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Method for producing an anti-infective coating on implants

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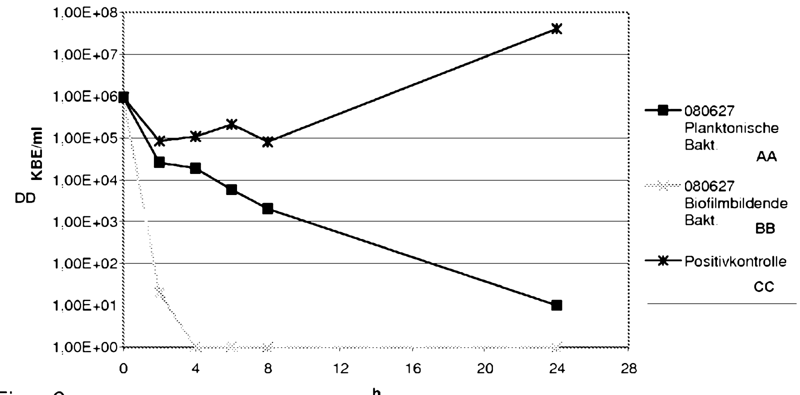
Erklärungen gemäß Regel 4.17:
— *Erfindererklärung (Regel 4.17 Ziffer iv)*

[Fortsetzung auf der nächsten Seite]

(54) Title: METHOD FOR PRODUCING AN ANTI-INFECTIVE COATING ON IMPLANTS

(54) Bezeichnung : VERFAHREN ZUR HERSTELLUNG EINER ANTIINFEKTIÖSEN BESCHICHTUNG AUF IMPLANTATEN



Figur 2

AA PLANKTONIC BACT.
BB BIOFILM-FORMING BACT.
CC POSITIVE CONTROL
DD CFU/MLU/ml

(57) Abstract: The invention relates to a method for producing an anti-infective coating on implants that contain titanium or are composed of titanium. The aim of the invention is to provide a coating method with which it is possible, for implants made of titanium or that contain titanium, to combine the optimization of the mechanical properties achieved with the anodic oxidation type II with the optimization of the anti-infective properties. According to the invention, the implants are anodically oxidized in an alkaline solution, then metal having anti-infective properties is electrodeposited on said surface, and afterwards the oxide layer containing metal is solidified.

(57) Zusammenfassung: Die Erfindung betrifft ein Verfahren zur Herstellung einer antiinfektiösen Beschichtung auf Implantaten, die Titan enthalten oder aus Titan bestehen. Es liegt die Aufgabe zugrunde, ein Beschichtungsverfahren aufzuzeigen, mit dem es möglich

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— *mit internationalem Recherchenbericht (Artikel 21 Absatz 3)*

ist, für Implantate aus Titan oder die Titan enthalten die mit der anodischen Oxidation Typ II erreichte Optimierung der mechanischen Eigenschaften mit der der antiinfektiösen Eigenschaften zu vereinen. Erfindungsgemäß werden die Implantate in einer alkalischen Lösung anodisch oxidiert, anschließend auf dieser Oberfläche Metall, das antiinfektiöse Eigenschaften aufweist, galvanisch abgeschieden und danach wird die metallhaltige Oxidschicht verfestigt.

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Method for producing an anti-infective coating on implants

Description

The invention relates to a method for producing an anti-infective coating on implants, that contain titanium or are composed of titanium.

State of the art

From DE 10243132 B4 is known to provide implants made of titanium or titanium alloys with a titanium oxide coating, in which metal ions such as silver and copper are homogeneously distributed, by using a sol-gel method. For the mechanical stabilization and densification of said coating a heat treatment is applied after drying. By means of heating to about 500 °C a ceramization of the coating takes place. For the implants the heat treatment has the disadvantage that it results in a loss of strength. Therefore, it is not suitable for implants which must have high fatigue strength.

It is known that an anodic oxidation type II of titanium results in a greater hardness and higher fatigue strength. In DE 20020649 U1 this coating is also described as an anti-infective coating because of its smooth surface, besides silver and copper. However, it is not possible to produce a sufficiently adherent coating of silver or copper on an anodic oxidation type II coating.

Genesis of the Invention

The genesis of the invention is a desire to provide a coating method by means of which it is possible for implants made of titanium or which contain titanium to combine the optimization of the mechanical properties achieved by the anodic oxidation type II with the optimization of the anti-infective properties.

According to an aspect of the invention there is provided a method for producing an anti-infective coating on an implant containing or comprising of titanium, the method comprising the steps of:

anodically oxidizing a surface of the implant in an electrolyte comprising an alkaline solution to form a porous titanium oxide layer on the implant surface,

wherein the pores of the titanium oxide layer have an electrical conductivity sufficient for electro-deposition of an anti-infective metal in the pores;

electro-depositing in the pores a metal having anti-infective properties to form a titanium oxide layer containing the anti-infective metal; and

blasting the anti-infective metal-containing titanium oxide layer to form a solidified anti-infective metal-containing titanium oxide layer.

According to another aspect of the invention there is provided a coated implant comprising a substrate and an anti-infective coating on a surface of the substrate, the substrate containing or consisting of titanium, and the anti-infective coating comprising a porous titanium oxide layer formed by anodic oxidation of the substrate surface, and an anti-infective metal electro-deposited in the pores of the titanium oxide layer, wherein the titanium oxide layer containing the anti-infective metal is solidified by blasting.

The genesis is addressed in that the implants are oxidized anodically in an alkaline solution, then metal having anti-infective properties is electrodeposited on the surface, and afterwards the metal-containing oxide layer is solidified.

In the anodic oxidation a conversion layer containing oxygen and optionally other atoms, and a porous titanium oxide layer having a sufficient conductivity, which is electrically conductive in the pores, such that metal can be electrodeposited in it, can be formed. By blasting, e.g. with glass beads, the metal-containing oxide layer is solidified, and more weakly bound oxide and metal particles are removed or more intensely connected with each other and with the surface of the implant.

In the preferred embodiments, there is provided a method for the combined modification of implants containing titanium or consisting of titanium, wherein in the first step a porous oxide layer is formed by anodic spark discharge in strongly alkaline electrolytes, in the second step the galvanic charge of metal into the porous layer takes place, and in a third step the metal-enriched oxide layer is solidified by blasting and can be more or less eliminated by removing more weakly bound oxide or metal particles or by connecting said particles more intensely with each other and the implant.

Preferably, the elution of the metal starting under physiological conditions may be adjusted in regard to its concentration by variation of the mean metal coating of the

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implant surface, so that an antimicrobial or rather antibacterial action without any substantial damage to the cells of the surrounding body tissue is achieved. In particular copper, silver and zinc are suitable as metals. In the case of the metal copper there can be additionally expected an improved blood flow of the newly formed body tissue due to the catalytic effect on the angiogenesis. The thickness of the metal-containing layer is advantageously 8 – 15 μm, preferably 10 μm.

Preferably solidifying the metal-containing oxide layer is advantageously carried out by blasting with glass beads.

Brief Description of the Figures

A preferred embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawings in which:

Figure 1 Partially copper-coated TiAlV-surface

Figure 2 Development of cell number of staphylococcus aureus ATCC25923 after culture on a partially copper-coated TiAlV-surface

Example 1: Coating

TiAl6V4-samples are coated with a porous oxide layer according to the principle of anodic spark discharge in a strongly alkaline electrolyte. For that, 50 g NaOH are dissolved in 500 ml of distilled water. In the solution heated to 40°C, the anodic spark oxidation of the titanium sample is conducted by slowly increasing the voltage to 40 volt. Then, copper from a saturated copper acetate solution is added into the porous oxide layer by means of cathode deposition. The resulting copper- enriched oxide layer is partially removed by glass bead blasting, and the copper contained in it is almost completely incorporated in an island-like manner into the sample surface (Figure 1).

Example 2: Description of the antibacterial mechanism

For testing the antibacterial action investigations were performed on clinically relevant bacterial strain staphylococcus aureus ATCC25923. For that, TiAlV-cylinders with a diameter of 8 mm and a length of 20 mm were coated with copper according to Example 1, and outsourced at 37 °C for various times in 6 ml of a PBS buffer solution which had been vaccinated with 150 μl of bacterial suspension. Thereafter, the concentration of the living bacteria on the cylinders and in the solution was determined by plating on agar culture media and by outsourcing (48 h) at 30°C. Even after only 4 hours no living bacteria were found on the cylinders and after 24 hours only very few living bacteria were found in the buffer solution (Figure 2).

The foregoing describes only one embodiment of the present invention and modifications, obvious to those skilled in the art, can be made thereto without departing from the scope of the present invention.

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The term "comprising" (and its grammatical variations) as used herein is used in the inclusive sense of "including" or "having" and not in the exclusive sense of "consisting only of".

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CLAIMS

1. A method for producing an anti-infective coating on an implant containing or comprising of titanium, the method comprising the steps of:

   anodically oxidizing a surface of the implant in an electrolyte comprising an alkaline solution to form a porous titanium oxide layer on the implant surface, wherein the pores of the titanium oxide layer have an electrical conductivity sufficient for electro-deposition of an anti-infective metal in the pores;

   electro-depositing in the pores a metal having anti-infective properties to form a titanium oxide layer containing the anti-infective metal; and

   blasting the anti-infective metal-containing titanium oxide layer to form a solidified anti-infective metal-containing titanium oxide layer.

2. The method according to claim 1, wherein the electrolyte for said anodic oxidation comprises sodium hydroxide.

3. The method according to claim 1, wherein the electrolyte for said anodic oxidation comprises sodium silicate.

4. The method according to any one of claims 1 to 3, wherein the metal is selected from the group consisting of copper, silver, zinc and mixtures thereof.

5. The method according to claim 1, wherein the anti-infective metal-containing titanium oxide layer has a thickness of from 8 to 15 μm.

6. The method according to claim 1, wherein the blasting step comprises blasting the anti-infective metal-containing titanium oxide layer with glass beads.

7. A coated implant comprising a substrate and an anti-infective coating on a surface of the substrate, the substrate containing or consisting of titanium, and the anti-infective coating comprising a porous titanium oxide layer

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formed by anodic oxidation of the substrate surface, and an anti-infective metal electro-deposited in the pores of the titanium oxide layer, wherein the titanium oxide layer containing the anti-infective metal is solidified by blasting.

8. The coated implant of claim 7, wherein the anti-infective metal is selected from the group consisting of copper, silver, zinc and mixtures thereof.

9. A method of producing an anti-infective coating on a titanium implant, the method being substantially as herein described with reference to any one of the accompanying drawings or examples.

10. An implant having an anti-infective coating provided by the method of any one of claims 1 to 6.

Dated this 12rd day of September 2013

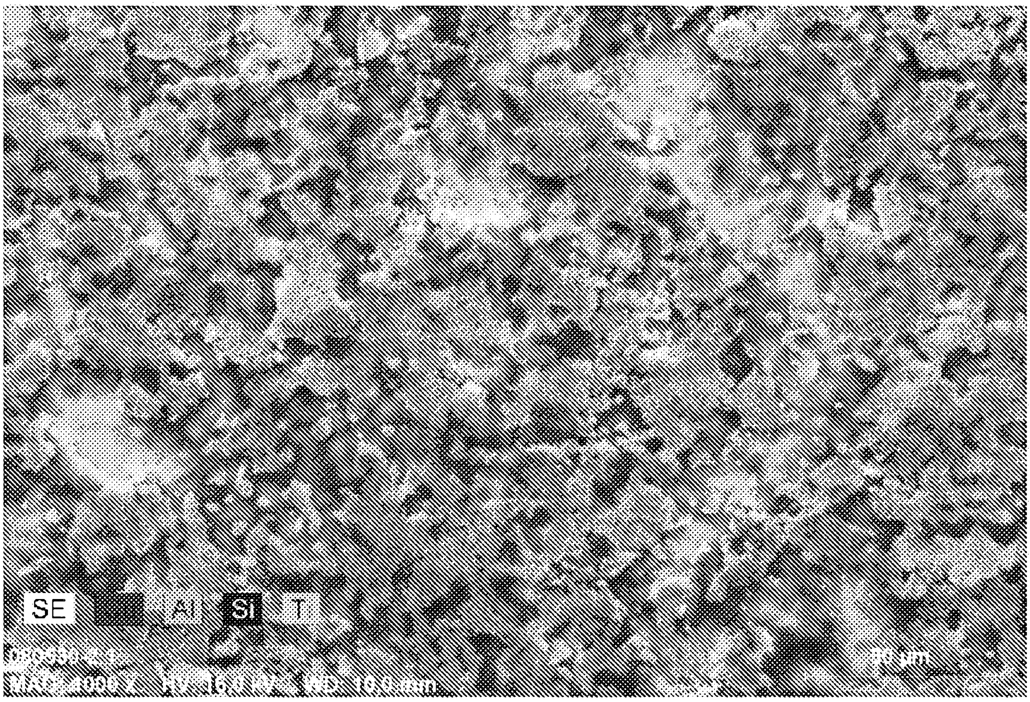
DOT GmbH

BY:

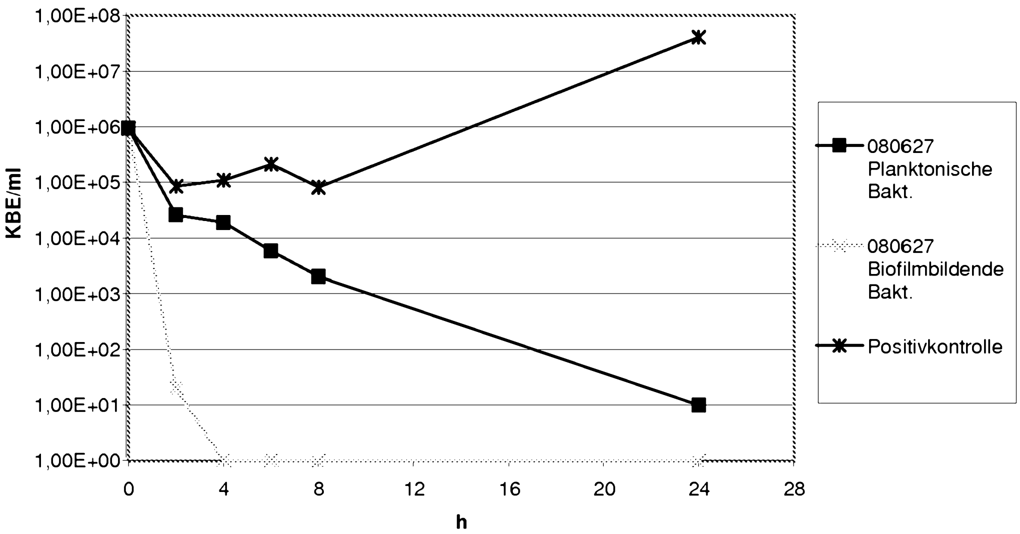
FRASER OLD & SOHN

Patent Attorneys for the Applicant

Zeichnungen



Figur 1



Figur 2