FORM 1

SPRUSON & FERGUSON

621427

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

APPLICATION FOR A STANDARD PATENT

Degussa Aktiengesellschaft, incorporated in the Federal Republic of Germany, of Rodenbacher Chaussee 4, D-6450 Hanau 1, FEDERAL REPUBLIC OF GERMANY, hereby apply for the grant of a standard patent for an invention entitled:

Process for the Production of a Sintered Denture

which is described in the accompanying complete specification.

Details of basic application(s):-

Basic Applic. No:	Country:	Application Date:
P3841902.5-24	DE	13 December 1988

The address for service is.-

Spruson & Ferguson
Patent Attorneys
Level 33 St Martins Tower
31 Market Street
Sydney New South Wales Australia

DATED this TWELFTH day of DECEMBER 1989

Degussa Aktiengesellschaft

By: J. Grein.

Registered Patent Attorney

TO: THE COMMISSIONER OF PATENTS
OUR REF: 112914
S&F CODE: 53300

SO11944 12/12/89

5845/3

88 227 DT

SPRUSON & FERGUSON

COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952

DECLARATION IN SUPPORT OF A CONVENTION APPLICATION FOR A PATENT

In support of the Convention Application made for a patent for an invention entitled:

Process for the Production of a Sintered Denture

I/~~We~~, Dr. Wolfgang Merk
[full name of declarant(s)]

of Bergstrasse 44, 6477 Limeshain 3, Federal Republic of Germany
[full address of declarant(s) - not post office box]

do solemnly and sincerely declare as follows:-

1. I am/~~We are~~ authorised by Degussa Aktiengesellschaft, the applicant for the patent, to make this declaration on its behalf.

2. The basic application as defined by Section 141 of the Act was made in Federal Republic of Germany on 13 December 1988 by Degussa Aktiengesellschaft

3. Werner Groll, Angela Klaus and Thomas Lange, of, respectively, Gartenstrasse 5, D-8755 Alzenau-Horstein; Feldstrasse 9, D-6450 Hanau 8; and Gerauer Strasse 86 A, D-6000 Frankfurt/M 71, all in the Federal Republic of Germany, are the actual inventors of the invention and the facts upon which the applicant is entitled to make the application are as follows:

   The said Applicant is the assignee of the actual inventors.

4. The basic application referred to in paragraph 2 of this Declaration was the first application made in a Convention country in respect of the invention the subject of the application.

DECLARED at Frankfurt/Main this 9th day of january 1990

Dr. Wolfgang Merk
Signature of Declarant

TO: THE COMMISSIONER OF PATENTS
AUSTRALIA

cjd:5535D

(12) PATENT ABRIDGMENT (11) Document No. AU-B-46136/89
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 621427

(54) Title
PROCESS FOR THE PRODUCTION OF A SINTERED DENTURE

International Patent Classification(s)
(51)[5] **A61C 005/10** **A61C 013/08** **A61K 006/04**

(21) Application No. : **46136/89** (22) Application Date : **12.12.89**

(30) Priority Data

(31) Number (32) Date (33) Country
3841902 **13.12.88** **DE FEDERAL REPUBLIC OF GERMANY**

(43) Publication Date : **21.06.90**

(44) Publication Date of Accepted Application : **12.03.92**

(71) Applicant(s)
DEGUSSA AKTIENGESELLSCHAFT

(72) Inventor(s)
THOMAS LANGE; ANGELA KLAUS; WERNER GROLL

(74) Attorney or Agent
SPRUSON & FERGUSON , GPO Box 3898, SYDNEY NSW 2001

(56) Prior Art Documents
AU 580687 62424/86 A61C 5/10 13/08 A61K 6/64

(57) Claim

1. A process for the production of a sintered denture having a metallic structural matrix consisting of a noble metal powder mixture having a bimodal or multimodal particle size distribution and predominantly spherical form, which mixture is stirred with a mixing liquid consisting essentially of water to give a slip which can be modelled and can be densified by expulsion of the mixing liquid, by means of which the denture is modelled on a cast of the teeth which will be provided and which serves as a firing support, using customary techniques for dental ceramics, and is then sintered on the model in a graphite box or under an inert gas, characterised in that the denture is modelled and initially dried in air for 5 to 25 minutes is heat-treated for 5 to 45 minutes between 100°C and 400°C, then heated to 800°C with a mean temperature increase of 50 to 300 K/min and brought, with a mean temperature increase of 20 to 200 K/min, in air or under an inert gas, in the graphite box, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa, without graphite box, to the sinter temperature T, which is between ($T_{solidus}$ -200°C) and ($T_{solidus}$ -70°C), $T_{solidus}$ being the solidus temperature of the sintered alloy, and sintered at this temperature for 5 to 45 minutes in the air or under an inert gas, in the graphite box, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa, without graphite box, and then cooling is carried out between 900°C and room temperature under an inert gas or, a vacuum of 50 to 1 HPa when a graphite box is used, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa when no graphite box is used.

S & F Ref: 112914

FORM 10

COMMONWEALTH OF AUSTRALIA 621427

PATENTS ACT 1952

COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE:

Class Int Class

Complete Specification Lodged:
Accepted:
Published:

Priority:

Related Art:

This document contains the amendments allowed under Section 83 (2) by the Supervising Examiner on

.....

and is correct for printing

: : .....

Name and Address of Applicant: Degussa Aktiengesellschaft
Rodenbacher Chaussee 4
D-6450 Hanau 1
FEDERAL REPUBLIC OF GERMANY

Address for Service: Spruson & Ferguson, Patent Attorneys
Level 33 St Martins Tower, 31 Market Street
Sydney, New South Wales, 2000, Australia

Complete Specification for the invention entitled:

Process for the Production of a Sintered Denture

The following statement is a full description of this invention, including the best method of performing it known to me/us

Process for the production of a sintered denture

Abstract

A denture having a very high density and produced by sintering a noble metal powder mixture having a bimodal or multimodal particle size distribution is obtained if drying and sintering are carried out in a certain manner. For this purpose, the dry denture produced from a slip is heat-treated for 5 - 45 minutes at between 100 and 400°C, then heated to 800°C with a mean temperature increase of 50 to 300 K/min and brought to the sinter temperature at 20 to 200 K/min. The said sinter temperature is between ($T_{solidus}$) -200°C) and ($T_{solidus}$ -70°C), $T_{solidus}$ being the solidus temperature of the sintered alloy. Cooling must be carried out in vacuo or in an inert gas.

Process for the production of a sintered denture

Description

The invention relates to a process for the production of a sintered denture having a metallic structural matrix consisting of a noble metal powder mixture or a noble metal alloy powder mixture having a bimodal or multimodal particle size distribution and predominantly spherical form, which mixture is stirred with a mixing liquid consisting essentially of water to give a slip which can be modelled and can be densified by expulsion of the mixing liquid, by means of which the denture is modelled, using customary techniques for dental ceramics, on a cast of the teeth which will be provided and which serves as a firing support, and is then sintered on the model in a graphite box or under an inert gas.

The production of a metallic denture as a prosthesis in tooth diseases or after loss of one or more teeth, such as, for example, inlays and bridges and crowns which are unfaced or can be faced with ceramic or plastic, is usually carried out using the so-called "wax burnout method", a precision casting technique which ensures high dimensional stability.

The advantages of the crowns and bridges thus produced, in addition to the dimensional stability, are in particular high strength and ductility, which must be guaranteed in the case of relatively large bridge structures in order to avoid overload breakages under an excessive load. On the other hand, the process itself is very time-consuming and requires a great deal of materials and apparatus. The necessity of using sprue channels and sprue cones results in the use of a substantially larger amount of material relative to the weight of the cast object, which material may lead

to changes in the alloy properties on a repeated use and - if it is not reused - remains as scrap. Another disadvantage of this technique is that, in the event of errors in the cast object, repair is not possible; instead, the entire production process must be repeated, starting with the wax modelling.

German Offenlegungsschrift 1,915,977 describes a process for the production of a metallic denture by sinter technology, in which process the denture is modelled on a cast of the teeth which will be supplied, using a paste which consists of metal powder having a particle size of between 2 and 25 μm and a binder acting as an adhesive, and is then sintered. The disadvantage of the process is the poor densification properties of the pastes described, since the binder acting as an adhesive cannot be expelled by compression processes, such as rippling or vibrating. Since, furthermore, a powder fraction can be used as the starting material, the density of the green compact is low. The result of this is that a great amount of shrinkage and accordingly an unacceptable inaccuracy of fit occur during sintering. The use of very fine powders between 2 and 25 μm ensures very high sinter activity but also entails high production costs.

The process, described in U.S. Patent 4,661,071, for the production of a metallic denture by sinter technology uses powder having a size of 5-90 μm - made into a paste with a suitable binder - for modelling the metallic denture on a cast of the teeth which will be supplied. A special castable and self-hardening die material which has to be fired before application of a metal powder at 1400°C - 1460°C is required for the production of the cast.

Since conventional firing kilns used for dental ceramics reach maximum temperatures up to about 1200°C, a special

kiln is required for this purpose. A liquid-phase sinter process in a vacuum of 1 HPa to 10^{-2} HPa is used for sintering the metal powder. Since conventional firing kilns used for dental ceramics do not achieve this vacuum, a special vacuum kiln is also necessary. Kilns with maximum temperatures of up to 1400°C and kilns that ensure good vacuum at high temperatures are very much more expensive than firing kilns normally used for ceramics, so that the use of this process by a dental laboratory technician necessitates high investment in equipment. The use of the liquid-phase sinter process also gives rise to problems with regard to the dimensional stability during sintering. To achieve densification as rapidly as possible, through a rearrangement of the solid constituents (particles), a liquid-phase fraction of at least 30-35% is necessary (R.M. German, Liquid Phase Sintering, Plenum Press, N.Y., pages 4, 6 and 80). Analogously to the behaviour of dental porcelain bonded to metal, rounding or flattening of very delicate details, for example of an occlusal surface, is to be expected, which may lead to problems with regard to contact points and necessitates considerable finishing in certain circumstances.

German Offenlegungsschrift 3,532,331 describes a process for the production of a metallic denture by sinter technology, which process, using a powder mixture having a multimodal size distribution, which is converted with water into a modellable and densifiable slip, achieves a specific high density of the green compact, and accordingly shrinkage during sintering remains small. This is advantageous for achieving good accuracy or fit. The use of water as a mixing liquid and of a consistency which is very similar to that of dental facing ceramic slips permits additional densification by expulsion of the liquid by a technique customarily used for dental ceramics (rippling, etc.). The sinter process can be carried out without great expense in a firing kiln

conventionally used for dental ceramics. This may be achieved on the one hand by using a graphite box in which the modelled denture, to be sintered, is located. This graphite box is placed in a firing kiln conventionally used for dental ceramics and ensures that non-noble metal constituents of the alloy are protected from oxidation at the sinter temperature. On the other hand, adequate reduction of the oxygen partial pressure can also be achieved by passing inert gas into the ceramics firing kiln. After sintering, the denture is cooled in air in the graphite box.

The process using powder mixtures of atomised, predominantly spherical noble metal alloys and precipitated, very fine, predominantly spherical noble metal powders has disadvantages and these were found to be that, with the sinter parameters described there, no maximum densities are achievable in the sintered state, and that, particularly in the case of repeated sintering, the density of the sintered denture clearly decreases. Repeated sintering may however be necessary in the production of bridges in several steps or for border corrections.

It was therefore the object of the present invention to develop a process according to the precharacterising clause of Patent Claim 1, by means of which optimum densities in the sintered state can be achieved, particularly in the case of repeated sintering, using conventional apparatus and techniques.

According to a first embodiment of the present invention there is provided a process for the production of a sintered denture having a metallic structural matrix consisting of a noble metal powder mixture having a bimodal or multimodal particle size distribution and predominantly spherical form, which mixture is stirred with a mixing liquid consisting essentially of water to give a slip which can be modelled and can be densified by expulsion of the mixing liquid, by means of which the denture is modelled on a cast of the teeth which will be provided and which serves as a firing support, using customary techniques for dental ceramics, and is then sintered on the model in a graphite box or under an inert gas, characterised in that the denture is modelled and initially dried in air for 5 to 25 minutes is heat-treated for 5 to 45 minutes between 100°C and 400°C, then heated to 800°C with a mean temperature increase of 50 to 300 K/min and brought, with a mean temperature increase of 20 to 200 K/min, in air or under an inert gas, in

the graphite box, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa, without graphite box, to the sinter temperature T, which is between ($T_{solidus}$ -200°C) and ($T_{solidus}$ -70°C), $T_{solidus}$ being the solidus temperature of the sintered alloy, and sintered at this temperature for 5 to 45 minutes in the air or under an inert gas, in the graphite box, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa, without graphite box, and then cooling is carried out between 900°C and room temperature under an inert gas or, a vacuum of 50 to 1 HPa when a graphite box is used, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa when no graphite box is used.

Advantageous embodiments of the process for the production of a sintered denture are evident from the measures of the subclaims.

The modelled and densified denture is dried in air (5 to 25 minutes) and then placed, for example, in a graphite box and heat-treated in a temperature range of between 100° and 400°C for 5-45 minutes. Thereafter, it is heated to 800°C with a mean temperature increase of 50 to 300 K and heated, at 20 to 200 K/min, in air or under an inert gas, from the preheating temperature to the sinter temperature T, which is between ($T_{solidus}$)-200°C) and ($T_{solidus}$ -70°C), $T_{solidus}$ being the solidus temperature of the sintered alloy. At this temperature, sintering is carried out for 5 to 45 minutes in air or under an inert gas, followed by cooling in the temperature range below 900°C in a vacuum of 1-50 HPa or under an inert gas. After cooling, the denture can be removed from the graphite box.

Heating to the sinter temperature and sintering in the graphite box are preferably carried out in air, and cooling in the temperature range below 900°C is preferably carried out in vacuo. This is particularly advantageous because it uses apparatus usually present in the dental laboratory and because of the cost.

Preferred times for the heat treatment between 100°C and 400°C and for sintering are 5-25 minutes and 10-30 minutes, respectively.

If a graphite box is not employed, the process must be carried out under an inert gas, and the oxygen partial pressure should be less than 5×10^{-2} HPa. This is ensured, for example, when technical grade argon is used. This can be realised by relatively simple conversion of a conventional ceramics kiln.

A particular advantage of this process is that a sufficiently high density coupled with closed porosity is achieved using the stated parameters - in particular for the vacuum cooling - even after repeated sintering.

In the process according to the invention, a mixture of predominantly spherical powders having a bimodal or multimodal distribution is used. This powder mixture is mixed, manually or with a stirrer suitable for this purpose, with a mixing liquid which predominantly consists of water but which may also contain small added amounts of electrolytes, such as, for example, strontium chloride, copper chloride or ammonium nitrate, monohydric or polyhydric alcohols, cellulose or polyethylene glycol, to give a slip whose consistency and modelling properties correspond to those of conventional dental or facing ceramics.

The slip thus prepared is applied to a cast of the teeth, which will be supplied, which is resistant to high temperatures, by a technique customarily used for dental ceramics, and is densified by known techniques (for example shaking with the rippling part of a casting instrument, ultrasound, etc.). During this procedure, the liquid comes to the surface and is sucked up using a cloth or dried in a warm air stream. Before application of the slip, it is advisable to impregnate the die with liquid or to insulate it, so that the moisture is not withdrawn from the slip by the die.

The green compact densified to high bulk density is first allowed to stand in the air on the cast for about 5-25 minutes to dry off slowly. This can also be effected on the top plate of a ceramics firing kiln, which normally has a temperature of $\geq$ 50°C. The denture on the cast is then placed in a graphite box which encloses it completely.

The denture is then heat-treated in a kiln at a temperature of between 100 and 400°C for 5-45 minutes. This treatment serves to remove any moisture or organic impurities still present. If this heat treatment is not

carried out before the actual sintering, cracks are formed in the crown walls. If the temperature of 400°C is exceeded and the temperature is maintained for the stated times, the result is a dramatic decrease in the density of the sintered denture.

After the heat treatment, the graphite box containing the denture is heated to the sinter temperature. To achieve a sufficiently high sinter density, it is necessary to bridge the temperature range between 400°C and 800°C at a mean heating rate of more than 50 K/min, in particular 50 to 300 K/min.

Lower heating rates lead to a reduced density. Above 800°C, the mean heating rate is advantageously chosen between 20 and 200 K/min, which leads to acceptable times even when the total sinter time is taken into account.

The density of the sintered denture is dependent on the sinter temperature T. Surprisingly, it has now been found that the density has a maximum in a temperature range between ($T_{solidus}$ -200°C) and ($T_{solidus}$ -70°C), the exact position depending in turn on the specific alloy. The maximum extends over a temperature range of 20-50°C, a very sharp decrease in the densities being observed after the maximum has been exceeded. Two typical curves for two powder mixtures are shown in the Figure.

Sintering of the denture in the graphite box can be carried out in air or under an inert gas. Sintering is preferably carried out in air since the results obtained are not inferior to those under inert gas and the cost of the apparatus is comparatively lower.

The sinter time is 5-45 minutes, the maximum density being achieved as a rule with sinter times of only

between 10 and 30 minutes.

It is true that cooling of the sintered denture in the graphite box in air leads to high densities after the first sinter step. However, for the production of bridges or for the correction of the occlusion, of the contact points or of the border seal, a second sinter step may have to be carried out subsequently. In spite of the same sinter cycle, the density of the sintered denture decreases dramatically when sintering is carried out twice.

Surprisingly, it has now been found that, by cooling the denture present in the graphite box in a vacuum of between 50 and 1 HPa, the decrease in density on repeated sintering can be prevented. It is critical that the required vacuum be present before the denture is cooled to a temperature of < 900°C.

Sintering without a graphite box is also possible, but in this case sintering must be carried out under an inert gas, the above-mentioned temperatures and times also being applicable.

The oxygen partial pressure in the inert gas must no exceed 5×10^{-2} HPa if a sufficiently high density is to be achieved. In this case, cooling must also be carried out under an inert gas.

The following Examples are intended to illustrate the process according to the invention in more detail:

1. The slip, consisting of the powder mixture 1 (Table 2), is converted to a consistency suitable for modelling by adding a mixing liquid consisting of 98% of H_2O and 2% of polyethylene glycol. The slip is now applied with a brush to a die impregnated with liquid.

The crown is completely modelled and the form is checked repeatedly in the articulator.

The slip is densified by rippling with the carving instrument. The liquid emerging from the surface is sucked up with a cloth. The good stability of the slip makes it possible to build up details of the occlusal surface, such as cusps or furrows. After densification (moisture no longer emerges from the surface), the surface can be finished by scraping or cutting, so that even fine fissures can be created prior to sintering. The completely modelled crown remains on the die during the entire sinter process. For drying, it is placed on the top plate of a ceramics firing kiln and introduced into a graphite box after 15 minutes. The graphite box consists of a graphite base with an appropriate holder for the die and a cup-shaped graphite lid. The graphite box with the modelled crown is placed in a kiln, which is simultaneously heated to 300°C. After 15 minutes, the graphite box is placed in a ceramics firing kiln preheated to 1000°C, and the temperature is increased to 1050°C. The sinter temperature of 1050°C is reached after 5 minutes, corresponding to a mean heating rate of 150 K/min. The sinter temperature of 1050°C is 160°C below the $T_{solidus}$ - temperature of 1210°C (see Table 2). After 20 minutes, the graphite box is removed from the kiln and cooled in air. Some relatively small corrections have to be made on the occlusal surface and in a border region. The regions to be corrected are then applied to the sintered crown, as described above. Thereafter, the crown is sintered again using the sinter cycle already described

above. When the density is checked, a value of 14.2 g/cm^3 is obtained. The crown is too large because cooling is carried out in air.

2. Another crown is produced analogously to Example 1. After the end of sintering at 1050°C, however, the graphite box with the crown is transferred to the cooling chamber which can be evacuated. Immediately after the graphite box has been transferred, the cooling chamber is evacuated to a vacuum of about 50 HPa. The cooled sample can be removed after about 15 minutes. As described in Example 1, a few small corrections have to be carried out. The crown is resintered and once again cooled in vacuo. The die material is blasted using a sand-blasting unit, and the density is determined. It is now 16.1 g/cm^3, and the pores are closed. The crown is finished and polished. The marginal leakage on the master cast is on average 50 µm.

3. For the production of inlays, the powder mixture 2 (Table 2) is used since this alloy is yellow and is preferred by many patients. In addition, it has a lower yield strength (0.2% offset) and greater ductility. This permits easier finishing of the borders in the month. The production is analogous to the process described under Example 2.

However, the sinter cycle is modified somewhat. After drying in the air, the inlay present on the die (in the graphite box) is placed on the firing table of an opened ceramics firing kiln preheated to 700°C. The firing table is at a temperature of about 250°C. After 9 min, the firing table moves in automatically and the kiln heats up to a sinter temperature of 940°C. After a further 15 min, the sample can be removed from the kiln, transferred to the cooling chamber and cooled there under a vacuum of 50 HPa. The die material is blasted, and the inlay is finished, placed on the master die and polished. The density is 17.1 g/cm^3 and the pores are closed. As a result of polishing, the pores at the surface are also closed. The marginal leakage is about 50 µm. The rate of temperature change between 400° and 800°C is on average about 120 K/min here, and the rate of temperature change between 800°C and the sinter temperature is on average 100 K/min. $T_{solidus}$ = 1040°C (Table 2).

Palladium alloys can also be processed in the same manner.

Table 1 gives the composition of the alloys used in the Examples, and their preparation, particle shape and particle size, while Table 2 shows the composition of the powder mixtures used in the Examples.

The Figure shows the dependence of the sinter density of dentures produced from the powder mixtures contained in Table 2 on the sinter temperature.

KWK:0732y

Table 1: Examples of powders used

	Composition (% by weight)	Particle shape	Preparation	Particle size/μm
Alloy 1	Au 65 Pt 15 Pd 13 In 2.5 + < 2% of each additive	Predominantly spherical	Atomisation	-*
Alloy 2	Au 87 Pt 11 + < 2% of each additive	Predominantly spherical	Atomisation	-*
Au powder 1	Au	"	Chemical precipitation	< 5
Au powder 2	Au	"	Chemical precipitation	< 10

* According to sieve fraction, always < 100 μm

Table 2:

	Component 1			Component 2			Component 3			$T_{solidus}$*
	Powder	Content %	PS**/µm	Powder	Content %	PS/µm	Powder	Content %	PS/µm	
Powder mixture 1	Alloy 1	80	< 63	Au 1	20	< 5				1210°C
Powder mixture 2	Alloy 2	85	< 50	Au 1	13	< 5	Au 2	2	< 10	1040°C

* $T_{solidus}$ of the sintered alloy

** PS = Particle size

The claims defining the invention are as follows:

1. A process for the production of a sintered denture having a metallic structural matrix consisting of a noble metal powder mixture having a bimodal or multimodal particle size distribution and predominantly spherical form, which mixture is stirred with a mixing liquid consisting essentially of water to give a slip which can be modelled and can be densified by expulsion of the mixing liquid, by means of which the denture is modelled on a cast of the teeth which will be provided and which serves as a firing support, using customary techniques for dental ceramics, and is then sintered on the model in a graphite box or under an inert gas, characterised in that the denture is modelled and initially dried in air for 5 to 25 minutes is heat-treated for 5 to 45 minutes between 100°C and 400°C, then heated to 800°C with a mean temperature increase of 50 to 300 K/min and brought, with a mean temperature increase of 20 to 200 K/min, in air or under an inert gas, in the graphite box, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa, without graphite box, to the sinter temperature T, which is between ($T_{solidus}$ -200°C) and ($T_{solidus}$ -70°C), $T_{solidus}$ being the solidus temperature of the sintered alloy, and sintered at this temperature for 5 to 45 minutes in the air or under an inert gas, in the graphite box, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa, without graphite box, and then cooling is carried out between 900°C and room temperature under an inert gas or, a vacuum of 50 to 1 HPa when a graphite box is used, or under an inert gas with an oxygen partial pressure below 5×10^{-2} HPa when no graphite box is used.

2. A process for the production of a denture according to Claim 1, characterised in that the heat treatment is carried out at between 100°C and 400°C for 5 to 25 minutes and sintering is carried out for 10 to 30 minutes.

3. A process for the production of a denture according to Claim 1 or 2, characterised in that heating to the sinter temperature, sintering and cooling are carried out under a protective gas, and the oxygen partial pressure in the furnace space must be less than 5×10^{-2} HPa.

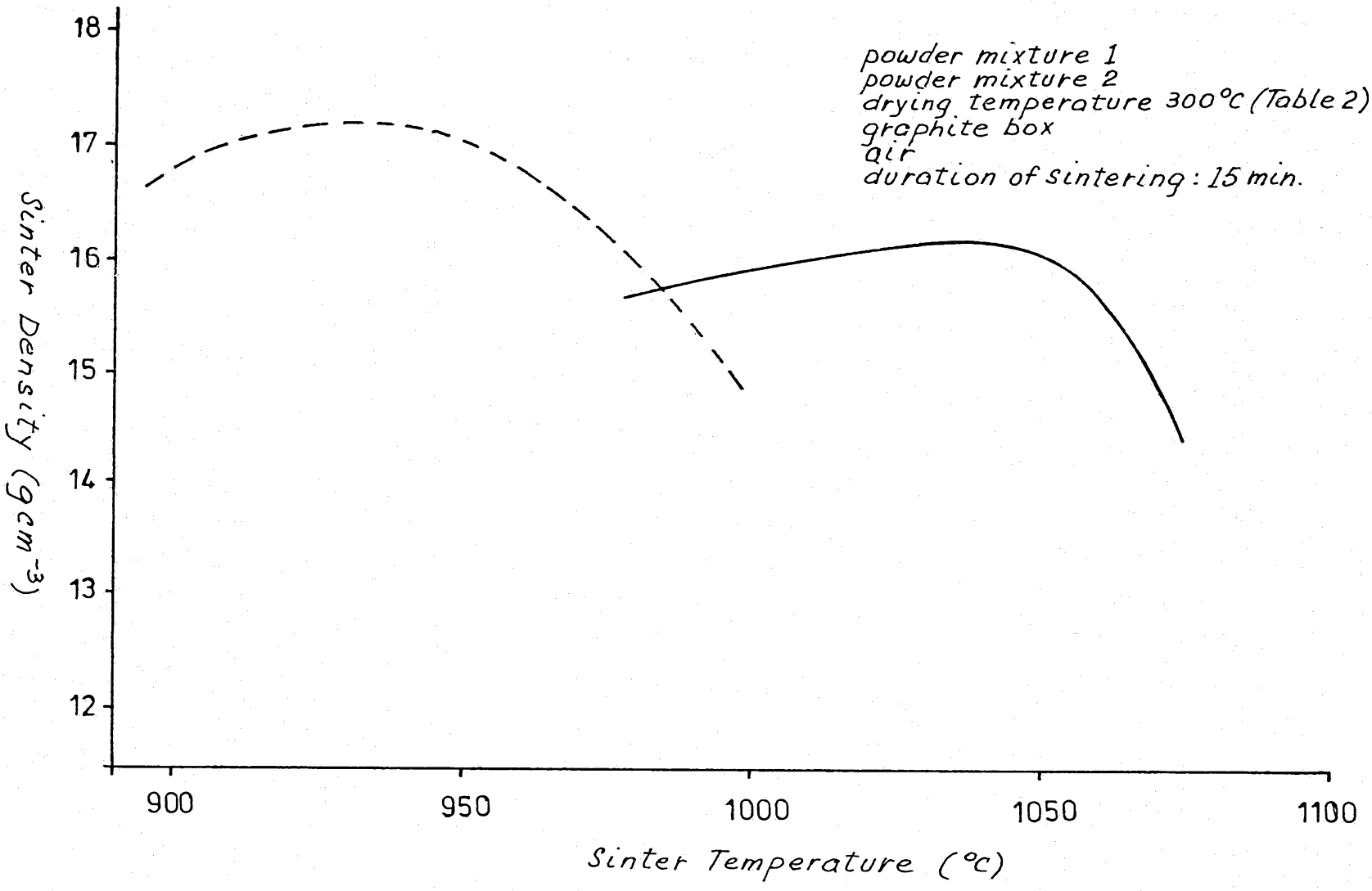
4. A process for the production of a sintered denture substantially as hereinbefore described with reference to the Examples.

5. A sintered denture whenever prepared by the process according to any one of claims 1 to 4.

DATED this TWENTY-THIRD day of DECEMBER 1991

Degussa Aktiengesellschaft

Patent Attorneys for the Applicant
SPRUSON & FERGUSON


18
17
16
15
14
13
12
Sinter Density (gcm⁻³)
900
950
1000
1050
1100
Sinter Temperature (°C)
powder mixture 1
powder mixture 2
drying temperature 300°C (Table 2)
graphite box
air
duration of sintering: 15 min.

Figure