Effect of Bulk-filling on the Bonding Efficacy in Occlusal Class I Cavities

Annelies Van Ende[a] / Jan De Munck[b] / Kirsten Van Landuyt[c] / Bart Van Meerbeek[d]

Purpose: To evaluate the effect of bulk filling Class I posterior cavities on bonding to cavity-bottom dentin.

Materials and Methods: Two flowable "base" bulk-fill composites (Filtek Bulk Fill Flowable, SDR), one paste-like "full-body" bulk-fill composite (Tetric EvoCeram Bulk Fill) and one conventional paste-like composite (Filtek Z100) were bonded (G-ænial Bond) to either a flat surface (3.5 x 3.5 x 4 mm; C-factor: 0.18) or a Class I cavity (3.5 x 3.5 x 4 mm; C-factor: 5.8). After 1-week water storage, the restorations were sectioned to obtain 4 rectangular microspecimens that were subjected to microtensile bond strength (μTBS) testing.

Results: No significant differences in μTBS were recorded between all composites when bonded onto a flat surface ($p > 0.05$). When bonded into a Class I cavity, the μTBS of all composites except SDR significantly decreased ($p < 0.001$).

Conclusion: Both the configuration factor and the type of bulk-fill composite were found to have a great impact on bonding to cavity-bottom dentin.

Keywords: adhesion, μTBS, shrinkage, configuration.

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Restorative materials necessitating fewer application steps are appealing for dental practitioners, as application errors can be avoided and valuable chairtime can be reduced. After the introduction of the simplest-to-use one-step self-etching adhesives,[24] bulk-fill composites are now strongly advocated.[8,9,13] However, since simplification of materials and/or application techniques often entails compromises,[4,6] concerns have been raised about possible shortcomings, such as depth of cure and shrinkage stress. Manufacturers have approached these problems using many different strategies, resulting in bulk-fill composites with highly variable properties.[10,18] Bulk-fill composites currently on the market are either flowable bulk-fill composites, which are intended to be used as a base covered by a conventional composite, or paste-like bulk-fill composites, which are intended to restore the entire body of the restoration. Reduced conversion in the deeper parts of the restoration and increased shrinkage stress are the most important issues with regard to bulk-filling techniques. Both of these factors can have a negative effect on the bond strength.[19,29]

Therefore, the objective of this study was to compare the bonding performance of two flowable "base" bulk-fill composites, one paste-like "full-body" bulk-fill composite and one conventional paste-like composite on flat surfaces and in Class I cavities. The null hypotheses were that the microtensile bond strength (μTBS) to dentin depends neither (1) on the configuration factor nor (2) on the composite used.

MATERIALS AND METHODS

The study setup is schematically illustrated in Fig 1 and the materials used are listed in Table 1. Sixty-four noncarious human third molars were stored in 0.5% chloramine solution at 4°C and used within 6 months after extraction (molars were collected following informed consent according to a procedure approved by the Commission for Medical Ethics of KU Leuven, file number S57622). All teeth were mounted in gypsum blocks to facilitate manipulation and were randomly subdivided into two groups. In the Class I cavity groups, the

[a] *PhD student, BIOMAT, Department of Oral Health Sciences, KU Leuven (University of Leuven) and Dentistry, University Hospitals Leuven, Leuven, Belgium. Conducted the experiments, wrote the manuscript.*

[b] *Postdoctoral Research Fellow, BIOMAT, Department of Oral Health Sciences, KU Leuven (University of Leuven) and Dentistry, University Hospitals Leuven, Leuven, Belgium. Statistical evaluation, contributed substantially to conducting the experiments, discussion.*

[c] *Research Professor, BIOMAT, Department of Oral Health Sciences, KU Leuven (University of Leuven) and Dentistry, University Hospitals Leuven, Leuven, Belgium. Contributed substantially to the discussion.*

[d] *Professor and Chair, BIOMAT, Department of Oral Health Sciences, KU Leuven (University of Leuven) and Dentistry, University Hospitals Leuven, Leuven, Belgium. Idea, hypothesis, reviewed the manuscript.*

Correspondence: *Professor Bart Van Meerbeek, BIOMAT, Department of Oral Health Sciences, KU Leuven (University of Leuven), Kapucijnenvoer 7, B-3000 Leuven, Belgium. Tel: +32-16-33-7587; e-mail: bart.vanmeerbeek@med.kuleuven.be*

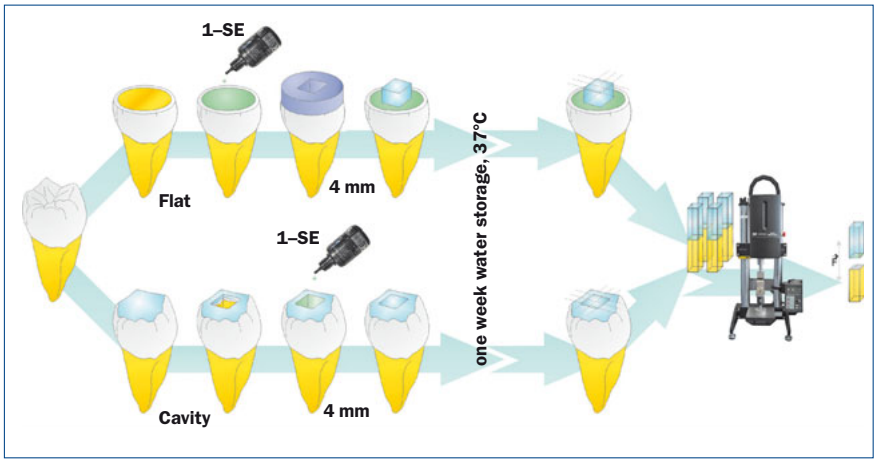

Fig 1 Schematic of the study setup.

Table 1 Materials investigated

	Product (acronym), shade (manufacturer)	Composition
Flowable base bulk-fill composites	Filtek Bulk Fill (FBF), A2 (3M ESPE)	Resin: bis-GMA, UDMA, bis-EMA, procrylat resin Filler: zirconia/silica, ytterbium trifluoride (42.5 vol%, 64.5 wt%)
	SDR (SDR), U (Dentsply)	Resin: modified UDMA, TEG-DMA, EBPDMA Filler: Ba–Al–F–B–Si–glass and Sr–Al–F–Si–glass (45 vol%, 68 wt%)
Paste-like full-body bulk-fill composite	Tetric EvoCeram Bulk Fill (TBF), IVA (Ivoclar Vivadent)	Resin: bis-GMA, UDMA Filler: Ba–Al–Si–glass, ytterbium fluoride, mixed oxide, prepolymer filler (60 to 61 vol%, 79 to 81 wt%)
Conventional paste-like composite	Z100 (Z100), A3 (3M ESPE)	Resin: bis-GMA, TEG-DMA Filler: zirconia/silica (71 vol%, 84.5 wt%)
One-step self-etching adhesive	G-ænial Bond (GC)	Phosphoric ester monomer, 4-MET, hydrophilic methacrylate monomer, water, acetone, photoinitiator, nanosilica

Bis-GMA: bisphenol A glycidyl dimethacrylate; bis-EMA: bisphenol A polyethylene glycol diether dimethacrylate; EBPADMA: ethoxylated bisphenol A dimethacrylate; TEG-DMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate; 4-MET: 4-methacryloyloxy ethyl trimellitic acid.

teeth were built up with the flowable composite G-ænial Flo (GC; Tokyo, Japan) after etching the enamel with 35% phosphoric acid (Scotchbond Universal Etchant, 3M ESPE; Seefeld, Germany) followed by the application of the adhesive (G-ænial Bond, GC), to create a flat surface at the height of the cusp tip. Thereafter, standard 4-mm-deep box-type Class I (3.5 x 3.5 x 4 mm) cavities were prepared. In the flat surface groups, the crown was cut 4 mm below the cusp tips, after which a smear layer was produced with the tip of the bur, similar to that produced in the cavities. Mid-coronal dentin was present both on the flat surfaces and in the cavities, ensuring that effects of regional variability on μTBS were negligible.[28,33] All preparations were made with a computer-controlled, custom-adapted automatic device (MicroSpecimen Former, University of Iowa; Iowa City, IA, USA), equipped with a cylindrical medium-grit diamond bur (835 314 010, 107 μm, Komet; Lemgo, Germany) mounted in a high-speed air turbine (650, KaVo; Biberach, Germany). A one-step self-etching adhesive (G-ænial Bond) was used in all experimental groups according to the manufacturer's instructions. Next, the cavities were filled in bulk and a 3.5 x 3.5 x 4 mm buildup was made in bulk on the flat dentin surfaces using a silicone mold. The teeth were randomly subdivided according to the composite used, resulting in 8 teeth per experimental group. Two flowable base bulk-fill composites (FBF and SDR), one paste-like full-body bulk-fill composite (TBF) and one conventional paste-like composite (Z100) were used. The restorations were light cured with a high-power LED light-curing device (Bluephase, Ivoclar Vivadent; Schaan, Liechtenstein) with an output above 1100 mW/cm^2 for 40 s. The light intensity was checked before each use with the accompanying Bluephase meter (Ivoclar Vivadent).

To estimate light irradiance at the bottom of the restorations, the same 4-mm-high silicone mold that was used to prepare the composite buildup on the flat dentin surfaces was placed on top of a 3.9-mm diameter sensor connected to a NIST referenced spectrometer (MARC PS, equipped with Ocean Optics ISO4000; Halifax, Canada). Next, the mold was filled with the uncured composite, and irradiance was measured while each of the four composites was cured following the same curing protocol mentioned above. Likewise, light irradiance was measured when similar 4-mm-

Table 2 μTBS results

Experimental group	Mean (SD)	ptf/n	Failure analysis
FBF cavity	4.0 (7.8)[b]	21/28	A: 96%; M: 4%
FBF flat	19.7 (7.8)[a]	0/28	A: 79%; M: 21%
SDR cavity	16.6 (7.7)[a]	0/25	A: 85%; M: 15%; C: 0%
SDR flat	26.7 (9.8)[a]	0/28	A: 73%; M; 22%; C: 5%
TBF cavity	3.9 (7.5)[b]	19/26	A: 96%; M: 4%
TBF flat	21.4 (9.0)[a]	0/30	A: 44%; M: 44%; C: 8%
Z100 cavity	0.0 (0.0)[b]	32/32	A: 100%; M: 0%; C: 0%
Z100 flat	26.0 (13.9)[a]	0/29	A: 52%; M: 48%; C: 0%

SD: standard deviation; ptf: pre-test failures; n: number of specimens; A: adhesive; M: mixed; C: cohesive. Means with same superscript are not statistically different from each other.

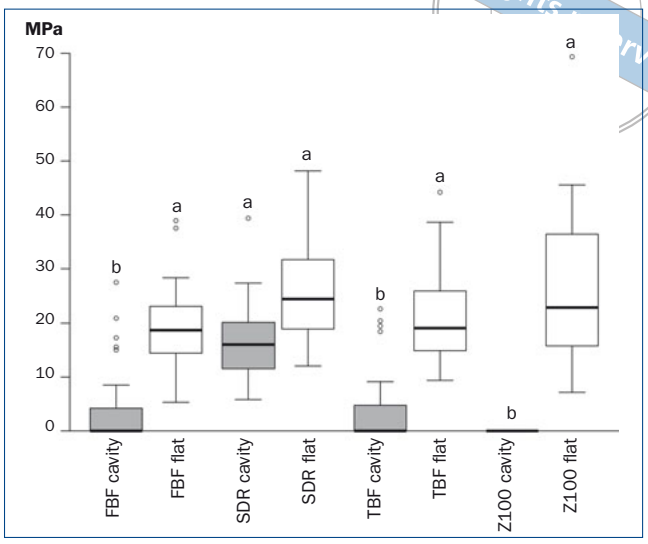

Fig 2 Boxplot of the μTBS results. Groups with the same letter are not statistically significantly different ($p < 0.05$). FBF: Filtek Bulk Fill; TBF: Tetric EvoCeram Bulk Fill.

deep "Class I cavity" tooth molds, from which the cavity bottom was removed, were positioned on the sensor, filled, and light cured accordingly.

After one week of water storage at 37°C, the teeth were sectioned perpendicular to the adhesive/tooth interface using an automated water-cooled diamond saw (Accutom-50, Struers; Ballerup, Denmark) to obtain rectangular 1- x 1-mm nontrimmed microspecimens for μTBS testing. The specimens were examined using a light microscope (MSA 166305 stereomicroscope, Wild; Heerbrugg, Switzerland) at a magnification of 50X to check for the presence of cavity corners or voids at the specimens' interface; all such samples were excluded from further testing. The specimens were kept moisturized until tested. They were attached to a notched BIOMAT-jig[30] with cyanoacrylate glue (Model Repair II Blue, Sankin Kogyo; Tochigi, Japan) and stressed until failure in a universal testing device (Instron 5848 Micro Tester; High Wycombe, UK) at a crosshead speed of 1 mm/min, using a load cell of 500 N. The μTBS was expressed in MPa, calculated by dividing the imposed force (N) at the time of fracture by the bond area (mm^2). The μTBS of specimens that failed before actual testing (pre-test failure: ptf), was assumed to be 0 MPa for further analysis.[12]

The mode of failure was assessed light microscopically (MSA 166305 stereomicroscope, Wild) at a magnification of 50X. Per microspecimen, the occurrence of adhesive failure, cohesive failure, or mixed failure was recorded. Representative fracture surfaces were processed using scanning electron microscopy (JSM-6610LV, JEOL; Tokyo, Japan) after common SEM preparation techniques.[23]

Microtensile bond strength data (μTBS per microspecimen in MPa) were analyzed using the Kruskal-Wallis test prior to post-hoc multiple comparisons at $p < 0.05$. All tests were performed at a significance level of $\alpha = 0.05$ using a software package (R2.12 and Weibull Toolkit 2.1, R Foundation for Statistical Computing; Vienna, Austria; and Weibull Toolkit 2.1, http://sourceforge.net/projects/weibulltoolkit).

RESULTS

The μTBS results are shown in Table 2 and presented graphically in Fig 2. Values ranged from 0 MPa (due to ptfs) to 70.1 MPa. There were no statistically significant differences between composites when bonded to a flat surface (FBF flat, SDR flat, TEBF flat, and Z100 flat: $p > 0.05$). When bonded in a cavity, the μTBS for all groups decreased significantly, except for SDR (SDR flat vs SDR cavity: $p > 0.05$). For the other composites, more than 50% ptfs occurred when bonded in cavities, with 100% ptfs for the conventional composite (Z100 cavity). There were no statistically significant differences between FBF cavity, TEBF cavity, and Z100 cavity ($p > 0.05$).

Failure analysis revealed predominantly adhesive failures. The incidence of mixed failures increased on flat surfaces. Representative SEM micrographs are shown in Fig 3.

Light irradiance measured at the bottom of the composite blocks (Fig 4) revealed that the light was attenuated the most in the conventional composite Z100; almost no difference in light attenuation was measured for the bulk-fill composites FBF and TBF; the highest irradiance at the bottom was measured for SDR. Except for Z100, light was attenuated to a significantly greater extent when using the silicone mold than when the composite was applied in the Class I cavity.

DISCUSSION

Bulk filling is highly desired in routine restorative practice, but concerns about shrinkage stress have caused it to be

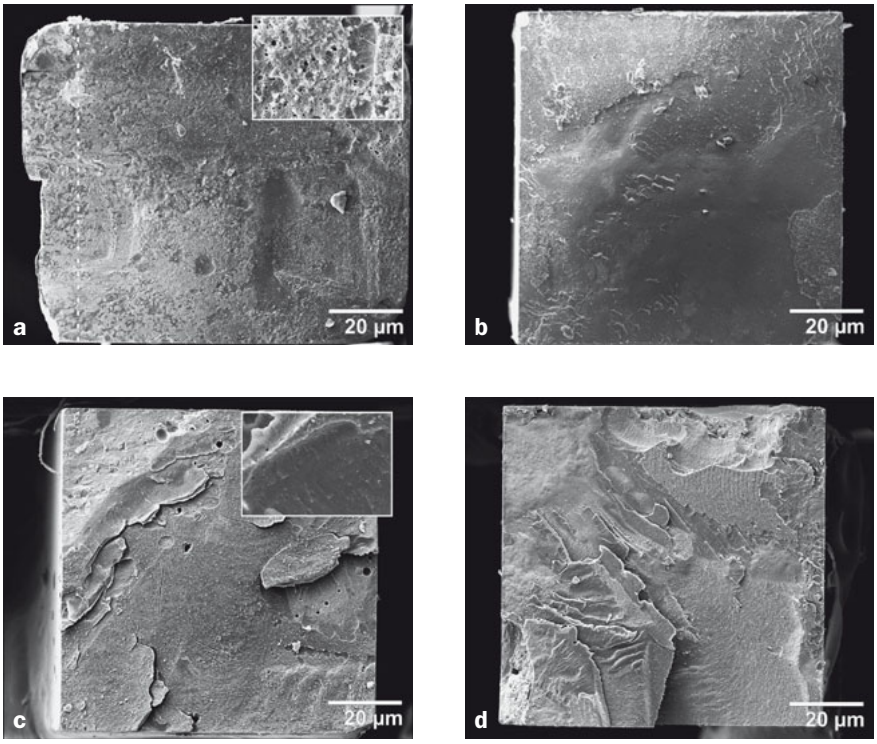

Fig 3 SEM photomicrographs of fractured surfaces. a) FBF cavity: adhesive failure. The specimen failed before the last cut was completed and was recorded as a pre-test failure (ptf). Insert: Despite thorough drying for 5 s, air bubbles were seen within the adhesive layer, most likely representing phase separation, as documented for the HEMA-free adhesive.[19] b) SDR cavity: adhesive failure. c) TBF flat: mixed failure. Air bubbles can be seen within the composite. Insert: on the flat surface, air bubbles within the adhesive could be avoided by thorough drying and spreading of the adhesive. d) Z100 flat: mixed failure.

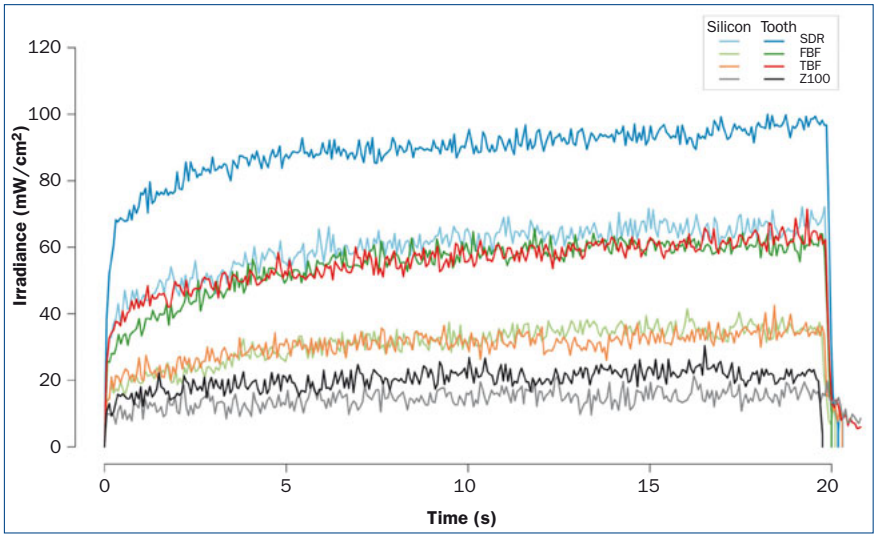

Fig 4 Irradiance measured at the bottom of 4-mm-thick composite specimens for the four experimental groups using either a silicone or tooth Class I cavity mold. FBF: Filtek Bulk Fill; TBF: Tetric EvoCeram Bulk Fill.

applied hesitantly. In this study, the decision was made to employ a microtensile bond strength (μTBS) protocol to evaluate the potential impact of bulk filling on the bond strength to cavity-bottom dentin in different C-factor configurations. A one-step self-etching adhesive was chosen to simulate the most simplified application protocol, since a faster, simpler procedure is the most important reason for choosing a bulk-filling procedure. The first null hypothesis had to be rejected, because the bond strength decreased significantly upon bonding in a Class I cavity ($p < 0.01$). Previous studies found a decrease in bond strength when Class I cavities were tested vs standard flat surface preparations.[2,20,22,27] This has mainly been attributed to shrinkage stress acting on the bond to cavity-bottom dentin and weakening it. However, other factors might have influenced the bond strength as well. The one-step self-etching adhesive used (G-ænial Bond) should be strongly air thinned to prevent phase separation.[31] In a cavity, spreading of the adhesive is limited, so that the occurrence of porosities (Fig 3) and pooling of adhesive in the cavity corners cannot be avoided.[6] Such an excess of adhesive has been reported to negatively influence bond strength.[5] Moreover, better adaptation can be obtained on flat surfaces. As the mold is not physically connected to the tooth surface, the

gap may serve as a sprue through which air can escape. In cavities, however, some air inclusions in the sharp angles of the cavity bottom were inevitable[21] and may have influenced stress distribution in the respective specimens.[1,3]

The second null hypothesis also had to be rejected, since significant differences between composites were found ($p < 0.05$). Differences in bond strength between composites have mainly been attributed to differences in shrinkage stress. Previous results have already indicated that the conventional composite Z100 induces high shrinkage stress.[17] This has been associated with a high percentag of pre-test failures,[19] which is in agreement with the finding of 100% ptfs in the Z100 cavity group. Sufficiently high bond strengths were obtained with SDR both on flat surfaces and in cavities, which is in line with previous results.[15,30] It has been demonstrated that SDR undergoes a peculiar polymerization pattern, resulting in stress levels even lower than those exerted by a silorane-based composite.[11] However, a considerable number of ptfs was recorded for the bulk-fill composites Tetric EvoCeram Bulk Fill and Filtek Bulk Fill. Kim et al[16] found that SDR induced significantly lower shrinkage stress than Tetric EvoCeram Bulk Fill and Filtek Bulk Fill.

However, in another study, no differences could be found,[7] whereas one study favored Tetric EvoCeram Bulk Fill over SDR.[14] In a previous study, Ilie et al[13] found a significant difference in bond strength of SDR vs Tetric EvoCeram Bulk Fill; however, these results were found on a flat surface and were mainly attributed to the better adaptability of SDR. Shrinkage stress is not a material's property, but is inherent to the compliance and configuration of the setup. Hence, results from different studies are conflicting and cannot simply be generalized. However, it has been repeatedly shown that Filtek Bulk Fill Flowable and Tetric EvoCeram Bulk Fill have a lower fracture toughness[9] and flexural strength[7,11,12,19] than SDR, which might render these composites more prone to crack induction/propagation and eventual failure during specimen preparation.

Insufficient curing and the associated decrease in mechanical properties have also been associated with lower bond strengths.[10,26] Indeed, when the light penetration at the bottom of the cavity was investigated, a clear trend of increasing bond strength with increasing translucency could be seen (Fig 4). Delayed cure at the bottom of the cavity may direct the shrinkage stress towards the already polymerized surface. Moreover, despite proper curing of the composite itself, the oxygen-inhibited layer of the adhesive may not receive sufficient energy to copolymerize adequately with the overlying composite,[22] creating a fragile zone at the interface. This might also explain the higher occurrence of adhesive failures in cavities (Table 2 and Fig 3). Surprisingly, the bulk restorations on flat surfaces were not negatively affected due to insufficient light curing; even Z100 performed equally well on flat surfaces, despite the fact that the effective depth of cure is limited to 2.5 mm. Considering the relative narrowness of the build-ups, ambient light exposure after removal of the mold might have enhanced polymerization as well, allowing the bond to mature in the absence of higher polymerization forces that are imposed on the bottom of Class I cavities.

Finally, it is known that variability increases with low bond strength. A high number of pre-test failures was found in this study; these were assigned a bond strength of 0 MPa and included in the analysis, as this is the most widely accepted method in the literature. However, this causes an underestimation of the bond strength and lack of discrimination between the true bond strength values.[19] Other adhesives that exhibit higher bond strengths and are less technique sensitive[32] might result in different outcomes.[10,20]

CONCLUSION

Although time-saving clinical procedures seem very attractive, simplification per se is not translated to less technique sensitivity. In combination with a one-step adhesive procedure, bulk filling may cause premature debonding of the interface, even when bulk-fill composites are used. Several factors may contribute to interfacial failure, such as polymerization shrinkage, insufficient depth of cure, and voids due to air inclusion or insufficient wettability. In short, the high variability in bond strength results emphasizes the need of further investigations.

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Clinical relevance: In combination with a one-step self-etching adhesive, bulk filling may cause bond detachment at the bottom of the restoration and thus compromise the longevity of the restoration.