

Three Point Bending Strength of In-Ceram Core Material Sinterized with Colloidal Alumina

In-Ceram Tozunun Kolloidal Alumina ile Sinterize Edilmesi Sonucu Dayanıklılığının İncelenmesi

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ABSTRACT

Aims: The aim of this study was to investigate the effect of the use of a colloidal alumina/inorganic binder on the shear strength of In-Ceram core material.

Methods: Two groups of samples (A & B) were fabricated in accordance with the relevant ISO specification; while the fabrication of group A was conducted in the conventional manner, group B samples were fabricated by using In-Ceram powder and colloidal alumina as binder. Three point bending test was applied on each group. The sample and the fracture surface were evaluated with SEM (Scanning Electro Microscope) x600, x800, x1200, and x2000 magnification.

Results: The mean fracture resistance for groups A and B were 963.75 MPa and 1164 MPa, respectively and the difference between two groups were statistically significant ($P<0.05$)

Conclusion: Within the limitations of this study, the use of colloidal alumina seems to be promising in reinforcing the In-Ceram core material.

KEYWORDS

In-Ceram, Colloidal Alumina, Three point bending

ÖZET

Amaç: Bu çalışmada In-Ceram kor materyalinin kolloidal alumina ile sinterize edilmesi sonucu dayanıklılığı araştırılmıştır.

Metod: Uluslararası standartlar organizasyonunun (ISO) belirlediği ölçülerde 2x4x25 mm boyutlarında In-Ceram kor materyalinden oluşan iki ayrı grup hazırlanmıştır. A grubu örnekleri geleneksel In-Ceram kor yapı hazırlama şekline uygun hazırlanmıştır. B grubu örnekleri ise In-Ceram tozuna inorganik bağlayıcı olarak (binder) kolloidal alumina kullanılarak hazırlanmıştır. Örnekler üç nokta eğilme testine tabi tutulmuştur, ve kırılma yüzeyleri SEM (Scanning Electro Microscope) ile x600, x800, x1200, ve x2000 büyütme ile incelenmiştir.

Bulgular ve Sonuç: A grubu örneklerinin kırılma kuvvetleri ortalama 963.75 MPa, B grubu örneklerinin kırılma kuvvetleri ortalama 1164 MPa olarak belirlenmiştir. İstatistiksel açıdan gruplar arasında fark olup olmadığı incelenmiş ve birbirleriyle karşılaştırılmıştır. İki grup arasında istatistiksel açıdan önemli fark ($p<0.05$) bulunmuştur.

Sonuç: Test sonuçları kolloidal alüminanın binder olarak kullanımının daha iyi sonuçlar verdiğini göstermiştir.

ANAHTAR KELİMELER

In-Ceram, Kolloidal Alumina, Üç nokta eğilme testi

INTRODUCTION

The word “ceramic” that we use extensively today is derived from the word “keramos” in Greek and means “earthen”. Ceramics are structurally modified inorganic materials used since the most early periods of human civilization that can be dated as early as 23 000 BC¹⁻².

Although metal-ceramic restorations are widely used in dentistry owing to considering their adequate strength and marginal fit properties, some of their disadvantages especially the limitations at cervical areas as a result of metallic color problems and opaque appearance because of thin porcelain layers, and also problems occurring because of corrosion byproducts induced the research and development of all ceramic restorations³⁻⁶.

In-Ceram restorations gained widespread acceptance and use owing to their high biocompatibility, high physical strength, good esthetics and marginal fit. At present, this material can be used for anterior and posterior crowns and anterior three unit bridge restorations⁷⁻¹¹. Several zirconium and yttrium based materials are developed to provide adequate strength to withstand chewing forces occurring at posterior areas. The main philosophy of further developing the physical strength of ceramic materials is generally based on increasing the amount of high strength ingredients like aluminum oxide, zirconium or yttrium¹²⁻¹³⁻¹⁴⁻¹⁵.

Colloidal alumina (Boehmite: $\text{Al}(\text{OH})3\text{Al}_2\text{O}_3$) is a monohydrated form of alumina and have a gelatinous appearance. The -OH groups in its structure are forced to transformation to $\gamma\text{-Al}_2\text{O}_3$ at 4500C and to $\alpha\text{-Al}_2\text{O}_3$ at 12000C. The most advantageous situation in this transformation is that the Boehmite will transform into $\alpha\text{-Al}_2\text{O}_3$ with a loss of 15% by weight and will be incorporated into the final ceramic¹⁶.

Three point bending tests are suggested test methods by ISO (1984) and ANSI/ADA (1992) for the evaluation of ceramic based materials. This method is simple and reliable and easy to

compare with the results of other studies. However, it must be noted that the values can greatly differ due to superficial cracks and defects, characteristic of ceramic failures.

Andrea et al (1989) reported that colloidal Boehmite gel is incorporated into the final porcelain when it is used as binder for aluminum oxide powder¹⁶.

The purpose of this study was to investigate the three point bending strength of colloidal alumina/inorganic binder matrix enriched In-Ceram core material.

MATERIALS AND METHOD

Two groups, each consisting of ten samples (A: Conventional In-Ceram; B: Modified binder-In-Ceram) (VITA Zahnfabrik Bad Säckingen, Germany) were fabricated in accordance with the relevant ISO specification; while the fabrication of group A was conducted in the conventional manner, group B samples were fabricated by using a powder including the experimental colloidal alumina / modified binder (Nyacol Products PQ Corporation, Megunco, Ashland). Three point bending test was applied on each group.

Silicone duplicates of test samples were prepared in a brass jig (2,1x4,1x25 mm) that was made for fabrication of standard samples. The duplicates were positioned on a glass slab and dental stone was poured and cast on.

Preparation of group A samples

In-Ceram powder and liquid was mixed according to the manufacturer's recommendations. 38 g powder, 5 ml liquid and one drop of binder was mixed in a glass mortar for 12 minutes. The material was further mixed for 7 minutes in ultrasonic vibrator and poured in the stone mold.

Preparation of group B samples:

In-Ceram powder was mixed with colloidal alumina (Nyacol AL-20, Nyacol Products, USA). 38 g In-Ceram powder, 5 ml colloidal alumina and one drop of binder were mixed in a glass

mortar for 12 minutes. The material was further mixed for 7 minutes in ultrasonic vibrator and poured in the stone mold.

The samples were pre-heated for 6 hours in the oven with the temperature rising gradually from 20°C to 120°C. During this period the stone material loses its moisture and shrinks leading to a total separation from In-Ceram core. Then the temperature was raised from 120°C to 1120°C with an increase of 10°C per minute, and the material was baked for two hours at this temperature. The core material was cooled to 400°C in the oven, then cooled down at room temperature.

Glass infiltration was performed according to the manufacturer's recommendations; the samples were subjected to 1100°C for 6 hours; then the In-Ceram glass powder/water slurry was applied on.

A special jig was prepared for three point bending tests (Figure 1).

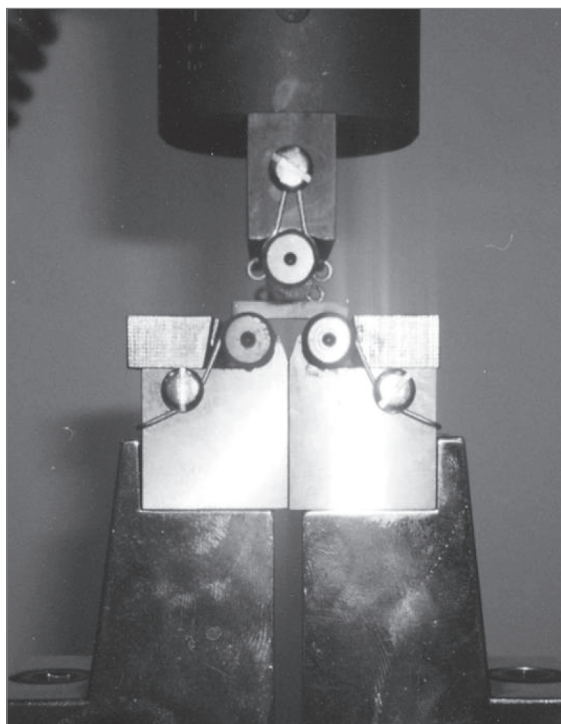


FIGURE 1

Three point bending test assembly

Three-point bending test:

The rectangular bar was placed on support 18mm apart and the test was conducted at a crosshead speed of 1mm/min by means of a universal testing machine (Instron Model1185, Instron, USA). The load applied to the test specimen and the corresponding deflection were measured until the specimen fractured. The three point bend stress were calculated by the following equations¹⁷.

$$\text{Stress } \sigma \text{ (MPa)} = \frac{3LF}{2WT^2}$$

Where

L= distance between supports (mm);

F= load (N);

W= width of specimen bar (mm);

T= thickness of specimen bar (mm);

The fracture surfaces were observed with SEM at x600, x800, x1200 and x2000 magnifications.

The data were analyzed with independent samples t-tests using a standard statistical program package (SPSS, Version Windows 6.0)

RESULTS

Descriptive statistics are summarized in Table I. The mean fracture resistance for groups A and B were 963.75 MPa and 1164 MPa, respectively and the difference between two groups were statistically significant ($t = -3.98$), ($p = 0.001$).

SEM (Scanning Electron Microscope) evaluation:

While there were microporosities in both groups before glass infiltration, lesser porosities were observed in colloidal alumina-binder group Figure (2,3,4,5,6,7,8). No porosities were observed after glass infiltration at both groups. Figure (9,10).

DISCUSSION

The aim of this study was to evaluate the effects of the use of colloidal alumina as a binder in In-Ceram core on flexural strength of the core.

TABLE I

Descriptive statistics for test groups (MPa)

	Mean	Std.Err.	Std.Dev.	Min.	Max	Median	n
Group A	963.75	25.2	79.6	779.85	1132.95	985.8	10
Group B	1164	22	70	970.5	1324.5	1140	10

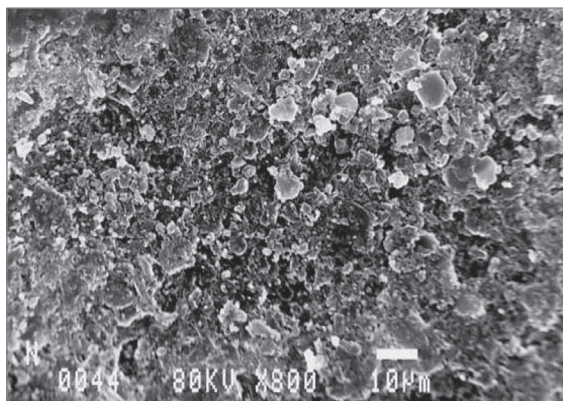


FIGURE 2

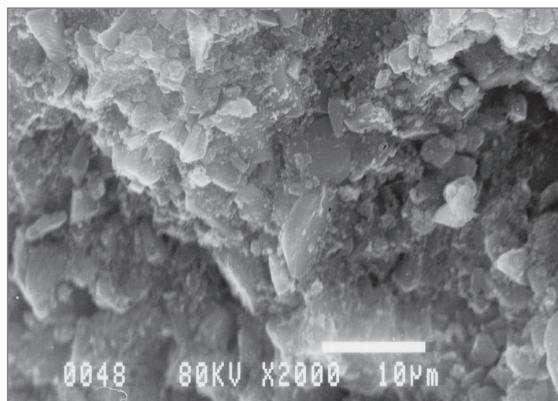
Group A before glass infiltration (x800)

FIGURE 4

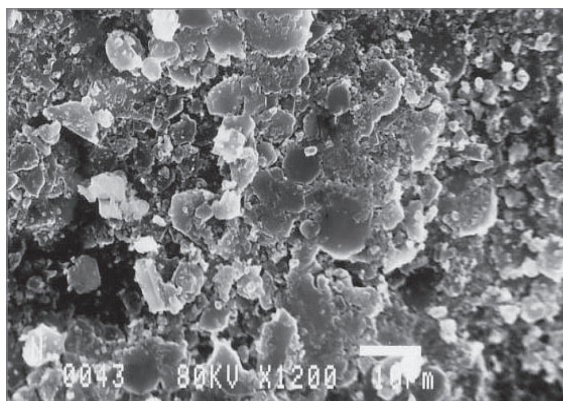
Group A before glass infiltration (x2000)

FIGURE 3

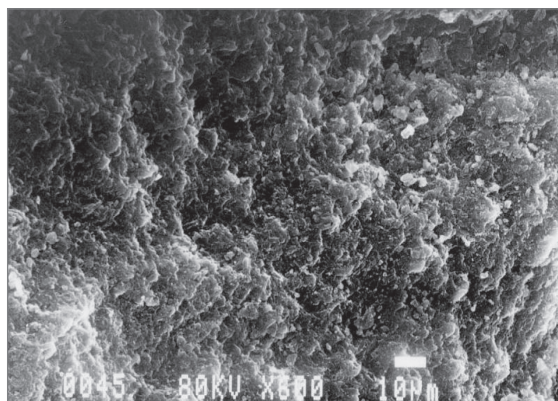
Group A before glass infiltration (x1200)

FIGURE 5

Group B before glass infiltration (x600)

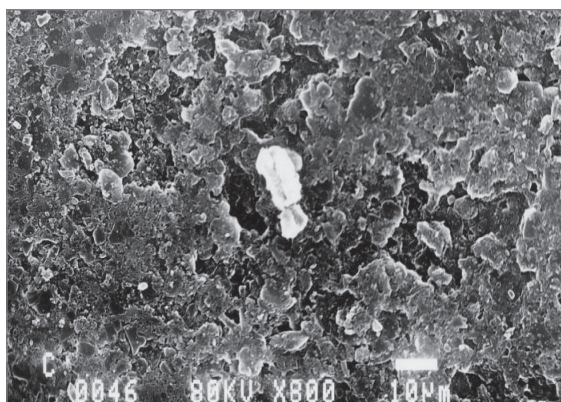


FIGURE 6

Group B before glass infiltration (x800)

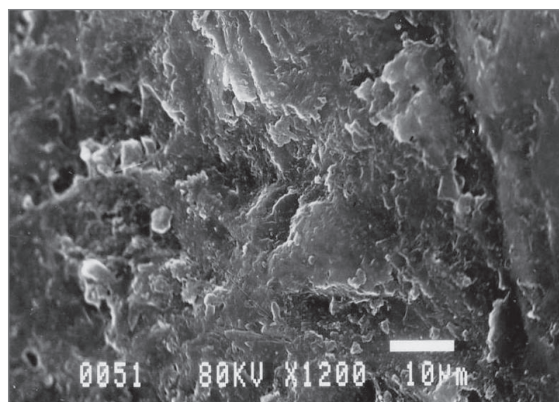


FIGURE 9

Group A after glass infiltration (x1200)

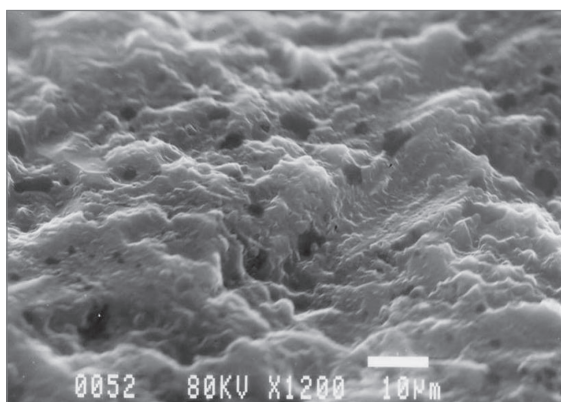


FIGURE 7

Group B before glass infiltration (x1200)

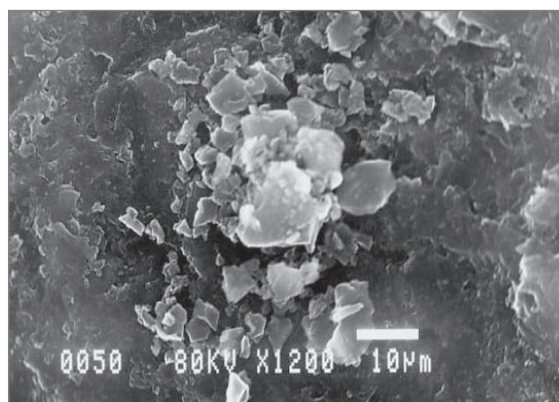


FIGURE 10

Group B after glass infiltration (x1200)

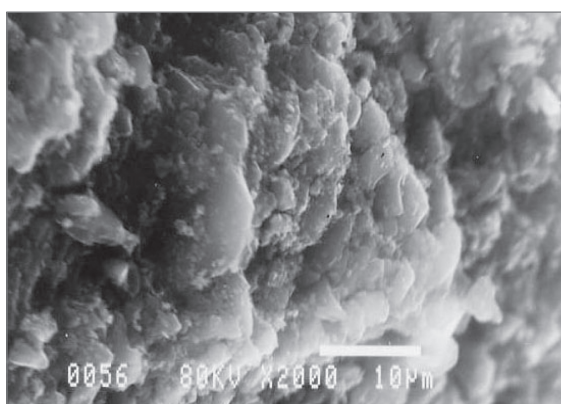


FIGURE 8

Group B before glass infiltration (x2000)

Various methods have been suggested to test the strength of dental ceramics^{18,19}, Torsion testing^{20,21}, Vickers indentation tests²², bi-axial flexural strength test, Diametrical compressive strength test²³ etc. Several authors also suggested using porcelain restorations from the standing point of establishing a more realistic test design²⁴.

The preference of a three-point bending strength test design relies on the International Standards Organisation (ISO, 1984) and American Dental Association's suggestions pointing out that this test design is reliable, appropriate

and easy for testing the ceramic materials. With this test design, the test values are easy to compare; however, it must be noted that the results can strongly be affected by the superficial fissures, cracks or defects^{24,25}.

It has been reported that with In-Ceram core porcelain crowns high flexural strength values can be observed similar to metal-ceramic restorations²⁶.

Castellani et al²⁷ investigated the diametral flexural strengths of castable glass ceramic, Hi-Ceram, In-Ceram and metal-ceramic restorations and reported mean values of 5,1 kg for castable glass ceramic, 8,4 kg for Hi-Ceram, 22,3 kg for In-Ceram and 17 kg for metal-ceramic restorations.

Grey et al²⁸ reported the mean strength of, 916 N for alumina crowns, 1609 N for full ceramic restorations with 0,7mm In-Ceram core, and 1557 N for conventional metal-ceramic restorations. Furthermore, they reported that most of the In-Ceram cores remained intact while their porcelain suprastructures were broken.

In another study²⁹, the mean three point bending values for most of full ceramic restoration systems were respectively; Vitadur-N 90 Mpa, Hi-Ceram 135 Mpa, Cerestore 145 MPa, Dicor 150 Mpa, Optec 175 MPa, IPS-Empres 200 MPa and In-Ceram 600 Mpa.

Giordano et al³⁰ reported three point bending values of 70 Mpa for feldspathic porcelain, 107 MPa for Dicor, and 236 Mpa for In-Ceram.

Pröbster et al¹⁰ reported three point bending values of 35 MPa for Vitadur-N, 70 MPa for Dicor, 170 MPa Alumina core material, 420 MPa for In-Ceram. They also concluded that by using In-Ceram core material, ten times greater strength values than conventional dental ceramic materials and four times greater strength values than alumina core material can be achieved.

Claus³¹, using 25x6x2 mm barshaped samples, reported three point bending strengths of 130 N/mm² for Vitadur-N, 160 N/mm² for Hi-Ceram, and 560 N/mm² for In-Ceram.

Bienek²⁵ reported that the porcelain materials can be classified in three groups according to their compressive strengths: Near 100 MPa (Cerec, Dicor, Duceram, IPS- Empress, Flexo-Ceram, LFG, Mirage I ve II, Optec HSP, Vitadur Alpha), near 150 MPa (Hi-Ceram, Syntho-Ceram, Vitadur-N), and In-Ceram with 222 MPa compressive strength.

The findings of these studies clearly show that the In-Ceram core material have significantly greater physical properties than other conventional or relatively recently developed ceramic materials. There are two basic theories explaining the reinforcing potential of In-Ceram core material through adequately distributing the alumina crystals into a glass matrix¹:

1. constant- strain theory
2. Limitation of microcrack propagation

It has been shown that as the microcrack length reaches 25-125 μm , instant decreases in mechanical strength occur³².

According to the constant strain theory, the alumina crystals are formed as a result of the stresses during the cooling process after the glass infiltration related to their thermal expansion coefficients. If the thermal expansion coefficient of the glass matrix is lower than that of the alumina crystals, the elasticity and strength of the resulting compound decreases³³. Thus, the thermal expansion coefficients of alumina and glass matrix must be similar in order to eliminate sudden volumetric changes that could eventually occur during the cooling process.

The limitation of microcrack propagation theory has first been suggested in 1966 by Haselman and Fulrath. The glass and alumina matrices having similar thermal expansion coefficients are thought to have a limiting effect on the dimensions of Griffith's flaws. In other words, the crack length is determined by the dimensions of the inter-alumina particle matrice.

Also in aluminous ceramics, the density of the compound significantly affects its strength^{1,34}.

One of the physical properties that colloidal alumina brings to alumina particles is the fluidity that can allow the mixture to be used in injection molding process.

Injection-molding, is a process allowing the adequate fabrication of complex geometrical structures^{35,36}.

The distribution of particles is affected by the porcelain particle-binder adhesion. The main binding force is hydrogen bonds provided by Lewis's acid-base reactions or covalent bonds, as the binding by Van der Waals forces is weak³⁷. Generally a surface active agent (Dispersant) is used to detect the bond between the powder and binder³⁸⁻³⁹⁻⁴⁰. In this study, colloidal alumina contained nitric acid as surface active agent.

Pinwil et al³⁵, investigated the effects of the atmosphere, time-temperature changes and the thicknesses of the models on the evaporation process of binders.

The materials and methods used in this study allows to provide a high Al_2O_3 content possibly explaining higher three point bending strength compared with the conventional In-Ceram method. Furthermore, the SEM evaluation revealed fewer porosities at the surface of the specimens prepared with colloidal alumina as binder.

Further in-vitro and in-vivo studies are needed to establish a good knowledge of the use of this trial method in fixed partial restorations with three or more units. This method could also be used with injection molding system in core preparation for fixed partial restorations.

CONCLUSION

Higher three point bending strength values were obtained with the group, in which colloidal alumina is used as binder. Also fewer microporosities were observed in the colloidal alumina core group.

Within the limitations of this study, the use of colloidal alumina as binder seems to be promising in achieving better flexural strength in fixed partial restorations with In-Ceram system.

REFERENCES

1. McLean JW. The science and art of dental ceramics. First ed. Quintessence Publishing Co, Chicago, 1979.
2. Jones DW. Development of dental ceramics, An historical perspective. *Dent Clin Nort Am*; 1985; 29:621-43.
3. Kelly RJ, Nishimura I, Champbell SD. Ceramics in dentistry. Historical roots and current perspectives. *J Prosthet Dent* 1996; 75:18-32.
4. Wohlwend A, Strub JR, Scharer P. Metal ceramic and all-porcelain restorations current considerations. *Int J Prosthodont* 1989; 2:13-26.
5. Hondrum SO. A review of the strength properties of dental ceramics. *J Prosthet Dent* 1992; 67:859-64.
6. Hobo S, Iwata T. A new laminate veneer technique using castable apatite ceramic material. I. Theoretical considerations. *Quintessence Int* 1985; 16:451-457.
7. Zeng K, Oden A, Rowolifle D. Flexure tests on dental ceramics. *Int J Prosthodont* 1996; 9:434-39.
8. Williamson RT, Kovarik RE, Mitchell RJ. Effect of grinding polishing, and overglazing on the flexure strength of a high-leucite feldspathic porcelain. *Int J Prosthodont* 1996; 9:30-7.
9. Geis-Gerstrofer J, Kanjantra P. Zum einfluss der prüfmethode auf die biegefestigkeit von IPS-Empress und In-Ceram. *Dtsch Zahnarzt Z* 1992; 47:618-21.
10. Pröbster L, Diehl J. Slip-casting alumina ceramics for crown and bridge restorations. *Quintessence Int* 1992; 23:1-8.
11. Sulaiman F, Chai J, Jameson LM, Wozniak WT. A comparison of the marginal fit of In-Ceram, IPS-Empress, and Procera crowns. *Int J Prosthodont* 1997; 10:478-84.
12. Claus H. VITA In-Ceram, a new system for producing aluminium oxide crown and bridge substructures. *Quintessenz Zahntechnik* 1990; 16:35-46.
13. Seghi RR, Sorensen JA, Engelman MJ, Roumanas E, Torres TJ. Flexural strength of new ceramic materials. *J Dent Research* 1990; 69:299 (Abstract).
14. Rinke S, Hüls A, Jahn L. Marginal accuracy and fracture strength of conventional and copy milled all ceramic crowns. *Int J Prosthodont* 1995; 8:303-10.
15. Shearer B, Grough MB, Setchell DJ. Influence of marginal configuration and porcelain addition on the fit of In-Ceram crowns. *Biomaterials* 1996; 17:1891-5.
16. Andrea Y, Cawley JD. Extrusion of Al_2O_3 ceramics with low organic content. *Ceram Eng Sci Proc* 1989; 10:773-783.
17. Mencik J. Strength and fracture of glass and ceramics. *Glass science and technology* 1992; 12:164-168.
18. Campbell SD, Pelletier DL, Poher RL, Giordano RA. Dimensional and formation analysis of a restorative ceramic and how it Works. *J Prosthet Dent* 1995; 74:332-40.
19. Kappert HF, Knode H, Schultheiss R. Festigkeitsverhalten der In-Ceram keramik bei mechanischer und thermischer wechsellast im kunstspeichel. *Dtsch Zahnarzt Z* 1991; 46:129-31.

20. Kern M, Fechtig T, Strub JR. Influence of water storage and thermal cycling on the fracture strength of all-porcelain resin-bonded fixed partial dentures. *J Prosthet Dent* 1994; 71:251-6.
21. Sherrill CA, O'Brein WJ. Transverse strength of aluminous and feldspathic porcelain. *J Dent Res* 1974; 53:683-90.
22. White SN, Zhao XY, Zhaokun Y, Li ZC. Cyclic mechanical fatigue of a feldspathic dental porcelain. *Int J Prosthodont* 1995; 8:413-20.
23. Seghi RR, Denry IL, Rosenstiel SF. Relative fracture toughness and hardness of new dental ceramics. *J Prosthet Dent* 1995; 74:145-150.
24. Miller A, Long J, Miller B, Cole J. Comparison of the fracture strengths of ceramometal crowns versus several all-ceramic crowns. *J Prosthet Dent* 1992; 68:38-41.
25. Bienek KW, Marx R. Die mechanische belastbarkeit neuer volkeramischer kronen-und brückenmaterialien. *Schweiz Monatsschr Zahnmed* 1994; 104:284-9.
26. Pröbster L. Compressive strength of two modern all-ceramic crowns. *Int J Prosthodont* 1992; 5:409-412.
27. Castellani D, Baccetti T, Giovannoni A, Bernardini DU. Resistance to fracture of metal ceramic and all-ceramic crowns. *Int J Prosthodont* 1994; 7:149-54.
28. Grey NJA, Piddock V, Wilson MA. In vitro comparison of conventional crowns and a new all-ceramic system. *J Dent* 1993; 21:47-51.
29. Ohyama T, Yoshinari M, Oda Y. Effects of cyclic loading on the strength of all-ceramic materials. *Int J Prosthodont* 1999; 12:28-37.
30. Giordano RA, Pelletier L, Campbell S, Rober R. Flexural strength of an infused ceramic, glass ceramic and feldspathic porcelain. *J Prosthet Dent* 1995; 73:411-8.
31. Magne P, Belser U. Esthetic improvements and in vitro testing of In-Ceram alumina and spinell ceramic. *Int J Prosthodont* 1997; 10:459-66.
32. Seghi RR, Denry IL, Rosenstiel SF. Relative fracture toughness and hardness of new dental ceramics. *J Prosthet Dent* 1995; 74:145-150.
33. Brackett SE, Leary JM, Turner KA, Jordan RD. An evaluation of porcelain strength and the effect of surface treatment. *J Prosthet Dent* 1989; 61:446-51.
34. Thompson JY, Anusavice KJ, Naman A, Morris HF. Fracture surface characterization of clinically failed all-ceramic crowns *J Dent Res* 1994; 73:1824-32.
35. Pinwill IE, Edirisinghe MJ, Bevis MJ. Development of temperature-heating rate diagrams for the pyrolytic removal of binder used for powder injection moulding. *J Materials Science* 1992; 27:4381-4388.
36. Doremus RH. Review Bioceramics. *Journal of Materials Science* 1992; 27:285-297.
37. Salamone AL, Reed JS. Extrusion Behaviour and Microstructure Development in Alumina. *American Ceramic Society* 1989; 58:1175-1178.
38. Paul LIN ST, German RM. The influence of powder loading and binder additive on the properties of alumina injection-moulding blends. *J Materials Science* 1994; 29:5367-5373.
39. Edmond H, David E. Removal of Polymethylmethacrylate binder from alumina using microwave energy. *Ceramic Engineering Science* 1993; 7:848-856.
40. Reed YS. Introduction to the principles of ceramic processing. *Ceramic Powder Science* 1988; 17:89-99.

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