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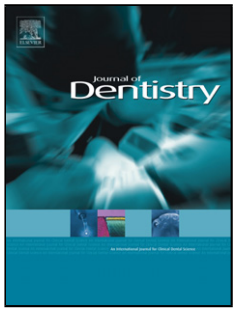

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Laminated ceramics with elastic interfaces: a mechanical advantage?

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ABSTRACT

Objectives: As CAD/CAM technologies improve we question whether adhesive lamination of ceramic materials could offer mechanical advantages over monolithic structures and improve clinical outcomes. The aim was to identify whether an adhesive interface (a chemically cured resin-cement) would influence the biaxial flexure strength (BFS) and slow-crack growth in a machinable dental ceramic.

Methods: Monolithic and adhesively laminated (with a chemically cured dimethacrylate resin-cement) feldspathic ceramic discs of identical dimensions were fabricated. BFS testing was performed on the Group A monolithic specimens (n=20), on Group B laminated specimens with the adhesive interface positioned below the neutral bending axis (n=20) and Group C laminated specimens with the adhesive interface positioned above the neutral bending axis (n=20). To study subcritical crack growth additional laminated specimens received controlled indentations and were exposed to thermo-mechanical fatigue. BFS data was analysed using parametric statistics (α=0.05). Fractographic analyses were qualitatively assessed.

Results: No significant differences between the mean BFS data of Groups A and B were observed ($p=0.92$) but the mean BFS of Group C was slightly reduced ($p<0.01$). Lamination reduced the stiffness of the structure and fractographic analysis demonstrated that energy consuming crack deflection occurred. Thermo-mechanical fatigue caused subcritical extension of radial cracks associated with indentations adjacent to the adhesive interface. Crack growth was limited to parallel to the interface and was arrested or deflected in a direction normal to the interface.

Conclusions: Ceramic lamination increased the damage tolerance of the structure and could limit or arrest subcritical crack growth at regions near the 'interlayer'.

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1. INTRODUCTION

Over the previous two decades there have been significant developments in Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) technologies for dental applications which now enable the fabrication of restorations from a range of materials, to a high level of dimensional accuracy [1,2]. More recently, ceramic CAD/CAM systems that employ a digital workflow to independently design and manufacture different layers of a dental restoration before they are subsequently joined together using an interface adhesive have been introduced [3,4]. The development allowed for ceramic core-veneer restorations to be manufactured with favourable residual stressing patterns [3,4,5]. The approach also reduces operator induced variability associated with the manual build-up of the veneering ceramic layer [6,7]. However, as CAD/CAM technologies improve further the question arises as to whether 'laminated structures consisting of multiple (possibly functionally graded) adhesively bonded ceramic layers could offer mechanical advantages that would improve clinical outcomes'?

The precedent for using adhesively laminated ceramics and glasses for structural purposes is widespread [8,9,10]. A large body of evidence from outside of the dental literature has demonstrated a modification of the mechanical properties of brittle materials when used in laminated structures such as thermal barrier coatings [11,12], architectural laminated glass [13] and automotive windscreens [14]. Researchers identified adhesive lamination changes the pattern of fracture when compared with monolithic structures of equal composition and dimensions [14,15]. Investigations on glass substrates identified adhesive lamination reduces the effective stiffness of the structure whilst maintaining, or in some cases improving, the

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flexural strength [16]. The selection of interface material and laminate design can be tailored to modify the load bearing capacity of the particular system [17]. However, it has also been recognised that mechanical improvements can be unpredictable because of the brittle nature of the glass and the sensitivity to pre-existing defects and residual stress states [17]. A consequence of the interfaces created can be the inadvertent introduction of new strength limiting flaws which may be absent in the monolithic substrate but may ultimately determine the strength of the system [17].

If processing routes can be identified to minimise the introduction of strength limiting defects, it is possible to 'toughen' a ceramic or glass structure by laminating with a polymer adhesive interface [14]. Therefore, on application of an external load, the laminating adhesive ('interlayer') can absorb energy elastically [17] and allow shear transfer, thereby, transporting the location of the load-reaction away from the concentrated point of application [14]. In the event where a crack does propagate through the 'interlayer', strain generated in the adhesive in the crack wake can act as a crack-bridge and arrest further extension [10]. Subsequently if one laminate layer fails, others can retain some load bearing capacity to retain overall function [10]. The flexural stresses generated in the 'interlayer' remain small in comparison due to a substantially lower elasticity of the typical 'interlayer' materials when compared with the ceramic or glass laminates, [10].

The overall objective of the current study was to investigate the concept of introducing polymer adhesive interfaces into dental ceramic materials to create laminated structures. The specific aim was to identify whether an adhesive interface (a chemically cured resin-cement) would influence the biaxial flexure strength (BFS) and slow-crack growth in a machinable feldspathic dental ceramic. Given the lack of evidence in this subject area the null hypotheses

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tested were that lamination would have no impact on both the BFS and the slow crack growth in a feldspathic dental ceramic.

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2. MATERIALS AND METHODS

2.1 Preparation of ceramic discs

Feldspathic ceramic blocks (40/19 VITA Mark II – VITA, Bad Säckingen, Germany, LOT 36990) were rounded to a 15 mm diameter cylinder using a diamond impregnated core drill under copious water lubrication. The cylinders were sectioned to produce circular discs using a low-speed diamond impregnated saw (IsoMet Low Speed, Buehler, Illinois, USA) with water as a lubricant. The discs were manually polished on one surface using P120 silicon carbide abrasive paper followed by P500, P800, and P1200 (Struers, Glasgow, UK) to achieve final thicknesses of 1.50 ± 0.01 mm (n=20); 1.00 ± 0.03 mm (n=43) and 0.50 ± 0.02 mm (n=43) measured using a digital micrometer accurate to 10 µm (Mitutoyo Corporation, Tokyo, Japan).

2.2 Preparation of ceramic samples for biaxial flexure strength (BFS) determination

Three different sample geometries were fabricated (Figure 1). The polished surface of each 1.0 mm and 0.5 mm ceramic discs was etched with 9.0 % hydrofluoric (HF) acid gel for 60 s (Ultradent Porcelain Etch, Ultradent Products, Cologne, Germany), thoroughly washed with water and allowed to air dry. The etched surface was silane coated (Ultradent Silane, Ultradent Products, Cologne, Germany) and allowed to air dry for 10 mins. Group A specimens were 1.5 mm thickness ceramic discs which received no HF acid gel or silane surface treatment. Group B specimens were fabricated by adhesively bonding the 1.0 mm ceramic discs on top of the 0.5 mm ceramic discs using a layer of chemically cured resin-cement (Panavia 21, Kuraray Co. Ltd., Tokyo, Japan). The chemically cured resin-cement was hand-mixed and applied to the centre of the silane primed surface of the 0.5 mm disc.

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The silane primed surface of the 1.0 mm ceramic disc was then placed onto the resin-cement so that the two discs were aligned directly one above the other. The discs were then loaded on a flat surface with a constant weight of 100 g for 10 mins. Group C specimens were fabricated by resin-cementing the 0.5 mm discs onto the top of the 1.0 mm discs and loaded according to the protocol for Group B. For the layered specimens in both Groups B and C, the excess resin-cement that exuded from the interface was carefully removed from the specimen periphery immediately after loading.

2.2.1 Determination of BFS

BFS was determined in a ball-on-ring configuration at a room temperature (23 ± 1 °C) using a universal testing machine (Instron 5544 with a 2 kN load cell, Instron Ltd, Bucks, England) with a loading rate of 1 mm/min. The ceramic samples were positioned centrally on a thin rubber sheet placed on top of a 10 mm diameter ring-support. The upper surface of each sample was centrally loaded with a 4 mm diameter stainless steel spherical ball indenter. The load at failure, the number of fracture fragments and the specimen thickness of each fracture fragment were measured. The BFS was calculated according to Timoshenko and Woinowsky-Kriegers' analysis (Eq. 1) [18] (n=20 per group).

$$\sigma = \frac{P}{t^2}\left\{(1+v)\left[0.485\ln\left(\frac{a}{t}\right)+0.52\right]+\ 0.48\right\} \qquad \text{Eq. 1}$$

where σ was the maximum biaxial flexure stress; P the measured load to fracture; a the radius of the ring-edged support; t the specimen thickness and v the Poisson's ratio where a value of 0.25 [19] was used for the ceramic investigated.

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Group B specimens were loaded in accordance with Figure 1B with the 1.0 mm disc uppermost so that the adhesive interface was closest to the ring support and below the neutral axis of bending (in tension). Group C specimens were loaded with the 0.5 mm disc uppermost so that so that the adhesive interface was closest to loading contact and above the neutral axis of bending (in compression) (Figure 1C).

2.2.2 Fractography

Fracture fragments derived from BFS testing were gold sputtered and imaged using scanning electron microscopy (SEM) (Zeiss EVO, Carl Zeiss GmbH, Jena, Germany) under high vacuum at operating voltages between 5 and 20 kV. The fracture surfaces were studied qualitatively at a range of magnifications to provide insight into the location of the fracture origin and the sequence of fracture events.

2.3 Qualitative measurement of crack extension in the region of ceramic-adhesive interfaces.

The remaining three untested laminate specimens were sectioned perpendicular to the resin cement interface using a diamond impregnated saw under water lubrication (IsoMet low speed saw, Buehler, Illinois, USA) to create semi-circular shaped specimens. The cut surfaces were gently sequentially polished using P4000 silicon carbide abrasive paper (Struers, Glasgow, UK) and a porous neoprene cloth (MD-Chem, Struers, Denmark) in combination with 0.04 µm colloidal silica abrasive suspension (OP-S Suspension, Struers, Denmark). The specimens were mounted in a stainless steel block so that the polished surface was horizontal and using the x40 objective of the microscope of a hardness indenter (Duramin, Struers, UK) to aid positioning, a series of Vickers indentations (10-20 µm) were created parallel to the adhesive interface with an indentation load of 1.961 N applied over 20 s. The indents were separated by a distance of 30-50 µm. SEM was then undertaken to visualise the location and

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proximity of each indent and associated cracks to the resin-ceramic interface prior to thermo-mechanical fatigue.

2.3.1 Thermo-mechanical fatigue

Following indentation the specimens were subjected to thermo-mechanical fatigue to study crack-extension in the proximity of the adhesive interface. Specimens were thermocycled in water between 4 ± 1 and 65 ± 1°C, for 10,000 cycles with a dwell time of 5 s and a transfer time of 3 s. Temperatures were chosen to represent the most extreme temperatures that may be encountered in the oral environment [20,21]. Subsequently, the test specimens were positioned such that the adhesive interface was parallel to the loading platen. The specimens were cyclically loaded on the upper surface (approximately central within the semi-circular specimen) for 30,000 cycles loading and unloading between 0 and 20 N at 1 mm/min using the universal testing apparatus (Instron 5544 with a 100 N load cell, Instron Ltd, Bucks, England). A 4 mm diameter stainless steel spherical ball indenter was used to apply the load and the specimen surface was protected using a thin rubber sheet to obviate any discrepancy in the flatness of the specimen or supporting surface and orientation. Following cyclic loading the specimens were subjected to a further 15,000 thermal cycles (4 to 65°C, 5 s dwell time, 3 s transfer time). SEM was conducted to capture images of the identical indents before and after the artificial fatigue process.

2.4 Statistical analysis

Comparisons of group means (Group A-C) were made utilising a one-way analysis of variance (ANOVA) and a post-hoc Tukey's multiple range test (α=0.05). Survival probability curves were examined to assess the distribution of flexure strengths values. Slow crack extension adjacent to adhesive interfaces was qualitatively assessed.

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3. RESULTS

The one-way ANOVA identified a significant difference between the mean BFS data of Groups A-C ($p=0.003$, $\beta=91\%$) (Table 1). Post-hoc Tukey tests identified Group C specimens which possessed an adhesive interface above the neutral axis of bending during BFS testing, exhibited a significantly lower BFS when compared with Group A ($p=0.01$) and Group B ($p<0.01$) specimens. There was no significant difference in the mean BFS of the monolithic specimens (Group A) and adhesively bonded specimens when the adhesive interface was positioned below the neutral axis of bending (Group B) during BFS determination ($p=0.92$). A similar distribution of the BFS data was observed for all three groups in the survival probability distributions (Figure 2). Plotting the mean load during testing against the deflection derived from the BFS test identified that load-deflection curve for the Group A monolithic specimens was steeper than those of Groups B and C which were coincident with each other (Figure 3). For an equivalent peak load, Group B and Group C specimens possessing an adhesive interface demonstrated a significantly increased deflection on loading. In addition, Group B and Group C specimens demonstrated similar numbers of fracture fragments following BFS testing with an increase in the number of fracture fragments observed for Group A specimens.

Fractography of the fracture fragments generated during BFS testing demonstrated that in all cases failure originated at the lower-most surface (Figure 4) of the test specimens (in-contact with the loading ring). Porosity was observed in the interface layer in the fracture plane for a number of specimens (Figure 4b and 4c) but could not be identified to be associated with fracture initiation. The fracture direction was observed to be modified by the presence of the

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resin-cement interface to varying extents and high strength specimens were generally associated with greater deflection in the crack direction (Figure 4d).

The introduction of indentations adjacent to the ceramic-resin interface resulted in the generation of median/radial cracks within the ceramic that were approximately parallel and normal to the adhesive-ceramic interface (Figure 5). On indentation, the crack lengths were longer in a direction parallel to the interface compared with cracks normal to the interface which appeared to have been arrested when they encountered the resin-cement (Figure 5a and c). Following thermo-mechanical fatigue, the cracks parallel to the adhesive interface were observed to increase in length often joining up with cracks from adjacent indents of 20 - 30 μm distance apart. No crack extension was observed in cracks normal to the interface through the 'interlayer' itself (Figure 5b and d). A modification in crack direction was observed when cracks originally normal to the resin-cement interface extended with a tendency towards growth in a direction parallel to the interface. No delamination of the interface was observed following thermo-mechanical fatigue.

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4. DISCUSSION

The introduction of an adhesive interface to create a laminated ceramic bilayer reduced the effective stiffness of the ceramic structure (Figure 3) but resulted in no significant strengthening when subjected to monotonic BFS testing. The mechanical response is consistent with other structural systems such as laminated safety glass where equivalent strengths to monolithic glass structures can be achieved [22]. However, it has been demonstrated elsewhere that the strength of a brittle laminate is highly sensitive to the elastic and viscoelasticity properties of the adhesive 'interlayer' which was not systematically considered in the current study [22].

A reduction in the stiffness of the ceramic structure through lamination as evident in the load-deflection plots (Figure 3) may offer a mechanical advantage when the ceramic is subjected to loading in the oral environment. The load-deflection data indirectly demonstrates a 'toughening' effect of lamination implying an increased energy requirement to cause fracture. The reduction in the number of fracture fragments generated during BFS testing of the laminated structures suggests reduced energy storage in the ceramic bulk (instead translated into elastic strain in the 'interlayer') prior to failure [23]. Additionally fractographic analyses identified that the adhesive interface can mediate crack deflection which has an increased energetic requirement and was more pronounced in 'high strength' specimens measured during BFS testing (Figure 5d) compared with 'lower strength' counterparts (Figure 5c).

The mean BFS data and toughening effects are promising as they suggests a damage tolerant system. It is important to recognise that the adhesive interface was generated using an

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existing adhesive system (chemically cured resin-cement) which is known to reliably bond to the etched and silane primed feldspathic ceramic surface [24]. The interface was not idealised and was shown to be susceptible to operator induced porosity. A small but significant strength reduction was observed in Group C specimens where the laminate was subjected to BFS testing with the adhesive interface positioned above the neutral axis of bending (Figure 1C). The mean load-deflection plot (Figure 3) was similar to that of Group B laminated specimens but this was a measure of deflection of the upper surface and is not indicative of equivalent strain at the lower surface in maximum tension. There will also have been a difference in the crack velocity when it reached the interface, namely the crack would be expected to be travelling faster in Group C specimens when compared with Group B specimens, thereby potentially mitigating the effect of the 'interlayer'.

Stable crack extension [25] of pre-existing defects in dental ceramic restorations usually precedes clinical failure [26]. In the current investigation, the effects of the presence of the adhesive layer on crack extension were studied by the introduction of controlled Vickers indentations into the ceramic - in close proximity to the 'interlayer'. Following indenter removal cracks form around the plastically deformed zone with some cracks extending radially outwards perpendicular to the surface [27]. At low indentation loads, such as those used in the current investigation, it would be expected that separate radial cracks would form and extend to their full length after load removal rather than 'halfpenny cracks' which constitute a single crack extending from one side of the indentation zone to the other [28]. Indents were introduced adjacent to the adhesive interface and orientated to create radial cracks both parallel and normal to the interface. Following indentation the radial cracks parallel to the adhesive interface were observed to be longer than cracks normal to the interface which appeared to be arrested. Importantly, cracks normal to the adhesive interface

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but extending away were also of reduced length which suggested that a residual stress state exists in the ceramic adjacent to the 'interlayer'. Residual stresses could be attributed to the polishing process [29] but would be expected to affect both parallel and normal cracks or may be attributed to the resin-cement through polymerisation shrinkage stresses [29].

Following thermo-mechanical fatigue cracks were observed to extend considerably parallel to the adhesive interface but not extend through the interface itself. Although cracks normal to the adhesive interface did extend, the crack direction was observed to change to a more parallel path before or at the adhesive interface. The observed pattern of slow crack growth would be clinically favourable preventing crack extension in particular in a radial direction towards the restoration surface.

5. CONCLUSIONS

The current study has identified that lamination of a dental ceramic with a polymeric 'interlayer' could offer toughening effects which could potentially delay or arrest sub-critical crack growth at regions near the interlayer. Conceptually the generation of a multilayer laminate structure demonstrated for illustrative purposes in Figure 6, could increase the toughness of the ceramic structure further. The adoption of technological knowhow from other engineering disciplines to idealise interfaces, interlayer materials and processing routes could further enable the fabrication of more damage tolerant structures which would be of significant clinical benefit. While incorporation of functionally and aesthetically graded layers would be realisable, considerable research would be required before the feasibility of the conceptual approach could be accurately assessed. Today the technological requirements in CAD/CAM to generate such structures for dental applications are no longer unrealistic.

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FIGURES

Figure 1. Schematic representation of the ceramic specimens from Group A (monolithic), Group B (adhesive interface below the neutral plane during BFS testing), Group C (adhesive interface above neutral plane during BFS testing) investigated in the study.

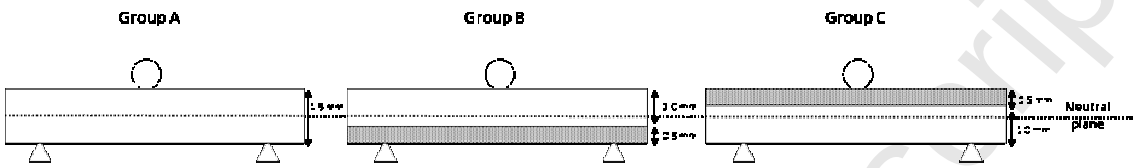

Figure 2. Plot of the individual biaxial flexure strengths (MPa) against survival probability for the Group A monolithic specimens (n=20), Group B laminated specimens with the adhesive interface tested in tension (n=20) and Group C laminated specimens with an adhesive interface tested in compression (n=20).

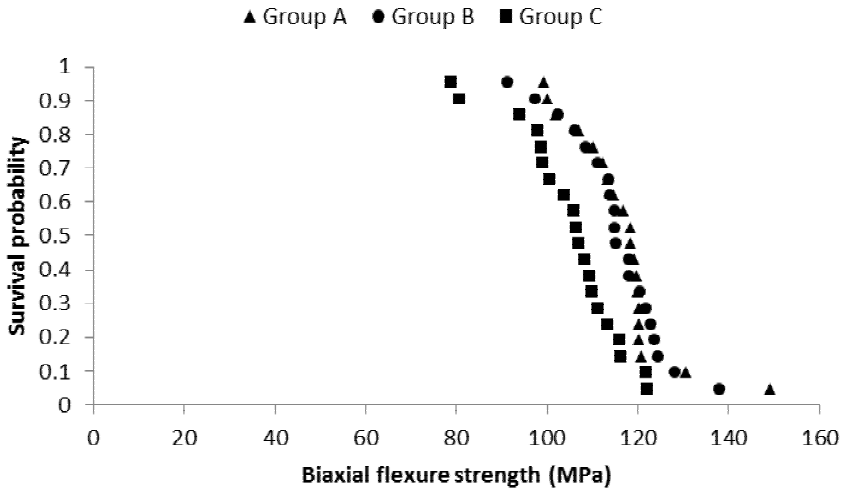

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Figure 3. Plot of load (N) against deflection (mm) derived from BFS testing of Group A monolithic specimens, Group B specimens with the adhesive interface tested in tension and Group C specimens with an adhesive interface tested in compression. Plots represent the mean (and associated standard deviation) of the specimens tested for each group.

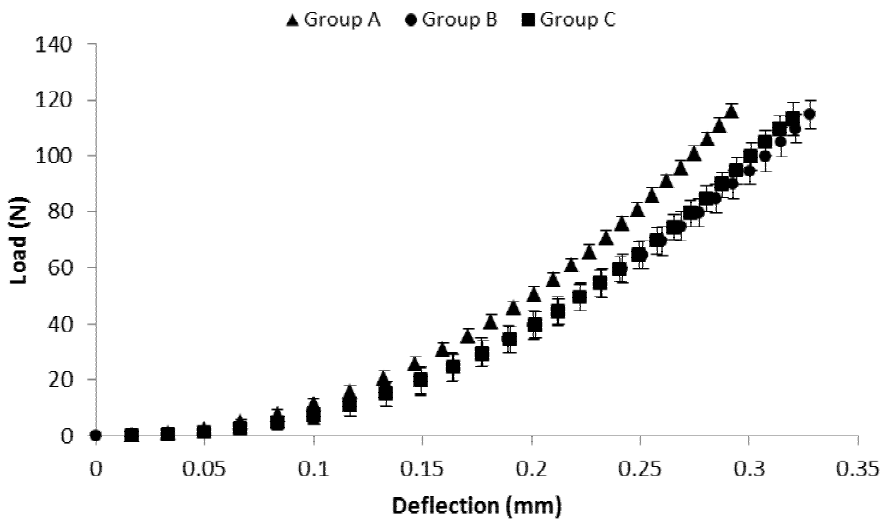

Figure 4. Scanning electron micrographs of fracture surfaces of the disc-shaped ceramic specimens following BFS testing. **(a)** demonstrates a monolithic specimen (Group A) with the fracture origin clearly evident at the lower surface. **(b)** is representative of a Group B specimen with the fracture origin again at the lower surface. Although porosity is present in the resin-cement adhesive no obvious effect on crack origin or crack direct was evident. **(c)** is illustrative of a low strength specimen from Group C. The fracture origin is situated on the lowermost surface almost vertically below the zone of contact loading. The fracture surface is not totally smooth in the transition across the adhesive interlayer suggesting a small degree of crack deflection. **(d)** In contrast a high strength specimen from Group C exhibits a clear change in the crack direction as it crosses the adhesive interlayer.

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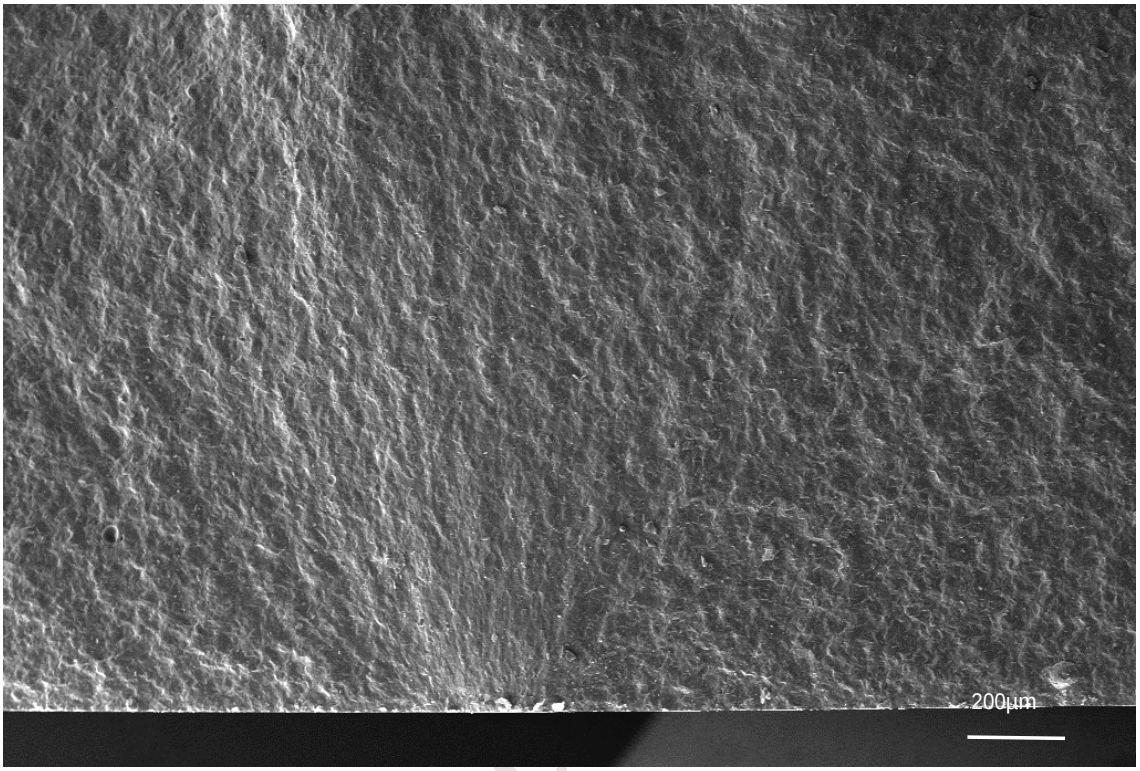
(a)



(b)

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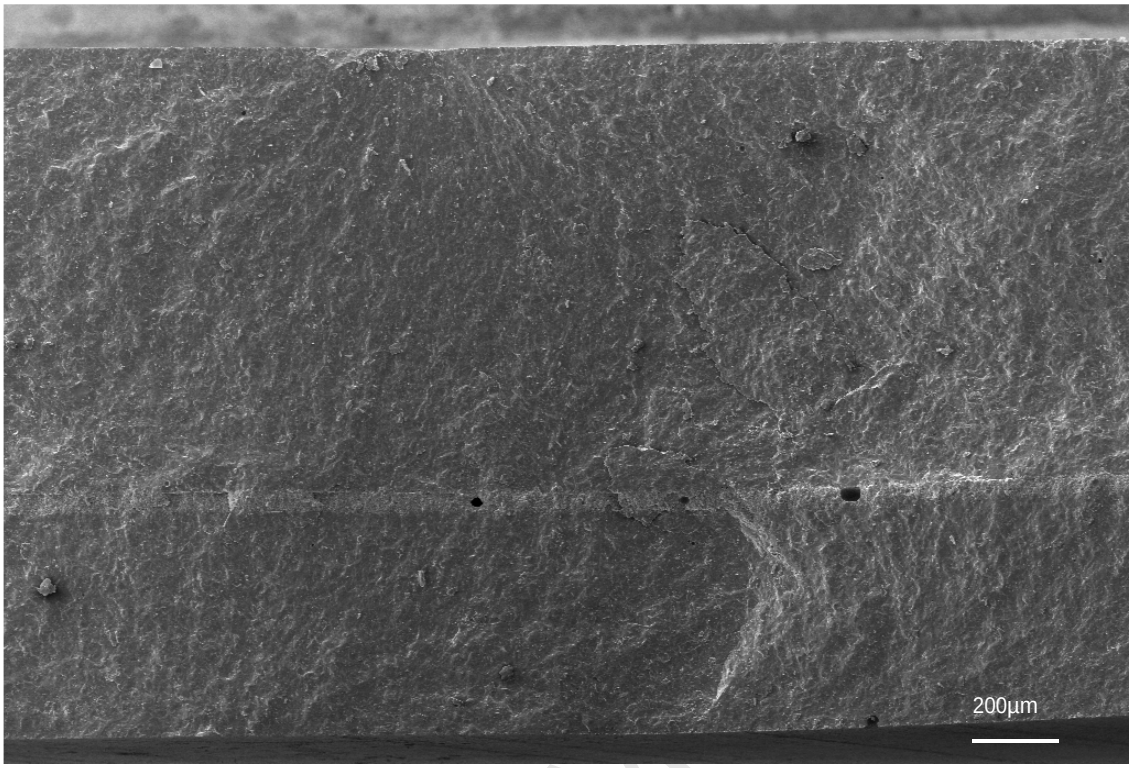
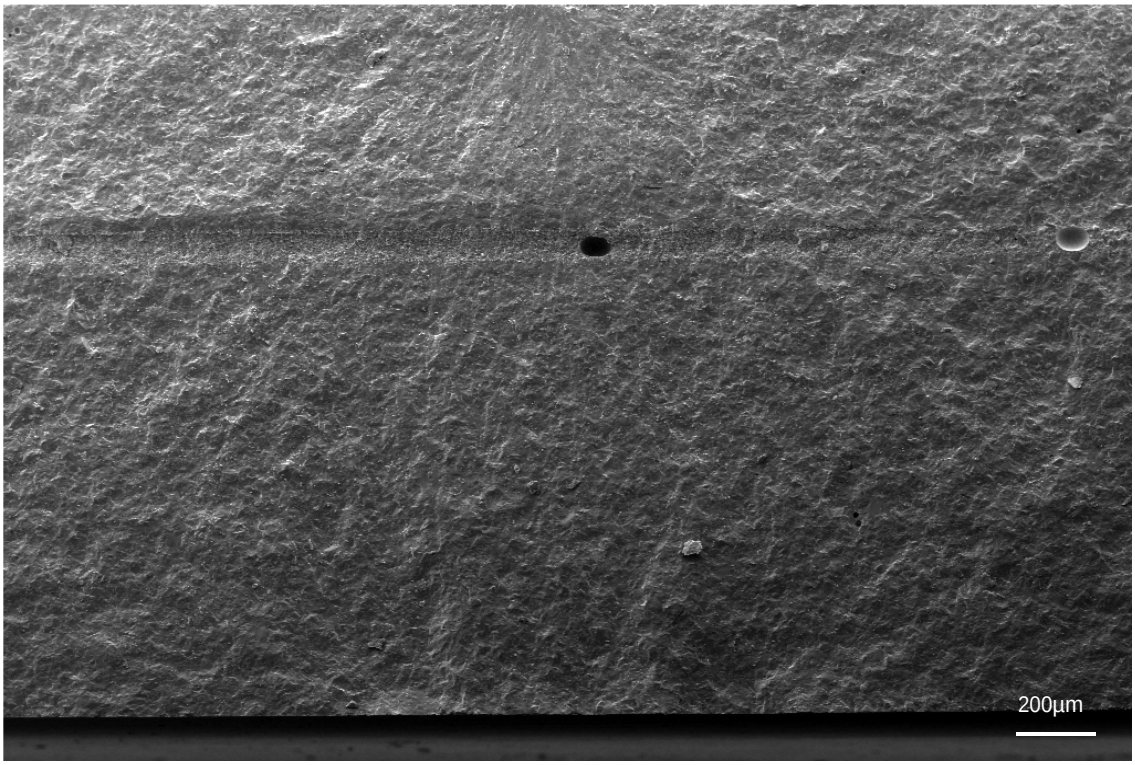


(c)



(d)

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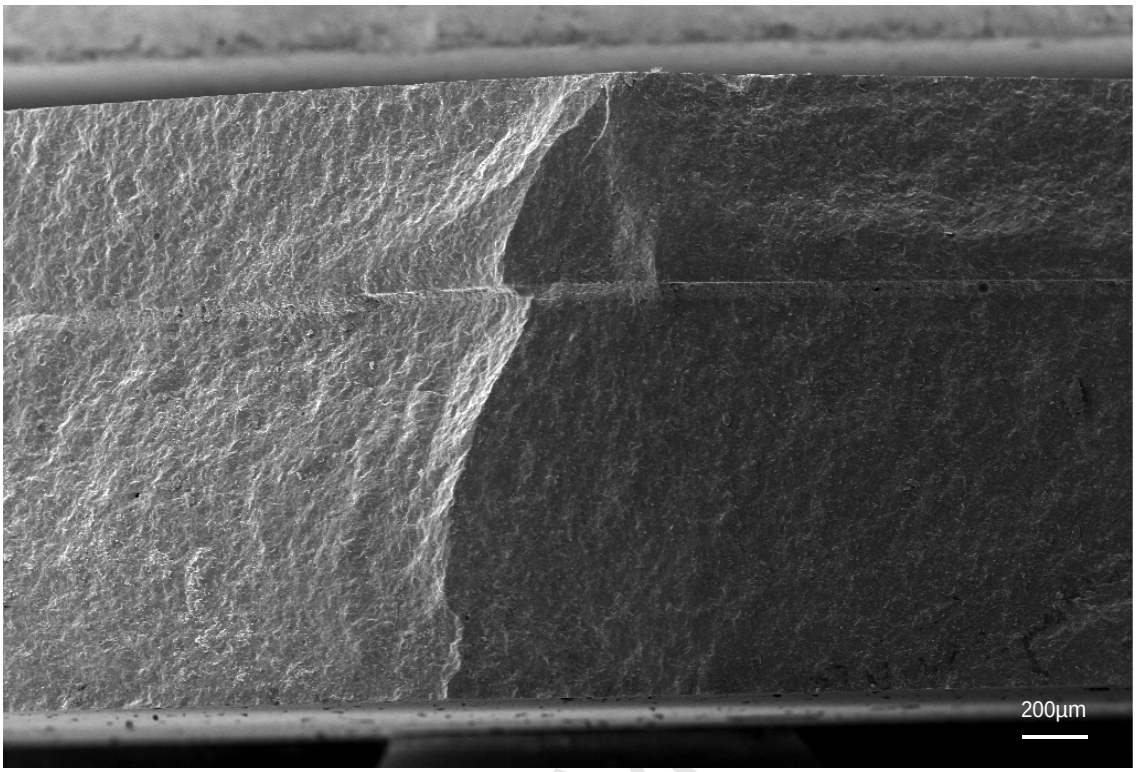

Figure 5. SEM images of the same regions of adhesively cemented ceramic laminates immediately following indentation **(5a,c)** and following 25,000 cycles of thermocycling and 30,000 cycles of cyclic loading **(5b,d).** The resin-interface was measured at between 10 and 30 µm in all micrographs, however the interface thickness demonstrated considerable regional variability in appearance. (**5a)** clearly demonstrates increased lengths of cracks parallel to the adhesive interfaces when compared with cracks in a normal direction. Crack extension in a normal direction to the adhesive interface appears to be arrested as the crack contacts the layer. Following thermo-mechanical fatigue crack extension occurred predominantly parallel to the interface **(5a,b)** and where cracks normal to the interface did extend there was evidence of a change in crack direction towards a more parallel path **(5c,d)**.

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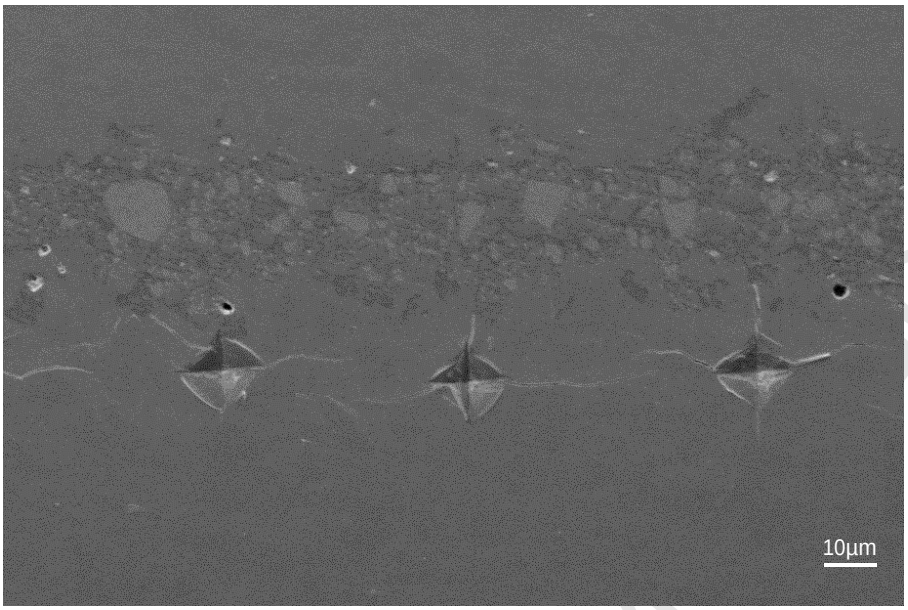
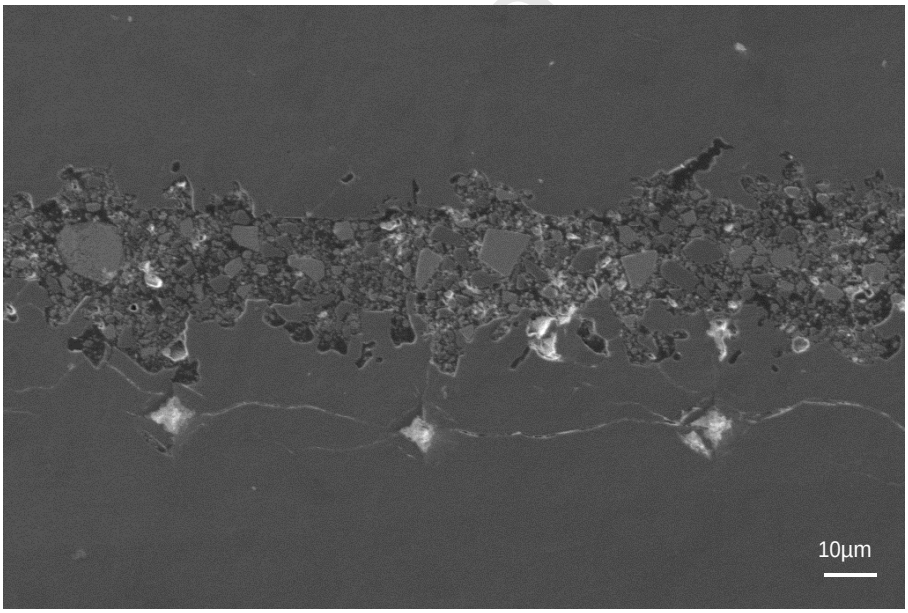
(a)



(b)



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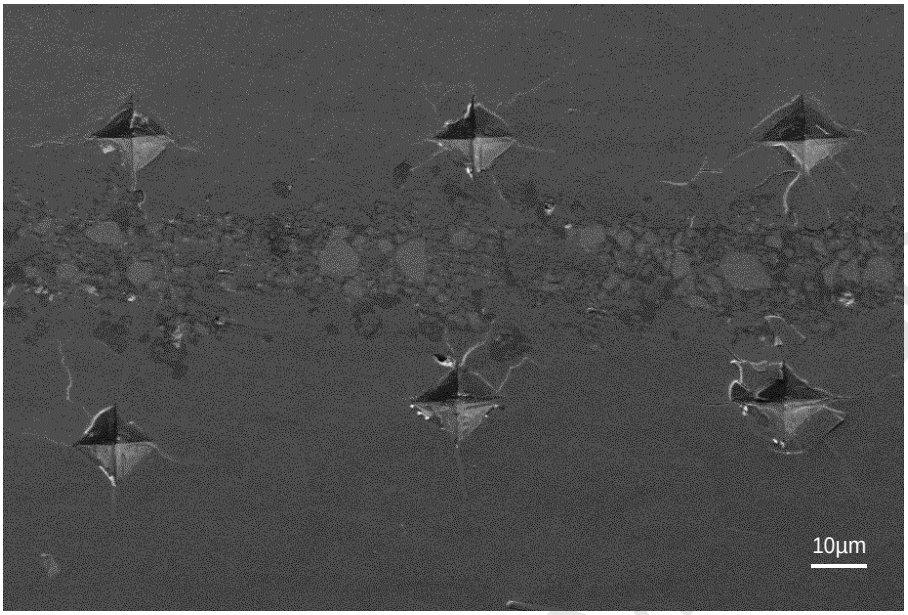
(c)



(d)

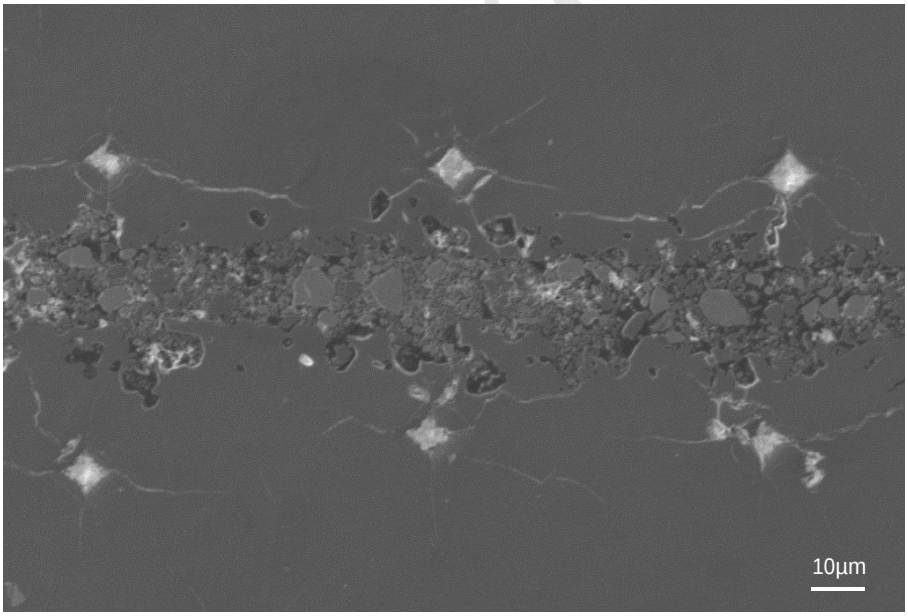


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Figure 6. (a) Scanning electron micrograph of a simulated 'concept' multilayer ceramic laminate (1.5 mm thickness) fabricated from 4 layers of Vita Mark II feldspathic ceramic adhesively bonded with Panavia 21 tested in BFS with the lower surface in maximum tension. Although there is obvious porosity in the interlayer adhesive there is clear evidence of deflection of the crack direction at multiple levels, both radially and laterally (*). There is evidence of crack arrest (□) and of Hertzian contact damage generated at the upper loaded surface (▲). **(b)** Load-deflection curve demonstrates further reduction in effective stiffness of the structure following incorporation of 4 layers.

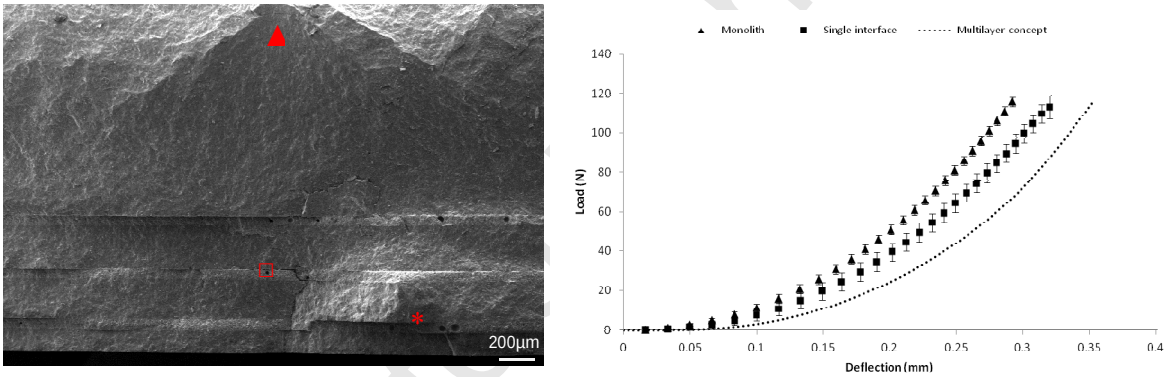

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TABLE

Table 1. Mean BFS (standard deviations), and number of fracture fragments derived from BFS testing for monolithic specimens (Group A), specimens with the adhesive interface tested in tension (Group B) and specimens with an adhesive interface tested in compression (Group C).

Group	**A**			**B**			**C**		
Mean BFS and standard deviation (MPa)	116.6 (11.0)[a]			115.2 (10.7)[a]			105.06 (12.0)[b]		
BFS range (MPa)	127.0-197.2			118.9-185.0			110.0-171.1		
Number of fracture fragments	2	3	4	2	3	4	2	3	4
	4	13	3	9	11	0	10	10	0

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