Effect of polymer amount on the mechanical behavior of polymer-infiltrated zirconia-ceramic composite at different pre-sintering temperatures

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Abstract
Polymer-infiltrated zirconia-ceramic composite (PIZC) comprises a pre-sintered zirconia-ceramic matrix and a polymer. In this work, pre-sintered zirconia-ceramic was infiltrated with varied amounts of methacrylate-based polymer. Therefore, this paper reports the effect of polymer amount on the mechanical behavior of PIZC at 1100 °C–1300 °C pre-sintering temperatures. Conventional mechanical tests were performed to obtain the elastic modulus and fracture toughness while Vickers micro-indentations were employed to extract the Vickers hardness. Advanced mechanical behaviour analysis was characterized by plastic deformation resistance, elastic and plastic deformation components and brittleness index. Increasing the amount of polymer from 0 to 42% led to the corresponding decrease in elastic modulus, hardness and fracture toughness by at least 78, 85 and 75%, respectively. As the temperature was increased, both elastic modulus and hardness increased while the fracture toughness initially increased but decreased at higher temperature. Mechanical properties and polymer amount were well modelled by connected-grain models which usefully explained the densification process occurring at higher temperatures. Plastic deformation resistance and component and brittleness index confirmed better plastic properties for PIZC at higher polymer amounts and lower temperature. Therefore, in order to utilize the plastic properties of PIZC during the CAD/CAM process, these findings recommended the processing of PIZC at not-lower-than 26% polymer amount and 1100 °C, which could greatly facilitate its precision ductile machining mode realization. Finally, the results provide a technical guidance for the selection of appropriate polymer amount when fabricating dental restorations from this novel ceramic-composite.

1. Introduction

Many computer-aided design/computer-aided manufacturing (CAD/CAM) machinable ceramic materials have been developed for medical and engineering applications. In particular, ceramic materials have been applied in orthodontic restorative dentistry as bridges, crowns, dentures and implants due to their excellent biocompatible mechanical properties. These ceramics are undergoing continuous refinements and enhancements for improvements in wear-resistance, esthetics, biocompatibility, color stability, flexural strength and toughness and ductility. The various ceramics applied as CAD/CAM machinable materials include feldspar porcelain, leucite- and lithium disilicate-glass ceramics and yttria-tetragonal zirconia polycrystals [1]. The mechanical properties of these dental ceramics, however, greatly differ from those of enamel and dentine, causing residual tensile stresses due to the mismatch of elastic moduli at the enamel/ceramic interface and resulting in the wear of the antagonist enamel. Therefore, the research goals in dental materials characterization and synthesis are being channeled towards developing materials with similar mechanical properties and...
behaviour to enamel and dentine, hence the introduction of polymer-infiltrated-ceramic networks to the restorative dental world [2–6].

Established for indirect CAD/CAM dental restorations, polymer-infiltrated-ceramic network, as a hybrid biomedical composite material, combines both properties of ceramic and polymer to imitate the biomechanical properties of natural teeth [5, 7]. It is often used to fabricate invasive restorations, inlays, onlays, and veneers for posterior and implant-supported crowns using the CAD/CAM process [3, 4, 8, 9]. Roughly, it comprises a pre-sintered ceramic matrix, which can be either an inorganic silicate-based glass ceramic or a zirconia, filled and reinforced with a polymer network [6, 10]. Thus, a hybrid microstructure is achieved by the infiltration of pre-polymerized liquid monomers into the pre-sintered ceramic matrix which is then followed by thermally activated crosslinking polymerization process [11]. Vita Enamic containing 80% and 20% of feldspar and polymer, respectively, and Crystal Ultra consisting of 70% inorganic silicate glass filler cross-linked with 30% polymer are the commercially available polymer-infiltrated-ceramic networks which find increasingly wide usage in dental clinics [12]. Their in-vitro survival performance over an historical clinical reflection of 2 or 3 years has been well documented [13, 14].

Many research works have characterized mechanical properties for polymer-infiltrated-ceramic network. The reported hardness, elastic modulus, flexural strength and fracture toughness in literature are very close to those of human enamel and dentine [3, 10, 15–18]. Also, it exhibits lower elastic modulus and hardness than leucite and feldspar glass ceramics, enabling it with higher damage and edge chipping tolerance [2, 19–25]. Further, several studies reveal its superior sliding-contact fatigue fracture resistance [24, 25], enamel-like wear properties [26, 27] and long-term hydrolytic degradation resistance [9]. Also, it can withstand clinically acceptable masticatory forces [8] as well as be conventionally cemented with human dentine [28, 29]. However, it suffers from low flexural strength (103.36 MPa) and fracture toughness (0.44–1.72 MPa m1/2) [10], necessitating the need for an improvement in properties.

Analogous to conventional composite fabrication from ceramics and polymers, the ceramic matrix can be adjusted via the pre-sintering process. Therefore, the improvement of the mechanical properties of composites is possible by manipulating their microstructural grain-size distributions and fractions of the ceramic matrix via adjusting their pre-sintering temperature and time [3, 5, 7, 30]. In this regard, Coldea et al [3] enhanced the flexural strength of polymer-infiltrated-ceramic network materials from 131 to 160 MPa by changing the amount of its feldspathic ceramic from 59%–72%. Also, Li et al [5] applied zirconia ceramic in their polymer-infiltrated zirconia–ceramic composite (PIZC) which is being used in restorative dentistry as crowns, veneers and bridges [5], changing the ceramic amount at different pre-sintering temperatures ranging from 1000 °C–1150 °C, resulting in optimal properties at 1150 °C. Further, Li and Sun [7] obtained different ceramic fractions of PIZC even at a single pre-sintering temperature by adding varied amounts of polyethylene glycol to the precursor green zirconia. They then filled the remaining fractions with a methacrylate-based polymer and investigated the influence of ceramic density on mechanical properties at different pre-sintering temperatures [7]. They obtained ceramic density- and temperature-dependent mechanical properties for PIZC materials.

All the aforementioned mechanical behaviour studies have majorly concentrated on properties (elastic modulus, Vickers hardness, fracture toughness, flexural strength and shear bond strength) of PIZC. However, there is a need to explore more beyond this realm of mechanical behaviour. In particular, the role played by the polymer amount which fills the porosity of the pre-cursor porous zirconia has not been adequately clarified. Therefore, it is highly important to elucidate the influence of polymer amount on the deformation and fracture behaviors of PIZC and determine the structure-property prediction relationships using the semi-mechanistic models.

Advanced mechanical properties can be characterized by material’s resistance to plastic deformation and the induced deformation modes [31]. In Vickers indentations, the elastic-plastic deformation responses have been reported for dense ceramics [32]. Thus, the Vickers hardness is a hybrid intrinsic mechanical property, containing an aggregate elastic and plastic deformation from which the individual component can be discerned. The Sakai model which assumes a series configuration of elastic deformation and plastic deformation can be applied to partition Vickers indentation-induced deformation into elastic and plastic components, allowing the realization of a dominant material deformation behavior to be obtained [31, 33]. Furthermore, the Sakai model can be applied to extract material’s plastic deformation resistance from Vickers hardness and plane strain modulus. The occurrence of low plastic deformation resistance and high plastic deformation component in materials in microscale deformation process is more favored in the precision ductile mode machining process [34–36]. Invariably, the Sakai model has been applied to characterize dominant deformation mechanisms for mineralized tissues [37], zirconia [38–40], and lithia-based glass ceramics [41]. In addition, material’s brittleness index represents a competition between deformation and fracture in the material’s mechanical response to indentation [42, 43] and can be physically related to its machinability [44]. Accordingly, low brittleness index indicates that the material is associated with higher deformation than fracture while high brittleness index leads to higher fracture than deformation [42].
Li and Sun [7] have published elastic modulus, Vickers hardness and fracture toughness data for PIZC obtained from three-point bending, micro-indentation and single-edge notched beam tests, respectively, at different pre-sintering temperatures. However, the plastic deformation resistance, the elastic and plastic deformations and the brittleness index of PIZC consolidated at different pre-sintering temperatures have received insufficient attention. In order to have a comprehensive study of the mechanical behavior of PIZC, this paper performs advanced analyses of the published mechanical properties of PIZC, revealing the effect of polymer amount on properties. Therefore, this study aims to investigate the effect of polymer amount on the mechanical behaviour of PIZC at different pre-sintering temperatures. Specifically, it formulates structure-property relations for elastic modulus, hardness and fracture toughness as functions of polymer amount. Also, it reports the effect of polymer amount on plastic deformation resistance, elastic and plastic deformation components and brittleness index. Next, it correlates the mechanical behaviours of PIZC to its machinability. Finally, it is envisaged that the results can be applied to provide scientific and technical guidance for the selection of appropriate polymer amount when fabricating dental dentures from this novel ceramic-composite.

### 2. Materials and methodology

#### 2.1. Materials

Material investigated is the polymer-infiltrated zirconia-ceramic composite (PIZC) which is the latest addition to dental biomedical ceramic composite applied in orthodontic restorative dentistry. The manufacturing processes of PIZC included compaction, pre-sintering, polymerization and final sintering processes [7]. Briefly, PIZC was prepared by mixing specific amounts of nano-sized 3 mol% Y-TZP powders and polyethylene glycol to form green porous zirconia-ceramic compact materials with different porosities before the pre-sintering process. These materials were then isostatic cold-pressed at a pressure of 200 MPa. It should be noted that this applied pressure was far greater than the threshold (70 MPa) suggested to provide better compactness of zirconia grains [45]. Thus, different porosities were formed from the compacts which were then pre-sintered at 1100 °C, 1200 °C and 1300 °C temperatures in a thermolyne furnace [7]. The Archimedes’ principle was applied to measure the density of the porous zirconia ceramics relative to the density of fully dense zirconia with 0% porosity. The porosity density of porous zirconia-ceramic was calculated from the following [46]:

\[
\rho_p = 1 - \rho_t
\]

where \(\rho_p\) and \(\rho_t\) represent porosity and porous zirconia-ceramic densities, respectively. Table 1 displays the porosity amounts of PIZC at different pre-sintering temperatures. The porous zirconia-ceramics were infiltrated and filled with triethylene glycol dimethacrylate and urethane dimethacrylate monomers with a mole ratio of 1:1 in a vacuum chamber at a pressure of 0.04 Torr for 4 h [47], forming PIZC structures [7, 47]. Methacryloyloxypropytrimethoxysilane coupling agent was used to condition and improve the adhesive bond between porous zirconia-ceramic and monomers [48]. Polymerization process was then carried out to form an interpenetrating PIZC materials, chemically bonding and cross-linking polymer networks to the ceramic matrices at a temperature and pressure of 180 °C and 300 MPa, respectively [7]. This high pressure was used to ensure that defect-free structure was obtained in which the polymer networks filled the pre-sintered zirconia-ceramic matrices [49], leading to the formation of PIZC materials [50] with a hybrid structure containing both organic (polymer) and inorganic (tetragonal) phases [51]. Thus, the PIZC materials with different porosities ranging from 8%–42% were filled through this process. The materials were consequently designated as PIZC–8 P, PIZC–15 P, PIZC–26 P and PIZC–42 P representing PIZC with 8, 15, 26 and 42% polymer amounts, respectively. In addition, the zero-porosity fully dense zirconia (ZC–0P) samples were prepared by compaction and sintering at 1500 °C which has a tetragonal phase structure [1, 21]. Thus, the ZC–0 P, PIZC–8 P, PIZC–15 P, PIZC–26 P and PIZC–42 P materials are displayed in Table 1.

### Table 1. Different porosities of polymer-infiltrated zirconia-ceramic composite (PIZC) at different pre-sintering temperatures filled with various polymer amounts and the PIZC designations.

| Pre-sintering temperature (°C) | Porosity (%) | ZC–0 P | PIZC–8 P | PIZC–15 P | PIZC–26 P | PIZC–42 P |
|-------------------------------|--------------|--------|----------|-----------|-----------|-----------|
| 1100                          |              | 0      | 8        | 15        | 26        | 42        |
| 1200                          |              | 0      | 8        | 15        | 26        | 42        |
| 1300                          |              | 0      | 8        | 15        | 26        | 42        |
2.2. Characterization of mechanical properties

Elastic modulus of PIZC at different pre-sintering temperatures and polymer amounts was measured from the three-point bending test in accordance with the ISO (6872) standard [52]:

\[
E = \frac{l^3}{4000wh^3 \left( \frac{d}{f} - \frac{d}{f_c} \right)}
\]  

(2)

where \(E, f, l, w\) and \(f\) represent elastic modulus, fracture load, tested span, specimen’s width and height, respectively, while \(d\) represents the deflection due to \(f\); \(f_c\) and \(d_c\) are the calibrated values corresponding to \(f\) and \(d\), respectively.

The fracture toughness of notched specimens of PIZC at different temperatures and polymer amounts was conducted using the single-edge notched beam method. Thus, the fracture toughness was calculated applying the ASTM (E1820–09E1) method [53]:

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

(3)

where \(K_{IC}\) represents mode I fracture toughness, \(a\) represents notch’s span, \(b\) is the specimen’s breadth and other parameters are as previously defined.

Vickers hardness of PIZC at different pre-sintering temperatures and polymer amounts was obtained using indentation techniques at a maximum load of 3 N [7]. The Vickers hardness from micro-indentation instrumented experiments is governed by [54]:

\[
H_v = \frac{P_{\text{max}}}{(\pi \tan^2 \alpha)h^2}
\]  

(4)

where \(\alpha\) represents the equivalent conical half angle which is 70.3° for the applied Vickers indenter, \(h\) is the contact depth (figure 1) and \(P_{\text{max}}\) represents the applied maximum load (3 N). It should be noted this load lied within the microscale region [43, 55].

2.2.1. Advanced characterization of mechanical properties

Sakai’s model based on a series configuration of elastic and plastic deformation components was used to extract plastic deformation resistance \((H_p)\) from micro-indentation tests. Briefly, the total contact depth \((h_t)\) at \(P_{\text{max}}\)
presumably resulted from contributions of both elastic and plastic deformation components. Consequently, this behavior could be modelled as a total sum of entirely elastic \((h_e)\) and plastic \((h_p)\) depths as \([31]\):

\[ h = h_e + h_p \tag{5} \]

The constitutive load equation for the entirely elastic component was defined as \([31, 33, 56]\):

\[ P_e = \frac{\pi E' h_e^2}{2 \cot \alpha} \tag{6} \]

where \(P_e\) represents the elastic load, \(\alpha = 70.3^\circ\) as defined above and \(E'\) represents the plane strain modulus defined by \([57]\):

\[ E' = \frac{E}{1 - \nu^2} \tag{7} \]

with \(\nu\) indicating the Poisson’s ratio of PIZC which was taken as 0.23 \([15]\). In a similar manner, the constitutive equation for the load of the entirely plastic component was defined as \([31, 33, 56]\):

\[ P_p = \pi \tan^2 \alpha H_f h_p^2 \tag{8} \]

where \(P_p\) and \(H_f\) represent the plastic load and plastic deformation resistance, respectively while \(h_p\) and \(\alpha\) are as defined above. At \(P_{\text{max}}\), both elastic and plastic loads are equal (i.e. \(P_e = P_p = P_{\text{max}}\)). Therefore, by assuming that \(\alpha_1 = \frac{\pi}{2 \cot \alpha}\) from equation \((6)\) and \(\alpha_1 = \pi \tan^2 \alpha\) from equation \((8)\), equation \((5)\) thus becomes \([31, 37]\):

\[ h = \frac{P_{\text{max}}}{(\alpha_1 E')^\frac{1}{2}} + \frac{P_{\text{max}}}{(\alpha_1 H_f)^\frac{1}{2}} \tag{9} \]

Hybrid Vickers hardness \((H_v)\) in micro-indentations has been expressed by equation \((4)\). Upon replacing \(h\) in equation \((4)\) with \(h_e\) in equation \((9)\), \(H_v\) becomes:

\[ H_v = \frac{1}{\alpha_1 E' + \alpha_2 H_f} \tag{10} \]

From equation \((10)\), \(H_f\) can then be found as \([38, 39, 41]\):

\[ H_f = \frac{H_v \alpha_2 E'}{(\alpha_1 E')^\frac{1}{2} - (\alpha_1 H_f)^\frac{1}{2}} \tag{11} \]

Therefore, the plastic deformation resistance of PIZC at different pre-sintering temperatures and polymer amounts was extracted using equation \((11)\).

At the maximum load, the corresponding elastic and plastic deformation depths \((h_e, h_p)\) were obtained from equations \((6)\) and \((8)\), respectively:

\[ h_e = \left( \frac{2P_{\text{max}} \cot \alpha}{\pi E'} \right)^\frac{1}{2} = \left( \frac{P_{\text{max}}}{\alpha_1 E'} \right)^\frac{1}{2} \tag{12} \]

\[ h_p = \left( \frac{P_{\text{max}}}{\pi \tan^2 \alpha H_f} \right)^\frac{1}{2} = \left( \frac{P_{\text{max}}}{\alpha_1 H_f} \right)^\frac{1}{2} \tag{13} \]

Elastic and plastic deformation components of PIZC at different polymer amounts and pre-sintering temperatures were obtained by normalizing equations \((12)\) and \((13)\) with equation \((5)\), achieving \(\frac{h_e}{(h_e + h_p)}\) % and \(\frac{h_p}{(h_e + h_p)}\) %, respectively.

2.2.2. Brittleness index

The brittleness index applied was the Quinn-Quinn model which related deformation energy per unit volume of materials to their fracture surface energy per unit area and expressed in terms as \([42]\):

\[ BI = \frac{H_f E'}{K_{IC}} \tag{14} \]

where all parameters are as defined previously. \(H_f\) was, however, used here instead of \(H_v\) because \(H_v\) is an elastic and plastic deformation property whereas \(H_f\) is a plastic property. Also, \(H_v\) could be load dependent, especially, for materials displaying normal indentation size effect behavior \([58]\) which was already reported for zirconia \([59, 60]\) while \(H_f\) is load-independent \([31]\).
3. Results and discussion

3.1. Analysis of mechanical properties

Figure 2 illustrates the plotted curves between the elastic modulus and polymer amount for polymer-infiltrated zirconia-ceramic composite (PIZC) at different pre-sintering temperatures from which the effects of both polymer amount and temperature can be reasonably observed. As the polymer amount is decreased at each pre-sintering temperature, the elastic modulus is increased similar to the dependent behaviour of porosity on elastic modulus. A decrease in porosity has led to an increment in elastic modulus of several porous ceramics \[32, 61, 62\]. Therefore, increasing the polymer amount from 0 to 42% leads to the corresponding decrease in elastic modulus by 83.2, 81 and 78.5% at 1100°C, 1200°C and 1300°C, respectively. In addition, with an increase in pre-sintering temperature, the elastic modulus of PIZC increases which can be attributed to the densification process taking place at higher pre-sintering temperatures \[5, 21, 38, 40, 45\].

Before determining the effect of polymer amount on the Vickers hardness of PIZC as reported in figure 3, the correlation between the micro-indentation hardness from equation (4) with contact depth \(h\) and measured hardness with average indentation diagonal length \(d\) should first be established. Using the Vickers indenter geometry in figure 1, the relationship between \(d\) and \(h\) has been established based on the principle of geometrical similarity as \[33\]:

\[
d = \frac{d_1 + d_2}{2} = 2h \tan \psi
\]

(15)

where \(\psi\) denotes the semi-apical angle of the Vickers indenter which is 74.05° \[33, 43\]. Upon making appropriate substitutions in equation (15), \(d\) becomes approximately \(7h\), which when substituted in equation (4) together with \(\alpha = 70.3°\), the equation (4) becomes:

\[
H_v = \frac{P_{\text{max}}}{(\pi \tan^2 \alpha) h^2} = 2 \frac{P_{\text{max}}}{d^2}
\]

(16)

Equation (16) is the established classical Vickers hardness equation using the projected rather than the real contact area with the former directly related to the stress intensity underneath the indentation contact \[43\]. By this analytical analogy, measured Vickers hardness data with projected area using \(d\) strongly correlate to those in micro-indentation instrumented tests with projected area using \(h\). Therefore, the two measurements can be applied interchangeably.

Figure 3 shows plotted curves of Vickers hardness measured with the projected area using the \(d\) versus polymer amount for PIZC at different pre-sintering temperatures. As observed, effects of both polymer amount and temperature on the Vickers hardness are similar to those observed for elastic modulus. Therefore, at each pre-sintering temperature, Vickers hardness increases with a decrease in polymer amount. Again, the hardness increase can be ascribed to an increasing ductility of polymer, lowering the deformation resistance at higher polymer amount. Thus, by increasing the polymer amount from 0 to 42%, the Vickers hardness decreases by
93.4, 92 and 85% at 1100 °C, 1200 °C and 1300 °C, respectively. Also, increasing the pre-sintering temperature correspondingly leads to the increase in the hardness PIZC. Again, this phenomenon is attributed to the densification process taking place at higher pre-sintering temperatures [5, 21, 38, 40, 45]. In addition, the Vickers hardness of PIZC–42 P, PIZC–26 P and PIZC–15 P materials ranges from 0.37–3.01 GPa at 1100 °C, implying its lower value in comparison with that reported for enamels (3.27–3.59 GPa) [63]. However, the Vickers hardness of PIZC–8 P material (4.84–5.95 GPa) is higher than the enamel. At 1200 °C, the Vickers hardness of PIZC–42 P and PIZC–26 P materials, ranging from 0.94–2.52 GPa, is still lower than that of enamel whereas the Vickers hardness values of PIZC–15 P and PIZC–8 P are higher than the enamel’s hardness. At 1300 °C, only the Vickers hardness of PIZC–42 P material (1.93–2.03 GPa) is lower than the Vickers hardness of enamel while those of PIZC–26 P, PIZC–15 P and PIZC–8 P materials are higher. This shows that higher polymer amount and lower pre-sintering temperature are the favorable conditions for PIZC to maintain dental property before the final sintering.

Figure 4 displays the plotted curves of fracture toughness versus polymer amount for PIZC materials at different pre-sintering temperatures. As the amount of polymer is decreased at each pre-sintering temperature, the fracture toughness is increased similar to the elastic modulus-polymer relationship above. Therefore, increasing the polymer amount from 0 to 42% leads correspondingly to the decrease in the fracture toughness by 80, 80.4 and 75% at 1100 °C, 1200 °C and 1300 °C, respectively. However, fracture toughness values at all polymer amounts at 1300 °C are higher than those at 1100 °C but lower than those at 1200 °C indicating the optimisation of fracture toughness at 1200 °C during the pre-sintering densification process of the material. It is worth noting that thermal induced phase transformation from monoclinic to tetragonal usually starts in zirconia-ceramic at 1170 °C [64]. So, pre-sintering PIZC at 1200 °C may have a heterogenous monoclinic, monoclinic-transformed tetragonal and polymeric structures all of which combine to slightly enhance the fracture toughness in comparison to a dual tetragonal and polymeric structures obtained when pre-sintered at 1300 °C. Further, the effect of polymer amount on the fracture toughness of PIZC can be appreciated specifically by comparing fracture toughness data of pre-cursor porous zirconia and PIZC at 1300 °C. These are 2.48, 2.26, 1.78 and 1.36 MPam$^{1/2}$ for PIZC–8 P, PIZC–15 P, PIZC–26 P and PIZC–42 P materials, respectively [7]. For the pre-cursor porous zirconia, they are 2.04, 1.78, 1.24 and 0.64 MPam$^{1/2}$ representing pre-cursor porous zirconia of 8, 15, 26 and 42% porosity, respectively [7]. It is obvious that PIZC has been toughened by 0.44, 0.48, 0.54 and 0.72 MPam$^{1/2}$ with the additions of 8, 15, 26 and 42% polymer amounts, respectively, to the pre-cursor porous zirconia. Similar toughening phenomenon occurs at other 1100 °C and 1200 °C pre-sintering temperatures. Therefore, ductile polymer reinforcements toughen zirconia-matrix composites by extrinsic mechanism, namely crack-bridging process acting behind an advancing crack [65]. When loaded, the higher stiff zirconia-ceramic constrains the plastic deformation of the ductile polymer network, facilitating ceramic-polymer debonding and bridging the crack in the process and higher polymer amount leads to higher toughness [5, 47, 66].

Since the porosities in pre-sintered zirconia-ceramics are infiltrated with polymeric material in PIZC, similar roles can envisagedly be played by both porosity and polymer amount on mechanical properties of PIZC.
Thus, it is normal to use the well-established porosity-mechanical property relations for this purpose. In this regard, different semi-empirical mechanistic models describing elastic modulus-porosity relationships reported in literature include the following [32, 61, 62, 67, 68]:

\[
E = E_o (1 - bP) \quad (17)
\]

\[
E = E_o (1 - bP + cP^2) \quad (18)
\]

\[
E = E_o \left( 1 + \frac{cP}{(1 - cP - P)} \right) \quad (19)
\]

\[
E = E_o \exp(-bP) \quad (20)
\]

\[
E = E_o \exp(-(bP + cP^2)) \quad (21)
\]

\[
E = E_o (1 - P)^m \quad (22)
\]

where \( P \), \( E \) and \( E_o \) represent porosity amount, predicted elastic modulus as functions of \( P \) and zero-porosity elastic modulus, respectively, while \( b \), \( c \), and \( m \) are non-negative empirical parameters from curve-fitting linear or non-linear regression equations. Note that when \( P = 0 \), \( E = E_o \) for all equations, which satisfies the first boundary condition in the absence of porosity. However, when \( P = 1 \), \( E = 0 \) for only equations (19) and (22) which satisfy the second boundary condition while \( E \approx 0 \) for equations (17), (18), (20) and (21) satisfying not the secondary condition. The implications of not satisfying the second condition \( (E \approx 0 \text{ at } P = 1) \) are that, a fully porous material is assumed to have an elastic modulus which is untrue and this predicted modulus will be superimposed on polymer when it is applied to fill the fully porous structure, complicating the prediction modeling process and yielding inaccurate results. Furthermore, equation (19) is limited inherently to low range of porosity \( (0 \leq P \leq 0.2) \) while equation (22) can be applied to many materials having a wide porosity range such as applied in this study \( (0 \leq P \leq 0.42) \) [32, 61, 62, 68]. In addition, equation (22), also known as connected-grain model, features important attributes like preserving randomness of grains’ distributions, relating porosity/grains and their distributions to ceramic properties. Besides, the power index \( (m) \) can be related to grain/pore size distribution [69]. Moreover, equation (22) has been adapted to porosity-dependent Vickers hardness as [32]:

\[
H_v = H_{vo} (1 - P)^m \quad (23)
\]

and fracture toughness of ceramics [69]:

\[
K_{fc} = K_{fco} (1 - P)^{m+0.5} \quad (24)
\]

where \( H_{vo} \) and \( K_{fco} \) represent zero-porosity Vickers hardness and fracture toughness, respectively, while other parameters are as previously defined.

Adapting \( P \) to mean polymer amount in equations (22)–(24), the connected-grain models describing the dependence of polymer amount on the elastic modulus, the Vickers hardness and the fracture toughness of PIZC, respectively, at different pre-sintering temperatures, are reported in figures 2–4. Table 2 lists the non-linear equations as well as empirically derived parametric data from these figures. Evidently, it is inferred in
that at zero polymer amount ($P = 0$), the values of these mechanical properties correspond to $E_{0} = 86.42$, 153 and 183 GPa at 1100, 1200 °C and 1300 °C, respectively, $H_{0} = 5.9$, 12 and 13 GPa at 1100 °C, 1200 °C and 1300 °C, respectively, and $K_{IC0} = 3.76$, 7.48 and 5.4 MPa m$^{0.5}$, at 1100 °C, 1200 °C and 1300 °C, respectively. These elastic moduli agree those reported for dense zirconia materials [7]. Meanwhile, 100% polymer amount predicts from equations (22)–(24) that $E = 0$, $H = 0$ and $K_{IC} = 0$ for dense zirconia materials at all pre-sintering temperatures, explicitly and intactly uncompromising the polymer properties. Furthermore, the $m$ index, which physically represents the grain/pore size distribution, decreases with increasing pre-sintering temperatures for $E$ and $H_{V}$ models in table 2. This may be used to explain the densification process occurring at higher temperatures. When porous ceramic composites become densified, there is always a reduction in grain/pore size distribution due to pore closure resulting from the ceramic grain growth [20, 21, 67]. In the same vein, the power indices of the fracture toughness equations of PIZC in table 2 obey equation (24), differing by 0.5 when compared with the elastic moduli indices at all temperatures in agreement with literature stipulations [69]. Therefore, the mechanistic connected-grain models (equations (22)–(24)) in table 2, perfectly predict mechanical properties-polymer amount relationships for PIZC at all considered pre-sintering temperatures. It is interesting to note that, to our best knowledge, this is the first research reporting such mechanical properties-polymer amount relationships for PIZC.

### 3.2. Analysis of advanced mechanical properties

The plastic deformation resistance ($H_{P}$) data obtained from equation (11) at different polymer amounts and pre-sintering temperatures for PIZC are displayed graphically in figure 5. As evidently shown, there is no specific correlation between $H_{P}$ and polymer amount for ZC–0 P, PIZC–8 P and PIZC–15 P materials at all pre-sintering temperatures. Specifically, for the PIZC–8 P material, the $H_{P}$ progressively reduces in the order of 1200 °C, 1300 °C and 1100 °C. Similar observations are made for the reduction of $H_{P}$ with respect to temperatures for ZC–0 P and PIZC–15 P materials. Additionally, the highest $H_{P}$, which corresponds to 297 GPa at 1200 °C for the

### Table 2. Curve fitting non-linear equations obtained from the connected-grain models for mechanical properties at different pre-sintering temperatures based on figures 3–5.

| Mechanical property | Pre-sintering temperature (°C) |
|---------------------|--------------------------------|
|                     | 1100  | 1200  | 1300  |
| Elastic modulus     | $E = 86.42(1 - P)^{0.8}$ | $E = 153(1 - P)^{3.6}$ | $E = 183(1 - P)^{4.8}$ |
| $R^{2}$             | 92%   | 91%   | 91%   |
| Vickers hardness    | $H = 5.9(1 - P)^{4.8}$    | $H = 12(1 - P)^{4.2}$  | $H = 13(1 - P)^{3.4}$  |
| $R^{2}$             | 95%   | 95%   | 98%   |
| Fracture toughness  | $K_{IC} = 3.76(1 - P)^{0.8}$ | $K_{IC} = 7.48(1 - P)^{3.4}$ | $K_{IC} = 5.4(1 - P)^{5.3}$ |
| $R^{2}$             | 85%   | 87%   | 84%   |

* $R^{2}$ represents correlation coefficient.
PIZC–8 P material, is lower than that of pre-sintered lithium disilicate glass ceramic (327 GPa) [41] and fused silica (350 GPa) [37] indicating that this material is considered less brittle than the two materials. Additionally, the $H_f$ value previously reported for zirconia-ceramic ranged from 33.63–52.81 GPa [38] while the $H_f$ values of the ZC–0 P material correspond to 36.74, 97.45 and 85.99 GPa, at 1100 °C, 1200 °C and 1300 °C, respectively. This reveals that only the $H_f$ of the material pre-sintered at 1100 °C lies within the reported literature value.

The effect of polymer amount on $H_f$ for PIZC can be inferred for PIZC–26 P and PIZC–42 P materials, where the $H_f$ scales down with increasing polymer amount and scales up with the increment in pre-sintering temperature for both materials (figure 5). Therefore, for both materials, their $H_f$ values range from 1–24.6 GPa. These values lie within the $H_f$ values extracted for of dentine and enamel (3.3–28.3 GPa) by Oyen [37], buttressing the strong compatibility between dentine and enamel and the two materials. In addition, it can be observed in figure 5 that the $H_f$ values of PIZC–42 P and PIZC–26 P materials are lower than those of ZC–0 P material at all pre-sintering temperatures, indicating their higher plastic deformation resistances. This is an important plastic property which can greatly facilitate the realization of precision ductile machining for the two materials during the CAD/CAM process [38, 39, 41].

Partitioning materials’ deformations into elastic and plastic deformations reveals the deformation mechanism that contributes dominantly to total deformations [37–39, 41]. Figure 6 shows elastic and plastic deformation components of PIZC normalized to total deformations versus polymer amount at different pre-sintering temperatures. Several important references can be made. As shown in figure 6, the elastic deformation overwhelms plastic deformation components at all pre-sintering temperatures for PIZC–26 P and PIZC–26 P materials, implying the dominant elastic deformation mechanism. Similarly, elastic deformation is the dominant mechanism for the ZC–0 P material at all pre-sintering temperatures which is consistent with the dominant deformation mechanism reported for zirconia [38]. Therefore, the dominance of elastic over plastic deformation in materials is associated with their proneness to brittle fracture during mechanical processing [38, 40].

In contrast, plastic deformation components are higher than the elastic deformation components for PIZC–42 P and PIZC–26 P materials at 1100 °C, suggesting the dominant plastic deformation mechanism at this temperature. Again, the dominance of plastic deformation is an indication that PIZC–42 P and PIZC–26 P materials can be precision machined in ductile mode material removal process at this pre-sintering temperature. However, as the pre-sintering temperature is raised above 1100 °C, the degree of plastic deformation dominance for these materials decreases, transitioning to elastic deformation dominance at 1200 °C especially for the PIZC–26 P material. For the PIZC–42 P material, the plastic dominance at higher pre-sintering temperatures is still maintained but it reduces to 50% at 1300 °C. This shows that pre-sintering temperature has highly impacted the densification process, increasing the elasticity of PIZC at higher temperatures [38, 40, 70]. In summary, the best plastic deformation properties are obtained for PIZC–42 P and PIZC–26 P materials at 1100 °C which are desired during mechanical fabrication processes. It is interesting to note that this temperature has been reported in the literature as the optimal temperature to achieve excellent quasi-plasticity for zirconia [22, 40] and PIZC [30]. However, this study has further established the quasi-plastic dependency of PIZC on polymer amount (26%–42%) at this temperature. Above this temperature and polymer amount range (26%–42%), the plastic

![Figure 6. Elastic and plastic deformation components of PIZC as a function of polymer amount at different pre-sintering temperatures.](image-url)
deformation properties of PIZC are not guaranteed. It is therefore recommended to perform dental CAD/CAM machining processes at 1100 °C on PIZC–42 P and PIZC–26 P materials to utilize by their better plastic deformation properties than those inherently offered by PIZC–15 P and PIZC–8 P materials.

The above assertion can further be buttressed by analyzing the brittleness index of PIZC at different polymer amounts and pre-sintering temperatures, which can as well be usefully applied to characterize its machinability [44]. In this sense, the brittleness index postulated for ceramics as expressed by equation (14) is adopted in this research [42]. Figure 7 shows the charts of brittleness index (BI) against polymer amount for PIZC at different pre-sintering temperatures. This figure follows similar trends to plastic deformation resistance versus polymer amount in figure 5. In particular, for the PIZC–8 P material, the BI progressively reduces in the order of 1300 °C 1100 °C and 1200 °C pre-sintering temperatures. For the ZC–0 P and PIZC–15 P materials, the BI increases with increasing pre-sintering temperatures (1100 °C, 1200 °C and 1300 °C). Similar increment of BI with pre-sintering temperature is also complied for PIZC–26 P and PIZC–42 P materials. In addition, the highest BI corresponds to 3037 μm−1 at 1300 °C for PIZC–8 P material while the lowest BI corresponds to 22.34 μm−1 at 1100 °C for PIZC–42 P material. Therefore, the BI ranks lowest for PIZC–42 P followed by PIZC–26 P, ZC–0 P, PIZC–15 P and PIZC–8 P. High and low BI values imply high susceptibility of the material to fracture and deformation, respectively [42]. Consequently, based on this study, the PIZC–42 P and PIZC–26 P materials are likely to be more apt to deformation than ZC–0 P material which, in turn, is more deformable than PIZC–15 P and PIZC–8 P materials at 1100 °C pre-sintering temperature.

Using the ZC–0 P material as a reference, Alao et al [71] have reported brittle fracture material removal mechanisms for the material during the CAD/CAM milling process. Since the BI value of PIZC–15 P and PIZC–8 P materials are far greater than that of ZC–0 P material, it is expected that PIZC–15 P and PIZC–8 P materials will display lower machinability than ZC–0 P material during the CAD/CAM process. Conversely, it is anticipated that PIZC–42 P and PIZC–26 P materials will display better machinability than ZC–0 P material during the CAD/CAM process due to their lower BI values. Finally, it can be inferred, from figure 7, that dental restorations can be fabricated from PIZC with polymer amount not lower than 26% at 1100 °C pre-sintering temperature. Therefore, future research aims at conducting machining science studies of all studied PIZC materials to reveal fundamental responses and material removal mechanisms during CAD/CAM machining process for the production of high-quality restorations.

4. Conclusions

This research investigated the effect of polymer amount on the mechanical behavior of polymer-infiltrated zirconia-ceramic composite (PIZC) at different pre-sintering temperatures. Pre-sintered zirconia-ceramics were fabricated and designated as PIZC–8 P, PIZC–15 P, PIZC–26 P and PIZC–42 P while fully dense zirconia was also fabricated and designated as ZC–0 P. Elastic modulus, Vickers hardness and fracture toughness of materials were characterized for all materials. Analysis of advanced mechanical properties was performed
through plastic deformation resistance, dominant mechanical deformation behavior and brittle index for all materials. In the light of the results reported in this research, the followings were concluded:

- Elastic modulus, Vickers hardness and fracture toughness at each pre-sintering temperature scaled down with the increase in polymer amount, conforming to the existing scaling relations between ceramic properties and porosity.
- Elastic modulus and Vickers hardness were enhanced with increasing pre-sintering temperature due mainly to the densification process occurring at higher pre-sintering temperatures.
- Optimal fracture toughness was reached for PIZC at 1200 °C pre-sintering temperature.
- Increment in polymer amount improved the fracture toughness of PIZC at all pre-sintering temperatures.
- All properties were perfectly modeled according to the connected-grain models, predicting mechanical properties-polymer amount relationships for PIZC at all pre-sintering temperatures.
- The higher plastic deformation resistances, the higher plastic deformation components and the lower brittleness indices exhibited by PIZC–42 P and PIZC–26 P materials at 1100 °C implied their superior plastic deformation properties in comparison to other PIZC materials.
- The findings recommended the processing of PIZC in CAD/CAM process at polymer amount not less than 26% and at 1100 °C pre-sintering temperature when used to fabricate dental dentures.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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