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(54) Titre : PROCEDE DE FABRICATION PAR METALLURGIE DES POUDRES DE PIECES EN TITANE OU EN ALLIAGES DE TITANE

(54) Title: METHOD FOR THE POWDER-METALLURGICAL PRODUCTION OF COMPONENTS FROM TITANIUM OR TITANIUM ALLOYS

(57) Abrégé/Abstract:

A method for the powder-metallurgical production of a component from titanium or a titanium alloy is disclosed. In this method, following the customary procedure, first a green part is formed by using metal powder formed from titanium or the titanium alloy and is densified and compacted in a subsequent sintering step. Metal powder of titanium or the titanium alloy with an average grain size of <25 µm is used for producing the green part and the sintering step is carried out at a sintering temperature of up to a maximum of 1100°C. for a sintering at a sintering duration of ≤5 hours in an atmosphere that is under a reduced pressure in comparison with normal pressure. These measures achieve the effect that the grain structure of the material obtained, and consequently also the material properties, can be selectively influenced. For example, by modifying the sintering conditions of the present method it is possible to create a primarily globular grain structure with high ductility.

ABSTRACT

A method for the powder-metallurgical production of a component from titanium or a titanium alloy is disclosed. In this method, following the customary procedure, first a green part is formed by using metal powder formed from titanium or the titanium alloy and is densified and compacted in a subsequent sintering step. Metal powder of titanium or the titanium alloy with an average grain size of <25 µm is used for producing the green part and the sintering step is carried out at a sintering temperature of up to a maximum of 1100°C. for a sintering at a sintering duration of ≤5 hours in an atmosphere that is under a reduced pressure in comparison with normal pressure. These measures achieve the effect that the grain structure of the material obtained, and consequently also the material properties, can be selectively influenced. For example, by modifying the sintering conditions of the present method it is possible to create a primarily globular grain structure with high ductility.

METHOD FOR THE POWDER-METALLURGICAL PRODUCTION OF COMPONENTS FROM TITANIUM OR TITANIUM ALLOYS

The present invention relates to a method for the powder-metallurgical production of a component from titanium or a titanium alloy, wherein first, using metal powder produced from titanium or the titanium alloy, a green part is formed and this is densified and compacted in a subsequent sintering step.

BACKGROUND

Various powder-metallurgical methods for producing true-to-size titanium components (here and in the following, "components from titanium" will be used here and in the following as a simplified term for components made from titanium (pure titanium) or one or more titanium alloys) are known, where in all methods, first a green part is produced, and this is densified and compacted in a subsequent sintering step. The green part can be produced in various ways, especially using additive production methods, metal powder injection molding, extrusion methods and non-pressurized powder-metallurgical production methods.

Because of the excellent properties of the material, titanium, plus the efficient and economical production method, the powder-metallurgical production of titanium is becoming increasingly more widely used. The good biocompatibility and the high specific strength of the material, titanium, play an important role especially in applications in medical engineering and air and space technology. The economically most significant alloy with sales figures accounting for more than 50% of the total titanium market 1 Ti6A14V.

As a rule, the following steps must be executed to produce a powder-metallurgically processed titanium component:

a) forming
b) debinding
c) sintering

The objective of forming is to bring the titanium powder particles into the tightest possible packing in a form close to the final contour. In this step, depending on the method employed, additives are used which must be removed in one or more subsequent debinding step(s). In the subsequent process step, frequently also the final one, sintering, the powder particles are consolidated by material transport.

Because of the high reactivity of titanium, all processing steps must take place under special process conditions. In patent EP 1 119 429 B1 [1], Gerling et al. describe necessary process conditions for sintering titanium. The combined implementation of debinding and sintering in a combined furnace design is described by Blüm in EP 1 496 325 A2 [2].

Titanium has two crystal modifications. The hexagonal α phase, which with pure titanium and normal pressure is present up to a temperature of 882.5°C, and the cubic space-centered β phase, which with pure titanium and normal pressure occurs above the aforementioned temperature. The presence of the different phases at room temperature is used to classify titanium alloys into α-Ti, (α+β)-Ti and β-Ti alloys. Ti6A14V, for example, is an (α+β)-alloy, i.e., both phases are present in the grain structure at room temperature. To produce components with a generally desired density >97% in the process of sintering titanium and titanium-alloy components, sintering temperatures of about 1100- 1 400°C at a sintering duration of about 2-5 h are needed. For pure titanium and Ti6Al4V this means that the materials are processed in the β-phase region, which leads to a massive β-grain growth.

In EP 1 119 429 B1 [1], Gerling et al. describe that the grain structure that becomes established has a β-grain growth of about 150 μm. Here, the nomenclature according to Sieniawski et al. [3], shown in Fig. 1, is used to describe the sizes of the various structures in the lamellar (α+β) alloys. The following designations are used:

D: grain size of the primary β phase
d: the size of a parallel α-lamella colony
t: the width of an α-lamella

In contrast to reforming processes, forming takes place as the first step on the powder metallurgy route. In the next process step, sintering, the compacted titanium alloy, previously brought into shape, is produced. In contrast to standard processing approaches, because of the reverse sequence of the process steps (1. forming, 2. material consolidation) in the powder metallurgy approach, the possibility of refining or optimizing the grain structure of the metal and thus its material properties by thermal/mechanical working before the forming step does not exist. For powder-metallurgical methods for producing components from titanium and/or titanium alloys, precisely the process-determined inverse sequence, combined with the very limited influence on the grain structure that develops during the familiar sintering process, is a limiting factor. As an example: the grain structure of a Ti6A14V sample produced in the standard manner from titanium powders commonly used in the prior art (with powder grain sizes $\leq$45 μm) and sintered under sintering conditions typically used in the prior art is shown in Fig. 2. Here it is possible to recognize the typical lamellar mixed grain structure for titanium components produced in the known way by powder metallurgy and sintered, made up of α phases and β phases, the (α+β) grain structure, with a mean primary β-phase grain size (D) of about 190 μm.

The production of powder-metallurgically processed titanium and titanium alloys with small grain sizes is described in US 4,601,874 [4] by Marty et al. Through the targeted admixtures of S, P, B, As, Se, Te, Y and lanthanoids, during the consolidation process a material is produced with grain sizes smaller by two orders of magnitude than the titanium powder particles used. The drawback of this approach is that the use of titanium and titanium alloys is widespread precisely in strictly regulated market segments. For these application purposes, the chemical compositions of the material and its mechanical properties are regulated by standards. For example, the material compositions and mechanical properties of Ti6A14V and pure titanium are regulated in the standards ASTM F2885 and ASTM F2889 respectively.

An additional procedure for producing fine-grained titanium and such titanium alloys by powder metallurgy is described in WO 2012/148471 A1. Here a green part made from titanium (alloy) powder with grain sizes of less than 325 mesh (less than 44 μm) is produced and then subjected to a multistep process of compaction and forming. In a first step the green part is sintered in a hydrogen atmosphere at temperatures of 1100 to 1500°C; in the embodiments, the processing temperature is always given as 1200°C. In this process titanium material in the β-phase forms. In a subsequent step of controlled cooling, a phase transformation occurs, in which restructuring occurs in the p-grains, resulting in a phase mixture of fine α-grains, β-grains and δ-phases. Then in a final step, the hydrogen must be expelled from the component obtained, which is done by applying a vacuum. With this procedure especially the use of hydrogen is especially problematic, since this gas can only be expelled from the component with great effort and often not completely expelled. Negative effects on the material properties and the stability of the material have been blamed on hydrogen remaining in the grain structure of the material. Outgassing from residual hydrogen from the finished component in various applications is also anything but desirable.

SUMMARY

One goal generally pursued with the invention is that of creating the possibility, in the case of powder-metallurgically produced and sintered titanium components, of manipulating the grain structure and optimizing the material properties. In particular the intention was to make it possible to adapt the material properties to the specific use case directly in the sintering process and/or to create, during the sintering process, an optimal starting point for further thermal treatment steps after sintering. For example, it should be possible, by modifying the sintering conditions, to create a primarily globular grain structure with high ductility.

To solve the problem, a process is suggested that comprises a method for the powder-metallurgical production of a component from titanium or a titanium alloy, wherein first, using metal powder from titanium or the titanium alloy, a green part is formed and this is densified and compacted in a subsequent sintering step, characterized in that for producing the green part, metal powder from titanium or titanium alloy with a mean grain size of <25 μm, measured using laser diffraction according to ASTM B822-10 is used and that the sintering step is performed at a sintering temperature up to a maximum of 1100° C., at a sintering duration of <5 h in an atmosphere under a reduced pressure in comparison with normal pressure. Advantageous embodiments of the invention are that the maximum grain size of the metal powder from titanium or the titanium alloy is <30 μm; that the sintering step is performed under a vacuum with a pressure of 10−3 mbar, especially at a pressure of 10−5 mbar; and that the sintering step is performed in an inert gas atmosphere, especially an argon atmosphere, at a pressure of <300 mbar. For producing the green part, metal powder from titanium or the titanium alloy with a mean grain size of <20 μm, in particular of <10 μm, preferably of <5 μm, is used. The sintering duration is 3.5 h, in particular of s 3 h, preferably of ≤2.5 h. Furthermore, the sintering duration is at least 1 h, preferably at least ≤2 h. The sintering temperature is up to a maximum of 1050° C., preferably up to a maximum of temperature up to a maximum of 1000° C., especially up to a maximum of 950° C. and the sintering temperature amounts to at least 860° C. The method is further characterized in that in the sintering step, the sintering temperature is adjusted in the range below a β-transition temperature of the titanium or titanium alloy material. The component after the sintering step has a material density of >97%, in particular >98%, preferably 99%. In the sintering step, a sintering temperature of below 950° C. is selected and that to achieve a material density in the component of >97%, after the sintering step this is exposed to an additional step with pressure and optionally a temperature, e.g., a step of cold isostatic pressing (CIP) and/or hot isostatic pressing (HIP). The component, following the sintering step, is subjected to a thermal aftertreatment that is conducted in the form of one or more of the following treatment procedures: hot isostatic pressing (HIP), quench, uniform rapid quench (URQ). An additional aspect to solving this problem lies in a titanium component that exhibits the properties that it has a globular α-structure with a grain size of <30 μm; that it has a grain structure with globular α-structure with mean grain size of <30 μm and lamellar (α+β) grain structure with a mean primary β-phase grain size of <90 μm; and/or that it has a lamellar (α+β) grain structure with a mean primary to β-phase grain size of <120 μm.

BRIEF DESCRIPTION OF THE FIGURES

Fig. 1 a representation of a lamellar $(\alpha+\beta)$ grain structure of a Ti6Al4V sample with description of the gran structure fractions according to Sieniawski et al. [3];

Fig. 2 an enlarged photomicrograph of a standard sintered Ti6A14V sample, produced by powder-metallurgically using powder particles <45 µm and standard-sintered and confirms a lame liar $(\alpha+\beta)$ grain structure for this;

Fig. 3 a schematic representation of the effect of reducing the grain size by half (using the example of spherical particles) on the number of particles required to fill a defined volume;

Fig. 4 a schematic representation of the reduction in size of the hollow space between adjacent particles due to reducing the grain size by half (using the example of spherical particles);

Fig. 5 an enlarged polished micrograph section of a powder-metallurgically produced and sintered Ti6Al4V sample made from powder particles <20 µm, confirming the formation of a distinct globular a-structure; and

Fig. 6 an enlarged polished micrograph section of a powder-metallurgically produced and sintered Ti6A14V sample made from powder particles <20 µm, confirming the formation of a bimodal grain structure with a globular a-structure and distinct lamellar $(\alpha+\beta)$ grain structure.

DETAILED DESCRIPTION

An essential prerequisite for implementing the process according to the invention and creating the possibility of influencing the material properties in the sintering process is the use of metal powder, produced from titanium or a titanium alloy, with a mean grain size of <25 µm, so-called fine powder. In such fine powder used for the process according to the invention, the maximum grain size may in particular be <30 µm. The maximum grain size is specified as a limit value by the manufacturers of such fine powders. At the same time, a small fraction of particles in such batches can always have grain sizes above this limit. Such a fraction, as a rule, is generally specified as a maximum of 1 to a maximum of 5 wt.-%.

The mean grain size may advantageously even be lower, especially <20 µm, advantageously <10 µm and particularly preferably even <5 µm. The smaller the grain size of the metal powder is, the more readily high final densities can be achieved even at sintering temperatures markedly reduced compared to the relatively high sintering temperatures previously used.

The measurement of the grain sizes essential for the invention and the distribution thereof is performed by grain size testing using laser diffraction according to ASTM B822-10 (published 2010), valid at the time of this application. The grain size distribution is determined by wt.-% and according to D10/D50/D90, wherein D50 is the mean grain size. Specifically, the grain sizes given here in comparison tests were measured using the COULTER® LS grain size analyzer made by Beckman Coulter and evaluated using the Fraunhofer theory according to ASTM B822-10.

For spherical particles, the grain size in the sense of the invention is specified as the particle diameter. For nonspherical particles, the grain size corresponds to the projected maximum particle dimension.

As a result of the reduced grain size, the surface area in the nonconsolidated component available for the sintering process increases, and thus so does the stored surface energy. Since the reduction of this energy is the driving force in the sintering process, the sintering process can then take place using little thermal energy.

An additional advantage of using fine powders of the sizes indicated above for forming the green part is that more powder particles can be introduced per unit volume. In addition to the enlarged surface, this leads to a higher number of contact points per unit volume, as shown in Fig. 3. There, in a schematic representation, the effect of reducing the grain size by half (using the example of spherical particles) on the particle count to fill a defined volume is shown.

The contact points of the particles in turn are the starting point and a necessary condition for the sintering process, which is driven by diffusion processes. The increased number of such contact points per unit volume therefore improves the starting conditions for the sintering process.

Through the use according to the invention of fine powders with mean grain sizes <25 μm, when considering the ideal packing density in addition to the aforementioned advantages, the result also occurs that the volume enclosed by the powder particles, as shown in an idealized representation in Fig. 4, is decreased. In Fig. 4, in a schematic representation the decrease in size of the hollow space between adjacent particles is illustrated by reducing the grain size by half (using the example of spherical particles). Since this hollow space must be closed to achieve the - high - material density desired for the component following the sintering process must be closed by material transport during the sintering process, a smaller volume to be covered is an additional decisive reason for an improvement in the process result.

The sintering step typically takes place in a reduced-pressure atmosphere. This can be a vacuum with a pressure of $\leq 10^{-3}$ mbar, especially $\leq 10^{-5}$ mbar. However, it may also be a reduced-pressure inert gas atmosphere with a pressure of, e.g., ≤ 300 mbar. Argon gas in particular is considered as the inert gas here.

The sintering temperatures according to the invention are below 1100°C. They can in particular be a maximum of 1050°C, a maximum of 1000°C, and even a maximum of only 950°C. Preferably, however, to achieve a good sintering result, the sintering temperature selected advantageously should not be below 860°C. The sintering temperature may be kept uniform. In particular, however, it is also possible and falls within the meaning of the invention to vary the temperature during the sintering processs. The sintering temperature is defined here as the temperature that the workpiece to be sintered has undergone. Depending on the sintering unit, in the unit control, an adapted process temperature is to be selected, which distinguishes the process temperature measured at a distance remote from the workpiece from the sintering temperature undergone by the workpiece.

The duration of sintering may especially be ≤ 3.5 h, often also ≤ 3 h or even ≤ 2.5 h. However, it was found that as a rule, for achieving good results, the sintering time should amount to at least 1 hour, preferably at least 2 hours.

After the sintering step, components from titanium or titanium alloys produced with the method of the invention generally have a final density of > 97%. However, final densities above 98% may also be reached, even ≥99%.

To achieve a globular grain structure, the titanium components are sintered at less than the β-transition temperature (e.g., at a temperature 30°C below the β-transition temperature.

For example, in initial experiments at a sintering temperature of 950°C, which is below the β-transition temperature, and with a sintering duration of less than three hours, components with a final density of >97% were produced. These had a globular grain structure with an α-grain size on average of 10.1 μm and a max. size of 29 μm. The grain structure of this material is shown in Fig. 5. These grain sizes fall in the order of magnitude of the powder particles used.

According to the literature, the β-transition temperature of Ti6A14V falls in the range of 985°C to 1015°C [3; 5]. This relatively wide range given in the literature is attributable, on one hand, to the distribution of the alloying elements in the titanium alloys. On the other hand, the ambient pressure is an additional influential factor. For example, Huang et al. describe that as a result of elevated process pressures (1500 bar), a reduction of the α-transition temperature can be observed in the alloy Ti4A18Nb [6].

The inventors now believe that depending on the process conditions, shifts in the β-transition temperature of only a maximum of 20°C will be observable due to pressure variations.

For creating a bimodal structure, the components were sintered close to the β-transition temperature, but still below this.

For example, in order also to produce the lamellar grain structure with reduced primary β-phase grain size of the Ti6A14V alloy, which is also advantageous for many use cases, initially samples were produced in which the titanium components were sintered at a sintering temperature of 1000°C (Fig. 6). As shown by studies of the samples obtained with respect to the grain structure formed, this sintering temperature was still below the β-transition temperature, although only slightly. The bimodal grain structure fanned is composed of globular α-structure and small portions of lamellar (α+β) structures, wherein the mean β-grain size is 81 μm.

The density measurement was performed according to the specifications of ASTM 8962 and ASTM B311. The grain size determination was performed according to the provisions of ASTM E112.

For creating a lamellar grain structure with the smallest possible grain size of the primary β-phase grains, the components were largely sintered, i.e., for the greatest part of the time, below the β-transition temperature, but with a minimal hold time that remained below 30 min, preferably below 20 min, especially below 10 min, and also above the β-transition temperature in phases, so that the β-phase is entirely present, in order thus to create the lamellar grain structure, but also the primary β-phase grain does not exceed the size range given in claim 16. The sintering above the β-transition temperature always took place at a temperature in excess of 1015°C. This temperature was always kept below 1080° C, but advantageously was below 1040°C and especially $\leq$1020°c was selected.

The possibilities mentioned above for influencing the phase composition in the sintered material by systematic adjustment of the sintering conditions at sintering temperatures below 1100°C, especially primarily below the β-transition temperature, present a particular advantage of the process according to the invention. The prerequisite for this variability is that sufficiently compact titanium components can be produced below the β-transition temperature, which is possible, as the inventors recognized, based on the use of the fine powder, essential to the invention, with grain sizes below <30 μm.

Thus it has been shown that according to the method of the invention, powder-metallurgical moldings from titanium and titanium alloys can be sintered at sintering temperatures below the usual mark of beyond 1100°C, generally 1200°C or more, advantageously below the β-transition temperature, and thereby components with good structural and other material properties can be obtained. It was possible to show that at distinctly lower set sintering temperatures compared with the sintering temperatures customary in the prior art - unexpectedly - components with high final densities of >97% can be obtained. In particular it was shown that the method according to the invention makes it possible to vary the grain structure of the titanium component in the sintering process and drastically reduce the grain size, which makes it possible to optimize the mechanical properties of the components, e.g., the tensile strength, ductility and fatigue strength.

For example, within the scope of the invention, a particularly low temperature may also be selected for sintering, e.g., a temperature below 950°C, can be selected, and if the desired material density in the finished component (generally >97%) is not yet achieved in such a sintering step, further compaction of the material can be performed in the subsequently performed pressing step, in which the material is subjected to pressure and optionally a temperature, especially by cold isostatic pressing (CIP) or hot isostatic pressing (HIP). Here, for example, the material density after sintering may be at <97%, and it may be compacted to >97% by the pressing step after sintering.

In addition, following the sintering step, components produced according to the method of the invention may be subjected to additional thermal aftertreatments to further modify the properties of the materials. Such additional thermal aftertreatments can, for example, be one or more of the following methods: hot isostatic pressing (HIP), quench, uniform rapid quench (URQ).

The lower sintering temperature compared to the sintering temperatures from the prior art also result in additional environmental/financial and process technology advantages. On one hand, less thermal energy is required in the sintering process, leading to lower costs but also to shorter processing times. On the other hand, the method in accordance with the invention performed with reduced sintering temperature also allows the use of how-wall furnace designs which are once again more economical than furnaces designed for process temperatures >1100°C, where cold-wall furnaces are typically used.

The selective combination of fine powders with mean grain size <25 μm, preferably also with maximum grain sizes <30 μm, and reduced sintering temperatures compared with the prior art, to be classified as low, allows the unrivaled manipulation of the grain structure and thus of the material properties.

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Claims

1. A method for the powder-metallurgical production of a component from titanium or a titanium alloy, wherein first, using metal powder from the titanium or the titanium alloy, a green part is formed and subsequently densified and compacted in a sintering step, wherein for producing the green part, metal powder from the titanium or the titanium alloy with a mean grain size of <25 µm, measured using laser diffraction according to ASTM B822-10 is used and the sintering step is performed at a sintering temperature up to a maximum of 1100°C, at a sintering duration of ≤ 5 h in an atmosphere under a reduced pressure in comparison with normal pressure.
2. The method according to claim 1, wherein the maximum grain size of the metal powder from the titanium or the titanium alloy is <30 µm.
3. The method according to claim 1 or 2, wherein the sintering step is performed under a vacuum with a pressure of $\leq 10^{-3}$ mbar.
4. The method according to claim 3, wherein the sintering step is performed at a pressure of $\leq 10^{-5}$ mbar.
5. The method according to either of claims 1 or 2, wherein the sintering step is performed in an inert gas atmosphere at a pressure of ≤ 300 mbar.
6. The method according to claim 5, wherein the sintering step is in an argon atmosphere.
7. The method according to any one of claims 1 to 6, wherein for producing the green part, the metal powder from the titanium or the titanium alloy with a mean grain size of <20 µm is used.
8. The method according to claim 7, wherein for producing the green part, the metal powder from the titanium or the titanium alloy with a mean grain size of <10 µm is used.
9. The method according to claim 8, wherein for producing the green part, the metal powder from the titanium or the titanium alloy with a mean grain size of <5 µm is used.
10. The method according to any one of claims 1 to 9, wherein the sintering duration is ≤ 3.5 h.
11. The method according to claim 10, wherein the sintering duration is ≤ 3 h.
12. The method according to claim 11, wherein the sintering duration is ≤ 2.5 h.
13. The method according to any one of claims 1 to 12, wherein the sintering duration is at least 1 h.

14. The method according to claim 13, wherein the sintering duration is at least ≤ 2 h.

15. The method according to any one of claims 1 to 14, wherein the sintering temperature is up to a maximum of 1050°C.

16. The method according to claim 15, wherein the sintering temperature is up to a maximum of temperature up to a maximum of 1000°C.

17. The method according to claim 16, wherein the sintering temperature is up to a maximum of temperature up to a maximum of 950°C.

18 The method according to any one of claims 1 to 17, wherein the sintering temperature is at least 860°C.

19. The method according to any one of claims 1 to 18, wherein, in the sintering step, the sintering temperature is adjusted in a range below a β-transition temperature of the titanium or the titanium alloy.

20. The method according to any one of claims 1 to 19, wherein the component after the sintering step has a material density of >97%.

21. The method according to claim 20, wherein the component after the sintering step has a material density of >98%.

22. The method according to claim 21, wherein the component after the sintering step has a material density of ≥ 99%.

23. The method according to any one of claims 1 to 22, wherein in the sintering step a sintering temperature of below 950°C is used and wherein, to achieve a material density in the component of >97%, after the sintering step a pressing step is performed in which the component is subjected to pressure or pressure and a temperature.

24. The method according to claim 23, wherein the pressing step comprises cold isostatic pressing and/or hot isostatic pressing.

25. The method according to any one of claims 1 to 24, wherein the component, following the sintering step, is subjected to a thermal aftertreatment.

26. The method according to claim 25, wherein the thermal aftertreatment is conducted in the form of one or more treatment procedures selected from the group consisting of: hot isostatic pressing (HIP), quench, and uniform rapid quench (URQ).

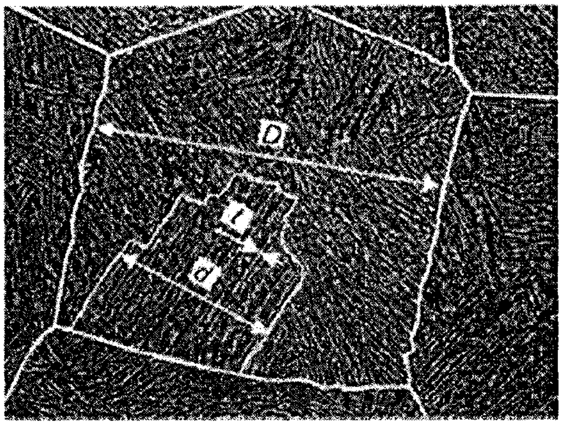

Fig. 1 **PRIOR ART**

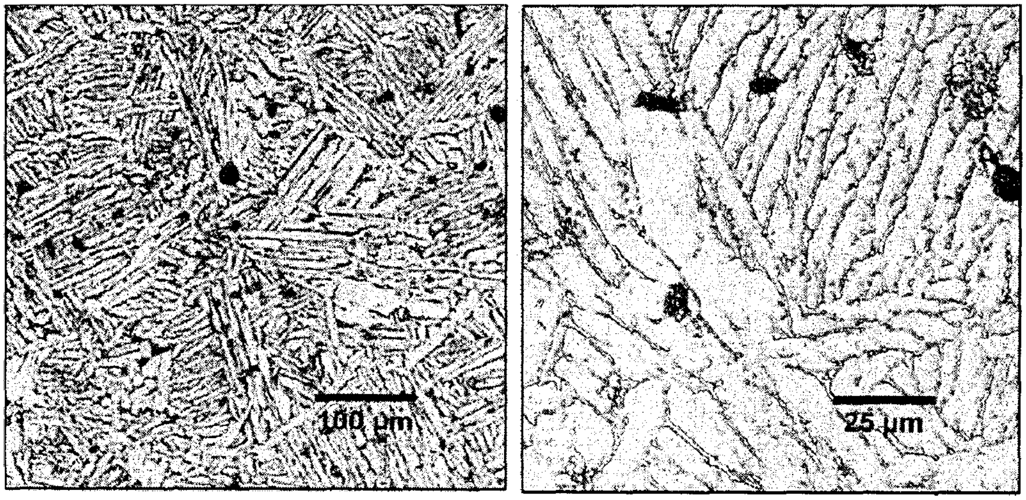

Fig. 2

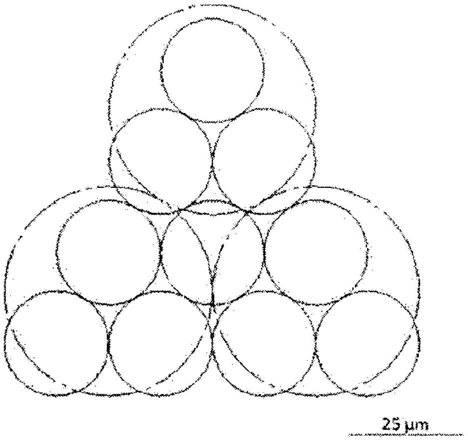

Fig. 3

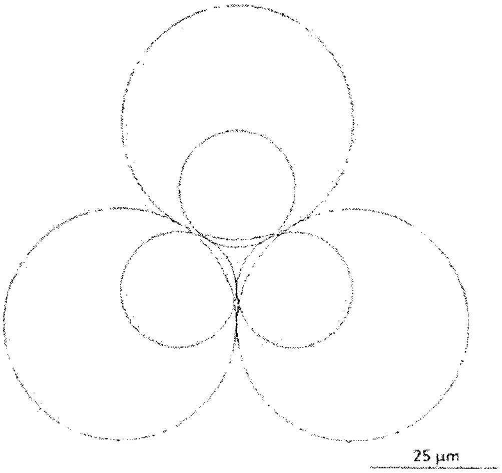

Fig. 4

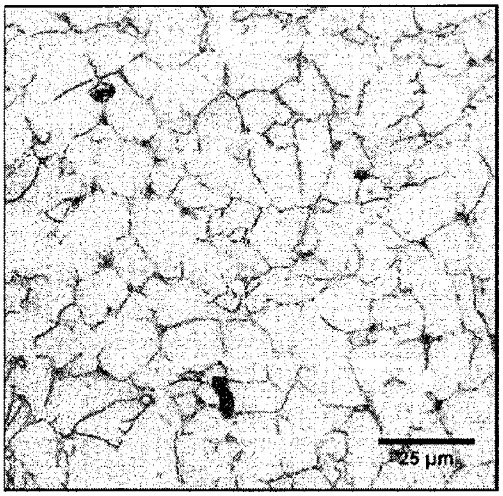

Fig. 5

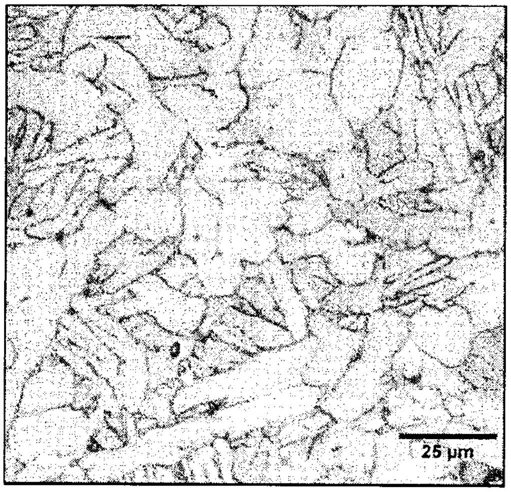

Fig. 6