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Microstructure characterization and SCG of newly engineered dental ceramics

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ABSTRACT

Objectives. The aim of this study was to characterize the microstructure of four dental CAD-CAM ceramics and evaluate their susceptibility to stress corrosion.

Methods. SEM and EDS were performed for microstructural characterization. For evaluation of the pattern of crystallization of the ceramics and the molecular composition, XRD and FTIR, respectively, were used. Elastic modulus, Poisson's ratio, density and fracture toughness were also measured. The specimens were subjected to biaxial flexure under five stress rates (0.006, 0.06, 0.6, 6 and 60 MPa/s) to determine the subcritical crack growth parameters (n and D). Twenty-five specimens were further tested in mineral oil for determination of Weibull parameters. Two hundred forty ceramic discs (12 mm diameter and 1.2 mm thick) were made from four ceramics: feldspathic ceramic – FEL (Vita Mark II, Vita Zahnfabrik), ceramic-infiltrated polymer – PIC (Vita Enamic, Vita Zahnfabrik), lithium disilicate – LD (IPS e.max CAD, Ivoclar Vivadent) and zirconia-reinforced lithium silicate – LS (Vita Suprinity, Vita Zahnfabrik).

Results. PIC discs presented organic and inorganic phases ($n = 29.1 \pm 7.7$) and Weibull modulus (m) of 8.96. The FEL discs showed $n = 36.6 \pm 6.8$ and $m = 8.02$. The LD discs showed a structure with needle-like disilicate grains in a glassy matrix and had the lowest value of n (8.4 ± 0.8) and $m = 6.19$. The ZLS discs showed similar rod-like grains, $n = 11.2 \pm 1.4$ and $m = 9.98$.

Significance. The FEL and PIC discs showed the lowest susceptibility to slow crack growth (SCG), whereas the LD and ZLS discs presented the highest. PIC presented the lowest elastic

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modulus and no crystals in its composition, while ZLS presented tetragonal zirconia. The overall strength and SCG of the new materials did not benefit from the additional phase or microconstituents present in them.

1. Introduction

New indirect restorative materials have been recently released in the dental market based on different microstructural approaches in comparison with those of previously available indirect materials. One example of these innovative materials is a polymer-infiltrated ceramic (PIC) CAD-CAM block that is claimed to have higher structural reliability as a result of the so-called crack-stop function mechanism, which occurs when a crack that is propagating through the polymer network halts due to the presence of the ceramic phase. Recent publications showed that this type of material is composed of a polymer-infiltrated ceramic network containing urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) cross-linked polymers [1]. In comparison with dental porcelains, this new material has been proven to have lower elastic modulus and higher damage tolerance [2].

Another recently released indirect dental material is a lithium-silicate-based glass-ceramic reinforced with zirconia particles (ZLS). According to the manufacturer, the zirconia particles added to this glass-ceramic are small and homogeneously distributed throughout the microstructure, improving its strength and providing good surface finish. This material is provided only for CAD-CAM technology, and, to the authors' knowledge, no laboratory or clinical data have yet been published regarding this product. The microstructure of this new glass-ceramic material is similar to that seen in lithium disilicate ceramics, which, for many years, were protected by a patent held by Ivoclar Vivadent and became well-known for their excellent optical properties [3]. These lithium disilicate ceramics are now commercially available as pressable ingots or CAD-CAM blocks, the latter having an intermediate stage of crystallization that, after milling, still requires heat treatment for crystal growth.

Ceramic restorations are constantly subjected to humidity and occlusal loads, and their time in service is controlled by slow crack growth (SCG), which is the environmentally assisted subcritical growth of cracks. SCG can be estimated by a power law relation in which a coefficient, n, expresses the material's susceptibility to stable crack growth. The higher the value of n, the lower the susceptibility to SCG. The method commonly used for measuring the subcritical crack growth parameter is the constant-stress-rate test, in which the material strength is obtained as a function of various stress rates in a certain environment [6].

The development of newly engineered indirect materials has led to a need for new studies to characterize their mechanical and fatigue properties, in attempts to predict their clinical behavior. Therefore, the primary objective of the present study was to characterize the microstructure and the slow crack growth (fatigue) parameters using constant-stress-rate testing of the following indirect restorative materials: polymer-infiltrated (PIC), zirconia-reinforced lithium silicate (ZLS), lithium disilicate (LD) and feldspathic (FEL) ceramics, all designed for CAD-CAM processing. Also, fracture toughness, elastic modulus and Weibull parameters were determined.

Table 1 – Cycle of crystallization of LD and ZLS ceramics.

	ZLS (Vita Suprinity)	LD (IPS e.max CAD)
Beginning chamber temperature (°C)	400	403
Time at the initial temperature (min)	8:00	6:00
Temperature rate increase (°C/min)	55	90
Crystallization temperature (°C)	840	820
Holding time (min)	8:00	7:00
Ending temperature (°C)	680	700

2. Materials and methods

Sixty discs of each of the following materials were prepared: feldspathic ceramic (Vita Mark II, Vita Zahnfabrik, Bad Säckingen, Germany), polymer-infiltrated ceramic (Vita Enamic, Vita Zahnfabrik), lithium disilicate ceramic (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) and zirconia-reinforced lithium silicate ceramic (Vita Suprinity, Vita Zahnfabrik).

2.1. *Specimen preparation*

A ring device was glued onto the top surfaces of the CAD-CAM blocks to round them until cylinders 12 mm in diameter were obtained. The cylinders were then cut into several discs approximately 12 mm in diameter and 1.35 mm thick, in a lathe (ISOMET 1000, Buehler, Lake Bluff, IL, USA). IPS e.max CAD and Vita Suprinity require a cycle of crystallization that was performed in their respective furnaces (Programat EP5000, Ivoclar Vivadent; and Vita Vacumat 6000MP, Vita Zahnfabrik) according to the temperature recommendations given by the manufacturers (Table 1). The specimens were then polished with SiC#400, 800 and 1200. According to ISO standard CD 6872, specimens attained final dimensions of 12 mm in diameter and 1.2 mm thick [4].