Acta Odontologica Scandinavica, 2012; 70: 471–477

informa
healthcare

ORIGINAL ARTICLE

Coating glass-ionomer cements with a nanofilled resin

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Abstract

Objectives. The objective of this study was to investigate the effect of a nanofilled resin coat on the flexural strength (FS) and the early wear (after 50 000 and 200 000 cycles) of the glass-ionomer cements Fuji IX GP Extra (FIXE) and Ketac Molar Aplicap (KM). ***Materials and methods.*** Specimens were prepared and half of them were coated with G-Coat plus. The uncoated specimens were used as controls. Flexural strength ($n = 10$) was evaluated after 24 h using a 3-point bending test on a universal testing machine (ISO 9917-2). Wear ($n = 20$) was evaluated after 50 000 and 200 000 cycles using the ACTA wear machine. One-way, two-way ANOVA and Tukey post-hoc tests were used to analyze differences in FS and wear. ***Results.*** For FIXE the coat significantly increased the FS and the wear along the two time spans. KM did not show a significant difference in FS with the coat. Improvements in wear were observed only after 50 000 cycles. ***Conclusion.*** Based on these laboratory results, it is concluded that G-coat Plus is indicated in association with GP IX Extra with the aim to improve the mechanical properties of the former. However, this study is limited to a short-term observation.

Key Words: *: glass ionomer cement, flexural strength, surface protection, wear*

Introduction

Glass-ionomer cements (GIC) are widely used in preventive and pediatric dentistry as they release fluoride and have chemical adhesion to dental structures [1]. This latter property is believed to be beneficial in terms of resistance to degradation of the interface between GIC and dental substrate [2], which also promotes advantages regarding microleakage and secondary caries formation [2]. Moreover, GIC have an expansion coefficient similar to the tooth structure, biocompatibility, chemical setting reaction and fluoride release/uptake, attributing a preventive character to them [1–5]. In the late 1990s, the conventional GIC were overtaken by the highly viscous GIC, which have a faster setting time and notably higher strengths [6]. They are mainly used in single surface restorations and in non-carious cervical lesions. They are also the material of choice for the atraumatic restorative treatment (ART) and for restorations that are at risk of early water contamination [7].

Some GIC's characteristics may impose limits to its indication for clinical use. The long setting reaction time and the water sensitivity during setting reaction (first 24 h) associated with a relatively low fracture strength and high occlusal wear rate in comparison to amalgam and modern resin materials are considered drawbacks of conventional GIC [6,8–10]. The setting reaction of conventional glass-ionomers needs water to form the polyacrylate matrix. This initial stage, which is the clinical setting reaction, occurs within the first 10 min after mixing. The second stage, involving the release of the calcium and aluminum cations within the matrix, is a slower continuation of the acid–base reaction that lasts 24 h [11]. During the first reaction, the material is very sensitive to water uptake, while during the second step the material is very susceptible to dehydration. This short-term sensitivity to water can result in lower strength of the surface and, as a consequence, in low wear resistance and low early flexural strength. This in turn restricts the full potential of GIC for dental applications [8,12–14]. To avoid problems within the first 24 h, it is recommended to protect the GIC's surface. Copal cavity varnish, light cured bonding resins, petroleum jelly, cocoa butter, or even nail varnish

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(Received 16 November 2010; revised 23 June 2011; accepted 3 August 2011)

ISSN 0001-6357 print/ISSN 1502-3850 online
DOI: 10.3109/00016357.2011.639307

can be used as such [11,15–18]. The longer this protective material is in contact with the restoration, the smaller the chance that the material will have its mechanical properties reduced [2]. Recently, a new generation of coating for GIC has been introduced. The main difference between this material (G-Coat plus) and the previous generations of coat for GIC is the content of nanofiller particles. A previous study showed no effect of the G-coat plus on the fracture toughness of Fuji IX, while fracture toughness of a resin-modified glass-ionomer (Fuji II LC) was positively affected [19]. On the other hand, it is expected that the conventional GIC, being more sensitive to water, would be more positively affected by the coat.

In contrast to single-surface, the success rate of multi-surface ART restorations is still not satisfactory [20]. Gross marginal defects due to wear and fracture of the restoration are among the most common failure reasons. It is interesting to know if the G-coat plus would have a positive influence on the fracture strength and the early wear on the most commonly used highly viscous GIC.

The aim of this study was to investigate the flexural strength and the early wear resistance of highly viscous GIC coated and uncoated with a nanofiller-containing fluid resin. The null-hypotheses tested were that there is no difference in (1) flexural strength and (2) wear between coated and uncoated specimens.

Materials and methods

The materials used in the study, their manufacturers, batch numbers, shade, and expiration dates are shown in Table I. The GIC were supplied in capsulated form and activated/mixed according to manufacturers' instructions.

Flexural strength

The flexural strength (FS) was determined according to the ISO Standard ISO9917-2 using 25 × 2 × 2 mm bar-shaped specimens. Twenty specimens were made for each material. The GIC were mixed by a Rotomix (3M ESPE) and set at room temperature for 10 min. After setting, the specimens were removed from the molds and randomly assigned in two groups ($n = 10$). The control group did not receive any surface protection. The experimental group had one surface coated with G-coat plus and light cured for 20 s in three sites along the bar (Elipar Highlight, 3M ESPE, power density of 750 mW/cm^2). Both groups were stored in liquid paraffin at 37°C [14]. After 24 h the height and width of the specimens were recorded with a digital caliper. The specimens were subjected to a 3-point bending test on a universal testing machine (Hounsfield Ltd, Redhill, Surrey, UK) at a crosshead speed of 0.75 mm/min. The FS was calculated with the following equation:

$$\mathrm{FS} = \frac{3Fl}{2wh^2}$$

where F is the load at fracture, l the distance between the supports (20.0 mm), w the specimen width, and h the specimen height. For the experimental group, the coated surface was placed opposite to the applied load.

Wear

Three-body wear was evaluated with the ACTA wear machine [21]. In short, this device consists of two motor-driven cylindrical wheels rolling over each other with a surface slip of 15% inside a bowl containing a third body medium, consisting of a slurry of rice and millet seed shells (pH = 7). Fuji IX GP Extra and Ketac Molar were mixed by a Rotomix (3M ESPE). For this experiment, new specimens were prepared to fit the wear machine. A total of eight specimens were used for this experiment, i.e. two experimental specimens and two control specimens of each material. The specimens were placed into one of these wheels and set at room temperature for 10 min. The specimen wheel consisted of 10 compartments, each containing ~ 1 g of cement. After setting, the specimens are removed from the mold and repositioned with cyanoacrylate ester (Super Bonder, Henkel Loctite Products, Rocky Hill, CT). The wheel with the specimens was kept at 37°C and 100% humidity for the whole period of the test. A test run consisted of 200 000 cycles of the specimen wheel (taking ~ 55.5 h) at a rotational speed of 1 Hz to simulate the chewing frequency [6].

The surface finishing was done 10 min after mixing by placing the specimen wheel on the fixed axis of the wear machine and a diamond-grinding wheel on the other axis, which was allowed to move slowly into the direction of the specimen wheel. The grinding procedure was carried out under water at 37°C with

Table I. List of the materials tested, manufacturers, batch numbers, shade, and expiration dates.

Material	Code	Manufacturer	Batch/shade/exp date
GC Fuji IX GP Extra	FIXE	GC Europe (Leuven, BE)	0609261/A3/2008-09 and 0812151/A2/2010-12
Ketac Molar (Aplicap)	KM	3M ESPE (Seefeld, GE)	70201103911/A3/2009-08 and 360334/A3/2011-09
G-Coat Plus	C	GC Europe (Leuven, BE)	0610241/-/2008-10 and 0809131/-/2010-09

grinding wheels with grits up to 1000. The grinding wheel was replaced by the 'antagonist wheel', which was pressed against the specimen wheel by a spring force of 15 N and rotated with a surface speed, which differed 15% from that of the specimen wheel (15% slip).

A wear-in run of 10 000 cycles was performed to adapt the two wheel surfaces and the worn-in surfaces were measured by profilometry (A1 - Baseline). A total of four specimens, two from Fuji IX GP Extra (FIXEC) and two from Ketac Molar (KMC), were coated with G-Coat Plus (specimens with C as last letter) and light cured for 20 s 3-times along the specimen (Elipar Highlight, 3M ESPE). After coating, the profile was measured again (B1) to establish the profile of the coating. Then, a wear run of 50 000 cycles was initiated. After 50 000 cycles, the profile was re-measured (C1) and followed by another wear run of 150 000 cycles, giving a total of 200 000 cycles. At the end of this run, which was 4 days after the preparation of the specimens, the final profiles were measured (D1). The procedure is schematically depicted in Figure 1. After completion of the sequence A1 → D1, the whole procedure was repeated (A2, B2, C2, and D2). For this part of the experiment, a fresh coating layer was applied again on the worn surfaces of FIXEC and KMC before the wear runs. Freshly prepared slurries were used for all three sequences.

After each run, 10 tracings were taken at fixed positions on the worn surface of each pair of specimens (PRK profilometer No. 720702, Perthen GmbH, Hannover, Germany) so the loss of material (μm) could be measured. The positioning of the specimen wheel and the direction in which the surface tracings were taken are shown in Figure 2.

The wear (in μm) after 50 000 cycles was calculated by subtracting the profile after the wear-in run of 10 000 cycles (A) from the profile after 50 000 cycles (C). The wear (in μm) at 200 000 cycles was calculated by subtracting the profile after the wear-in run of 10 000 cycles (A) and the profile after 200 000 cycles (D). It should be noted that after completion of the first sequence (A1 → D1) a fresh coating layer was applied, before measuring B2, C2 and D2. The coating thickness was calculated by subtracting the profile after the wear-in run of 10 000 cycles (A) from the profile after coating (B).

Scanning electron microscopy (SEM)

Representative cross-section of the specimens from the FS test were selected for SEM images. Replica impressions were taken using President™ JET light-bodied (Coltene-Whaledent, Alsätten, Switzerland) as the impression material. Replicas were made with epoxy resin (Epo-thin® Buehler Ltd, Lake Bluf, IL, USA) mounted on aluminum stubs, gold-sputtered and examined using a scanning electron microscope (XL 20: Philips, Eindhoven, The Netherlands) in order to illustrate the interaction between the GIC and G-Coat Plus.

Statistical analysis

Two-way ANOVA and Tukey *post-hoc* test were used to test differences in wear of the GIC with or without the coat and the effect of time/experiment. The

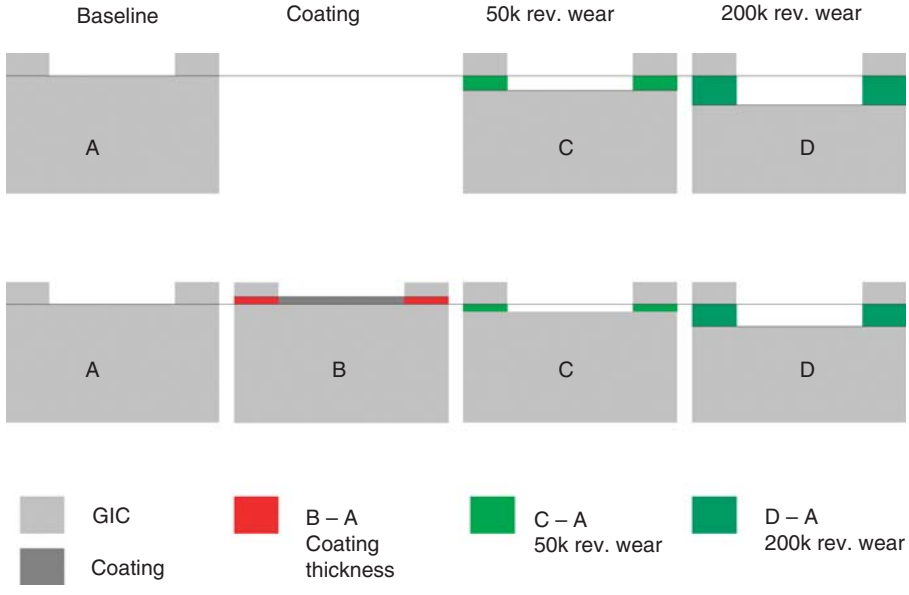

Figure 1. Top: Schematic representation of the wear after 50 000 cycles (C–A) and after completion of 200 000 cycles (D–A) of the uncoated specimens. Bottom: Schematic representation of coating thickness in μm (B–A), wear after 50 000 cycles (C–A), and after completion of 200 000 cycles (D–A) of the coated specimens after the initial coat.

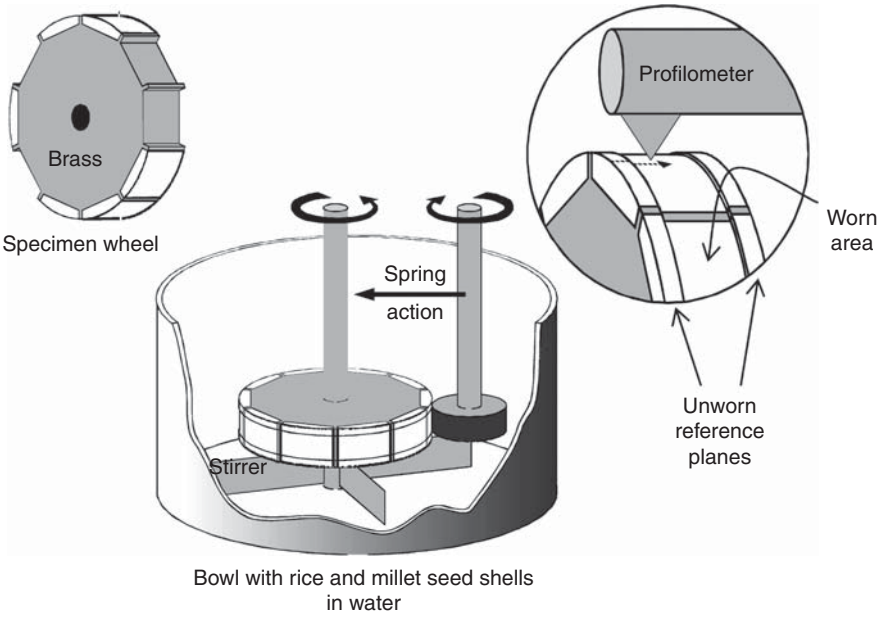

Figure 2. Schematic representation of the various steps in the wear experiment. Upper left: Specimen wheel with specimens cured and glued in the wheel. Middle: Antagonist wheel and specimen wheel rolling over each other in a slurry of rice and millet seed shells to produce 3-body (abrasive) wear. For standard experiments the antagonist wheel is pressed against the specimen wheel by a spring force of 15 N, and the surface velocity is adjusted to obtain a slip rate of 15%. The rotational speed of the specimen wheel is fixed at 1 Hz. Upper right: Profile tracings from one unworn reference to the other across the worn surface to measure wear.

flexural strength was analyzed with one-way ANOVA and Tukey *post-hoc* test. Pearson correlation test was performed to investigate a possible correlation between the tests, for both tests. The software used was Sigma Stat 3.1 (SPSS inc., Chicago, IL).

Results

The flexural strength data are summarized in Table II. One-way ANOVA ($F = 31.2$; $p < 0.001$) and *post-hoc* Tukey's test ($p < 0.01$) showed a significant improvement on the flexural strength of Fuji IX GP Extra when the G-Coat Plus was applied. For the Ketac Molar no differences were observed between the groups with and without the coat.

The wear data are summarized in Table III and graphically depicted in Figure 3. The mean coating thickness (SD) for Fuji IX GP Extra was 25.3 (4.7) μm (50 000 cycles) and 48.4 (10.6) μm (200 000 cycles) and for Ketac Molar 26.1 (5.5) μm (50 000 cycles) and 42.4 (9.9) μm (200 00 cycles). Two-way ANOVA showed that time ($F = 4327.2$; $p < 0.001$) and the different GIC with or without coat ($F = 319.4$; $p < 0.001$) had a significant effect on wear. Tukey's *post-hoc* test ($p < 0.05$) showed that wear decreased significantly after 200 000 cycles comparing with 50 000 cycles for all groups. The coated specimens showed significantly lower wear than the uncoated specimens, except for the Ketac Molar groups after 200 000 cycles, which showed the same wear irrespective of the coating application.

Table II. Mean (SD) flexural strength (F_s) in MPa for investigated materials.

	Flexural strength (F_s)	
Material	without G-Coat Plus	with G-Coat Plus
FIXE	20.2 (4.1)[c]	34.9 (6.7)[b]
Ketac Molar	44.1 (5.6)[a]	36.1 (5.8)[a,b]

Different letters indicate a statistically significant difference ($p < 0.01$).

Pearson correlation test showed no correlation between the materials in both tests. The SEM micrographs showed that there is micro-mechanical interlocking between the tests, for both tests and the coat (Figures 4 and 5).

Discussion

The null hypothesis, stating that there was no difference in flexural strength between coated and uncoated specimens, was rejected. Ketac Molar showed no difference in FS, irrespective of the coat application, but Fuji IX Extra showed statistically significant difference in FS depending on the use of the coat. Regarding the wear, the null-hypothesis was also rejected. There was a statistically significant difference in the use of the coat for both materials with all experiments, except for the one of Ketac Molar after 200 000 cycles.

Coating specimens with G-coat results in improved material properties in the laboratory. Despite some beneficial properties, the poor wear-resistance and the

Table III. Wear in μm after 50 000 cycles (C–A) and after completion of 200 000 cycles (D–A) at two time spans. Negative values indicate that there is still presence of coating. Standard deviations in parentheses are from 10 tracings at different locations on the worn surfaces.

		Material			
	Cycles	Ketac Molar	Ketac Molar C	FIXE	FIXEC
(C–A)	50,000	18 (2)^{bB}	7 (2)^{aB}	22 (2)^{bB}	6 (2)^{aB}
(D–A)	200,000	83 (3)^{cD}	72 (4)^{bD}	93 (2)^{dD}	54 (5)^{aD}
			recoat		recoat
(C–A)	50,000	7 (1)^{cA}	−5 (15)^{aA}	9 (1)^{cA}	0 (3)^{bA}
(D–A)	200,000	57 (2)^{bC}	59 (6)^{bC}	70 (3)^{cC}	46 (4)^{aC}

Different lowercase letters indicate a statistically significant difference between the materials within the same experiment (in the same line) ($p > 0.01$). Different uppercase letters indicate a statistically significant difference between the experiments (in the same column) within the same material ($p < 0.05$).

low fracture strength are still the most challenging properties of GIC, which limit their use to low-stress-bearing areas [22,23]. Using a coat might help to overcome these problems. Besides the coat, the long setting time can have a great influence on the sensitivity to water uptake [15]. To avoid differences in this characteristic Fuji IX GP Extra was selected for this study as its setting time (2 min and 30 s) is the same as for Ketac Molar, which is much faster than the conventional Fuji IX GP (4 min and 30 s).

The wear rate of the uncoated specimens of Ketac Molar and Fuji IX GP Extra were consistent with those previously published [6]. With the exception of Ketac Molar after 200 000 cycles all groups showed significant differences between the coated and uncoated specimens. For all materials, with or without coat, a decrease in wear rate as a function of time was observed. After 200 000 cycles the specimens showed a lower wear rate than after 50 000 cycles. The re-application of the G-Coat Plus reduced the wear rate for Fuji IX GP Extra after 200 000 cycles, but for Ketac Molar the wear rate was only lower in the short-term experiment. One can say that this might be due to the fact that the coating did not chemically interact with Ketac Molar but only superficially penetrated the material, forming an outer layer (Figure 4), which was totally removed in the longer wear experiment (200 000 cycles). This hypothesis is supported by Figure 5, in which the GIC detached from the coating despite the existence of micro-mechanical interlocking.

It has been shown that the 3-point bending test can represent the clinical situation [24], e.g. when the opposing cusp of the opposing tooth exerts forces onto the restoration. The ISO 9917 standard for dental water-based cements included only compressive strength as a mechanical property. The flexural strength is included in the ISO 9917-2 standard (for light-activated dental water-based cements). These standards were designed at a time when conventional GIC were not considered for use in occlusal and approximal cavities [25] and highly viscous GIC were not developed yet. However, an evaluation of

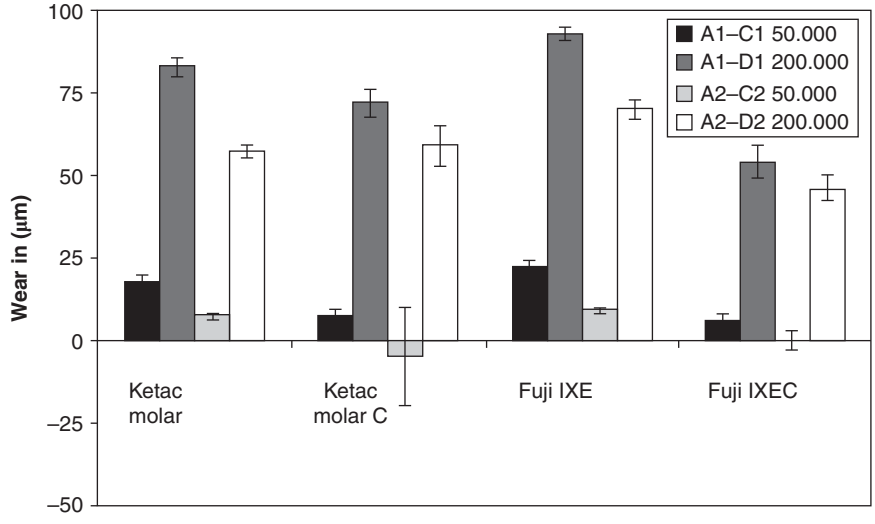

Figure 3. Wear in μm per 50 000 and 200 000 cycles for the investigated materials at slip of 15% and 15 N force. The bars represent standard errors.

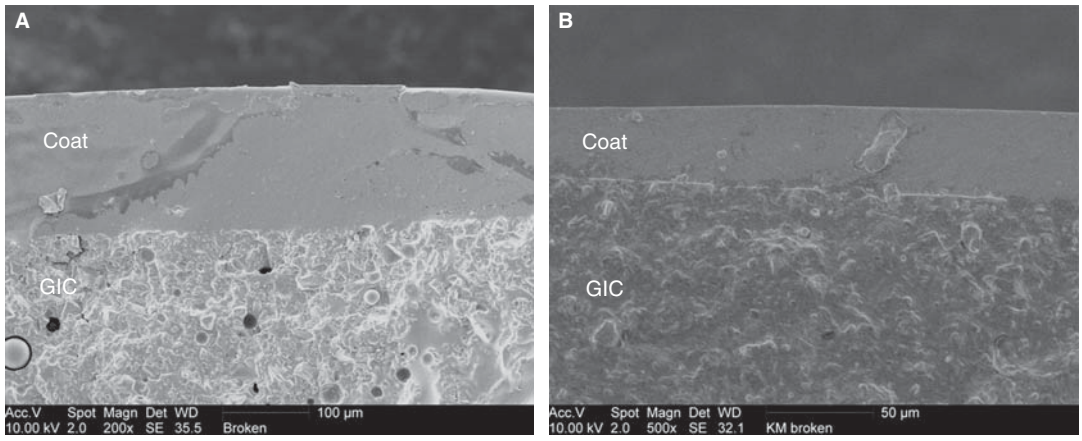

Figure 4. SEM micrographs of the specimens' cross-section of FIXE (A) and KM (B), where it is observed that there is micromechanical interlocking between the coat and the GIC.

the flexural strength seems to be more appropriate than compressive strength, as the bulk fracture is one of the main reasons for failure of approximal GIC restorations [25,26]. The coated surface in this experiment was placed opposite the applied force as it is the most stressed surface in which the crack will start. The results showed higher flexural strength compared to the previous version of those materials, the so-called low and medium viscosity GIC [27,28]. The flexural strength for these high viscosity versions of Ketac Molar and Fuji IX GP Extra are in line with previously reported values from hand mixed versions of those materials [29]. While Fuji IX GP Extra showed significant improvement in its flexural strength with G-Coat, the same was not observed for Ketac Molar. Although Fuji IX GP Extra improved its flexural strength, this material seems to have lower strength than Ketac Molar. In the 3-point bending test the highest tension is on the opposite side of the applied force. Applying a coating at this side might prevent the existing flaws to serve as areas of stress concentration and crack propagation. Once a crack sets off, the specimen will fail due to its brittle nature. Apparently, the crack starts from within the specimen and not from the surface, meaning that the coat does not play a role when applied on materials which are intrinsically more resistant to flexural stresses. For some restorative materials, a correlation is observed between the surface damage and flexural strength [30]. In the present study, we did not observe any correlation between the wear and the flexural strength, as the materials behaved differently for each property when the coat was used.

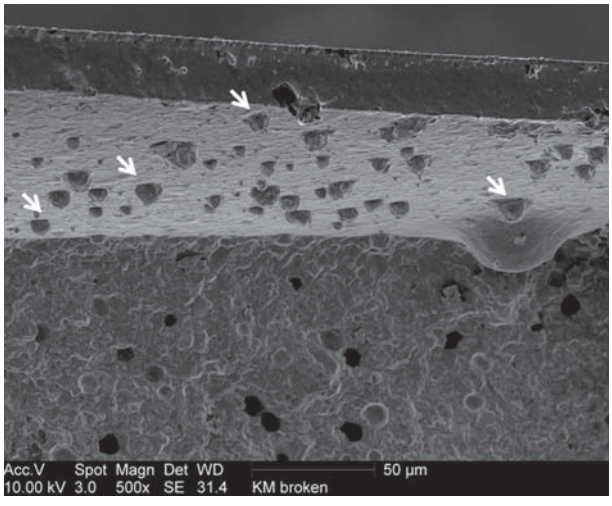

Figure 5. SEM micrograph of the specimens' cross-section of KM. Part of the GIC fell off and the coat was left intact. The arrows indicate areas where the coat penetrated the voids of the GIC.

The manufacturer's instructions recommend 20 s of light curing for G-Coat Plus. However, due to the length of the specimens (25 mm) in the FS experiment and the diameter of the curing light (10 mm), the curing was done in 3-times each 20 s along the bar. The coating was applied just after the first stage of the setting reaction. In the specimens of the wear test, it was re-applied after the first experiment. The aim of the reapplication of the coating was to mimic a clinical situation when the coating has disappeared because of wear. This was done to evaluate if the reapplication of the coat can be done also in old restorations. According to our results, Fuji IX GP Extra showed significant improvement in wear-resistance and flexural strength when the G-Coat Plus was applied. Even after a long-wear experiment, when the coat was reapplied after 200 000 cycles, Fuji IX GP Extra with G-Coat Plus showed significantly better wear results than Fuji IX GP Extra without coat. This means that the coating can be used to protect the Fuji IX GP Extra restoration for at least 24 h in the present experimental design. Recently, research by Bagheri et al. [19] evaluated the fracture toughness of different esthetic restorative materials. Fuji IX GP Extra was either coated with G-Coat plus or non-coated. The coated group showed significant differences compared to the non-coated group after 4 and 8 weeks of aging in water. Their results agree with the statement that water uptake in conventional GIC results in a

decrease in strength and elasticity of the material [31,32]. Another research by Schmage et al. [10] presented the wear rates for restorative composites, measured under similar circumstances. The values ranged between 18–50 μm after 200 000 cycles. Usually, restorative composites are required to have wear values lower than 50 μm per year [33]. Fuji IX GP Extra with G-Coat Plus showed an average wear of 54 μm after 200 000 cycles, suggesting that the wear properties of these materials combined may approach the ones of composites.

Conclusions

G-coat plus is indicated for concomitant use with Fuji IX GP Extra to decrease the early wear rate and increase its fracture strength. However, it should be considered that this study is limited to a short-term observation.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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