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# Characterization of a polymer-infiltrated ceramic-network material

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## ABSTRACT

**Objectives.** To characterize the microstructure and determine some mechanical properties of a polymer-infiltrated ceramic-network (PICN) material (Vita Enamic, Vita Zahnfabrik) available for CAD–CAM systems.

**Methods.** Specimens were fabricated to perform quantitative and qualitative analyses of the material's microstructure and to determine the fracture toughness ( $K_{Ic}$ ), density ( $\rho$ ), Poisson's ratio ( $\nu$ ) and Young's modulus ( $E$ ).  $K_{Ic}$  was determined using V-notched specimens and the short beam toughness method, where bar-shaped specimens were notched and 3-point loaded to fracture.  $\rho$  was calculated using Archimedes principle, and  $\nu$  and  $E$  were measured using an ultrasonic thickness gauge with a combination of a pulse generator and an oscilloscope.

**Results.** Microstructural analyses showed a ceramic- and a polymer-based interpenetrating network. Mean and standard deviation values for the properties evaluated were:  $K_{Ic} = 1.09 \pm 0.05 \text{ MPa m}^{1/2}$ ,  $\rho = 2.09 \pm 0.01 \text{ g/cm}^3$ ,  $\nu = 0.23 \pm 0.002$  and  $E = 37.95 \pm 0.34 \text{ GPa}$ .

**Significance.** The PICN material showed mechanical properties between porcelains and resin-based composites, reflecting its microstructural components.

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## 1. Introduction

Microstructural characterization and determination of the material properties are the first steps to understand the behavior of the materials used in restorative dentistry. Scanning electron microscopy (SEM) is a useful tool to provide information on topography [1–4] and microstructural parameters

(stereology) such as particle size and shape [1,3,5]. When SEM is associated with energy dispersive spectroscopy (EDS), the information is enhanced by semi-quantitative chemical data of the material's phases. In addition, the density ( $\rho$ ), the Young's modulus ( $E$ ) and the Poisson's ratio ( $\nu$ ) play an important role in the material's behavior [4], thereby are essential for finite element analysis [6,7], which has increased in popularity in dental research. Nevertheless, the fracture toughness

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( $K_{Ic}$ ) has been reported as one of the key properties associated with the clinical performance of dental materials [8].  $K_{Ic}$  indicates the ability of a material to resist crack propagation and, consequently, catastrophic failure. It has special relevance to fracture of brittle materials [9,10]. For ceramic materials, the recommended method to determine  $K_{Ic}$  is the pre-crack-induced-test named single edge V-notched beam (SEVNB), which was found to be user friendly, repeatable, reliable and accurate, except for ceramics with pronounced R-curve behavior [11]. This method is based on notched bar-shaped specimens that are subsequently tested in flexure. The  $K_{Ic}$  value is calculated considering the failure load and the dimensions of the specimen and the notch. The notch design, specially the size of the notch root radius, affects the  $K_{Ic}$  value, so it is important to sharpen the notch until the root radius becomes approximately of the same size than the major microstructural feature size [12]. Yet, bar-shaped specimens fabricated from small size blocks, usually used for milling crowns in the CAD–CAM systems, present an additional challenge to determine the  $K_{Ic}$  value using V-notched specimens.

Ceramics and resin-based composites are the two main classes of dental restorative materials. Resin-based composites are composed of an organic polymer matrix and reinforcing inorganic filler particles [1,13]. The amounts of filler particles are directly related to the Young's modulus and the hardness of the composites [1]. Development of filler technology has resulted in considerable improvements of the composites properties [1]. An important consideration to select the filler particles is the optical characteristic, and silica-based particles meet well this requirement [13]. On the other hand, the dimensional changes resulting from the polymerization are determined by the monomers of the polymer matrix, and the most common monomers are Bis-GMA, UDMA, UTMA, and Bis-EMA [13]. Nevertheless, the clinical performance of direct composites is still inferior to the performance of indirect ceramic restorations considering marginal adaptation, color match, and anatomic form [14]. A 3-year clinical study showed that indirect resin-based composite restorations have inferior esthetic and wear resistance compared to all-ceramic restorations [15]. Dental ceramics are essentially inorganic materials commonly composed of a crystalline phase and/or glass matrix [8]. Stronger and tougher ceramics, e.g. zirconia-based ceramics and alumina-based ceramics, have higher crystalline content and are more opaque than esthetic porcelains, e.g. silica-based ceramic [16]. However, the low  $K_{Ic}$  and high susceptibility to slow crack growth of the porcelains limit their clinical application [17].

Associating the Young's modulus of resin-based composites, which is similar to the dentin Young's modulus, with the long lasting esthetics of ceramics would be ideal for a restorative material. The newly developed polymer-infiltrated-ceramic-network (PICN) may offer an alternative solution. The fabrication process of this material requires two steps: first, a porous pre-sintered ceramic network is produced and conditioned by a coupling agent; second, this network is infiltrated with a polymer by capillary action [18]. The flexural strength, elastic modulus, hardness and strain at failure of PICN structures were reported in a

previous study [18], showing similar properties to the tooth structure and encouraging further studies on this material. Thus, the aim of the present study is to characterize the microstructure and determine some mechanical properties of a PICN material available for CAD–CAM systems, testing the hypothesis that the new material has properties ranging between porcelains and resin-based composites. In addition, this study applies the short beam toughness method to determine  $K_{Ic}$ .

## 2. Materials and methods

A PICN material (Vita Enamic, Vita Zahnfabrik, Bad Sackingen, Germany) was used to fabricate all specimens.

### 2.1. Microstructural characterization

Specimens were fabricated ( $n=5$ ) to perform quantitative and qualitative analyses of the microstructure. CAD–CAM blocks (17.5 mm × 14 mm × 12 mm) of the material were sectioned with a precision cutting machine (Isomet 1000, Buehler, Lake Bluff, USA), polished with metallographic papers (600, 800 and 1200-grit SiC) to the final dimension (2 mm × 14 mm × 12 mm) and finished with 1 μm alumina abrasive (Mark V Laboratory, East Granby, CT, USA). The specimens were sonically cleaned in acetone bath for 5 min, and then in isopropyl alcohol bath for additional 5 min before gold coated (SC7620 Sputter Coater, Quorum Technologies, Laughton, United Kingdom) and examined under the SEM (Jeol JSM-5310, Jeol, Japan) for the qualitative (SEI and BSI images) and quantitative (electron dispersive spectroscopy – EDS) analyses. Images in three different magnifications (1500×, 5000× and 20,000×) were recorded. Material composition, oxides and element concentrations (above 1 wt%) were recorded from three different locations in each specimen using EDS. Average values were calculated.

### 2.2. Material properties

Four properties of the material were examined: fracture toughness ( $K_{Ic}$ ), density ( $\rho$ ), Poisson's ratio ( $\nu$ ) and Young's modulus ( $E$ ).

$K_{Ic}$  was evaluated using the V-notched-beam test according to the ASTM C1421-10 standard [19]. Bar-shaped specimens (17.5 mm × 4 mm × 3 mm) were fabricated ( $n=7$ ) from CAD–CAM blocks using a precision cutting machine (Isomet 1000). The specimens were polished and positioned side-by-side on a flat holder, with the 3-mm wide face up, to be notched. The V-notch was created using a razor blade adapted in a notching machine (Equitecs, São Carlos, SP, Brazil). The machine applied a constant load of 10 kg on the razor blade, with a constant back-and-forth movement. A 6-μm diamond paste was used as an initial lubricant followed by a 1-μm diamond paste (Mipox Abrasives India, Bangalore, India). The final depth of the notch was approximately 1.1 mm. The specimens were removed from the holder and cleaned using alcohol in a sonic bath for 5 min. The notch root radius of each specimen was measured using SEM at 1000× magnification.

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