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Brittleness index of machinable dental materials and its relation to the marginal chipping factor

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Abstract

Objectives: The machinability of a material can be measured with the calculation of its brittleness index (BI). It is possible that different materials with different BI could produce restorations with varied marginal integrity. The degree of marginal chipping of a milled restoration can be estimated by the calculation of the marginal chipping factor (CF). The aim of this study is to investigate any possible correlation between the BI of machinable dental materials and the CF of the final restorations.

Methods: The CERECTM system was used to mill a wide range of materials used with that system; namely the Paradigm MZ100TM (3M/ESPE), Vita Mark II (VITA), ProCAD (Ivoclar-Vivadent) and IPS e.max CAD (Ivoclar-Vivadent). A Vickers hardness Tester was used for the calculation of BI, while for the calculation of CF the percentage of marginal chipping of crowns prepared with bevelled marginal angulations was estimated.

Results: The results of this study showed that Paradigm MZ100 had the lowest BI and CF, while IPS e.max CAD demonstrated the highest BI and CF. Vita Mark II and ProCAD had similar BI and CF and were lying between the above materials. Statistical analysis of the results showed that there is a perfect positive correlation between BI and CF for all the materials.

Conclusions: The BI and CF could be both regarded as indicators of a material's machinability. Within the limitations of this study it was shown that as the BI increases so does the potential for marginal chipping, indicating that the BI of a material can be used as a predictor of the CF.

Keywords: Brittleness index, Chipping factor, Machinability, Marginal chipping, CAD/CAM, CEREC

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1. Introduction

The application of CAD/CAM technologies for the fabrication of dental prosthesis had as a result the introduction of new methods of processing materials, many of which would be unavailable to the restorative dentist via other processing routes.^{1,2} Many of the materials available with these systems can be adhesively bonded to the tooth, providing the dentist with the prospect of less invasive dentistry. This would refer though to restorations with more conservative marginal angles and wall thicknesses to that of traditional restorations.

In a previous study it was reported that the CERECTM system (Sirona Dental Systems GmbH, Bensheim, Germany) can produce restorations with different marginal angles (shoulder, chamfer or bevel) with clinically acceptable marginal gaps.³ However, the integrity of the margins is another important factor for the longevity of a dental restoration. Since CAD/CAM systems utilize abrasive machining processes (i.e. grinding and milling), there is a potential for generation of machining induced damage that could reduce the integrity of the final restoration.⁴ Consequently the machinability of the chosen material can influence the integrity of a minimally designed restoration, prohibiting the application of certain minimal designs (i.e. bevelled margins).

A common observation of a materials' surface damage due to machining are chipping defects. These defects can reduce the accuracy of fit of a restoration and can potentially contribute to the reduction of mechanical strength overtime.^{5,6}

The machinability of a material can be simply assessed qualitatively as the ease with which a given material is cut. However, its accurate quantitative measurement is more difficult. Various parameters have been suggested as the “measurement” of the machinability, such as tool wear, surface roughness, cutting force, cutting energy, drilling rates, etc.⁷⁻⁹ Another method for determining machinability has been suggested by Boccaccini who proposed the brittleness of a material as a parameter for estimating its machinability.¹⁰ One useful approach for the quantification of the brittleness of materials has been proposed by Lawn and Marshall,¹¹ consisting of a simple index of brittleness that can be derived from the hardness (H) and fracture toughness (K_{Ic}) of the material (Eq. (1)):

$$B = \frac{H}{K_{Ic}} \quad (1)$$

More recently Sehgal and Ito,^{12,13} have shown that the brittleness of glasses can be more easily determined from the following equation:

$$B = \gamma P^{2/4} \left[\frac{C}{\alpha} \right]^{3/2} \quad (2)$$

In this equation, B is the brittleness in $\mu\text{m}^{1/2}$, P the indentation load (N) for median cracking, γ equals to $2.39 \text{ N}^{1/4}/\text{mm}^{1/2}$, C is the median crack length and α is the contact diagonal of the indent in mm. According to their method to define the brittleness of a glass, the material has to be indented at a constant load of 49 N.¹³ Boccaccini has also shown using Eq. (2) that C/α alone can give an estimate of brittleness for composites and glass ceramics.¹⁴ However, the brittleness index of dental machinable materials, glass ceramics or composites, has not yet been calculated.

In the present study the concept of chipping factor (CF) is introduced as an estimation of the degree of marginal chipping, which can be derived by estimating the ratio of overall marginal chipping over the total marginal circumference of the restoration multiplied by 100 to give the percentage of chipping Eq. (3):

$$CF = \left[\frac{L}{P} \right] * 100 \quad (3)$$

In this equation L is the amount of marginal chipping and P is the marginal circumference of the restoration.

The question posed in this study is whether it would be possible to predict the susceptibility of a CAD/CAM material to marginal chipping simply from the knowledge of the brittleness index of the material. The aim of this study is to measure the brittleness index (BI) and the chipping factor (CF) of a range of dental materials used with the CEREC system and to assess their relationship. The null hypothesis is that there is a positive correlation between these two parameters and as the BI increases so does the potential for marginal chipping.

2. Materials and methods

A broad spectrum of materials was examined in this study, including a glass ceramic (ProCAD, Ivoclar-Vicadent AG, FL- 9494 Schaan-Liechtenstein), a feldspar ceramic (VITA MKII, VITA Zahnfabrik, Bad Sackingen, Germany), a hybrid composite (ParadigmMZ100, 3M ESPE Dental Products, St. Paul, USA) and a lithium disilicate ceramic material in a pre-crystallized (blue) stage (IPS e.max,

Ivoclar-Vivadent AG, FL-9494 Schaan- Liechtenstein). Details of their chemical structure, brand name and manufacturer, are shown in Table 1.

Table 1 Details of the materials used in the study

Brand Name	Manufacturer	Basic Chemical Structure	Milling stage
VITA Mark II	VITA Zahnfabrik	Fine particle feldspar ceramic	Fully sintered
ProCAD	Vivadent-Ivoclar	Leucite reinforced glass ceramic	Crystallized
IPS e.max	Ivoclar-Vivadent	Lithium silicate glass ceramic (milling phase)	Pre-crystalline stage
Paradigm MZ100	3M ESPE	Resin composite with 85wt% ultrafine zirconia-silica ceramic particles (size of 0.6 µm)	Cured

2.1. Measurement of brittleness index

Specimens were cut from the commercially available blocks for CAD/CAM processing. Each block was cut with a diamond wheel (LECO VC-50, LECO Instrument (UK) Ltd., Stockport, Cheshire, England) in thinner sections, so that all specimens had an approximate width of 3 mm. Two specimens of each material were produced. A grinding and polishing device (Buehler Metaserv™, Grinder-Polisher, Buehler UK Ltd., Coventry, England) was used to sequentially grind all specimens with 120, 400, 600 and 1200 grit SiC papers to ensure parallel surfaces and a uniform thickness of 2 mm. Finally, a mirror polish was produced using 6 and 1 mm diamond pastes (Buehler® Metadi® Diamond Suspensions, Buehler UK Ltd., Coventry, England).

Each specimen was indented using a Vickers hardness tester (Vickers Armstrong Engineers Ltd., Serial No.: 255002, Grayford, Kent, England) with an applied load of 49 N in ambient air until five acceptable indents were obtained. The criteria for acceptability were: (1) all cracks emanated from the corners of the indent, (2) presence of only four radial cracks, (3) no crack chipping and (4) no crack branching¹⁵ (Fig. 1). In total 10 indents were produced for each material. Readings were performed as soon as possible after indentation before any time dependent cracking occurred.¹⁶ An optical microscope (Polyvar-MET, Reichert Ophthalmic

Instruments, New York, USA) connected to a PC (Athlon 1200, 256 MB) was used to obtain images of the indents. Image analysis software (KSRUn 400 Imaging System, v.3.0, Carl Zeiss Vision GmbH, Göttingen, Germany) was used to measure the crack lengths (C) and the contact diagonals (α). The brittleness index (BI) was calculated according to the equation of Sehgal and Ito (Eq. (2)), which relates the indentation load (P), the size of the median cracks (C) and the indentation diagonal lengths (α).

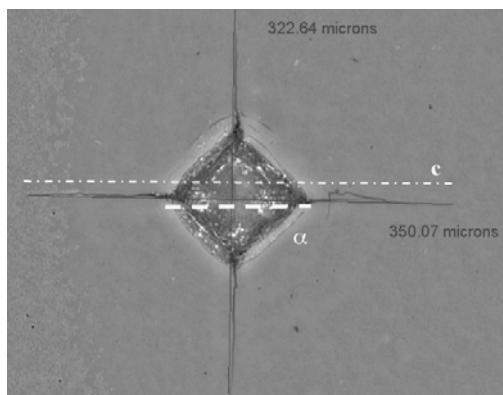


Figure 1 A typical indent produced with the Vickers tester. The top dashed line represents the crack length C and the bottom dashed line the contact diagonal α .

2.2. Calculation of chipping factor

In order to determine the chipping factor of the machinable materials the marginal finish of crowns prepared with bevelled marginal angulations was evaluated. Crowns with a 30° bevel finishing line were prepared. To minimize variations in crown shape, dimension or thickness a master model was fabricated in brass and was machined to approximate the dimensions of a molar abutment, with a 2 mm occlusal reduction and a 1.2 mm axial reduction. For the designing process it was found that the presence of a neighbouring tooth was convenient and for that reason a premolar phantom tooth was attached to the brass model to resemble a clinical situation. An addition curing vinyl polysiloxane material (Dublisil-HC, Dreve-Dentamid GmbH, Unna, Germany) was used to duplicate the master brass model. A duplicate cast was fabricated with the use of a special die stone (CAM-base®, Dentona, Dortmund, Germany). The CEREC Scan system was used for the scanning of the cast, designing and fabrication of the crowns. The duplicate cast was fixed on a special model holder provided with the CEREC Scan and scanning was performed according to the manufacturers' instructions. The Cerec 3D software (V2.10 R1500) was used for designing a crown restoration and three crowns were milled for each material. The default milling mode and the default milling burs (CEREC Cone-shaped Cylinder

Diamond 1.6, Art. No. 5855734 D3329, CEREC Cylinder Diamond 1.6, Art. No. 5466193b D3268, Sirona Dental Systems GmbH, Bensheim, Germany) were used to mill the crowns. A new set of burs was used for each material. A lubricant (Dentatec Lubricant, Sirona, Bensheim, Germany) provided with the system was used during cutting according to manufacturer's instructions.

Quantitative analysis of the amount of marginal chipping of the crowns was performed, by taking a series of images of the perimeter of the crowns. The edge of each crown was divided in eight sections so that when the distance between two points was observed axially would be a straight line (Fig. 2a). Also a top view image of the margins of each crown was taken to measure the circumference (maximum crown margins periphery, P) (Fig. 2b). The images were taken using a 32-bit digital camera (Kodak/Nikon DCS 410). The camera was connected to a PC (Pentium CX1 [2x 800MHzCPU], Viglen Ltd., UK) and the acquisition software was Adobe Photoshop (V5.0, Adobe Systems Ltd., Europe). The images were next imported into Image Pro Plus software (V4.01, Media Cybernetics, USA) for analysis of the marginal finish. Each image was calibrated with a steel rule adjacent to the crown surface. The length (L) of the chipped margins and the perimeter (P) of the crowns were measured and the chipping factor (CF) for each crown was calculated using Eq. (3).

The same observer determined the marginal chipping of the specimens. To calculate the intra-examiner error the measurements were repeated twice. The second measurements were made a week later and without the examiner referring to the first measurements. The differences were calculated and a reliability analysis was performed.

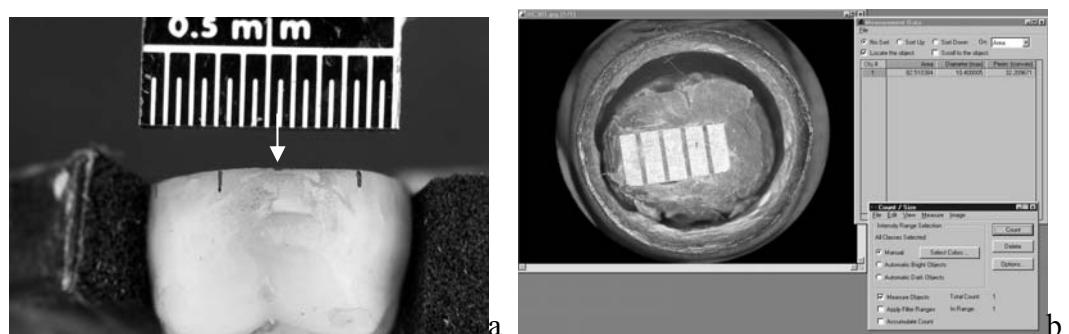


Figure 2 Axial and top view images of a crown sample showing the length (L) of a chip as indicated by the arrow (a), and measurement of the maximum crown margins periphery (P) of the sample (b) using image analysis software.

2.3. Data analysis

The statistical package SPSS 14 was used to perform a reliability analysis of the CF measurements and a statistical analysis of the results. For the reliability analysis the intraclass correlation coefficient (ICCC) was measured. A one-way ANOVA analysis was performed to find any statistically significant differences between the brittleness indices (BI) and chipping factors (CF) reported for the four materials. Finally, the correlation between BI and CF was calculated using Spearman's r correlation coefficient (r_s).

3. Results

3.1. Reliability analysis

The reliability analysis of the two measurements of the chipping of the crowns gave an intra-class correlation coefficient equal to 0.994, which indicated a very good correlation and consequently a very small inter-examiner error.

3.2. Brittleness index

The brittleness index for each material is shown in Table 2. It can be seen that the composite material (Paradigm MZ100) had the lowest brittleness index. Vita Mark II and ProCAD had similar indices, while IPS e.max CAD demonstrated the highest brittleness index. A one-way analysis of variance and pair wise multiple comparisons showed that there was a statistically significant difference between the BI of the four materials ($p < .05$) (Table 2).

3.3. Chipping factor

The average CF and standard deviations (S.D.) of each material are given in Table 2. The IPS e.max demonstrated the poorest margins with a 70% chipping over the total circumference of the crowns, while the Paradigm MZ100 gave margins with the fewest defects (0.86% chipping). A one-way analysis of variance and pair wise multiple comparisons were performed between the CF of the four materials, which showed that there was a statistically significant difference between the four materials ($p < .05$) (Table 2).

3.4. CF and brittleness index

A plot of CF versus BI is shown in Fig. 3, where it can be seen that the CF increases as the BI increases. A correlation analysis was performed in order to measure the strength of the relationship between brittleness and chipping factor. The Spearman's r correlation coefficient (r_s) was used to determine the strength of correlation which showed a perfect correlation between the two parameters ($r_s = 1$).

Table 1 Average CF with standard deviations (SD) and brittleness index BI. The statistically significant different groups are indicated with a different background colour (materials in the same background colour group showed no statistically significant differences).

Materials	CF(%)	BI ($\mu\text{m}^{-1/2}$)
Paradigm MZ100	0.86±0.8	0.50±0.03
VITA	8.5 ±2.8	1.60±0.15
ProCAD	8.7±2.5	1.70±0.14
IPS e.max	69.8 ±5	2.90±0.54

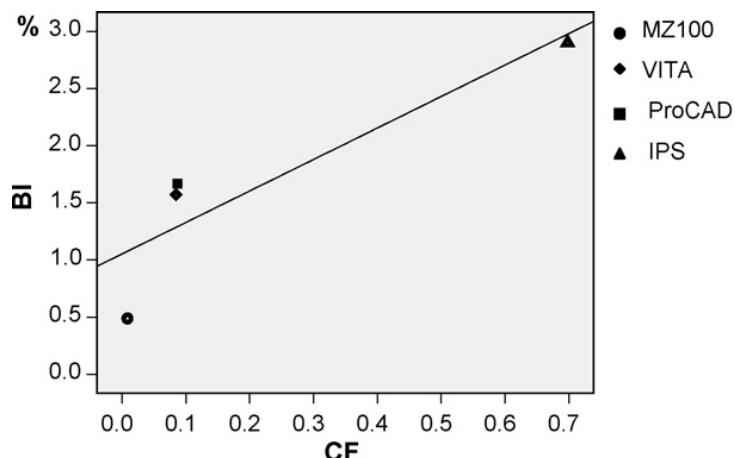


Figure 3 Plot of CF vs. BI where it is clear an increase of CF as the BI increases

4. Discussion

One of the consequences of computer aided machining is the creation of surface flaws due to the machining process. All available materials for CAD/CAM machining and especially ceramics potentially suffer from surface chipping and subsurface defects.⁶ It has also been found in the present study that these defects can get more prominent if a more conservative tooth reduction is attempted by preparation of bevelled margins especially with specific materials. Up till now there is no clear

way of measuring this machining parameter other than direct experimentation. The chipping factor (CF) was introduced as a direct method to measure the degree of marginal chipping. However, this is not a particularly convenient method to predict the marginal chipping of a material as CF can only be calculated after milling. In contrast, the brittleness index is a relatively easy measurement of a material's machinability.

The reason to identify any possible relationship between CF and BI lies in the fact that both these parameters relate to machinability and BI could be an easier parameter to calculate. It has already been mentioned that even though machinability is broadly understood as the ease with which a material is cut, it is not readily measured in quantitative terms. Various parameters have been suggested by different researchers to quantify machinability, which depend on the microstructure and properties of the material. In particular fracture strength, hardness and fracture toughness have been considered for the prediction of machinability.⁷⁻⁹

Boccaccini's proposal of using the brittleness index as an estimate for the machinability of glass ceramics is based on the rationale that brittleness is a measure of the relative susceptibility of a material to deformation and fracture.¹⁰ The brittleness index provides a relationship between the hardness (H), which quantifies the resistance to deformation, and the toughness (K_{Ic}), which quantifies the resistance to fracture. Since machinability involves deformation and microfracture, the brittleness index, combining the response of the material to both of these phenomena, should be a better parameter for its quantification than either hardness or fracture toughness taken separately.¹⁰

However, unlike other mechanical properties the concept of brittleness is not so well defined. The knowledge of the factors that govern brittleness is largely empirical and there is little literature dealing specifically with this material characteristic. A variety of techniques and parameters have been suggested to characterize brittleness. A report by Quinn and Quinn contains some information on the previous work on the brittleness of glass with a variety of techniques and parameters that have been proposed to characterize brittleness.¹⁶ A more convenient method was proposed by Lawn and Marshall which was later evolved by Sehgal and Ito to an equation where only the indentation load, the crack length and the contact diagonal are used for the calculation of brittleness.¹¹⁻¹³ According to Sehgal and Ito the ratio C/α can be used as an index of brittleness for glasses and isotropic

monocrystals, however, it may be inadequate for polycrystalline and composite materials especially if they are anisotropic. The validity, though, of this equation will also depend on the type of cracks emanated around the indentation. Two types of cracks can result from the indentation, namely a median/radial crack and a Palmqvist crack. The former consists of two halfpenny cracks perpendicular to the plane of indentation, whilst the latter consists of semi-elliptically shaped cracks.^{17,18} The equation of Sehgal and Ito only applies in the case of a median/radial crack.¹⁹ Using the technique of grinding back from the surface it was possible to deduce that we obtained a median/radial crack. Hence the equation was valid for the calculation of the brittleness index.¹⁹

The results of the current study showed that as expected all materials had a brittleness index lower than $4.3 \text{ mm}^{1/2}$, which is within the range that show good machinability.¹⁴ However, the composite material had the lowest brittleness index, which indicated that it had better machinability compared to the ceramic materials.¹⁴

There are a few studies that have examined the chipping of machinable materials as a result of machining. Flanders et al. tested the scratch hardness and degree of chipping of machinable dental ceramics with respect to the effect of the cutting environment.²⁰ In this study the degree of chipping was determined by calculating the lengths that chipped out along a scratch made on the material's surface. In the present study it was decided to determine the degree of chipping by calculating the total lengths of the edge margins of machined restorations that chipped out during milling, as it would more accurately represent the machining process. The chipping factor of a material could be regarded as another indicator of machinability, as it is the result of this process.

The results of this study showed that the chipping factor varies according to the material used. It was found that the composite material (ParadigmMZ100) had the lowest chipping factor while the lithium disilicate glass ceramic (IPS e.max) had the highest CF. The feldspathic (VITA MKII) and leucite reinforced glass ceramic (ProCAD) showed similar chipping factors. A material with greater level of chipping during milling is likely to have a reduced quality of marginal fit because of greater damage to the margins. This might be the case for the IPS e.max ceramic whose physical properties are improved by subsequent firing.

When the brittleness index (BI) and the chipping factor (CF) of the tested materials were compared it was clear that there was a correlation between them as the

chipping factor was increasing as the brittleness index increased. Correlation analysis verified this, giving a perfect positive correlation relationship between BI and CF ($r_s = 1$).

5. Conclusions

The null hypothesis was accepted, that is as the brittleness of a material increases so does the chipping factor. The significance of this finding is that by knowing the brittleness of a material the degree of chipping during milling can be predicted and consequently the degree of conservation with regard to tooth reduction can be determined. Within the limitations of this study we could conclude that a material with a high brittleness index would not represent a favourite candidate when a minimal preparation design is desired, as it would result in a restoration with high chipping factor which would compromise its marginal fit.

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References

1. Mehl A, Hickel R. Current state of development and perspectives of machine-based production methods for dental restorations. International Journal of Computerized Dentistry 1999;2:9–35.
2. Kelly JR, Luthy H, Gougoulakis A, Pober RL, Mormann WH. Machining effects on feldspathic porcelain and glassceramic: fractographic analysis.. In: Mormann WH, editor. State of the art of the CEREC method international symposium on computer restorations. Zurich, Switzerland: Quintessence;1991. p. 253–73.
3. Tsitrou EA, Northeast SE, van Noort R. Evaluation of the marginal fit of three margin designs of resin composite crowns using CAD/CAM. Journal of Dentistry 2007;35:68–73.

4. Rekow ED. High-technology innovations- and limitations for restorative dentistry. *Dental Clinics of North America* 1993;37:513–24.
5. Shearer AC, Heymann HO, Wilson NH. Two ceramic materials compared for the production of CEREC inlays. *Journal of Dentistry* 1993;21:302–4.
6. Sindel J, Petschelt A, Grellner F, Dierken C, Greil P. Evaluation of subsurface damage in CAD/CAM machined dental ceramics. *Journal of Materials Science Materials in Medicine* 1998;9:291–5.
7. Baik DS, No KS, Chun JS, Yoon YJ, Cho HY. A comparative evaluation method of machinability for mica-based glass ceramics. *Journal of Materials Science* 1995;30:1801–6.
8. Xu HHK, Jahanmir S, Scratching. Grinding of a machinable glass-ceramic with weak interfaces and rising T-curve. *Journal of the American Ceramic Society* 1995;78:497–500.
9. Taira M, Yamaki M. Ranking machinability of 9 machinable ceramics by dental high-speed cutting tests. *Journal of Materials Science Letters* 1994;13:480–2.
10. Boccaccini AR. Machinability and brittleness of glassceramics. *Journal of Materials Processing Technology* 1997;65:302–4.
11. Lawn BR, Marshall DB. Hardness, toughness, and brittleness—indentation analysis. *Journal of the American Ceramic Society* 1979;62:347–50.
12. Sehgal J, Nakao Y, Takahashi H, Ito S. Brittleness of glasses by indentation. *Journal of Materials Science Letters* 1995; 14:167–9.
13. Sehgal J, Ito S. A new low-brittleness glass in the soda-lime–silica glass family. *Journal of the American Ceramic Society* 1998;81:2485–8.
14. Boccaccini AR. Assessment of brittleness in glass-ceramics and particulate glass matrix composites by indentation data. *Journal of Materials Science Letters* 1996;15:1119–1121.
15. Scherrer SS, Denry IL, Wiskott HW. Comparison of three fracture toughness testing techniques using a dental glass and a dental ceramic. *Dental Materials* 1998;4:246–55.
16. Quinn JB, Quinn GD. Indentation brittleness of ceramics: a fresh approach. *Journal of Materials Science* 1997;32:4331–46.
17. Ray KK, Dutta AK. Comparative study on indentation fracture toughness evaluations of soda–lime–silica glass. *British Ceramic Transactions* 1999;98:165–71.

18. Ogilvy IM, Perrott CM, Suiter JW. On the indentation fracture of cemented carbide. Part 1. Survey of operative fracture modes. *Wear* 1977;43:239–52.
19. Hand R.J. Personal communication, Faculty of Engineering, Department of Engineering Materials, University of Sheffield; 2005.
20. Flanders LA, Quinn JB, Wilson Jr OC, Lloyd IK. Scratch hardness and chipping of dental ceramics under different environments. *Dental Materials* 2003;19:716–24.