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Flexural Properties of Chairside CAD/CAM Materials

Własności sprężyste materiałów CAD/CAM w warunkach gabinetu stomatologicznego

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A – research concept and design; B – collection and/or assembly of data; C – data analysis and interpretation; D – writing the article; E – critical revision of the article; F – final approval of article

Abstract

Background. New blocks for milling crowns using CAD-CAM technology were introduced to the profession. It is important to determine mechanical properties of such materials since they are used for the fabrication of crowns used in stress-bearing areas.

Objectives. This study determined the flexural strength (FS) and the flexural modulus (FM) of 2 glass-ceramic and 2 nanoceramic resin composite CAD/CAM blocks used for chair-side crown fabrication

Material and Methods. Rectangular specimens were cut from 4 different CAD-CAM blocks. Specimens were 3 mm wide, 1.2 mm thick 14 mm long. Specimens were subjected to 3-point bending test following ISO guidelines (ISO 6872) at cross-head speed of 0.5mm/min. The flexural strength (FS) and the flexural modulus (FM) were calculated and the data statistically-analyzed with one-way ANOVA and Games Howell multiple comparison tests at 95% confidence interval.

Results. Means and SDs of FS (MPa) for VE, LU, E-max, E-max-U, CD, CD-U were: 123.97(14.84), 168.07(16.70), 334.10(54.3), 128.90(17.6), 177.32(37.54) and 147.61(26.62) respectively. For FM means and SDs were: 17.18(2.03), 9.75(0.51), 44.8(5.52), 35.14(7.46), 32.96(6.55) and 38.90(8.03) for VE, LU, E-max, E-max-U, CD, CD-U, respectively. ANOVA revealed a highly significant difference among group means ($p < 0.0001$) for both FS and FM. E-max had significantly highest mean FS and FM values among all groups, while VE showed lowest FS and LU lowest FM means. Firing and or crystallization positively affected both flexural properties of E-max, but only FS of CD.

Conclusions. A wide variability in mean FS and FM was observed among the tested materials. Generally, glass-ceramic based materials had superior flexural properties (**Dent. Med. Probl. 2016, 53, 2, 230–235**).

Key words: flexural modulus, flexural strength, flexural properties of milling blocks.

Słowa kluczowe: moduł sprężystości, wytrzymałość na zginanie, właściwości sprężyste wyfrezowanych klocków.

All-ceramic dental materials are a remarkable alternative to other restorative materials due to their unique biocompatibility and superior esthetic appeal [1, 2]. They have excellent esthetics, high strength and acceptable fit, especially with the introduction of new processing methods [3], such as the Computer-Aided Design/Manufacturing (CAD/CAM) systems. The availability of manufactured blocks has enabled the fabrication of indirect esthetic restorations in a single visit [4,5]. According to the mode of laboratory processing, highly accurate ceramic restorations are ei-

ther pressed, milled, slip-cast or sintered; and according to their chemical composition, they can be further classified into high- and low-leucite, lithium disilicate glass-ceramic and magnesia and alumina core reinforced types [6]. Due to the wider range of available CAD/CAM blocks having diverse properties, it can be sometimes challenging to make the proper selection for specific clinical situations [2].

Dental ceramics, known to be brittle in nature with low tensile strengths [7], are extremely sensitive to inherently growing surface defects and

flaws resulting from exposure to complex stresses during function [1, 8–10]. These defects may result in subsequent crack propagation and final fracture of the restoration. The density, location and severity of flaws affect the durability of the restorations [8]. The use of ceramic restorations have been limited to low stress-bearing areas in an attempt to prevent propagation of defects under stress which can cause catastrophic failure [1].

At least 2 types of flaws develop in ceramics, those known as fabrication defects as well as surface cracks [11]. Conventional laboratory fabrication procedures and clinical restoration adjustments initiate the development of subcritical flaws and different defects within the cut materials. Similarly, different finishing procedures of ceramic restorations may cause surface roughness to occur, thus concentrating stresses in the restoration and consequently reducing their fracture resistance [12]. Several test methods have been employed for brittle materials. Among them flexural testing proved to be a practical and cost-effective method to examine mechanical strength of brittle materials either using the transverse (3- and 4-point flexure strength) tests [13–16] loading rectangular beam-shaped specimens to fracture, or the biaxial flexure test [17].

The biaxial flexure test with its disk-shaped specimens was proven to be insensitive to specimen geometry and independent of flaw direction, thus avoiding the effects of edge flaws commonly found with rectangular bars of the 3- and 4-point flexure tests [18, 19]. However, the 3-point flexure test has been the standard test used for testing strength of ceramics, cements and polymers due to its ease of specimen preparation [20], this is in spite of its increased sensitivity and dependence on flaws and defects alongside the specimen edges [15, 19]. In a 3-point bending test, and in contradiction to the 4-point flexure test, only the lower surface of the specimen located between the 2 supporting rollers under tension [21] is subjected to the maximum tensile stress [15], showing even higher failure stress values compared to values obtained for the 4-point test [15, 22]. Stress finally results in fracture of the specimen due to crack initiation from the central undersurface of the beams [23]. However, each of these tests has its limitations and none of them really reproduce the complex intraoral stresses that dental

restorations are subjected to while in use [24]. Therefore, properties of ceramics should rather not be described only by their mean fracture strength [25].

This study aimed to determine flexural strength (FS) and flexural modulus (FM) of 2 groups of CAD/CAM materials: glass-ceramic and nanoceramic resin composite.

Material and Methods

Sixty bar-shaped specimens ($n = 10$ /material) were cut from 4 different CAD/CAM blocks (15mm length) using a water-cooled low-speed diamond saw (Buehler Ltd., Lake Bluff, IL, USA). The materials comprised 2 nanoceramic resin composites (NCRC): Lava-Ultimate (LU) (3M/ESPE) and Vita Enamic (VE) (Vita); a partially-crystallized lithium disilicate glass-ceramic: IPS e.max CAD (Ivoclar Vivadent), which was tested before and after crystallization firing under vacuum (E-max-U, E-max, respectively); and a zirconia-reinforced fully crystallized lithium silicate glass-ceramic, Celtra Duo (Dentsply), which was also tested before and after firing under vacuum (CD-U, CD, respectively) in a furnace at 860°C (Table 1).

Specimens were $3 \pm .5$ mm wide, $1.2 \pm .05$ mm thick and 14 mm long. Specimen dimensions were verified with a digital micrometer (Mitutoyo digimatic caliper 500-136, Kawasaki, Japan) with an accuracy of $\pm .01$ mm at 3 separate locations. Cut specimens were placed on a 3-point bending test device (ISO 6872) with a span of 12 mm distance between supports (2 rods of 2mm diameter) [4]. (Fig. 1) Compressive load was applied at the center of the specimens at a cross-head speed of .5 mm/min [14] in an Instron universal testing machine (Model 4301, Instron Corporation, Canton, MA, USA).

Fracture load was recorded and flexural strength (FS) as well as flexural modulus (FM) were respectively calculated from equations (1) and (2):

$$FS = 3Fl/2bh^2 \quad (1)$$

and

$$FM = Fl^3/4 dlin bh^3 \quad (2)$$

Where: F = ultimate force [N], l = distance between supports (span length) on the tension surface

Table 1. Materials used

Material	Type of Material	Lot/Batch No.	Manufacturer
Celtra Duo® (CD)	Zirconia-reinforced lithium silicate	18017606	Dentsply
IPS e.max CAD® (E-max)	Lithium disilicate glass ceramic	N44759	Ivoclar Vivadent
Lava Ultimate® (LU)	Nanoceramic resin composite	N316515	3M/ESPE
Vita Enamic® (VE)	Nanoceramic resin composite	43441	Vita

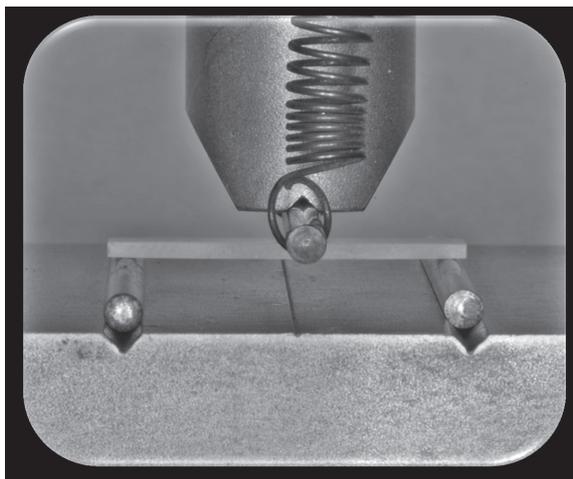


Fig. 1. Compressive load was applied in Instron

[mm], b = mean specimen width [mm], h = mean specimen height (thickness) [mm], F_{lin} = force in the linear part of the stress/strain curve [N] and d_{lin} = corresponding deflection at F_{lin} [mm]

Flexural modulus (FM) was determined from the load deformation outlines created during the 3-point flexural strength (FS) testing with previously mentioned equations.

Means and SDs were calculated and data statistically analyzed with one-way ANOVA at 95% confidence interval and Games Howell multiple comparison tests (IBM SPSS Statistics 20).

Results

Means and standard deviations of FS (MPa) and FM (GPa) for tested materials are presented in boxplot charts (Figs. 2 and 3). ANOVA revealed a highly significant difference between the means ($p < .0001$) for FS and FM. Further analysis with Games-Howell test revealed E-max's mean FS value (334.1 ± 54.3 MPa) to be significantly highest ($p < .001$). However, FM mean value (44.8 ± 5.54 GPa) was not significantly higher than that of CD ($p > .05$). Both nanoceramic resin composites, Vita Enamic (VE) and Lava Ultimate (LU), had significantly lower FM means of 17.18 ± 2.03 GPa and 9.75 ± 0.51 GPa, respectively.

Discussion

Flexural strengths and flexural moduli of 4 tooth-colored restorative CAD/CAM materials, glass ceramics and nanoceramic resin composites, were tested. Specimens were cut from different CAD/CAM blocks, which were manufactured by industrially standardized processes. These blocks

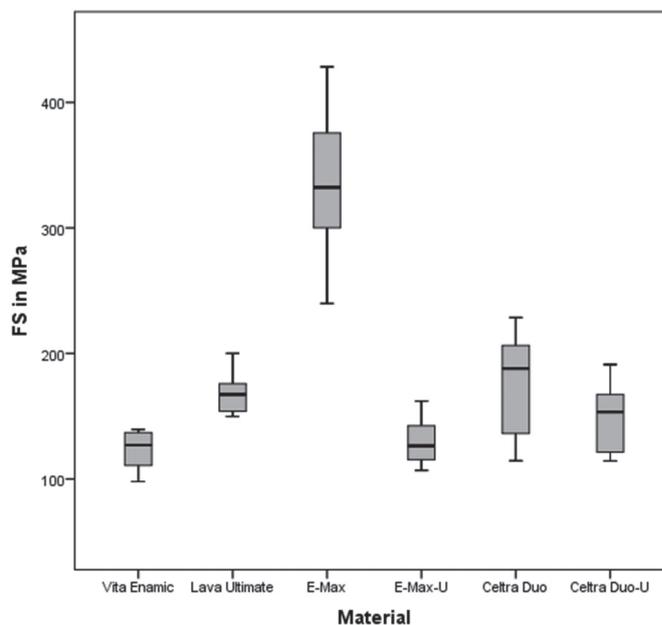


Fig. 2. Means and standard deviations of FS (MPa)

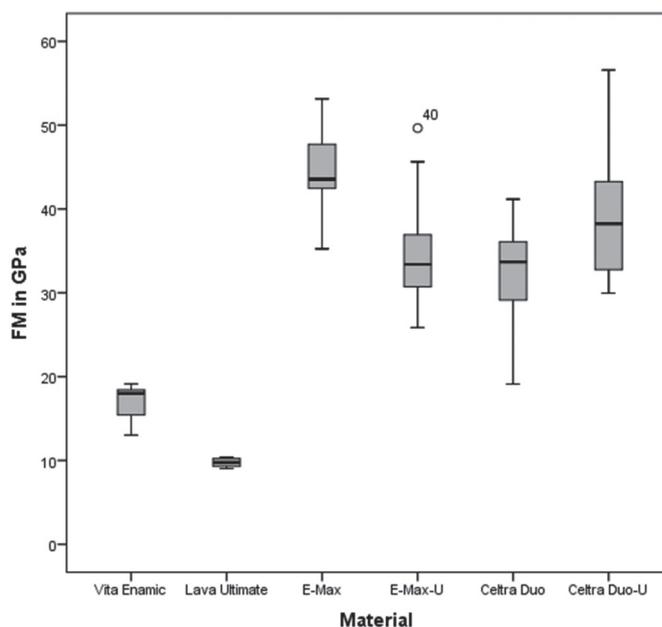


Fig. 3. Means and standard deviations of FM (MPa)

are superior to conventional powder: liquid methods, as they provide products of more uniform microstructure and minor porosity [26] with better resistance to crack growth and possessing enhanced mechanical properties [4].

Both glass ceramics, Celtra Duo and IPS e-max CAD, were tested after firing (CD), (E-Max) and also without firing (CD-U), (E-Max-U) to determine the effect of firing on their flexural properties. Obtained FS values of fired IPS e-max CAD (E-Max) were consistent with data reported by Thornton 2014 (FS: 359.87 ± 30.93 MPa) [27], while

mean FM values (44.80 ± 5.52 GPa) of the same material were slightly lower (FM: 69.29 ± 5.19 GPa).

Flexural properties differed significantly between materials having different microstructures [10]. Highest FS and FM values were recorded for crystallized E-Max (334.1 ± 54.3 MPa and 44.8 ± 5.54 GPa, respectively). These values are significantly higher than those of the rest of materials for FS ($p < 0.0001$). But for mean FM values, IPS e.max CAD only differed significantly from means for both nanoceramic resin composites, (VE): 17.18 ± 2.03 GPa and (LU): 9.75 ± 0.51 GPa.

The increase in flexural properties of partially-crystallized (E-Max-U) lithium disilicate following crystallization under vacuum at 860°C is most probably due to the change in structure and development of a higher crystalline content of finer highly interlocked lithium disilicate crystals, embedded in the glassy matrix of E-Max [28, 29]. According to the manufacturer, the partially-crystallized lithium disilicate material, IPS e.max CAD has a 40 vol% of 0.2–1.0 mm crystal size, and 70 vol% of 1.5 mm grain size [30]. The structure of crystallized E-max resulted in significantly higher FS proving the importance of crystallization for that type of material. An increase in FM values from 35.14 ± 7.46 GPa to 44.80 ± 5.52 GPa before and after crystallization can be attributed to the newly developed interlocking structure of the elongated lithium disilicate crystals [30]. The increase in FS may also be attributed to the development of tangential compressive stresses within the material leading to crack deflection, a condition which resists crack propagation [14].

Although Celtra Duo is also a glass ceramic, however, it showed statistically none significant means for fired (CD); FS: 177.32 ± 37.54 MPa, FM: 32.96 ± 6.55 GPa) and unfired (CD-U); FS: 147.61 ± 26.62 MPa, FM: 38.89 ± 8.03 GPa conditions. The differences in flexural properties between the IPS e.max CAD and Celtra Duo are most probably due to their different composition.

Celtra Duo is composed mainly of silica (SiO_2), terbium oxide, lithium composition (lithium-meta-silicate, lithium disilicate and lithium-phosphate crystals), alumina (Al_2O_3), phosphates, ceria (CeO_2) and zirconia (ZrO_2). The 10% zirconia (ZrO_2) content available in a highly dispersed form is completely dissolved in a glassy matrix. Incorporation of zirconia into ceramics is known for improving their load resistance compared to that of alumina- or lithium-disilicate ceramics [31]. Alumina added to the ceria-stabilized zirconia-based material was expected to enhance the mechanical and physical properties of the material as well [32, 33]. The ultrafine nano-sized glass ceramic crystals trapped inside ZrO_2 and/or Al_2O_3

grains further divide them into finer sized particles to form sub-grain boundaries. Such fine microstructure allows for easy and fast processing of a zirconia-reinforced lithium silicate, resulting in reduced flaw size and increased strength [34]. Nevertheless, Celtra Duo demonstrated lower flexural properties when compared to the IPS e.max.

Nano-ceramic resin composites (NCRCs) are hybrid materials containing a polymer, which imparts more resilience to the material. NCRCs were expected to fracture at higher loads, due to their hybrid nature which imparted resilience to the material [35]. However, NCRCs showed lower FS compared to the rest of materials assessed, with values only differing significantly with E-Max. NCRCs also had significantly lower FM values compared to the two glass-ceramic materials examined, for both fired and unfired specimens.

These findings were in accordance with a report in the literature that indicated minimal structural improvement of physical properties of resin composites fabricated by CAD/CAM blocks [36]. Another report suggested better mechanical properties of polymer-ceramic materials, with less brittleness, better edge stability as well as superior machinability and esthetics [37]. Nguyen et al. [38] stated that mechanical properties, including FS, hardness and fracture toughness, were significantly improved in NCRCs using the method of manufacturing pre-polymerized materials under high pressure and high temperature.

Due to their polymer incorporation, these nanoceramic resin composites show more resilience, with lower tendency to brittle fracture compared to conventional types [39]. However, their elastic moduli, density and fracture toughness of VE were reported to lie between those of resin-based composites and porcelains [40].

Flexural properties obtained in this study for (LU) and (VE) were within a range of values reported by Ilie and Hickel for resin composites (FS: ranged from 62.9–160.8 MPa; FM: 2.4–12.5 GPa) for both flexural properties [41].

Lava Ultimate revealed significantly higher FS values (168.07 ± 16.70 MPa), and insignificantly lower FM values (9.75 ± 0.51 GPa) compared to the values of VE (FS: 123.97 ± 14.84 MPa; FM: 17.18 ± 2.03 GPa). Differences between both materials may be due to the different manufacturing methods of the blocks, resulting in different organization of components, the polymers (resin matrix) and the nanoceramic particles (ceramic fillers).

Lava Ultimate contains a polymer matrix formed of Bis-GMA, UDMA, Bis-EMA and TEGDMA monomers [42], and a high inorganic content of 80 wt% nanoceramic particles of 4–11 nm zirconia and 20 nm silica, as well as 0.6–10 μm

nanoceramic particles bonded in clusters. These nanoceramic filler particles form 20% of the entire resin composite material and are tightly bound in a highly cross-linked matrix [43]. Nano-scale particles dispersed in the matrix of nanoceramic resin composites revealed better mechanical properties [38, 44]. As ceramic particles exist in the form of crystal clusters, available microcracks coalesce into larger cracks, finally weakening the nanoceramic [45]. Enhanced crack propagation significantly lowers its FS values compared to those of (E-Max) the crystallized lithium disilicate material. FS values of (LU) were even higher than those of (CD-U) (FS: 147.61 ± 26.62 MPa), the unfired Celtra Duo glass ceramic and the FS values declared by Thornton (155.31 ± 17.28 MPa) [27].

Vita Enamic has a dual network structure containing high inorganic ceramic contents of 86

wt% and 14 wt% organic polymers. This structure is presented as a hybrid ceramic framework with pores existing in a sintered ceramic matrix that become polymer-filled by capillary action after application of a coupling agent [39]. Presence of alumina as one of the dopants was also expected to improve fracture strength of the material. However, (VE) revealed lowest FS values of all materials examined.

Within limitations of this *in vitro* study it is concluded that the highest flexural properties were recorded for crystallized lithium disilicate glass-ceramic. Firing significantly improved flexural properties of lithium disilicate glass ceramic, but had no effect on flexural properties of the lithium silicate glass ceramic. The lowest FM values were recorded for both nanoceramic resin composites.

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