A novel technique for preparing dental CAD/CAM composite resin blocks using the filler press and monomer infiltration method

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The authors have developed a new technique for preparing dental CAD/CAM composite resin blocks (CRBs): the filler press and monomer infiltration (FPMI) method. In this method, surface-treated filler is molded into a green body in which the filler particles are compressed to form an agglomeration. The green body is then infiltrated with a monomer mixture before being polymerized. It is possible to produce CRBs using this method through which densely packed nanofiller is uniformly dispersed. The greater the pressure of the filler molding, the more filler in the CRB, resulting at high pressure in a very dense CRB. A CRB obtained by applying 170 MPa of pressure contained up to 70 wt% of nano-silica filler and had a flexural strength of 200 MPa, as well. It is anticipated that CRBs obtained using the FPMI method will be useful as a dental CAD/CAM material for the fabrication of permanent crown restorations.

Keywords: CAD/CAM, Composite resin block, Filler content, Monomer infiltration

INTRODUCTION

The applications of CAD/CAM systems in dentistry are growing apace, especially because the systems facilitate the fabrication of restorations using machinable materials. Ceramics, including crystallized glass and zirconia, are widely used as esthetically pleasing, long-lasting restorative materials. On the other hand, milling composite resin blocks (CRBs) has become common practice as a means of fabricating permanent dental restorations, such as crowns and inlays[1-3]. CRBs have clinical advantages, like superior esthetics, ease of milling, simple intraoral repair, and —because this is a more yielding material— less wear on opposing teeth[1,2].

Dental composite resin is a particle-dispersed composite with a sea-island structure of organic resin and inorganic filler, which has a long track record of clinical application as an esthetic filling and restorative material[4,5]. The first generation CRB for CAD/CAM was produced by polymerizing a conventional filling composite resin at the manufacturer's site[6,7]. In recent years, attempts have been made to produce very strong CRBs by polymerizing composite resin paste under conditions of high temperature and pressure[6,7]. Current CRBs are usually prepared by first polymerizing a mixture of composite resin paste obtained by mixing inorganic filler and liquid monomers, which is then molded into a block (Fig. 1). In general, the smaller the filler particles, the better the polishability and surface smoothness of the composite. Therefore, 0.1 μm or smaller particle nanofillers are often used with dental composite resins[8-13]. However, when these small nanofillers are incorporated into the monomer, the composite becomes so viscous that it is difficult to add sufficient filler[14,15]. Thus, it is difficult to use the current method to produce a CRB containing closely packed nanofillers.

The authors have developed the filler press and monomer infiltration (FPMI) method, a totally new method of preparing CAD/CAM CRBs. In this method, a powdered inorganic filler is compressed in a die into a green body block which is then infiltrated with a monomer mixture, before being polymerized to produce a CRB (Fig. 2). This method makes it possible to prepare a CRB easily, within which the nanofiller is densely packed and uniformly dispersed. In this paper, a consolidated powder in the desired shape produced by pressing in a die is called "green body" which is a commonly-used technical term in ceramic manufacturing field.

The purpose of this study is to report our new method of preparing CRBs, and to examine the effects that the conditions under which the filler powder is compressed have upon the physical properties of the resultant CRB. That is, in this study we evaluated the amount of inorganic filler contained, and the flexural strength of the CRBs obtained using the FPMI method. The hypotheses tested were: (1) the FPMI (Fig. 2) produces CRBs containing more inorganic filler than the conventional method (Fig. 1); and (2) the greater the pressure, the more inorganic filler contained, thus resulting in a much stronger CRB.

MATERIALS AND METHODS

Raw materials
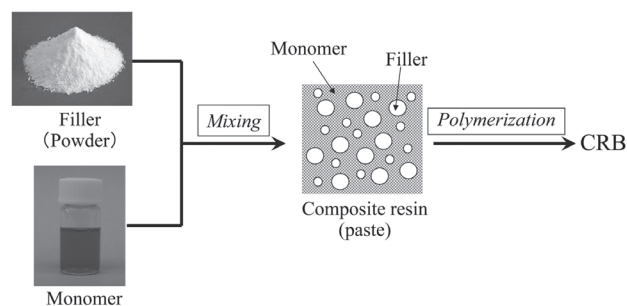A fine powder of silica nanofiller (Aerosil OX-50, Nippon Aerosol Co., Ltd., Tokyo, Japan) with an average

Fig. 1   System for preparing CRBs using the polymerization of composite resin paste (the currently used method).

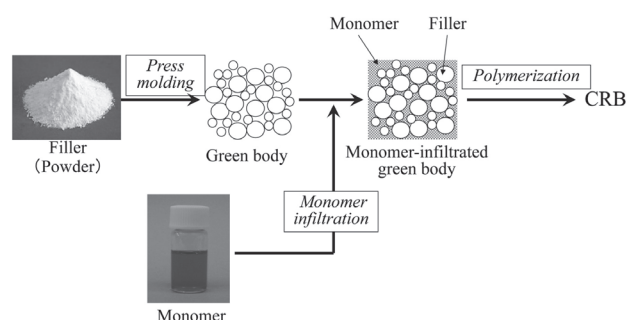Fig. 3   A stainless die with an internal capacity of 33 mm×24 mm and two punches for a uni-axial press.

Fig. 2   System for preparing CRBs using the filler press and monomer infiltration (FPMI) method.

Fig. 4   Pictures of the step using the uni-axial press.
(a) Inorganic filler is placed in a uni-axial press stainless die.
(b) Inorganic filler is compressed from both the top and bottom using two punches in a table press (Type TB-110H; NPa System Co., Ltd., Osaka, Japan).
(c) The resultant press molded inorganic filler block (the nano-silica green body).

particle size of 40 nm and a specific surface area of 50 m^2/g, was surface-treated using a silane coupling agent (3-methacryloxypropyl trimethoxysilane; Shin-Etsu Chemical Co., Ltd., Tokyo, Japan) and used as the inorganic filler. The monomer mixture was comprised of 49.48 wt% urethane dimethacrylate (UDMA; Kyoeisha Chemical Co., Ltd., Osaka, Japan), 49.48 wt% triethyleneglycol dimethacrylate (TEGDMA; Shin-Nakamura Chemical Co., Ltd., Wakayama, Japan), 0.99 wt% benzoyl peroxide (BPO; NOF Corporation, Tokyo, Japan) as a heat-curing catalyst, and 0.05 wt% acylphosphine oxide compound (Lucirin TPO; BASF Japan Ltd., Tokyo, Japan) as a light-curing catalyst. All of these materials were thoroughly and uniformly mixed together for use.

Preparing a CRB using the FPMI method
1. Press molding the filler
A stainless die for a uni-axial press (Fig. 3) with an internal capacity of 33 mm×24 mm and two punches were used. A 5.5 g charge of inorganic filler was placed in a uni-axial press stainless die (Fig. 4a), which was compressed from both the top and bottom using two punches. It was then press molded using a table press (Fig. 4b, Type TB-110H; NPa System Co., Ltd., Osaka, Japan). The filler was compressed for 60 s at one of two
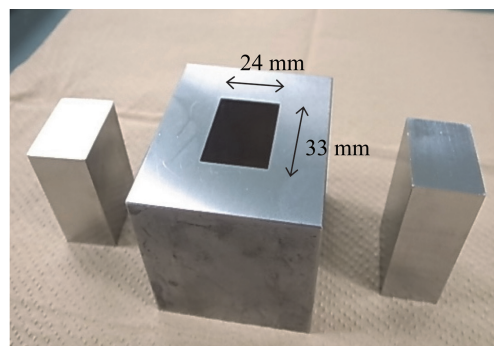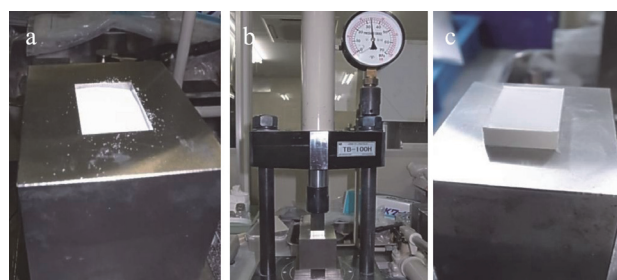
levels of pressure, 30 kN (38 MPa) or 60 kN (76 MPa). After pressing, the green body block of inorganic filler was removed from the die (Fig. 4c, Fig. 5a). Some blocks pressed at 60 kN were vacuum sealed in a vinyl bag (Fig. 5b) and subjected to cold isostatic pressing (CIP) at 170 MPa for 60 s, in order to mold a green body at even higher pressure. That is, sample blocks were prepared using three successively higher levels of compression (Table 1). The CIP device used was Type N6022-01 (Fig. 5c, NPa System Co., Ltd., Osaka, Japan).

2. Infiltration of the monomer
Each inorganic filler green body was immersed in a beaker containing the monomer mixture (Fig. 6a). Translucent monomer-infiltrated green bodies were obtained five days later (Fig. 6b).

3. Polymerization
The polymerization of the monomer-infiltrated green bodies thus obtained was performed in two stages: first

light curing and then heat curing. Cracks often occur when a monomer-infiltrated green body is heated in an oven for heat curing. However, if a press-molded green body is gently light-cured and then heated at 120ºC, a hard, crack-free, well-polymerized CRB can be obtained.

The monomer-infiltrated green body was put on a glass plate and light-cured for 3 min using a dental light-curing unit (α-Light 3; J. Morita Corp., Osaka, Japan) and then heated at 120ºC in the open atmosphere for 2 h using an oven chamber. This resulted in a hard translucent composite resin block. Since, after curing, the blocks tended to be covered with a layer of unfilled cured monomer, this layer was ground from the surface and the new surface was polished to obtain the target uniform CRB (Fig. 6c). In this study, CRBs were prepared using three levels of compression, 38 MPa, 76 MPa and 170 MPa, and thus the CRBs were identified as P1, P2 and P3, respectively (Table 1).

Preparation of CRBs from composite resin paste
To make the control samples, the same monomer mixture and 50 wt% of the same inorganic filler as mentioned above were mixed by hand in a glass mortar. The composite resin paste thus obtained was polymerized in a die in the same manner as was used in Fig. 1 to produce CRB. The CRBs obtained by this method are referred to as CP (Table 1). During the process of producing the CP, the paste became so viscous and resistive that it was difficult to work more than 50 wt% of filler into the monomer mixture.

Another series of hard blocks (designated M, Table 1) were also prepared. These were made of monomer without any inorganic filler.

The inorganic filler component of the blocks
Each of the hard composite resin blocks was put in a porcelain crucible and heated at 600ºC in an electric furnace to burn off the organic components. Then the residue was measured to determine the amount of inorganic filler in the CRB. The average value of five
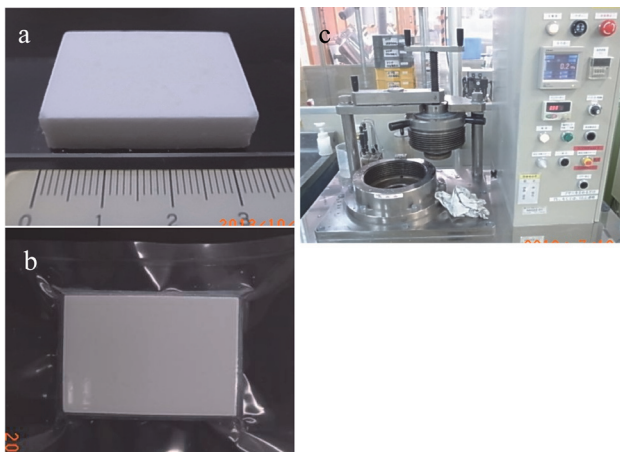

Fig. 5   Pictures of the cold isostatic pressing (CIP) step.
(a) Nano-silica green body made by uni-axial compression at 76 MPa.
(b) Green body vacuum-sealed in a vinyl bag.
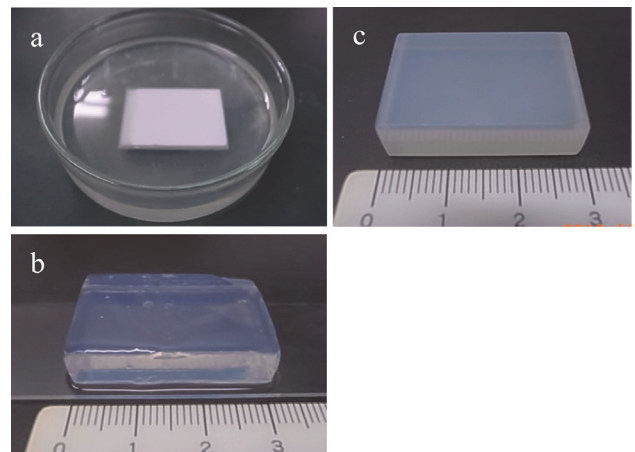(c) The cold isostatic press used for this study (Type N6022-01; NPa System Co., Ltd., Osaka, Japan).

Fig. 6   Pictures of the monomer infiltration step.
(a) Nano-silica green body immersed in liquid monomer.
(b) Monomer infiltrated nano-silica green body.
(c) CRB resulting from the polymerization of the monomer-infiltrated green body.

Table 1   CRBs tested in this study

CRB (Code)	Filler	Preparation method	Compression method	Molding pressure (MPa)
M	none	Polymerization of monomer	—	—
CP	nano-silica	Polymerization of composite paste	—	—
P1	nano-silica	FPMI	uni-axial	38
P2	nano-silica	FPMI	uni-axial	76
P3	nano-silica	FPMI	uni-axial+CIP	76+170

specimens was used as the inorganic filler content of the block being tested.

To observe the dispersion of the nanofiller through the composite resin blocks, P3 and CP were observed using a transmission electron microscope (TEM) (Type JEM-2100F; JEOL Ltd., Tokyo, Japan).

Sections 50 nm thick were cut from the CRBs using a diamond knife (Diatome, Bienne, Switzerland) in an ultra microtome (Reichert ULTRACUT-UCT, Leica, Vienna, Austria). The sections were then observed with a TEM at an accelerating voltage of 200 kV.

Flexural strength

The measurement of the flexural strength of the CRBs was carried out by the 3-point bending method according to ISO4049 (2009)[16]. Long specimens, 2×2×25 mm, were cut from each block using a diamond saw and used for the flexural strength test. The surface of each specimen was polished with 3,000 grit emery paper to create a glossy surface. The measurements were performed using a universal testing machine (Autograph Type AG-1; Shimazu Corp., Kyoto, Japan) at a crosshead speed of 1 mm/s. The flexural strength (FS) in MPa was calculated as:

$$FS = 3Fl/2bh^2$$

Where F is the maximum load in Newtons exerted on the specimen at the point of fracture, l is the distance in mm between the supports, b and h are, respectively, the width and thickness of the specimen in mm. The elastic modulus was also determined from the slope of the initial linear part of the stress-strain curve. The average value of five specimens was used as the flexural strength of the block being tested.

The fracture surfaces of test specimens P3 and CP were observed using a scanning electron microscope (SEM) (Type S-3500N; Hitachi Kyowa Engineering Co. Ltd., Ibaragi, Japan). The surfaces were ion-coated with gold for 30 s. using an ion sputter apparatus (Type NST-1S, Vacuum devise Co., Ltd, Ibaragi, Japan). Then SEM observation was performed at an accelerating voltage of 15 kV.

Statistics

The data obtained from the flexural strength measurements were analyzed using the Tukey HSD test run from a software package (Excel Statistics, 2006, SSRI, Tokyo, Japan). The level of statistical significance was set at $p<0.05$.

RESULTS

Fabrication of the blocks

Figure 5a shows an example of a compressed inorganic filler green body and Fig. 6b shows a translucent monomer-infiltrated green body, as prepared for this study. The press-molded inorganic filler green body maintained its shape when handled; thus it was intact enough for subsequent operations.

The infiltration of monomers was achieved by immersing compressed powder green bodies in a

Table 2　Inorganic filler in the CRBs

CRB (Code)	Inorganic filler	
	Ignition residue (wt%)	Calculated volume ratio (vol%)
CP	48.0 (0.3)	33.5
P1	56.5 (0.6)	41.5
P2	60.4 (0.3)	45.4
P3	70.1 (1.7)	56.0

monomer mixture solution and leaving them for a few days, during which time the monomers thoroughly infiltrated the green bodies. Figure 6b shows a green body completely infiltrated by the monomer. There was macroscopically no difference in size between the compressed filler green body and the monomer-infiltrated green body. The monomer-infiltrated green body is uniform and looks almost transparent. This is probably because the monomers have completely penetrated the gaps between filler particles and because there is only a slight difference in the refractive indexes of the inorganic filler and the monomer.

Inorganic filler content

Table 2 shows the results of the measurement of the residue left after burning. During this test all the organic components contained in the CRB are burned away so completely that the amount of inorganic filler components alone can be measured. If the specific gravity of fumed silica is assumed to be 2.2 g/cm³, as stated by the manufacturer[17,18], and the specific gravity of the matrix resin is 1.21 g/cm³ (the same value as the specific gravity of CRB M, obtained by using the gas density measurement method), one can calculate the volume percent of inorganic filler contained in the CRB. The calculated values thus obtained are also shown in Table 2.

When the residue left after burning was measured, the amount of filler in the CP was found to be 48 wt%, a lower value than the targeted 50 wt%. This is because the surface treatment agent in the surface treated filler was burned off as an organic component.

On the other hand, the CRBs (P1, P2 and P3) obtained by the FPMI method all contained more inorganic filler than did the CP. The greater the pressure, the more inorganic filler each of the three CRBs contained, with P3 blocks produced by 170 MPa CIP containing as much as 70 wt% (56 vol%) of filler. The TEM images of the P3 and CP blocks (Fig. 7) reveal that the nanofiller is uniformly and densely distributed through the P3 CRB.

Flexural strength

Figures 8 and 9 show the results of the flexural strength measurements. The flexural strengths of the P1, P2

and P3 blocks, obtained using the FPMI method, were approximately 200 MPa in all cases, regardless of the amount of filler they contained. There were no significant differences among the three types of block ($p>0.05$). The flexural strength of the CP blocks was a low 140 MPa. That is, the CRBs obtained by the FPMI method were significantly stronger in terms of flexural strength than the CP blocks ($p<0.05$). The flexural strength of the hard resin blocks (M) containing only the monomer mixture was 110 MPa, even significantly lower than that of the CP blocks ($p<0.05$).

The flexural modulus of the CRBs is shown in Fig. 9. The modulus of the M blocks was a low 2 GPa, and that of the CP blocks was 6 GPa, attributable to the nanofiller loading. The flexural modulus of the CRBs obtained by the FPMI method was in the range of 8–10 GPa, and significantly increased as the filler content

increased ($p<0.05$).

The fracture surfaces of the CP and P3 test specimens are shown in Fig. 10. There were no significant differences in the fracture surfaces. In addition, no voids were observable on any fracture surface.
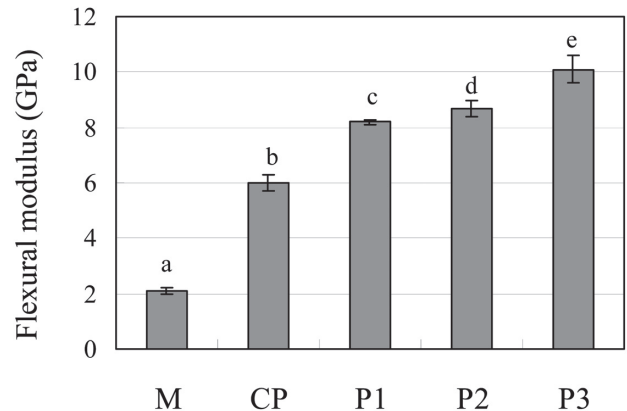

Fig. 9 Mean flexural moduli of the CRBs.
M: polymerized monomer without inorganic filler, CP: CRB from composite resin paste, P1, P2 and P3: CRBs made using the FPMI method. Different characters indicate statistically significant difference at $p<0.05$.

Fig. 7 TEM images of a CRB, original magnification ×10,000, bar=200 nm.
(a) CRB from composite resin paste containing 48 wt% of nano-silica (CP).
(b) CRB by the FPMI method containing 70 wt% of nano-silica (P3).

Fig. 8 Mean flexural strengths of CRB samples.
M: polymerized monomer without inorganic filler, CP: CRB from composite resin paste, P1, P2 and P3: CRBs made using the FPMI method. Different character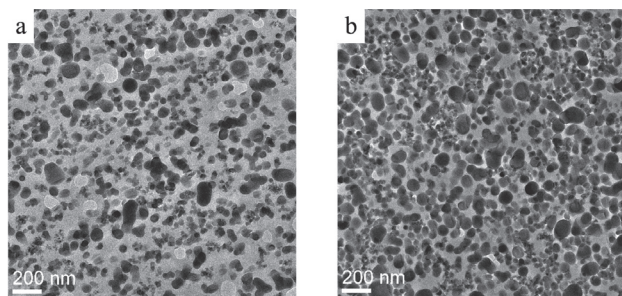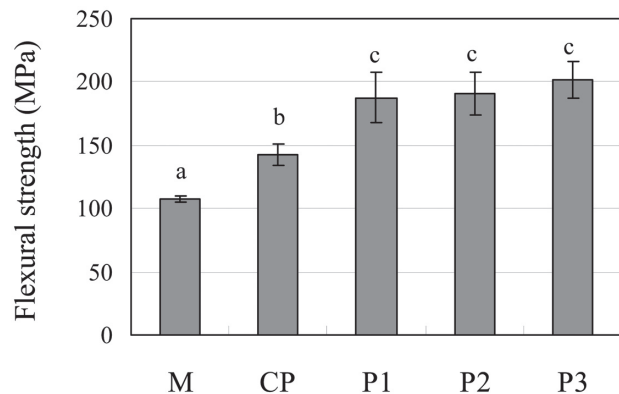s indicate statistically significant difference at $p<0.05$. Same characters indicate no significant difference at $p>0.05$.

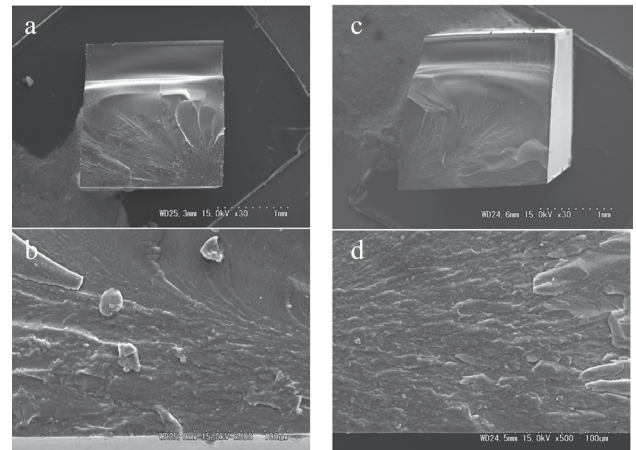Fig. 10 SEM images of the fracture surfaces of specimens after the flexural strength test.
(a) CP; whole image of fracture surface, original magnification ×30.
(b) CP; magnified image of the bottom of the specimen (tension side), original magnification ×500.
(c) P3; whole image of a fracture surface, original magnification ×30.
(d) P3; magnified image of the bottom of the specimen (tension side), original magnification ×500.

DISCUSSION

Preparation of CRBs

Willems et al.[4] have roughly divided commercial dental composite resins into two types: the dense-filled type containing micro-glass filler as a main ingredient and the micro-fine type containing microfiller (nanofiller). He points out that when these small nanofillers are incorporated into the monomers, the composite becomes so viscous and the paste becomes so sticky that it is difficult to add sufficient filler. In this study, we found it difficult to work more than 50 wt% of silane-treated nanofiller into the monomer for the same reason. Therefore, the inorganic filler content (ignition residue) of the CRB (CP) resulting from the composite resin paste and hand mixing was only 48 wt%. In the conventional method for preparing the CRB, wherein a powerful mixing machine is used, it might be possible to prepare a composite resin paste containing 50 wt% or more of inorganic filler. However, the resultant paste would be so viscous and sticky that it would be difficult to prepare a uniform composite resin block without any air bubbles.

The FPMI method does not require the process of mixing monomers and fillers to create a composite resin paste. Thus, it does not pose the problem of increased paste viscosity when the filler is nano-sized. In other words, it is not difficult, using this system, to manufacture composite resin paste containing a large quantity of nanofiller. In addition, the FPMI method makes it possible to compact the filler particles even closer together by press molding the filler. Press molding inorganic powder into a green body is a common practice in the ceramic manufacturing field and the uni-axial press and CIP are also widely used in that field[19,20].

The measurement of residue left after burning revealed that the amount of inorganic filler contained in RCBs (P1, P2, P3) obtained by press molding was higher than in CRBs (CP) obtained using composite resin paste. The greater the molding pressure, the more closely was the inorganic filler compressed in the green body, and the more inorganic filler each of the three CRBs obtained using FPMI method contained. P3 blocks produced by 170 MPa CIP containing as much as 70 wt% (56 vol%). This is a very large amount of inorganic filler to find in a dental composite resin consisting of only nanofiller.

During the monomer infiltration step, a pressed green body composed of silane-treated nanoparticle silica powder was immersed in a liquid monomer mixture, to allow the monomers to penetrate thoroughly into the green body. The monomers apparently penetrated deep into the narrow gaps between filler particles through capillary action. It took a few days to completely infiltrate the entire green body with the monomer mixture, but the infiltration of monomers throughout the green body could be confirmed visually, because where there is complete infiltration of monomers the body is translucent, with no air bubbles (Fig. 6b). In addition, no voids were observable on the fracture surface of the strength-test specimens examined under SEM (Fig. 10). Nor was there any difference in size between the compressed filler green body and the monomer-infiltrated green body. Thus, an entirely new method of manufacturing CRBs has been established, mainly thanks to the discovery that a densely compressed nanofiller green body can be deeply infiltrated with monomer.

There are presently some commercially available CRBs containing nanofiller. However, the filler content of a CRB with a homogeneous distribution of nanofiller can be as low as 14 wt%[21]. For this reason, these CRBs are mostly used for temporary restorations, and they are not suitable for permanent restorations where great mechanical strength is required. There are commercial CRBs containing 80 wt% nanofiller. However, the nanofillers (silica and zirconia) are aggregated and incorporated into the composite as nanocluster filler (with an average nanocluster particle size of 0.6 to 10 µm)[22]. That is, these nanofillers are not added in a monodispersed state in the form of primary particles. A polymer-infiltrated ceramic network (PICN) material for dental CAD/CAM has also been proposed[23-25]. The PICN material is produced by first infiltrating a monomer mixture into porous ceramic that was obtained by sintering inorganic powder and then polymerizing the monomer-infiltrated ceramic. The PICN material involves monomer infiltration followed by polymerization; however the PICN material obtained is a porous ceramic network material, not a particle-dispersed composite material; that is, it is a completely different type of composite material from the CRBs produced for this study.

As stated above, the CRBs prepared for this study using the FPMI method are a totally new nanocomposite material with a homogeneous distribution of nanofiller having a higher than 50 wt% content in a polymer matrix.

Flexural strength

Matsumoto[26] has reported that the higher the filler content, the greater the flexural strength. One might be tempted to conclude that the difference in strength between the CP and P1 blocks can be explained by the fact that P1 contains more filler. However, the P2 and P3 blocks contain more filler than P1, but they are not significantly stronger. Therefore, the difference in flexural strength may instead be attributable to the difference in the method of preparing the composite resin, not to the amount of filler contained. For example, press molding may cause the filler to be more uniformly dispersed than does hand mixing. It is known that it is difficult to disperse nanofiller uniformly when it is mixed into resin[27]. In our samples, the flexural modulus of elasticity did increase as the filler content increased, consistent with Matsumoto's report[26].

Karabela et al.[28] prepared light-cured composites based on a Bis-GMA/TEGDMA (50/50 wt/wt) matrix mixed with 55 wt% silane-treated nanosilica with the particle size controlled to between 7 and 40 nm,

including Aerosil OX-50, the same filler used in our study. He reported that each nanocomposite had a flexural strength in the range 90–103 MPa. The flexural strengths of the CRBs (P1, P2 and P3), obtained by the FPMI method, were approximately 200 MPa, significantly stronger than the previously investigated homogeneous dental nanocomposite materials.

CRBs containing densely loaded nanoparticles, like those obtained in this study, should be useful as a dental CAD/CAM material for the fabrication of permanent crown restorations. Containing a great deal of filler, they have good mechanical strength and durability. In addition, containing minute particle-sized nanofiller, they will probably make crown restorations with especially smooth surfaces that are less wearing to the opposing teeth. In the future, we will evaluate other physical properties appropriate for clinical purposes including wear resistance, the extent of damage to the opposing teeth, fatigue characteristics, surface smoothness and durability.

CONCLUSIONS

A dental CAD/CAM composite resin block, with a nanocomposite structure that included having nanofiller densely and uniformly dispersed throughout, was manufactured on a trial basis using the filler press and monomer infiltration (FPMI) method. With this method, it was possible to substantially increase the filler content over that of a CRB obtained by polymerizing composite resin paste in the conventional manner. A CRB obtained by applying a 170 MPa molding pressure contained up to 70 wt% of homogeneously dispersed nano-silica filler and had a flexural strength of 200 MPa, as well. It is anticipated that CRB blocks like the trial samples obtained in this study will be useful as a CAD/CAM material for the fabrication of permanent crown restorations.

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