Relative fracture toughness and hardness of dental ceramics

Sharifa Al-Shehri, BDS, MS

مقاومة المادة لانتشار الصدع تعرف بمقياس مقاومة الانكسار. كما انها مقياس لقدرة المواد سهلة الكسر على امتصاص طاقة الإنفعال.

في هذه الدراسة استخدم الفحص بطريقة النقر فيكرز هاردنس تست لمقارنةمعامل مقاومة الانكسار (Fracture toughness K_{IC}) ومعامل الصلابة"Hardness" لست مواد . هذه المواد كانت بورسلين فيتا ف م ك ٦٨ المستخدم لبناء الطبقة العاجية للاسنان، أوبتك اتش اس بي، دايكور وآي بي اس امبرس القابلة للصب وبورسلين فيتادور ان وبورسلين الان سيرام.

النتائج الإحصائية بإستخدام تحليل الاختلاف واختبار توكي للمقارنة اظهرت وجود فرق معنوي بين المجموعات . البورسلين المدعم بالألومنيا كريستالز "الانسيرام والفيتادور ان " كانت الاعلى من حيث مقاومة الانكسار يتبعها الماده المدعمة بالفلور والميكا كرستالز "دايكور" والمواد المدعمة باللوسيت كرستالز أوبتك اتش اس بي والآي بي اس امبرس واللتي كان لها معامل صلادة متوسط ولكن اكبر من بورسلين فيتا ف ا م ك ٦٨ الفلدز باثي وزجاج الصودا لايم المستخدمة كمجموعة شاهده . هذه الفروق في الصلادة عائدة الى الفروق في التركيب لهذه المواد على المستوى الميكروسكوبي.

معامل الصلابة Hardness لمادة الدايكور كان الاقل بالرغم من ان معامل مقاومة الانكسار "Fracture Toughness K_{IC} " كان متوسطاً مقارنة ببقية المواد مما يدل على ضعف العلاقة بين هاتين الخاصيتين.

The resistance of material to crack propagation is defined as fracture toughness (K_{IC}) and is one measure of the strain energy absorbing ability of brittle materials. An identation technique was used to compare the apparent fracture toughness (K_{IC}) and hardness of six dental ceramic materials to a control soda-lime-silica glass. The materials studied included Vita VMK 68 body porcelain, Optec HSP body porcelain, Dicor, IPS- Empress castable ceramics, Vitadur N core porcelain and In-Ceram core porcelain. Alumina reinforced materials (In-Ceram core) resulted in the highest fracture toughness values followed by Vitadur N core porcelain. IPS-Empress and Optec HSP materials showed moderate but statistically significant ($P \leq 0.0001$) greater values compared to conventional feldspathic porcelain Vita VMK68 and a control soda-lime-silica glass. These differences in K_{IC} were attributed to differences in the nature of crack microstructure interaction. The hardness of Dicor castable ceramic was significantly lower than all materials tested including soda-lime-silica glass suggesting a lack of direct correlation between those two properties.

Introduction

Dental ceramics, like all brittle materials, suffer from an inability to absorb appreciable quantities of plastic strain energy prior to fracture. This liability is manifested in such behavior as flaw sensitivity, low tensile strength, and catastrophic failure.[1] One measure of the strain energy absorbing ability of a brittle material is the critical stress intensity factor "fracture toughness" or K_{IC}. The fracture toughness of a material is related to the level of tensile stress which must be attained in the vicinity of a crack tip before a catastrophic fracture process is initiated.[1] Mechanical properties as diverse as strength, thermal shock resistance, and susceptibility to erosive wear are all controlled by this parameter. Unlike fracture strength, K_{IC} is intrinsic to the material and is less sensitive to such variables of specimen preparation as the size and density of surface flaws.[2]

Because of the importance of fracture toughness in determining all aspects of brittle material mechanical behavior, knowledge of K_{IC} for dental ceramics is an essential starting point if the resistance to fracture of ceramic base dental prostheses is to be improved. K_{IC} for dental materials is usually determined through the use of an indentation technique.[3,4] This method is particularly suited to relatively expensive materials like dental ceramics since small samples are required. The basis of this technique is the series of cracks that form around a Vickers hardness indentation in a brittle material. (Fig.1) These cracks, which are termed radial cracks, appear to emanate when viewed from above, from each of the corners of the indentation. The size of these cracks, expressed by the surface dimension c (measured from the center of the indentation), increases with increasing indentation load and is

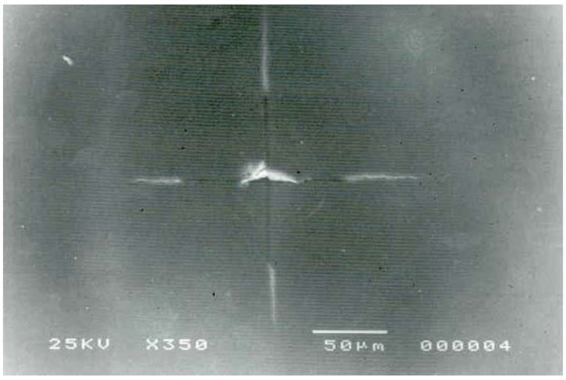

Fig. 1A. Scanning electron micrograph of typical Vickers produced indentation fracture system. B. Schematic of A: a is the indentation half-diagonal, c: is the crack size (measured from the center of the indentation).

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Lecturer, Department of Prosthetic Dental Sciences, College of Dentistry, King Saud University, Riyadh, KSA

Address reprint requests to:
Dr. Sharifa Al-Shehri
P.O. Box 60169
Riyadh 11545 KSA

an inverse function of fracture toughness. The technique has been used to obtain K_{IC} values for a variety of dental materials, including dental amalgam,[5,6] commercial dental porcelains,[2,7] and composite resins,[8] and for indirectly measuring stresses on the interface of metal ceramic restorations.[9] The rapid introduction of new dental restorative ceramic materials makes the selection of the appropriate material difficult. This investigation selected the indentation technique to determine the relative fracture toughness and hardness of several recently introduced dental ceramic materials and compared the results with a traditional feldspathic porcelain and soda-lime-silica glass.

Materials and Methods

The ceramic materials used in this study are listed in Table 1.

Table 1. Product information.

Code	Product Name	Type	Crystalline Reinforcing Component	Manufacturer
VMK	Vita VMK 68 (body)	Feldspathic porcelain	Leucite crystals	VitaZahnfabrik Bad Sackingen, Germany
DI	Dicor	Castable ceramic	Fluormica plates	Dentsply, Int. Inc. York, Pa.
EMP	IPS Empress	Castable ceramic	Leucite	Ivoclar, Schaan, Liechtenstein
OP	Optec HSP	Feldspathic porcelain	Leucite	Jeneric/Pentron Inc. Walling Ford, Conn.
VNC	Vitadur N (core)	Aluminous porcelain	Alumina	Vita Zahnfabrik Bad Sackingen Germany
INC	In-Ceram Core	Aluminous porcelain	Alumina	VitaZahnfabrik

Ceramic specimens for fracture toughness determination were circular discs 10.0 ± 0.6 mm in diameter and 1.5 ± 0.4 mm in thickness. The conventional powder systems VMK68, Optec HSP, and Vitadur N core were formed into discs by vibrating and condensing porcelain slurry into a brass mold. Excess moisture was eliminated from the porcelains by blotting with absorbent tissues. After dynamic under ambient conditions for a minimum of 4 hours, the discs were fired according to the manufacturer's recommended firing schedule using a commercial porcelain furnace (Vicumat200, Vita Zahnfabrik).

Discs of Dicor (DI) and IPS-Empress (EMP) were prepared with the lost wax technique and were cast or hot pressed, respectively. The In-Ceram core porcelain discs were made by pouring the slip material into a special silicone mold over a uniform thickness (approximately 0.5 mm gypsum material supplied by the manufacturer. After setting, the discs were sintered and glass infiltrated according to the manufacturer's instruction. Each disc specimen was mounted in acrylic resin and the exposed surface was ground flat with 150 grit aluminum oxide and polished with a sequence of steps that ranged from 180 to 600 grit. Final polish was done using 1 μm diamond paste.

Ten specimens of each material were fabricated. Polished samples were placed in an oven at 600°C for 30 minutes to relieve residual stresses that may have developed because of polishing procedures. Samples were coated with approximately 40 nm of gold using a vacuum vaporization process to allow the cracks to be more clearly seen. Also included in the study were specimens of soda-lime-silica glass, which is used in fracture studies as a reference standard. The reference sample was obtained from a glass microscope slide. It was prepared with the same coating and testing procedure as used on the ceramic samples.

Determination of fracture toughness

Fracture toughness was determined by the indentation technique. Ten to fifteen indentations were made on each sample at widely separated locations with a load of 1 kg (9.8N) for 15 seconds in a microhardness tester. The fracture toughness was calculated with the following formula $K_{IC} = 0.016\ (E/H)^{0.5}\ (P/c^{1.5})$, where K_{IC} is fracture toughness, c is the crack length (measured from

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Micromet II model 1600-9000, Buehler, Lake Bluff, Illinois the center of indentation), P is the applied indenter load, H is the Vickers hardness (0.47 P/a^2), a is the half diagonal of the indentation, and E is the elastic modulus.

A total of 50 cracks were measured for each material with crack size data obtained on 10-15 cracks per specimen. Measurements were made within 2-4h following indentation. The elastic modulus[7] for each material was determined using a three-point bend test of five rectangular specimens with the dimensions of 3x2x25mm.

All the specimens were loaded to failure. Load and displacement were monitored during testing using a precise computer program which also calculated Youngs modulus from the load displacement curves taking into account sample geometry. The average E value for each material was used in the formula to calculate fracture toughness. The fracture toughness data were subjected to analysis of variance (ANOVA) and Tukey's multiple comparison tests to determine significant differences among group means.

Results

Mean elastic modulus (E) and hardness (H) values for each material are reported in Table 2. The mean values for E were 279 ± 6.4 GPa for In-Ceram, 116.8 ± 4 GPa for Vitadur N core, and ranged from 70 ± 3.5 to 59.2 ± 3.3 GPa for the other materials. Hardness values ranged from 4.5 to 9.5 GPa. Results of the one-way ANOVA for hardness and fracture toughness K_{IC} data are summarized in Table 4 and indicate that very highly significant differences ($P \leq 0.0001$) existed among group means. Table 2 lists materials in order of increasing mean hardness values and vertical lines connect group means that were not significantly different at the 95% level of confidence. Dicor castable ceramic resulted in the lowest measured hardness values and In-Ceram core ceramic resulted in the greatest hardness.

Means and standard deviations of the measured crack length and calculated fracture toughness values for each of the ceramic materials tested are listed in Table 3. The K_{IC} value obtained for the soda-lime-silica glass was 0.8 ± 0.04 MN/m3/2 which compared favorably with the literature.[10] Mean fracture toughness values for tested dental ceramics ranged from 0.95 ± 0.09 MN/m3/2 for VMK68 feldspathic porcelain to 4.58 MN/m3/2 for In-Ceram core porcelain. Results of the ANOVA for the fracture toughness values are summarized in Table 4 and indicate that a very highly significant difference (P 0.0001) occurred among group means. The vertical lines in Table 3 connect group means that were not significantly different at the 95% confidence level. Alumina reinforced ceramic materials, In-Ceram core and Vitadur N core porcelain, resulted in an appreciably greater fracture toughness than the other materials tested. The two castable ceramic materials tested, Dicor and IPS-Empress, and Optec HSP feldspathic porcelain resulted in an appreciably greater fracture toughness than conventional feldspathic porcelain (VMK68) which had a significantly higher K_{IC} than the reference glass.

Table 2. Relative elastic modulus (E) and hardness (H) mean values and their strandard for deviation.

Material Code	No. of Specimens	E (GPa)	No. of Specimens	H (GPa)
DI	5	70±3.5	10	4.46±0.31
GL	5	73.4±3	10	5.2±0.21
EMP	5	69.8±3.7	10	6.53±0.34
VMK	5	59.2±3.3	10	6.56±0.32
Vita	5	63.6±4.2	10	6.7±0.45
OP	5	116.8±4	10	7.9±0.56
Inc	5	279.6±6.4	10	9.5±0.46

Table 3. Relative crack length (c) and fracture toughness (K_{IC}) mean values and their standard deviation.

Material Code	No. of Specimens	C (μm)	K_{IC} (MN / m$^{3/2}$)
Inc	10	95.7±7.5	4.58±0.42
VNC	10	46.5±6.2	1.95±0.36
OP	10	51.9±3	1.29±0.07
EMP	10	55.5±4.5	1.26±0.16
Di	10	62.1±4.4	1.28±0.09
VMK	10	62.7±3.4	0.95±0.08
GL	10	81.3±3.9	0.8±0.04

Table 4. Summary of one way ANOVA for hardness and fracture toughness values.

		Sum of Sqaures	df	Mean square	F	Sig.
v. hardness	Between groups	165.880	6	27.647	177.565	.000
	Within groups	9.809	63	.156		
	Total	175.689	69			
f toughness	Between groups	102.479	6	17.080	331.716	.000
	Within groups	3.244	63	5.149E-02		
	Total	105.723	69			

Discussion

The indentation derived fracture toughness and hardness values for conventional feldspathic porcelain, alumina reinforced porcelain, fluormica glass ceramic, Lucite reinforced glass ceramic and reference soda-lime-silica glass were in agreement with similar values reported previously.[2,7,10-12]

Hardness is a property of restorative materials that is generally considered important to applications involving friction and wear, although a direct correlation between matched hardness to minimize wear cannot be made. Intraoral conditions limit this; and indeed, a hard material will scratch, abrade, and wear away opposing tooth structure. It is desirable for a restoration to resist abrasion and have a wear rate equal to that of enamel. Materials with low hardness, like Dicor castable ceramic, will probably not damage the natural antagonists. However, the rate of tooth substance wear has been found to be a function of roughness.[13]

This investigation indicated that substantial improvements were apparent in fracture toughness of several new dental ceramics relative to conventional feldspathic porcelain, but the fracture resistance of these new materials was still far below that of alumina reinforced materials. Although the optical properties of ceramic materials were not measured in this investigation, optical effects are critical for selection of materials. Nevertheless, it could be generally stated that materials with intermediate fracture toughness and translucency similar to feldspathic porcelain (i.e. Optec HSP, Dicor,and IPS- Empress) are often preferred over materials with greater fracture resistance and high opacity (namely, Vitadur N core and In-Ceram core) when higher esthetic properties are required as in anterior crowns and veneers. On the other hand, if resistance to fracture is the primary concern as for posterior crowns and bridges, higher fracture toughness materials should be selected.

Several approaches to strengthening ceramics have been recognized in the past two decades. The ways leading to increased fracture toughness or specific fracture energy of ceramics generally involve manipulation and tailoring of the microstructure. The microstructure can be improved by optimizing the proportions of the individual phases, including crystalline and glassy phases, by adjusting their shape and size distribution, by optimizing the size and shape of crystalline grains, and by improving their mutual bonds. If the ceramic body contains a relatively high proportion of glassy phase, the crack will primarily propagate through this phase and the properties of the other phases will have little effect. VMK68 and Optec HSP are both leucite reinforced porcelains, though Optec HSP showed higher K_{IC} in this study. This could be the manifestation of improved microstructure by incorporating a higher amount of the crystalline phase for Optec HSP (48% vol) as compared to VMK (19% vol).[14] Another possible reason for the increased K_{IC} of Optec as compared to VMK68 is the difference in grain size of raw material (10-15 m for Optec and 15-20 m for VMK).[14]

A fine homogeneous microstructure with the smallest grain is another requirement for increasing fracture toughness of ceramics. The probability of defects occurring in the crystal lattice or in the grains decreases with decreasing grain size, as does the energy of microstresses which has accumulated in grains and is available for possible formation of microcracks. In addition, grain boundaries always represent an obstacle to crack propagation from one grain to another.[15] One favorable property of polycrystalline ceramics is that cracks can only propagate over certain lattice planes inside the grains in the case of transgranular fracture and through the narrow space between the grains for intergranular fracture. The crack propagation is therefore spatially restricted with the result of higher forces necessary and of a larger total fracture area and thus a higher overall energy consumed. The inhomogenous stress distribution in front of the crack due to anisotropy of the elastic properties of the individual crystals also plays a role. If the grain size is too small, these effects cease to influence the process.[15] Evidence for this is given by the generally easy crack propagation in glass whose structure is almost ideally disarranged.

A significant part is also played by the sintering process and the presence of pores. For this reason, a special technique for ultrasonic mixing of In-Ceram core porcelain was introduced with the system. Residual pores are then removed after sintering through glass infusion. This technique seems to have a positive effect in reducing the amount of total porosity of this material. As far as pores are concerned, these should not arise at grain boundaries where they increase the stress concentration at points where weakened bonds already occur. In contrast to this, pores of spherical shape arising for example in the glassy phase may even have a favorable effect by blunting the crack front so that greater energy is required for its growth.[15]

Particle sizes and the amount of crystalline phase are believed to affect the resulting toughness. In-Ceram core and Vitadur N core are similar materials that differ mainly in crystalline size (1-5 m for In-Ceram and 10-25 m for Vitadur N core)[14] and the amount of crystalline phase in the glass matrix (85+% for the In-Ceram core and 30% (vol) for Vitadur N core). The improved fracture toughness of In-Ceram core supports the concept that increased amounts and finer grains tend to improve toughness.

A glass ceramic is initially a glass in which, at some stage, the formation of nuclei is enhanced either by the addition of nucleating agent of by using special compositions which are self-nucleating. The resulting material contains very small crystals in a residual glassy phase. If the crack, whenever nucleated, takes a tortuous path through the glass ceramic, then more energy will be absorbed in surface production, and fracture toughness will be improved.[16] The residual glass phase will be of major importance because stresses developed at the interface between crystal and glass will, to a large extent, determine the crack path. Stresses between the two phases arise from thermal expansion mismatch, and consideration of this leads to expectations for improved toughness. For this, suitable oxide additions can be made to the original base glasses so that the residual glass will have a lower coefficient of thermal expansion than the crystalline phase.

Residual stress in the interface is tensile, and fracture will always be expected to be intergranular, with longer path length and higher fracture toughness.[16] This is the primary strengthening mechanism in the case of Dicor and IPS Empress glass ceramics. The leucite reinforced IPS Empress is further strengthened through the residual stresses created by volume reduction associated with the high to low temperature phase transformation of leucite. This, in addition to the high coefficient of thermal contraction, creates a situation that causes leucite crystals to contract substantially more than the glass matrix that surrounds the particles and are frozen during the cooling phase. Tensile forces are created in the particles leading to microcracking in the leucite phase. Branching of a primary crack into a greater number of secondary cracks or simultaneous growth of many cracks leads to an increase in the fracture surface and volume, reducing the stress peaks.[17,18]

Conclusion

1. In-Ceram had substantially greater hardness and fracture toughness than all other materials.
2. Dicor had a moderate fracture toughness and the lowest hardness, suggesting a weak correlation between the two properties.
3. Alumina was the most effective toughening phase currently used in dental ceramics.
4. Leucite and fluor mica were both effective at increasing the toughness of ceramic materials (Optec HSP, IPS-Empress, and Dicor castable ceramics) over that of conventional feldspathic porcelain but the effect was not as pronounced as with alumina.

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