Effects of Ceramic Density and Sintering Temperature on the Mechanical Properties of a Novel Polymer-Infiltrated Ceramic-Network Zirconia Dental Restorative (Filling) Material

Authors' Contribution:
Study Design A
Data Collection B
Statistical Analysis C
Data Interpretation D
Manuscript Preparation E
Literature Search F
Funds Collection G

BCDEF 1,2   **Weiyan Li**
AG 1,2   **Jian Sun**

1 Department of Prosthodontics, Ninth People's Hospital, Shanghai JiaoTong University School of Medicine, Shanghai, P.R. China
2 Shanghai Key Laboratory of Stomatology and Shanghai Research Institute of Stomatology, National Clinical Research Center of Stomatology, Shanghai, P.R. China

Corresponding Author:
Source of support:
Jian Sun, e-mail: doctorsunjian74@aliyun.com
This study was supported by Key Project of Scientific Research Innovation, Shanghai Municipal Education Commission (No.12ZZ116), by the Shanghai Summit and Plateau Disciplines and by Science and Technology Development Fund of Pudong New Area (PKJ2015-Z05)

Background:
Polymer-infiltrated ceramic-network (PICN) dental material is a new and practical development in orthodontics. Sintering is the process of forming a stable solid mass from a powder by heating without melting. The aim of this study was to evaluate the effects of sintering temperature on the mechanical properties of a PICN zirconia dental material.

Material/Methods:
A dense zirconia ceramic and four PICN zirconia dental materials, with varying porosities, were sintered at three different temperatures; 12 PICN zirconia dental materials based on these porous ceramics were prepared, as well as a pure polymer. After the specimen preparation, flexural strength and elastic modulus values were measured using the three-point bending test, and fracture toughness were determined by the single-edge notched beam (SENB) method. The Vickers hardness test method was used with an indentation strength (IS) test. Scanning electron microscopy (SEM) was used to examine the microstructure of the ceramic surface and the fracture surface.

Results:
Mechanical properties of the PICN dental materials, including flexural strength, elastic modulus, fracture toughness, and hardness, were more similar to the properties of natural teeth when compared with traditional dental ceramic materials, and were affected by the density and sintering temperature. SEM showed that the porous ceramic network became cohesive and that the length of cracks in the PICN dental material was reduced.

Conclusions:
PICN zirconia dental materials were characterized by similar mechanical properties to natural dental tissues, but further studies are required continue to improve the similarities with natural human enamel and dentin.

MeSH Keywords:
Ceramics • Dental Materials • Prosthodontics

Full-text PDF:
https://www.medscimonit.com/abstract/index/idArt/907097

📄 3594   🗂 4   📊 6   📖 31

Background

Computer-aided design and computer-aided manufacturing (CAD/CAM) are currently gaining popularity in esthetic dentistry and prosthodontics with dental materials under development to support the emerging restorative (filling) dental materials market [1]. Currently, ceramics and polymers are the two main restorative materials in esthetic dentistry [2]. Ceramic materials show excellent biocompatibility, good wear resistance, and good corrosion resistance, with similar aesthetic performance to natural teeth. However, brittleness is the weakness of ceramic materials, and when the deformation is between 0.1–0.3%, fracture failure may occur. When subjected to repeated chewing and non-axial loading, a tiny crack inside the material will expand and cause the ceramic material to undergo fatigue damage, eventually leading to the destruction and failure of the restoration [3–5]. Also, the elastic modulus and hardness of ceramic materials are much greater compared with those of natural teeth, which can result in excessive wear on the opposing teeth during daily use [6]. Sintering, the process of forming a stable solid mass from a powder by heating without melting, can be used with ceramics.

However, resin materials used in dentistry have high ductility (malleability), are simple to manufacture and use, and are antagonist cusp friendly. However, the curing shrinkage of resin materials, low mechanical properties, and poor wear resistance restrict their further development and application. Although attempts have been made to improve resin restorations, and their mechanical properties have improved to some extent, the use of direct dental composites is accepted only for small restorations, and the results of the use of large resin restorations remain poorly understood [7]. Therefore, there is a need to develop a new dental restorative material that incorporates the benefits of both ceramic and resin materials and provides characteristics similar to those of natural teeth. This goal has attracted recent research on the use of dental materials, and several relevant products have been developed recently.

In 2014, a new generation of ceramic dental materials was developed, including Vita Enamic (Vita Enamic Co., Vita Zahnfabric, Germany), which is the world's first hybrid dental ceramic, having a dual-network structure that combines the best properties of ceramic and composite materials [8,9]. The hybrid ceramic, known as a polymer-infiltrated ceramic-network (PICN) dental material, consists of a sintered ceramic matrix containing pores that are filled with a polymer material, resulting in flexural strength, hardness, and fracture toughness values of 150–160 MPa, 2.5 GPa, and 1.5 MPam$^{1/2}$, respectively. Since 2014, more PICN dental materials have been developed, with differences between their mechanical properties, which are affected by parameters that include the density of the ceramic network and the sintering temperatures used.

The aim of this study was to evaluate the effects of sintering temperature on the mechanical properties of a PICN zirconia dental material and to compare these mechanical properties with other PICNs. The hypothesis that drives this study is that the optimal performance of PICN zirconia dental material will be achieved at a specific ceramic network density and sintering temperature.

Material and Methods

Manufacturing of samples

Nano-sized zirconia powder (TZ-3YB-E) (Tosoh, Japan) was used to prepare specimens by the cold isostatic press (CIP) and dry press methods. Different porosities of the samples were achieved by adding varying amounts of polyethylene glycol (PEG). The nano-sized zirconia powder and PEG were mixed in specific ratios, and the mixtures were then uniaxially compacted at 20 MPa in a stainless steel die for 30 s, and recompressed by the CIP method at a pressure of 200 MPa for 5 min. After pressing, blocks of the materials were selected for sintering at increasing temperatures of 1,100°C, 1,200°C, and 1,300°C in a Thermolyne high-temperature tube furnace (Motoyama NH-2025D/MS 1368, Japan). The sintering temperature was first increased at a ramp rate of 5°C/min, from room temperature to 500°C, held for one hour, heated at the same rate to the peak temperature, held for six hours, and then furnace-cooled.

After sintering, some of the blocks were filled with polymer to form the polymer-infiltrated ceramic-network (PICN) zirconia dental material. The porous zirconia ceramic network structure was infiltrated with a mixture of two monomers, triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA), which were mixed in a 1: 1 (mol) ratio in a vacuum chamber [10].

To improve the adhesion between the ceramic and polymer, the porous ceramic network was conditioned with the coupling agent, methacryloxypropyltrimethoxysilane (MPS), before the monomer was infiltrated [11,12]. Therefore, the polymer network was chemically cross-linked to the ceramic network to form an interpenetrating network system. Then, polymerization reactions were conducted at 180°C and 300 MPa for one hour to form a polymer network, leading to the formation of the PICN material [1].

PICN dental materials tested

Twenty-eight materials were tested in this study: three dense zirconia ceramics and twelve PICN zirconia dental materials with varying porosities sintered at three different

Table 1. Tested porous materials and PICN based on porous materials.

Sintered temperature/density	Dense ceramic [%]	Porous ceramic [%]	Porous ceramic [%]	Porous ceramic [%]	Porous ceramic [%]
1100°C	100	92	85	74	58
1200°C	100	92	85	74	58
1300°C	100	92	85	74	58

temperatures; twelve PICNs based on the porous ceramics mentioned described above; and the pure polymer. The densities of the ceramics were measured using a method based on the Archimedes' principle of water displacement, and the dense zirconia ceramics were considered to be 100% dense. The densities of the porous ceramics were determined based on the corresponding dense ceramics. Details of the tested materials are provided in Table 1.

Flexural strength and elastic modulus

After manufacturing, each group of specimens (n=5) were cut out of blocks with a diamond saw to enable measurements of the flexural strength and static elastic modulus. All bending bars (20×3×2.5 mm³) were polished using diamond-embedded wheels of 45 μm grit size with water and then to 2.5 and 1 μm with polycrystalline diamond paste. The flexural strength of the specimens was evaluated via the three-point bending test (Instron 5566) (Instron, Norwood, MA, USA) with an across-head speed of 0.5 mm/min. The load and the corresponding deflection were recorded. The flexural strength δ_f was calculated according to the formula (ISO 6872) [13]:

$$\delta_f = \frac{3fl}{2wh^2} \tag{1}$$

Where f was the load at fracture, l was the span tested (here, 16 mm), w the width and h the height of the specimen.

The elastic modulus was calculated according to the three-point bending results using the equation [8]:

$$E_f = \frac{l^3}{4000wh^3\left(\frac{d}{f} - \frac{d_c}{f_c}\right)} \tag{2}$$

Where d was the deflection corresponding to the load f, and dc and fc were the calibration values of d and f, respectively. The mean values and standard deviations (SD) of the flexural strength and elastic modulus are listed in Tables 2–4.

Fracture toughness

After polishing, the beams (20×3×6 mm³) for the fracture toughness test were pre-notched to a depth of 3 mm through a width of 6 mm at the mid-span of each bar, using a high-speed

cutting machine. The final length of the notch was measured using a microscope. The fracture toughness of the notched beam was determined by the standard single-edge notched beam (SENB) method [14], using a stroke rate of 0.05 mm/min, and a three-point bending apparatus (Instron 5566) (Instron, Norwood, MA, USA) with a span of 16 mm.

The *KIC* was calculated using the equation:

$$K_{IC} = \frac{0.0474fl\left(\frac{a}{w}\right)^{\frac{1}{2}}\left(1.99 - \frac{a}{w}\left(1 - \frac{a}{w}\right)\left(2.15 - 3.93\left(\frac{a}{w}\right) + 2.7\left(\frac{a}{w}\right)^2\right)\right)}{bw^{\frac{3}{2}}\left(1 + \frac{2a}{w}\right)\left(1 - \frac{a}{w}\right)^{\frac{3}{2}}} \tag{3}$$

where f was the fracture load, l was the roller span, b was the breadth of the bar, w was the width of the bar, and a was the length of the notch.

Vickers hardness test (method HV5)

The specimens (20×12×5 mm³) were polished to a high-luster finish. The Vickers hardness test (method HV5) was then used with an indentation strength (IS) test to measure the indentation strength at a load of 3N with a dwell time of 20 s (Tukon 2100B) (Instron, Norwood, MA, USA). The hardness was determined using the equation [15]:

$$H = 1.854 \cdot \frac{f}{d^2} \tag{4}$$

where f was the load and d was the diagonal indentation length.

Scanning electron microscopy (SEM)

Representative samples were gold-coated for scanning electron microscopy (SEM) using a SU3500 microscope (Hitachi High Technologies, Japan). SEM observations were made on the morphology of both the polished and the fractured samples.

Statistical analysis

The results were expressed as the mean ± standard deviation (SD). One-way analysis of variance (ANOVA) was used for comparison of parametric data. Statistical analysis was performed using SPSS for Windows, version 22.0 (SPSS Inc., Chicago, IL, USA). A p-value <0.05 was considered to be statistically significant.

Table 2. Measured physical properties of the PICN and porous materials sintered at 1100°C.

Material/density	Flexural strength [MPa]	Elastic-modulus [GPa]	Fracture toughness (MPam$^{1/2}$)	Hardness HV5 [GPa]
Dense ceramic 100%	252.59±9.99	86.42±3.99	3.76±0.10	5.89±0.06
PICN 92%/	96.23±0.76	38.80±1.62	1.40±0.06	4.90±0.06
Porous ceramic 92%	46.34±4.15	26.14±2.59	1.15±0.10	1.65±0.03
PICN 85%/	84.02±2.03	25.62±0.80	1.16±0.03	2.93±0.08
Porous ceramic 85%	36.70±1.71	4.75±0.14	0.85±0.06	0.98±0.03
PICN 74%/	68.51±2.58	18.54±0.94	0.97±0.04	1.01±0.03
Porous ceramic 74%	29.53±2.58	3.29±0.14	0.68±0.05	0.45±0.01
PICN 58%/	58.00±1.14	14.56±0.51	0.83±0.03	0.39±0.02
Porous ceramic 58%	23.88±1.64	1.57±0.19	0.51±0.03	0.29±0.01
Polymer	97.08±1.87	3.09±0.17	1.08±0.03	0.23±0.00

Table 3. Measured physical properties of the PICN and porous materials sintered at 1200°C.

Material/density	Flexural strength [MPa]	Elastic-modulus [GPa]	Fracture toughness (MPam$^{1/2}$)	Hardness HV5 [GPa]
Dense ceramic 100%	538.55±2.58	155.22±6.47	7.48±0.27	12.26±0.05
PICN 92%/	178.12±2.70	77.48±2.28	2.97±0.17	9.83±0.06
Porous ceramic 92%	130.89±7.58	37.37±2.75	2.18±0.12	3.92±0.03
PICN 85%/	135.92±1.54	56.05±2.42	2.39±0.16	6.86±0.06
Porous ceramic 85%	93.28±1.95	24.53±0.96	1.97±0.10	1.96±0.01
PICN 74%/	111.39±2.00	44.92±1.31	1.91±0.06	2.46±0.06
Porous ceramic 74%	68.61±5.63	14.52±1.65	1.30±0.12	1.19±0.02
PICN 58%/	94.28±2.19	29.67±0.76	1.47±0.17	0.97±0.03
Porous ceramic 58%	59.37±4.79	10.55±0.74	0.69±0.03	0.59±0.01
Polymer	97.08±1.87	3.09±0.17	1.08±0.03	0.23±0.00

Table 4. Measured physical properties of the PICN and porous materials sintered at 1300°C.

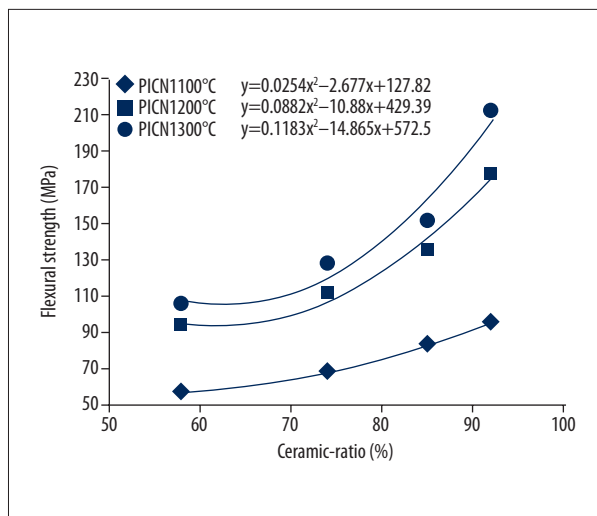| Material/density	Flexural strength [MPa]	Elastic-modulus [GPa]	Fracture toughness (MPam$^{1/2}$)	Hardness HV5 [GPa]
Dense ceramic 100%	576.15±1.42	186.79±1.81	5.50±0.23	13.14±0.04
PICN 92%/	211.77±2.37	100.52±1.04	2.48±0.12	10.79±0.06
Porous ceramic 92%	139.11±1.75	56.83±0.78	2.04±0.18	4.81±0.03
PICN 85%/	152.84±12.80	70.01±1.64	2.26±0.06	7.87±0.06
Porous ceramic 85%	122.95±2.70	34.00±1.20	1.78±0.19	2.74±0.03
PICN 74%/	127.67±6.78	57.55±1.17	1.78±0.06	3.93±0.03
Porous ceramic 74%	103.75±2.80	21.63±0.75	1.24±0.16	1.86±0.02
PICN 58%/	106.81±1.30	40.24±0.63	1.36±0.06	1.97±0.04
Porous ceramic 58%	68.42±5.03	13.95±2.89	0.64±0.04	0.88±0.01
Polymer	97.08±1.87	3.09±0.17	1.08±0.03	0.23±0.00

Figure 1. Comparison of flexural strength of four polymer-infiltrated ceramic-network (PICN) dental materials sintered at three temperatures.
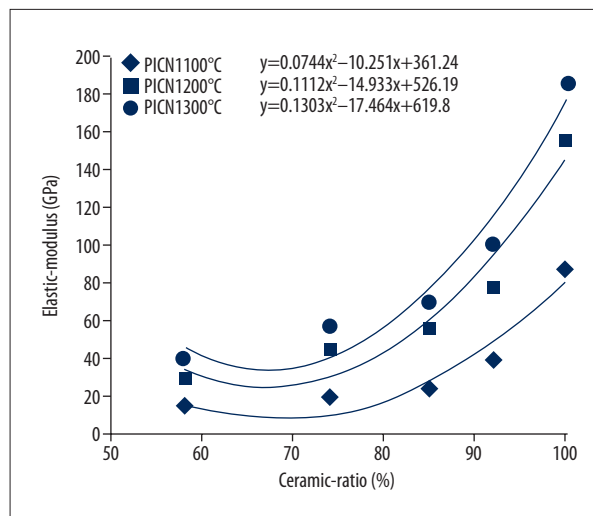

Figure 2. Elastic modulus of polymer-infiltrated ceramic-network (PICN) dental materials based on three sintered temperatures.

Results

Flexural strength and elastic modulus of the sintered materials

The flexural strength of the materials sintered at the three different temperatures is shown in Tables 2–4, and Figure 1. With decreasing density, the flexural strength of the samples in each temperature group was reduced. The flexural strength of the four porous ceramics was reduced from 139.1 MPa to 68.4 MPa; for example, the flexural strength of the polymer-infiltrated ceramic-network (PICN) material was reduced from 211.8 MPa to 106.8 MPa for the 1,300°C specimen. The flexural strength values obtained for PICN 85% and 74% for the 1.300°C group were not significantly different at a 95% confidence interval (CI) level ($p=0.112$). However, all the other flexural strength values were significantly different, when analyzed by single-factor ANOVA (Tables 2–4). Also, Tables 2–4 show that the when analyzed by one-way ANOVA, the flexural strengths of the PICN material were significantly greater compared with those of the pre-infiltrated ceramics ($p<0.05$).

The flexural strengths of the materials sintered at the three different temperatures are shown in Figure 1. The flexural strength increased with increasing sintering temperature, but the degree of increase tapered off. One-way ANOVA analysis indicated significant differences between the three groups with respect to the flexural strength. The elastic modulus values of the materials tested are shown in Tables 2–4 and Figure 2.

An increase in material density resulted in higher elastic modulus values at the same sintering temperature. The 100-value (x-axis) of 186.79 GPa corresponded to the elastic modulus of

the dense ceramic sintered at 1,300°C, as shown in Figure 2. As the density decreased, the elastic modulus of the PICN material dropped to 40.24 GPa. Tables 2–4 show that as the sintering temperature increased, the elastic modulus values increased, in both the porous ceramics and PICN. Statistical analysis showed that there was a significant difference in the elastic moduli between both groups with different densities at the same sintering temperature ($p<0.05$), and the groups with different sintering temperatures at the same density ($p<0.05$).

Fracture toughness

The values of the fracture toughness derived from Equation (3) are shown in Tables 2–4. Except for 1300°C PICN 92% and PICN 85% ($p=0.161$) and the 1300°C porous ceramic 92% and 85% ($p=0.586$), significant differences in the fracture toughness were found. In contrast to the other mechanical properties, Figure 3 shows that an increase in the sintering temperature, from 1100°C to 1200°C resulted in a significant increase in the fracture toughness and then a slight decline from 1200°C to 1300°C ($p<0.05$).

Hardness

The results of the hardness test were similar to those of the elastic modulus. The data on PICN materials sintered at 1300°C from the indentation strength (IS) system showed important differences in the hardness values between the upper and lower boundaries at 13.14 GPa and 0.23 GPa for the dense ceramic and polymer, respectively. The hardness values were significantly different ($p<0.05$) (Tables 2–4; Figure 4).
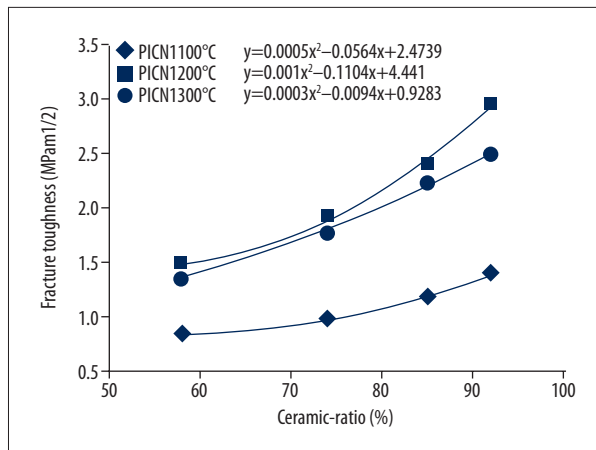
Figure 3. Comparison of fracture toughness of polymer-infiltrated ceramic-network (PICN) dental materials sintered at different temperatures with varying ceramic ratio.

Figure 4. Hardness of three different temperature sintered polymer-infiltrated ceramic-network (PICN) zirconia dental materials for various ceramic fractions.

Scanning electron microscopy (SEM)

Figure 5 shows the scanning electron microscope (SEM) images of the pre-infiltrated porous ceramic with 74% sintered at 1,300°C (Figure 5A), PICN with 74% sintered at 1,300°C (Figure 5B), fracture surfaces of dense zirconia ceramic sintered at 1,300 °C (Figure 5C) and fracture surfaces of the PICN material with 74% sintered at 1,300°C (Figure 5D).

Figure 5A shows that the PICN material was composed of irregularly-shaped ceramic phases, resulting in irregular pores that were connected with each other. Figure 5B shows that the dominant ceramic network structure and the reinforcing polymer network structure were merged fully with one another. On the SEM images, the light gray areas represented the ceramic network structure, and the dark gray areas showed the polymer network structure. Typical cracks for the dense zirconia ceramic were detected (Figure 5C), but no propagating cracks were detected for the PICN with 74% sintered at 1,300°C (Figure 5D). The remaining specimens had similar microstructures and fractographic features, except that the ratio between the porous ceramic and polymer contents was different; these findings have not been included.

Discussion

The hybrid polymer-infiltrated ceramic-network (PICN) zirconia dental material described in this study represents an important development of computer-aided design and computer-aided manufacturing (CAD/CAM) of dental restorative (filling) materials. This study was primarily conducted to evaluate the mechanical properties of the new PICN materials and to identify the parameters that could influence their mechanical
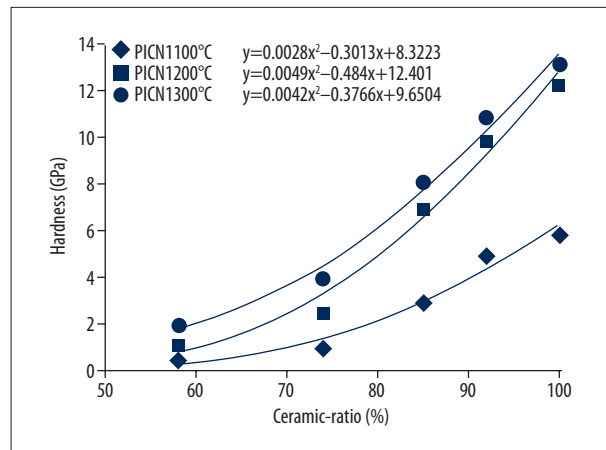
properties. The findings of this study support the previously reported findings of Coldea et al., on PICN zirconia dental material [2]. In this present study, four different PICNs and three different sintering temperatures were evaluated, which showed that the mechanical behaviors of the PICNs were similar to those of human enamel and dentin. A review of the literature on the physical properties of human teeth has shown that enamel has an elastic modulus, hardness, and fracture toughness of 48–105.5 GPa, 3–5.3 GPa, and 0.6–1.5 MPam$^{1/2}$, respectively; dentin has an elastic modulus, hardness, and fracture toughness of 16–20.3 GPa, 0.6–0.92 GPa, and 2.2–3.1 MPam$^{1/2}$, respectively [2].

Tables 2–4, show that the higher initial density of the pre-infiltrated ceramic at the same sintering temperature gave both the porous ceramic and PICN superior mechanical properties (flexural strength, elastic modulus, fracture toughness, and hardness), although there was no significant difference between the groups. Also, the mechanical properties of the PICNs were significantly greater than those of the pre-infiltrated ceramics.

Flexural strength is a meaningful mechanical property for brittle materials [16]. The PICNs sintered at 1,300°C showed flexural strength values in the range of 106.81–211.77 MPa for the four different porosities. These values were comparable with that of dentin, 212.9 (±41.9) MPa [17]. Therefore, PICN dental material should be able to resist the shear and compressive forces of the anatomic system that includes the teeth, jaws and soft tissues (the stomatognathic system), allowing the PICN dental material to be used in a variety of dental applications.

In this study, similar trends were found for the elastic modulus. The elasticity values of 40.2–100.5 GPa (1,300°C PICN) are slightly lower than those of the human enamel (between
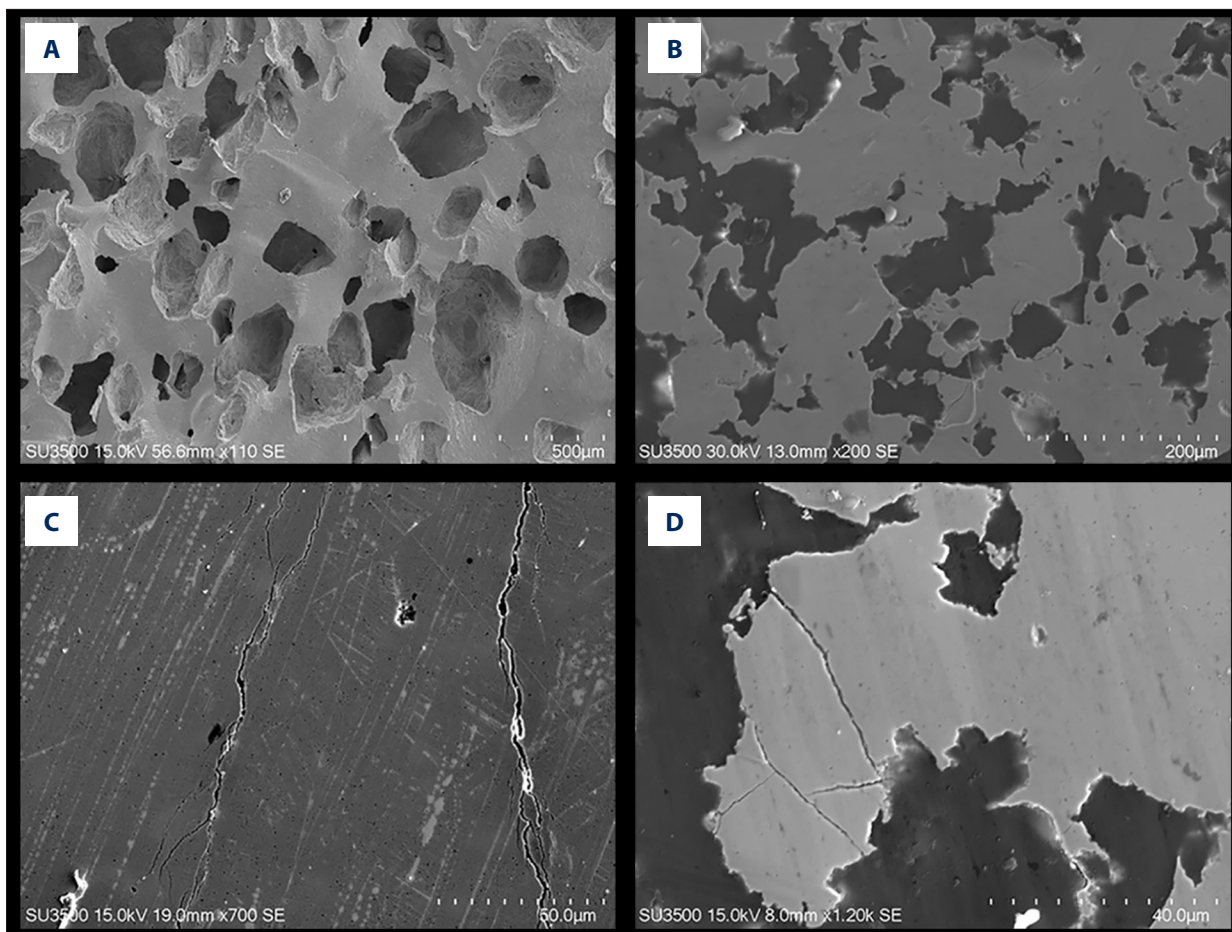
Figure 5. Scanning electron microscopy (SEM) images of the polymer-infiltrated ceramic-network (PICN) zirconia dental material. (**A**) Polymer-infiltrated ceramic-network (PICN) zirconia dental material 74% at 1,300°C. (**B**) PICN 74% at 1,300°C. (**C**) Fracture surfaces of dense zirconia ceramic at 1,300°C. (**D**) Fracture surfaces of PICN 74% at 1,300°C.

48–105.5 GPa) [18,19]. To date, no dental restoration material has exhibited values in this elastic modulus range. The other two temperature groups had lower elastic modulus values. A high elastic modulus material absorbs less stress, and the excess stress will concentrate near the interface due to the modulus mismatch between the restorative material and the human teeth, causing tooth damage during mastication loading [20]. Owing to the elastic modulus match with enamel, the PICN may offer a more uniform stress distribution, especially during loading, and the tooth will be protected from cracking.

Tables 2–4 also show the fracture toughness of the materials. Fracture toughness has been reported as one of the key properties associated with the clinical performance of dental materials [21]. In comparison with other restorative dental materials, almost all the infiltrated-ceramic materials have high fracture toughness, which is slightly greater than that of the Vita Enamic PICN zirconia dental material (K1c~1.5 MPam$^{1/2}$). Therefore, it is not surprising that, in this study, the PICN with a greater polymer fraction was characterized by lower fracture
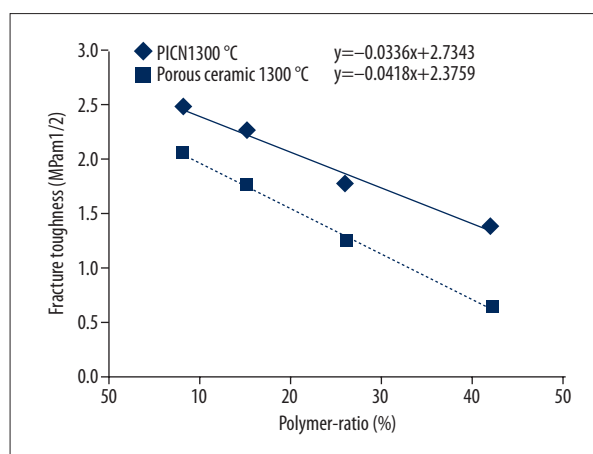

Figure 6. Comparison of fracture toughness of polymer-infiltrated ceramic-network (PICN) dental materials and porous ceramics with a 1,300°C sintered temperature.

toughness. The reason for this phenomenon was explained by the ceramic network. When the sintering temperature was 1,100°C, the influence of the ceramic precursor was more significant. Figure 6 shows that the fracture toughness of the PICN with increasing polymer content decreased, although the degree of this decrease tapered off. This result suggests that, due to the addition of the polymer, the fracture toughness of the PICN was reinforced.

To explain the strengthening mechanism of sintering and polymer infiltration, previous studies have shown that the crack growth in the PICN was hindered by different strengthening mechanisms [22]. In previous studies that have tested PICN, the reinforcement was found to be mainly attributed to polymer bridges [11]. Other strengthening effects typical of ceramic-polymer composites are crack branching and crack deflection [23]. The microstructures of the propagating crack surfaces of the PICN zirconia dental material are shown in Figure 5C and 5D, indicating that the polymer infiltration significantly changed the fracture pattern of the ceramic. The scanning electron microscope (SEM) imaging (Figure 5C) of the dense zirconia ceramic cracks did not show any obvious branching and deflection, whereas that of the PICN did (Figure 5D), which indicates that the polymer infiltration successfully hindered the crack growth and gave the materials high fracture toughness. In summary, the tendency for the brittle fracture of the PICN is lower than that of the pure ceramics, and excellent CAD/CAM handling can also be achieved [9]

In this study, the level of hardness of the PICN 74% sintered at 1,300°C was approximately 3.93 GPa, which is intermediate between that of dentin (0.6–1 GPa) [24,25], and enamel (3–5 GPa) [19,26]. The hardness levels of the dense ceramics sintered at 1,300°C (13.14 GPa) were significantly greater than that of enamel. A dental material that is harder than natural enamel may cause more severe wear on the opposing dentition [27]. Therefore, with the lower hardness levels of PICNs when compared with most commercial dental ceramics, the opposing teeth will be protected from excessive wear.

As shown in Figures 1–4, all the mechanical properties, except the fracture toughness, of the materials in this study had similar increasing trends as the sintering temperature increased from 1,100°C to 1,300°C. When the sintering temperature was increased, the increased grain growth or inter-particle contact may occur, resulting in increased density and strength. Therefore, the density and strength of the samples that were sintered at high temperature was greater when compared with those at a low temperature. Also, the flexural strength, elastic modulus, and hardness increased rapidly from 1,100°C to 1,200°C, and then slowly increased to 1,300°C. This result demonstrates that the 1,100°C temperature results in a below normal zirconia ceramic densification, whereas 1,200°C was near

the peak temperature for densification. The fracture toughness significantly increased, when the temperature rose from 1,100°C to 1,200°C, although when the temperature was increased to 1,300°C, the fracture toughness decreased slightly. The ceramic transition temperature ranged from 1200°C to 1300°C. The reason for the rising fracture toughness may be similar to that mentioned above. The slight decrease in the trend of fracture toughness from 1,200°C to 1,300°C may have originated from the constantly increasing grain size and decreasing pore size associated with the higher processing temperature.

A previously published study showed that the fracture toughness of the zirconia ceramic was greatest when the grain size of the zirconia was approximately equal to the critical grain size [10]. Considering the trend of the fracture toughness, it is reasonable to suggest that the critical grain size of the zirconia ceramic appears at a temperature between 1,200°C and 1,300°C. An explanation for the decreased fracture toughness is residual stress from the polymerization shrinkage of the heat-cured polymer [10]. The tension stress has to be overcome in the process of rupture, and the crack tip can also be shielded because of the residual stress around the ceramic particles. Residual compressive stress can counteract tensile stress, which prevents the crack from propagating and enhances the strength. A higher sintering temperature of 1,300°C results in smaller pore sizes than 1,200°C, and small pores are generated when less compressive force exists after the curing process. Therefore, the fracture toughness of PICN at 1,300°C was slightly lower than that at 1,200°C.

The findings of this study have shown that PICN zirconia dental material has the potential to be successful in the dental restorative materials market. Comparing the results of this study with the characteristics of the structure of natural teeth, a promising step towards replicating the composition of natural teeth has been realized by optimizing the ceramic network density and sintering temperature with PICNs. However, the main drawback is that the traditional method used to produce the PICN results in an irregular porous ceramic network, which gives the ceramic body low homogeneity and thus decreases the mechanical strength of the materials [28]. Three-dimensional printing is an alternative route that offers versatility and high precision, and is also environmentally friendly, and allows tailored pore structures [29]. It is accepted that greater structural stability results in improved structural properties of dental materials [30,31]. Therefore, further studies are required to optimize the microstructure of the porous ceramic network through three-dimensional printing, to replicate the structural and mechanical properties of dentin and enamel. These further studies are ongoing in our laboratory.

Conclusions

The findings of this study showed that polymer-infiltrated ceramic-network (PICN) zirconia dental materials, sintered at the same temperature, were characterized by similar mechanical properties to natural dental tissues, including flexural strength, elastic modulus, fracture toughness, and hardness, were directly proportional to the ratio between the porous ceramic and polymer content. All the mechanical properties of the polymer-infiltrated ceramics were significantly greater compared with those of the pre-infiltrated ceramics. The flexural strength, elastic modulus, and hardness of the materials showed similar increasing trends as the sintering temperature increased from 1,100°C to 1,300°C. Also, the fracture toughness increased as the temperature increased from 1,100°C to 1,200°C and then

decreased slightly at 1,300°C. The findings indicated that ceramic network density and sintering temperature influence the mechanical properties of the PICN zirconia dental material.

Acknowledgments

Conflict of interest

The authors declare that they have no conflict of interest.

References:

1. Nguyen J, Ruse D, Phan A, Sadoun M: High-temperature-pressure polymerized resin-infiltrated ceramic networks. J Dental Res, 2014; 93(1): 62–67

2. Coldea A, Swain MV, Thiel N: Mechanical properties of polymer-infiltrated-ceramic-network materials. Dental Materials, 2013; 29(4): 419–26

3. Kim JW, Bhowmick S, Hermann I, Lawn BR: Transverse fracture of brittle bilayers: Relevance to failure of all-ceramic dental crowns. J Biomed Materials Res Part B: Appl Biomater, 2006; 79(1): 58–65

4. Niu X, Yang Y, Soboyejo W: Contact deformation and cracking of zirconia/cement/foundation dental multilayers. Materials Sci Engineering A, 2008; 485(1): 517–23

5. Donovan TE: Factors essential for successful all-ceramic restorations. J Am Dental Assoc, 2008; 139: S14–18

6. Heintze S, Cavalleri A, Forjanic M et al: Wear of ceramic and antagonist – a systematic evaluation of influencing factors *in vitro*. Dental Materials, 2008; 24(4): 433–49

7. Jongsma L, Kleverlaan C, Feilzer A: Clinical success and survival of indirect resin composite crowns: Results of a 3-year prospective study. Dental Materials, 2012; 28(9): 952–60

8. Dirxen C, Blunck U, Preissner S: Clinical performance of a new biomimetic double network material. Open Dent J, 2013; 7: 118–22

9. Della Bona A, Corazza PH, Zhang Y: Characterization of a polymer-infiltrated ceramic-network material. Dental Materials, 2014; 30(5): 564–69

10. Chaiyabutr Y, Giordano R, Pober R: The effect of different powder particle size on mechanical properties of sintered alumina, resin and glass infused alumina. J Biomed Mat Res Part B: Applied Biomaterials, 2009; 88(2): 502–8

11. Steier VF, Koplin C, Kailer A: Influence of pressure-assisted polymerization on the microstructure and strength of polymer-infiltrated ceramics. J Materials Sci, 2013; 48(8): 3239–47

12. Sideridou ID, Karabela MM. Effect of the amount of 3-methacyloxyprop-yltrimethoxysilane coupling agent on physical properties of dental resin nanocomposites. Dental Materials, 2009; 25(11): 1315–24

13. ISO I. 6872: Dentistry-ceramic materials. Switzerland: International Organization for Standardization (IOS). 2008

14. ASTM International. Standard test method for measurement of fracture toughness. ASTM International. 2011

15. Kayahan M, Nekoofar MH, Kazandağ M et al: Effect of acid etching procedure on selected physical properties of mineral trioxide aggregate. Int Endodontic J, 2009; 42(11): 1004–14

16. Petrini M, Ferrante M, Su B: Fabrication and characterization of biomimetic ceramic/polymer composite materials for dental restoration. Dental Materials, 2013; 29(4): 375–81

17. Plotino G, Grande NM, Bedini R et al: Flexural properties of endodontic posts and human root dentin. Dental Materials, 2007; 23(9): 1129–35

18. Ausiello P, Rengo S, Davidson CL, Watts DC: Stress distributions in adhesively cemented ceramic and resin-composite Class II inlay restorations: A 3D-FEA study. Dental Materials, 2004; 20(9): 862–72

19. He LH, Swain MV: Nanoindentation derived stress–strain properties of dental materials. Dental Materials, 2007; 23(7): 814–21

20. Costa A, Xavier T, Noritomi P et al: The influence of elastic modulus of inlay materials on stress distribution and fracture of premolars. Operative Dentistry, 2014; 39(4): 160–70

21. Della Bona A: Bonding to ceramics: Scientific evidences for clinical dentistry; Artes Médicas. Sao Paulo, Brazil, 2009

22. Bowling GD, Faber KT, Hoagland R: Computer simulations of R curve behavior in microcracking materials. J Am Ceramic Soc, 1987; 70(11): 849–54

23. Kinloch A, Maxwell D, Young R: Micromechanisms of crack propagation in hybrid-particulate composites. J Materials Sci Letters, 1985; 4(10): 1276–79

24. Lawn BR, Lee JJ: Analysis of fracture and deformation modes in teeth subjected to occlusal loading. Acta Biomaterialia, 2009; 5(6): 2213–21

25. Mahoney E, Holt A, Swain M, Kilpatrick N: The hardness and modulus of elasticity of primary molar teeth: An ultra-micro-indentation study. J Dentistry, 2000; 28(8): 589–94

26. Park S, Quinn J, Romberg E, Arola D: On the brittleness of enamel and selected dental materials. Dental Materials, 2008; 24(11): 1477–85

27. He L-H, Swain M: A novel polymer infiltrated ceramic dental material. Dental Materials, 2011; 27(6): 527–34

28. Preiss A, Su B, Collins S, Simpson D: Tailored graded pore structure in zirconia toughened alumina ceramics using double-side cooling freeze casting. J Eur Ceramic Soc, 2012; 32(8): 1575–83

29. Zhang X, Zhang Y: Tissue engineering applications of three-dimensional bioprinting. Cell Biochem Biophys, 2015; 72(3): 777–82

30. Yi H, Wu L, Wu L et al: Crystal structure of high-temperature phase β-NaSrBO3 and photoluminescence of β-NaSrBO3: Ce3+. Inorg Chem, 2016; 55(13): 6487–95

31. Bauer J, Hengsbach S, Tesari I et al: High-strength cellular ceramic composites with 3D microarchitecture. Proc Nat Acad Sci, 2014; 111(7): 2453–58