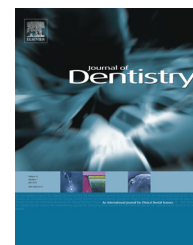


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Bending strength of zirconia/porcelain functionally graded materials prepared using spark plasma sintering

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ABSTRACT

Objectives: The purpose of this study was to fabricate functionally graded materials (FGMs) consisting of yttria-stabilised tetragonal zirconia polycrystal (Y-TZP) and porcelain using spark plasma sintering (SPS) and examine the influence of their microstructures and thermal stress on their bending strengths.

Methods: Two types of four-layered Y-TZP/porcelain FGMs having a constant layer thickness and a varying layer thickness, Y-TZP/porcelain composite materials having a microstructure corresponding to each layer in FGMs and monolithic materials of Y-TZP and porcelain were fabricated by SPS. The Y-TZP/porcelain volume fraction of each layer in FGMs was varied over 100/0–70/30. Three-point bending test, X-ray diffraction, density measurement, microstructure observation, and thermal stress estimation were performed to characterise the materials. **Results:** The bending strength of the Y-TZP/porcelain composite materials decreased with the volume fraction of the porcelain. About FGMs, when the 100%Y-TZP layer was on the tensile stress side during the bending test, the bending strength was almost the same as that of the 100%Y-TZP monolithic material. On the other hand, when the 100%Y-TZP layer was on the compressive stress side, the bending strength of FGM having a constant layer thickness was almost the same as that of the 70%Y-TZP + 30%porcelain composite material, while the bending strength of FGM with a varying layer thickness was significantly higher than that of the 70%Y-TZP + 30%porcelain composite material.

Clinical significance: The FGMs prepared and analyzed in this research can potentially be used for crowns and bridges as well as for inlays and onlays.

Conclusion: The SPS method could effectively fabricate the Y-TZP/porcelain FGMs, and the bending strength results revealed that the graded structure was very efficient to raise the bending strength.

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1. Introduction

Increasing concern with aesthetics and biocompatibility has led to a gradually increasing demand for metal-free and translucent tooth colour restorations. Conventional metal-ceramic restoration supports the low fracture strength of porcelain, which is the usual aesthetic restorative material.^{1,2} Advances in computer-aided design/computer-aided manufacturing (CAD/CAM)³ have led to the feasibility of using yttria-stabilised tetragonal zirconia polycrystal (Y-TZP) as a core material to replace metal in dental prostheses.⁴ Y-TZP was developed as an engineering material and a biomaterial^{5,6} since Garvie et al.⁷ reported that the tetragonal-monoclinic transformation in Y-TZP improved the mechanical strength and toughness of ZrO₂.

However, because of high mechanical strength and toughness, the removal of Y-TZP from the abutment tooth by cutting is very difficult. Moreover, a high failure rate has been reported for veneered porcelain,^{8,9} and the survival rates of Y-TZP core-porcelain veneer crowns have been reported to be lower than for metal-ceramic restorations.^{2,10} Residual stress within the veneer porcelain and occlusal loading may cause chipping¹¹ or fracture of the porcelain.^{12,13} Guazzato et al.^{14,15} reported that cracks propagate along the porcelain/Y-TZP interface in porcelain bonded to Y-TZP. However, others have reported that cracks initiate in the porcelain fused to Y-TZP substrate and then extend throughout the porcelain.^{16,17} White et al.¹⁸ reported that layered Y-TZP-porcelain beams with the Y-TZP layer on the tensile stress side have high bending strengths of 636–786 MPa, while beams with the porcelain layer on the tensile stress side have low bending strengths of 77–85 MPa. Moreover, cracks often propagate along the porcelain/Y-TZP interface. Thus, veneering the porcelain on a Y-TZP core is not sufficient to prevent the expansion of cracks.

Zhang et al.^{19,20} reported that a graded Y-TZP/glass structure has good resistance to veneer fracture. They hypothesised that gradation of the elastic modulus may mitigate the fracture.^{21,22} Their fabrication method infiltrated silicate glass into a Y-TZP plate. The graded layer was only 120 µm thick.

We are developing restorative functionally graded materials (FGMs) using spark plasma sintering (SPS).^{23–25} SPS charges the gaps between powder particles with electrical energy and instantaneously applies a high-temperature spark plasma to carry out the sintering. Thus, SPS is a fairly new process that enables sintering at lower temperatures and for shorter times compared with hot pressing, hot isostatic pressing, and pressureless sintering. Furthermore, near net-shape forming of the Y-TZP/porcelain FGMs is possible by using SPS with a powder stacking method in which the volume fraction of each powder is changed. Both Y-TZP and functionally graded HA/Y-TZP composites have benefitted from the use of SPS.^{26–28} However, the Y-TZP/porcelain FGMs prepared using SPS has not previously been reported.

The purpose of this study was to fabricate four-layered FGMs which were varied the Y-TZP/porcelain volume fraction over 100/0–70/30 by using SPS, and to examine the influence of their microstructures and thermal stress on their bending strengths.

2. Materials and methods

2.1. Materials

Yttria-stabilised tetragonal zirconia (Y-TZ) powder (3 mol% Y₂O₃, TZ-3Y-E grade; Tosoh Co., Tokyo, Japan) was used; it had an average particle size of 40 nm. High-fusing porcelain powder (average particle size: 12.23 µm) (SI-HF10901; Shofu Inc., Kyoto, Japan) having a high fusing temperature (1290 °C) was milled for 9 h at 300 rpm in ethanol using a planetary ball mill (Pulverisette 6; Fritsch Co., Idar-Oberstein, Germany). The ball milling media was a mixture of two types of Y-TZP balls which diameters were 2 and 10 mm. The resultant fine porcelain powder (average particle size: 0.53 µm) was used. The experimental Y-TZP/porcelain volume fractions were 100/0, 90/10, 80/20, 70/30, and 0/100. The Y-TZ and porcelain powders listed in Table 1 were mixed by vibration ball milling with Y-TZP balls.

2.2. Sintering

The powder was loaded into a cylindrical graphite die having an inside diameter of 20 mm. FGMs were made by mixing different volume fractions of the Y-TZP and porcelain components; layers were stacked sequentially in the cylindrical graphite die (Fig. 1). The powder for the bottom layer was inserted into the die, followed by levelling the surface to be the flat with a brush, and then pressed lightly by punch. Successively, the powder for the next layer was stacked on the bottom layer as well. By repeating this operation, four kinds of powders were stacked. Finally the powders were pressed at 2 MPa at room temperature using a graphite punch. A commercial SPS device (SPS-515S; Fuji Electronic Industry Co., Saitama, Japan) was performed in partial vacuum under a 10 MPa compressive stress. The heating rate was 88 °C/min and the holding time at the sintering temperature was 4 min. Sintering temperature of the 100%Y-TZP monolithic material used for measurement of a bulk density was 1100–1350 °C, while sintering temperature of materials used for other characterisation was 1350 °C only. Pulsed electricity was supplied during the heating and holding periods. The final dimensions of the specimens were 1 mm thickness and 20 mm diameter.

2.3. Evaluation of properties

X-ray diffraction (XRD) (Ultima IV, Rigaku Corporation, Tokyo, Japan) was used to characterise the crystal structures of porcelain powder, 70%Y-TZP + 30%porcelain composite

Table 1 – Specimen.

Specimen	Microstructure
100%porcelain	Monolithic
70%Y-TZP + 30%porcelain	Composite
80%Y-TZP + 20%porcelain	Composite
90%Y-TZP + 10%porcelain	Composite
100%Y-TZP	Monolithic
FGM (with constant layer thickness)	4 layers
FGM (with varying layer thickness)	4 layers

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