

# Influence of dentin and enamel on the fracture resistance of restorations at several thicknesses

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**ABSTRACT: Purpose:** To investigate the effect of substrate and thickness on the fracture resistance of bonded dental restorative materials. **Methods:** Cylindrical restorations ( $d = 4.0$  mm) of resin composites Filtek Supreme XTE, Clearfil AP-X, Lava Ultimate and glass-ceramic IPS e.max CAD were fabricated at thicknesses of 0.5 mm, 1.0 mm and 2.0 mm respectively ( $n = 10$  per group) and adhesively bonded to bovine enamel or dentin. The load to failure (LtF in N) of all specimens was determined in a universal testing machine and two one-way ANOVAs with a post hoc LSD tests and separate independent samples t-tests, performed at a significance level of 5%. **Results:** At 0.5 and 1.0 mm, direct resin composites bonded to dentin showed a higher LtF than when bonded to enamel, while the indirect materials showed reversed results ( $P < 0.05$ ). At 2.0 mm there was no difference except for LU. A direct relationship between LtF and increasing thicknesses on enamel was found, while on dentin the LtF of direct resin composite restorations was less dependent on the thickness. (*Am J Dent* 2018;31:34-38).

**CLINICAL SIGNIFICANCE:** For restorations up to 1 mm thickness, a substrate with a matching elastic modulus has a positive effect on the fracture resistance of glass-ceramics and resin composite restorations. When bonded to enamel, restoration thickness plays an important role in the fracture resistance. When bonded to dentin, thickness only affects the fracture resistance of indirect restoratives.

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## Introduction

Nowadays, bonded tooth-colored restorations are a preferred treatment option as they have esthetic and biologic advantages over cast metal and amalgam restorations. The disadvantage of these materials was always their susceptibility to fracture or wear. The improved properties of ceramics and resin composites and the increased knowledge of bonding to dentin and enamel has led to a decrease of these problems and to an increased use of tooth-colored partial restorations. However, fractures still occur and the differences in mechanical properties between ceramics and resin composites give them each their own indication area. However, a clear overview of the properties and circumstances that determines their indication area seems not yet available in current literature.

The most common used materials for tooth-colored adhesive dental restorations are glass-ceramics and resin composites. Glass-ceramics like lithium disilicate are used for their optical and mechanical properties.<sup>1,2</sup> Clinical studies<sup>3-6</sup> show promising results regarding longevity for single unit glass-ceramic restorations. When compared to more traditional indirect restorations, like full metal (gold alloy) or porcelain fused to metal, glass-ceramics seem a reliable and esthetic alternative.<sup>7,8</sup> The reliable techniques available to achieve a durable bond of resin composite cements to glass ceramics,<sup>9,10</sup> created the possibility of these materials to be used for partial restorations, and meanwhile enhances a minimally invasive indirect approach by limiting the indication of full crowns.<sup>11,12</sup> Although clinical studies<sup>4,7</sup> showed promising results for partial ceramic restorations, laboratory studies<sup>13,14</sup> reported mixed results for thin (until 0.6 mm) occlusal (stress bearing) restorations. Therefore the question remains on which thickness of a ceramic restoration remains a reliable treatment option.

Resin composites have been proven in clinical studies<sup>15-18</sup> as reliable direct restorative materials to restore even large cavities and worn dentitions, but their longevity is negatively influenced by increasing restoration size and in areas of increased occlusal stresses.<sup>15,16</sup> Their elastic modulus is significantly lower than that of glass ceramics (resp. 14 GPa and 70 GPa) which is beneficial for relatively thin occlusal restorations.<sup>19</sup> Additionally, fatigue appears to have less effect on resin composites compared to ceramics.<sup>20</sup> Disadvantages of resin composites are susceptibility to water sorption,<sup>21</sup> and wear and esthetic degradation.<sup>22</sup> In an attempt to combine the advantages of ceramics and composites, so-called hybrid materials were developed, like industrially made resin composite blocks that can be used as a CAD/CAM fabricated indirect composite restoration.

The fracture resistance of composite and ceramic restorations depends not only on the material properties but also on its match to the elastic modulus of the substrate to which it is bonded.<sup>23-27</sup> Because enamel showed superior bonding capacities over dentin,<sup>28-30</sup> the substrate as such also influences the bonding to the restoration. The quality of the bonding affects the fracture resistance of the restoration.<sup>31-34</sup> Finally, thickness appears to play an important role in the fracture resistance of resin composite and especially ceramic restorations.<sup>26,27,34-36</sup>

It is assumed for this study that the elastic modulus and strength of the restorative material and the tooth substrate as well the restorative thicknesses and the quality of the adhesion between the restorative and substrate might play important roles in the fracture resistance of indirect tooth-colored restorations.<sup>28-30,37</sup> Therefore, this study investigated the combined influence of tooth substrate, thickness and bonding on the fracture resistance of several bonded restorative materials. The null-hypothesis was that there was no influence of these factors on the load to failure.

Table 1. Materials properties according manufacturers data.

Code	Material	Composition (in weight%)
MF	Clearfil Majesty Flow	Hydrophobic aromatic dimethacrylate, TEGDMA, 3 $\mu$ m, silanated barium glass + 20 nm silanated colloidal silica
AP	Clearfil AP-X	3 $\mu$ m Barium glass filler (70% vol.), Bis-GMA, TEGDMA, silica, pyrogenic SiO <sub>2</sub>
LU	Lava Ultimate	20 nm silica filler, 4 to 11 nm zirconia filler (80% vol.),
EM	IPS e.max CAD	SiO <sub>2</sub> , Li <sub>2</sub> O, K <sub>2</sub> O, MgO, AlO <sub>3</sub> , P <sub>2</sub> O <sub>5</sub> , ZrO <sub>2</sub> , ZnO
	Clearfil SE Bond Primer	MDP, HEMA, hydrophilic dimethacrylate, photo-initiator, water
	Clearfil SE Bond Bond	MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, photo-initiators, silanated colloidal silica
	Panavia F2.0 ED Primer Liquid I	HEMA, MDP, 5-NMSA, water, accelerator
	Panavia F2.0 ED Primer Liquid II	5-NMSA, accelerator, water, sodium benzene sulfinate
	Clearfil Porcelain Bond Activator	Hydrophobic dimethacrylate, $\gamma$ -MPS
	Panavia F2.0 Paste A	MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators
	Panavia F2.0 Paste B	Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, surface treated sodium fluoride, catalysts, accelerators, pigments

TEGDMA: triethylene glycol dimethacrylate, Bis-GMA: bis-phenyl glycidylmethacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, HEMA: hydroxyethyl methacrylate; 5-NMSA: N-Methacryloyl-5-aminosalicylic acid,  $\gamma$ -MPS: gamma methacryloyloxy propyltrimethoxy silane.

## Materials and Methods

The load to failure (LtF) of four restorative materials was tested for three different restoration thicknesses adhesively placed on dentin and enamel. All materials used in this study are listed in Table 1. The restorative materials were the flowable resin composite Clearfil Majesty Flow<sup>a</sup> (MF), the direct resin composite Clearfil AP-X<sup>a</sup> (AP), the indirect resin composite Lava Ultimate<sup>b</sup> (LU) and a lithium disilicate ceramic IPS e.max<sup>c</sup> CAD (EM). For each of these materials three groups of 20 specimens were made at a thickness of 0.5 mm, 1.0 mm and 2.0 mm respectively. Each group of 20 specimens was then again divided into two groups (n = 10): 1) Bonded to dentin, and 2) Bonded to enamel.

The crowns of cleaned bovine incisors, stored in 0.5% chloramine at 4°C, were separated from their root along the cemento-enamel junction. The buccal coronary part was ground to a flat enamel surface. The root was cut vertically and ground to create two flat dentin surfaces. All specimens were embedded in molds of polymethyl methacrylate<sup>d</sup> and wet polished with abrasive paper of 400 grit (Ecomet<sup>e</sup> polisher).

To make identical specimens of MF and AP, plastic molds (d= 4.0 mm) with the required thicknesses were made. First, bovine tooth specimens were prepared by applying Clearfil SE Bond<sup>a</sup> primer and bonding according to the manufacturer's instructions. Then, a mold was placed on the specimen and slightly overfilled with the composite. Then a glass plate was placed on top under finger pressure and the composite was photopolymerized for 20 seconds by blue light (Elipar S10<sup>b</sup>). Afterwards, the mold was removed and the exact thickness of each composite cylinder was measured.

For LU and EM, cylinders were cut out of blocks as delivered by the manufacturer for CAD/CAM milling devices, using a hollow cylinder drill (internal diameter = 4.0 mm). Discs of the required thickness were sliced from the cylinders using a diamond coated saw (Isomet 1000<sup>e</sup>). The EM specimens were sintered afterwards in a porcelain furnace (Progamat P100<sup>e</sup>), according to the manufacturer's instructions and both the EM and LU specimens were wet polished using 600 grit. The EM restorations were etched for 20 seconds using 9% hydrofluoric acid (Ultradent Porcelain Etch<sup>f</sup>), extensively

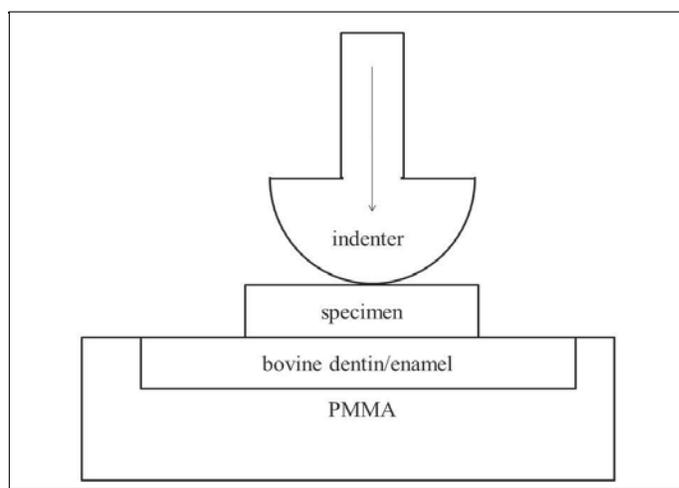


Fig. 1. Schematic drawing of the test procedure.

rinsed with water and air-dried. The LU restorations were air-abraded using aluminum oxide (50  $\mu$ m, 2,000 hPa) for 10 seconds at a distance of 10 mm, well rinsed and air dried. Both restoration types were chemically pretreated with Clearfil Porcelain Bond Activator,<sup>a</sup> mixed with Clearfil SE Bond primer, according to the instructions of the manufacturer.

Bovine enamel specimens were pretreated by 30-second etching with 37% phosphoric acid (Ultradent Porcelain Etch), followed by applying Panavia F2.0 ED Primer<sup>a</sup> for 30 seconds. Dentin specimens were treated only with the ED primer. After pretreatment, the EM and LU discs were cemented with resin composite cement Panavia F2.0, under a continuous pressure of 80 gr for 20 seconds. After removal of the cement excess, the cement was photopolymerized for 20 seconds from two sides. Oxygen-inhibiting gel (Panavia F2.0 Oxyguard<sup>d</sup>) was applied on the margins while chemical polymerization took place. After 5 minutes the gel was rinsed off with water.

All specimens were stored in 37°C at 100% humidity for 48 hours and then tested in a universal testing machine<sup>g</sup> using a load cell of 10 kN at a crosshead speed of 1 mm/minute. The load was applied on the samples by a stainless steel indenter with a diameter of 3.5 mm fixed on a stylus (Fig. 1). The definition of failure was determined as the value at the point

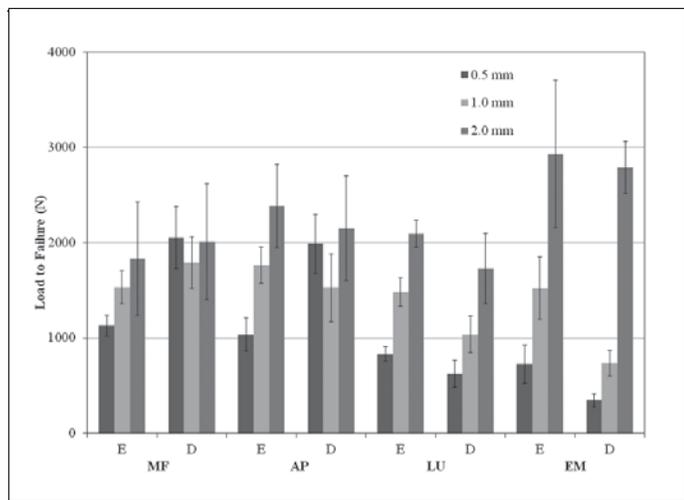


Fig. 2. Load to Failure (in N) of Clearfil Majesty Flow (MF), Clearfil AP-X (AP), Lava Ultimate (LU), and IPS e.max (EM) at different specimens thicknesses bonded to enamel (E) or dentin (D).

where the first crack occurred. This was registered as the first drop in the graph during loading and recorded in Newtons.

To analyze the statistical differences between all groups, two separate One-Way ANOVAs with a post hoc LSD test were performed for each substrate. To analyze the influence of the subsurface, separate independent samples t-tests were performed, all at a significance level of 5%. IBM SPSS Statistics 23<sup>h</sup> was used for the data analysis.

## Results

The loads to failure (LtF) are shown in Fig. 2 and Table 2. Figure 2 shows the failure loads and the standard deviations for each group. Table 2 displays the statistical differences between the groups. On both enamel and dentin, EM2.0 showed the highest LtF, and EM0.5 the lowest LtF. On enamel, the LtF for all groups significantly increased with increasing thickness. On dentin, this was only found for LU and EM. Additionally, the results in Table 2 show a significantly higher LtF of FL0.5, FL1.0 and AP0.5 on dentin, compared to enamel, while a significantly higher LtF on enamel was found for LU on all thicknesses and EM at 0.5 and 1.0 mm.

The substrate resulted in significant differences in LtF for all materials at 0.5 mm and 1.0 mm, except for AP1.0 mm. At 2.0 mm there were no significant differences between enamel and dentin, except for LU2.0.

## Discussion

The results of this study showed that tooth substrate and restoration thickness have a significant influence on the load to failure of all tested restorative materials. Therefore, the null-hypothesis could be fully rejected.

The substrate affected the load to failure significantly for all materials at 0.5 mm. While the direct resin composites MF and AP showed a higher LtF on dentin, indirect restorations LU and EM showed a higher LtF on enamel. The same effect was seen at 1.0 mm, except for AP. One explanation for the effect of the substrate can be found in the results of previous studies<sup>23-26</sup> that a more matching elastic modulus of the restoration and the substrate has a positive effect on the fracture resistance. For the directly bonded resin composites, the elastic moduli of MF and

Table 2. Load to Failure (in N) and standard deviations in parentheses of Clearfil Majesty Flow (MF), Clearfil AP-X (AP), Lava Ultimate (LU), and IPS e.max (EM) at different specimens thicknesses (0.5, 1.0, and 2.0 mm) bonded to enamel or dentin.

Material	Thickness	Enamel	Dentin
MF	0.5	1,129 (109) AB *	2,051 (329) E *
	1.0	1,531 (170) BC *	1,789 (270) DE *
	2.0	1,830 (596) CD	2,009 (609) DE
AP	0.5	1,032 (176) AB *	1,988 (308) DE *
	1.0	1,760 (189) CD	1,528 (358) CD
	2.0	2,382 (440) E	2,151 (553) E
LU	0.5	830 (74) A *	625 (141) AB *
	1.0	1,481 (145) BC *	1,036 (191) BC *
	2.0	2,093 (143) DE *	1,728 (369) DE *
EM	0.5	722 (199) A *	344 (71) A *
	1.0	1,522 (326) BC *	730 (133) AB *
	2.0	2,933 (773) F	2,789 (277) F

Characters indicate significant differences within columns, while \* indicates significant differences within rows.

AP (respectively 10.5 GPa and 16.8 GPa, according to manufacturer) approach dentin (18.0<sup>37</sup>), explaining the overall higher LtF on dentin than on enamel.

This rationale also explains the higher LtF of lithium disilicate EM (95.0 GPa, according to manufacturer) on enamel compared to dentin, as its elastic modulus is more similar to enamel (90.0 GPa<sup>37</sup>). The effect of the substrate on the strength of ceramics was described by Rhee et al<sup>27</sup> with an equation that showed that the critical load to failure of ceramic materials is dependent on the difference in elastic modulus between the ceramic and its substrate. They state that the difference in elastic modulus causes biaxial stresses in the bonded surface directly below the contact point of the load, which led to radial cracks.<sup>23,26,27</sup> These radial cracks are described as small initial cracks deriving from minor flaws at the bonded surface, propagated towards the outer surface and seen as the most common reason for failure of ceramic crowns.<sup>25,26,35,38</sup>

An exception to this theory is the indirect composite resin LU (12.8 GPa, according to manufacturer), that has an elastic modulus comparable to dentin, but a significantly lower LtF on dentin than on enamel. This might be explained by the effect of adhesion on the fracture strength of a restorative material. A comparable study<sup>34</sup> to the current one showed an increase of the fracture resistance of LU, when the bonding was improved by air-abrading the material prior to cementation. Also, the shear bond strength of resin composite cement to enamel is better than to dentin.<sup>28,29</sup> This might explain the higher LtF of LU on enamel in this study.

The effect of bonding on the fracture resistance of a restoration can also explain the results of the other materials, like the higher LtF on dentin of both directly bonded resin composites MF and AP. According to a laboratory study,<sup>30</sup> SE Bond had a higher  $\mu$ TBS to dentin than to enamel. Also, the superior adhesion of direct composites compared to that of indirect ceramics as found by others,<sup>10,39</sup> might play a role in the significantly higher LtF of both resin composites compared to EM and LU at 0.5 mm thickness.

The effect of bonding on the LtF can also be found in the results of EM; because the higher fracture resistance when bonded to enamel instead of dentin can be related to the better adhesion of the luting agent to enamel.<sup>28,29</sup> This increase in fracture resistance of lithium disilicate with adhesive cementa-

tion was also seen in a previous laboratory study<sup>31</sup> and of an earlier glass ceramic tested.<sup>33</sup> The strengthening effect of resin composite cement on glass ceramics was described as crack bridging.<sup>32</sup> Because of the high elastic modulus of ceramics, the shrinkage of resin cement creates stresses in the cement and the substrate that result in additional strengthening of the restoration.

In addition to the elastic modulus and adhesion to the substrate, the results of this study showed a significant effect of thickness of the restoration on the LtF, with AP and MF on dentin as the only exceptions. Except these two situations, the resin composites, including LU, showed a linear increase of the LtF with increasing thickness. Because of the relatively low elastic modulus of resin composites, they fail on quasi-plasticity instead of radial crack formation.<sup>26</sup> This is a deformation which is less affected by the thickness of the material. The ceramic EM showed a more quadratic increase of LtF. This is in line with the study of Rhee<sup>27</sup> which reported that the formation of radial cracks depends on the square of the layer thickness, making the fracture resistance of the ceramic more dependent on its thickness than composite. This confirms previous in vitro studies, that direct resin composites can better withstand forces at small thicknesses than ceramics.<sup>14,19,26,35</sup>

Another finding in this study is that with increasing thickness, the influence of the substrate became less significant. At 0.5 mm and 1.0 mm, almost all materials showed a significant influence of the substrate, while at 2.0 mm there were no significant differences anymore between dentin and enamel within the same material, except for LU. This phenomenon is described in literature as 'bulk properties' of the material that become dominant over the influence of the substrate.<sup>25,26,35</sup>

The results of this in vitro study showed significant differences in the performance of all materials on dentin and enamel. Therefore, it can be assumed that comparable findings will be seen in the clinical situation. Nevertheless, this laboratory study did not take into account all the challenging conditions of the oral cavity. These conditions imply that factors like fatigue, water sorption and aging can degrade the quality of restorations.<sup>21,40</sup> Especially cyclic loading could alter the results of this study, because the adhesive layer will experience more stresses and radial cracks forming, as described for ceramics, and will become more significant during fatigue testing.<sup>26,38</sup> Additionally, for the reproducibility of the results, the investigators chose a simplified set-up with disk-shaped specimens. These do not resemble the shape of a dental restoration, because restorations have a very specific occlusal pattern that results in varying thicknesses and a geometry creating different stress distribution. Dental restorations also have a mixed support of tooth substrate, varying in enamel, dentin and resin composite and they have a varying distance to the pulp chamber. These factors cause a variation in Young's modulus and adhesive properties of the substrate and thus have a direct effect on the fracture resistance. Finally, most of the forces applied in this study do not occur during function as they exceed the maximum bite force of healthy young adults.<sup>41</sup> Therefore, the straightforward set-up of the present study clearly shows the influence of enamel and dentin on the fracture strength of a restoration. These results encourage further research on this topic considering additional factors like fatigue.

Under the tested conditions, tooth substrate, restoration type and restoration thickness all have a significant influence on the fracture resistance of the four dental restorative materials tested. A matching elastic modulus between substrate and restorations resulted in higher load to failure at thicknesses up to 1.0 mm. When restorations are thicker, the role of the substrate become less significant.

- a. Kuraray Medical Inc., Okayama, Japan.
- b. 3M ESPE, St. Paul, MN, USA.
- c. Ivoclar Vivadent, Schaan, Liechtenstein.
- d. Vertex Dental, Zeist, The Netherlands.
- e. Buehler Ltd., Lake Bluff, IL, USA.
- f. Ultradent Products Inc., South Jordan, UT, USA.
- g. Instron, Norwood, MA, USA.
- h. IBM, Chicago, IL, USA.

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