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Polymerization contraction stress in dentin adhesives bonded to dentin and enamel

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ABSTRACT

Objective. In a previous study on of polymerization contraction stress determinations of adhesives bonded to dentin a continuous decline of stress was observed after the adhesives had been light-cured. The decline was ascribed to stress relief caused by diffusion into the adhesive layer of water and/or solvents, left in the impregnated dentin surface after drying and/or evaporation in the application procedure. The purpose of the present study was to test the hypothesis that the contraction stress of adhesives bonded to enamel will not decline after light-curing, based on the assumption that water and/or solvents are more efficiently removed from impregnated enamel surfaces in the drying and/or evaporation step.

Materials and methods. Contraction stress was determined in a tensilometer for three total-etching adhesives Scotchbond multi-purpose, Single bond and One-step plus and four self-etching adhesives Clearfil SE Bond, Clearfil Protect Bond, AdheSE, and Xeno III. The adhesives were placed in a thin layer between a glass plate and a flat dentin or enamel surface pre-treated with phosphoric acid or self-etching primer and light-cured under constrained conditions.

Results. All adhesives bonded to enamel showed a stress decline, but significantly less than for dentin with the exception of two self-etching adhesives. The greatest decline was found for the total-etching adhesive systems bonded to dentin. The presence of hydrophobic monomers in the adhesives had a significant influence on the decline.

Significance. The experiments indicate that fluids are withdrawn from the resin impregnated tooth structures, which may result in small defects in the tooth–resin interfaces.

1. Introduction

Although dental resin composites are used in restorative dentistry for several decades now the polymerization shrinkage [1,2] remains a matter of concern. Loss of marginal integrity [3,4], cusp deflection [5,6] and tooth fracture [7,8] are still frequently occurring problems caused by induced contraction stresses when the polymerization shrinkage takes place under constrained conditions with the composite bonded between cavity walls.

When contraction stresses are measured in a universal testing machine with the composite bonded between surfaces of glass or steel, the course of stress development commonly shows a continuous increase from the start of light-curing

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[9,10]. However recent studies demonstrated that when one of these surfaces was replaced by dentin, the stress only increased during light-curing, but showed soon after that a continuous decrease [11,12]. The decrease of stress has been attributed to stress relaxation by fluid movement from the dentinal tubules and pores into the adhesive layer. The fluids may consist of water and/or solvents from the bonding systems, which were still present after drying and/or evaporation at the moment of light-curing. Movement of fluid across dentin and the resin–dentin bond during bonding procedures may be held responsible for the formation of nanosized spaces and channels [13] that can fill-up with water and contribute to degradation of resin bonds [14–19].

In contrast to etched dentin that consists of a complicated network of collagen fibrils, partially demineralized collagen and partially or fully opened tubules, etched ground enamel shows a regular honeycomb like etch pattern [20,21]. The sound enamel below the etch pattern is highly dense and does not contain pathways, like tubules in dentin, through which fluids or solvents can penetrate deeper into the enamel. Although sound enamel is permeable for salts and water [22] the diffusion rates are so extremely low [23] that any penetration through this structure during application procedures is most unlikely. Therefore compared to etched dentin, where water and/or solvents are captured in a complicated demineralized network and in tubules from which they are difficult to remove, we expect that drying of etched enamel surfaces and/or evaporation will remove most of the water and/or solvents. As water and/or solvents left in dentin surfaces have shown to cause a substantial decline of the contraction stress [12] we hypothesized that for adhesion to enamel the contraction stress will more closely follow the normal course of a continuous increase with time [9,10], based on the assumption that most of the solvents are removed from the enamel surface in the drying or evaporation step.

The aim of this study was to test this hypothesis by investigating the course of polymerization contraction stress development of thin adhesive layers bonded to enamel for three total-etching systems and four systems with self-etching primers. In addition, as a control, similar experiments with these adhesive systems were conducted with dentin.

2. Materials and methods

2.1. *Adhesives*

Three total-etching adhesives Scotchbond multi-purpose (SBMP) (3 M Espe, St. Paul, MN, USA), Single bond (SIB) (3 M Espe, St. Paul, MN, USA) and One-step plus (OSP) (Bisco Inc., Schaumburg, IL, USA), and four self-etching adhesives Clearfil SE Bond (CSE) (Kuraray Co., Ltd., Osaka, Japan), Clearfil Protect Bond (CPB) (Kuraray Co., Ltd., Osaka, Japan), AdheSE (ADH) (Ivoclar Vivadent, Schaan, Liechtenstein), and Xeno III (XIII) (Dentsply deTrey, Konstanz, Germany) were used in this study (Table 1). The experiments with OSP and CSE bonded to dentin were performed in an earlier study [12] and the results are included in this study.

2.2. *Preparation of dentin and enamel specimens*

Seventy central bovine incisors within 2 months of extraction were selected and the crown sections were cut from the roots at the proximal cemento-enamel junction, using a low-speed water-cooled saw (Buehler, Evanston, IL, USA). Dentin and enamel cores, 7.0 mm in diameter were cut normal to flat

Table 1 – Adhesive systems used in this study and their composition

Material (manufacturer)	Etchant	Primer or adhesive
Adper Scotchbond multi-purpose	35% H_3PO_4 gel	Primer: HEMA, PAA co-polymer and water Adhesive: HEMA and BisGMA
Single bond	35% H_3PO_4 gel	Primer + adhesive: HEMA, BisGMA, DMA, PAA co-polymer, water and ethanol
One step plus	37.5% H_3PO_4 gel	Primer + adhesive: HEMA, BisGMA, BPDMA, acetone and filler
Clearfil SE bond	–	Primer: HEMA, MDP, hydrophilic DMA and water Adhesive: HEMA, BisGMA, MDP, hydrophobic DMA and filler
Clearfil protect bond	–	Primer: HEMA, hydrophilic DMA, MDP, MDPB and water Adhesive: HEMA, BisGMA, MDP, hydrophobic DMA and filler
Xeno III	–	A: HEMA, water, ethanol and filler B: Pyro-EMA, PEMA-F and UDMA
AdheSE	–	Primer: PPAA, bis-acrylamide and water Adhesive: HEMA, BisGMA, GDMA and filler

Abbreviations—BisGMA: bisphenol A diglycidylmethacrylate; BPDMA: biphenyl dimethacrylate; DMA: dimethacrylate monomers; GDMA: glycerol dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; MDPB: 12-methacryloyloxydodecylpyridinium bromide; PEMA-F: pentaethylmethacrylate fluoride (pentamethacryloxyethyl cyclophosphazene monofluoride); PAA: polyalkenoic acid co-polymer; PPAA: phosphonic acid acrylate; Pyro-EMA: pyrophosphate ethylmethacrylate (tetramethacryloxyethyl pyrophosphate); UDMA: urethane dimethylacrylate.