



(22) Date de dépôt/Filing Date: 2004/05/14

(41) Mise à la disp. pub./Open to Public Insp.: 2004/11/16

(45) Date de délivrance/Issue Date: 2010/02/09

(30) Priorités/Priorities: 2003/05/16 (US60/471,463);  
2004/05/12 (US10/843,916)

(51) Cl.Int./Int.Cl. *A61C 8/00* (2006.01),  
*A61F 2/02* (2006.01), *A61F 2/28* (2006.01),  
*A61K 6/04* (2006.01), *A61L 27/06* (2006.01),  
*A61L 27/50* (2006.01), *C22C 14/00* (2006.01)

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(54) Titre : PROCÉDE DE TRAITEMENT DE SURFACE D'IMPLANTS A BASE D'ALLIAGE DE TITANE

(54) Title: SURFACE TREATMENT PROCESS FOR IMPLANTS MADE OF TITANIUM ALLOY

(57) **Abrégé/Abstract:**

A titanium 6 Al/4V alloy is provided with a surface topography that is similar to the Osseotite® surface produced on commercially pure titanium. Native oxide is removed from the Ti 6Al/4V alloy, followed by contacting the metal at ambient temperature with an aqueous hydrochloric acid solution containing a relatively small amount of hydrofluoric acid.

**ABSTRACT**

A titanium 6 Al/4V alloy is provided with a surface topography that is similar to the Osseotite® surface produced on commercially pure titanium. Native oxide is removed from the Ti 6Al/4V alloy, followed by contacting the metal at ambient  
s temperature with an aqueous hydrochloric acid solution containing a relatively small amount of hydrofluoric acid.

# **SURFACE TREATMENT PROCESS FOR IMPLANTS MADE OF TITANIUM ALLOY**

## **FIELD OF THE INVENTION**

This invention relates generally to the surface of metal implants, such as those placed in the human body.

## **BACKGROUND OF THE INVENTION**

5 This invention principally relates to the surface of titanium alloy dental implants, although it has application to other types of implants made of titanium alloys. More specifically, the invention relates to roughened surfaces provided on dental implants to improve the osseointegration of the implant surface with the bone,  
10 thereby shortening the time between initial insertion of the implant and the installation of a prosthetic tooth.

Various techniques have been suggested for roughening implants, each producing a unique surface. One approach has been to apply materials to the surface of the implant, for example hydroxyapatite, a material that is considered to improve  
15 the bonding of the implant to bone because the hydroxyapatite is chemically related to bone. In a related approach, titanium particles have been sprayed onto a titanium implant to roughen the surface. Anodization to add titanium oxides to the surface has also been proposed. Roughening also can be done by removing some of the surface. Grit blasting with fine particles has been proposed to create dents and to abrade away  
20 some of the surface. Another method is the use of acid etching to create a roughened surface. At least one supplier of dental implants has proposed grit blasting to create a coarse roughened surface, followed by acid etching to form a superimposed fine roughening.

Etching the surface of titanium with acids has been included in many  
25 processes proposed for manufacturing dental implants. In many cases, only general reference to selecting from a list of mineral acids, in other instances specific acids are used. For example, Toho Titanium Co. disclosed in Japanese Published Patent Application JP3146679A1 a two step treatment in which aqueous hydrofluoric acid was used to etch the surface, followed by a second treatment with a solution  
30 hydrofluoric acid and hydrogen peroxide. Another example is found in U.S.



Published Application 2003/0135282A1, in which an implant is treated with a sequence of three acids – hydrofluoric, sulfuric and hydrochloric acid. The etched surface is coated with plasma before use to improve integration with bone.

In a series of U.S. patents, including U.S. Patent Nos. 5,603,338; 5,876,453; 5,863,201; and 6,652,765 assigned to Implant Innovations Inc., a unique two-step acid treatment was disclosed, which is used on dental implants to produce an Osseotite® surface. The first acid treatment uses aqueous hydrofluoric acid to remove the “native oxide”, that is, the titanium oxide found on titanium metal surfaces. Removing the native oxide makes the metal surface more accessible to etching by other acids, assuring uniform etching of the titanium surface. Other methods of removing the native oxide could be used, such as plasma treatment, but the initial treatment with aqueous hydrofluoric acid was preferred. The second acid treatment preferably used a mixture of hydrochloric and sulfuric acids to etch the exposed titanium surface. A relatively fine etching was achieved, having peak-to-valley heights of 10  $\mu\text{m}$  or less. The peak-to-peak distance typically is about 1-3  $\mu\text{m}$ . This Osseotite® surface has achieved commercial success, having reduced the time required for osseointegration of the titanium implant with bone.

Previous U.S. patents have shown the titanium surface obtained by scanning electron microscopy (SEM). Another method of describing the surface is surface mapping microscopy (SMM), which produces a computer-generated three-dimensional picture of the region being examined, and several calculated measures of the roughness of the surface. It will be understood by those skilled in the art that acid treatment produces a surface that appears very uniform to the naked eye, but contains variations that become evident only when greatly magnified, as in the photomicrographs. Each region will not be precisely the same as the others, but nevertheless, the variations are small and fall within the general limits discussed above. By carefully controlling the treatment process, each implant has substantially the same surface.

It has more recently been found that, while a consistent response to the two-step acid treatment is obtained on commercially pure titanium, the same treatment process produces non-uniform results on titanium alloys. Because titanium alloys have some benefits over commercially pure titanium, it would be desirable if the



topography of the Osseotite® surface were to be duplicated on a titanium alloy surface. To achieve this goal, the inventors found that the process used for commercially pure titanium required unexpected revisions to achieve the desired surface on titanium alloys. Their new process will be described in detail below.

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#### Summary of the Invention

A process for producing on a titanium alloy a desired surface topography similar to the Osseotite® surface removes the native oxide on the titanium alloy and thereafter, and before the titanium alloy reoxidizes significantly, the surface is etched  
10 in an aqueous solution of hydrofluoric and hydrochloric acids to produce the desired surface. The native oxide maybe removed by immersing for about 40 to 60 seconds in an aqueous solution of hydrofluoric acid containing about 7.9 to 9.0 wt% hydrofluoric acid. In a preferred embodiment, the surface is etched for about 19-21 minutes at room temperature in an acid mixture containing from about 0.053 to about 0.105 wt%  
15 hydrofluoric (HF) acid and from about 19 to about 21 wt% hydrochloric (HCl) acid. The conditions for both acid treatments are chosen to provide the desired surface topography, while minimizing the loss of titanium metal.

In one specific embodiment, the native oxide is removed by immersing the implant in an 8.45 wt% HF solution at ambient temperature for 45 seconds. After  
20 rinsing to remove the residual acid, the implant is immersed for 19.5 minutes at ambient temperature in a 20 wt% HCl solution containing 0.0845 wt% HF.

The conditions for both acid treatments are chosen to provide the desired surface topography, while minimizing the loss of titanium metal.

In one aspect, the invention is a dental implant that has been treated according  
25 to the process described above to provide the desired surface topography in predetermined regions of the implant.

#### Brief Description of the Drawings

Fig. 1A shows a commercially pure titanium machined surface.

30 FIG. 1B shows the surface of FIG. 1A after being treated with HF

FIG. 1C shows the surface of FIG. 1B after being etched with HCl and H<sub>2</sub>SO<sub>4</sub> so as to produce an Osseotite® surface on pure titanium.

Fig. 1D is a surface map of the Osseotite® surface of FIG. 1C.

Fig. 1E shows the effect of the treatment of Fig. 1A-C on Ti 6/4 alloy.

Fig. 2A-E show several etching processes on Ti 6/4 alloy.

Fig. 3A-E show the effect of etching with Keller's reagent and Kroll's reagent.

5 Fig. 4A-B show the effect of etching with HCl alone.

Fig. 5A-D show the effect of etching with HCl plus HF.

Fig. 6 shows a typical dental implant.

### Detailed Description of the Preferred Embodiments

#### 10 Titanium and Titanium Alloys

Although other metals, and ceramics have been proposed for use in dental implants, titanium has been generally used. Particularly commercially pure titanium, which contains trace amounts of carbon, iron, oxygen, hydrogen, and nitrogen. Titanium alloys have also been used since they are stronger than the commercially  
15 pure grades of titanium. One commonly used titanium alloy, Ti-6Al-4V, contains 6 wt % aluminum and 4 wt % vanadium, hereafter referred to as Ti 6/4.

A characteristic of titanium and its alloys is the rapid formation of tenacious titanium oxide films on the surface, a feature which contributes to titanium's resistance to corrosion. This oxide film is considered to be a combination of various  
20 oxides of titanium, including TiO, TiO<sub>2</sub>, Ti<sub>2</sub>O<sub>3</sub>, and Ti<sub>3</sub>O<sub>4</sub>. It has been referred to the "native oxide" film. Measurement of the native oxide film by Auger spectrometer indicates that it typically has a depth of 70 to 150 Angstroms.

As previously disclosed, removing the native oxide is important if a uniformly roughened surface is to be produced by acid etching. Experience has shown that most  
25 acids are not capable of removing the native oxide sufficiently so that a uniform roughness can be produced. Titanium surfaces are often pickled in mixtures of hydrofluoric acid and nitric acids to clean the surface. Aqueous solutions of hydrofluoric acid alone, without the addition of oxidizing acids such as nitric acid, are very aggressive toward titanium and its native oxide film. A relatively brief exposure  
30 to a dilute solution of hydrofluoric acid will remove the native oxide. Since after removing the native oxide, the hydrofluoric acid will begin to consume the metal as well, an undesirable result, the titanium implant is removed from the acid and rinsed



to stop further attack. However, as is well known, the titanium metal surface will begin to oxidize quickly. Consequently, the exposed metal surface should be protected against oxygen exposure until the titanium implant is immersed in an acid bath to uniformly etch the surface, creating the desired surface topography. Other  
5 methods of removing the native oxide could be used, such as plasma treatment, but the use of hydrofluoric acid is preferred.

The rate at which titanium is etched depends on the concentration of the hydrofluoric acid. A hydrofluoric acid solution containing about 15 vol. % of 49 wt % hydrofluoric acid was found to permit complete removal of the native oxide within  
10 about one-half minute, but with minimal consumption of the metal. This is illustrated in Figures 1A and B which show at 2000X magnification the surface of a commercially pure titanium metal dental implant after machining (producing macro-features such as threads or grooves) and then after being exposed to hydrofluoric acid to remove the native oxide. The machining marks have disappeared and the  
15 hydrofluoric acid has left the titanium grains exposed after the native oxide has been removed and some of the grain boundary material has been removed.

In Figure 1C, the surface of the commercially pure titanium (after native oxide has been removed) has been etched with a solution of 19.55 wt % hydrochloric acid and 72.29 wt % sulfuric acid at 60-70°C for about 7 minutes. This desirable surface  
20 topography has been clinically demonstrated to achieve enhanced osseointegration. Implants having this surface are sold under the Osseotite® trademark by the assignee of the present invention. This desirable surface has a generally uniform set of sharp peaks with a maximum peak-to-valley height of 10 µm or less. The average peak-to-peak distance is about 1-3 µm. The result of a typical examination of an Osseotite®  
25 surface by surface mapping microscopy is shown in Figure 1D.

Figures 1A-1D illustrate the process and results produced on a commercially pure titanium dental implant. Clinical success of the Osseotite® surface in improving osseointegration of the implants has been confirmed and it is well accepted in the marketplace. Therefore, the present inventors had expected to create the same surface  
30 topography on titanium alloy Ti 6/4 using the same treatment. However, they were surprised to discover that the process providing uniform results on commercially pure

titanium failed to produce the characteristic surface topography when applied to Ti 6/4 alloy.

Other etching solutions were tested. In some instances, a surface similar to the Osseotite ® surface was obtained, but in other cases, acid etching was ineffective. It was found also that the effect on Ti 6/4 alloy varied from batch to batch, so that each batch had to be tested to determine its suitability. After further investigation of this problem, the inventors found that certain acid etching solutions were capable of consistently producing the desired surface on Ti 6/4 alloy.

#### Acid Etching of Ti 6/4 Alloy

Figures 1E, and 2C-E, 3A-E, 4A, B, and 5A-D illustrate the results of some of the acids tested on Ti 6/4 E.L.I. alloy, as defined by ASTM B348 Grade 23 or ASTM F136. In each case, the implants had been given the same treatment in a hydrofluoric acid solution to remove the native oxide on the surface. In particular, the implants were immersed in 8.45 wt% hydrofluoric acid at room temperature. The results of the etching processes shown in Figures 1E, and 2C-E, 3A-E, 4A, B, and 5A-D can be compared with Figure 1C, the Osseotite® surface produced on commercially pure titanium metal by an acid treatment with an initial mixture of 19.55 wt% hydrochloric acid and 72.29 wt% sulfuric acid at 60-70°C for 7 minutes.

Experiments were carried out with a series of acid compositions, the results being shown in Figures. The acid compositions and treatment conditions are summarized in the following table.



**Table 1**

Figure	Ti:	Native Oxide Removal Treatment		Etching					
				Acid Composition <sup>(1)</sup>					
		8.45 wt% HF	Time, min.	HF	HCl	H <sub>2</sub> SO <sub>4</sub>	HNO <sub>3</sub>	Time min.	Temp °C
1C	CP	Yes	1.0	—	19.55	77.29	—	7	60-70
1E	6/4	Yes	1.0	—	19.55	77.29	—	7	60-70
2A	6/4	No	—	0.284	1.062	—	2.297	1	61
2B	6/4	No	—	0.284	1.062	—	2.297	8	61
2C	6/4	Yes	0.5	0.284	1.062	—	2.297	0.5	61
				—	19.55	77.29	—	1.0	61
2D	6/4	Yes	0.5	0.284	1.062	—	2.297	0.5	61
				—	19.55	77.29	—	7	61
2E	6/4	Yes	0.17	0.284	1.062	—	2.297	1.5	61
				1.143	—	—	1.923	1.5	ambient
3A	6/4	Yes	1.0	0.284	1.062	—	2.297	7	ambient
3B	6/4	Yes	2.5	0.284	1.062	—	2.297	7	ambient
3C	6/4	Yes	1.0	0.284	1.062	—	2.297	10	ambient
3D	6/4	Yes	2.5	0.284	1.062	—	2.297	10	ambient
3E	6/4	Yes	2.5	0.284	1.062	—	2.297	10	ambient
4A	6/4	Yes	1.5	—	20	—	—	14	ambient
4B	6/4	Yes	1.5	—	20	—	—	21	ambient
5A	6/4	Yes	1.0	0.26	20	—	—	20	ambient
5B	6/4	Yes	1.0	0.175	20	—	—	20	ambient
5C	6/4	Yes	1.0	0.09	20	—	—	20	ambient
5D	6/4	Yes	1.0	0.09	20	—	—	20	ambient

<sup>(1)</sup> wt% acid, remainder water

The above table generally follows the progress of experiments carried out to determine the acid etching needed to produce the desired surface topography on Ti 6/4 alloy. To produce the surface of FIG. 1C, the native oxide on the commercially pure titanium was removed by exposure to an 8.45 wt % HF solution for 1 minute at ambient temperature. After rinsing in deionized water containing baking soda to neutralize the residual acid and a further rinse in deionized water, the titanium was immersed in an aqueous solution of 19.55 wt % HCl and 77.29 wt % H<sub>2</sub>SO<sub>4</sub> for 7 minutes at 60-70°C to produce a uniformly roughened surface, i.e. the Osseotite® surface.

Figure 1E illustrates the surprising result when the same procedure was carried out on Ti 6/4 alloy. As will be seen in the photograph, the characteristic Osseotite surface was not obtained on Ti 6/4 alloy. The machining marks were still visible. It



was concluded that a different etching process was needed for use with Ti 6/4 alloy if the Osseotite® surface was to be provided on the Ti 6/4 alloy.

Figures 2A-E show the results obtained when two known etching acid mixtures were used. One was Keller's solution, containing HF, HNO<sub>3</sub>, and HCl, and the second was Kroll's solution, containing HF and HNO<sub>3</sub>. The compositions used are shown in Table 1 above. Figures 2A and 2B show that Keller's solution alone did not produce the Osseotite surface, although some pitting can be seen. Since the pre-treatment with HF solution to remove the native oxide was not done, it is presumed that the native oxide interfered with the attempted etching with Keller's solution.

Figures 2C to 2E show the results achieved when the native oxide was removed by pre-treatment with an HF solution and thereafter the titanium surface was exposed to two acid solutions in sequence. In FIG. 2C and 2D, Keller's solution was used, followed by the mixture of HCl and H<sub>2</sub>SO<sub>4</sub>, known to be successful in etching chemically pure titanium. In FIG. 2E, Keller's solution was used first, followed by immersion of the Ti 6/4 alloy in Kroll's solution. None of these tests produced a surface topography like that shown in FIG. 1C on the Ti 6/4 alloy.

Figures 3A-3E show the results obtained when the native oxide was removed with an HF solution, and Keller's solution was used for etching, but at ambient temperature rather than at 61°C previously used. It was found that this process was capable of providing a surface similar to FIG. 1C on some samples of Ti 6/4 alloy, but not on others (compare FIG. 3 D with FIG. 3 E). The difference in response of the samples appeared to be associated with the machining or the alloy heat (i.e., the conditions associated with a specific batch of titanium alloy). Therefore, additional experimentation was undertaken. However, it was concluded that etching with Keller's solution may be useful also, provided that control of the quality of the Ti 6/4 alloy can be achieved.

Figures 4 A and B report the surfaces produced when the native oxide was removed by the usual method and then the surface was etched with an HCl solution. Although some pitting occurred, it was evident that HCl alone was not sufficient to produce a surface like that of FIG. 1C.

Figures 5 A-D illustrate the improved results that were obtained when small amounts of HF were added to the 20 wt % HCl etching solution. It was concluded



that a small amount of HF should be used if the desired surface topography was to be obtained. The surfaces of FIGS. 5C and 5D were given the same treatment and produced substantially the same surface, even though the C and D samples had different machining and heats. Thus, it was concluded that the process was broadly applicable to Ti 6/4 alloys.

In the presently preferred process, Ti 6/4 alloy is immersed in an aqueous solution of hydrofluoric acid for the length of time required to remove the native oxide while not removing a significant amount of metal. A preferred solution, suitable for commercial application would contain about 7.9 to 9.0 wt% HF.

However, more or less concentrated solutions could be used, with appropriate adjustment of the exposure time, provided that the native oxide was removed to prepare the surface for subsequent etching needed to create the desired surface topography.

The etching step immerses the Ti 6/4 alloy, from which the native oxide had been removed, in an aqueous solution at room temperature containing about 0.053 to 0.105 wt % HF and 19-21 wt % HCl. Such solutions have been found to produce the desired surface topography on Ti 6/4 alloy within about 20 minutes and using only ambient temperatures. Again, some adjustment of the acid concentrations, temperature, and exposure time is believed to be possible, while still obtaining the desired surface. It is believed that equivalent results may be obtained within the broader range of 0.005 to 1.0 wt% HF and 10-30 wt% HCl.

### Dental Implants

The etching process of the invention may be used to prepare the surface of various styles of dental implants. A typical example is illustrated in Fig. 6. The implant 10 will be placed in a pre-drilled hole in a patient's bone to replace the root of a missing tooth. The threaded portion 12 engages the bone, while at least some of the upper portion 14 contacts tissue. In many cases, the etching process will be applied to the threaded portion 12 of the implant 10, while the upper portion 14, shown in Fig. 6 to include a head 16 portion for engaging dental prosthesis components and a neck portion 18, remains relatively smooth. In some cases, the roughened area may be extended upward into the neck and head regions, or even to the top of the implant 10.

10

In other cases, only a portion of the threads will be roughened to improve osseointegration of the metal with bone, while the upper section of the threaded region will remain relatively smooth.



The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of producing a uniformly roughened surface on Ti 6/4 alloy for contact with living bone comprising:
  - (a) removing native oxide from said Ti 6/4 alloy to expose metal; and
  - (b) contacting said exposed metal with an aqueous solution of hydrofluoric acid and hydrochloric acid for a suitable period of time to create a desired surface topography having irregularities with peak-to-valley heights of less than 10 microns.
2. A method of claim 1, wherein the roughened surface has peak distances of about 1-3 $\mu$ m.
3. A method of claim 1 or 2, wherein said native oxide is removed in step (a) by contacting said Ti 6/4 alloy with an aqueous solution of hydrofluoric acid for a suitable period of time.
4. A method of claim 3, wherein said aqueous solution of step (a) contains about 7.9 to 9.0 wt% hydrofluoric acid.
5. A method of any one of claims 1 to 4, wherein said aqueous solution of step (b) contains about 0.005 to 1.0 wt% hydrofluoric acid and about 10 to 30 wt% hydrochloric acid.
6. A method of claim 5, wherein said aqueous solution of step (b) contains about 0.0845 wt% hydrofluoric acid and about 20 wt% hydrochloric acid.
7. A method of producing a uniformly roughened surface on Ti 6/4 alloy for contact with living bone comprising:
  - (a) removing native oxide from said Ti 6/4 alloy to expose metal by contacting said alloy with an aqueous solution of hydrofluoric acid for a suitable period of time;
  - (b) rinsing said alloy after said native oxide removal of (a);

(c) contacting said exposed metal in an aqueous solution of about 0.005 to 1.0 wt% hydrofluoric acid and about 10 to 30 wt% hydrochloric acid at room temperature for a suitable period of time to create a desired surface topography having irregularities with peak-to-valley heights of less than 10 microns; and

(d) rinsing the surface produced in step (c) to remove the residual aqueous solution of (c).

8. A method of claim 7, wherein said aqueous solution of step (a) contains about 7.9 to 9.0 wt% hydrofluoric acid.

9. A method of claim 8, wherein said aqueous solution of step (a) contains about 8.45 wt% hydrofluoric acid.

10. A method of claim 7, 8 or 9, wherein said aqueous solution of step (c) contains about 0.053 to 0.105 wt% hydrofluoric acid and about 19 to 21 wt% hydrochloric acid.

11. A method of claim 10, wherein said aqueous solution of step (c) contains about 0.0845 wt% hydrofluoric acid and about 20 wt% hydrochloric acid.

12. A dental implant of Ti 6/4 alloy for contact with living bone comprising:  
a. a head section for mounting dental prosthesis components;  
b. a threaded section for engaging said living bone;  
c. a neck section disposed between said head section and said threaded section;  
wherein at least a portion of said threaded section has a desired roughened surface topography having irregularities with peak-to-valley heights of less than 10 microns created by etching said threaded portion with an aqueous solution of about 0.005 to 1.0 wt% hydrofluoric acid and about 10 to 30 wt% hydrochloric acid.

13. A dental implant of claim 12, wherein native oxide is removed from said threaded portion prior to said etching with an aqueous solution of hydrofluoric acid and hydrochloric acid.



14. A dental implant of claim 13, wherein said native oxide is removed by contacting said threaded portion with a second aqueous solution of hydrofluoric acid.

15. A dental implant of claim 14, wherein said second aqueous solution contains about 7.9 to 9.0 wt% hydrofluoric acid.

16. A dental implant of claim 12, wherein said aqueous solution contains about 0.0845 wt% hydrofluoric acid and about 20 wt% hydrochloric acid.

17. A method of producing a uniformly roughened surface of Ti 6/4 alloy for contact with living bone comprising:

(a) removing native oxide from said Ti 6/4 alloy to produce an exposed metal surface; and

(b) etching said exposed metal surface with Keller's Solution to create a desired surface topography having irregularities with peak-to-valley heights of less than 10 microns.

18. A dental implant assembly comprising:

a threaded section comprising Ti 6/4 alloy adapted to engage living bone; and

a head section located above the threaded section;

wherein at least a portion of the threaded section has a roughened surface topography having irregularities with peak-to-valley heights of less than 10 microns created by etching with an aqueous solution comprising hydrofluoric acid and hydrochloric acid.

19. The dental implant assembly of claim 18, further comprising a neck section positioned between the head section and the threaded section, the head section being adapted to engage dental prosthesis components, wherein the threaded section directly below the neck section has a roughened surface topography having irregularities with peak-to-valley heights of less than 10 microns.

20. A method of producing a uniformly roughened surface on a dental implant for contact with living bone, the dental implant being made of Ti 6/4 alloy and having a head section, a neck section below the head section, and a threaded section below the neck section, the method comprising:

(a) removing native oxide from the neck section and the threaded section to create an exposed metal surface; and

(b) contacting the exposed metal surface including the neck section and the threaded section directly below the neck section with an aqueous solution of hydrofluoric acid and hydrochloric acid for a suitable period of time to create a desired surface topography having irregularities with peak-to-valley heights of less than 10 microns.



Application number / numéro de demande: 2467320

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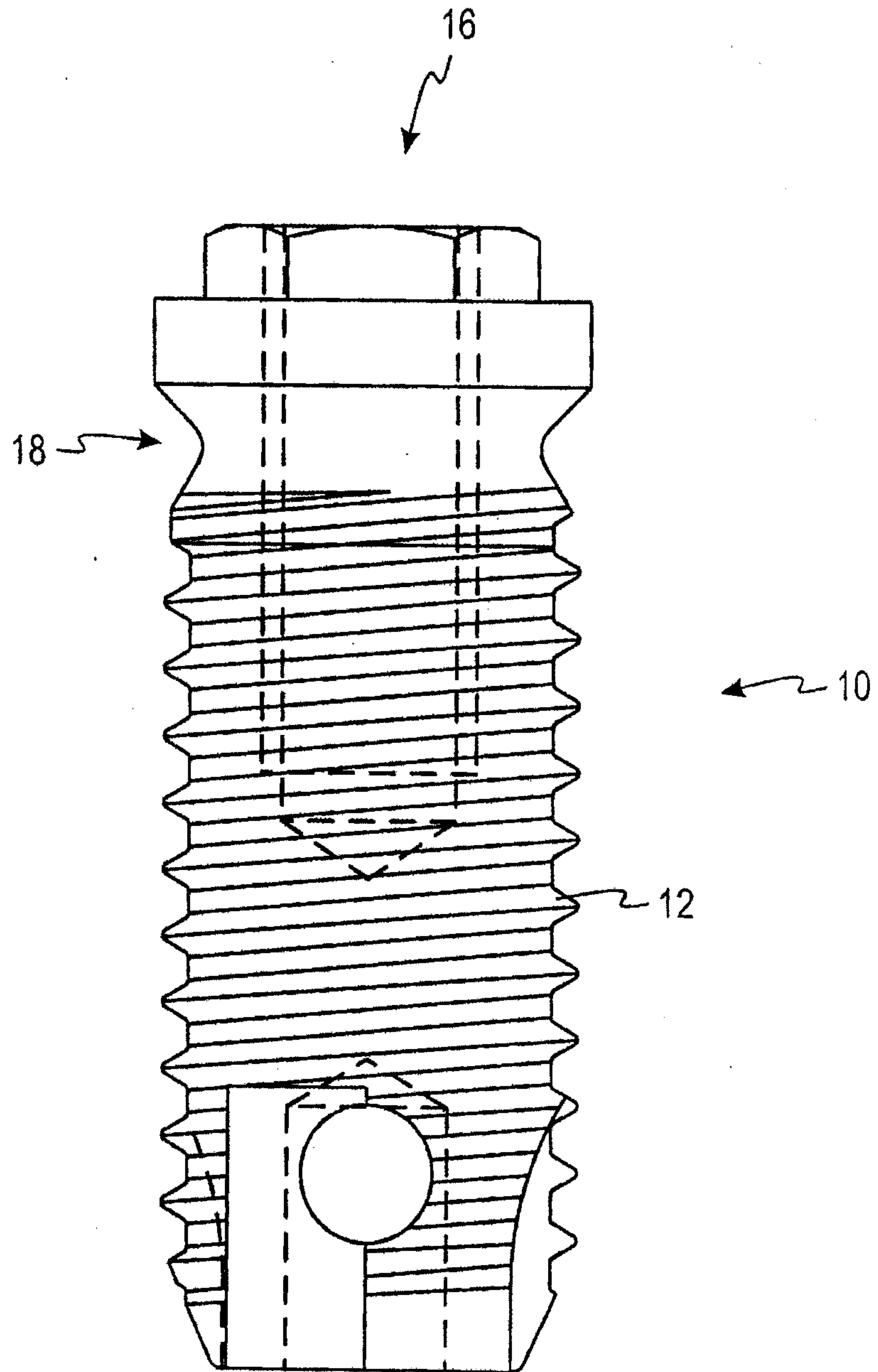
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*Fig. 6*