Titanium Dental Copings Prepared by a Powder Metallurgy Method: A Preliminary Report

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Purpose: The purpose of this study was to determine if the Procera pressed-powder method can be used to fabricate titanium copings. **Materials and Methods:** Commercially pure titanium powder was used to prepare the copings. The powder was pressed onto an enlarged tooth preparation die of aluminum using cold isostatic pressing. The outer shape of the coping was formed using a Procera milling machine, and the copings were vacuum sintered. **Results:** Titanium copings could be prepared using this method. The density of the sintered copings reached 97% to 99%+ of theoretic density, and the copings showed ductile behavior after sintering. Enlarging the tooth preparation die to compensate for the sintering shrinkage could optimize the final size of the copings. **Conclusion:** Ductile and dense titanium dental copings can be produced with powder-metal processing using cold isostatic pressing, followed by milling and sintering to final shape. The forming technique has, if properly optimized, a potential of becoming a more cost-efficient production method than spark erosion. *Int J Prosthodont 2004;17:11–16.*

The dental coping is a substructure onto which veneering porcelain is fused to create a dental crown, which is cemented on a residual prepared dental root. Various metals have been used to create the dental coping.[1] More recently, titanium has been used because it is considered to have good biocompatibility and mechanical properties.[2] Most research involving the use of titanium has focused on finding a method for casting this metal.[3] Difficulty has been experienced in casting titanium because of its high melting point and the very high reactivity of the titanium melt.[4,5] Several commercial systems have been used.[6–8] There have been three major obstacles to overcome when developing a method for casting titanium.[9] It is important to avoid the creation of an alpha layer, eliminate porosity, and achieve a good fit of the casting onto a die. Work on these problems has been reported.[10–12]

The fabrication of a titanium coping has also been investigated using the computer-aided design/manufacturing (CAD/CAM) approach. One example is the Digitizing Computer System (DCS Production).[13] Another commercially available system is the Procera system (Nobel Biocare), from which the spark-erosion process was developed. Carbon electrodes were used to spark erode the inner profile of the titanium coping.[14] This approach has proven successful in the fabrication of titanium copings.[15] Today, the Procera system also manufactures all-ceramic copings using pressed-powder technology.[16–18] The copings shrink to their intended dimensions during the sintering process. The powder-based methods for making aluminum oxide dental copings have undergone clinical tests.[19]

Adaptation of the current Procera CAD/CAM method using pressed-powder technology in the fabrication of titanium copings has several advantages compared to the original Procera method. The original method required at least four milling operations, the outer surface milling of the titanium blank and milling of three electrodes for spark erosion of the inner form. The powder

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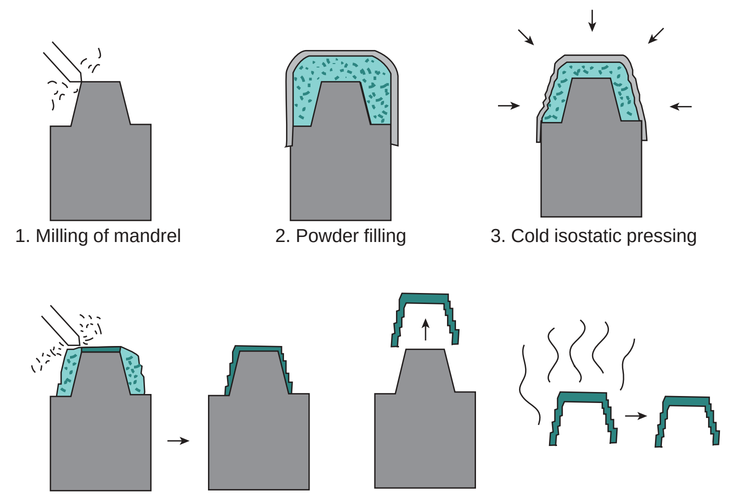


Fig 1 Fabrication of a titanium dental coping using powder metallurgy.

method needs only two milling operations, one for the enlarged refractory die and one for the outer contour of the coping. Using the spark-erosion process, the milling of the outer contour of the coping is performed on a titanium blank. With the pressed-powder method, the powder is compacted onto the surface of the refractory die and then milled before the sintering, which speeds up the process and reduces wear of the milling tools. The spark-erosion process requires careful alignment of the carbon electrodes with the titanium blank in fabricating the coping. With powder technology, the alignment is not critical in the same way. The spark-erosion process is a time-consuming and relatively costly operation. The pressed-powder technology could become a very cost-effective operation. The sintering process does take time; however, when the process is used commercially in creating multiple copings at the same time, it could become cost effective. Several approaches have been reported in the literature using powder technology in dentistry.[20–23]

The success realized with the current Procera method in creating all-ceramic copings has led to this investigation, which addressed the research question, "Can the Procera pressed-powder method be used to fabricate titanium copings?"

Materials and Methods

Tooth Preparation

A tooth-preparation die was made using conventional dental techniques and scanned using a mechanical scanner (Procera). The three-dimensional digitized die data were recorded in a computer. A computerized 3-D shape of the preparation was generated from the data on the computer screen. In forming the inner surface of the coping, titanium powder was to be pressed onto a refractory die having a shape that was enlarged from the form of the original preparation to compensate for shrinkage of the titanium during sintering. The refractory die could be made of various materials; for this investigation, aluminum was used. One end of an aluminum bar was milled with a milling machine (Procera) to the enlarged shape of the preparation die. A commercially pure fine-grained hydride/dehydride titanium powder was used (–270 mesh, < 53 µm grain size) in the pressed-powder method. To prevent contamination, no organic pressing aid was added (the powder was not granulated).

Forming and Sintering

A few grams of titanium powder were placed in a flexible latex cover around the refractory die at the end of the aluminum bar with the preparation surface. The powder was pressed onto the refractory die using cold isostatic pressing (Fig 1). The fluid pressed on the flexible latex cover, which in turn pressed the powder body isostatically against the die. Several copings were pressed at various pressures ranging from 300 to 900 MPa (holding time 60 seconds). The flexible latex cover was removed after pressing, and the powder body had become firmly attached to the die during pressing. The inner surface of the coping (the surface of the powder body against the die) had now been shaped. The green (unsintered) body was hard and

Table 1 Density and Sintering Shrinkage of Titanium Made by Powder Metallurgy*

Cold isostatic pressing pressure (MPa)	Green density (%)†		Sintered density, 1,200°C 2 h (%)†		Linear sintering shrinkage (%)
	Mean	SD	Mean	SD	
300	77.1	0.16	97.2	0.17	7.4
500	84.4	0.03	98.5	0.03	5.0
700	89.0	0.06	99.1	0.07	3.5
900	91.8	0.06	99.6	0.07	2.7

*Theoretic density used for calculations was 4.506 g/cm^3.
†Every density value is a mean with the standard deviation (SD) of three similar samples.

showed a significant green strength (it could not easily be broken by hand). The individual metal grains interlocked with each other and were deformed plastically and pressed into each other during the pressing.

The die with the firmly attached powder body was repositioned in a Procera milling machine, and the outer surface of the powder body was milled to give the shape of the outer surface of the enlarged coping. After this operation, the shaping of the coping was finished, and the green body had been transformed into a coping attached to the die. The thickness of the coping was approximately 0.6 mm. The coping was separated from the die by immersing the die in liquid nitrogen for 60 seconds, whereupon the coping could easily be removed. The difference in linear thermal expansion coefficient between aluminum and titanium made the aluminum die shrink more than the titanium coping, facilitating separation of the two.

The copings were placed on molybdenum foil in a crucible and placed into a graphite resistance furnace. The sintering temperature used was 1,200°C, with a 2-hour dwell time, after which the copings were cooled in the furnace to room temperature. The heating rate was 20°C/min up to 800°C and 10°C/min up to 1,200°C. The sintering was performed in a vacuum (0.05 mbar, 5 Pa), which was maintained during the entire sintering process.

Density and Shrinkage

The green (unsintered) and sintered density of the titanium material were measured with the water-intrusion method on cylinder-shaped samples (2 g each) made by cold isostatic pressing and sintering using the same parameters (pressing, sintering) as for the copings (Table 1). The sintering shrinkage was calculated from the density data according to the following equation:

$$\Delta L/L_0 = 1 - (\rho_g/\rho_s)^{1/3}$$

where $\Delta L/L_0$ = linear shrinkage; ρ_g = green density; and ρ_s = sintered density.

Visual Appearance

After sintering, the visual appearance of the copings was described. Fitting tests were performed on the original tooth preparation die to get a view of the general fit, but proper fitting tests will be conducted in future work when the process has been optimized.

Preliminary Ductility Test

Measuring ductility directly on copings is difficult because of the small size and irregular form. Rods or cylinders made by the same method may give a different result than the material in a coping because the surface-to-volume ratio is much higher for a coping. That may influence the properties during sintering. Therefore, the ductility of the sintered copings was tested by deforming the copings with hard blows on the edge of the coping to observe if cracks appeared where the sintered metal was deformed.

SEM Observation

Some of the sintered copings were cut in half, and the cross-sections were polished and chemically etched and examined using a scanning electron microscope (SEM).

Results

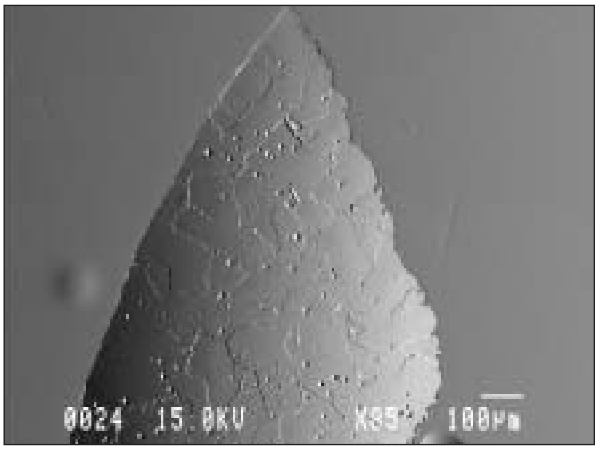
Density and Shrinkage

The level of green density of the titanium depended on the pressure used. An increase in green density was observed during the whole pressing range from 300 to 900 MPa. After 300 MPa and 900 MPa, the green density was 77% and 92%, respectively, of theoretic density. The sintered (1,200°C, 2 hours) densities reached 97% to 99%+, and the level depended on the pressure used (Table 1). The linear sintering shrinkage (Fig 2) was about 7% for 300 MPa and about 3% for 900 MPa. The density levels between

Fig 2 *(left)* Titanium dental copings made by powder metallurgy (green body, *left,* and sintered coping, *right*).

Fig 3 *(below left)* Sintered coping deformed by a blow. A plastic deformation without cracks shows that the sintered material is ductile.

Fig 4 *(below right)* Cross-section of a sintered coping. The grains are well-sintered, and the upper edge is sharp.



99.0% and 100.0% are somewhat uncertain because of the nature of the water-intrusion method, but they were at least 99%.

Visual Appearance

A few differences were observed when comparing sintered copings with copings produced by the spark-erosion method. The sintered copings showed silvery bright, glittering surfaces (typical of sintering) on both the inside and the outside, whereas the spark-eroded copings were more shiny on the outside (as a result of the milling) but darker and uneven on the inside (because of the spark erosion). The outer surface of the sintered coping was more uneven because it was milled in the green state, thus roughening the surface somewhat. The inner surface was smoother than a spark-eroded coping because the green body was pressed against the relatively smooth die surface. The sintered copings gave a slightly brighter appearance. The fit could be improved by optimization of the process, which will be performed in future work.

Preliminary Ductility Test

The titanium material in the sintered copings was ductile. The edges of a sintered coping were deformed plastically when hit with a hard object (Fig 3). The blows deformed the coping without causing visible cracks, showing that the sintered coping was ductile, not brittle.

SEM Observation

The SEM analysis of polished and etched cross-sections of sintered copings showed that the grain size of the sintered coping was around 100 μm, compared to around 50 μm for the typical grade 2 titanium material used for spark-eroded copings. The grain boundaries of the sintered coping were dense, and no cracks or open porosity were detected. Some closed porosity remained (around 1 vol% to 3 vol%, depending on the density), represented as a few, small, fairly round pores (approximately 10 μm in diameter), evenly spread in the material. The edge of the sintered coping had a good level of sharpness (Fig 4), which is desirable in typical copings made by spark erosion.

Discussion

Several factors could influence the results and quality of the sintered copings, including type of titanium powder, grain size, level of isostatic pressure, sintering temperature, and time and other sintering- and milling-related factors. Powder types with irregular grains will give a higher green strength compared to round powder grains. Different powders will give various densities because of different packing behavior, and the grain size will affect the sintering efficiency. Commercially pure titanium is a common material for use in dental implants.[24] Various titanium powders with various properties have been described in the literature.[25,26]

A lower pressure gave a somewhat lower density (both green and sintered density). No lubricant was necessary when a flexible mold and isostatic pressing were used.[27] Contamination, mainly from C, H, N, and O, could transform the titanium into another grade and raise its strength, but impurities could also increase hardness and brittleness, thus lowering[28–30] the ductility, which would be a disadvantage for the application. Others have described cold isostatic pressing of titanium.[31]

The sintered density could be raised somewhat with a higher sintering temperature ($>$ 1,200°C) or a longer dwell time (longer than 2 hours) at the top temperature, but then the sintered grains became larger, which gave a less appealing visual appearance. Too-large sintered grains might also affect the mechanical properties negatively. Molybdenum has been reported as a suitable support material when sintering titanium.[32] Sintering of titanium in nitrogen, argon, and vacuum has been conducted; vacuum was found to give the best densification.[33] Studies on pressing and sintering of titanium have been conducted,[34] and, for instance, cardiac valve prostheses have been prepared.[35] A method to make sintered dental restorations of titanium alloys using isostatic pressing and carving has also been presented.[36]

A coping prepared by spark erosion generally has full density (100%), and a similar density (99%+) could be reached after sintering of the copings if the right parameters were used (pressure level and sintering parameters). The edge of the sintered coping was almost equal in sharpness compared to the spark-eroded coping, and the very small difference that may exist between them was considered less important because some grinding adjustments are usually done at the edge to give the final fitting before use.

There was a difference in visual appearance between a sintered and spark-eroded coping, at both the outer and inner surfaces. The sintered outer surface was somewhat more rough than the spark-eroded one. The outer surface is, however, always sandblasted before final use, roughening the surface for better porcelain adherence. This makes the appearance less important for the outer surfaces. The sintered inner surface was somewhat smoother than the spark-eroded inner surface. Further study will be needed to determine the extent to which that difference will affect the cementation of the crown to the dental surface.

The fitting behavior could vary for different pressures used. Each pressure gives its own shrinkage level. To achieve a good fit, the (enlarged) mandrel size must be adjusted exactly for the powder quality, pressure level, and sintering cycle used. This can be done but demands further work.

Conclusion

The goal of this study was to produce ductile titanium copings with a powder metallurgy–forming process, and this goal was achieved. Titanium powder was pressed at 300 to 900 MPa, milled to the right shape, and sintered at 1,200°C for a 2-hour holding time. The copings were silvery bright in appearance and showed sharp edges. The titanium in the sintered copings was ductile, dense (97% to 99%+), and free from open porosity after sintering. If the method is properly optimized, aiming to give the copings an improved fit, and adjusted from lab scale to production, the fabrication of copings with this sintering method could have a potential of becoming a very cost-efficient method of producing dental crowns.

Acknowledgments

The authors wish to thank the Swedish National Board for Industrial and Technical Development and the Knowledge Foundation for financial support. The authors also want to thank the following people at SCI. B. Nyberg, for initial studies: L. Eklund for SEM work, B. Ekeholt for photography, and M. Jansson for preparation of the manuscript. The authors also wish to thank A. Pettersson, The Swedish Institute for Food and Biotechnology, for pressing experiments; B. R. Lang, The University of Michigan, for good advice and reviewing of the text; and finally, the following people at Nobel Biocare: R. Holmersson, U. Nilsson, and C. Berggren for technical participation; and A. Eesmäe for preparation of the manuscript.

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