DOI 10.1007/s11029-016-9551-x
Mechanics of Composite Materials, Vol. 51, No. 6, January, 2016 (Russian Original Vol. 51, No. 6, November-December, 2015)

DAMAGE OF THE INTERFACE BETWEEN AN ORTHODONTIC BRACKET AND ENAMEL – THE EFFECT OF SOME ELASTIC PROPERTIES OF THE ADHESIVE MATERIAL

B. H. Durgesh,[1*] A. A. Alkheraif,[2] M. Al Sharawy,[3] J. Varrela,[4] and P. K. Vallittu[5]

Keywords: *adhesives, debonding, shear, strain*

The aim of this study was to investigate the magnitude of debonding stress of an orthodontic bracket bonded to the enamel with resin systems having different elastic properties. For the same purpose, sixty human premolars were randomly divided into four groups according to the adhesive system used for bonding brackets: G Fix flowable resin (GFI) with Everstick NET (ESN), GFI, G Aenial Universal Flow (GAU) with ESN, and GAU. The brackets were stressed in the occlusogingival direction on a universal testing machine. The values of debonding load and displacement were determined at the point of debonding. The elastic modulus of the tested materials was determined using nanoindentation. An analysis of variance showed a significant difference in the loads required to debond the bracket among the groups tested. The GAU group had the highest elastic modulus, followed by the GFI and ESN groups. ARI (Adhesive Remnant Index) scores demonstrated more remnants of the adhesive material on the bracket surface with adhesives having a higher elastic modulus. Taking into consideration results of the present in-vitro study, it can be concluded that the incorporation of a glass-fiber-reinforced composite resin (FRC) with a low elastic modulus between the orthodontic bracket and enamel increases the debonding force and strain more than with adhesive systems having a higher elastic modulus.

---

[1] Dental Health Department, Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia
[2] Dental Health Department, Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia
[3] Bio-Medical Technology, Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia
[4] Department of Oral Development and Orthodontics, Institute of Dentistry, University of Turku, Turku and City of Turku Welfare Division, Oral Health Care, Turku, Finland
[5] Department of Biomaterials Science and Turku Clinical Biomaterials Centre-TCBC, Institute of Dentistry, University of Turku and City of Turku Welfare Division, Oral Health Care, Turku, Finland and King Saud University, Visiting Professor Program
[*] Corresponding author; e-mail: drdurgesh19@gmail.com

Russian translation published in Mekhanika Kompozitnykh Materialov, Vol. 51, No. 6, pp. 1141-1154 , November-December, 2015. Original article submitted April 20, 2015.

Introduction

Dental adhesives are claimed to be user-friendly because of the improvements achieved in the last three decades [1]. In bonding orthodontic brackets to the enamel, the conventional adhesive system comprises three separate agents: an enamel conditioner, a primer solution, and an adhesive resin [2]. To reduce the chair time, simplified, or self-etching, adhesives were developed with the advantage of simple application technique, since they are intended to combine the etching, priming, and bonding steps of the conventional etch-and-rinse adhesives in one solution [3]. Bonding results are influenced by brackets, adhesive systems, and the composite between the bracket and enamel. By reducing the number of steps during the procedure of bonding, clinicians can save time and reduce potential errors [4]. Introduction of light-curing flowable composites have had an influence on orthodontic bonding. The use of flowable composites for orthodontic bonding has met with success [4-6]. Unlike such orthodontic bonding system as Transbond XT (3M Unitek, Monrovia, California), flowable composites can be applied to an acid-etched enamel without the use of intermediate bonding resins because of their low filler loading and improved flowability, which enable the penetration of monomer resins into the microscopic irregularities of etched enamel. The adhesive interface between a bracket and enamel is based on the mechanical retention of the bracket to the bonding surface. Debonding of the bracket can occur either from the interface to the enamel or to the bracket, or at the interface material. It is well understood that the debonding stress is typically of shear type [7].

The mechanism of debonding is related to the adhesion strength, elasticity, and viscosity of the adhesive resin and thickness of the adhesive resin layer. Dental resin systems are typically made from dimethacrylate monomers and particulate fillers with light, dual, or chemical curing initiator systems. In polymerization, dimethacrylate-based resins form a highly cross-linked polymeric matrix, which is typically characterized by its brittleness related to the high elastic modulus of the resin matrix.

Glass fibers are the common choice of reinforcement for dental applications, because they adhere well to dimethacrylates by silane coupling agents [8-10]. Glass fibers have been used in different forms to strengthen dental polymers, including continuous bidirectional woven ones, unidirectional rovings, and discontinuous short-fiber reinforcements [11-15]. Fibers have been used in fixed prosthodontic appliances, in endodontic posts and cores, and in orthodontic retainers, space maintainers, and active appliances [16-22]. The bonding strength of an orthodontic bracket in the case of incorporation of a glass-fiber-reinforced composite between the bracket and enamel has also been reported [23].

However, no attention was paid to the debonding mechanism of the bracket in relation to the elastic properties of the polymeric matrix of the glass-fiber-reinforced composite. The polymeric matrix is a semi-interpenetrating polymer network (semi-IPN) system of bisphenol-A-glycidylmethacrylate (bis-GMA) and polymethymethacrylate (PMMA) [24, 25]. This system has a lower modulus of elasticity than the corresponding cross-linked polymer due to the plasticizing effect of PMMA chains in the polymer structure. No studies have been done to examine the bonding behavior of a bracket to the enamel incorporating a glass-fiber-reinforced composite with a semi-IPN polymeric matrix.

Therefore, the purpose of this study was to investigate the magnitude of debonding stress (the so-called shear bond strength) of an orthodontic bracket bonded to the enamel with resin systems having different viscoelastic properties. A special problem was to find a correlation between the elastic modulus of the interfacial resin material to the debonding force and the percentage of strain.

Materials and Methods

Sixty human premolars extracted for orthodontic purposes were used for this study. Teeth with a hypoplastic enamel, fractures or caries were excluded from the study. Each tooth was mounted vertically in a self-curing acrylic resin to have the buccal bond surface parallel to the force applied during the debonding test. Enamel surfaces were etched with a 37% phosphoric acid for 15 s, washed, and dried. Acid-etched teeth were randomly divided into four groups ($n = 15$), as follows.

Group 1 (GFI-F): Standard premolar metal brackets (Lancer Orthodontics, Milano, Italy) with a surface mesh area of 11.86 mm^2 were bonded at the center of the buccal surface of premolar teeth by using a continuous bidirectional glass-fiber weave between the tooth and bracket. The diameter and thickness of a single glass fiber of the weave was 6 and 60 µm,

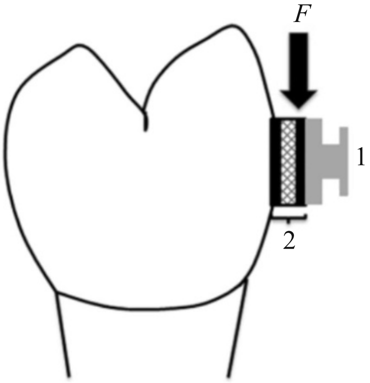

Fig. 1. Schematic representation of the bracket-tooth interface and direction of the debonding force *F* (arrow).

TABLE 1: List of the Materials Used in the Study

Name	Batch No	Composition	Manufacturer
Everstick NET	1407021	Fiber impregnated with polymethy methacrylate	Stick Tech Ltd, Finland
Stick resin	1212191	Triethyleneglycol dimethacrylate 2-dimethylaminoethylmethacrylate	
G-Fix	1302262	Particulate glass fillers, dimethacrylate monomers, phosphoric ester monomer, silica nanoparticles	GC Corporation, Tokyo, Japan
G Aenial universal flo	1404091	Urethane dimethacrylate, bisphenol A polyethoxymethacrylate, Y-methacryloxypropyltrimethoxysilane, triethyleneglycol dimethacrylate	

respectively. The resin matrix of the fiber prepreg (everStick NET, (ESN)) contained bis-GMA and PMMA, which formed a semi-IPN for the glass fibers. A glass-fiber prepreg sheet was cut to bracket size and wetted from the surface with the Stick Resin before using the prepreg. A thin layer of a flowable resin (G-FIX (GFI)) was applied to the tooth enamel, and the fibers were bonded at the center of the facial surface of the tooth, with a pressure sufficient to expel the excess adhesive, and light-cured for 5 s. A thin layer of adhesive was used to cover the fiber net, and a bracket was placed on the resin and pressed hard to expel any excess adhesive. The specimen was light-cured for 20 s on both medial and distal sides.

The light curing was performed by a hand light-curing unit (Elipar free light, 3M ESPE, Germany) with a radiance power of 1505 mW/cm^2 and a maximum wavelength between 420-540 nm (MARC System; Blue Light Analytics Inc., Halifax, Canada).

Group 2 (GFI): Brackets were bonded using GFI without fibers.

Group 3 (GAU –F): Brackets were bonded using a G Aenial Universal Flow (GAU) flowable resin composite incorporating a fiber layer, the same as for group 1.

Group 4 (GAU): Brackets were bonded using GAU without fibers.

A complete list of the materials used in the study is given in Table 1.

Debonding Test

The specimens were mounted in a universal testing machine (Instron Corporation, Canton, Massachusetts, USA), with the enamel surface parallel to a shearing rod (Fig. 1), and stretched in the occlusogingival direction at a crosshead speed of 1 mm/min. The maximum load necessary to debond the bracket was recorded. The corresponding debonding stress is called

TABLE 2. Mean and Standard Deviations of the Load (N) and Strain (%) of Tested specimens

Groups ($n = 15$)	Load, N, mean ± SD	Strain, %
GFI	74.21 ± 1.89^{A}	11.80 ± 0.91^{A}
GFI+F	90.00 ± 4.96^{B}	16.50 ± 1.77^{B}
GAU	58.82 ± 0.84^{C}	9.56 ± 0.46^{C}
GAU+F	69.26 ± 1.19^{D}	14.56 ± 0.73^{D}

Note. Different capital letters in columns mean statistical significant differences between the groups ($P < 0.05$).

the shear adhesion strength. In addition to the values of debonding load, the displacement was also recorded, and the strain was calculated.

Evaluation of Fracture Sites

Bracket bases and enamel surfaces were inspected by using a light stereomicroscope (Nikon SM2-10, Tokyo, Japan) at a 20× magnification to determine fracture sites and to establish the character of debonded surface. The sites were classified as Type 0, 1, 2, or 3 according to the Adhesive Remnant Index (ARI) [26]:

0 = No adhesive remaining on the enamel;
1 = Less than 50% of the adhesive remaining on the enamel;
2 = More than 50% of the adhesive remaining on the enamel;
3 = All adhesive remaining on the enamel with a distinct impression of bracket mesh.

Scanning Electron Microscopy (SEM) Observations

The representative micrographs of tested teeth and brackets after debonding were taken using a scanning electron microscope (Jeol JSM-5900 LV SEM, Tokyo, Japan) at an operating voltage of 10 kV and a 300× magnification.

Nanoindentation Test to Determine the Elastic Modulus

Three disk-shaped specimens of diameter 12 mm and thickness 2 mm per each resin material tested were prepared in a metallic mold and cured according to manufacturer's instructions. For the ESN specimens, fiber nets wetted with a stick resin was stacked in layers to obtain a 2-mm thickness. By using a light-curing unit, the specimens were irradiated in multiple areas to ensure optimum polymerization. Before testing, they were kept dry at room temperature for 24 h. The elastic modulus of the specimens was determined using a nanoindentation tool (Bruker, Tucson, AZ, USA) equipped with a Berkovich diamond indenter. The testing was performed at a controlled temperature of 23°C in low noise conditions with loading and unloading rates of 0.01 and 0. 02 mN/s and a 5-s rest period at the maximum load, by varying the load between 0 and 10 mN.

Statistical Analysis

The statistical software Statistical Package for Social Sciences version 22.0 (SPSS Inc., Chicago, IL) was used to analyze the experimental data obtained. An analysis of variance was employed to calculate the differences of mean values of the debonding force and strain between the groups, and multiple comparisons of the means were performed by using Tukey's multiple comparisons *post hoc* analysis at a significance level $P < 0.05$.

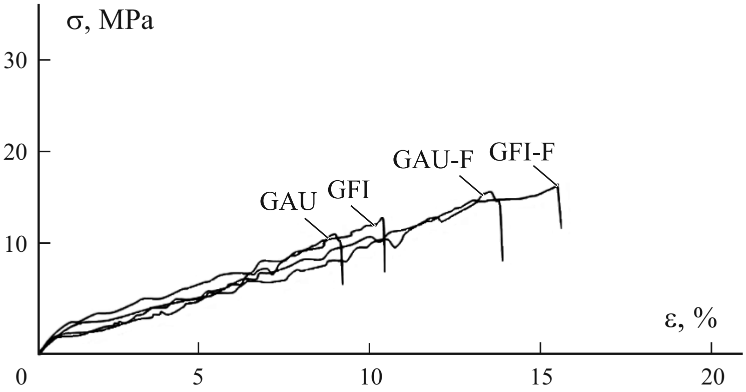

Fig. 2. Representative stress–strain curves σ–ε of debonding of brackets from the enamel surface.

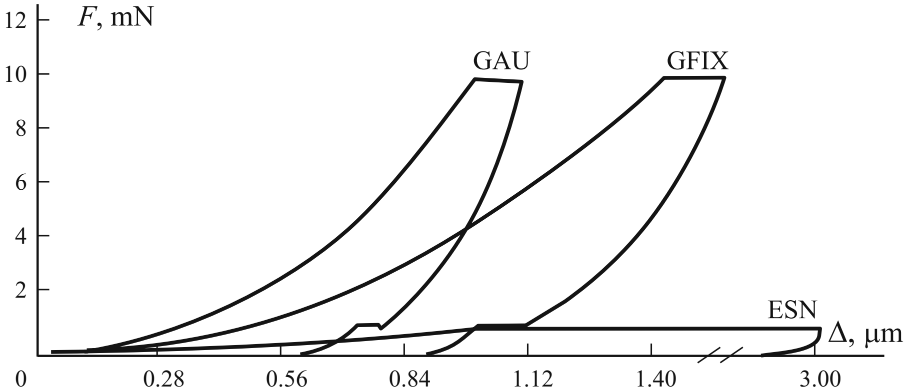

Fig. 3. Load– penetration depth graphs F–Δ of nanoindentation tests on cured adhesive resin systems.

Results

The analysis of variance showed significant differences in the values of strains and loads for all the groups tested (Table 2). The load required to debond a bracket was found to be the highest for the GFI-F group, followed by the GAU-F, GFI, and GAU groups (Fig. 2). The nanoindentation load–penetration depth curves F–Δ of the specimens tested are shown in Fig. 3. The GAU group had the highest elastic modulus, followed by GFI and ESN.

SEM images (Fig. 4) showed that the glass fibers (GFI-F and GAU-F groups) had been left on the surface of bracket, and an impression of the texture of fiber surface could be detected on the surface of adhesive resin of the bonded site of bracket. In the GFI group, the composite was found on the surface of bracket, but in the GAU group, composite remnants were detected on both surfaces.

Discussion

This study was aimed to investigate the elastic modulus of the interface material between an orthodontic bracket and a tooth, and especially the possible differences in the magnitudes of debonding force and strain to loosen the bracket. This question was found to be important for a deeper understanding of the mechanism of the failure which may occur during a orthodontic treatment and in the case the bracket is removed intentionally. Our study was also encouraged by the increasing use of light-curing flowable composite resins by orthodontists and by specialists in other fields of dentistry [4, 23, 27]. The addition of a glass-fiber-reinforced composite resin significantly increased the bond strength and the load necessary to

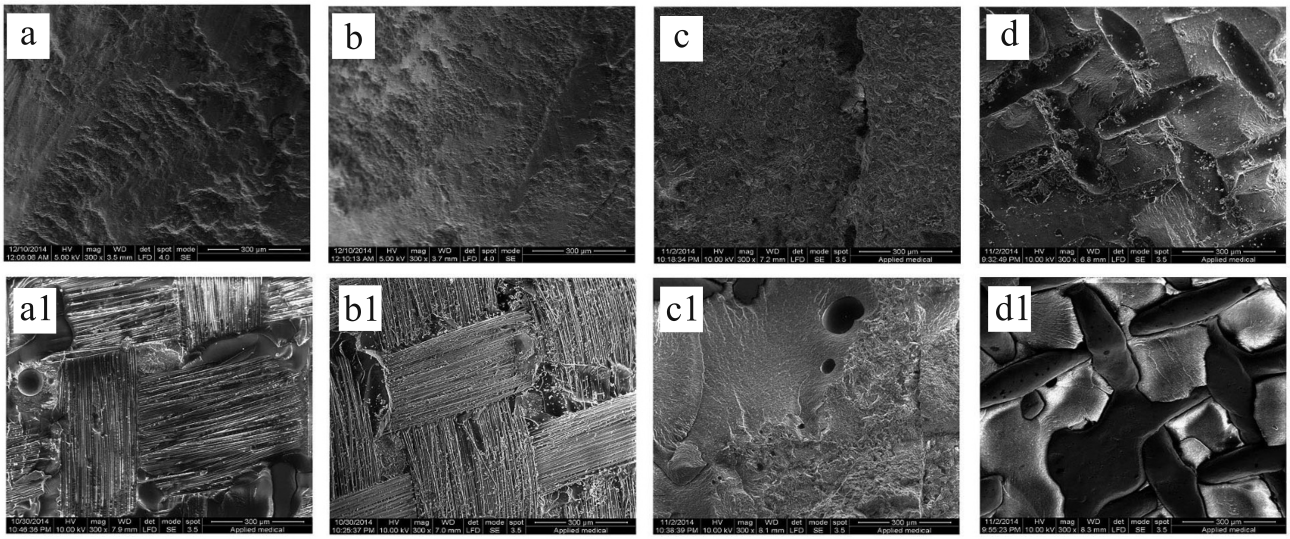

Fig. 4. SEM micrographs of representative debonded tooth (a, b, c, d) and bracket (a1, b1, c1, d1) surfaces bonded with GFI-F, GAU-F, GFI, and GAU, respectively.

debond brackets from the enamel. The composite, with a bidirectional orientation of fibers, had a thickness of 60 μm, which effected the thickness of the interface layer more than the other interface materials used in this study [28]. A shortcoming of the experimental part of this work was the fact that the thickness of the adhesive interface was not the same in all the groups investigated. However, the study followed the way of clinical work.

The results of the present study showed that the incorporation of a bidirectional glass-fiber-reinforced composite between a bracket and a tooth increased the debonding force and strain compared with those of other materials, namely the flowable GFI and GAU composite resins. The elastic modulus of the polymeric matrix of the materials also varied depending on the nanoindentation measurements performed. It should be noted that the nanoindentation measurements were performed for the polymeric matrix of the composite resin (a flowable particulate composite or a fiber-reinforced composite), which, on the average, made up half the volume of the material. Therefore, the values of elastic modulus contributed to the elastic properties of the composite material only partly. A significant correlation between the debonding force and strain and elastic modulus of the polymeric matrix of the composite located between the bracket and a tooth was found to exist. This suggests that the interface with a lower elastic modulus behaves as a stress absorber allowing a strain to develop before debonding. An analysis of ARI scores (Table 3) revealed more remnants of the adhesive material on the bracket surface with adhesives with a polymeric matrix having a low modulus of elasticity. The fracture in the groups with glass fibers occurred near the bracket surface. This fracture mode suggests less chance for the fracture of enamel during debonding. No instances of enamel fracture were reported during the debonding process. It is known that the resin matrix of the ESN glass-fiber-reinforced composite has a high viscosity before the cure and formation of a semi-IPN polymer [24]. The high viscosity is due to the presence of PMMA macromolecules in the resin of bis-GMA monomer liquid.

The values of debonding load found in the present study are in accordance with the results of previous studies [29, 30]. A bond strength of 6-8 MPa is reported to be enough for clinical orthodontic needs [31, 32]. In [29], it is stated that the shear bond strength should be less than 21 MPa to avoid the damage of enamel. Moreover, the maximum bond strength should be lower than the tensile strength of enamel, which ranges between 11-25 MPa, depending on the prismatic orientation of enamel [30]. The groups with glass fibers showed a higher bond strength than the other groups, but it was low enough to exclude any undue effect on the enamel. However, it is important to emphasize that the so-called shear bond strength tests are sensitive to any sources of inaccuracies and the values of stresses obtained cannot be considered as exact ones for the maximum shear stress necessary for debonding. Therefore, the values of shear stress cannot be used to compare results for the bond between a bracket and tooth found in different studies. Our recommendation would be to use the debonding force instead of the shear bond strength.

TABLE 3. Fracture Types According to the Adhesive Remnant Index (ARI)

Groups (*n* = 15)	ARI SCORE			
	0	1	2	3
GFI	3	4	3	5
GFI+F	1	2	4	8
GAU	3	6	6	2
GAU+F	2	3	3	7

Clinically, the results of the present study suggest that the probability of loosening of a bracket during an orthodontic treatment can be lowered by selecting an adhesive material with a low elasticity. The energy necessary to debond a bracket was high for the groups incorporating glass fibers. However, a higher debonding force does not necessarily make an intentional removal of a bracket more difficult by a bracket plier. In the clinical context, it should be remembered that the force exerted by orthodontic wires is static and long-term. Therefore, the understanding of possible viscoelastic properties of the interface of the IPN-type resin a under constant load are important when the undesired debonding of brackets under forces caused by orthodontic wires and devices are considered. Thus, an insight into the elastic and viscoelastic properties of the resin under long-term static forces would be beneficial to better understand how properties of the adhesive interface of cross-linked semi-IPN polymer systems could contribute to the bonding of brackets to the tooth surface. In addition, more information is needed to verify the degree of cure of light-curing monomer systems under brackets.

Conclusions

The present *in vitro* study showed that incorporation of a FRC with a low elastic modulus between the bracket and enamel increased the debonding force and strain before debonding more than adhesive systems with a higher elastic modulus. A correlation between the elastic modulus of the polymer matrix of the adhesive composite material tested and the debonding force of the bracket is found.

Acknowledgments. The project was financially supported by the Vice Deanship of Research Chairs, King Saud University. The investigation was carried out at the Turku Clinical Biomaterials Centre-TCBC, Institute of Dentistry, University of Turku, Finland and Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia.

REFERENCES

1. M. Ekambaram, C. K. Y. Yiu, and J. P. Matinlinna, “An overview of solvents in resin–dentin bonding,” Int. J. of Adhesion and Adhesives, **57**, 22-33 (2015).
2. S. E. Bishara, et al., “Comparison of the shear bond strength of 2 self-etch primer/adhesive systems,” Am. J. Orthod Dentofacial Orthop., **125**, No. 3, 348-350 (2004).
3. M. Peumans, et al., “Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials,” Dent. Mater., **21**, No. 9, 864-881 (2005).
4. D. B. Ryou, et al., “Use of flowable composites for orthodontic bracket bonding,” Angle Orthod., **78**, No. 6, 1105-1109 (2008).
5. M. D’Attilio, et al., “Shear bond strength, bond failure, and scanning electron microscopy analysis of a new flowable composite for orthodontic use,” Angle Orthod., **75**, No. 3, 410-415 (2005).
6. S. Tecco, et al., “A new one-step dental flowable composite for orthodontic use: an in vitro bond strength study,” Angle Orthod., **75**, No. 4, 672-677 (2005).
7. T. R. Katona, “Stresses developed during clinical debonding of stainless steel orthodontic brackets,” The Angle Orthodontist, **67**, No. 1, 39-46 (1997).

8. S. Bouillaguet, et al., “Hydrothermal and mechanical stresses degrade fiber-matrix interfacial bond strength in dental fiber-reinforced composites,” J. Biomed. Mater. Res. B, Appl. Biomater., **76**, No. 1, 98-105 (2006).
9. P. K. Vallittu, “Comparison of two different silane compounds used for improving adhesion between fibres and acrylic denture base material,” J. Oral. Rehabil., **20**, No. 5), 533-539 (1993).
10. Y. K. Lung and J. P. Matinlinna, “Silanes for adhesion promotion and surface modification,” in Silane: Chemistry, Applications and Performance, 2013, Nova Sci Publ.: Hauppauge. p. 87.
11. S.Garoushi, P. K. Vallittu, and L. V. J. Lassila, “Use of short fiber-reinforced composite with semi-interpenetrating polymer network matrix in fixed partial dentures,” J. Dent., **35**,No. 5, 403-408 (2007).
12. D. C. Smith, “Recent developments and prospects in dental polymers,” J. Prosthet. Dent., 12, No. 6, 1066-1078 (1962).
13. P. K. Vallittu, “Compositional and weave pattern analyses of glass fibers in dental polymer fiber composites,” J. Prosthodont, **7**, No. 3, 170-176 (1998).
14. T. J. Nohrstrom, P. K. Vallittu, and A. Yli-Urpo, “The effect of placement and quantity of glass fibers on the fracture resistance of interim fixed partial dentures,” Int. J. Prosthodont, **13**, No. 1, 72-78 (2000).
15. K. H. Chong and J. Chai, “Strength and mode of failure of unidirectional and bidirectional glass fiber-reinforced composite materials,” Int. J. Prosthodont, **16**, No. 2, 161-166 (2003).
16. D. Edelhoff, H. Spiekermann, and M. Yildirim, “Metal-free inlay-retained fixed partial dentures,” Quintessence Int., **32**, No. 4, 269-281 (2001).
17. T. N. Gohring, P. R. Schmidlin, and F. Lutz, “Two-year clinical and SEM evaluation of glass-fiber-reinforced inlay fixed partial dentures.” Am. J. Dent., **15**, No. 1, 35-40 (2002).
18. C. Kolbeck, et al., “In vitro examination of the fracture strength of 3 different fiber-reinforced composite and 1 all-ceramic posterior inlay fixed partial denture systems,” J. Prosthodont., 11, No. 4, 248-253 (2002).
19. C. Monaco, et al., “Clinical evaluation of fiber-reinforced composite inlay FPDs,” Int. J. Prosthodont., **16**, No. 3, 319-325 (2003).
20. Petersen, R.C., Discontinuous fiber-reinforced composites above critical length. J. Dent. Res., **84**, No. 4, 365-370 (2005).
21. P. Valyi, et al., “Effect of occlusal therapy with FRC splint on periodontal parameters in maintenance phase,” Fogorv Sz, **98**,No. 4, 159-163 (2005).
22. V. Cacciafesta, et al., “Flexural strengths of fiber-reinforced composites polymerized with conventional light-curing and additional postcuring,” Am. J. Orthod. Dentofacial Orthop., **132**, No. 4, 524-527 (2007).
23. A. Scribante, V. Cacciafesta, and M. F. Sfondrini, “Effect of various adhesive systems on the shear bond strength of fiber-reinforced composite,” Am. J. Orthod. Dentofacial Orthop., **130**, No. 2, 224-227 (2006).
24. T. M. Lastumaki, T. T. Kallio, and P. K. Vallittu, “The bond strength of light-curing composite resin to finally polymerized and aged glass fiber-reinforced composite substrate.” Biomaterials, **23**, No. 23, 4533-4539 (2002).
25. P. K. Vallittu, “Interpenetrating polymer Networks (IPNs) in dental polymers and composites,” J. of Adhesion Sci. and Technol., **23**, No. 7-8, 961-972 (2009).
26. J. Artun and S. Bergland, “Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment,” Am. J. Orthod., **85**, No. 4, 333-340 (1984).
27. B. Pick, et al., “Are flowable resin-based composites a reliable material for metal orthodontic bracket bonding?” J. Contemp. Dent. Pract., **11**, No. 4, E017-E024 (2010).
28. M. Shinya, et al., “Enhanced degree of monomer conversion of orthodontic adhesives using a glass-fiber layer under the bracket,” Angle Orthod., **79**, No. 3, 546-550 (2009).
29. G. V. Newman, et al., “Adhesion promoters, their effect on the bond strength of metal brackets,” Am. J. Orthod. Dentofacial Orthop., **108**, No. 3, 237-241 (1995).
30. R. M., Carvalho, et al., “Effects of prism orientation on tensile strength of enamel,” J. Adhes. Dent., **2**, No. 4, 251-257 (2000).
31. I. R., Reynolds, “A review of direct orthodontic bonding,” Br. J. Orthodont., **2**, 171-178 (1975).
32. M. D. Turgut, et al., “Comparison of shear bond strengths of orthodontic brackets bonded with flowable composites,” Dent. Mater. J., **30**, No. 1, 66-71 (2011).