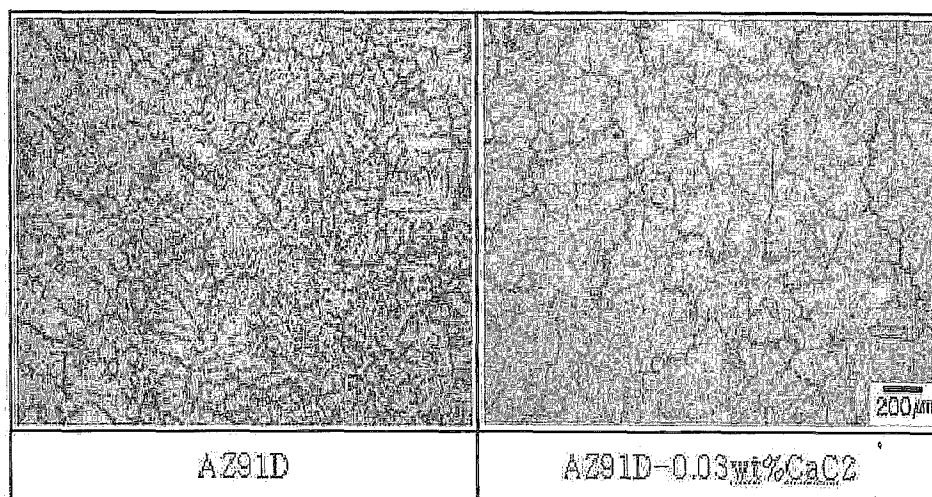
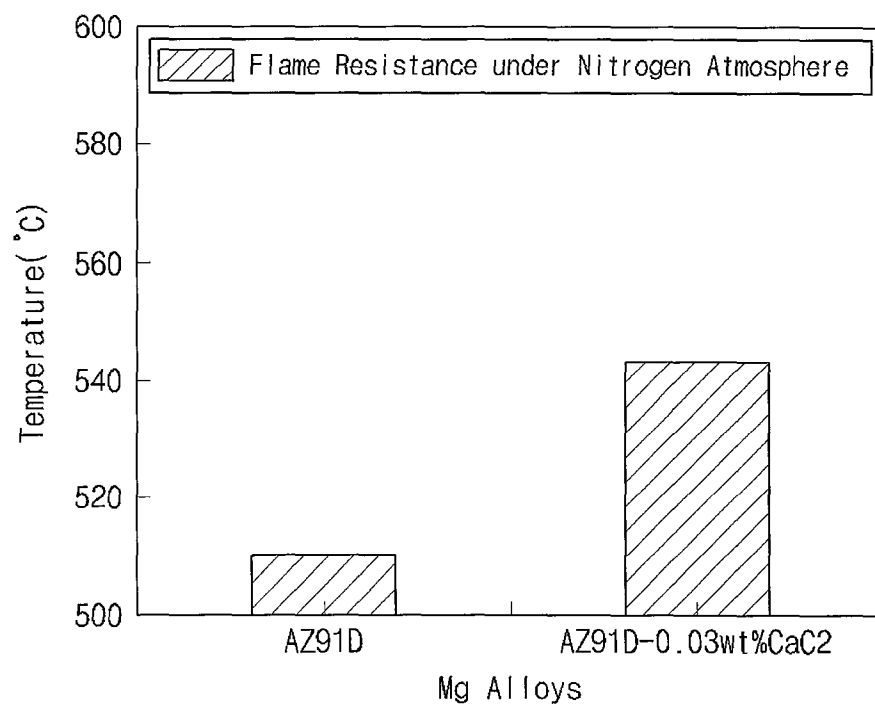


Fig. 29

	1	2	3	4	5	Average
AZ91D-0.03wt%CaC ₂	66	69	63	66	64	65.3

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Fig. 30



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Fig. 31

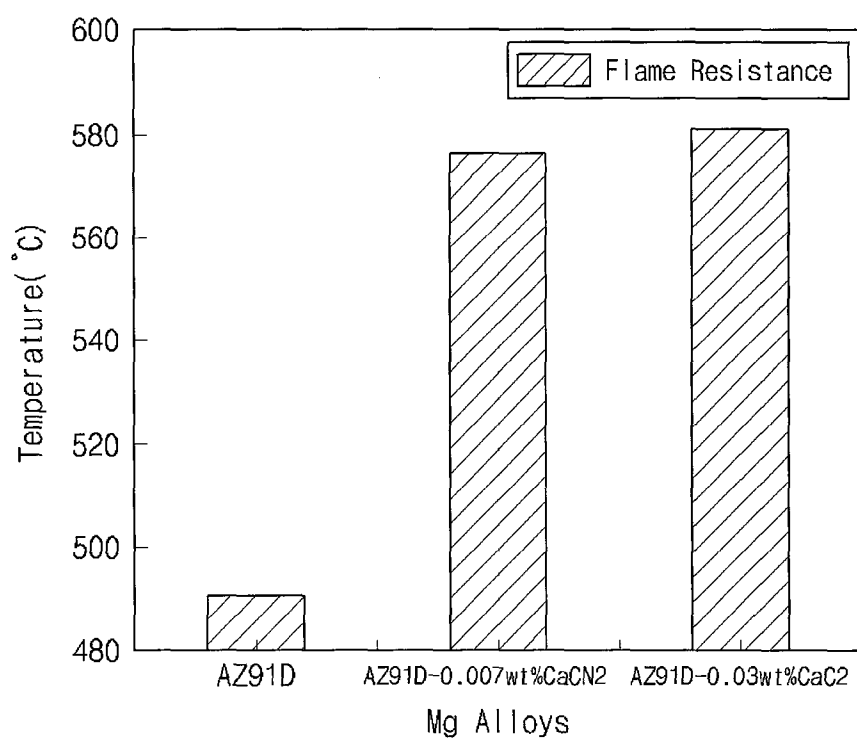
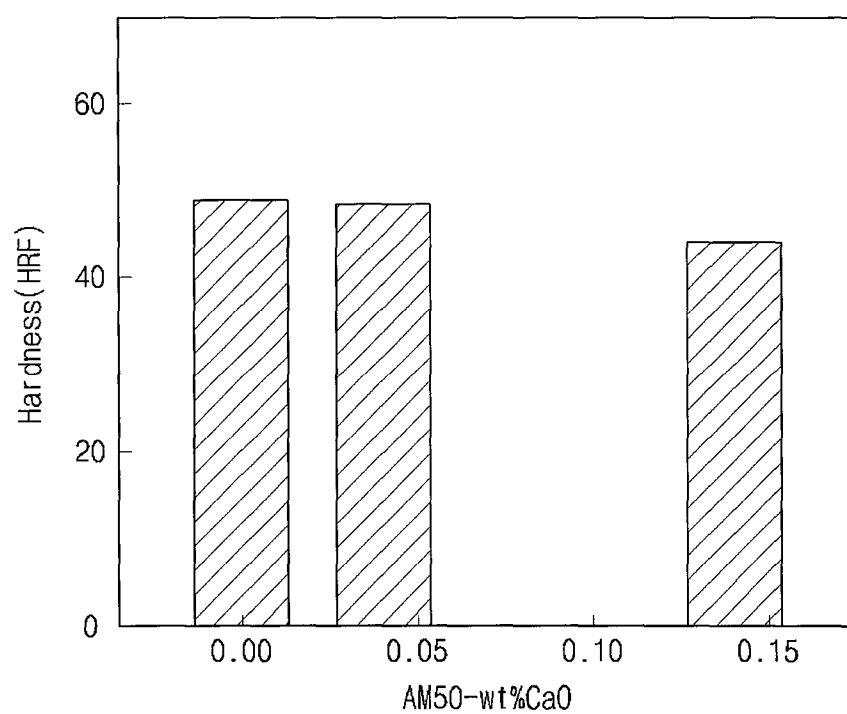
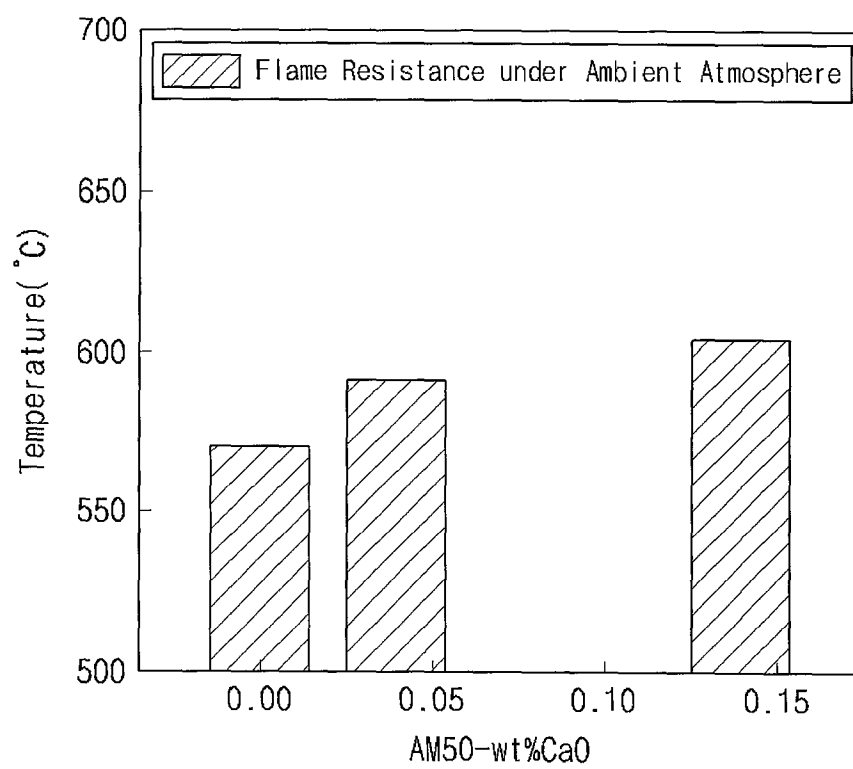


Fig. 32



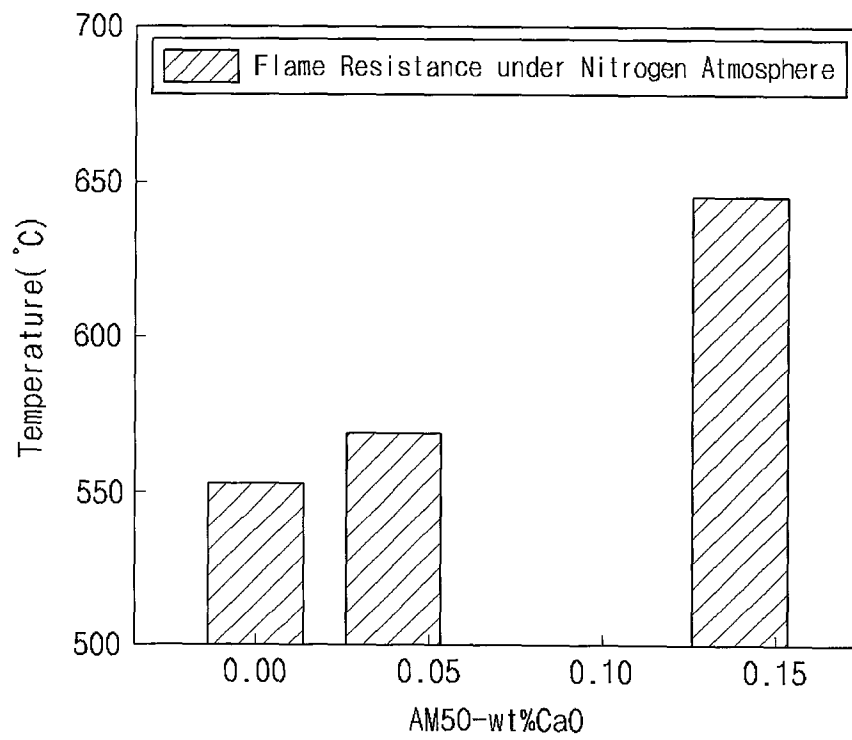
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Fig. 33



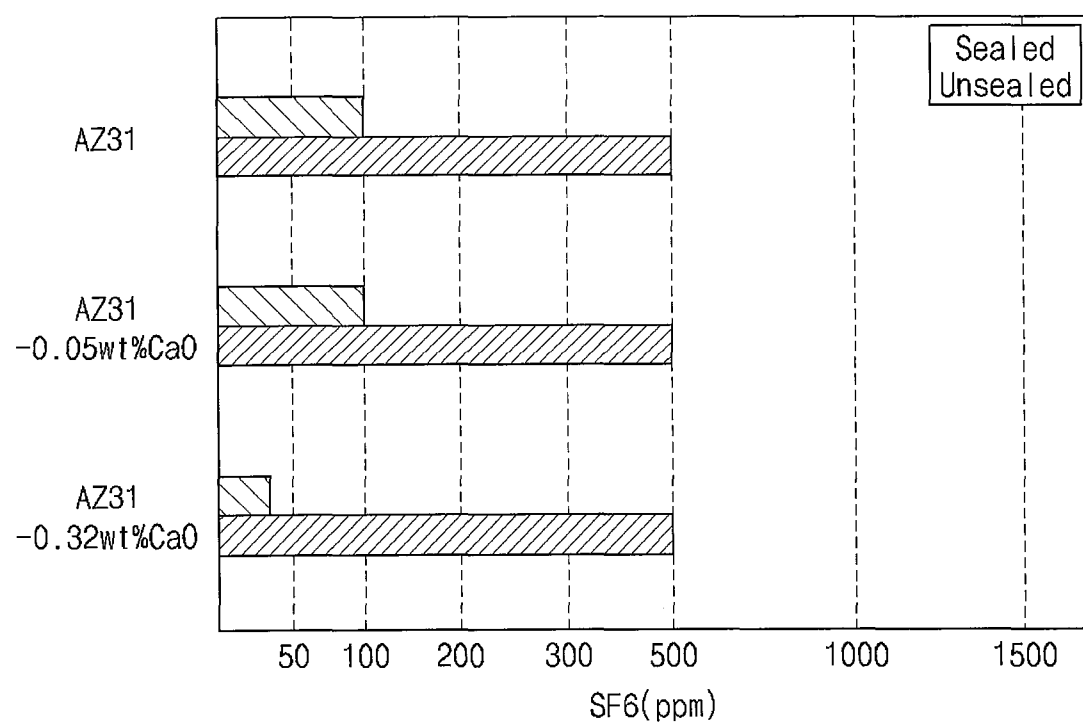
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Fig. 34



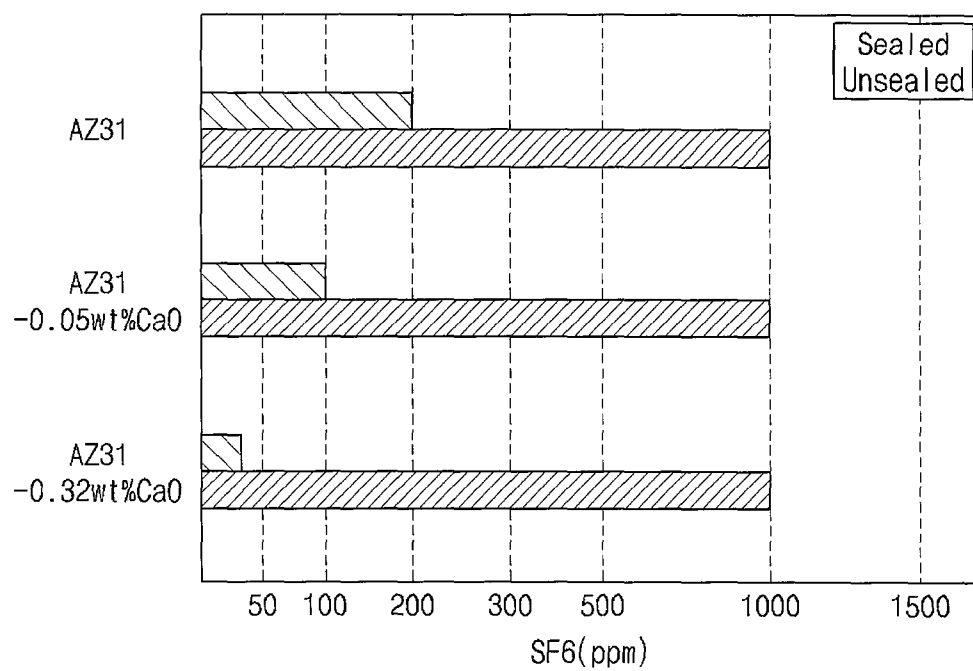
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Fig. 35



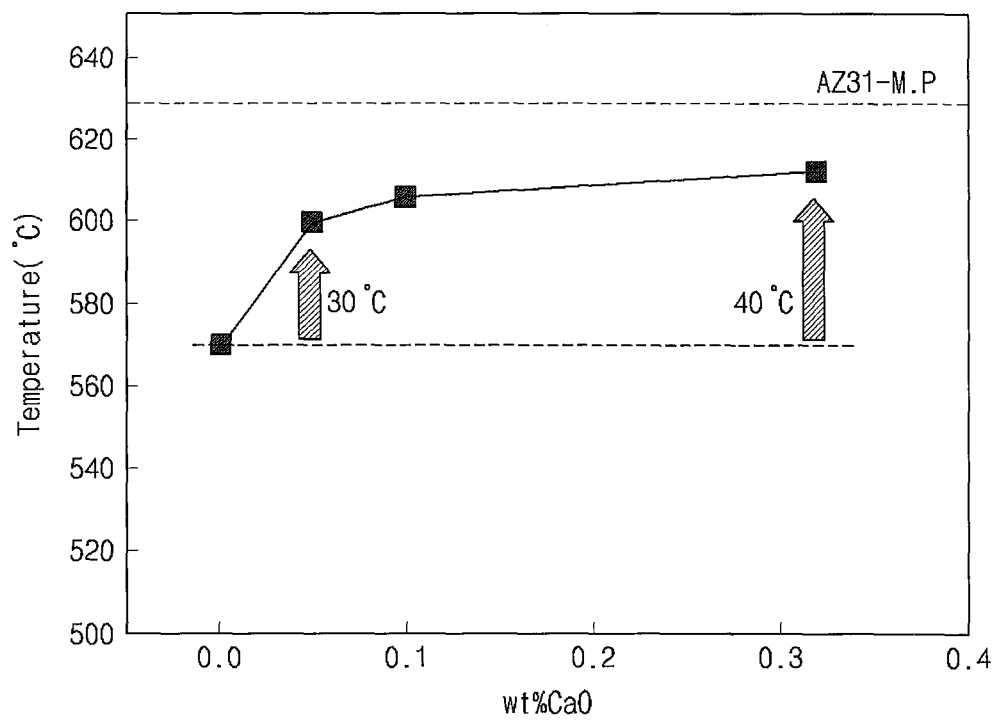
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Fig. 36



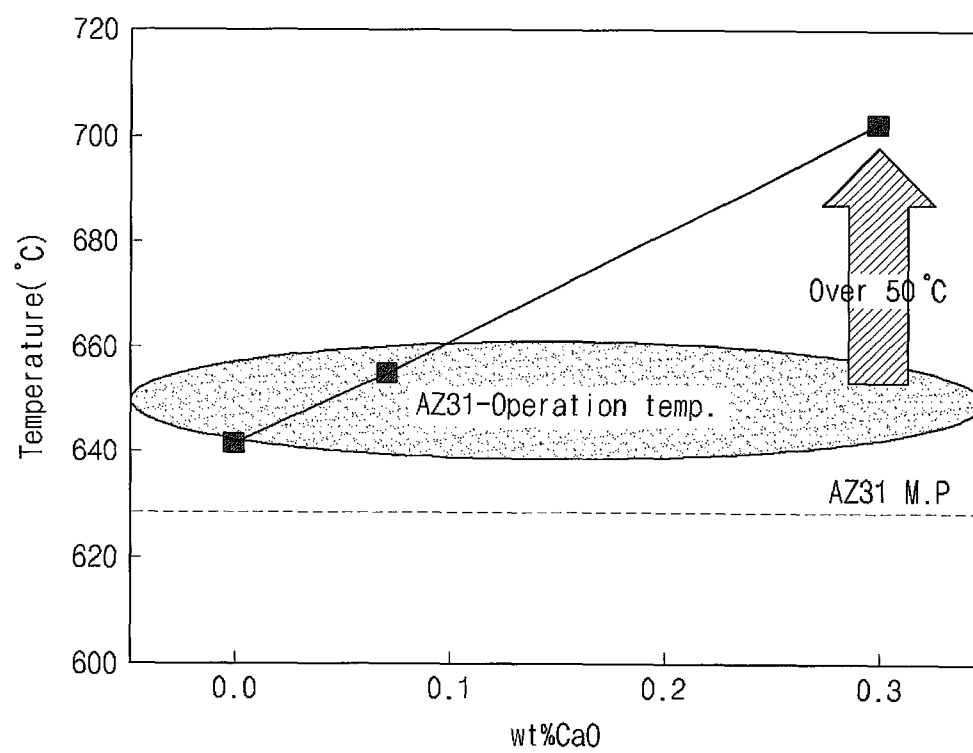
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Fig. 37



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Fig. 38



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Fig. 39

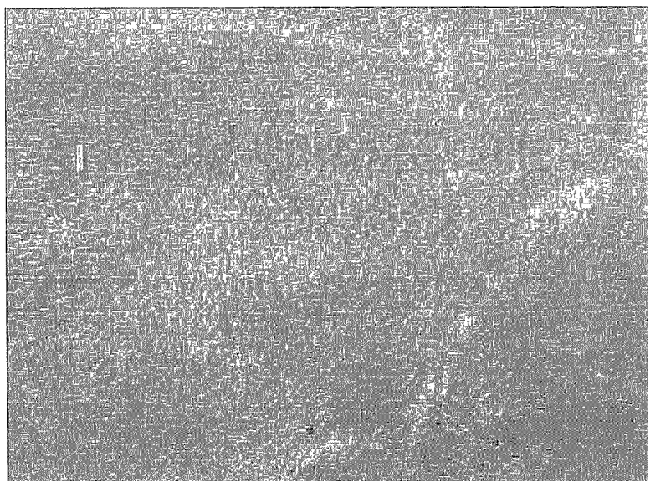


Fig. 40



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Fig. 41



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2008/005655**A. CLASSIFICATION OF SUBJECT MATTER***C22C 23/00(2006.01)i*

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C22C

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

Korean Utility models and applications for Utility models since 1975

Japanese Utility models and applications for Utility models since 1975

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

eKIPASS (KIPO internal), "Keywords: Mg alloy, CaO, grain refinement, ignition temperature, and casting"

Google Scholar, <http://scholar.ndsl.kr>**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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X ----- A	HIRAI, K. et al. 'Effects of Ca and Sr addition on mechanical properties of a cast AZ91 magnesium alloy at room and elevated temperature.' Materials Science & Engineering A. 25 August 2005, Vol. A403, Issues 1-2, pp. 276-280.	1-4, 7 ----- 5, 6, 8-16
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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

25 MAY 2009 (25.05.2009)

Date of mailing of the international search report

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Authorized officer

OH, Jun Cheol

Telephone No. 82-42-481-8506



INTERNATIONAL SEARCH REPORT

International application No.

PCT/KR2008/005655

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ----- A	US 6719857 B2 (OHORI, K et al.) 13 April 2004 see abstract; column 2 lines 36-67, column 3, column 4 lines 1-40, column 7 lines 4-9; claims 1-4	1-4, 7 ----- 5, 6, 8-16
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X ----- A	LI, P. et al. 'Microstructure and properties of AZ91D alloy with Ca additions.' Materials Letters. March 2005, Vol. 59, Issue 6, pp.671-675.	1-4, 7 ----- 5, 6, 8-16

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/KR2008/005655

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