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## Bond strength of restorative materials to hydroxyapatite inserts and dimensional changes of insert-containing restorations during polymerization



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#### ABSTRACT

*Objective.* To determine the shear bond strength (SBS) between synthetic controlled porous hydroxyapatite (HAP) inserts and restorative materials and dimensional changes of insert-containing restorations during curing.

Methods. Cylinder-shaped HAP inserts (4 mm in diameter, 1.6 mm thick) were cemented in dentin discs (5 mm  $\times$  1.6 mm), cut mid-coronally from human third molars, using one of the following materials: universal microhybrid composite Filtek Z250, flowable composite Filtek Ultimate or glass-ionomer Vitrebond (all 3M ESPE). SBS of the same materials to HAP inserts was tested in a universal testing machine. Three-dimensional digital image correlation system Aramis (GOM) was used to measure strains and displacements. Data were statistically analyzed using one-way ANOVA with Tukey's post-test ( $\alpha$  = 0.05).

Results. SBS of restorative materials to HAP inserts ranged between  $12.2 \pm 2.1$  MPa (Filtek Z250) and  $0.7 \pm 0.4$  MPa (Filtek Z250 without an adhesive). The 'total-etch' approach of adhesive application significantly increased SBS of both Filtek Z250 ( $12.2 \pm 2.1$  MPa) and Filtek Ultimate flowable ( $9.5 \pm 2.5$  MPa) compared to the 'self-etch' approach ( $8.2 \pm 1.6$  MPa and  $4.4 \pm 0.9$  MPa, respectively) (p < 0.05). HAP inserts reduced polymerization shrinkage to below 0.5% as well as displacements in the central region of the restorations. Peripheral shrinkage of restorative materials was similar with and without HAP inserts as were displacements of Filtek Z250 and Vitrebond.

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Significance. Replacing major part of dentin clinically, especially in large cavities, HAP inserts may shorten clinical working time, improve dimensional stability of the restoration by reducing central shrinkage and displacements and provide adhesive bonding to universal composites following a 'total-etch' approach.

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#### 1. Introduction

Attempts to overcome the adverse properties of resin-based dental materials, polymerization shrinkage and the associated stress [1] can either be material-related (e.g. low shrinkage monomers [2,3] or dental inserts [4]), technique-related (incremental layering versus bulk technique [5–7]) and light source-related ('soft-start' or 'pulse' light-curing regimes [8]).

The idea behind the use of dental inserts, prefabricated parts of a restoration similar to inlays, is to reduce the amount of unset material in the cavity. Early inserts were megafillers [4,9] and later were ceramic, manufactured in different sizes for different cavities [10,11]. Inserts were produced mostly from IPS Empress ceramics (Cerena),  $\beta$ -quartz ceramics ( $\beta$ -quartz glass ceramic inserts) and leucite-reinforced ceramic (SonicSys and Cerafil inserts). To the best of our knowledge, hydroxyapatite (HAP)-based inserts have not been tested in restorative dentistry.

HAP may be more beneficial than ceramics due to similar mechanical properties to dentin, e.g. HAP fracture toughness of  $1.30 \pm 0.015 \,\text{MPa}\,\text{m}^{1/2}$  [12] falls in the range of dentin fracture toughness values ( $1.13-2.02 \,\text{MPa}\,\text{m}^{1/2}$ ) [13] whereas the trend is to increase fracture toughness of ceramics [14,15]. Another advantage of synthetic HAP over ceramics is the ability to chemically interact with functional monomer groups in adhesives [16].

Dimensional changes of insert-containing restorations were tested using dye penetration tests [11,17–19], microscopic evaluation of microgaps [10,11,20–22], strain gauges and mercury bath [9]. The use of  $\beta$ -quartz ceramic inserts instead of 31–38 vol% of composite reduced polymerization shrinkage [9]. Clinical investigation of  $\beta$ -quartz inserts has shown contradicting results. Kiremitci et al. have shown satisfactory and improved clinical performance of restorations according to the USHPS criteria [22]. An 8-year clinical study confirmed the beneficial effect of these inserts on composite restoration properties [23].

Inserts are clearly advantageous in large cavities due to a reduction in the number of clinical steps. In general, it has been shown that ceramic inserts do have some positive effects on marginal adaptation but further improvements are required. A possible negative effect of increasing the number of interfaces between materials with different elastic moduli needs to be investigated. Dentin exhibits lower elastic modulus (2 to 15 GPa) [24] than most contemporary composites (10 to 24 GPa) [25]. Although the elastic modulus of synthetic HAP may vary significantly [26], modulating this property to be comparable to dentin may in fact have a positive rather than a negative effect on the restoration despite the increased number of interfaces.

Digital image correlation is a non-contact method for measuring volumetric shrinkage, based on tracking the position of surface markers by specialist software before and after polymerization and calculating strains and displacements. Earlier studies employed one-camera systems [27–30] which provide information only for in-plane movements. Micro-computed tomography was also used for 3D measurements [31–33]. However, radiopaque markers required in micro-computed tomography often exceed filler size in dental composites and may disturb polymerization. Two-camera systems for obtaining actual 3D data were seldom used [34,35]. In these previous studies, no dental bonding systems were used in order to allow unrestricted shrinkage.

The aim of this study was to measure the shear bond strength (SBS) between HAP inserts and various restorative materials as well as the effect of HAP inserts on dimensional changes of restorations during material setting/polymerization. The null hypotheses were: (1) there is no difference in SBS to HAP inserts between restorative materials and (2) there is no difference in shrinkage strains and displacements between groups with and without HAP inserts.

#### 2. Materials and methods

#### 2.1. Insert preparation and properties

Starting HAP powder, composed of spherically agglomerated nanosized rod-like particles, was obtained using the modified hydrothermal synthesis described earlier [36,37], starting from a solution of  $CaCl_2 \cdot 2H_2O$ ,  $Na_2H_2EDTA \cdot 2H_2O$ ,  $NaH_2PO_4 \cdot 2H_2O$ and urea. The synthesis parameters and concentration of precursors were the same as described previously [12]. The powder was further isostatically pressed in a stainless steel mold at 400 MPa into disc compacts, 4 mm in diameter and 1.6 mm thick, and additionally sintered at 1200  $^\circ$ C over 2 h at a heating rate of 20 °C/min. Processed controlled porous insert material (with calculated mean pore diameter of 0.74 µm, observed at the fractured surface of inserts), consisted of HAP as the dominant phase, with the presence of lower amounts of  $\alpha$ - and  $\beta$ -TCP as a second crystalline phase [12]. Insert material was characterized with the density value of  $2.64 \pm 0.02$  g/cm<sup>3</sup>, fracture toughness of  $1.30 \pm 0.01 \,\text{MPa}\,\text{m}^{1/2}$  and Vickers hardness of  $3.05 \pm 0.05$  GPa.

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