



Research

Evaluation of physical properties of esthetic brackets after clinical use: Study in situ

Matheus Melo Pithon^{a,*}, Daniel Santos Fonseca Figueiredo^b, Dauro Douglas Oliveira^c, Rogerio Lacerda dos Santos^d

^a Professor, Southwest Bahia State University, Department of Health, Bahia, Brazil

^b Former Orthodontic Resident, Pontifical Catholic University of Minas Gerais, Department of Orthodontics, Belo Horizonte, Brazil

^c Program Director of Orthodontics, Pontifical Catholic University of Minas Gerais, Department of Orthodontics, Belo Horizonte, Brazil

^d Professor of Health and Technology, Rural Center at the Federal University of Campina Grande, Department of Health, Patos, Brazil

ARTICLE INFO

Article history:

Received 20 January 2013

Received in revised form

17 June 2013

Accepted 15 July 2013

Keywords:

Biodegradation

Bracket

Orthodontics

ABSTRACT

Aim: The aim of this study was to investigate the surface morphology, dimensional stability, and frictional behavior of ceramic brackets with metal-inserted slots after different intervals of intraoral use.

Methods: Eighty-eight brackets were evaluated. The sample was divided into four groups ($n = 22$ per group): group C (control, as received from the manufacturer) and groups T12, T24, and T36 (brackets recovered after 12, 24, and 36 months of treatment, respectively). Surface morphology was analyzed with optical and scanning electron microscopy. Dimensional stability was verified with a measuring microscope, and sliding resistance on 0.019×0.025 -in stainless steel wires was evaluated using a universal testing machine.

Results: Signs of corrosion and wear occurred gradually from the 12th to the 36th month, being more significant at the metal slot base and at the porcelain/metal junction. The depth of the slot and the internal height between the tie-wings increased after clinical use, showing a significant difference at 36 months ($P < 0.05$). There was a progressive increase in the coefficient of friction versus time of clinical use and a maximum increase of 22% after 36 months ($P < 0.05$).

Conclusions: Ceramic brackets with metal-inserted slots had significant changes in physical properties during clinical use. The dimensional changes encountered were small and appeared clinically nonsignificant. However, the progressive increase in the coefficient of friction must be taken into consideration because it may compromise the clinical performance of the appliance.

© 2013 World Federation of Orthodontists.

1. Introduction

With an increasing number of adult patients referred to orthodontic care in recent years [1], the demand for more esthetic appliances that would minimally compromise patients' social and professional lives has increased. Therefore, the acceptability and use of esthetic brackets has been consistently increasing since their introduction into orthodontics [2]. However, the long-term biodegradation of these materials in the oral environment is unknown. There is a lack of studies evaluating how the biodegradation of esthetic brackets affects their clinical performance.

The multifaceted intraoral environment cannot be ideally simulated in vitro with the currently available research

methodologies [3]. Variations in temperature and pH caused by diet, food decomposition, cell debris, and oral flora and their byproducts could affect the properties of some biomaterials [4]. Orthodontic brackets are constantly under multiaxial loads arising from wire insertion and action, as well as from masticatory forces [3]. Some studies have evaluated the intraoral aging pattern of orthodontic materials, the resultant surface alterations, and possible morphologic changes and variations on mechanical properties [4–7].

Nevertheless, in the majority of these studies [5–8], investigation was limited to the changes in the properties of the orthodontic wires, such as the increase in the coefficient of friction after intraoral use [5,8]. However, wires are susceptible to cleaning methods capable of decreasing friction [8] and are frequently replaced during orthodontic therapy. Conversely, brackets are usually maintained throughout the entire treatment. Therefore, it seems important to better understand the long-term changes on the physical properties of orthodontic brackets.

Ceramic brackets with metal-inserted slots were developed to combine the frictional properties of stainless steel with the good

* Corresponding author: Av. Otávio Santos, 395, sala 705, Centro Odontomédico Dr. Altamirando da Costa Lima, Bairro Recreio, CEP 45020-750, Vitória da Conquista, Bahia, Brazil.

E-mail address: matheuspithon@gmail.com (M.M. Pithon).

esthetic characteristics of ceramics and their as-received, out-of-the-box mechanical properties are well known [9,10]. Nevertheless, the alterations that these brackets undergo during treatment and their impact on clinical performance are mostly unknown. Thus, the aim of this study was to investigate the surface morphology, dimensional stability, and frictional behavior of ceramic brackets with metal-inserted slots retrieved after different intervals of intraoral use.

2. Materials and methods

The sample consisted of ceramic brackets with metal-inserted slots (Clarity, 3 M/Unitek, Monrovia, CA) that were exposed to the intraoral environment during the orthodontic therapy of 15 patients (mean age 22 years and 6 months) treated at the same private practice. A total of 90 premolar and canine brackets from both maxillary and mandibular arches were selected after use (12, 24, and 36 months; $n = 30$ per group). The slot size used was 0.022×0.028 in (0.559×0.711 mm), as informed by the manufacturer. All patients received instructions on oral hygiene of the orthodontic appliance before installation and every 3 months thereafter. The brackets were carefully debonded with bracket-removing pliers, with force applied only to the bracket base, and were kept in receptacles filled with distilled water. They were brushed with an electric brush for 10 seconds and rinsed with distilled water to remove any loosely attached debris. The samples were then kept in self-sealed sterilizing packs until testing. The months of intraoral bracket use were precisely registered, and a group of brackets as received from the manufacturer were used as control samples.

The sample was divided into four groups, as follows: group C (control, as received from the manufacturer), group T12 (brackets after 12 months of treatment), group T24 (24 months), and group T36 (36 months). Throughout the orthodontic therapy of all 15 patients, the edgewise technique was used, with mechanical sliding. Twist-flex wires (0.018 in, 3 M/Unitek) and stainless steel wires (0.016, 0.018, 0.020, 0.018×0.025 , and 0.019×0.025 in, 3 M/Unitek) were used for a period that ranged from 1 to 7 months for each type and thickness of wire. Wires were ligated with either elastomeric or steel ligatures, as needed.

An optical reflective light microscope (BX40, Olympus, Hamburg, Germany) was used to verify the surface morphology of all 90 recovered brackets. Brackets were evaluated using images acquired in a bright field at various magnifications (50–200 \times). After the exclusion of brackets with deformation in the metal slot, signs of wear, or plastic deformation, 66 retrieved brackets remained ($n = 22$ per group, and $n = 22$ controls) and were effectively evaluated in this study.

The micromorphologic characteristics of the slot surfaces were analyzed with a scanning electron microscope (SSX-550, Shimadzu, Kyoto, Japan). The brackets were evaluated after different intervals of exposure to the intraoral environment and compared to controls. Backscattered electron images were acquired at various magnifications (20–2000 \times).

A measuring microscope (Model MM-40, Nikon Corporation, Tokyo, Japan) was used to evaluate the dimensions of all 66 retrieved brackets and their as-received counterparts. The internal height between the tie-wings (occlusal and cervical) and the slot depth were measured in all samples. To evaluate the internal depth, the mean of the measurements of the right and left tie-wings was analyzed. The same operator performed all measurements using a holder with a 0.0215×0.028 -in wire, which was used to position the slots of the brackets perpendicular to the microscope table. To evaluate the method error, the measurements of 12 specimens were repeated after a 1-week interval. The systematic error was evaluated using a paired *t* test, and random error was calculated

from the Dahlberg formula. No systematic error of measurements was observed ($P = 0.43$). The average random error was 0.001, which is considered acceptable.

A sliding resistance analysis with stainless steel wires was conducted on all brackets in a universal testing machine (AME-2Kn, Oswaldo Filizola, São Paulo, Brazil). Test specimens were obtained by bonding the brackets with a cyanoacrylate adhesive to a $4 \times 15 \times 50$ -mm acrylic plate with a holder in a standardized way. This guaranteed that the brackets' slots remained parallel to the vertical axis of the testing machine. Stainless steel wire segments (3 M/Unitek) with a cross-section of 0.019×0.025 in (0.4826×0.635 mm) and a length of 11.5 cm were used. Before testing, the wires were cleaned with 70% alcohol to remove oily substances or impurities that could interfere with the results. They were ligated in their middle portion to the brackets with 3.0-mm elastomeric ligatures (Mini-StiK, 3 M/Unitek) immediately before testing to standardize the ligation force. Each wire segment was used only once.

Test specimens were mounted in the device and assembled in the test machine. A 300-gf weight was attached to the lower extremity of the wire to keep it under tension. The wire was pulled along the bracket at a rate of 5 mm/min for 1 minute. The force levels were registered by a 10-kgf load cell. The sliding resistance was calculated by averaging the forces registered between the 1st and 5th millimeters of displacement, irrespective of the initial static friction. For means of comparison among the different time intervals evaluated, the percentage differences in the sliding resistance between the retrieved brackets (T12, T24, and T36) and the control brackets were calculated with the following equation: $\text{DifSR}(\%) = [(\text{SR}_{\text{retrieved}} \times \text{SR}_{\text{as received}}) / \text{SR}_{\text{as received}}] \times 100$, where SR is sliding resistance and *DifSR*(%) is the percentage difference in sliding resistance.

2.1. Statistical analysis

The Kolmogorov-Smirnov and Levene tests were used to assess the normality of the data distribution and the homogeneity of variance, respectively. The data showed non-normal distribution, and only the friction of the control brackets had a homogeneous variance. The internal heights of the tie-wings were compared among the different treatment intervals using ANOVA and the post hoc test of Games-Howell. The effects of treatment on the physical properties of the brackets, such as slot depth and friction, were compared among the different treatment intervals using the Mann-Whitney *U* test. The level of significance for all statistical tests was predetermined at 5%.

3. Results and discussion

The results of the morphologic analysis indicated a trend toward an increase in the internal height of the tie-wings between the brackets after clinical use. There were no statistically significant differences after 12 and 24 months of intraoral use. However, there was a statistically significant difference in cervical internal height (Table 1) between the T36 specimens and the controls ($P < 0.05$).

Regarding the depth of the slot, there were statistically significant differences between the brackets in the T36 group and those in the control group ($P < 0.05$). Conversely, no significant differences were registered among the samples in groups C, T12, and T24 ($P < 0.05$). These results indicated an increase in the slot depth after longer periods of clinical use. This increase was similar to those observed in the cervical internal height after 36 months of intraoral use (Tables 1 and 2). Conversely, the occlusal internal height showed minimum variation at the different time intervals tested (Table 2).

Table 1
Internal height between the tie-wings, by interval of intraoral use (n = 22)

Time interval, months	Internal height, mean, mm	SD
Occlusal		
As received	0.594 ^{a,c}	0.011
Retrieved		
12	0.596 ^{a,c}	0.014
24	0.600 ^{a,c}	0.019
36	0.605 ^a	0.018
Cervical		
As received	0.567 ^b	0.014
Retrieved		
12	0.571 ^{b,c}	0.017
24	0.577 ^{b,c}	0.020
36	0.586 ^c	0.024

Same letters mean no statistically significant difference ($P > 0.05$) (ANOVA and the Games-Howell post hoc test).

The T36 group showed a 22.2% increase in the coefficient of friction compared with the control samples. This amount of change was statistically significant ($P < 0.05$). Although T12 and T24 presented a numeric increase in the coefficient of friction compared with the control brackets, the results were not statistically significant (Table 3). The mean percentage increases in friction coefficients of the T12 and T24 brackets were 6.8% and 8.3%, respectively.

Gradual changes in surface morphology were registered from the 12th to the 36th month. Corrosion, wear, and debris deposition were observed in the retrieved brackets. Signs of corrosion and wear were more intense in the base of the inserted metal slot (Fig. 1A) and in the porcelain/metal junction (Fig. 1B). However, the deposition of debris and precipitated biofilm reached different extensions (Fig. 1C), varying even in brackets retrieved from the same patient.

The backscattered electron images showed a dark surface that indicated the presence of precipitated biofilm in some areas (Fig. 1D) in contrast to the bright surface of the slot alloy. These areas suggest the presence of elements with low atomic numbers, such as carbon, oxygen, and calcium, consistent with crystalline particle formation, and other areas of bright surface that may be related to silica, aluminum, barium, iron, and silver (Fig. 1E), as described by other investigators [4,11,12].

The metal slot surface of the T24 (Fig. 2E and F) and T36 brackets showed significant roughness and the presence of crevices and gaps. These features were absent or negligible in brackets retrieved after 12 months of intraoral use (Fig. 2C and D) as well as in the control specimens (Fig. 2A and B). The associations between precipitated biofilm, narrow grooves, and gaps at the metal slot surface were more extensive in the T36 samples (Fig. 2G and H).

Some properties of the orthodontic materials as received from the manufacturers have already been extensively described in the literature, such as the friction coefficient [9,10,13–16]. However, variations in temperature and pH caused by diet, food debris

Table 2
Slot depth by interval of intraoral use (n = 22)

Time interval, months	Slot depth, mm			
	Mean	SD	Median	IQR
As received	0.715 ^a	0.018	717	21
Retrieved				
12	0.719 ^a	0.025	723	32
24	0.728 ^a	0.022	729	36
36	0.749 ^b	0.029	753	39

IQR, interquartile range.

Same letters mean no statistically significant difference ($P > 0.05$) (Mann-Whitney U test).

Table 3
Friction by interval of intraoral use (n = 22)

Time interval, months	Slot depth, mm				
	Mean	SD	Median	IQR	Increase
As received	81.60 ^a	3.06	82	5.6	–
Retrieved					
12	87.20 ^a	3.51	89	6.2	6.8
24	93.08 ^a	3.92	91	6.8	13.9
36	99.71 ^b	4.29	97	8.0	22.2

IQR, interquartile range.

Same letters mean no statistically significant difference ($P > 0.05$) (Mann-Whitney U test).

decomposition, and oral flora and their byproducts may cause biodegradation of these materials, modify some physical properties, and compromise clinical performance [3,5].

The synergistic action of these biological factors may significantly alter the surface integrity of stainless steel brackets [3,4]. However, there are few studies reporting on the changes of the physical properties of esthetic brackets [11], especially those with metal-inserted slots.

In this study, the evaluated brackets showed a trend of increasing the internal height between the tie-wings according to the duration of clinical use. However, the only significant difference was in cervical internal height between group T36 and controls. There was an increase of 3% (0.019 mm) in this variable, whereas the occlusal internal height was increased 1.85% (0.011 mm). These findings are similar to those found with metal brackets, which showed variations of 2.5% (0.029 mm) in cervical internal height and 1.9% (0.022 mm) in occlusal internal height [4].

There was also a trend of increasing the slot depth versus duration of clinical use. However, the only statistically significant difference was between group T36 and controls. This dimensional increase was of 3% (0.00094 in, or 0.024 mm). Although statistically significant, this change was small and appears to have been clinically nonsignificant. These findings are similar to those reported by Gkantidis et al. [11].

Proper dimensional stability of ceramic brackets may be due to the brittleness of the material, which does not allow significant deformation before fracturing [17]. Structural deformation is more relevant when plastic brackets are used [11]. Intraoral aging reduced the hardness of plastic brackets compared with that of their out-of-the-box counterparts [18]. In the present study, to eliminate any possible influence of the method of debonding on the values of the internal measurements of the bracket slot, debonding was accomplished by carefully applying force only to the bracket base, thus preserving the area of interest.

This study demonstrated an increase in the friction coefficient versus time and surface alterations. Friction progressively increased from T12 to T36, reaching a maximum change of 22.2%. In a similar study that evaluated the intraoral aging of different brands of metal brackets, an increase in friction coefficient of up to 18% was reported [4]. In this context, similar percentages of change in coefficient of friction were observed with metallic or ceramic brackets with metal-inserted slots. This phenomenon appears to be caused by the increased surface roughness arising from the presence of grooves, gaps, and crevices and the deposition of debris during their intraoral use [3,5,19]. The small increase in friction coefficient found in T12 and T24 may still be clinically relevant, because in the early stages of the orthodontic treatment, low levels of sliding resistance are desirable to increase alignment and space-closure efficiency [20].

Previous studies [5,8] reported that stainless steel wires presented a 20.8% increase in frictional force after 8 weeks of intraoral

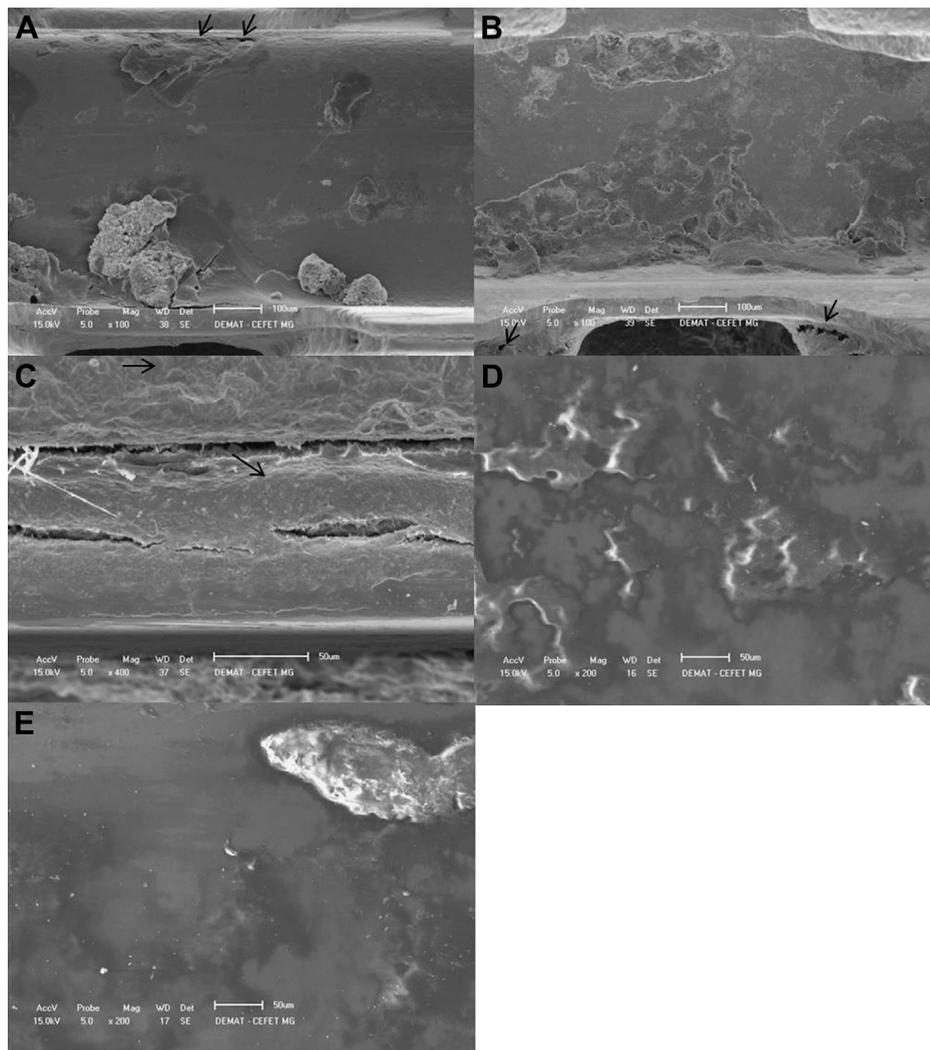


Fig. 1. Scanning electron microscopy of recovered slot brackets. (A) (Group T36) Areas of corrosion and deposition of debris (arrows) (original magnification, 100 \times). (B) (Group T24) Junction porcelain/metal (arrows) (original magnification, 100 \times). (C) (Group T36) Cracks and debris deposition in different extensions (arrows) (original magnification, 400 \times). (D) (Group T24) Dark areas biofilm precipitate (original magnification, 200 \times). (E) (Group T36) Bright surface area related to the presence of metallic elements and debris (original magnification, 200 \times).

use, and increased friction also was observed with nickel-titanium [21]. Thus, during orthodontic therapy, an orthodontist should expect an increase in friction on both bracket and wire surfaces. Orthodontic tooth movement occurs only when the applied forces overcome the friction at the bracket–wire interface [22].

Therefore, it is important to know the force levels of all systems because if the frictional forces are higher than the applied force, the efficiency of the system is affected [23]. Part of the increased friction registered on the orthodontic wires after their clinical use can be eliminated with simple cleaning methods [8]. However, a practical way to prevent this increase in the orthodontic brackets remains unknown.

Clinical use caused changes in the surfaces of the brackets, as seen in scanning electron microscope images. The as-received brackets already presented some surface irregularities, as found by Gkantidis et al. [11]. The brackets exposed to longer durations of intraoral use showed greater signs of corrosion, wear, and debris deposition [3]. The presence of precipitated biofilm found on retrieved ceramic brackets was also reported with metal brackets [4], orthodontic wires [5,6], and intraoral headgear components [12].

Certain areas of the metal-inserted slot surface suggest the presence of elements with low atomic numbers, such as carbon, oxygen, calcium, phosphorus, consistent with the formation of crystalline particles, and other areas of bright surface may be related to silica, barium, aluminum, iron, and silver, as reported by Regis et al. [4], Gkantidis, et al. [11], and Eliades et al. [12]. This calcification is a common finding in materials used in the intraoral cavity [3]. Oxygen, aluminum, and carbon are primary constituents of ceramic brackets, which explains their presence [11]. The presence of silica and barium might be attributed to the contamination of the slot by adhesives or composite resins [24]. According to Regis et al. [4], although mass transfer can occur with the sliding of a metallic surface, the silver-containing incrustations found on retrieved brackets could not be attributed to any clinical finding. The calcium and aluminum inclusions are strongly related to corrosion [4,11].

The integuments found in this study were similar to the results of studies [4] assessing the biodegradation of metal brackets, which seems to indicate that the integuments deposited on the surface of the brackets are related to the environment in which they are used and not only to the type of bracket material used.

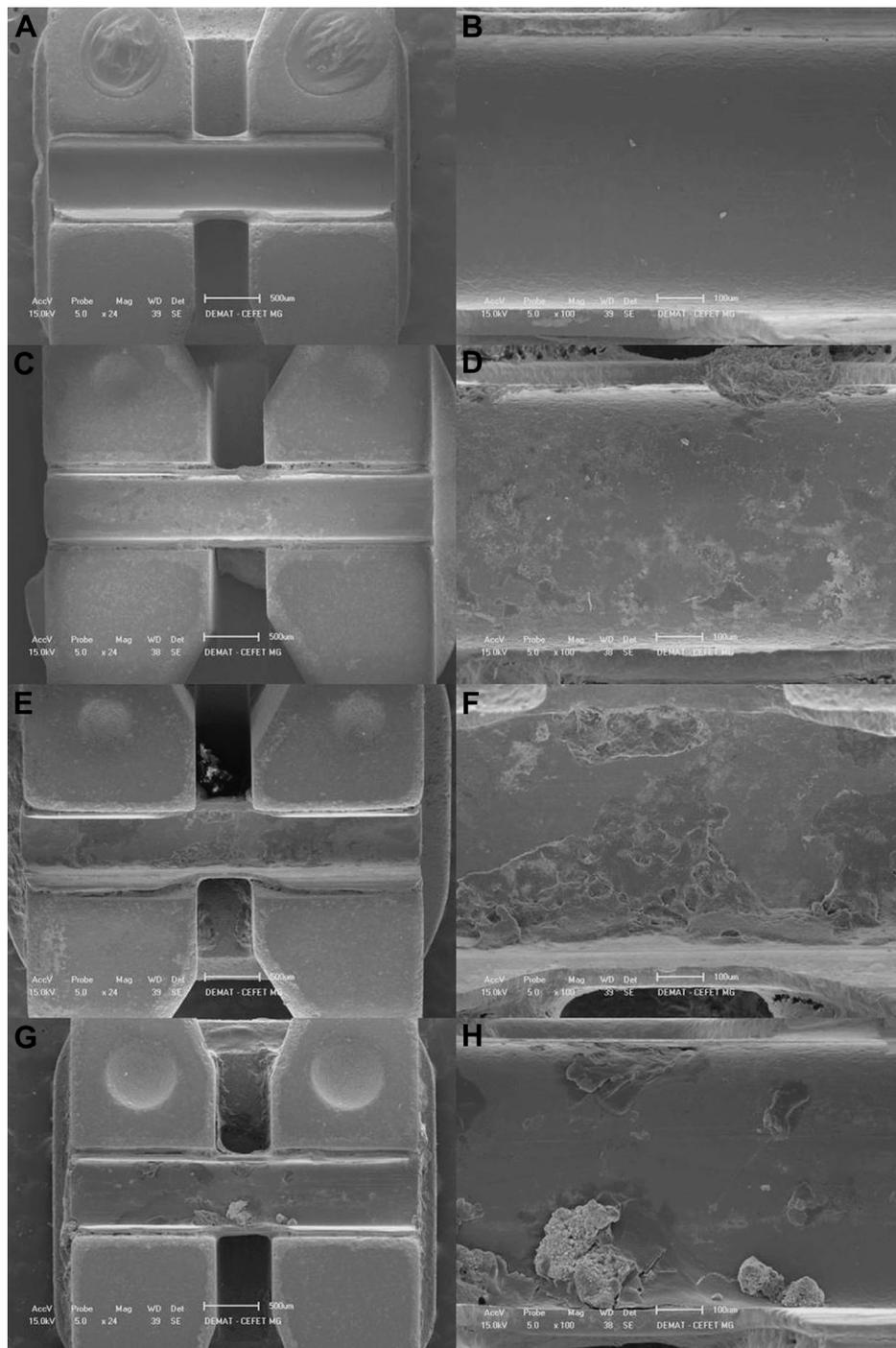


Fig. 2. Scanning electron microscopy of recovered and control brackets. (A) Group (C) Bracket as received (original magnification, 24 \times). (B) Group (C) Slot of a bracket as received (original magnification, 100 \times). (C) Group (T12) Bracket recovered after 12 months of treatment (original magnification, 24 \times). (D) Group (T12) A slot bracket recovered after 12 months, with few superficial changes and debris (original magnification, 100 \times). (E) (Group T24) Bracket recovered after 24 months of treatment (original magnification, 24 \times). (F) (Group T24) A slot bracket retrieved after 24 months, with alterations of surface and greater accumulation of debris (original magnification, 100 \times). (G) (Group T36) Bracket recovered after 36 months of treatment (original magnification, 24 \times). (H) (Group T36) Slot of a bracket recovered after 36 months, with the presence of debris, grooves, and gaps most significant (original magnification, 100 \times).

In general, the clinical use of ceramic brackets with a metal-inserted slot influenced the variables evaluated in this study. Surface morphology, dimensional stability, and resistance to sliding increased with the duration of intraoral exposure. However, the changes in dimensional stability appeared to have been clinically nonsignificant. The increase in the coefficient of friction may have

important clinical significance considering the total duration of a long-term orthodontic treatment. Still, the percentage of friction increase was similar to that with retrieved metallic brackets [4]. The impact of biological and physical properties on the clinical performance of these orthodontic materials must be further investigated in other studies.

4. Conclusions

Ceramic brackets with metal-inserted slots undergo significant changes in their physical properties during clinical use. The dimensional changes observed were small and appeared to have been clinically nonsignificant. However, the coefficient of friction must be taken into consideration in treatment durations of more than 36 months, which could compromise the clinical performance of the appliance.

References

- [1] Kokich VG. Adult orthodontics in the 21st century: guidelines for achieving successful results. *World J Orthod* 2005;6(Suppl.):14–23.
- [2] Rosvall MD, Fields HW, Ziuchkovski J, Rosenstiel SF, Johnston WM. Attractiveness, acceptability, and value of orthodontic appliances. *Am J Orthod Dentofacial Orthop* 2009;135:276.e1–276.e12 discussion 276–7.
- [3] Eliades T, Bourauel C. Intraoral aging of orthodontic materials: the picture we miss and its clinical relevance. *Am J Orthod Dentofacial Orthop* 2005;127:403–12.
- [4] Regis Jr S, Soares P, Camargo ES, et al. Biodegradation of orthodontic metallic brackets and associated implications for friction. *Am J Orthod Dentofacial Orthop* 2011;140:501–9.
- [5] Marques IS, Araujo AM, Gurgel JA, Normando D. Debris, roughness and friction of stainless steel archwires following clinical use. *Angle Orthod* 2010;80:521–7.
- [6] Eliades T, Eliades G, Athanasiou AE, Bradley TG. Surface characterization of retrieved niti orthodontic archwires. *Eur J Orthod* 2000;22:317–26.
- [7] Edie JW, Andreasen GF, Zaytoun MP. Surface corrosion of nitinol and stainless steel under clinical conditions. *Angle Orthod* 1981;51:319–24.
- [8] Normando D, Araujo AM, Marques ID, Barroso Tavares Dias CG, Miguel JA. Archwire cleaning after intraoral ageing: the effects on debris, roughness, and friction. *Eur J Orthod* 2013;35:223–9.
- [9] Cacciafesta V, Sfondrini MF, Scribante A, Klersy C, Auricchio F. Evaluation of friction of conventional and metal-insert ceramic brackets in various bracket-archwire combinations. *Am J Orthod Dentofacial Orthop* 2003;124:403–9.
- [10] Nishio C, da Motta AF, Elias CN, Mucha JN. In vitro evaluation of frictional forces between archwires and ceramic brackets. *Am J Orthod Dentofacial Orthop* 2004;125:56–64.
- [11] Kkantidis N, Zinelis S, Karamolegkou M, Eliades T, Topouzelis N. Comparative assessment of clinical performance of esthetic bracket materials. *Angle Orthod* 2012;82:691–7.
- [12] Eliades T, Eliades G, Watts DC. Intraoral aging of the inner headgear component: a potential biocompatibility concern? *Am J Orthod Dentofacial Orthop* 2001;119:300–6.
- [13] Bednar JR, Gruendeman GW, Sandrik JL. A comparative study of frictional forces between orthodontic brackets and arch wires. *Am J Orthod Dentofacial Orthop* 1991;100:513–22.
- [14] Tecco S, Di Iorio D, Cordasco G, Verrocchi I, Festa F. An in vitro investigation of the influence of self-ligating brackets, low friction ligatures, and archwire on frictional resistance. *Eur J Orthod* 2007;29:390–7.
- [15] Drescher D, Bourauel C, Schumacher HA. Frictional forces between bracket and arch wire. *Am J Orthod Dentofacial Orthop* 1989;96:397–404.
- [16] Kusy RP, Whitley JQ. Coefficients of friction for arch wires in stainless steel and polycrystalline alumina bracket slots. I. The dry state. *Am J Orthod Dentofacial Orthop* 1990;98:300–12.
- [17] Eliades T, Eliades G, Brantley WA. Orthodontic brackets. In: Brantley WA, Eliades T, editors. *Orthodontic materials: scientific and clinical aspects*. New York, NY: Thieme; 2001. p. 143–72.
- [18] Eliades T, Gioka C, Zinelis S, Eliades G, Makou M. Plastic brackets: hardness and associated clinical implications. *World J Orthod* 2004;5:62–6.
- [19] Doshi UH, Bhad-Patil WA. Static frictional force and surface roughness of various bracket and wire combinations. *Am J Orthod Dentofacial Orthop* 2011;139:74–9.
- [20] Harradine NW. Self-ligating brackets: where are we now? *J Orthod* 2003;30:262–73.
- [21] Wichelhaus A, Geserick M, Hibst R, Sander FG. The effect of surface treatment and clinical use on friction in NiTi orthodontic wires. *Dent Mater* 2005;21:938–45.
- [22] Rossouw PE. Friction: an overview. *Semin Orthod* 2003;9:218–22.
- [23] Kuramae M. Evaluation of frictional forces in vitro between brackets and orthodontic wires in upper cusp distalization according to the Tweed-Merrifield sequential directional force technique [thesis]. São Paulo, Brazil: Faculdade de Odontologia de Piracicaba, Universidade Estadual de Campinas; 2006.
- [24] Soderholm KJ, Yang MC, Garcea I. Filler particle leachability of experimental dental composites. *Eur J Oral Sci* 2000;108:555–60.