Sintering behavior and mechanical properties of machinable zirconia/mica composites

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1. Introduction

Zirconia ceramics, particularly tetragonal zirconia polycrystalline (TZP) stabilized with 3 mol% yttria, have excellent mechanical properties (i.e. high strength, fracture toughness and wear resistance), chemical durability, biocompatibility and aesthetics [1–14]. They are therefore the most promising materials for metal-free dental restorations; however, they also have some issues. Low-temperature degradation (LTD), or “aging”, is well-known as the main issue for the long-term reliability of zirconia ceramics [5–7,9,11,12,14–18]. Aging is the slow transformation of zirconia crystals from the tetragonal phase to the much more stable monoclinic phase in the absence of mechanical stress. This causes surface roughening and micro-cracking [5,7,17] and exposes zirconia-based dental restorations to the risk of spontaneous catastrophic failure [10,11]. Zirconia-based dental restorations with layers of ceramic veneer added to improve their translucency or aesthetics increase the risk of chipping because of the compatibility of zirconia and ceramic veneers [1,2,4,5,8,9,11,12,14,18]. In order to provide higher restorative quality, a highly precise occlusal fit (i.e. high dimensional accuracy) is required [7,12,13]. As one of the issues to zirconia-based dental restorations, however, zirconia ceramics have much lower machinability, which is regarded as a key mechanical property for precise fitting [7,12], although the precision of zirconia-based dental restorations is dependent on various factors [12,13].

Most zirconia-based dental restorations are manufactured using a computer-aided design and computer-aided manufacturing (CAD/CAM) technique called “soft machining” [11,12,14,15,18,19]. In this technique, pre-sintered zirconia blanks are machined because fully densified zirconia ceramics are very hard and brittle. Thus, machined blanks are densified through shrinkage by sintering them at higher temperatures. Shrinkage must be precisely controlled to assure the required dimensions after sintering [13]. The major drawback of “soft machining” is the difficulty of matching the sintering shrinkage of the machined zirconia blanks to the degree of enlargement programmed by the CAD software as precisely as possible [12]. By contrast, another CAD/CAM technique called “hard machining” can be used [11,12,14,18,19]. In this method, post-sintered, fully densified and homogeneous zirconia blanks are machined directly as dental restorations. In cases of complex prosthetic geometries, therefore, post-sintered, “hard machining” is the most predictable manufacturing system [12,13] leading to a superior marginal fit [14]. However, “hard machining” requires a much longer time and a more expensive production procedure [12–14,18,19]. In addition, it introduces surface damage, such as micro-cracks and phase transformations [12,14,18]. If zirconia ceramics have machinability, zirconia-based dental restorations with high
dimensional accuracy can be easily fabricated, not only by "hard machining" but also by "soft machining".

As machinable zirconia ceramics, zirconia/CePO$_4$ [20,21] and zirconia/LaPO$_4$ [22] composites, which utilize the weak interface between zirconia and phosphate, have been reported. They can be cut and drilled using conventional machining tools. While composites prepared by infiltrating a CePO$_4$ liquid precursor and hot pressing have a relatively high bending strength of 500–700 MPa [21], zirconia/LaPO$_4$ with its excellent machinability has significantly degraded bending strength [22]. For similar machinable Al$_2$O$_3$/LaPO$_4$ [20,23–27] composites, machinability is enhanced and bending strength substantially decreases with increases in the LaPO$_4$ content [23,25,26]. Thus, bending strength is improved by spark plasma sintering (SPS) [27].

As other machinable ceramics utilizing the machinability of h-BN or MAX phase ceramics (e.g. Ti$_3$SiC$_2$ and Ti$_6$AlC$_3$), machinable Al$_2$O$_3$/h-BN [28], Si$_3$N$_4$/h-BN [29–31], SiC/h-BN [32], α-SiAlON/BN [33], AlN/h-BN [34], Al$_2$O$_3$/Ti$_3$SiC$_2$ [35] and hydroxyapatite/Ti$_3$SiC$_2$ [36] composites are also being fabricated by hot pressing or SPS. In fact, machinable Si$_3$N$_4$/h-BN nanocomposites with a bending strength of greater than 1 GPa have been reported [31].

On the other hand, mica glass-ceramics are well-known machinable ceramics [37] that are applied as dental materials [37,38]. Also, machinable cordierite/mica [39], spinel/mica [40] and alumina/mica [41] composites have been successfully fabricated utilizing the excellent cleavage of mica. While machinable composites containing the h-BN [28–34] and MAX phases [35,36] are fabricated using special sintering methods with expensive apparatuses (e.g. hot pressing and SPS), machinable composites containing mica [39–41] can be fabricated without such expensive sintering apparatuses. Compared with machinable composites containing rare-earth phosphates [20–27], machinable composites containing mica [39–41] can be produced with low-temperature sintering because of sintering through a liquid phase formed by melting raw mica materials.

In this study, in order to fabricate machinable zirconia ceramics with higher mechanical properties for dental restorations, zirconia/mica composites were prepared by mixing TZP and fine mica powders, compacting the powder mixtures and then conducting pressure-less sintering of green powder compacts. Thus both the sintering behavior and mechanical properties of the zirconia/mica composites were investigated.

Zirconia-toughened and zirconia-strengthened mica glass-ceramics whose base materials are mica glass-ceramics are known as mica-zirconia composites [42–45]. In this study, mica (fluorophlogopite) was added to zirconia base material, and machinable zirconia/mica composites, which are different from the zirconia-toughened and zirconia-strengthened mica glass-ceramics, were successfully fabricated for the first time by the sintering method.

2. Materials and methods

The reagents of K$_2$CO$_3$, MgO, Al$_2$O$_3$, SiO$_2$ and MgF$_2$ were mixed in a chemical composition corresponding to the stoichiometric fluorophlogopite (K$_4$Mg$_6$Al$_2$Si$_3$O$_{10}$F$_2$) composition, calcined at 900°C, melted in a sealed platinum container at 1450°C and cooled in the furnace. Fluorophlogopite is the best-known synthetic mica and is easily synthesized by the melting method. Therefore, it was first used as mica to fabricate zirconia/mica composites. A lump of mica was obtained and crushed using a mortar and then pulverized in ethanol by ball milling for 48 h. The large mica particles in the obtained slurry were removed by centrifugation at 5000 rpm for 6 min. The particle size distribution of the remaining mica in the slurry (centrifuged mica) is shown in Figure 1. The size range of the mica particles was 0.04 µm to 0.2 µm, and the average particle size was 0.09 µm as measured using a particle size analyzer (Partica LA-950, Horiba Ltd., Japan). The sizes were confirmed with the scanning electron microscopic image shown in Figure 2. In addition, X-ray diffraction (XRD) patterns of the as-prepared mica and the centrifuged mica powders are shown in Figure 3. A large amount of mica crystal was precipitated as only a crystal phase. A minimal amount of glass might have remained in the as-prepared mica specimen. The crystallinity of the mica was lowered by ball milling. The slurry obtained after centrifugal separation was mixed with 3 mol% Y$_2$O$_3$ containing zirconia powder (HSY-3F-J-LA, Daiichi Kigensyo Kagaku Kogyo Co., Ltd., Japan) in ethanol by ball milling for 24 h. The obtained slurry was then dried and passed through a 100-mesh sieve. Thus, four powder mixtures were prepared. These mixtures contained 0%, 17 wt% (30 vol%), 21 wt% (36 vol%) and 31.5 wt% (49 vol%) mica powder and were denoted as 0MZ,
30MZ, 36MZ, and 49MZ, respectively. The powder mixtures were calcined at 600ºC for 1 h, passed through a 100-mesh sieve, compacted by cold isostatic pressing at 190 MPa and then fired in a sealed platinum container at 1150-1250ºC for 2 h. Both the heating and cooling rates of the firing were 10ºC/min.

The crystalline phases of the prepared composites were determined using an XRD analyzer (MiniFrez II, Rigaku Corp., Japan). The bulk and true densities were measured by the Archimedes and pycnometer methods, respectively. The microstructures were observed using field emission-scanning electron microscopy (FE-SEM; S-4100, Hitachi High-Technologies Corp., Japan) and scanning transmission electron microscopy (STEM; HD2300A, Hitachi High-Technologies, Corp., Japan). The Vickers hardness and Young’s modulus of the composites, finally polished to a mirror finish, were measured by the Vickers indentation fracture method using a hardness tester (VMT-7, Matsuzawa Co. Ltd., Japan) and the pulse-echo method using an ultrasonic thickness meter (PANAMETRICS-NDT 35, Olympus Corp., Japan), respectively. When measuring the Vickers hardness, a 196.1 N load was applied to the polished surface for 10 s. In addition, the fracture toughness was determined by the indentation-fracture (IF) method. The mechanical strength of the 3 mm × 2 mm × 22 mm composites was measured using a three-point bending tester (Autograph AG-300kNXplus, Shimadzu Corp., Japan), with a crosshead speed of 0.5 mm/min and a span length of 20 mm. The machinability was qualitatively evaluated using a bench-drilling machine (TB-1131K, Ryob Ltd., Japan). A drill with a 3 mm diameter was used, and the rotation frequency was 620 rpm.

3. Results and discussion

3.1. Densification and phase change

The relative densities of the prepared composites and monolithic zirconia are shown in Figure 4. While the monolithic zirconia reached approximately full density at 1300ºC, all the composites reached it at 1150ºC, although the relative densities of the composites became slightly higher with increases in the amount of mica added. The addition of mica enhanced the densification of zirconia and allowed the densification temperature to be lowered by approximately 150ºC.

Figure 2. SEM photographs of (a) centrifuged mica and (b) zirconia powders.

Figure 3. XRD patterns of (a) as-prepared mica and (b) centrifuged mica powders.

Figure 4. Relative densities of (■) 0MZ, (●) 30MZ, (▲) 36MZ and (○) 49MZ composites.
XRD patterns of the composites fired at 1200°C and the monolithic zirconia fired at 1300°C are shown in Figure 5. All the sintered specimens were powdered and analyzed. The main crystal phase of all the composites was tetragonal zirconia. Monoclinic zirconia was observed as the minor phase. The diffraction peak was strengthened further with increase in the amount of mica added. Mica was observed only in the 49 MZ composite. No other crystal phases appeared in any of the composites fired at below 1200°C, suggesting that 3 mol% Y_2O_3 containing zirconia and mica almost did not react.

DTA and TG curves of the as-prepared mica are shown in Figure 6. The mica was analyzed in air, although the composites were fired in a sealed container. Beginning at approximately 1000°C, an endothermic transformation started in the DTA curve as the temperature was elevated and weight loss also started in the TG curve, indicating that the mica was thermally decomposed and melted and fluoride or fluorine was simultaneously volatilized [39,41]. Hence, the liquid phase formation reported herein resulted in low-temperature sintering of zirconia. As sintering aids to densify the zirconia at low temperatures, Bi_2O_3 [46,47] and Fe_2O_3 [47–51] are reported; the melting of Bi_2O_3 at 825°C, in particular, achieved densification of zirconia at temperatures below 1000°C [46]. The melting of Bi_2O_3 caused liquid phase sintering, which induced grain slipping and rearrangement and accelerated the mass-transport and diffusion rates of zirconia through solution-precipitation [46,47]. Meanwhile, Fe_2O_3 caused not only liquid phase sintering but also formation of lattice defects in the zirconia crystals [47–51], thereby resulting in low-temperature sintering. In the same manner, the melting of mica at above 1000°C induced rearrangement of the zirconia particles and accelerated the mass-transport of zirconia through solution-precipitation, thereby leading to rapid densification in the temperature range of 1000°C to 1150°C.

### 3.2. Microstructure development

STEM images of the 30MZ composite fired at 1200°C are shown in Figure 7. The dark grains represent zirconia, while the light grains depict mica. Striped patterns can be observed in some elongated light grains, proving that the grains are a layered compound, namely, mica. The mica therefore closely contacted the neighboring zirconia grains, as shown in Figure 7(c). The zirconia grains were 0.2–0.3 µm, which was not much larger than the particles in the zirconia powder shown in Figure 2(b). Figure 7(b) reveals that the boundaries between the zirconia grains were flat and smooth, especially the grain boundaries in the circle drawn in Figure 7(b), which was caused by grain shape accommodation [52]. This result indicates that the densification of zirconia was promoted through the solution-precipitation mechanism of liquid phase sintering. The yttrium ions in zirconia grains might dissolve in the liquid phase moreover, as well as the zirconium ions. Zirconia, which was precipitated through solution-precipitation and which might contain yttrium ions precipitated with zirconium ions, became a monoclinic phase but did not maintain a tetragonal phase during cooling. Thus, more monoclinic zirconia were formed in the composites containing larger amounts of mica and the composites fired at higher temperatures.

FE-SEM images of the polished and fracture surfaces of 30 MZ composite fired at 1150-1250°C are shown in Figure 7.
Figure 8. The dark grains in the FE-SEM image of the polished surface of the composite (Figure 8(a)) depict mica, while the light grains represent the zirconia. The FE-SEM image shows that the dark grains were less than 1 µm and uniformly distributed. That is, the mica grew slightly in the composite at 1150ºC compared with the centrifuged mica powder shown in Figure 2(a) and were uniformly dispersed, although the mica was melted once in the composites at above 1000ºC and then precipitated again during cooling. The mica grains grew mainly with increases in the sintering temperature and reached approximately 2 µm at 1250ºC. By contrast, the zirconia grains were less than 0.5 µm, even in the 49 MZ composite, with increases in the amount of mica added. That is to say, the solution-precipitation of zirconia slightly promoted densification and grain growth as the liquid phase increased during firing.

3.3. Mechanical properties

Photographs of drilling tests conducted on 30MZ and 36MZ composites fired at 1200ºC using a drill made of hard metal and on 49MZ composite fired at 1150ºC using a drill made of high-speed steel are shown in Figure 10. The drills penetrated these composites with a thickness of approximately 3 mm. Glass-ceramics containing more than two-thirds of mica by volume are normally machinable, regardless of the morphology of the mica crystals, and those containing as little as one-third of mica by volume are machinable if the aspect ratio of the crystals is large enough to cause a high degree of crystal interlocking [37,53]. The composites fabricated herein contained more than approximately one-third of mica by volume. In addition, an interlocking microstructure of flake-like mica crystals was observed in these composites (Figures 8 and 9). Thus, 30 MZ and 36 MZ composites could be machined with a drill made of hard metal. 49 MZ composite containing a large amount of mica and forming a pronounced interlocking mica structure could be sufficiently machined with a conventional high-speed steel tool.

The mechanical properties of each composite are shown in Table 1. The bending strength of monolithic zirconia was approximately 690 MPa, which was lower than that of standard TZP. However, the origin of this strength is currently unclear. The bending strength of the composites decreased as the mica content increased. Yet machinable 30 MZ composite showed a bending strength of over 500 MPa. The bending strength of composites may be enhanced by combining finer mica particles with zirconia. Similarly, the Young’s modulus, Vickers hardness and fracture toughness of the composites decreased as the content of softer mica increased. The fracture toughness degradation, especially, was caused by a lessening of the effect of the martensite
transformation of the tetragonal zirconia due to a decrease in not only the zirconia content but also in the ratio of the tetragonal zirconia-to-zirconia content.

Figure 8. FE-SEM photographs of (a) polished surface of 30MZ composite fired at 1150°C and fracture surfaces of 30MZ composite fired at (b) 1150°C, (c) 1200°C and (d) 1250°C for 2 h.

Figure 9. FE-SEM photographs of fracture surfaces of (a) 39MZ and (b) 49MZ composites fired at 1150°C for 2 h.

Figure 10. Photographs of drilling test for (a) 30MZ and (b) 36MZ composites fired at 1200°C using a drill made of hard metal and for (c) 49MZ composite fired at 1150°C using a drill made of high-speed steel.

4. Conclusions

In this study, machinable zirconia/mica composites were successfully fabricated using slurry containing
Table 1. Mechanical properties of the composites.

| Specimen | Mica content /vol% | Firing temp. /ºC | Relative density /% | Bending strength /MPa | Young's modulus /GPa | Vickers hardness /GPa | Fracture toughness /MPa·m° 1/2 |
|----------|-------------------|------------------|---------------------|-----------------------|---------------------|----------------------|-------------------------------|
| 0MZ      | 0                 | 1350             | 100                 | 687 ± 37              | 218                 | 11.4 ± 0.1           | 5.1 ± 0.1                     |
| 30MZ     | 30                | 1200             | 100                 | 552 ± 91              | 153                 | 8.1 ± 0.1           | 3.9 ± 0.1                     |
| 36MZ     | 36                | 1200             | 100                 | 470 ± 49              | 146                 | 6.8 ± 0.2           | 3.9 ± 0.1                     |
| 49MZ     | 49                | 1150             | 99.8                | 400 ± 60              | 123                 | 5.3 ± 0.1           | 3.5 ± 0.3                     |

fine mica particles with an average size of 0.09 µm and zirconia powder containing 3 mol% yttria. The composites containing 30–49 vol% mica reached approximately full density at a low temperature of 1150ºC, which was approximately 150ºC lower than the densification temperature of monolithic zirconia. The mica melted during firing at above 1000ºC and recrystallized during cooling. The recrystallized mica grains made close contact with the zirconia grains. The liquid formed by melting of the mica resulted in low-temperature sintering of the composites. The rearrangement and solution-precipitation, which are the mechanisms of liquid phase sintering, enhanced the densification of zirconia in the temperature range of 1000ºC to 1150ºC. The zirconia grains grew slightly, even though liquid phase sintering occurred. The zirconia grains became slightly larger, moreover, with increases in the amount of mica added. By contrast, the mica grains in the composite containing 30 vol% mica were fine grains, even at 1150ºC, and the sizes were less than 1 µm. Larger mica grains were formed with increases in the firing temperature and the amount of mica added. Dense composites containing 30–36 vol% mica were machinable using a drill made of hard metal, while those containing 49 vol% mica could be machined sufficiently with a conventional high-speed steel tool. The machinable composite containing 30 vol% mica had a bending strength of 552 MPa and a fracture toughness of 3.9 MPa·m° 1/2. The mechanical properties were lowered, however, with increases in the amount of mica added.

Disclosure statement

No potential conflict of interest was reported by the authors.

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