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Porous ceramic lamellae for orthodontic ceramic brackets

Part II *In vitro performance testing*

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This study was undertaken to test a new and original orthodontic bracket base, consisting of a porous lamella, which was designed to facilitate removal of ceramic brackets from the enamel surface after treatment. In the phase of the study presented here, two types of lamella and the adhesive resin used to bond them to brackets and teeth, were evaluated *in vitro.* Two types of test were carried out on bracketed teeth. The tensile bond strength was measured for specimens that had been either kept in water for 24 h at 37 °C or subjected to 18 000 cycles in water between 6 °C and 55 °C. The stress required to remove brackets with debracketing pliers was measured and the mode of failure recorded for specimens that had been kept in water for 24 h at 37 °C. The results indicate that bracket/lamella assemblies can be bonded to enamel sufficiently strongly for clinical application and can be safely removed without damage to enamel.

1. Introduction

In the first phase of this study, the use of a crushable porous ceramic lamella was proposed as a means of solving the problems associated with the removal of ceramic orthodontic brackets [1]. The fabrication and characterization of porous alumina ceramics with different porosity levels for this application was also undertaken. The second phase of the study, described in this paper, was conducted to see if the use of lamellae *in vitro* would prove to be satisfactory.

Tensile bond strength tests were performed on the bracket/lamella assemblies bonded to bovine teeth after the specimens had either been kept at 37 °C in distilled water for 24 h or after thermal cycling between 6 °C and 55 °C for 18 000 cycles. A debonding test which simulated the use of conventional debracketing pliers was also carried out.

2. Materials and methods

2.1. Experimental lamellae

The two lamellae which gave the highest tensile bond strengths in the first phase of this study, namely FA2a and CA2b with porosities of 37% and 45%, respectively, were selected for further study. After processing and machining, these lamellae were attached to ceramic brackets by mechanical adhesion using an adhesive composite resin (Concise, 3M Dental Products, St. Paul, Minnesota, USA). The steps followed at this stage were the same as those described earlier [1]. The ceramic brackets were of the polycrystalline aluminium oxide type for use on maxillary central incisors (Transcend, Unitek Corp., Monrovia, California, USA). The bracket base was coated with a silane for chemical retention.

2.2. Sample preparation

Selected bovine incisor teeth, free of dental caries, were randomly assigned to one of the test groups. Before bonding, the labial surfaces of the crowns were polished using a pumice and water slurry in a rubber cup for 10 s. They were then rinsed with water for 15 s and blown dry with oil-free compressed air. A 37% phosphoric acid liquid, Concise etching agent, was applied to the labial surface for 60 s. Finally the teeth were washed with water for 30 s to remove the orthophosphoric acid and dried with compressed air. The labial surfaces of the teeth appeared chalky white in colour, as is normal after etching.

After the enamel preparation, the bracket/lamella assemblies were bonded to the teeth at room temperature by strictly following the adhesive manufacturer's suggested procedure. A mixture of the Concise paste A and paste B was immediately applied onto the lamella surface in a thin layer and the bracket/lamella assembly was positioned on the enamel surface. Pressure was applied to the bracket, simulating clinical chairside procedures, to express any excess adhesive from between the lamella and the enamel surfaces. The excess adhesive around the lamella was removed carefully with a dental scaler.

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For tensile bond strength tests, the samples were mounted in plastic cups filled with a low-temperature-setting resin as described previously [1]. Two major groups, FA2a (FA) and CA2b (CA) were tested. These two groups were divided into two subgroups of 30 samples each, and tested after either 24 h at 37 °C immersion in distilled water or thermal cycling. Thermal cycling in water between 6 °C and 55 °C for 18 000 cycles was used to measure the effects on bond strength of prolonged exposure to moisture at temperatures encountered in service and to simulate accelerated ageing by thermally induced stresses.

In the simulated debonding test, the experimental lamella groups FA and CA were split into two subgroups and tested with two types of debonding plier blades. The sample size for each subgroup was 20 for this test, and the specimens were kept in water at 37 °C for 24 h before testing.

2.3. Test equipment

The tensile bond strength and simulated debonding tests were carried out using a Lloyd M 5K testing machine (Lloyd Instruments Plc., Fareham, Hampshire, England). During testing, the slowly increasing force level could be observed on the digital display on the machine. The force to pull the system apart was automatically recorded and the mean bond strength, its range and standard deviation were calculated in MPa by dividing the force at failure by the mean value of the bonding surface areas of the lamellae. This value, calculated after measuring thirty bonding surfaces (fifteen from each group), was 12.54 mm^2.

Tensile bond strength was measured using the system illustrated in Fig. 1. Force was applied to the bracket using a holder that had been cast to fit the brackets very closely to reduce the chance of bracket tie-wing failure. To minimize the peeling forces, which are inevitable during tensile testing, the surface of the bracket base was oriented perpendicular to the line of force by inserting the mounted specimen into a steel cylinder which was pinned to the lower jaw of the machine. The crosshead speed of the machine was set at 1 mm/min, which is commonly used in this type of testing.

The purpose of the simulated debonding test was to determine bond failure sites and the stress levels required for both types of sharp-edged blades, which were used to crush the porous lamellae during the debonding of the bracket/lamella assemblies. The first pair of stainless steel blades used were 3.2 mm wide. These were made for use with ETM 345-6 RT conventional debracketing pliers (ETM Corporation, Monrovia, CA, USA). The second pair were a modification of these blades with the tips ground to give a pointed edge. It was thought that the pointed blades would localize and intensify the crushing force to give more progressive and controlled lamella failure.

A pair of blades were secured in two opposing steel cylinders by means of screws so that the tips were centralized in the compression jig. For testing, every specimen was positioned freely between the two blades by the operator in the incisal-gingival plane until the blades touched the lamella from both sides (Fig. 2). The crosshead speed of the machine was set at 5 mm per minute.

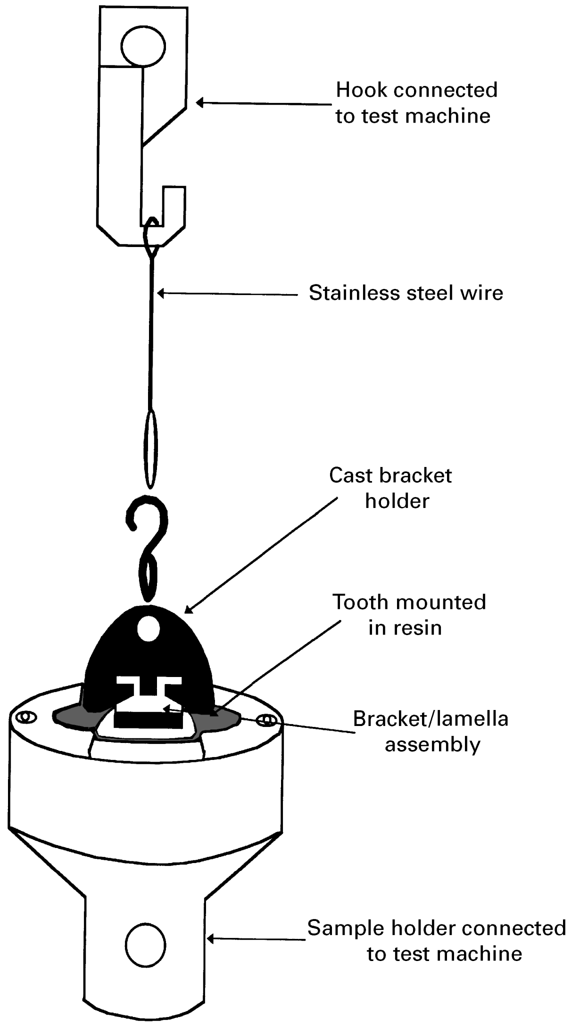

Figure 1 Tensile test equipment used in this study.

2.4. Classification of failure sites

In addition to measurements of the tensile bond strengths and debonding stresses, the bond failure sites were determined and the teeth examined for visible enamel damage. The classification of failure sites is illustrated in Fig. 3.

After testing, the separated assemblies were recovered and examined under an optical microscope at × 20 magnification to determine the site of failure. Failure sites of some of the separated assemblies were further examined using scanning electron microscopy (SEM).

3. Results

The mean tensile bond strengths and standard deviations for each group are shown in Fig. 4. It can be seen that the mean values were lower and the standard deviations were higher for the two thermally cycled groups (FA/Th and CA/Th) than their counterparts immersed in water for 24 h at 37 °C (FA/24 and

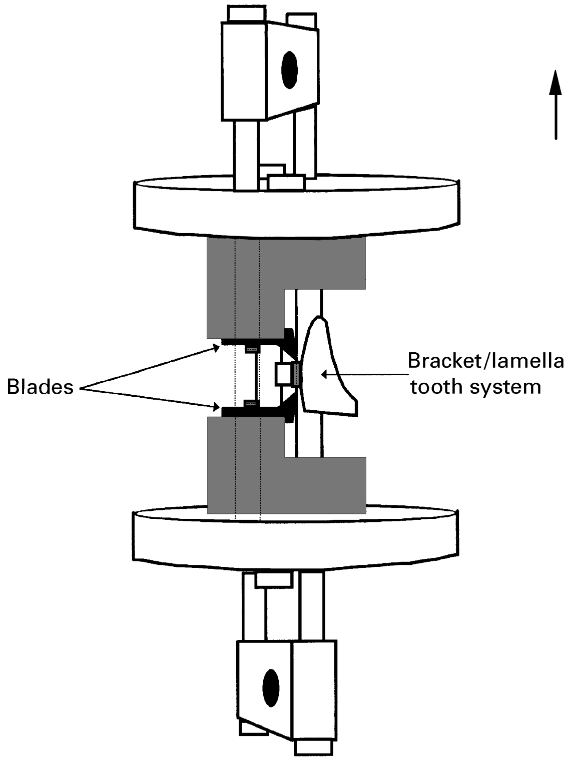

Figure 2 Equipment for simulated debonding test.

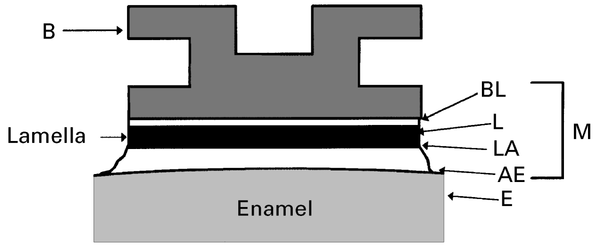

Figure 3 Classification of possible failure sites. B: bracket fracture, BL: bracket/lamella interface (bracket separates from lamella), L: lamella failure (more than 50% of failure occurs within lamellae), LA: lamella/adhesive interface (adhesive may remain within the pores of lamella; however, a continuous layer of adhesive remains on the enamel surface), AE: adhesive/enamel interface (more than 50% of the bonded enamel surface is free of adhesive), M: mixed type of failure (failure occurs at more than one interface, except B and E), E: enamel fracture.

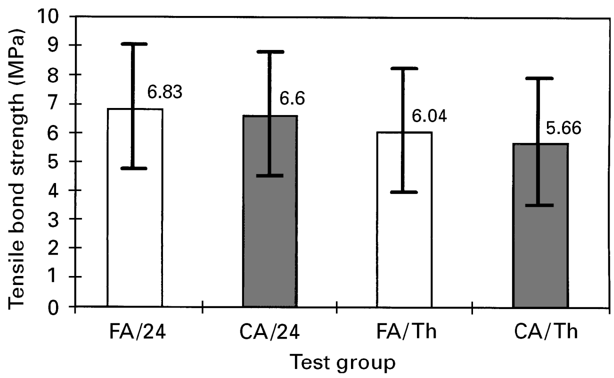

Figure 4 Mean tensile bond strengths (± standard deviations) of the groups tested after 24 h storage in water at 37 °C (24) and thermal cycling in water between 6 °C and 55 °C (Th).

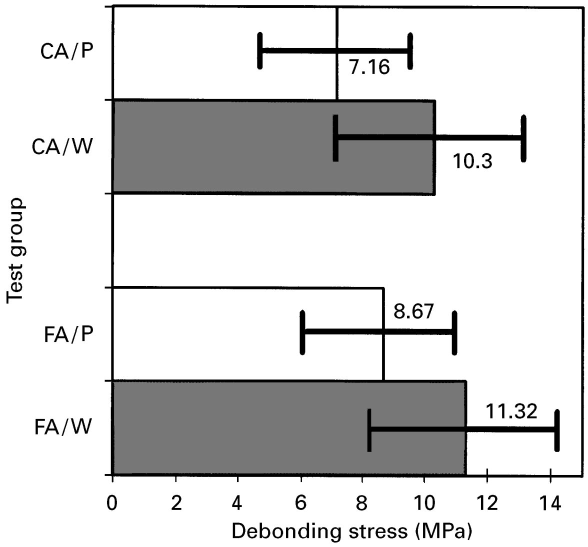

Figure 5 Mean debonding stresses at failure when using wide (W) and pointed blades (P).

CA/24), but the ranking order of the main groups remained the same. FA/24 had the highest mean tensile bond strength followed by CA/24, with values of 6.83 MPa and 6.6 MPa, respectively. The lowest value of 5.66 MPa was obtained for CA/Th.

The mean stress levels at failure and standard deviations for each group in the simulated debonding test are given in Fig. 5. The FA group had higher debonding stresses with both types of blades than the CA group. For both lamellae, use of the pointed blades resulted in lower mean debonding stresses than with the wide blades.

Table I shows the distribution of failure sites expressed both as a frequency of occurrence as well as a percentage for the two test types. Under tensile forces, both lamella groups predominantly underwent bracket/lamella interface (BL) failures (~ 50% of the total). In the simulated debonding test, both lamella groups exhibited a higher incidence of BL failures with the pointed blades than with the wide blades.

In no case was evidence of any macroscopic enamel damage found in either type of test.

4. Discussion

4.1. The bracket/lamella assembly

Although the attachment of the lamellae to the ceramic brackets was meticulously performed by the same operator, some inconsistencies may have appeared at this stage. First, the amount and viscosity of the adhesive resin applied to the bracket/lamella interface during construction of the assembly may affect the adhesive thickness at this interface. Even though the application force was constant, it cannot be claimed that all samples had the same adhesive thickness. Second, trimming of the lamella and the excess adhesive around the base with the dental burr may have affected the nominal base area as it was impossible to keep a constant working angle between the dental burr and the bracket. Therefore the bonding surface

TABLE I Failure sites in tensile and simulated debonding tests

Test type	Group				Failure	Sites			
	code	Size	B	BL	L	LA	AE	M	E
Tensile	FA/24	30	6 (20%)	13 (44%)	0 (0%)	6 (20%)	1 (3%)	4 (13%)	0 (0%)
Tensile	FA/Th	30	5 (17%)	15 (50%)	1 (3%)	4 (13%)	3 (10%)	2 (7%)	0 (0%)
Tensile	CA/24	30	6 (20%)	16 (53%)	0 (0%)	2 (7%)	2 (7%)	4 (13%)	0 (0%)
Tensile	CA/Th	30	4 (13%)	15 (50%)	2 (7%)	1 (3%)	2 (7%)	6 (20%)	0 (0%)
Tensile	Total	120	21 (18%)	59 (49%)	3 (3%)	13 (11%)	8 (7%)	16 (13%)	0 (0%)
S. Debonding	FA/W	20	0 (0%)	7 (35%)	2 (10%)	7 (35%)	3 (15%)	1 (5%)	0 (0%)
S. Debonding	FA/P	20	0 (0%)	12 (60%)	0 (0%)	8 (40%)	0 (0%)	0 (0%)	0 (0%)
S. Debonding	CA/W	20	0 (0%)	6 (30%)	4 (20%)	5 (25%)	2 (10%)	3 (15%)	0 (0%)
S. Debonding	CA/P	20	0 (0%)	15(75%)	0 (0%)	5 (25%)	0 (0%)	0 (0%)	0 (0%)
S. Debonding	Total	80	0 (0%)	40 (50%)	6 (8%)	25 (31%)	5 (6%)	4 (5%)	0 (0%)

Key: 24, kept in water at 37 °C for 24 hours; Th, thermally cycled; W, wide (3.2 mm) blades; P, pointed blades.

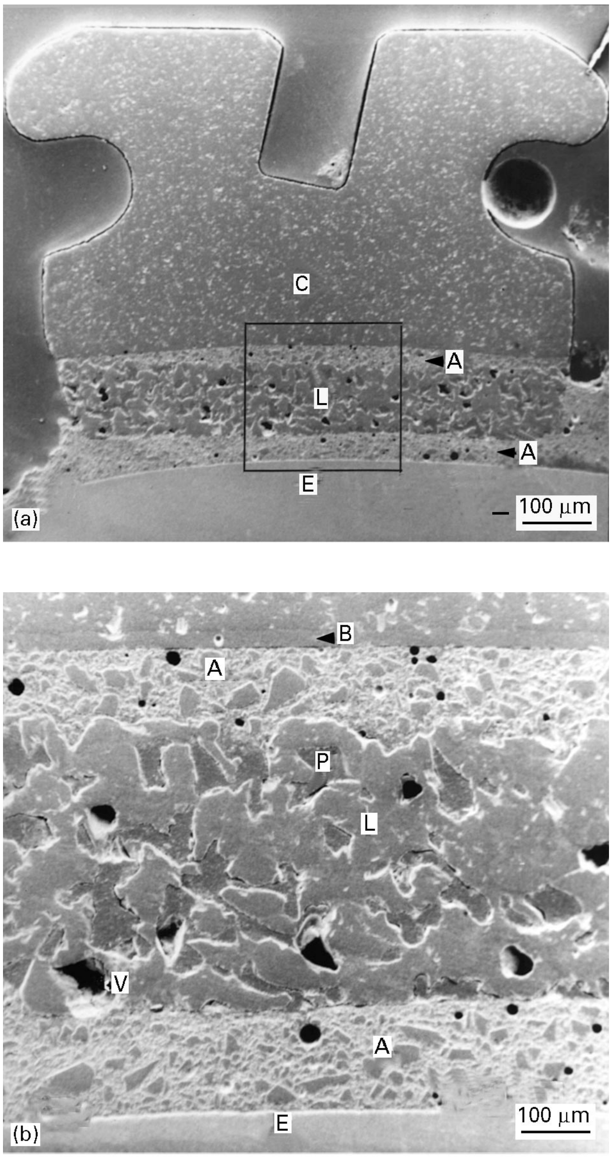

Figure 6 Scanning electron micrographs of (a) cross-section of the bracket/lamella assembly on tooth. (b) enlargement of the area outlined in Fig. 6a. A: adhesive resin layer, B: silane-coated glass bracket base, C: ceramic bracket, E: enamel, L: lamella, P: pores filled with adhesive, V: voids or unfilled pores.

areas of the thirty bracket/lamella assemblies, prepared in the same way, were measured and the average was taken and used for bond strength calculations.

Fig. 6a shows a cross-section of a bracket/lamella assembly on a tooth surface. Fig. 6b shows an enlargement of the lamella and its interfaces with the bracket and the enamel surface. The mechanical interlocking between the adhesive and lamella can clearly be seen. Although some pores appear not to be filled with resin, in most cases resin forms a continuous phase from the base of the bracket to the enamel. Although it was anticipated that during debonding the separation would occur within the lamella, it was found that the wedge action of the blades tended to cause separation at the bracket/lamella interfaces (BL).

4.1. Tensile bond strengths

In spite of the use of standardized bonding and testing methods (performed by one investigator), some inconsistencies would have been introduced by variations of enamel prism micromorphology, thickness of adhesive layer and the distribution of the porosity in the lamellae. Nevertheless, the standard deviations in the bond strengths obtained in this study were in the range which would normally occur in this type of test [2–4].

It has been reported that a tensile bond strength of 7 MPa would be adequate to withstand the forces encountered in treatment [5]. It has also been stated that *in vitro* experiments with brackets giving tensile bond strengths of 4.9 MPa have proved clinically acceptable [5]. Although following thermal cycling the tensile bond strengths of both bracket/ lamella groups were reduced, they were still within the guidelines given in the literature.

The decrease in the bond strengths of thermally cycled specimens relative to those that were not cycled may possibly be explained by the absorption of water and the alternating stressing of the system resulting from the large mismatch of the thermal expansion coefficient of the adhesive with those of the bracket, lamella and enamel. The former is likely to affect adversely the adhesion of the resin to other parts of the system. The alternating stressing may cause any debonded regions to grow progressively in size.

4.2. Debonding stresses

The mean debonding stress obtained with the pointed blades was lower than that for the wide blades in both the FA and CA groups, being 24% less in the former case and 30% less in the latter. This probably means that use of pointed rather than wide blades would lead to a reduction in the stresses on the enamel surface during debonding.

It has been stated that it would be best to avoid bond strengths larger than 138 kg/cm^2 (13.53 MPa) during removal of brackets by conventional debonding pliers [6]. This suggests that debonding with pliers fitted with pointed blades would be a safe and satisfactory method since none of the specimens debonded with these blades had a bond strength value higher than 13 MPa.

4.3. Failure sites

The predominant failure site for both groups in the tensile tests was at the bracket/lamella (BL) interface. Although the application of tensile type forces is not the intended technique for the debonding of the bracket/lamella assemblies, the occurrence of this type of failure site with the lamellae may offer a clinical advantage in protecting the adhesive/enamel interface from damage if excessive tensile forces were accidentally applied.

Fracture of the tie-wings of ceramic brackets is a common problem during *in vitro* tensile testing [3,4]. Bracket fracture occurred in 18% of the test groups in tensile testing. A possible explanation for this high incidence of fracture could be the introduction of flaws during the preparation of bracket/lamella assemblies. However, since this study advocates the use of a debonding technique which places no stress on the bracket tie-wings, indeed does not require them to be present, tie-wing fracture during debonding should be reduced to negligible levels.

The results of the simulated debonding test in the present study showed that applying the load to the two sides of the lamella using the sharp-edged or pointed blades starts a crack which propagates in the brittle lamella or at the interfaces between the lamella and adhesive. Although it was assumed *a priori* that the crack would propagate in the lamella structure alone, this did not happen in practice. In fact, the propagation of the crack, which began in the lamella, frequently shifted towards to the bracket/lamella interface. This is thought to occur because of the shape of the debonding blades (Fig. 7). After the sharp edge of the blade, particularly for the pointed blades, had penetrated into the lamella for a certain distance, the interior slope of the blade came into contact with the corner of the bracket and acted as a wedge lifting it away from the lamella on which it was bonded. This could explain why 50% of the specimens in this test failed at the bracket/lamella interface.

It is important to note that there was no failure at the adhesive/enamel interface in the debonding test with the pointed blades. Failure at the adhesive/enamel interface is not desirable since it places debonding stresses directly on the enamel surface, which in turn increases the chance of damaging the enamel [6].

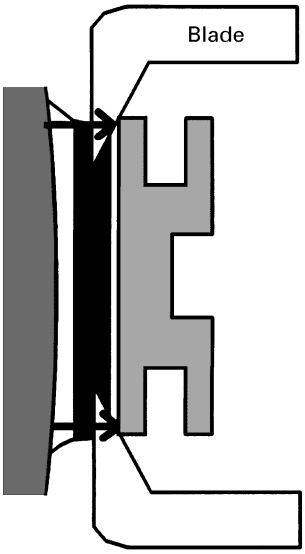

Figure 7 Debonding action of blades penetrating lamella and lifting bracket.

4.4. Clinical implications

Since clinical (*in vivo*) studies of any dental material are time consuming, expensive and involve patients, a manufacturer of dental products and researchers rely largely on laboratory (*ex vivo*) testing to predict the clinical performance of materials. However, extrapolation of laboratory data to the clinical situation should always be done with care because of the complexity of the oral environment. The changes in temperature, humidity and acidity as well as the mechanical and masticatory stresses placed on a bracket "on duty" in the oral cavity cause deterioration of the adhesive bond, and are impossible to simulate in a laboratory [7]. Nevertheless, laboratory testing can be used as a screening mechanism for predicting clinical performance [8].

The forces required to move a tooth orthodontically through bone usually vary from between 0.5 N and 4 N [9]. It was also reported that 45 N of applied orthodontic force is rarely exceeded in clinical conditions [10]. With a bond area of around 12 mm^2, as used in this study, such a force would translate to a pressure of around 3.7 MPa. As is evident from the results of this study, the bracket/lamella assemblies bonded to teeth should withstand these orthodontic forces.

The bracket/lamella assembly may be suitable for clinical application because it produced low stress levels on the enamel and a satisfactory bond failure site during debonding with conventional debonding pliers. Furthermore, it exhibited a slow and progressive type of bond failure pattern. During *in vitro* debonding with the method used, the majority of the CA specimens failed in a manner in which the bracket slowly separated from the lamella. The FA specimens, however, showed a somewhat more sudden type of failure. This may have been caused by the difference in the porosities of the two lamellae.

As stated before, 50% of the specimens in the debonding test failed at the bracket/lamella interface, and a small trial showed that the lamellae or lamella fragments remaining on the teeth could easily and safely be removed with the conventional debonding pliers. This would be a time–saving procedure for the clinician.

The results of the present study show that clinical application of the bracket/lamella assembly would provide the potential advantages listed below during debonding with conventional debonding pliers at the end of orthodontic treatment.

1. The chance of enamel damage is minimized. The reduction in potential for enamel damage is directly related to the type of bond failure that occurs during debonding.
2. Less discomfort for the patient due to a reduction in the rate of change of debonding stress.
3. Potentially safer debonding for both the patient and orthodontist. Sudden failure of the bond or the bracket and related risks, such as that of aspiration of ceramic bracket fragments by patient or operator, or eye injuries, during debonding is minimized (particularly with the CA lamellae).
4. Less time will be required for debonding and clean-up procedures than with other debonding methods. It is unnecessary to remove all adhesive flash from the bracket base prior to debonding in order to properly seat the debonding instrument, and also the remnant lamella parts can be removed with the same debonding pliers as are used for debonding. Ceramic fragments are not left on the tooth so it is unnecessary to remove remaining ceramic bracket fragments with a diamond burr in a high-speed hand piece which bears a potential risk of enamel damage, and is time consuming.
5. Since the debonding of a bracket/lamella assembly does not use the bracket tie-wings, as do the latest lift-off or electrothermal debonding devices, debonding can be carried out even when the tie-wings of the bracket have been lost during the active phase of the orthodontic treatment.
6. The approach is economical, because there is no failure of the ceramic bracket body during debonding, and therefore recycling of the ceramic bracket body is possible. It also does not require expensive and complicated debonding tools as do several of the other debonding methods.

However, the ceramic bracket/lamella assembly has some recognized disadvantages.

1. The lamella increases the thickness of ceramic bracket. In order to eliminate this disadvantage the bracket base should be reduced in thickness. This is turn may weaken the bracket structure.
2. Those pores in the lamella structure which are not filled by the adhesive resin during bonding may cause problems with aesthetics and oral hygiene because of debris accumulation. This problem will arise at the margins of the lamella (mesial, distal, occlusal and gingival) where the adhesive is not directly applied. Although some of the overflowing adhesive during the bonding stage may fill some of the pores at these margins, this problem could be prevented by isolating the lamella from the oral fluids with adhesive resins or other biocompatible materials.
3. The machining of individual lamellae is not an ideal fabrication method for commercial production. It is possible that the use of tape casting could be employed, coupled with subsequent moulding of the plasticized sheet, when required, to yield appropriately curved surfaces.

5. Conclusions

On the basis of the data that were collected and statistically analysed in this study, the following conclusions may be drawn.

1. Mean tensile bond strengths for both of the bracket/lamella assemblies (FA, CA) with Concise adhesive resin indicate that they should be adequate for clinical use.
2. Thermal cycling in water between 6 °C and 55 °C for 18 000 cycles resulted in a reduction in the tensile bond strengths. This reduction was, however, not considered sufficient to be important clinically since all groups still had adequate bond strength to withstand normal orthodontic forces.
3. The simulated conventional debonding method easily removed the ceramic bracket/lamella assemblies from the teeth surfaces without fracture of the ceramic bracket or evidence of damage to the enamel.
4. The pointed debonding blades developed for the present study, when compared with the wide blades, gave a lower mean debonding stress for both lamellae. Therefore it would be advantageous to debond the bracket/lamella assemblies with the pointed blades because of the reduced stresses transmitted to the enamel surface.

It is concluded therefore that ceramic brackets fitted with porous lamellae are potentially able to resist orthodontic forces in clinical use and should be easily removable with simple instruments at the completion of treatment. It is also suggested that there would be a substantially reduced risk of damage to the enamel during debonding. It is postulated that the major clinical objection to the use of ceramic brackets may thus be overcome.

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References

1. S. ARICI, C. J. MINORS and P. F. MESSER, *J. Mater. Sci. Med.* (Submitted)

2. A. J. OSTERTAG, V. B. DHURU, D. J. FERGUSON and R. A. MEYER, *Amer. J. Orthod.* **100** (1991) 251.
3. J. M. BORDEAUX, R. N. MOORE and M. D. BAGBY, *ibid.* **105** (1994) 552.
4. S. J. CHACONAS, A. A. CAPUTO and G. S. NIU, *Angle Orthod.* **61** (1991) 35.
5. I. R. REYNOLDS, *Br. J. Orthod.* **2** (1975) 171.
6. S. E. BISHARA, J. M. FONSECA, D. E. FEHR and D. B. BOYER, *Angle Orthod.* **64** (1994) 277.
7. G. ØILO, *Int. Dent. J.* **43** (1993) 492.
8. P. GANGE, *J. Clin. Orthod.* **29** (1995) 429.
9. A. A. GIANELLY and H. M. GOLDMAN, In "Biologic basis of orthodontics" (Lea & Febiger, Philadelphia, 1971) p. 159.
10. G. V. NEWMAN, *Amer. J. Orthod.* **51** (1965) 901.

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