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(54) METHOD FOR DEGASSING AND SOLIDIFYING ALUMINUM ALLOY POWDER

VERFAHREN ZUM ENTGASEN UND ERSTARREN VON ALUMINIUMLEGIERUNGSPULVER

PROCEDE POUR DEGAZER ET SOLIDIFIER DES POUDRES D'ALLIAGE D'ALUMINIUM

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(56) References cited:

JP-A- 1 075 604	JP-A- 1 156 402
JP-A- 3 281 748	JP-A-61 194 101
JP-A-63 121 628	JP-B-57 008 842
US-A- 4 435 213	

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89-148936 & SU-A-1 414 501 (DOVYDENKOVA A
V) , 7 August 1988
- **PATENT ABSTRACTS OF JAPAN vol. 011 no. 141**
(C-421) ,8 May 1987 & JP-A-61 281834
(SUMITOMO ELECTRIC IND LTD) 12 December
1986,

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Description

This invention relates to a method of degassing and consolidating a rapidly solidified aluminium alloy powder.

A number of methods of forming and consolidating a rapidly solidified aluminium alloy powder are known, for example extrusion, HIP and powder forging. In order to consolidate a powder, it is necessary to heat the powder, during which the structure obtained on quenching may be lost with a corresponding deterioration in the properties. Prevention of such a deterioration is achieved by rapid heating in a short time. A method relating to carrying out rapid heating for the purpose of consolidating a rapidly solidified aluminium alloy powder is disclosed in US Patent No. 4435213; a method relating to induction heating of general powder, not limited to aluminium, is disclosed in US Patent No. 5134260; and a method relating to rapid heating by hot air is disclosed in Japanese Patent Laid-Open Publication No. 158401/1991.

In the above described powder forging, extrusion and HIP methods, well known in the art, heating before consolidation is vital to decrease the deformation resistance of the powder and to allow the powder to be shaped with a low stress and, additionally, for the purpose of degassing.

In particular, degassing is an indispensable means for preventing the formation of bubbles in a solidified article, known as blistering, and, in the case of powder forging, for forming strong bonds between the grains. The methods described in Japanese Patent Laid-Open Publication No. 224602/1987 and "Kei-Kinzoku (Light Metals)" 37 (10) 1987, page 656-664 are referred to in this regard.

In the prior art techniques, degassing has generally been carried out by can-sealing a CIP (cold isotactic pressing) body and heating in a vacuum or in an inert gas atmosphere at a temperature of 400 to 600°C. The prior art methods have aimed to effect sufficient degassing by raising the temperature for 0.5 to 2 hours and maintaining it at a predetermined temperature for 0.5 to 2 hours, amounting to 1 to 4 hours, using an ordinary resistance heating furnace.

However, it has hitherto been pointed out that the above described degassing method has the disadvantage that the structure obtained on quenching, i.e. the effect of precipitating an element or phase finely and uniformly, which tends to be coarsely precipitated at an ordinary cooling rate, or the effect of rendering fine crystal grains, is lost by heating for a long time. This results in a deterioration in the properties of a shaped and consolidated body. Moreover, prevention of oxidation requiring a controlled atmosphere, results in higher productions costs.

SU-A-1414501 relates to a method of forming articles from aluminium alloy powders, wherein pressed blanks are heated in an electric furnace to 530°C at 50°C/min.

Rapid and uniform heating of a material having a low heat conductivity, such as green compacts, has generally been considered difficult to achieve. Ordinarily, the most suitable method for rapid heating on a commercial scale is induction heating. For example, it has been reported in Japanese Laid-Open Publication No. 134503/1974 that high frequency induction heating may be used for heating and sintering a green compact of a ferrous metal. Up to the present time, such high frequency induction heating has been utilised to effect sintering in a short time or sinter forging (forging for increasing the density of a preform which is being sintered).

However, induction heating has not been used for degassing a green compact of aluminium powder or aluminium alloy powder. The reasons for this are as follows.

Firstly, it has been considered that the presence of a stable alumina film (Al_2O_3) with a low electric conductivity on surfaces of aluminium powder or aluminium alloy powder results in an increase in the resistance of the powder and a decrease in the electric conductivity of a green compact. Consequently, effective heating is impossible by induction heating since Joule heating is not readily achieved in a material with a low electric conductivity, such as aluminium, and eddy currents are not readily generated in the green compact. Additionally, aluminium has a smaller magnetic permeability than ferrous materials.

Secondly, it has been considered that even if the induction heating of the powder pressed compact could be carried out, a temperature difference between the surface and central part thereof would be so large, owing to the low heat conductivity thereof, that it would be impossible to impose a uniform temperature.

The present invention provides a method of degassing aluminium powder or an aluminium alloy powder comprising utilising induction heating for a degassing means in a step of forming and consolidating the aluminium powder or aluminium alloy powder, whereby the above described disadvantages of the prior art can be overcome.

When consolidating a rapidly solidified aluminium alloy powder, the following points should be taken into consideration.

(A) The thermal history of the powder is accounted for so as to minimise deterioration of the texture of the powder when it is heated for consolidation.

(B) Bonding of the aluminium powder granules with each other is rendered as strong as possible.

(C) The consolidation is carried out at low cost.

Above all, for accomplishing (A), rapid heating by the induction or hot air methods disclosed in the aforementioned three patents is favoured. In the rapid heating method, however, there arises a problem in that bonding of the aluminium

powder granules with each other (B) does not readily occur. Therefore, heating in air, as described in US-4435213, results in a decrease in the fracture elongation, even if extrusion is carried out. In order to compensate for this problem, rapid heating is performed in an inert gas, vacuum degassing is carried out before consolidation and extruding or swaging working is performed to cause a large plastic deformation after the consolidation to increase the elongation or fracture toughness value of the consolidated material. In JP-158401/1991 an apparatus for rapid heating in a vacuum is disclosed. However, (C) above, i.e. consolidation at a low cost, is not thereby accomplished.

The present invention provides means for addressing the above described points (A), (B) and (C) and provides a consolidated body having a higher strength and toughness, without a reduction in other mechanical properties, compared with the consolidating methods of the prior art. A consolidating method for obtaining the same in an economical manner is also provided.

The inventors have made various studies to solve the above described problems and, consequently, have devised a method whereby degassing of aluminium or aluminium alloy powder can be carried out by the use on induction heating, whilst suppressing a deterioration in the microstructure thereof. The heating time can also be decreased to about 1/10 of the prior art. The present invention is based on this finding.

Accordingly, the present invention provides a method of degassing aluminium powder, aluminium alloy powder or aluminium composite alloy powder or mixed powders thereof, optionally containing non-metallic grains, before consolidation thereof, characterised by preforming the powder body to give a specific electric resistance of at most $0.2\Omega\text{cm}$, subjecting the preform directly to induction heating in a stagnant atmosphere of air at atmospheric pressure, raising the temperature to in the range of from 400 to 600°C at a temperature gradient of at least 0.8°C/sec when a temperature of 300°C has been reached, whereby heat-decomposable volatile components are removed to obtain a hydrogen content of at most 10ppm.

In the present invention, the above described induction heating is carried out in air.

In the present invention, moreover, re-adsorption of moisture can be prevented by subsequent cooling of the preform in an inert gas atmosphere.

In addition, the inventors have made various studies to solve the above described problems and, consequently, have found that the following procedures, differing from the prior art, are the most suitable for achieving the above described points (A), (B) and (C), leading to the present invention.

(i) As to heating the powder, rapid heating is employed as usual, but the heating temperature is maintained at at least 30°C higher than in the prior art.

(ii) As to consolidating the powder, it is preferable to use a powder forging method, not a HIP or extrusion method.

(iii) The atmosphere for rapid heating should not be a vacuum nor an inert gas atmosphere, but an inexpensive stagnant atmosphere of air at atmospheric pressure (the stagnant atmosphere).

(iv) Rapid cooling is preferably carried out after powder forging.

That is, the present invention consists in preforming aluminium powder, aluminium alloy powder or aluminium composite alloy powder or mixed powders thereof, optionally containing non-metallic grains, to give a specific electric resistance of at most $0.2\Omega\text{cm}$, subjecting the preform directly to induction heating in a stagnant atmosphere of air at atmospheric pressure, maintaining a temperature-raising gradient of at least 0.8°C/sec when a temperature of 300°C has been reached and raising the temperature to in the range of from 400 to 600°C, corresponding to a temperature of at least 30°C higher than the vacuum degassing temperature applied in the case of extruding the above described powder, whereby heat decomposable volatile components are removed to obtain a hydrogen content of at most 10ppm, and then directly subjecting the thus obtained product to hot working and thus consolidating the product.

At to the heating temperature, a higher temperature, i.e. 400°C to the melting point, can be chosen in the case of an alloy containing only an alloying element (Fe, Ni, etc) which does not lower the melting point of Al (MP 660°C). As a preferred embodiment of the present invention, a powder forging method can be used as the above described hot working.

In the present invention, the above described induction heating can be carried out in an inexpensive stagnant atmosphere of air and, moreover, both the strength and toughness can be improved compared with the prior art without carrying out degassing in a vacuum before consolidation, without subjecting the material to plastic working, such as extrusion, after consolidation, and without lowering the elongation and fracture toughness.

The present invention preferably furthermore comprises quenching at a rate of at least 10°C immediately after forging, or reheating at a temperature of at most the forging temperature and at least (the forging temperature - 50°C) without cooling to room temperature and subjecting to a quenching and solution treatment.

In addition, a particularly preferred embodiment of the present invention comprises carrying out the preforming of the above described powder after coating the inner wall of a metallic mold with a wetting agent without adding an organic wetting agent to the powder.

Infrared radiation heating or direct electric heating can also be used instead of the above described induction heat-

ing.

Brief Description of the Drawings

5 Figure 1 is an SEM photograph of a texture of a forged body obtained in Example 2-1) of the present invention.
 Figure 2 is an SEM photograph of a texture of a forged body obtained in Example 2-3) of the present invention.
 Figure 3 is an SEM photograph of a texture of a forged body obtained in Comparative Example 2-6) of the present invention.

10 Considering that it is difficult to heat the whole body of a green compact at a uniform temperature in a short time, heating for a long time, e.g. at at least 1 hour, has ordinarily been carried out using a resistance heating furnace. However, the microstructure obtained on quenching a powder is lost because the powder is exposed to a high temperature for a long time. Since a H₂O component in the air hinders the above described H₂O release reaction, and an O₂ component in the air oxidizes the powder, heating has generally been carried out in a vacuum at a low dew point and low O₂ concentration atmosphere, or in an inert gas atmosphere, so as to prevent this phenomenon.

15 According to conditions found as a result of the inventors' studies, it is rendered possible to rapidly heat a formed body of aluminium powder or aluminium alloy powder by induction heating, which has hitherto been considered unsuitable, and to effect sufficient degassing by induction heating in air. Removal of absorbed water or crystallization water can be sufficiently effected by heating in a short time and, because of the shorter heating time, contact time with the atmosphere at a high temperature can be decreased.

20 That is, the above described conditions are that the compacting pressure of a pressing mold is increased by about 20% as large as the prior art so as to increase electric contact of powder particles with each other, and an incident direction of magnetic induction flux of a high frequency and the frequency of the high frequency are optimised.

25 Examples of the aluminium alloy powder used in the present invention include not only rapidly solidified alloy powders but also those prepared by other methods. The composition thereof is not limited, but can be an aluminium composite alloy powder (aluminium or aluminium alloy powder in which a non-metallic or intermetallic compound is dispersed). Aluminium powder can also be used. In addition, non-metallic grains such as SiC or Al₂O₃ grains can be mixed with these powders.

30 Firstly, aluminium powders, aluminium alloy powders, aluminium composite alloy powders or mixed powders thereof, optionally containing non-metallic grains, are formed into a preform with an increased density to give a specific electric resistance of at most 0.2Ωcm. The forming in this case can be carried out by a mold pressing method, such as uniaxial compression, a CIP method or other methods, without using heat-decomposable organic lubricants. The powder grains are thus subjected to micro-shearing forces with each other so that they have metallic contact areas with each other.

35 When the specific electric resistance exceeds 0.2Ωcm, eddy currents are not readily generated and the temperature of the preform is not readily raised even by induction heating. If the output of an electric source is increased to raise the temperature rapidly, the temperature gradient between the surface of the preform and interior part thereof is increased due to the low heat conductivity of the preform owing to the large electric resistance, and cracks tend to occur due to thermal strain. The specific electric resistance of at most 0.2Ωcm can generally be accomplished by a compacting pressure of 4 to 6 tons/cm². When this is not accomplished within this pressure range, the mold pressing is carried out at a high pressure or the temperature of the powder is subsequently raised to decrease the deformation resistance thereof.

40 The preform is then subjected directly to induction heating using an electric source and rapidly heated to 400 to 600°C while maintaining a temperature-raising rate of 0.8°C/sec when a temperature of 300°C has been reached, during which the frequency is preferably adjusted to 3 kHz according to the inventors' experiments, although an optimum frequency can suitably be chosen depending on the particular material.

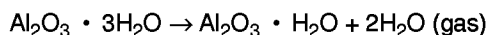
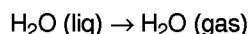
45 During consolidation of a rapidly solidified powder, its behaviour in its interior differs from that at its surface. The state of the interior primarily governs tensile strength and hardness, so if the thermal history for consolidation is reduced, the tensile strength and hardness of the powder itself are naturally increased. On the other hand, properties such as fracture elongation and fracture toughness are primarily governed by the state of the surface of the rapidly solidified powder.

50 An oxide film, i.e. alumina (Al₂O₃), on the surface of the aluminium powder is such a stable compound that it is hardly removed by reduction. This oxide film hinders strong bonding of the aluminium alloy powder grains with each other.

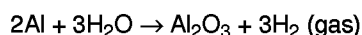
55 Accordingly, a method has been proposed comprising subjecting the powder to plastic flow working, e.g. extrusion or upset working, thus mechanically breaking the oxide film, and exposing and bonding fresh surfaces of aluminium. It has been known up to the present time, however, that even when using the extrusion method, only a product with a low elongation and low fracture toughness value is obtained if degassing before the consolidation is insufficient. Now, the degassing method will be illustrated.

A gas-atomised and rapidly solidified aluminium alloy powder has an oxide film with a thickness of 50 to 100Å covered on the surface thereof, the surface oxide film further containing absorbed water or crystallisation water, which causes a decrease in the elongation or fracture toughness value of the solidified material.

These water components can be removed by the following reactions:



These removal reactions take place at 100 to 400°C. At a temperature of 300°C or higher, water vapour generated by the above described reactions reacts directly with aluminium to evolve hydrogen. That is, the following reaction takes place,



A method employed to accelerate these reactions comprises heating for a long time (a longer time allows the reactions to proceed further), heating in a vacuum (a lower atmospheric pressure moves the equilibrium of these reactions to the right) or heating in an inert gas with a low dew point (equilibrium is moved to the right because of less H₂O (gas) at a low dew point). The object of using the inert gas atmosphere is to prevent the powder from oxidising.

From this point of view, it can be understood that rapid heating is effective for breaking the structure of the interior part of the powder, but is disadvantageous from such a point of view that release of the water absorbed on the surface oxide film of the powder and the crystallisation water is accelerated. It is probably due to this reason that in Examples 1 and 2 of JP-158401/1991, the tensile strength is improved but the elongation and fracture toughness values are lowered. In Example 3, both the tensile strength and elongation are improved, which is probably due to heating in an inert gas and subsequent degassing in vacuum. However, it is assumed that an ordinary heat-treatment (T7) is finally carried out in this Example and the effect of the rapid heating is decreased to half.

The inventors have made various examinations as to methods whereby a sufficient degassing can be carried out in an economical manner even when using rapid heating and, consequently, have found that this problem can be solved by utilising the hydrogen gas evolved by the above described release reaction. The above described generation of hydrogen gas takes place in particular, at a high temperature. The amount of the thus generated hydrogen gas, depending on the heating temperature, is generally about 30ppm. There are pores of about 25% in a green compact of the powder and the volume of the hydrogen generated amounts to about 10 times as much as that of the pores. In this case, it is required to hold the generated hydrogen in the pores of the green compact and to introduce an inert gas (not within the scope of the present invention) into and around the green compact without stirring the atmosphere, in particular, to maintain a stagnant atmosphere, so as to impart to the hydrogen a function of turning out harmful steam or oxygen present in the pores of the green compact and allowing the foregoing reactions to proceed. Furthermore, in order to generate a large quantity of hydrogen at once, heating when a temperature of 300°C has been reached for generating hydrogen should be carried out at a rate of at least 0.8°C/sec, and in order to generate hydrogen in a large quantity in a series of degassing reactions, it is required to heat to a temperature of as high as possible. Therefore, the heating temperature should be at least 30°C, preferably at least 50°C, higher than that of the vacuum degassing method carried out before extrusion in the prior art (generally heated at about 450°C). In this way, the structure of the powder surface tends to be fixedly bonded.

As a measure of the tendency of bonding of the powder, it is required that the amount of the residual hydrogen is at most 10 ppm.

When the heating temperature is higher, the structure of the interior part of the powder tends to be coarse even if rapid heating is effected and it is required to carry out (i) heating in a short time, (ii) consolidation in a short time and (iii) quenching after consolidation.

(i) For the purpose of rendering most advantageous "heating in a short time", the object to be heated needs to be as small as possible. In this respect, in the extrusion method, the end part and residual part are discarded and a large green compact is used to obtain a plurality of products in one extrusion and to increase the yield, so that the rapid heating is naturally limited. On the other hand, in the present invention, a green compact is small and rapid heating is possible. A green compact is generally subjected to CIP (cold isotactic pressing), but in the powder forging method, uniaxial compression by a metallic mold is applied. In this case, shearing of the powder when compressed uniaxially is more effective than when compressed isotropically and contact of the powder grains is increased by the newly exposed surfaces. Thus, inductive eddy currents are increased and heat generated in the

vicinity of the compact surface is more rapidly propagated to the interior part. Therefore, the forging method is more advantageous in this respect.

(ii) The most effective method for consolidating in a short time is also the powder forging method. The time required for powder forging is about 0.7 second as compared with about 5 minutes required for extrusion and about 20 minutes required for HIP (hot isotactic pressing).

(iii) For quenching after consolidation, it is required to separate a product from a tool used for working after hot working and powder forging is advantageous for this purpose. As to the cooling rate, about 100 °C/sec can be accomplished by water cooling, but in this case, there is a problem of cracking, in particular, when using a brittle material. In such a case, blowing of cooling air (cooling rate of about 10 to 20 °C/sec) should be carried out and the cooling rate is thus adjusted to at least 10 °C/sec. Since it is thought that a sufficient solution treatment cannot be effected sometimes by only direct cooling after forging in the case of alloys of heat-treatment type, it is preferable to reheat just after forging, rather than reheating after cooling to room temperature, so as to reduce the thermal history to as small as possible during the same time. The reheating temperature during the same time is specified to be at the forging temperature (the forging temperature - 50 °C) for the purpose of preventing generation of blisters and obtaining a sufficient annealing.

When plastic working is carried out after consolidation in order to reduce the thermal history, heating is necessary for the plastic working. Accordingly, this is not preferable. An organic lubricant is not added because it lowers the heat conductivity during heating of the green compact and hinders a rapid rise in the temperature owing to the evaporation heat thereof.

A consolidated body according to the present invention has a feature such as to be more changeable (concerning the structure distribution of a precipitate, obtained by X-ray diffraction, shape of a precipitate, size of a precipitate, tendency of coarsening) for the same composition at a higher temperature (substantially the same as the powder forging temperature) because it contains more non-equilibrium phases than those prepared by other methods. When a powder is heated for a long time in an inert gas and then subjected to extrusion or powder forging so as to turn out the air (predominantly consisting of nitrogen) contained in pores or gaps by hydrogen released from the powder surface, N₂ or Ar can be detected, while in the consolidated body of the present invention, such elements are contained only in an amount of at most the detectable limit.

The degassed powder obtained according to the present invention, having such a clean surface as having little adsorbed water or crystalline water, can be subjected to powder forging as heated. Accordingly, this is forged by a known forging method just after degassing. However, an induction heating has the disadvantage that the temperature of a body to be heated is more non-uniform as compared with an ordinary atmospheric heating furnace and accordingly, when the temperature gradient is large, the temperature thereof can be rendered uniform by holding at a predetermined temperature in an atmospheric heating furnace after temperature raising, during which the atmosphere should be of an inert gas.

The preform rapidly heated and degassed in this way is immediately charged in a metallic mold at about 200 °C and subjected to forging at a compacting pressure of 2 to 12 tons/cm².

Examples

The present invention will now be illustrated in detail by the following examples without limiting the same. In the following Experimental Examples and Examples, an induction heating is carried out by about 3 kHz.

Experimental Example A

About 250 g of an air-atomized powder (mean grain diameter: about 50 μm) with a composition of Al-25Si-2.5Cu-1Mg (by weight) was compacted at a compacting pressure of 4 tons/cm² in a diameter 100 mm x height 20 mm to give a specific electric resistance of 0.02 Ω cm, heated to 500 °C under the following conditions A-1) to A-5), removed into a can having an Ar atmosphere when the heating was finished, cooled to 50 °C within 1 minute in an Ar stream, and then subjected to measurement of the quantity of oxygen and the quantity of hydrogen in the powder, the hardness (mHv) and the grain diameter of primary crystal Si. The results are shown in Table 1.

A-1) Induction heating in the air (32°C /sec).....Present Invention

A-2) Induction heating in the air (8.0 °C/sec).....Present Invention

A-3) Induction heating in the air (4.0 °C/sec).....Present Invention

A-4) Induction heating in the air (0.8 °C/sec).....Present Invention

A-5) Induction heating in the air (0.2 °C/sec).....Outside Present Invention

For comparison, the same compacted body was heated to 500°C under the following conditions A-6) to A-8)

using a resistance heating furnace.

A-6) Resistance furnace heating in vacuum (maintained for 1 hour) Outside Present Invention

A-7) Resistance furnace heating in N₂ atmosphere (maintained for 1 hour) Outside Present Invention

A-8) Resistance furnace heating in the air (maintained for 1 hour) Outside Present Invention

The properties of the alloy powders thus obtained are shown in Table 1.

Table 1

Heating Conditions	Oxygen Quantity (wt%)	Hydrogen Quantity (ppm)	Powder Hardness (mHv)	Primary Crystal Si Grain Diameter (μm)
Within Present Invention				
A-1	0.27	3	172	3.2
A-2	0.28	4	153	3.1
A-3	0.30	3	130	4.0
A-4	0.28	5	115	7.8
Outside Present Invention				
A-5	0.33	6	100	10.2
A-6	0.28	3	92	11.2
A-7	0.28	9	95	10.7
A-8	0.38	17	102	10.0
Note 1: Powder Hardness (mHv): mean value of five points				
Note 2: Primary Crystal Si Grain Diameter: mean value of thirty samples				

From the results of Table 1, it is apparent that 1) the degree of degassing can substantially be obtained as in degassing in vacuum and 2) the structure is not coarsened and the hardness is high because of little thermal history.

Experimental Example B

The procedures under the conditions of Experimental Examples A-1), A-4), A-5), A-7) and A-8) were repeated except using a mixed powder of air-atomized, industrial grade pure aluminum powder (mean grain diameter: 50 μm) and 30 volume % of SiC grains with a mean grain diameter of 1.5 μm, as a raw material powder. The properties of the resulting powders are shown in Table 2, in which the powder hardness is measured as to the aluminum powder.

Table 2

Experimental Example	Heating Conditions	Oxygen Quantity (wt%)	Hydrogen Quantity (ppm)	Powder Hardness (mHv)	Primary Crystal Si Grain Diameter (μm)
Within Present Invention					
B-1	A-1	0.20	4	95	-
B-2	A-4	0.19	5	93	-
Outside Present Invention					
B-3	A-5	0.26	7	85	-
B-4	A-7	0.21	8	63	-
B-5	A-8	0.32	15	62	-
Note 1: Powder Hardness (mHv): mean value of five points					

Experimental Example C

The procedures under the conditions of Experimental Examples A-1), A-4), A-5), A-7) and A-8) were repeated except using a mixed powder of air-atomized, Al-20Si-5Fe-2Ni alloy powder (mean grain diameter: 50 μm) and alumina powder with a mean grain diameter of 0.5 μm , as a raw material powder. The properties of the resulting powders are shown in Table 3. The quantity of oxygen is a quantity from which the quantity of oxygen contained in the alumina grains has been removed by calculation. The powder hardness is measured as to the aluminum alloy powder.

Table 3

Experimental Example	Heating Conditions	Oxygen Quantity (wt%)	Hydrogen Quantity (ppm)	Powder Hardness (mHv)	Primary Crystal Si Grain Diameter (μm)
Within Present Invention					
C-1	A-1	0.26	4	186	2.6
C-2	A-4	0.29	3	179	2.4
Outside Present Invention					
C-3	A-5	0.32	5	145	5.6
C-4	A-7	0.28	10	108	6.8
C-5	A-8	0.40	19	113	6.5
Note 1: Powder Hardness (mHv): mean value of five points Note 2: Primary Crystal Si Grain Diameter: mean value of thirty samples					

Experimental Example D

About 500 g of an air-atomized powder with a composition of Al-20Si-5Fe-1Ni (mean grain diameter: 50 μm) was compacted in a diameter of 100 mm and height of 40 mm while varying the compacting density as shown in Table 4, and then subjected to measurement of the specific electric resistance. In the central part and outer circumferential part of the green compact were respectively made two holes each having a diameter of 1.0 mm in which a thermocouple is to be inserted and the temperature raising gradient was sought in which the temperature gradient between both the sites was not 70 $^{\circ}\text{C}$ or higher and the fastest temperature raising could be obtained.

Table 4

Green Compact No.	Specific Electric Resistance of Green Compact ($\Omega\text{ cm}$)	Maximum Temperature-Raising Gradient in Which Temperature Gradient between Central Part and Peripheral Part of Green Compact is not 70 $^{\circ}\text{C}$ or Higher ($^{\circ}\text{C}/\text{sec}$)
D-1	0.001	25
D-2	0.005	16
D-3	0.01	8
D-4	0.02	3.2
D-5	0.05	2
D-6	0.1	0.9
D-7	0.2	0.4
D-8	0.5	0.2
D-9	1.0	0.09
D-10	2.0	not reached 500 $^{\circ}\text{C}$

As shown in Table 4, the temperature-raising efficiency is not good at a specific electric resistance of about 0.2Ω cm or more.

Example 1

An air-atomized powder (mean grain diameter: about 50μm) with a composition of Al-25Si-2.5Cu-1Mg (by weight, same hereinafter) was compacted in a diameter 100 mm x height 20 mm to give a specific electric resistance of 0.02 Ω cm and heated in the air to 500 °C from room temperature for 4 minutes by induction heating. The product was immediately charged in a metallic mold (200 °C) lined with graphite lubricant, powder-forged at a compacting pressure of 8 tons/cm² and just after the forging, cooled by immersing in water at room temperature. The forged body was subjected to natural ageing for 4 days, after which Rockwell hardness B scale (H_R B) was measured to obtain an H_R B of 86.

For comparison, the green compact prepared in the similar manner to Example 1 was heated for 1 hour in a nitrogen atmosphere at 500°C in a resistance furnace and after heating, forged, cooled and then subjected to natural ageing and measurement of the hardness to obtain an H_R B of 79 (Comparative Example 1).

Example 2

250 g of an air-atomized powder (mean grain diameter: about 50 μm) with a composition of Al-25Si-2.5Cu-1Mg was compacted at a compacting pressure of 4 tons/cm² in a diameter 100 mm x height 20 mm to give a specific electric resistance of 0.02 Ω cm, heated to 500 °C under the following conditions 2-1) to 2-5), charged into a mold heated at 200 °C when the heating was finished, subjected to powder forging at a compacting pressure of 8 tons/cm², immediately cooled by immersing in water. Thereafter, the product was subjected to natural ageing for 4 days.

In the case of 2-3'), "moistened", the green compact was exposed to an atmosphere at a temperature of 40 °C and a humidity of 90 % for 24 hours, before heating and degassing, thus adsorbing a large amount of water on the surface of the powder, and then subjected to the steps after the heating and degassing in a similar manner.

2-1) Induction heating in the air (32°C /sec).....Present Invention

2-2) Induction heating in the air (8.0 °C/sec).....Present Invention

2-3) Induction heating in the air (4.0 °C/sec).....Present Invention

2-3') Induction heating in the air moistened (4.0 °C/sec).....Present Invention

2-4) Induction heating in the air (0.8 °C/sec).....Present Invention

2-5) Induction heating in the air (0.2°C/sec).....Outside Present Invention

For comparison, the same compact was heated to 500 °C under the following conditions 2-6) to 2-7) using a resistance heating furnace, forged, then heated at 485 °C for 2 hours and immersed in water to effect a solution treatment and thereafter, subjected to natural ageing for 4 days.

2-6) Resistance furnace heating in N₂ atmosphere (maintained for 1 hour) Outside Present Invention

2-6') Resistance furnace heating in N₂ atmosphere (maintained for 1 hour) moistened Outside Present Invention

2-7) Resistance furnace heating in the air (maintained for 1 hour) Outside Present Invention

2-7') Resistance furnace heating in the air (maintained for 1 hour) moistened Outside Present Invention

The properties of the alloy powders thus obtained are shown in Table 5.

Table 5

Sample No.

Degassing Conditions

	<u>Heating Means</u>	<u>Atmosphere</u>	<u>Temperature Conditions</u>	<u>Moistened</u>
Example				
2-1	Induction Heating	In The Air	Temp. Raising at 32°C /sec	No
2-2	Induction Heating	In The Air	Temp. Raising at 8.0 °C /sec	No
2-3	Induction Heating	In The Air	Temp. Raising at 4.0 °C /sec	No
2-3'	Induction Heating	In The Air	Temp. Raising at 4.0 °C /sec	Yes
2-4	Induction Heating	In The Air	Temp. Raising at 8.0 °C /sec	No
Comparative Example				
2-5	Induction Heating	In The Air	Temp. Raising at 0.2 °C /sec	No
2-6	Resistance Furnace Heating	In N ₂ Atmosphere	Maintained For 1 Hour	No
2-6'	Resistance Furnace Heating	In N ₂ Atmosphere	Maintained For 1 Hour	Yes
2-7	Resistance Furnace Heating	In The Air	Maintained For 1 Hour	No
2-7'	Resistance Furnace Heating	In The Air	Maintained For 1 Hour	Yes

Table 5 (continued)

Sample No.	Properties of Forged Body				
	Amount of Oxygen (%)	Amount of Hydrogen (ppm)	Tensile Strength (kg/mm ²)	Elongation (%)	Hardness H _R B
<u>Example</u>					
2-1	0.23	3	48	1.2	89
2-2	0.30	4	50	2.0	85
2-3	0.29	3	52	3.0	86
2-3'	0.76	4	51	1.8	88
2-4	0.32	5	50	1.8	85
<u>Comparative Example</u>					
2-5	0.38	8	45	1.0	83
2-6	0.28	9	49	1.8	87
2-6'	0.38	11	32	0.2	82
2-7	0.42	17	30	0.0	75
2-7'	0.93	23	23	0.0	65

From the results of Table 5, it is apparent that according to the present invention, effective degassing is achieved and forged bodies having a good balance of properties, such as hardness, tensile strength, elongation, etc. are obtained without a deterioration in the microstructure obtained on quenching the raw material powder.

With regard to the results of 2-3') and 2-6'), it is apparent that in the effective degassing method of the present invention, degassing (removal of adsorbed water) can sufficiently be carried out even if there is a large amount of adsorbed water (becoming crystalline water of alumina during heating), while in the degassing method 2-6') of the prior art, it is difficult to remove such a large amount of the adsorbed water and the resulting forged body has inferior properties.

The forged bodies obtained in Examples 2-1) and 2-3) according to the present invention and Comparative Example 2-6) according to the prior art, as described above, were cut and polished, and then after etching strongly, subjected to observing of the structure thereof by SEM (scanning electron microscope), thus obtaining SEM photographs as shown in Fig. 1 to Fig. 3. It is apparent from these photographs that the structures of the forged bodies according to the present invention are clearly finer than that of the prior art.

Example 3

The procedures under the conditions of Examples 2-1) and 2-4) and Comparative Examples 2-6) and 2-7) were repeated except using a mixed powder of air-atomized, Al-20Si-5Fe-2Ni alloy powder (mean grain diameter: 50 μ m) and alumina powder with a mean grain diameter of 0.5 μ m, as a raw material powder, thus obtaining forged bodies 3-1) and 3-2) of the present invention and comparative articles 3-3) and 3-4). The properties measured in the similar manner to Example 2 are shown in Table 6. The quantity of oxygen is a quantity from which the quantity of oxygen contained in the alumina grains has been removed by calculation.

Table 6

Example	Heating Condition	Amount of Oxygen (%)	Amount of Hydrogen (ppm)	Tensile Strength (kg/mm ²)	Elongation (%)	Hardness H _R B
3-1	2-1	0.26	4	62	0.8	108
3-2	2-4	0.35	3	59	0.6	110
Comparative Example						
3-3	2-6	0.25	10	48	0.4	89
3-4	2-7	0.53	19	28	0.0	68

It is apparent from the results of Table 6 that the forged bodies of the present invention have good properties.

Example 4

The procedures under the conditions of Examples 2-1) and 2-4) and Comparative Examples 2-6) and 2-7) were repeated except using an air-atomized, Al-12Si- 5 vol % (mean grain diameter: 2 μm) SiC aluminum composite alloy powder (mean grain diameter: 50 μm) as a raw material powder, thus obtaining forged bodies 4-1) and 4-2) of the present invention and comparative articles 4-3) and 4-4). The properties measured in a similar manner to Example 2 are shown in Table 7.

Table 7

Example	Heating Condition	Amount of Oxygen (%)	Amount of Hydrogen (ppm)	Tensile Strength (kg/mm ²)	Elongation (%)	Hardness H _R B
4-1	2-1	0.26	4	62	0.8	108
4-2	2-4	0.35	3	59	0.6	110
Comparative Example						
4-3	2-6	0.25	10	48	0.4	89
4-4	2-7	0.53	19	28	0.0	68

It is apparent from the results of Table 7 that the forged bodies of the present invention have good properties.

Example 5

About 250 g of an air-atomized powder (mean grain diameter: about 50 μm) with a composition of Al-25Si-2.5Cu-1Mg was compacted at a compacting pressure of 4 tons/cm² in a diameter 100 mm x height 20 mm to give a specific electric resistance of 0.02Ω cm, heated to 500 °C under the following conditions 5-1) to 5-5), charged into a mold heated at 200°C when the heating was finished, subjected to powder forging at a compacting pressure of 8 tons/cm², and immediately cooled by immersion in water. Thereafter, the product was subjected to natural ageing for 4 days.

5-1) Induction heating in the air (32°C/sec).....Present Invention

5-2) Induction heating in the air (8.0 °C/sec).....Present Invention

5-3) Induction heating in the air (4.0 °C/sec).....Present Invention

5-4) Induction heating in the air (0.8 °C/sec).....Present Invention

5-5) Induction heating in the air (0.2 °C/sec).....Outside Present Invention

For comparison, the same compact was heated to 500 °C under the following conditions 5-6) to 5-7) using a resistance heating furnace, forged, then heated at 485 °C for 2 hours and immersed in water to effect a solution

treatment and, thereafter, subjected to natural ageing for 4 days.

5-6) Resistance furnace heating in N₂ atmosphere (maintained for 1 hour) Outside Present Invention

5-7) Resistance furnace heating in the air (maintained for 1 hour) Outside Present Invention

5-8) Resistance furnace heating in vacuum (maintained for 1 hour) moistened Outside Present Invention

The properties of the alloy powders thus obtained are shown in Table 8. From the results of Table 8, it is apparent that according to the present invention, forged bodies having well balanced properties such as hardness, tensile strength, elongation, etc. can be obtained without a deterioration in the microstructure.

Table 8

Sample No.	Degassing Conditions			
	<u>Heating Means</u>	<u>Atmosphere</u>	<u>Temperature Conditions</u>	<u>Moistened</u>
Example				
5-1	Induction Heating	In The Air	Temp. Raising at 32°C /sec	No
5-2	Induction Heating	In The Air	Temp. Raising at 8.0 °C /sec	No
5-3	Induction Heating	In The Air	Temp. Raising at 4.0 °C /sec	No
5-4	Induction Heating	In The Air	Temp. Raising at 0.8 °C /sec	No
Comparative Example				
5-5	Induction Heating	In The Air	Temp. Raising at 0.2 °C /sec	No
5-6	Resistance Furnace Heating	In N ₂ Atmosphere	Maintained For 1 Hour	No

5-7	Resistance Furnace	In The Air	Maintained For 1 Hour	No
	Heating			
5-8	Resistance Furnace	In Vacuum	Maintained For 1 Hour	No
	Heating			

Table 8 (continued)

Sample No.	Properties of Forged Body				
	Amount of Oxygen	Amount of Hydrogen	Tensile Strength	Elongation (%)	Hardness H _R B
<u>Example</u>	<u>(%)</u>	<u>(ppm)</u>	<u>(kg/mm²)</u>		
5-1	0.26	4	52	1.0	99
5-2	0.29	4	55	1.2	98
5-3	0.30	3	58	1.8	98
5-4	0.35	5	57	2.0	83
<u>Comparative Example</u>					
5-5	0.40	8	51	1.5	80
5-6	0.27	8	19	0.0	73
5-7	0.45	20	*	*	65
5-8	0.38	3	42	0.0	83

Example 6

An atomized powder with a composition of Al-25Si-2.5Cu-1Mg (by weight %) was formed in a shape of $\varnothing$ 50 mm x 50 mm t under a pressure of 4 tons/cm² by a die wall lubricating mold, heated to a forging temperature for 4 minutes by induction heating and forged in a shape of $\varnothing$ 53 mm. The forging conditions were a heating temperature of 500 °C and a forging pressure of 5 tons/cm².

After the forging, the product was subjected to a T6 heat treatment (comprising holding at 490 °C for 1.5 hours, immersing in water and subjecting to an ageing treatment at 180 °C for 6 hours) and subjected to estimation of the strength. The tensile strength was estimated in n = 2 to obtain 53 kg/mm² and 51 kg/mm².

For comparison, the same powder was subjected to powder forging by mixing with a lubricant and heating in an electric furnace, thus obtaining a tensile strength of 48 kg/mm² in n = 2.

It will be understood from these results that better results are obtained when a raw material powder is previously formed without adding a lubricant thereto and coating an inner wall of a mold.

The foregoing Examples are in relation to a rapidly solidified powder, but the method of the present invention can also be applied to the degassing of other powders with a reduction in production costs.

Example 7

A gas atomized powder (Al-7.3Ni-2.9Fe) was pressed at a compacting pressure of 4 tons/cm² to prepare three samples each having a shape of $\varnothing$ 70 mm x 25 mm t, heated to 550 °C for 2 minutes by induction heating for one sample, by radiation heating for another sample and by direct electric heating for a further sample, and then forged in $\varnothing$ 72

mm at a forging pressure of 8 tons/cm² and, after forging, water-cooled. The properties of the products at room temperature were as follows:

Induction-heated product: tensile strength 62.3 kg/mm², elongation 13.5 %, K_{IC} 28.0 kg/mm²√m

Radiation-heated product: tensile strength 60.1 kg/mm², elongation 13.0 %,

Direct electrically-heated product: tensile strength 63.4 kg/mm², elongation 13.6 %

Example 8

A gas atomized powder (Al-8.8Fe-3.7Ce) was pressed at a compacting pressure of 4 tons/cm² to prepare a sample having a shape of Ø 70 mm x 25 mm t, induction-heated to 550°C for 1.5 minutes and then forged in Ø 72 mm at a forging compacting pressure of 8 tons/cm² and after the forging, water-cooled. The properties of the product at room temperature were as follows:

Tensile strength: 65.2 kg/mm² and elongation: 16.2 %

Example 9

A gas atomized powder (Al-8Zn-2.5Mg-1Cu-1.6Co) was pressed at a compacting pressure of 4 tons/cm² to prepare a sample having a shape of Ø 70 mm x 25 mm t, induction-heated to 530 °C for 1 minute and then forged in Ø 72 mm at a forging pressure of 8 tons/cm². After forging, the temperature was lowered to 460°C and the product was reheated to 520 °C in 1 minute by induction heating, water-cooled, then subjected to natural ageing for 4 days, followed by an examination of the properties at room temperature.

Tensile strength: 70.2 kg/mm² and elongation: 12.5 %

Example 10

10 g of an air-atomized powder with a composition of Al-25Si-3Cu-1Mg was compacted at a compacting pressure of 4 tons/cm² in a shape of 10 x 18 x 30 mm and heated to 510 °C for 4 minutes by infrared radiation heating in a stagnant atmosphere, followed by forging. A metallic mold of 10.5 x 10.5 mm was used at a mold temperature of 400 °C. The forging pressure was 8 tons/cm². After forging, the product was water-cooled and then subjected to an examination of the properties without heat-treatment.

Tensile strength: 58 kg/cm², fracture elongation: 3.0 % (at room temperature)

The same green compact was heated to 510°C for 4 minutes in a nitrogen stream (7 liters/min) and then forged under the same conditions as described above.

Tensile strength: 51 kg/cm², fracture elongation: 2.1 % (at room temperature)

Example 11

20 kg of an air-atomized powder with a composition of Al-17Si-5Fe-3Cu-1Mg was subjected to CIP (compacting pressure: 2 tons/cm²) to prepare a green compact with a dimension of Ø 180 x 300 mm.

The resulting compact was subjected to:

① ambient heating in N₂ atmosphere (450 °C x 4 hours)
(490 °C x 4 hours)

② induction heating in the air (temperature raising to 460 °C in 16 minutes)
(temperature raising to 500 °C in 16 minutes)

These samples were extruded in Ø 44 (extrusion ratio: 21) by a container with a diameter of Ø 200, cooled after the extrusion and then subjected to an examination of the properties of an F material and then to a T6 treatment (470°C x 2 hours → water-cooled 175 °C x 6 hours) to examine the properties thereof.

Furthermore, after the extrusion, the sample was charged in a furnace at 485 °C for 10 minutes, water-cooled, subjected to an ageing treatment of 175 °C x 6 hours and reheated to obtain a T6 material.

③ Similarly, 250 g of the powder was compacted in Ø 80 mm (metallic mold with lubricating wall: pressure 4 tons/cm²), induction-heated in the air (temperature raising of to 520 °C in 2.5 minutes), charged in a metallic mold

with $\varnothing$ 82 and subjected to powder forging at a pressure of 8 tons/cm². After the forging, the product was immediately water-cooled to obtain an F material.

After the forging, the product was induction-heated to 485 °C for 1 minute, water-cooled and subjected to an ageing treatment of 175 °C x 6 hours to obtain a rapidly reheated T6 material.

After the forging, the product was charged in a furnace at 485 °C for 10 minutes, water-cooled and subjected to an ageing treatment of 175°C x 6 hours to obtain a reheated T6 material.

After the forging, the product was directly water-cooled and then subjected to a T6 treatment (i.e. subjected to 485 °C x 2 hours, water-cooling and a treatment of 175 °C x 6 hours) to obtain a T6 material.

The above described samples were subjected to examination of the properties, thus obtaining the results shown in Table 9.

Table 9

Sample No.	Solidified Material	Heating Temp. (°C)	H _R B	Tensile Strength (kg/cm ²)	Elongation (%)	Amount of Hydrogen (ppm)	Remarks
1	①-F material	450	78	45	0.4	18	A
2	①-T6 material		88	49	1.0	-	A
3	①-F material	490	75	41	1.4	15	A
4	①-T6 material		85	43	1.2	-	A
5	②-F material	460	89	53	1.3	14	A
6	②-Reheated T6		93	55	0.9	-	A
7	②-T6 material		92	50	1.1	-	A
8	②-F material	500	88	57	2.6	9	B
9	②-Reheated T6		91	59	2.1	-	B
10	②-T6 material		89	54	1.8	-	B
11	③-F material	520	92	61	2.8	7	B
12	③-Rapidly Reheated T6		96	62	2.2	-	B
13	③-Reheated T6		93	60	2.0	-	B
14	③-T6 material		88	57	1.9	-	B
Note) ① ~ ③ of Solidified Material correspond to the treatments ① ~ ③ in Example 11. A: Comparative Example B: Example							

The following matters are apparent from the foregoing results.

(1) In extrusions, the rapid heating method according to the present invention is also useful.

(2) The product obtained by rapid heating to a lower temperature in the extrusion exhibits a smaller elongation.

(3) The product obtained by rapid heating to a lower temperature in the extrusion exhibits a larger amount of hydrogen.

(4) When rapidly heated and extruded according to the present invention, the reheated T6 gives better properties than the ordinary T6.

(5) Even the F material gives sufficient properties when rapidly heated and powder forged according to the present invention.

(6) When rapidly heated and powder forged according to the present invention, the reheated T6 material gives better properties than the T6 material and the rapidly reheated T6 material gives better properties than the reheated

T6 material.

(7) In the articles of the present invention, both the tensile strength and fracture elongation can simultaneously be improved more than the prior art materials.

5 Example 12

The above described Sample Nos. 2 and 11 were subjected to examination of the tensile strength and elongation at 300 °C :

10 Sample No. 2 Material.....22 kg/mm², 3.5 % elongation Comparison
Sample No. 11 Material.....28 kg/mm², 5.6 % elongation Present Invention

Accordingly, it is apparent that the article of the present invention also has an excellent heat resistance.

15 Example 13

250 g of a rotary disk atomized powder with a composition of Al-8Fe-4Mo was compacted in Ø 80 mm (metallic mold with lubricating wall surface) and heated under the following conditions.

20 induction heating in the air (temperature raising to 510 °C in 1.0 minute)
(temperature raising to 650 °C in 1.0 minute)

The product was charged in a metallic mold of Ø 82 mm and subjected to powder-forging at a pressure of 8 tons/cm². After forging, the product was cooled to examine the properties. The results are shown in Table 10.

Table 10

Heating Temperature (°C)	Tensile Strength (kg/mm ²)	Fracture Elongation (%)
510	67	4.5
650	66	12.3

35 As described above, in an aluminum alloy with a high melting point, heating at a temperature exceeding 600 °C sometimes gives good results.

Since according to the present invention, sufficient degassing can be carried out in a simpler and more economical manner than in the prior art, the tensile strength, elongation and fracture toughness values can be improved without carrying out heating in an inert atmosphere, degassing in vacuum and plastic deformation after consolidation.

40

Claims

1. A method of degassing an aluminium alloy powder, comprising the steps of preforming an aluminium powder, aluminium alloy powder or aluminium composite alloy powder or mixed powders thereof, optionally containing non-metallic grains, to give a specific electric resistance of at most 0.2Ωcm, wherein the preform is subjected directly to induction heating in a stagnant atmosphere of air at atmospheric pressure, raising the temperature to in the range of from 400 to 600°C at a temperature gradient of at least 0.8°C/sec when a temperature of 300°C has been reached, whereby heat-decomposable volatile components are removed to obtain a hydrogen content of at most 10 ppm.
2. A method as claimed in claim 1, wherein the preform is cooled in an inert gas atmosphere after degassing by induction heating.
3. A method of consolidating a rapidly solidified aluminium alloy powder, comprising the steps of preforming an aluminium powder, aluminium alloy powder or aluminium composite alloy powder or mixed powders thereof, optionally containing non-metallic grains, to give a specific electric resistance of at most 0.2Ωcm, wherein the preform is subjected directly to induction heating in a stagnant atmosphere of air at atmospheric pressure, raising the temperature to in the range of from 400°C to the melting point at a temperature-raising gradient of at least 0.8°C/sec when a

temperature of 300°C has been reached, whereby heat-decomposable volatile components are removed to obtain a hydrogen content of at most 10 ppm, and then directly subjecting the thus obtained product to hot working and consolidating the product.

- 5 4. A method as claimed in claim 3, wherein the hot working is powder forging.
5. A method as claimed in claim 3 or claim 4, wherein immediately after forging, the product is quenched at a rate of at least 10°C/sec or reheated at a temperature of at most the forging temperature to at least (the forging temperature - 50°C) without cooling to room temperature.
- 10 6. A method as claimed in any one of claims 3 to 5 wherein the preforming of the powder is carried out by coating an inner wall of a forming metallic mold with a wetting agent without adding an organic wetting agent to the powder.

Patentansprüche

- 15 1. Verfahren zum Entgasen eines Aluminiumlegierungspulvers bei dem man:

ein Aluminiumpulver, Aluminiumlegierungspulver oder Aluminiumverbundlegierungspulver oder deren Pulvergemisch, enthaltend wahlweise nichtmetallische Körnchen, vorformt, um einen spezifischen elektrischen Widerstand von höchstens 0,2 Ω cm zu erhalten, wobei der Vorformling in einer stagnierenden Luftatmosphäre bei atmosphärischem Druck direkt induktionserhitzt wird,

die Temperatur auf einen Wert im Bereich von 400 bis 600°C bei einem Temperaturgradienten von mindestens 0,8°C/sec erhöht, wenn eine Temperatur von 300°C erreicht ist, wodurch die durch Hitze zersetzbaren flüchtigen Komponenten entfernt werden, um einen Wasserstoffgehalt von höchstens 10 ppm zu erhalten.
2. Verfahren nach Anspruch 1, bei dem die Vorform nach dem Entgasen durch Induktionserhitzen in einer Inertgasatmosphäre gekühlt wird.
- 30 3. Verfahren zum Verfestigen eines rasch erstarrten Aluminiumlegierungspulvers bei dem man:

ein Aluminiumpulver, Aluminiumlegierungspulver oder Aluminiumverbundlegierungspulver oder deren Pulvergemisch, wahlweise enthaltend nichtmetallische Körnchen, vorformt, um einen spezifischen elektrischen Widerstand von höchstens 0,2 Ω cm zu erhalten, wobei der Vorformling in einer stagnierenden Luftatmosphäre bei atmosphärischem Druck direkt induktionserhitzt wird,

die Temperatur auf einen Wert im Bereich von 400°C bis zum Schmelzpunkt bei einem Gradienten der Temperaturerhöhung von mindestens 0,8°C/sec erhöht, wenn eine Temperatur von 300°C erreicht ist, wodurch die durch Hitze zersetzbaren flüchtigen Komponenten entfernt werden, um einen Wasserstoffgehalt von höchstens 10 ppm zu erhalten,

und dann das so erhaltene Produkt direkt heißbearbeitet und verfestigt.
4. Verfahren nach Anspruch 3, bei dem die Heißbearbeitung durch Pulverschmieden erfolgt.
- 45 5. Verfahren nach Anspruch 3 oder 4, bei dem das Produkt unmittelbar nach dem Schmieden mit einer Geschwindigkeit von mindestens 10°C/sec abgeschreckt oder ohne Abkühlen auf Raumtemperatur bei einer Temperatur von höchstens der Schmiedetemperatur bis zu mindestens der Schmiedetemperatur minus 50°C erneut erhitzt wird.
- 50 6. Verfahren nach einem der Ansprüche 3 bis 5, bei dem das Vorformen des Pulvers durch Beschichten einer Innenwand einer Metallform mit einem Benetzungsmittel ohne Zusatz eines organischen Benetzungsmittels zum Pulver erfolgt.

Revendications

- 55 1. Procédé de dégazage d'une poudre d'alliage d'aluminium, comprenant les étapes consistant à préformer une poudre d'aluminium, une poudre d'alliage d'aluminium ou une poudre d'alliage composite d'aluminium, ou des poudres mixtes de celles-ci, contenant éventuellement des grains non métalliques, pour conférer une résistance électrique

spécifique d'au plus 0,2 Ωcm , dans lequel on soumet l'ébauche directement à un chauffage par induction dans une atmosphère stagnante d'air, à la pression atmosphérique, augmenter la température jusqu'à 400 à 600°C avec un gradient de température d'au moins 0,8°C/s quand on atteint la température de 300°C, moyennant quoi on élimine les composants volatils décomposables sous l'effet de la chaleur pour obtenir une teneur en hydrogène d'au plus 10 ppm.

2. Procédé selon la revendication 1, dans lequel on refroidit l'ébauche dans une atmosphère de gaz inerte après le dégazage par chauffage par induction.

3. Procédé de consolidation d'une poudre d'alliage d'aluminium rapidement solidifiée, comprenant les étapes consistant à préformer une poudre d'aluminium, une poudre d'alliage d'aluminium ou une poudre d'alliage composite d'aluminium, ou des poudres mixtes de celles-ci, contenant éventuellement des grains non métalliques, pour conférer une résistance électrique spécifique d'au plus 0,2 Ωcm , dans lequel on soumet l'ébauche directement à un chauffage par induction dans une atmosphère stagnante d'air, à la pression atmosphérique, augmenter la température jusqu'à 400°C et le point de fusion, avec un gradient d'augmentation de la température d'au moins 0,8°C/s quand on atteint la température de 300°C, moyennant quoi on élimine les composants volatils décomposables sous l'effet de la chaleur pour obtenir une teneur en hydrogène d'au plus 10 ppm, et ensuite, on soumet le produit ainsi obtenu directement à un façonnage à chaud et on consolide le produit.

4. Procédé selon la revendication 3, dans lequel le façonnage à chaud est un forgeage de la poudre.

5. Procédé selon la revendication 3 ou la revendication 4, dans lequel, immédiatement après le forgeage, on refroidit brusquement le produit à une vitesse d'au moins 10°C/s, ou on le réchauffe à une température valant au plus la température de forgeage, jusqu'à au moins (la température de forgeage - 50 °C) sans refroidissement à la température ambiante.

6. Procédé selon l'une quelconque des revendications 3 à 5, dans lequel on effectue le préformage de la poudre en revêtant la paroi interne d'un moule métallique de façonnage avec un agent mouillant sans ajouter d'agent mouillant organique à la poudre.

FIG. 1

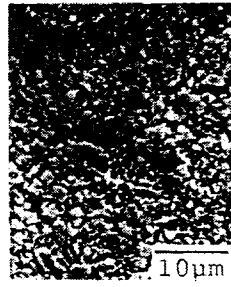


FIG. 2



FIG. 3

