Clin Oral Invest (2004) 8:75–80
DOI 10.1007/s00784-003-0242-3

ORIGINAL ARTICLE

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Interfacial adaptation of a calcium aluminate cement used in class II cavities, in vivo

Received: 26 May 2003 / Accepted: 10 November 2003 / Published online: 6 December 2003

Abstract The aim of this in vivo study was to evaluate the interfacial marginal adaptation of a calcium aluminate cement, Doxadent (DD), and to compare it intra-individually with a resin composite, Tetric Ceram/Syntac Single-Component (TC/SS), in Class II cavities. Sixteen Class II box-shaped, enamel-bordered cavities were prepared in eight premolars scheduled to be extracted after 1 month's service for orthodontic reasons. The interfacial marginal adaptation (internal surfaces) of the restorations was evaluated by a quantitative scanning electron microscope analysis using a replica method. DD showed a statistically significant, lower degree of gap-free adaptation to enamel compared with TC/SS: 84% vs. 93%. To dentin, DD showed a significantly better adaptation than TC/SS: 72% vs. 49%. A high frequency of enamel fractures perpendicular to the margins was observed for the DD restorations, which may be explained by an expansion of the calcium-aluminate cement. It can be concluded that DD showed a better adaptation to dentin while TC/SS showed a better adaptation to enamel. The dimensional changes of DD have to be investigated before clinical use can be recommended.

Keywords Clinical · Dental restoration · Dental cement · Interfacial adaptation

Introduction

Commonly used direct restorative materials for Class I and II cavities are resin composites and amalgams [12, 13]. The disadvantages of amalgam are its mercury content and the need for macro-mechanical retentive cavity preparations. This is in contrast to resin composite, which enables micro-mechanical retention by the use of different bonding techniques. Gap formation along the internal border between the restoration and the cavity walls can enhance microleakage, which may result in postoperative sensitivity and recurrent caries. A central goal in adhesive dentistry is to obtain a permanent intimate adaptation between the cavity walls and the restorative material to secure a long-term clinical sealing of the restoration.

Doxadent (Doxa AB, Uppsala, Sweden) is a calcium aluminate cement that has been developed in Sweden and marketed recently as an alternative dental restorative to amalgam and resin composite. It received CE marking 2000. The cement represents a new kind of dental material and the clinical outcome as restorative is yet unknown since no clinical longevity study has been published. Doxadent (DD) is marketed as a "bioceramic" and a moderate amalgam preparation is recommended. Ceramics may be defined as materials that are inorganic and nonmetallic or as materials that have in common that they are compounds of metals and nonmetals [3, 20]. Thus, according to the composition and setting reactions earlier described, DD may be classified as a ceramic material, as is the case with typical zinc phosphate cement [16, 17, 18]. In contrast to other dental ceramic materials which are heat-sintered and therefore used indirectly, e.g., dental feldspathic porcelains, DD sets directly in the cavity as a consequence of an acid-base reaction.

DD is delivered as small tablets based on calcium aluminate ($3CaO{\cdot}Al_2O_3$), which also contains small amounts of ZrO_2 and SiO_2. The supplied liquid, which consists of water and small amounts of Li^+-ions, saturate the calcium aluminate tablet. An acid-base reaction is initiated during which water acts as a weak acid and calcium aluminate dissolves to form Ca^{2+}, Al $(OH)_4^-$ and OH^-. The solutes precipitate to form a gel. Gradually, the amorphous gel changes into a crystalline phase of mainly katoite $[(CaO)_3(Al_2O_3)(H_2O)_6]$. According to the manufacturer, hardening starts within 2 min and the restoration will be sufficiently hard for chewing after 60 min. The setting process is supposed to be fully completed after

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2 weeks. The material is placed in macro-mechanical retentive cavities. No conditioning or priming of the cavity walls is recommended.

The material is claimed to have a setting expansion of 0.05–0.1%, which would provide gap-free restorations according to the manufacturer. The aim of the study was to evaluate the interfacial adaptation of DD in class II restorations, in vivo. The null-hypothesis stated was that DD and the resin composite restorations show a similar degree of interfacial adaptation to enamel and dentin.

Materials and methods

Eight sound and caries-free premolars scheduled for extraction because of orthodontic reasons were used in the study. The three patients, with a mean age of 12 years (range 10–14), were offered to participate in the study at a period of time which was coincidental with the start of the study. Each patient provided informed and parental consent to participate in the study, which was approved by the Ethics Committee of the University of Umeå. The teeth were anaesthetised with 3% Citanest-Octapressin (Astra, Södertälje, Sweden). In each tooth a mesial and distal box-shaped Class II cavity was prepared with a cylindrical diamond bur in a high-speed hand-piece using copious water-cooling. No bevels were prepared and all margins were placed in enamel. Both the bucco-lingual distance of the preparation and the axial depth was 4 mm (±0.5–1 mm).

The prepared cavities of each tooth were arbitrarily assigned to one of the two experimental groups, DD or TC/SS (Tetric Ceram/ Syntac Single-Component) (Table 1). The operative field was isolated with cotton rolls and suction device. Metal matrix bands were used in combination with careful application of wooden wedges. The DD restorations were produced as follows: a tablet was partially immersed in the supplied liquid and was allowed to absorb the liquid for 5 s. Subsequently, the tablet was totally immersed in the liquid for another 5 s, and then blot dried on a piece of absorbing tissue, distributed in the cavity, and condensed with special packing instruments (Doxa AB) under maximum hand pressure. The procedure was repeated with new tablets until the cavity was filled with excess. Excess material was carved away and the patient was asked to close the lips for 10 min. Matrix band and wooden wedge were then carefully removed and the restoration was finished with polishing stones (Shofu, Kyoto, Japan). The cavities were etched with 35% phosphoric-acid (Ultra-Etch, Ultradent Products, South Jordan, UT, USA), 15 s for enamel and 5 s for dentin, followed by water rinsing for 20 s and brief air drying, allowing the wet bonding technique to be used. Syntac Single-Component (Vivadent, Schaan, Liechtenstein) was placed in two layers with a disposable brush. The first coat was applied during 20 s; the surfaces were slightly dried to remove the solvent and the resin was light cured for 20 s. A second coat was applied, dried and light cured for another 20 s. The cavity was then filled with Tetric Ceram (Vivadent) in layers not exceeding 2 mm, each layer light cured for 40 s. A light-tip was used for the first layer. The light-curing unit used was a Demetron 2000 (Demetron, Danbury, CT, USA) at an intensity of 500 mW/cm^2. Before each treatment session, the intensity was verified with a radiometer Optilux 100 (Demetron). The metal matrix band and wooden wedge were carefully removed and the restoration was finished with polishing diamonds (Drendel+Zweilling, Berlin, Germany). All restorations were made by one operator.

After 1 month functioning time the premolars were extracted. Care was taken not to damage the restorations by using an initial elevation technique, followed by careful application of forceps to the root surfaces. Immediately after extraction, the teeth were carefully cleaned in pouring water and thereafter stored in a chlorhexidine digluconate solution (Corsodyl 2 mg/ml, SmithKline Beecham, Brentford, England) for 1 week prior to preparation of the teeth for SEM.

Scanning electron microscopy

To be able to observe interfacial marginal adaptation, the extracted premolars were sectioned by use of a diamond disc (Horico; Hopf, Ringleb & Co, Berlin, Germany) in a hand-piece with copious water spray. The sectioning was performed in a buccal-lingual direction beginning at the outermost part of the restoration and continuing with five sequential sections providing six different surfaces of the restoration, from the most superficial surface to the deepest part [1]. Irregularities of every sectioned surface were levelled with fine and medium polishing discs (Soflex, 3 M Dental Products, St. Paul, MN, USA) under water spray. To remove smear layer, the tooth sections were etched with 35% phosphoric-acid (Ultra-Etch, Ultradent) for 5 s and thereafter rinsed with water for 20 s and gently dried. Immediately after conditioning, impressions were made of each section with a polyvinylsiloxane impression material (President light body, Coltene, Altstätten, Switzerland). Positive replica models were fabricated of all sectioned restoration surfaces by pouring epoxy resin (EPON embedding resin, Fluka, Buchs, Switzerland) into the negative impression. The models were prepared for scanning electron microscopy (SEM) by mounting on metal stubs and coating with gold by a standard metal evaporation technique [4]. The replicas were studied by SEM (Cambridge Stereoscan 360 ixp, LEO Electron Optics, Cambridge, UK). The interface of each restoration to enamel and dentin was evaluated at ×275 and ×1400 and supplemented when necessary with other magnifications. The quality of the interfacial marginal adaptation between the restoration material and enamel or dentin was judged according to a five-point rating scale with increasing degree of openings and breakdown (Table 2) [8]. A score of 1–3 represents acceptable adaptation with an increase of irregularities at the interface. Scores 4 and 5 represent non-acceptable adaptation with

Table 2 Interfacial breakdown scores

1.	Good adaptation, no interfacial opening, no deficiencies
2.	Slight interfacial marginal irregularities
3.	Severe interfacial marginal irregularities, no crack visible
4.	Hairline crack, wider gap with bottom visible
5.	Severe gap, bottom hardly or not visible

Table 1 Investigated materials and their manufacturers

Group	Material	Type	Batch no/Shade	Manufacturer
1	Doxadent	Calcium aluminate hydrate	SB002 / B	Doxa AB, Uppsala, Sweden
2	Tetric Ceram	Bis-GMA, uretandimetakrylat, trietylenglykoldimetakrylat, inorganic fillers, catalysts, stabilizers, pigments	B37704 / A3	Vivadent, Schaan, Liechtenstein
2	Syntac Single-Component	Maleic acid, HEMA, methacrylate modified polyacrylic acid, initiators, stabilizers, water	A 15134	Vivadent, Schaan, Liechtenstein

crack and gap formation. The scoring was performed on microphotographs at a magnification of ×275 by two operators. Quantitative data were then obtained by measuring the length of each evaluation score expressed as percentage of the total length of the examined interface. The outer proximal surface (marginal) of each restoration was not counted for, since only the interfacial border between tooth and restoration was studied. The proportion of dentin to enamel was 1:3 for both DD and TC/SS. Fractures in enamel or dentin were recorded as well.

Statistical analysis

The interfacial adaptation scores are given as relative frequencies of the total lengths of the evaluated interfaces for the two restorative materials used. The Statistical Package for Social Sciences, version 10.0 (SPSS, Chicago, USA) was used to process the data. Differences between the DD and TC/SS groups were statistically analysed by Mann-Whitney U-test and exact test (Montecarlo). The level of significance was set at $P<0.05$.

Results

The results of the interfacial marginal adaptation scores are shown in Table 3. DD showed a higher percentage of acceptable adaptation (score 1–3) to dentin compared with the TC/SS-group: 72% vs. 49%. The acceptable adaptation to enamel for the DD group compared with TC/SS was lower: 84% vs. 93% (Figs. 1, 2, 3 and 4). The gap-free adaptation to both dentin and enamel was significantly different between the two investigated materials.

Two different types of fractures, parallel and perpendicular oriented, were observed. When scoring fractures, the most superficial part of the restorations was also counted for. The mean length of evaluated enamel boarder per restoration measured (all sections counted) was 2.90±1.38 cm for the TC/SS group and 2.98±1.13 cm for the DD group. Parallel fractures were observed for TC/SS in 0.19±0.14 cm per restoration and for DD in 0.02±0.02 cm per restoration. No parallel fractures were observed in dentin. Perpendicular enamel fractures radiating from the restoration margins were frequently seen in the DD group but never observed in dentin (Figs. 5 and 6). The total amount of these perpendicular fractures registered in all DD restorations was 80 fracture sites, in contrast to three fracture sites for the TC/SS restorations.

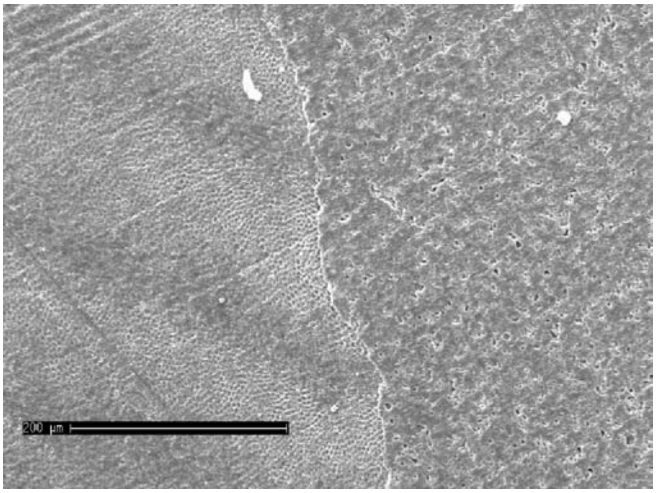

Fig. 1 Excellent interfacial adaptation in enamel of a Class II DD restoration. Original magnification ×275

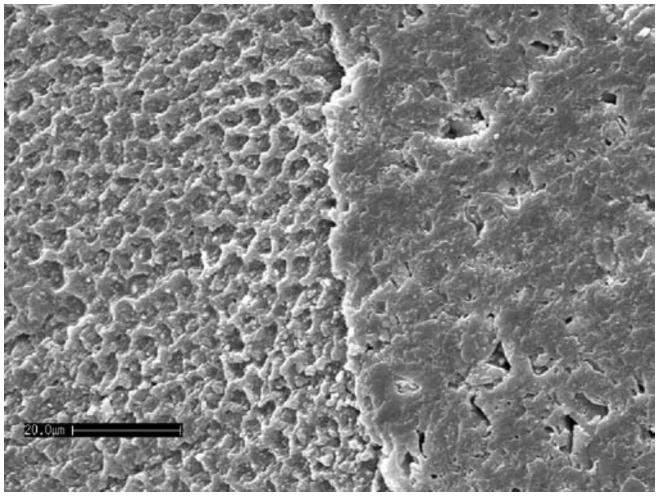

Fig. 2 Higher magnification of the interfacial adaptation shown in Fig. 1. Original magnification ×1400

Table 3 Interfacial marginal adaptation scores to enamel and dentin for Doxadent and Tetric Ceram/Syntac Single Component restorations determined as percentages of the length of interfaces examined (%) mean and standard deviation. Score 1–3: acceptable adaptation; 4–5: unacceptable adaptation. In parenthesis 25, 50 and 75 percentiles are given

Group/material		n	Scores				
			1	2	3	4	5
Doxadent	Enamel	8	62.8±28.2 (40.4/69.3/85.7)	15.7±23.8 (0.0/2.6/29.9)	5.6±7.4 (0.0/2.7/9.0)	11.2±10.6 (1.4/9.9/18.2)	4.6±7.8 (0.0/2.2/5.0)
	Dentin	8	56.3±27.1 (42.2/54.5/76.5)	11.3±19.4 (0.0/0.0/18.1)	4.4±7.7 (0.0/0.0/5.7)	14.8±11.6 (3.0/14.2/23.8)	13.3±26.2 (0.0/4.5/11.4)
Tetric Ceram/Syntac Single Component	Enamel	8	84.1±14.2 (73.4/87.8/94.1)	1.7±3.8 (0.0/0.0/0.0)	7.3±8.9 (0.3/4.5/12.0)	4.7±4.6 (0.6/3.4/7.3)	2.3±4.2 (0.0/0.2/2.8)
	Dentin	8	38.6±26.1 (17.7/38.8/54.7)	2.4±9.0 (0.0/0.0/0.0)	7.9±10.9 (0.0/2.8/12.6)	26.4±23.9 (4.8/17.1/46.5)	24.7±35.9 (0.0/7.1/44.1)

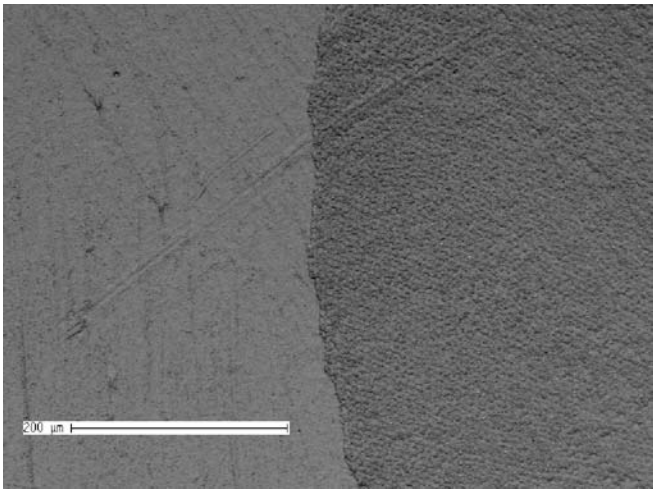

Fig. 3 Excellent interfacial adaptation in enamel of a Class II TC/SS restoration. Original magnification ×275

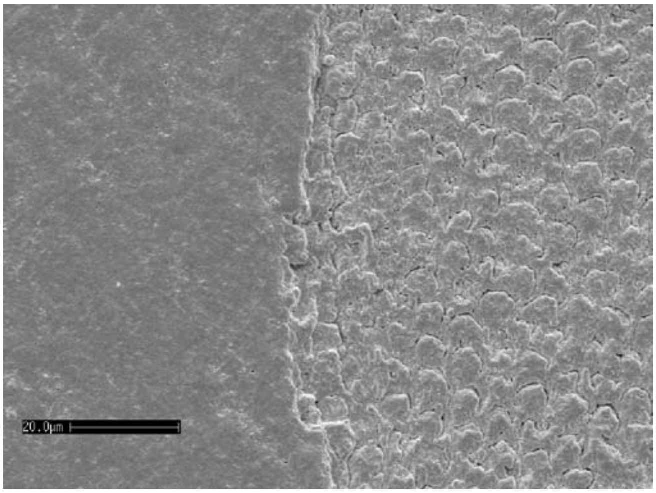

Fig. 4 Higher magnification of the interfacial adaptation shown in Fig. 3. Original magnification ×1400

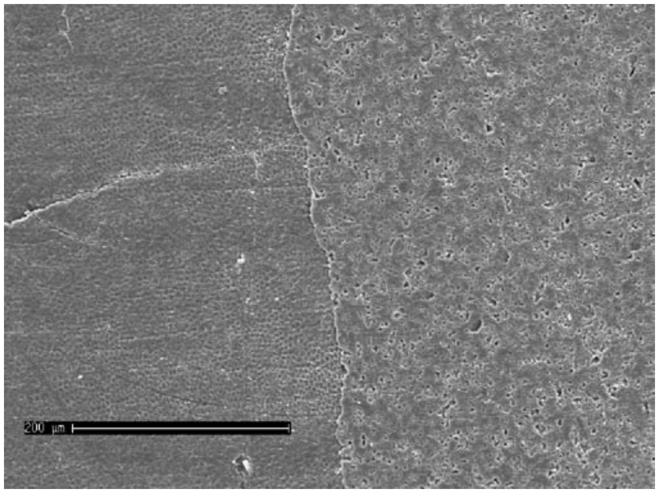

Fig. 5 Perpendicular fractures extending into enamel from the interfacial boarder between a DD restoration and enamel. Original magnification ×275

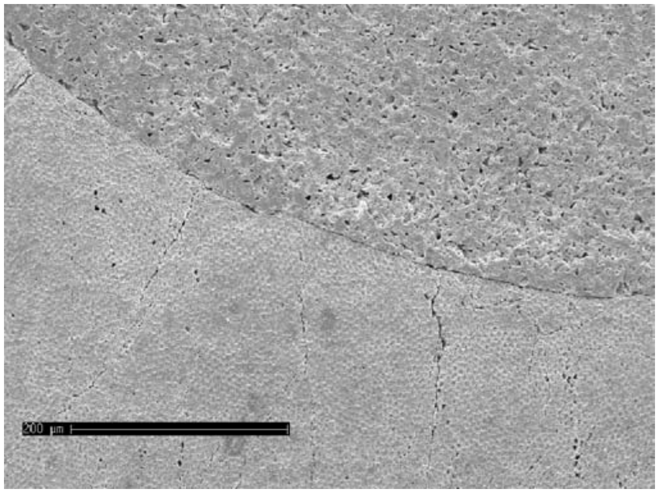

Fig. 6 Perpendicular fractures extending into enamel from the interfacial boarder between a DD restoration and enamel. Original magnification ×275

Discussion

Interfacial marginal adaptation can be studied both in vitro and in vivo. A common test is dye penetration evaluation. The experimental teeth are immersed into a dye solution and, after sectioning the teeth, the degree of dye penetration is evaluated with different types of microscope [6, 15, 21]. SEM is another widely used method for evaluating interfacial marginal adaptation. Direct observation by SEM is difficult due to presence of the liquid phase in the tooth tissues. The vacuum procedure during SEM causes artefacts such as cracks, which can look like true gap formation, if the liquid is not removed in a proper way [10]. The SEM replica method is another way to avoid artificial gap formation. Grundy showed a high degree of agreement when he compared specimens observed directly with replica models using SEM [7]. The SEM analysis used in this study is both qualitative and quantitative. A similar method has been described earlier by Roulet et al. [14] and has also been used in earlier in vivo investigations [1, 2, 4, 5, 8].

The two materials were evaluated intra-individually, which means that both restorations have been subjected to the same clinical environment concerning occlusal load, temperature and pH changes. The quality of the interfacial marginal border between the restoration and the tooth structures was judged according to an ordinal scale with increasing degree of deficiencies. This scoring system is highly dependent on the evaluator's degree of reproducibility. Calibration was therefore conducted on regular intervals both between and within the authors, and the inter-examiner reliability gave a kappa value of 0.77. The quantitative character of the analysis is presented by different adaptation scores measured on SEM pictures and transformed to percentages of the total length measured. The SEM observes a three-dimensional reality while measurements are made on a two-dimensional picture. To decrease projection distortion, as flat a surface as possible should be used. Therefore, all tooth sections were planed by the diamond wheel followed by the Sof-Lex system. The outer surface of each restoration was excluded because of its convex shape. Planed surfaces also make it easier to look at the interfacial boarder in a perpendicular angle.

Both restorative materials used in this study showed a relative high frequency of gap-free interfacial marginal adaptation to enamel. The TC/SS group (93%) showed a significantly better adaptation to enamel compared with the DD group (84%). In another study, a high degree of perfect marginal seal was observed in the enamel part of Class V resin composite restorations, which confirms the present observations [9]. However, in that study all Class V cavities were prepared with an enamel bevel, which increases the surface area of bondable enamel, and exposes the enamel rods end-on. A good adaptation of resin composite restorative systems to enamel is easier to obtain than to dentin with both hydrophilic and hydrophobic systems, as shown in earlier reports [4, 5]. Dentin is more sensitive than enamel and the adaptation obtained depends more on the adhesive system used [5]. Compared with enamel, a lower degree of adaptation was observed to dentin for both DD and TC/SS. The adaptation to dentin for DD (72%) was significantly better than for TC/SS (49%). The different degree of interfacial marginal adaptation to enamel and dentin observed between the two investigated materials could not confirm the null-hypotheses. In score 2, a distinction could be noticed between the two material groups. This can depend on the fact that one material is more brittle than the other, resulting in loss of material during cutting, or on the different application techniques of the investigated restoratives. The adaptation to dentin for resin composite materials in earlier reported studies was higher than in this study, which is probably due to the use of more effective dentin adhesive systems [5, 8]. Syntac-Single Component was chosen in this study since it was frequently used at clinics in northern parts of Sweden at the time of conducting the study. The one-bottle adhesive system used has shown low shear bond strength in another study [19]. Manhart et al. [9] investigated marginal quality and microleakage of several restorative systems in class V cavities. Within dentin cavity segments, the adhesive (Syntac Single-Component) showed statistically more leakage than a filled single bottle adhesive. Microleakage in dentin was found to be significantly lower compared with enamel sections for the investigated restorative systems. Perdigão et al. investigated the infiltration pattern of different single bonding agents into dentin, including Syntac SC, a water-based adhesive which did not penetrate and saturate the collagen network thoroughly enough [11]. Adhesive systems that thoroughly infiltrate the demineralised dentin to its full depth are more likely to produce a long-term stable bond compared with those that only partially infiltrate the demineralised zone. Vargas et al. demonstrated that single bottle adhesives produced hybrid layers of varying thickness, ranging from no discernible layer for Syntac SC to a 50-μm thick layer when using filler loaded primer systems [19]. A newer version of this dentin bonding agent in which water is combined with an organic solvent to improve the infiltration capacity has been introduced.

Vital teeth were used in the study to imitate the clinical situation as close as possible. What influence the use of vital teeth have on the results can only be speculated, since DD is a material which needs a moist environment during its setting reaction. The non-adhesive character of DD made the mesial-distal sectioning technique not useful due to the loss of several restorations during sectioning. Instead, the teeth were sectioned in a buccal-lingual direction providing five sections per tooth, in contrast to other similar studies using only two sections [2, 5, 8]. Interfacial marginal adaptation to dentin and enamel for DD is dependent on the handling of the material, like all dental restoratives. The handling characteristic of the cement is difficult because of the crumbly character of the material during packing. In a parallel SEM study (unpublished), Class II calcium aluminate hydrate restorations were performed by an experienced general practitioner who had become familiarised with the material during a short period. Compared with the present results, a decreased adaptation quality was observed, in most cases by inclusion of large air pores during packing.

Parallel fractures contiguous to TC/SS restorations were expected and have also been observed in earlier studies [1, 2, 5, 8]. They are explained as a result of the polymerisation shrinkage of the bonded TC/SS restorations. The amount of parallel enamel fractures differed greatly between the two investigated materials, being less in the DD group, (0.19±0.14 and 0.02±0.02 cm per restoration, respectively). An unexpected finding in this study was the high frequency of perpendicular enamel fractures contiguous to all DD restoration boarders (Figs. 5 and 6). The total amount of these fractures seen for the DD group was 80, in contrast to three for the TC/SS group. The non-presence of these fractures in the TC/SS group was confirmed in other similar SEM investigations of Class II restorations in vivo but using different resin composite systems than in this study [1, 2, 5, 8]. The fractures extended in a more or less perpendicular direction from the restorations into the enamel and were never observed in dentin. Part of these fractures could be followed through the different sections of the restoration, while other fractures only were present at one specific level. A possible reason for crack formation may be the use of forceps during extraction. However, each tooth received one restoration of both materials studied, which means that both restorations are exposed to the same force. Whether the expansion of DD was due to an ongoing chemical reaction and/or water absorption is unclear. It is also unclear for how long the expansion proceeds and how it will influence the dimensional changes of the cement. A continuing expansion may result in clinical consequences such as an increasing frequency of tooth and/or material fractures. Therefore, the dimensional changes of the cement should be further investigated. An expansion of DD may, on the other hand, also contribute to the relatively high degree of gap-free interfacial marginal adaptation observed. More knowledge is needed to understand how the tooth interacts with dental materials. Different mechanical properties may influence the clinical behaviour of tooth and restorative in different ways. In a previous study the calcium aluminate hydrate, being a more brittle material, was shown to have a higher modulus of elasticity compared with several resin composite materials [18]. Brittle materials lack the ability to deform plastically compared with ductile materials.

Conclusions

It can be concluded that DD had a significantly higher gap-free interfacial marginal adaptation to dentin but lower to enamel compared with TC/SS. Parallel and perpendicular fractures were observed contiguous to the investigated materials. Parallel enamel fractures occurred to a greater extent around TC/SS, and are explained by its polymerisation shrinkage. Perpendicular enamel fractures were observed around all the DD restorations and are suspected to be caused by the expansion of the material. Further investigation of the dimensional stability of DD is needed before the material can be recommended for dental use.

Acknowledgements This study was supported by Doxa AB, Sweden, and the County Council of Västerbotten, Sweden.

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