



(19) **United States**

(12) **Patent Application Publication**
HACHENBERG et al.

(10) **Pub. No.: US 2019/0321147 A1**

(43) **Pub. Date: Oct. 24, 2019**

(54) **METHOD FOR MANUFACTURING A DENTAL PROSTHESIS**

A61K 6/04 (2006.01)

B22F 3/16 (2006.01)

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B22F 3/11 (2006.01)

B22F 3/02 (2006.01)

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(52) **U.S. Cl.**

CPC *A61C 13/0022* (2013.01); *C22C 19/07* (2013.01); *C22C 19/053* (2013.01); *C22C 19/05* (2013.01); *B22F 3/24* (2013.01); *B22F 3/1021* (2013.01); *B22F 2003/247* (2013.01); *B22F 3/16* (2013.01); *C22C 19/055* (2013.01); *B22F 3/11* (2013.01); *B22F 3/02* (2013.01); *B22F 2301/15* (2013.01); *B22F 2003/248* (2013.01); *A61K 6/04* (2013.01)

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(21) Appl. No.: **16/452,809**

(57)

ABSTRACT

(22) Filed: **Jun. 26, 2019**

Related U.S. Application Data

(60) Continuation of application No. 15/178,697, filed on Jun. 10, 2016, which is a division of application No. 13/292,132, filed on Nov. 9, 2011, now Pat. No. 9,393,088.

A method for manufacturing a shaped body, comprising creating a mixture of a metal powder and binding agent, compacting the mixture to form a green compact, heating the green compact to a debinding start temperature T_1 , debinding the green compact by controlled heating of the green compact from start temperature T_1 to end temperature T_2 at a heat-up rate R_1 , presintering the debinded green compact to the presinter end temperature T_{VS} at a heat-up rate R_{HVS} , cooling the green compact from the presinter end temperature T_{VS} at a cool-down rate R_{KVS} , whereby at least the heat-up rate R_{HVS} , the presinter end temperature T_{VS} , and the cool-down rate R_{KVS} are tuned relative to each other in such a way that the presintered green compact forming a blank has a surface porosity of 16% to 22% after presintering, and machining and sintering of the blank to form the shaped body.

(30) **Foreign Application Priority Data**

Nov. 9, 2010 (EP) 10 19 0512.3

Publication Classification

(51) **Int. Cl.**

A61C 13/00 (2006.01)

C22C 19/07 (2006.01)

C22C 19/05 (2006.01)

B22F 3/24 (2006.01)

B22F 3/10 (2006.01)

METHOD FOR MANUFACTURING A DENTAL PROSTHESIS

[0001] This application is a continuation of U.S. application Ser. No. 15/178,697, filed Jun. 10, 2016, which is a divisional application of U.S. application Ser. No. 13/292,132, filed Nov. 9, 2011, now U.S. Pat. No. 9,393,088, which claims priority to EP application number 10 19 0512.3, filed Nov. 9, 2010, the contents of which are incorporated herein by reference.

[0002] The invention relates to a method for the manufacture of a shaped body, in particular of a dental prosthesis or part thereof, by way of mixing of a metal powder.

[0003] The invention also relates to a green compact for the manufacture of a dental prosthesis or of a part thereof.

[0004] In recent times CAD/CAM technology (Computer Aided Design, Computer Aided Manufacturing) is more frequently being used in the manufacture of dental prostheses such as crowns or bridges, whereby CAD/CAM is applied in particular in the area of ceramics. EP-B-1 067 880 should be referenced as an example for this.

[0005] DE-C-199 38 144 describes a method for the manufacture of dental prostheses, whereby a ceramic-based presintered moulded blank is machined using a milling process and subsequently is sintered to full density.

[0006] WO-2009/120749 discloses the use of a CAD/CAM milling process in the manufacture of a dental prosthesis. For this purpose, at first a metal powder is mixed with a binding agent, whereupon a moulded blank is produced by way of metal powder injection moulding. A milling process is used to create a shaped body from this, which corresponds to the dental prosthesis to be produced taking into account the contraction occurring during sintering.

[0007] In accordance with EP-A-1 764 062, a shaped body produced from a dental alloy consists of a dental alloy powder that is sintered to full density by hot-isostatic pressing.

[0008] In accordance with DE-A-103 42 231 it is known to manufacture a shaped body using powder-metallurgical processes, whereby during the machining the body is open-pored and has not been sintered to full density. Only after the final shape has been created, the open pores of the shaped body are filled with a second alloy in a further processing step by means of an infiltration process. The use of two alloys is a disadvantage.

[0009] US-A-2005/0023717 discloses a method for the manufacture of dental restorations using a free-forming process, in particular a rapid-prototyping process. Favoured materials to be used are powders of non-oxidizing metals. Preferred use is given to noble metals.

[0010] The reference Rodrigues et al.: "Powder metallurgical Processing of Co-28% Cr-6% Mo for dental implants: Physical, chemical and electrochemical properties" Powder Technology, 2006 (2011), 233-238, describes a method for the manufacture of restoration elements. In this, a biocompatible cobalt-chromium-molybdenum alloy is mixed with a moulding agent, is heated to a temperature suitable for burning out the moulding agent, and is subsequently sintered to full density.

[0011] U.S. Pat. No. 4,996,022 discloses a method for the manufacture of a sintered body. As starting material one uses a powdered metal such as iron or nickel.

[0012] An iron-powder mixture that contains up to 1% organic binding agent is used in the manufacture of a

sintered moulded part in accordance with AT-A-505 698. A final sintering is performed after presintering and cooling.

[0013] The objective of the present invention is to further develop a method and a presintered green body of the above-mentioned type so as to provide a shaped body, in particular a dental prosthesis or part thereof, that can be manufactured with very low tolerances and allows problem-free wet or dry machining, whereby in particular it should be possible to use a ceramic material for veneering purposes. Disadvantages known in the art should be avoided.

[0014] Another objective is to provide a green compact that can be machined with high accuracy in a simple manner, in order to subsequently be able to use it to manufacture a highly precise shaped part, in particular a dental prosthesis or a part thereof.

[0015] In accordance with the invention, this objective is met chiefly through a method for the manufacture of a shaped body, in particular a dental prosthesis or a part thereof, that is characterized by the following process steps:

[0016] Producing a mixture of a metal powder and a binding agent,

[0017] Compacting the mixture to form a green compact,

[0018] Heating the green compact from room temperature to a debinding start temperature T_1 ,

[0019] Debinding the green compact by controlled heating of the green compact from the debinding start temperature T_1 to a debinding end temperature T_2 at a heat-up rate R_1 in a manner that rules out damage to the green compact,

[0020] Presintering the debinded green compact, whereby the green compact is heated to a presinter end temperature T_{VS} at a heat-up rate R_{HVS} ,

[0021] Cooling the green compact from the presinter end temperature T_{VS} at a cool-down rate R_{KVS} , whereby at least the heat-up rate R_{HVS} , the presinter end temperature T_{VS} , and the cool-down rate R_{KVS} are tuned relative to each other in such a way that the presintered green compact forming a blank possesses a surface porosity between 16% and 22% after presintering,

[0022] Material-removing machining of the blank, and

[0023] Sintering the machined blank to final density to form the shaped body.

[0024] Surface porosity here denotes the fraction of the surface that is not filled with material if viewed in a metallographic section.

[0025] In particular it is intended to use as metal powder a dental metal alloy in form of a cobalt-chromium or nickel-chromium alloy.

[0026] For a cobalt-chromium alloy the composition should be chosen as follows:

Cobalt: 50% to 70% by weight

Chromium: 20% to 35% by weight

Molybdenum: 0% to 10% by weight

Tungsten: 0% to 20% by weight

Other elements: less than 10% by weight, whereby the sum total is 100% by weight.

[0027] Also an option is the use of a nickel-chromium alloy of the following composition:

Nickel: 50% to 70% by weight

Chromium: 20% to 35% by weight

Molybdenum: 0% to 10% by weight

Tungsten: 0% to 20% by weight

Other elements: less than 10% by weight,
with a sum total of 100% by weight.

[0028] Other elements that may be considered are in particular manganese, silicon, and nickel in case of the cobalt-chromium alloy, cobalt in case of the nickel-chromium alloy, and beryllium, cadmium, lead, iron, aluminum, titanium, carbon, nitrogen, oxygen, sulphur and other elements with a weight fraction of less than 1%.

[0029] In particular it is intended that the mixture compacted into the green compact possess a surface porosity, corresponding to the volume porosity, of between 16% and 27%, preferably between 18% and 22%. This porosity is created by the areas of the green compact that are filled with air or binding agent in between the metal powder particles.

[0030] It is also intended that after heating of the green compact to the presinter end temperature T_{VS} , the green compact be held at the presinter end temperature T_{VS} for the duration of a holding time t_{VS} and subsequently be cooled at a cool-down rate R_{KVS} .

[0031] In particular it is suggested that the green compact be cooled at the cool-down rate R_{KVS} to a temperature T_3 , whereby in particular $T_3 \leq T_2$, $450^\circ \text{C.} \leq T_3 \leq 650^\circ \text{C.}$, and preferably T_3 is approximately 600°C.

[0032] In this, debinding and presintering should be performed in the absence of oxygen, in particular under an inert gas atmosphere, particularly preferred under an argon atmosphere. Other options are a reducing atmosphere or vacuum.

[0033] For the debinding, the green compact preferably is heated to a debinding start temperature T_1 with $350^\circ \text{C.} \leq T_1 \leq 550^\circ \text{C.}$ After reaching a temperature T_1 , in particular reaching the temperature $T_1 \approx 450^\circ \text{C.}$, a slow heating takes place, whereby the heat-up rate during the debinding process should not exceed 20 K/min. A preferred range for the heat-up rate is 1 K/min to 5 K/min. In particular it is intended that in the region above 500°C. , in particular from above 550°C. to the debinding end temperature T_2 , with $550^\circ \text{C.} \leq T_2 \leq 650^\circ \text{C.}$, in particular $T_2 \approx 600^\circ \text{C.}$, one choose a heat-up rate between 1 K/min and 5 K/min. After reaching the debinding end temperature T_2 the green compact should be held at this temperature for a duration t_2 with $1 \text{ min} \leq t_2 \leq 20 \text{ min}$. However, this is not obligatory and mainly dependent on the chosen heat-up rate.

[0034] Irrespective of the preferred parameters, which were provided as examples and shall not limit the scope of protection of the invention, the heating must be performed in such a manner that the debinding takes place in a controlled manner, so that the green compact is not damaged and rendered unserviceable. This controlled heating, which is essential to prevent damage to the green compact, can be performed by an average expert without any problems after carrying out several simple trials.

[0035] After debinding, heating to the presinter end temperature T_{VS} takes place, whereby in principle the heating rate R_{HVS} may be chosen freely.

[0036] In order to obtain the desired surface porosity of the presintered green compact of between 16% and 22%, in particular between 18% and 20%, the invention intends that the presinter end temperature T_{VS} , the heat-up rate R_{HVS} , possibly the holding time t_{VS} at the presinter end temperature T_{VS} , and the cool-down rate R_{KVS} be tuned relative to each other. In case of a very slow heating to the presinter end temperature T_{VS} , e.g. using a heat-up rate between 1 K/min

and 10 K/min, it is not required that the debinded green compact be held at the presinter end temperature for a time period t_{VS} .

[0037] The cool-down rate may also be used to influence the holding time t_{VS} , in particular to the extreme degree that cool-down commences immediately upon reaching the presinter end temperature T_{VS} .

[0038] The relative tuning of the parameters for the purpose of obtaining the desired surface or volume porosity of between 16% and 22%, in particular between 18% and 20%, can be performed taking into account the details provided as examples in the following.

[0039] If for example the presinter end temperature T_{VS} is in a range between 650°C. and 750°C. , the heat-up rate R_{HVS} and/or the cool-down rate R_{KVS} should be between 1 K/min and 200 K/min, preferably between 1 K/min and 50 K/min, and particularly preferred between 1 K/min and 20 K/min, whereby after reaching the presinter end temperature T_{VS} , a holding time t_{VS} of between 10 min and 200 min, in particular between 30 min and 100 min, particularly preferred between 50 min and 80 min, should be adhered to.

[0040] If the presinter end temperature T_{VS} is between 750°C. and 850°C. , the heat-up rate R_{HVS} and/or the cool-down rate R_{KVS} should be between 5 K/min and 200 K/min, in particular between 5 K/min and 20 K/min. After reaching the presinter end temperature T_{VS} , one should preferably choose a holding time t_{VS} between 5 min and 60 min, in particular between 10 min and 30 min.

[0041] However, it is also possible to set the presinter end temperature T_{VS} in the range between 850°C. and 950°C. , for example. In this case, the heat-up rate R_{HVS} and the cool-down rate R_{KVS} should be in the range between 15 K/min and 200 K/min, preferably between 15 K/min and 50 K/min. Preferred holding times t_{VS} at this presinter end temperature T_{VS} are in the range between 5 min and 30 min, in particular between 10 min and 20 min.

[0042] In order to achieve the desired surface porosity or volume porosity of the presintered blank, the presinter end temperature may also be in the range between 950°C. and 1100°C. In this case, the heat-up rate R_{HVS} and the cool-down rate R_{KVS} should be between 30 K/min and 200 K/min, preferably between 30 K/min and 100 K/min. For the above parameters one preferably sets a holding time t_{VS} $5 \text{ min} \leq t_{VS} \leq 20 \text{ min}$.

[0043] For a cobalt-chromium alloy of the above-described composition one preferably chooses a presinter end temperature between 650°C. and 750°C. and a holding time between 50 min and 70 min at the presinter end temperature T_{VS} , whereby the heat-up rates are in the range between 10 K/min and 30 K/min.

[0044] In other words: various heat-up rates, presinter temperatures, holding times, and cool-down times may be chosen, which must be tuned relative to each other in such a way that they yield a surface porosity of the presintered green compact, which may also be referred to as blank, of between 16% and 22%.

[0045] In particular, the parameters should be tuned in such a way that they yield a surface porosity between 18% and 20%.

[0046] In particular it is intended that prior to debinding the green compact possess a porosity that is not more than 5% higher than the surface porosity after the presintering. In particular, this difference should not exceed 2%.

[0047] Consequently, the invention is also characterized by the fact that the compacted green compact that is used in the manufacture of the shaped body possesses a porosity between 16% and 27%, in particular between 18% and 22%.

[0048] If the porosity of the compacted green compact prior to debinding is always above the porosity of the presintered green compact, i.e. the blank, then the porosity may also be equal without leaving the scope of the invention.

[0049] A correlation exists between the heat-up rate, the presinter end temperature, the holding time, and the cool-down rate. For example for lower heat-up and cool-down rates one should select shorter holding times. The reverse also applies. All rates and holding times are decisively determined by the choice of presinter end temperature. Holding times shorter than 5 min are less suitable, since in particular for blanks of larger sizes a homogeneous heat penetration and presintering can not be ensured for shorter holding times. Holding times in excess of 60 min are also detrimental, since a longer dwell time favours the undesired formation of an oxidation layer.

[0050] Shaped bodies with a corresponding surface porosity possess excellent machining characteristics to allow production of in particular a dental prosthesis or part thereof. Highly precise machining can be performed with low tool wear.

[0051] After cooling the blank to room temperature, material-removing machining is performed to create the shaped body, whereby the processes of milling and grinding shall be named.

[0052] The final step performed is the one of sintering to full density.

[0053] It is in particular intended to use as metal powder a nickel-chromium-based or cobalt-chromium-based metal powder, in particular a dental alloy powder in the form of a cobalt-chromium alloy, preferably a cobalt-chromium-molybdenum alloy.

[0054] Preferred binding agents are wax- and/or cellulose-based binding agents.

[0055] In particular it is intended that for the purpose of achieving a surface porosity between 16% and 22%, in particular between 18% and 20%, the green compact be held at the presinter end temperature T_{VS} for a time period t_{VS} in accordance with the relation

$$t/2 < t_{VS} < 2t \quad (1)$$

t is computed using the equation:

$$t = t_0 \cdot \ln\left(\frac{c_0}{c}\right) \cdot \exp\left(\frac{T_0}{T_{VS}}\right) \quad (2)$$

[0056] with

[0057] c =desired surface porosity fraction of the green compact after presintering,

[0058] c_0 =surface porosity fraction of the green compact after debinding,

[0059] t_0 =material constant in min,

[0060] T_0 =material constant in Kelvin,

[0061] T_{VS} =presinter end temperature at the holding time t_{VS} with $650^\circ \text{C.} \leq T_{VS} \leq 1100^\circ \text{C.}$

[0062] c_0 , i.e. the surface porosity fraction of the green compact after debinding, can be determined by interpolation of measurement results, whereby on principle the following

relation applies: $c_0 - c \leq 5\%$, in particular $c_0 - c < 2\%$. Preferably, one should specify as additional condition: $c < c_0$.

[0063] The material constant t_0 can also be determined by interpolation of measurement results. When using cobalt-chromium-based metal powder or equivalent materials one finds $t_0 = 0.0125$ min.

[0064] When using cobalt-chromium-based or equivalent metal powder the corresponding material constant will be $T_0 = 11000$ K.

[0065] It should be noted that T_{VS} is to be entered in the above equation in Kelvin rather than in degree Celsius.

[0066] If the presintering in accordance with this relation is performed by holding the presinter end temperature for the duration of a holding time t_{VS} , then cool-down and heat-up rates should be chosen to be so short that the bulk of the presintering will take place during the holding time. In particular, during heating with a nearly constant heat-up rate R_{HVS} and cooling with a nearly constant cool-down rate R_{KVS} , the heat-up period between 650°C. and the presinter end temperature T_{VS} and the cooling period between the presinter end temperature T_{VS} and 650°C. should satisfy the following relation:

$$\frac{T_{VS} - 650^\circ \text{C.}}{R_{HVS}} + \frac{T_{VS} - 650^\circ \text{C.}}{R_{KVS}} < 2t, \quad (3)$$

whereby temperatures should be specified in degree Celsius. An additional condition that must be satisfied is that the presinter end temperature T_{VS} and the difference between c_0 and c are tuned relative to each other so that negative heat-up and cool-down rates are ruled out. Moreover, the maximum presinter end temperature T_{VS} should not exceed 1100°C.

[0067] Consequently, relation (3) represents a condition that must be satisfied by the specified parameters in order to be able to use a cobalt-chromium-based or equivalent metal powder to produce a presintered blank that possesses a surface porosity between 16% and 22%.

[0068] Surprisingly it has been realized that a presintered blank—irrespective of the existing surface porosity—not only can be machined with the desired accuracy, but that in addition after the final sintering an absolutely void-free veneering is possible irrespective of the residual surface porosity. The reason for this most likely is that the presinter steps and specified parameters according to the invention result in a residual surface porosity that after complete sintering does not form a connected system but exists in isolated occurrences. This does not only provide the option of a non-porous ceramic veneering, as already mentioned, but also ensures the necessary corrosion resistance. It could further be determined that the necessary dimensional accuracy can be achieved after machining and the subsequent dense-sintering, i.e. that the contraction is uniform and shape-preserving.

[0069] As a further development of the invention, it is intended that the mixture of alloy powder and binding agent be subjected to axial or isostatic pressing at a pressure p with $100 \text{ MPa} \leq p \leq 1,000 \text{ MPa}$, in particular with $200 \text{ MPa} \leq p \leq 600 \text{ MPa}$.

[0070] It is further preferred and intended that the green compact or the debinded green compact and the presintered green compact be heated under an inert-gas or forming-gas atmosphere or in vacuum. These measures ensure that only a very thin oxide layer accumulates on the surface,

which can be easily removed, e.g. by polishing, without the polishing after the complete-sintering being affected by the residual surface porosity.

[0071] The blank produced in this manner can subsequently be machined using in particular wet- or dry-working tools, whereby in particular with CAM technology the blank can be used to create any desired number of shaped bodies with corresponding dimensions, in particular of dental prostheses such as crowns or bridges, in particular by milling or grinding. Profiling also represents a viable option.

[0072] The invention is further characterized by a green compact intended for the manufacture of a dental prosthesis or part thereof, whereby the green compact is a presintered green compact made from a dental metal alloy and possesses a surface porosity of between 16% and 22%. It is particularly intended that the dental metal alloy be a nickel-chromium or cobalt-chromium alloy.

[0073] In particular it is intended that as dental alloy metal powder one use a mixture of 50% to 70% by weight of cobalt, 20% to 35% by weight of chromium, 0% to 10% of weight by molybdenum, 0% to 20% by weight of tungsten, less than 10% by weight of one or several other elements, in particular one or several elements from the group comprising manganese, silicon, nickel, beryllium, cadmium, lead, iron, aluminum, titanium, oxygen, nitrogen, and sulphur, with possible use of other elements with a weight fraction of less than 1% by weight, whereby the sum total adds up to 100%.

[0074] The invention is further characterized by the fact that one uses as dental alloy metal powder a mixture of 50% to 70% by weight of nickel, 20% to 35% by weight of chromium, 0% to 10% by weight of molybdenum, 0% to 20% by weight of tungsten, and less than 10% by weight of one or several other elements, in particular one or several elements from the group manganese, silicon, cobalt, beryllium, cadmium, lead, iron, aluminum, titanium, oxygen, nitrogen, sulphur, and possible other elements with a weight fraction of less than 1% by weight, whereby the sum total is 100% by weight.

[0075] Further details, advantages, and features are not only found in the claims and the characteristic features specified therein, but also in the following description of preferred embodiment examples.

[0076] In the manufacture of a dental prosthesis we used a metal alloy with the composition

[0077] 26 to 30% by weight of Cr,

[0078] 5 to 7% by weight of Mo,

[0079] in total between 0.01 and 1.5% by weight of at least one of

[0080] the elements Mn, Si, Fe, C, Ni,

[0081] remainder Co (61.5% to 68.99% by weight)

whereby the sum total is 100% by weight. To produce the powder, we at first produced, melted, and atomized a metal alloy. The mean grain size was in the region between 5 μm and 50 μm . Subsequently a wax-based binding agent was added, specifically approximately 2% by weight of the metal powder. The mixture produced in this manner was subjected to axial pressing to produce green compacts with a disk-shaped geometry. The diameter was approximately 10 cm and the thickness approximately 1 cm. Different dimensions are feasible.

[0082] This was followed by debinding. For this, the green compacts were at first heated to 450° C. using any desired heat-up rate. Heat-up above 450° C. occurred slowly,

whereby we chose 3 K/min as preferred heat-up rate. After reaching the temperature T_2 , which was approximately 600° C., the green compacts were held there for a time t_1 of approximately 10 min. These parameters are in principle sufficient to ensure elimination of the binding agent.

[0083] A green compact that had been subjected to debinding was subsequently presintered to create a CoCrMo blank. For this purpose, the green compact—in accordance with an alternative method—was rapidly heated to a temperature in the region of approximately 800° C. (heat-up rate in the region of 90 K/min) and was held at this temperature for a time period of approximately 20 min. This was followed by cooling, which initially took place at a constant rate and then at a lower rate.

[0084] This method satisfied the relations and conditions of equations (1), (2), and (3):

$$t = t_0 \cdot \ln\left(\frac{c_0}{c}\right) \cdot \exp\left(\frac{T_0}{T_{VS}}\right) \\ = 0.0125 \text{ min} \cdot \ln\left(\frac{0.20}{0.19}\right) \cdot \exp\left(\frac{11000\text{K}}{1073\text{K}}\right) \approx 18 \text{ min}$$

and thus $t/2 < t_{VS} < 2t$, in this case $9 \text{ min} < t_{VS} < 36 \text{ min}$ and

$$\frac{T_{VS} - 650^\circ \text{ C.}}{R_{HVS}} + \frac{T_{VS} - 650^\circ \text{ C.}}{R_{KVS}} < 2t$$

here:

$$\frac{800^\circ \text{ C.} - 650^\circ \text{ C.}}{90 \text{ K/min}} + \frac{800^\circ \text{ C.} - 650^\circ \text{ C.}}{90 \text{ K/min}} \approx 3.3 \text{ min} < 36 \text{ min.}$$

[0085] Micrographs of blanks produced in this manner showed an open surface porosity in a range between 16% and 22%, with a large number between 18% and 20%. These blanks were easy to work with, without any risk of high tool wear, which is known to have a detrimental effect on the precision of the machining.

[0086] The surface porosity allowed an uncomplicated processing using a CAM machine. For this, the blank was mounted in the CAM machine using a holding device. This was followed by material-removing machining, whereby regions of the blank were machined using a wet system and a dry system. When milling dry, the dust generated during the machining was removed by means of a class-H vacuum cleaner. The wet processes used were grinding processes. In particular when using the wet machining, no disadvantages were encountered.

[0087] The bodies machined from the blank possessed dimensions that took into account the contraction occurring during sintering to final density. After contraction we determined that the contraction took place uniformly and in a shape-preserving manner subsequently, the surface of the shaped body was polished or a ceramic veneer was attached in the usual manner, which could be achieved in an absolutely void-free manner, without the existing residual surface porosity causing any problems.

[0088] Even though the invention was explained using the example of dental prostheses, this shall not place any limitations on the invention.

What is claimed is:

1. A method of manufacturing a dental prosthesis, or a part thereof, the method comprising:

preparing a green compact from a cobalt-chromium alloy consisting of:

cobalt: 50 to 70% by weight;

chromium: 20 to 35% by weight;

molybdenum: 0 to 10% by weight;

tungsten: 0 to 20% by weight;

other elements: less than 10% by weight;

wherein a sum total of the elements adds up to 100% by weight;

heating the green compact;

debinding the heated green compact;

presintering the debinded green compact to form a blank having a surface porosity of from 16% to 22%;

processing the blank; and

sintering the processed blank to a final density to form the dental prosthesis, or the part thereof.

2. The method according to claim **1**, wherein the blank is processed by machining.

3. The method according to claim **1**, wherein the blank is processed using computer-aided manufacturing technology.

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