

# An *in vitro* investigation of marginal dentine caries abutting composite resin and glass ionomer cement restorations

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## Abstract

**Background:** There are a number of studies citing the primary reason for replacing auto cure glass ionomer cements was due to recurrent caries. The purpose of this study was to use an *in vitro* model to measure caries at the dentine restoration interface of bonded composite resin and auto cure glass ionomer cement restorations and to measure the amount of surface degradation occurring in the restorative materials.

**Methods:** Specimens of auto cure glass ionomer cements (Riva Fast, Fuji IX Fast, Ketac Molar Quick and Fuji VII) and bonded composite resin restorations (Ice, SDI) were placed separately at the dentino-enamel junction of 10 recently extracted human third molar teeth, disinfected and placed into the overflow from a continuous culture of *S. mutans* for two weeks. Restorations were sectioned and prepared for scanning electron microscopy (SEM) and electron probe microanalysis (EPMA). Restoration tooth interfaces were photographed and the distance from the surface of the teeth to the surface of the restorations measured. EPMA of percentage weights of calcium, phosphorous and fluoride were made outwards from the restoration surface 130µm at a depth of 10µm below the surface of the dentine.

**Results:** There were significant differences between the surface heights of composite resin, auto cure glass ionomer cements compared to teeth surfaces. Percentage weights of calcium and phosphorus levels were similar to non-demineralized dentine in the auto cure glass ionomer cement samples but there were significant reductions in mineral content of dentine adjacent to bonded composite resin restorations. Fluoride levels were mixed.

**Conclusions:** This study shows that placing a bonded composite resin restoration into dentine affords little protection to the surrounding tooth from caries attack although insignificant degradation of the restorative surface occurs. Placing a glass ionomer cement restoration into dentine protects the surrounding tooth from caries but degradation of the restoration surface occurs.

**Key words:** Dentine caries, auto cure glass ionomer cement, composite resin.

**Abbreviations and acronyms:** EPMA = electron probe microanalysis; GIC = glass ionomer cement; SEM = scanning electron microscopy.

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## INTRODUCTION

A number of papers observing the clinical performance of auto cure glass ionomer cement (GIC) have cited the primary reason for replacing these restorations have been marginal caries.<sup>1,4</sup> This is in contrast to Mount who found no marginal caries associated with GIC restorations up to five years.<sup>5</sup> In a recent paper, Tyas analysed the clinical criteria 28 general practitioners used to place approximately 100 consecutive restorations.<sup>6</sup> The failure of GIC restorations due to secondary caries ranged from 17 to 36 per cent. The design of this study did not involve any standardization of clinical assessment criteria amongst the practitioners.

Whilst the anecdotal experience of many Australian dental practitioners suggests that GICs resist caries formation in adjacent tooth structure,<sup>7</sup> a systematic review of the literature does not support this.<sup>8</sup>

Nicholson and co-workers have shown GICs capable of buffering plaque acids<sup>9,10</sup> and although the cement breaks down as a consequence of this process, this may have the effect of maintaining the pH above the levels required to demineralize the surrounding dentine.

The purpose of this study was to set up an *in vitro* model to measure the incidence of caries at the dentine restoration interface in composite resin and auto cure GIC restorations and to measure the amount of surface degradation occurring in composite resins and GICs during the period of the study.

## MATERIALS AND METHODS

### Preparation of demineralized dentine slabs

Ten recently extracted human third molar teeth that had been stored in 0.5% chloramine were cut horizontally into sections about 5mm thick so as to

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incorporate the cemento-enamel junction. Teeth were collected within the guidelines set by the Committee for the Ethics of Human Experimentation at The University of Adelaide.

The enamel and root surfaces were polished with a fine Emery disc so as to remove any remaining biofilm and cementum on the root surfaces. Five cavities of about 2mm diameter and 2.5mm deep were prepared in each tooth at the enamel dentine interface so that half the preparation was in enamel and half in dentine.

### Restoration placement prior to experimentation

Each cavity preparation was restored as follows: (1) The cavity was treated with SE Bond (Kuraray, Tokyo, Japan) according to manufacturer's instructions and a composite resin (Ice, Southern Dental Industries, Melbourne, Australia) was placed into the preparation and photo cured for 10 seconds; (2) The cavity was conditioned with GC conditioner (GC Corporation, Tokyo, Japan) for 10 seconds, washed and dried with oil-free air. Into this preparation a GIC (Riva Fast, SDI) was placed and allowed to set; (3) The cavity was conditioned with GC conditioner for 10 seconds, washed and dried with oil-free air. Into this preparation a GIC (Fuji IX Fast, GC) was placed and allowed to set; (4) The cavity was conditioned with GC conditioner for 10 seconds, washed and dried with oil-free air. Into this preparation a GIC (Ketac Molar Quick, 3M ESPE, Minneapolis, USA) was placed and allowed to set; (5) The cavity was conditioned with GC conditioner for 10 seconds, washed and dried with oil-free air. Into this preparation a GIC (Fuji VII, GC) was placed and allowed to set. Each restoration was then finished flush to the tooth surface using fine Emery discs and polished using fine Softflex discs (3M) and returned to the chloramine solution.

### Experimental method

Medium was prepared by weighing out as (w/v) 3% Tryptone Soya broth (Oxoid, Basingstoke, UK), Yeast Extract 0.5% (Oxoid Basingstoke, UK) and 20% sucrose.<sup>11</sup>

The sections were placed into a sterile glass container that was connected to the outflow from a Chemostat system. This provided a constant supply of viable *Streptococcus mutans* (strain Inbritt) grown by continuous culture. Growth was maintained under anaerobic conditions at an imposed dilution rate of  $0.1\text{h}^{-1}$  (mean generation time = 7 hours) and the pH was maintained at pH 7.4 by the automatic addition of KOH (2N). The pH of the flask containing the vials was uncontrolled and remained at ca. pH 4.5 throughout the two-week duration of the experiment.

At completion of the experiment the samples were sectioned so that each restoration was surrounded by dentine. These sections were then cut through the restoration to expose a cross-section of the restoration dentine interface. Following this each section was placed in a fixing solution containing 1.25%

glutaraldehyde, 4% sucrose, 4% paraformaldehyde in PBS at pH 7.2 (Adelaide Microscopy, Adelaide, Australia) for 12 hours, placed into a washing buffer solution, containing PBS, 4% sucrose (Adelaide Microscopy) for 1.5 hours with changes every 30 minutes. Samples were then dehydrated with ascending grades of ethanols (25% ethanol for 20 minutes, 50% ethanol for 20 minutes, 75% ethanol for 20 minutes, 95% ethanol for 30 minutes, 100% ethanol for 1 hour).

### Electron probe microanalysis (EPMA) and scanning electron microscopy (SEM) evaluation

Dehydrated specimens were placed face down on the base of cylindrical mounting blocks into which epoxy resin 100:25 (Epoxy resin LC 191: Epoxy hardener HY 956) was poured under vacuum and left to set for 24 hours at room temperature. After setting, the specimens were polished using an Abramin polishing machine (Struers, Denmark). The upper and lower surfaces of the mounting blocks were made parallel to each other using a leveling device and the samples mounted on a polisher. The surfaces in which the specimens were embedded were polished using a p80 grit silicone carbide disc (Struers, Denmark) at 150 revolutions per minute (rpm), lubricated with water for 30 seconds under a load of 100N. The specimen containing surfaces were then polished using p500 grit silicon carbide discs at 150rpm, lubricated with water under a load of 100N to grind away the surface resin.

Once the specimen containing surfaces were flat, the surfaces were further polished using diamond polishing discs (Struers) with diamond paste (Kemet, UK). First the surface was polished with 15 $\mu\text{m}$  diamond paste on a 15 $\mu\text{m}$  diamond polishing disc at 150rpm for 5 minutes at 200N, lubricated with DP-lubricant Green (Struers). After that the surfaces were polished with 3 $\mu\text{m}$  (Kemet) and 1 $\mu\text{m}$  (Kemet) diamond polishing discs and diamond pastes respectively. Both cycles were at 150rpm for 3 minutes at 200N and lubricated with DP-Lubricant Green. The surfaces were cleaned with water, air dried and viewed under a stereo microscope (Zeiss, West Germany) at 25x magnification to determine that the embedded specimens had been polished adequately.

The samples were carbon coated for SEM analysis (Philips XL30 Field Emission Scanning Electron Microscope, Netherlands) and EPMA investigation (CAMECA, SX51, France). Line scans were carried out on the specimens to measure the relative percentage weights of the following elements: calcium (Ca), phosphorus (P) and fluoride (F). Scans were conducted from the dentine restoration interface at 10 $\mu\text{m}$  below the surface of the dentine every 10 $\mu\text{m}$  to a distance of 130 $\mu\text{m}$ . Three scans were made at each location and the three readings averaged. Measurements were expressed as a relative percentage weight of the identified element as part of the total weight of the sample where the measurement was taken.

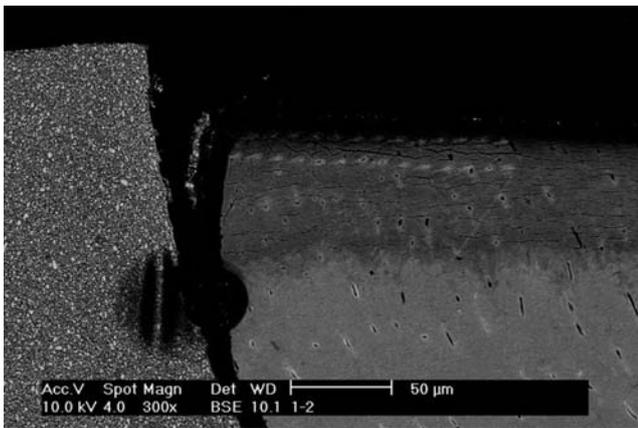


Fig 1. Typical dentine composite resin restorative interface showing dentine surfaces below the surface of the composite resin. Caries are apparent across the dentine surface up to the surface of the restoration.

### Data analysis

Since the data were not normally distributed, the Kruskal-Wallis Test was used to determine if there was a difference amongst the groups. *Post hoc* testing was used to make pairwise comparisons with no adjustment made for multiple comparisons.

## RESULTS

### Scanning electron microscopy

SEMs of the restorative dentine interfaces generally showed that restorations restored with composite resin were above the dentine surface (Fig 1) and restorations restored with glass ionomer cement were generally below the dentine surface (Figs 2–5).

There were significant differences ( $p < 0.05$ ) between the level of composite resin and all the GICs. Amongst the GICs there was significantly less ( $p < 0.05$ ) surface loss of the Riva Fast and Fuji IX Fast restorations than Ketac Molar Quick and Fuji VII restorations (Fig 6).

### Electron probe microanalysis

The graphs in Figs 7 and 8 show the mean values of calcium and phosphorus in dentine for each of the experimental groups 10µm below the surface from the

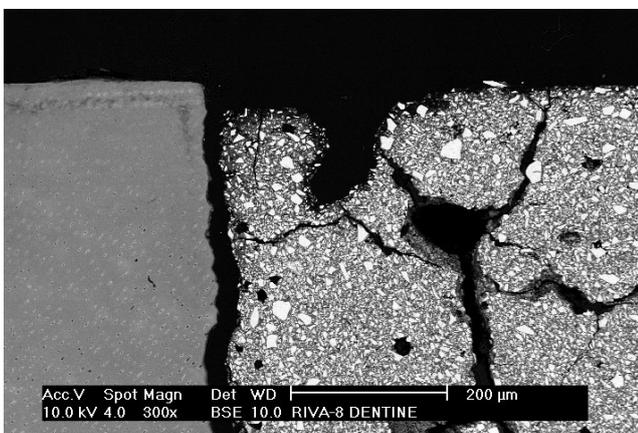


Fig 2. Typical dentine glass ionomer restorative interface restored with Riva Fast showing dentine surfaces above the surface of the GIC.

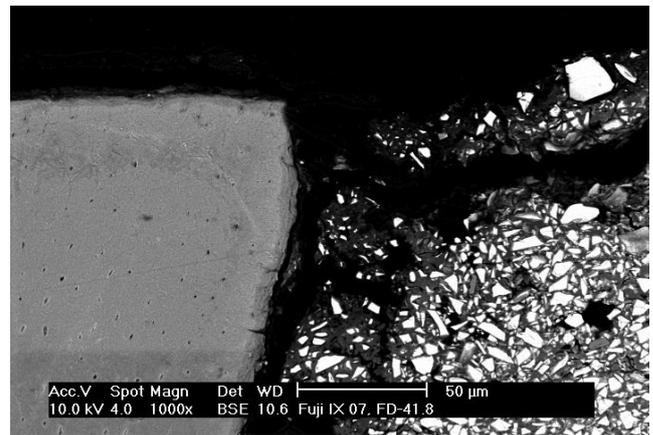


Fig 3. Typical dentine glass ionomer restorative interface restored with Fuji IX Fast showing dentine surfaces above the surface of the GIC.

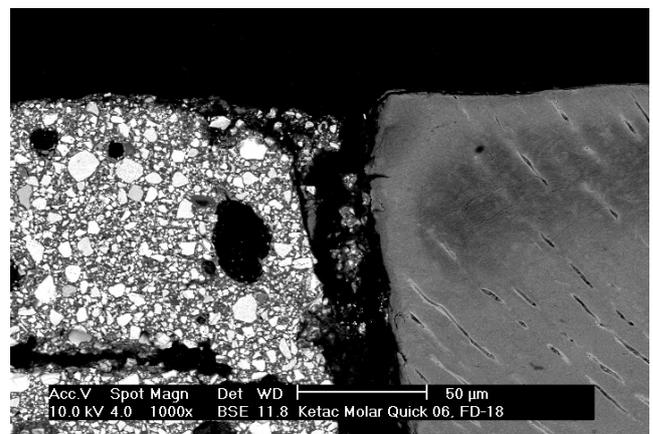


Fig 4. Typical dentine glass ionomer restorative interface restored with Ketac Molar Quick showing dentine surfaces above the surface of the GIC.

restoration dentine interface to a distance of 130µm. The percentage weights of each element are represented on the left-hand scale. There were significant differences ( $p < 0.05$ ) in loss of calcium and phosphorus between bonded composite resin and Riva Fast, Fuji IX Fast and Ketac Molar Quick but not Fuji VII, although there were no significant differences in calcium and phosphorus loss amongst the GICs as a group.

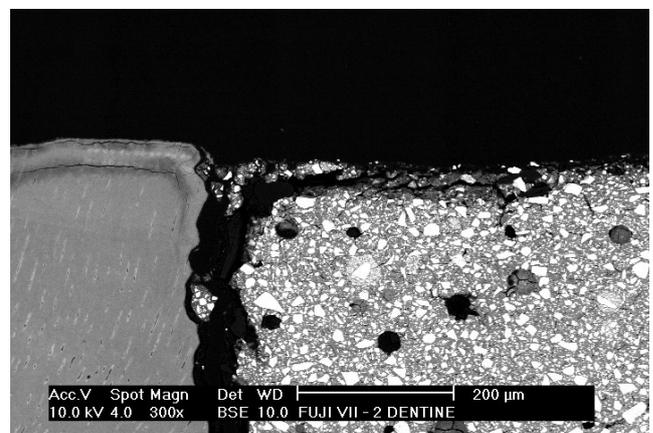


Fig 5. Typical dentine glass ionomer restorative interface restored with Fuji VII showing dentine surfaces above the surface of the GIC.

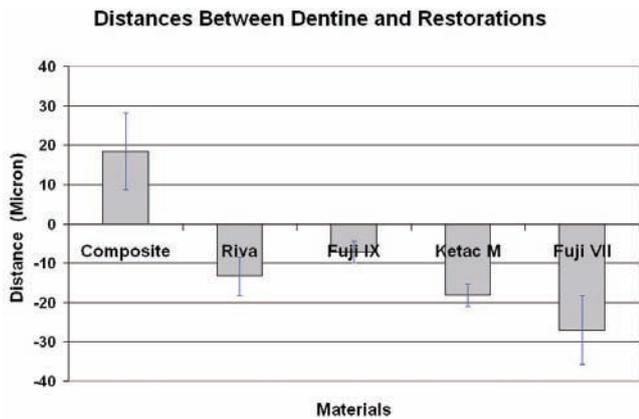


Fig 6. Graph depicting the mean values and standard deviations of the distances of the various restorative materials either above or below the dentine surface.

*Statistical analysis: Change in restoration surface height. Differences at the 0.05 level. NS = not significant. S = significant. Composite versus Riva Fast (S); Composite versus Fuji IX Fast (S); Composite versus Ketac Molar Quick (S); Composite versus Fuji VII (S); Riva Fast versus Fuji IX Fast (NS); Riva Fast versus Ketac Molar Quick (S); Riva Fast versus Fuji VII (S); Fuji IX Fast versus Ketac Molar Quick (S); Fuji IX Fast versus Fuji VII (S); Ketac Molar Quick versus Fuji VII (NS).*

There were significant differences in fluoride levels ( $p < 0.05$ ) in the surrounding dentine between composite resin and Fuji IX Fast but not Riva Fast, Ketac Molar Quick or Fuji VII. There were significant differences ( $p < 0.05$ ) between Riva Fast and Fuji IX Fast and Fuji VII but not composite resin or Ketac Molar Quick. There were significant differences ( $p < 0.05$ ) between Ketac Molar Quick and Fuji IX Fast but not composite resin, Riva Fast or Fuji VII (Fig 9).

## DISCUSSION

It is unlikely there would be a destruction of the dentine surface over the two-week period of the study.

### Dentine Calcium Content (10 um sub surface)

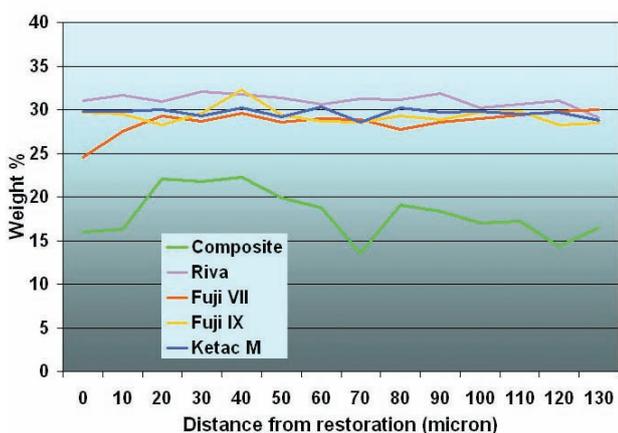


Fig 7. EPMA graph of the average percentage weights of calcium 10um below the surface from the restoration interface of each material out to 130um.

*Statistical analysis: Calcium. Differences at the 0.05 level. NS = not significant. S = significant. Composite versus Riva Fast (S); Composite versus Fuji IX Fast (S); Composite versus Ketac Molar Quick (S); Composite versus Fuji VII (NS); Riva Fast versus Fuji IX Fast (NS); Riva Fast versus Ketac Molar Quick (NS); Riva Fast versus Fuji VII (S); Fuji IX Fast versus Ketac Molar Quick (NS); Fuji IX Fast versus Fuji VII (NS); Ketac Molar Quick versus Fuji VII (S).*

### Dentine Phosphorus Content (10 um sub surface)

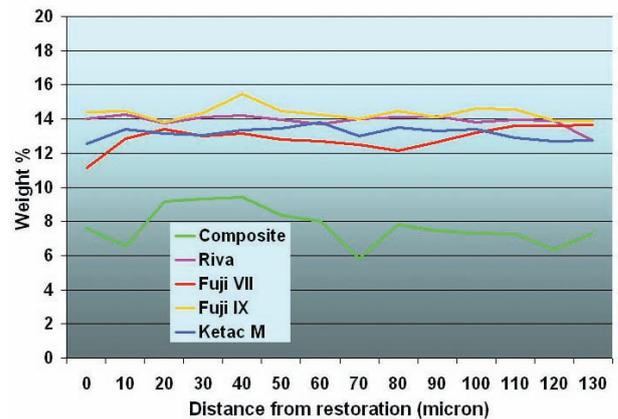


Fig 8. EPMA graph of the average percentage weights of phosphorus 10um below the surface from the restoration interface of each material out to 130um.

*Statistical analysis: Phosphorus. Differences at the 0.05 level. NS = not significant. S = significant. Composite versus Riva Fast (S); Composite versus Fuji IX Fast (S); Composite versus Ketac Molar Quick (S); Composite versus Fuji VII (NS); Riva Fast versus Fuji IX Fast (NS); Riva Fast versus Ketac Molar Quick (NS); Riva Fast versus Fuji VII (NS); Fuji IX Fast versus Ketac Molar Quick (NS); Fuji IX Fast versus Fuji VII (NS); Ketac Molar Quick versus Fuji VII (NS).*

Surface loss of the dentine surrounding the composite resin restorations can be attributed to the demineralization of the surface dentine and collapse following dehydration of the specimens for SEM and EPMA. As the percentage weights of calcium and phosphorus adjacent to the GIC restorations were similar to sound dentine it can be assumed that surface collapse due to dehydration was less around the GIC restorations. A surface loss of about 10um for Riva Fast and Fuji IX Fast and losses of about 20um for Ketac Molar Quick and Fuji VII over the two-week period of this study has clinical implications that may need to be considered when placing GIC restorations. Some SEMs of Fuji VII restorations showed a slight

### Dentine Fluoride Content (10 um sub surface)

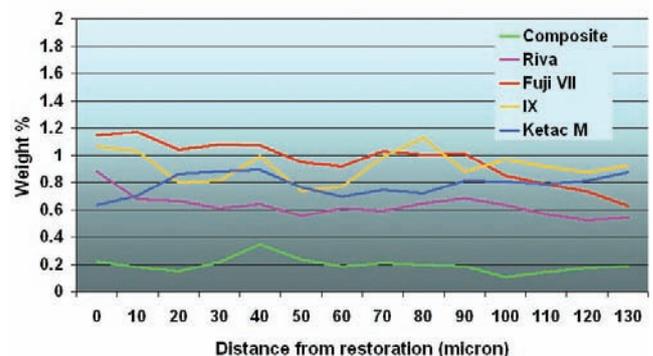


Fig 9. EPMA graph of the average percentage weights of fluoride 10um below the surface from the restoration interface of each material out to 130um.

*Statistical analysis: Fluoride. Differences at the 0.05 level. NS = not significant. S = significant. Composite versus Riva Fast (NS); Composite versus Fuji IX Fast (S); Composite versus Ketac Molar Quick (NS); Composite versus Fuji VII (NS); Riva Fast versus Fuji IX Fast (S); Riva Fast versus Ketac Molar Quick (NS); Riva Fast versus Fuji VII (S); Fuji IX Fast versus Ketac Molar Quick (S); Fuji IX Fast versus Fuji VII (NS); Ketac Molar Quick versus Fuji VII (NS).*



**Fig 10.** Glass ionomer cement restoration placed nine years earlier. Observation of the lesion shows it is due to loss of the restoration rather than breakdown of associated dentine. Furthermore, surrounding dentine resisted probing, unlike a carious lesion.

subsurface breakdown. This may be attributed to the higher liquid powder ratio this material has compared to the other GICs.

The *in vitro* caries model used in this study maintained pH levels of about 4.5, causing significant surface degradation of the GIC surfaces. This may explain clinically why GIC restorations appear to be lost more readily interproximally where lower pH levels can be maintained than restorations on occlusal, buccal or lingual surfaces subject to the effects of saliva flow where sustained drops in pH are less likely to occur.

The EPMA data show that GICs are capable of substantially reducing demineralization of dentine adjacent to the cavo restoration interface. Percentage weights of calcium and phosphorus adjacent to GICs were similar to sound dentine yet percentage weights adjacent to bonded composite resin restorations were about half that. Furthermore, cavities restored with GIC have this protection extended out to at least 130µm from the cavo restoration surface.

The release of fluoride from glass ionomer cements is greater at lower pH than at higher pH.<sup>12</sup> The pH levels experienced by the specimens in this study (pH 4.5) would have been conducive to fluoride release. Other laboratory studies have confirmed the ability of fluoride to prevent or inhibit demineralization of dentine adjacent to glass ionomer cement restorations.<sup>13-15</sup>

However, the effects of fluoride on preventing demineralization in this *in vitro* caries model are not clear cut. One glass ionomer cement material tested, Riva (SDI), was associated with lower fluoride uptake in the dentine than the other glass ionomer cement materials, but it was also associated with quite high resistance to demineralization as evidenced by the high calcium and phosphorus levels in the adjacent dentine. The possibility exists that other ionic species eluted from the material may have conferred additional protection. In addition, fluoride can inhibit biofilm

formation by *S. mutans* and other bacterial species.<sup>16</sup> The extent of this happening in this study is not known.

Another protective factor associated with glass ionomer cement is its excellent buffering capacity in an acidic environment.<sup>9,10</sup> The loss of surface material may have helped in maintaining a higher pH in the vicinity of the restorations.

Irrespective of the causal factors protecting surrounding dentine from caries, these findings support the clinical impressions of Australian dentists who observed little caries activity associated with GIC restorations,<sup>7</sup> although loss of a GIC restoration could be interpreted by a clinician as recurrent caries at that site (Fig 10).

The results of this study show that placing a bonded composite resin restoration into dentine affords little protection of the surrounding tooth from caries attack although no destruction of the restorative surface occurs. Placing a glass ionomer cement restoration into dentine protects the surrounding tooth from caries but degradation of the restoration surface occurs.

As the dental profession moves away from the surgical management of caries to a pharmacologically-based model, the findings of this paper raise questions concerning the use of bonded composite resin as a restorative material abutting dentine. This study also identifies the problems of surface breakdown associated with GIC as a restorative material particularly when used in interproximal restorations where sustained periods of low pH are likely to occur. Manufacturers of GICs may wish to address this aspect of the restoration's clinical performance.

Resin modified GICs may offer surrounding protection from caries without surface breakdown of the restorative material and this is currently being evaluated. Alternatively, surface treatment of auto cure GICs after placement may prevent surface degradation and offer enhanced protection for surrounding tooth structure.

## CONCLUSIONS

In this study, composite resin and auto cure GIC restorations were placed into dentine followed by two weeks in an *S. mutans* inoculated growth medium. Bonded composite resin restorations afforded little protection to the surrounding tooth from *in vitro* caries attack although no degradation of the restorative surface occurred. Auto cure GIC restorations protected the surrounding tooth from *in vitro* caries out to a distance beyond 130µm but degradation of the restoration surfaces occurred.

## ACKNOWLEDGEMENTS

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## DISCLOSURE

The corresponding author was associated with the development of Fuji VII and has a financial interest in this product.

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