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Measurement of micro-strain across resin-dentin interfaces using microscopic moiré interferometry

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Abstract

Little is known about the mechanical behavior of resin-dentine interfaces during loading. The presence of relatively compliant hybrid and adhesive layers between stiffer dentin and resin composite should deform more during compressive loading.

Objective—The objective of this study was to measure changes in microstrain across bonded dentine interfaces in real time using a recently developed microscope moiré interferometer.

Method—This system used a miniature moiré interferometer, using two CCD cameras for simultaneous recording of longitudinal and transverse deformation fields, a piezotransducer for fringe shifting and use of a microscope objective with magnification up to 600×. Small beams (1 × 2 × 6 mm) of moist resin-bonded dentine covered with a diffraction grating replica were placed in a miniature compression tester to allow controlled loading from 2–37 N while imaging the interference fringe patterns.

Results—Dentine beams bonded with Single Bond/Z100 under compressive loading of resin-dentine interfaces exhibited comparatively large strains across the adhesive-hybrid layer interface. When the wrapped phase maps were unwrapped to permit conversion of fringe order to displacements, the 2-D profiles of strain fields revealed nonuniform strains across the adhesive interface. In the adhesive/hybrid layer zone, the negative strain was larger (i.e. -6000 µε) than that seen in the adjacent resin composite or underlying mineralized dentin. The changes were elastic because they disappeared when the load was removed.

Significance—Microscopic moiré interferometry can be very useful in revealing real-time strain of bonded interfaces under load.

Keywords

Composite; Compression; Dentin; Dentin bonding agent; Mechanical properties; Moiré interferometry

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1. Introduction

Resin-dentin bonding is achieved via removal of some or all of the mineral phase from the top 4-5 μm of mineralized dentin surfaces, followed by infiltration of solvated monomer mixtures into the demineralized region to create a hybrid layer that is about half collagen fibrils and half adhesive resin [1]. The quality of the hybrid layer is determined, in part, by how deep the comonomers can infiltrate into the 4-5 μm deep demineralized zone and if there is any molecular sieving of larger dimethacrylates from monomethacrylate [2,3]. The excess adhesive lying on top of the hybrid layer is called the adhesive layer (Fig. 1) and serves to couple highly-filled resin composite to the underlying dentin via the hybrid layer. The adhesive layer should not contain residual solvent from the solvated comonomer mixtures or phase changes and should be free of water-filled voids and channels that have been reported in bonded interfaces [4-7]. Generally, in etch-and-rinse bonding systems, the hybrid layer is only 4-5 μm thick, while the adhesive layer can vary from 0-50 μm thick.

Currently, several laboratories are measuring the apparent modulus of elasticity of resin-dentin interfaces using nanoindentors, modified AFMs or acoustical microscopes [8-10]. These provide static measurements that provide little information about how the interface responds to tensile or compressive stresses. It is highly likely that collagen fibrils at the base of hybrid layers that are not enveloped by adhesive resins will undergo far more strain than collagen fibrils perfectly enveloped by resin [11]. Similarly, if most dimethacrylates are confined to the upper half of hybrid layers, and the lower half is mainly monomethacrylates [2,3], the degree of cross-linking in the top vs. bottom halves of hybrid layers should be different and their water sorption, modulus of elasticity, etc. would be expected to be different.

New techniques need to be developed that allow real-time quantitative measurements of the interfacial properties of bonds made under ideal conditions, versus those made under over-wet or over-dry conditions, or with incomplete polymerization or with residual solvents. These less than ideal conditions produce lower bond strengths, but the mechanisms involved remain unknown. Why do some bonds fail at the base of the hybrid layer? Is it because resin did not infiltrate to the depth of the acid-etch, causing the naked collagen fibrils to undergo more tensile and compressive strain during mastication? Can such strains be measured *in vitro* during compressive or tensile loading [12]?

One promising technique is moiré interferometry [13-15] where replicas of high frequency (i.e. 2400 lines/mm) cross-lined diffraction gratings are applied to flat cross-sections of resin hard tissue interfaces. Using an appropriate laser, the frequency distribution of the grating on a specimen is compared to a reference grating. Any differences create interference fringes that are proportional to displacement/microstrain in the specimen [13]. The major advantage of moiré interferometry is the potential for simultaneous acquisition of full U and V displacement fields in real-time. However, to measure deformation across 5 μm thick hybrid layers and 50 μm thick adhesive layers, one must be able to image interference fringes that are separated by only 1-2 μm . This requires development of a moiré interferometry microscope [15]. The ability to deliver enough light to the specimen and to construct the microscope in such a manner that one can place a tooth slice in a compression or tensile loading device under the objective lens is a daunting task.

The purpose of the work was to demonstrate the first use of a recently developed moiré interferometry system to measure real-time deformation across resin-dentin interfaces under stepwise compressive loads. The test null hypothesis was that deformation across the hybrid and adhesive layers under compressive load is not uniform.

2. Materials and methods

2.1. Experimental method

A high sensitivity microscopic moiré interferometry (MMI) system was developed to assess the behavior of biological/biomimetic materials at the micro scale. With the use of fringe shifting, this system is theoretically capable of providing full-field displacement data with high resolution. MMI provides the ability for the analysis of full-field material behavior in sections ranging from a few millimeters down to a few micrometers while loading the specimens.

The general optical configuration of the advanced microscopic moiré interferometry system is shown in Fig. 2a. In the system, green light ($\lambda = 514$ nm), emitted from an argon laser, enters an objective lens, where it is spatially filtered by a precisely positioned pinhole. The light is then collimated and transferred to a custom-designed fringe shifting device consisting of two 10° glass wedges and a piezoelectric transducer. A 50-50 beam splitter is used for beam separation into U and V fields. After the interference of the polarized beams in a custom-made mini-interferometer (Fig. 2b), the two fringe fields are directed through a microscope objective and are then transferred to two CCD cameras (Pulnix, San Jose, CA) via a polarized beam splitter (Fig. 2c). Simultaneous acquisition of the in-plane fields enhances the ability to monitor viscoelastic behavior of specimens.

The resolution of the MMI was enhanced by using double cross-line gratings that created less optical internal reflections than single cross-line gratings. The sensitivity of the developed moiré system is significantly increased through the implementation of fringe-shifting. As was previously mentioned, only half of the collimated beam is sent through the wedge setup (Fig. 2d), which provides for beam retardation and allows for fringe shifting when the top wedge is translated by the piezoelectric transducer (PZT). LabVIEW software (National Instruments, Austin, TX) is used for the control of the PZT and the acquisition of the shifted interferograms. This process relies on a five-step phase-shifting algorithm (Schwider-Hariharan) resulting in the computation of a wrapped phase distribution. Finally, the phase maps are unwrapped and displacement/strain is determined.

The design of the micro-mechanical testing apparatus was primarily governed by the intrinsic miniature size of dental specimens and the necessity for proper hydration levels mandated by biological tissues. Figure 2e shows the configuration of the loading slots in the developed system. The rectangular nature of the shaft design removes any possibility of relative rotation of the two loading components and permits purely compressive loading. Furthermore, the slot in the upper part of the shaft provides a place for a wet sponge and thus allows for proper specimen hydration during the investigation phase. Fig. 2f shows a schematic of the hydration arrangement. The final arrangement of the compression fixture allows for automated stepwise loading ranging from 0 to 350 N (measured by a miniature load cell [ELFS-T4-100lb. Model, Entran, Fairfield, NJ]) with a compression rate of roughly 0.15 mm/min. The reliability of the developed apparatus was established through experiments on specimens with known mechanical properties. Conducted tests involved materials such as aluminum alloys (6061-T6 and 3000 series), pure lead, and nylon 6,6 in order to properly ascertain the potential range of the constructed system. Beams of these materials were cut to $8 \times 2 \times 1$ mm (L $\times$ W $\times$ H). A 2400 line/mm double cross-line grating (Spectrogon US, Inc., Parsippany, NY, USA) was replicated on the top surface of these materials (Fig. 3) using a thin layer (ca. 1-2 μ m) cement (M-Bond 200, Vishay Micro-Measurements, Raleigh, NC). These beams were then placed in the compression-loading device and analyzed in the elastic zone. In addition, samples of unbonded mineralized dentin with varying tubule orientation (relative to the loading direction) were also examined in a similar manner. Proper hydration levels were maintained during the entire analysis.

2.2. Tooth preparation and resin bonding

Extracted human third molars were stored in phosphate buffer saline (PBS) containing 0.02% sodium azide at 4°C until used. The occlusal enamel and superficial dentin of the teeth were removed (Fig. 4a) using a copper blade embedded with diamond particles (Isomet saw, Buehler Ltd., Lake Bluff, IL) irrigated with water, turning at 20 rpm [3,7,11]. The exposed flat tooth surfaces were further ground with 320 grit SiC abrasive wet paper (Ecomet Polisher, Buehler Ltd). The smear layer was removed by applying 37% phosphoric acid gel for 15 s followed by rinsing for 10 s with water. The surface was blot-dried with cotton pellets to leave a visibly moist surface. Two layers of Single Bond (3M-ESPE, St. Paul, MN) were applied to the moist dentin surface in quick succession, followed by the use of an air stream to evaporate the solvent for 10 s and irradiation with a dental light-curing unit using a halogen bulb delivering light from 390-505 nm with a peak output at 470 nm and an energy density of 600 mW/cm² for 20 s [3,7,11,13]. After light-curing the adhesive, two separate 1.5 mm thick increments of a resin composite (Z100, 3M-ESPE, St. Paul, MN) were applied and light-cured for 40 s each (Fig. 1). The teeth were placed in water at 37°C for 6 months to permit maximum water sorption of the resin composite [16]. Then the bonded teeth were vertically sectioned into 1 mm thick slabs to expose the composite-adhesive-dentin interface (Fig. 4a). Several beams 6 × 2 × 1 mm (L × W × H) were cut from the center of the bonded interface. The top half of each beam consisted of resin composite and the bottom half was mineralized dentin (Fig. 4a). At the interface of these two dissimilar materials was an adhesive layer about 40-80 μm thick and a hybrid layer about 5 μm thick (Fig. 1). Each beam was briefly polished with 1000 grit SiC to remove any grinding debris. After sonication in water, it was briefly dried with air and a tiny drop of cyanoacrylate (M-Bond, Vishay Micro-Measurements, Raleigh, NC, USA) applied to the surface followed immediately by a polydimethyl siloxane replica of a 2400 line/mm cross-lined diffraction grating that had been sputter-coated with aluminum [13]. This spread the cyanoacrylate into a very thin film. After the cyanoacrylate had set, the polydimethylsiloxane carrier was removed leaving the double-cross aluminum replica of the diffraction grating embedded in the thin layer of cyanoacrylate covering the polished specimen surface (Fig. 3).

By compressing the composite-adhesive-dentin beams with known stresses (2-18 MPa), each of the four materials (composite, adhesive, hybrid layer and underlying dentin) undergo different strains that are proportional to their Young's modulus. The advantage of the MMI system is that it can measure the deformation of all of the dissimilar materials simultaneously in both x and y planes.

2.3. Finite element modeling (FEM)

A simple finite element model was developed to simulate the deformation field within the dentin beams resulting from compressive loading. A simple finite element model was developed to simulate the deformation field with the resin-bonded dentin beams resulting from compressive loading. The finite element model was developed using a commercial finite element software (ABAQUS, Version 6.6-1) with 3-dimensional 8-noded reduced integration point elements (C3D8R). The total model was comprised of approximately 7800 elements and nearly 10,000 nodes. Due to the partitioning of regions associated with material (and unique elastic constants) and expected non-uniform strain near the interfaces, the element sizes were different within the distinct regions. For both the resin composite and dentin, the average element size (edge length) was about 10 μm. For the resin adhesive, the average element size was about 4 μm and for the hybrid layer, the average element size was approximately 2 μm. The adhesive and hybrid layers were modeled with 75 μm and 5 μm thickness, respectively, which were selected according to the dimensions of a specific specimen. All of the interfaces (dentin/hybrid, hybrid/adhesive and resin adhesive/resin composite) were treated as perfectly bonded and the compressive load was distributed over

the specimen cross-section. The dentin, hybrid zone, resin adhesive and resin composite were considered linear elastic with moduli of 20, 5, 4, and 18 GPa, respectively. Poisson's ratio of dentin was assigned 0.26 according to the experimental results, whereas all other materials were assigned a value of 0.3 for convenience.

3. Results

Using the MMI system, the load-displacement and stress-strain curves for aluminum (6061-T6 and 3000 series), pure lead and nylon 6,6 were linear in the expected elastic zone (data not shown). Both moduli of elasticity and Poisson's ratios that were measured for these materials were comparable to the well-established properties of these materials (Table). Tests utilizing mineralized dentin alone showed that both the modulus of elasticity and Poisson's ratio are dependent on the orientation of the tubules relative to the applied loading direction (Table). The Young's modulus values for dentin were similar to the values published by others [17].

The utility of the MMI system is in providing full-field deformations across microscopically dissimilar materials in real-time. Figure 4b shows the progressive changes in fringe pattern across the composite/adhesive/hybrid layer/dentin interface of a single specimen created under ideal bonding conditions (i.e. acid-etched for 15 s, moist-bonding). As the compressive stress increased from 4-18 MPa, the fringe patterns come closer together in the U fields, showing that the compressive deformation increased across the bonded interface. These panels image a $200 \times 150 \mu\text{m}$ area that covers the bonded interface that is located in the center of the panels. Although microstrain distributions were calculated for every 4-5 MPa increase in compressive stress, only representative images are shown here. Note the lack of compressive deformation in the V field.

In Fig. 5, the wrapped phase maps were unwrapped to permit conversion of displacements into 2-D strain distributions. In Fig. 5a, the strain in the U field (ϵ_{xx}) is plotted in the x direction across a $400 \times 300 \mu\text{m}$ area of a resin-bonded dentin specimen undergoing a compressive stress of 16 MPa. In Fig. 5a, as the bonded interface is approached from the resin composite (i.e. left) side, the composite shows a gradual compressive strain that increases from near $-1500 \mu\epsilon$ to about $-3000 \mu\epsilon$ over a $150 \mu\text{m}$ distance. However, at about $150 \mu\text{m}$ from the left side, the compressive strain begins to increase rapidly to nearly $-6000 \mu\epsilon$, falls and then rises over the next $100 \mu\text{m}$, which corresponds to the adhesive layer. The compressive strain then decreases sharply from $-6000 \mu\epsilon$ to approximately $-3000 \mu\epsilon$ at $300 \mu\text{m}$, and then decreases gradually again over the remaining distance to $-1500 \mu\epsilon$. Note that results from the FEM are also presented in Figures 5(a) and 5(b) as dotted lines. While exhibiting a consistent spatial distribution in strain as the experimental results, the expected strain per the FEM is considerably smaller. The U-field strain distribution is shown again in Figure 5(b), along with additional finite element results where the elastic modulus of the materials was decreased by 25 % from that in Figure 5(a); there is far better agreement in the experimental and FEM results.

In contrast to the U-field, there was no distinct strain profile in the V field (not shown). Instead, the strains were randomly distributed. Shear strains were randomly distributed rather than uniform. These strains were elastic because the specimens returned to near zero strains in both U and V fields when the load was removed.

4. Discussion

The results of this study show the utility of the MMI in providing full-field microstrain maps across dissimilar material interfaces in real time. Strains were measured in both U and V fields simultaneously to avoid any effects of creep [18] if they were measured sequentially.

Unexpectedly, there were nonuniform axial strains across both the hybrid layer and the adhesive layer regions at stresses of 18 MPa which were due to relatively large ($-6000 \mu\epsilon$) compressive strains in the hybrid and adhesive layers/composite junction. These results require acceptance of the test null hypothesis that microstrains across the bonded resin-dentin interface are not uniform.

The near-interface non-linear strain profile seen in the U field across the resin-bonded dentin interfaces, and within the adhesive layer is a new observation. As the compressive stress was uniaxial and uniform along the length of the specimen, the strain should be inversely proportional to the elastic modulus. Therefore, the large negative strains in the resin composite and dentin (near the bonded interfaces) suggests that there has been a reduction in their elastic moduli. The use of lower elastic moduli in the FEM simulation present in Figure 5(b) results in better agreement, but there remains appreciable deviation near the interfaces. In the Table, the elastic moduli varied from 16 to 27 GPa depending on tubule orientation relative to loading. In this study, the tubules oriented parallel to the direction of loading and gave a elastic modulus of 16 GPa. Previous attempts to measure the stiffness across bonded interfaces by nanoindentation have reported that unfilled resin layers and the underlying hybrid layers have elastic moduli of about 3-4 GPa [8,9]. Even lower values (1.84 GPa) were reported by Katz *et al.* [10] using a scanning acoustical microscope. Yet, less attention has been placed on the elastic properties of the dentin and resin composite bounding the adhesive layer. Future evaluations of the microstrain distribution across the interface should incorporate a complimentary analysis comprised of mapping the elastic modulus distribution using nanoindentation. Thus, the presence of these relatively compliant materials sandwiched between the resin composite (Z100) and underlying dentin supports our speculation that strain distribution is non-uniform, but there are additional concerns remaining to be addressed.

Even within hybrid layer created by 37% phosphoric acid, there is a gradient of dimethacrylate resin (i.e. Bis-GMA) that has a higher concentration at the top of the hybrid layer, but is almost absent from the bottom of the hybrid layer [2,3] using Single Bond, the adhesive system used in the current study. That is, the bottom of the hybrid layer is thought to be filled primarily with very hydrophilic HEMA that may absorb more water [19] than the top half of the hybrid layer.

The complex relatively large (i.e. $-6000 \mu\epsilon$) axial strains observed in the U field reveal that Single Bond resin-dentin bonds undergo complex elastic deformations during loading. These may fatigue the structural components of the bonded interface, leading to plastic deformation over time. The relatively large (i.e. $-6000 \mu\epsilon$) microstrains observed across the resin-dentin interface may involve expression of absorbed fluid [19] from nanometer-sized porosities within these structures that could accelerate leaching of unreacted monomers over time. In addition, Poisson's effects were evident in the surface strains of the adhesive layer. The experimental U-field strains in Figure 5 between 150 and 250 μm show a concave distribution and results from the out-of-plane deformation or "bulging" of the adhesive resin/hybrid layer complex under compressive stress at the surface; the bulging causes development of a small tensile strain which reduces the magnitude of compressive strain through superposition. Similarly, tensile U-field strains would cause "cupping" of the adhesive layer due to Poisson's effects. These complex Poisson's effects could facilitate pumping of absorbed fluids and accelerated degradation of the adhesive. These complex nonuniform interfacial strains may fatigue the structures involved and contribute to the relatively rapid decrease in resin-dentin bonds made with the same adhesive, *in vivo*[20].

Because microstrains were simultaneously measured in U and V fields, it was theoretically possible to calculate Poisson's ratios of the materials across the interface. Two problems

confound such attempts: Calculations of the ratio of lateral to axial strain assume that the stress is purely axial (σ_{xx}) and that lateral stress is zero ($\sigma_{yy} = 0$). However, plots of $du/dy - dv/dx$ were not uniform. This did not occur in the resin composite or mineralized dentin sides of the bonded interface. Thus, the only uncertainty about Poisson's ratios is in the middle compliant zone. The second problem is that Poisson's ratio is a material property, but the hybrid layer is not a pure material but a construct of resin infiltration into a biopolymer network (i.e. collagen fibril mesh). Although dentin has been described as a biological composite, the number of dentinal tubules per mm [21], and the number of lateral branches of tubules varies with location [22] and hence creates different biological composites in different parts of each tooth. Thus, when it becomes possible to measure Poisson's ratios, they may vary with location. This may complicate FEM attempts to model stress-strain relationships in hybrid layers [23,24].

When the results of our FEM were compared to the actual microstrain results (Figs. 5a,b), the predicted square profile of the compressive strain across the adhesive/hybrid complex was in sharp contrast to the gradual negative and positive slopes that were actually measured across the bonded interface. We speculate that when resin composites are applied to the oxygen-inhibited adhesive resin surface, the adhesive comonomers diffuse into the composite matrix, thereby lowering its filler content and hence its elastic modulus. Additionally, recent work [25] shows that water flows from dentin into the overlying adhesive after bonding. This local water sorption lowers the elastic modulus of the adhesive below its predicted values. Finally, during acid-etching of dentin, 37% phosphoric acid, the acid is thought by some [26] to create a porous zone in the underlying mineralized dentin that may extend 10 μm below the 5-10 μm thick hybrid layer. All of these events may summate to produce graded elastic moduli at the interfaces of these different materials. Finite element modeling is based on interfaces, whereas reality involves interphases that have depth.

The MMI instrument may provide important new information on strain profiles across one-step all-in-one adhesives, two-step self-etching primer adhesives, and etch-and-rinse three-step adhesive systems. While nanoindentation instruments have provided useful information on the mechanical properties of bond interfaces, they give no insight into how these interfaces respond to loading. The MMI instrument can provide such important information.

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References

1. Nakabayashi, N.; Pashley, DH. Hybridization of Dental Hard Tissues. Quintessence Publishing Co., Ltd.; Chicago: 1998.
2. Wang Y, Spencer P. Quantifying adhesive penetration in adhesive/dentin interface using confocal Raman microspectroscopy. *J Biomed Mater Res.* 2002; 59:46–55. [PubMed: 11745536]
3. Spencer P, Wang Y. Adhesive phase separation at the dentin interface under wet bonding conditions. *J Biomed Mater Res.* 2002; 62:447–456. [PubMed: 12209931]
4. Tay FR, Pashley DH, Suh BI, Carvalho RH, Itthagarun A. Single-step adhesives are permeable membranes. *J Dent.* 2002; 30:371–382. [PubMed: 12554121]
5. Tay FR, Pashley DH. Water treeing - a potential mechanism for degradation of dentin adhesives. *Am J Dent.* 2003a; 16:6–12. [PubMed: 12744405]
6. Tay FR, Hashimoto M, Pashley DH, Peters MC, Lai SCN, Cheong C. Aging effects two modes of nanoleakage expression in bonded dentin. *J Dent Res.* 2003b; 82:537–541. [PubMed: 12821715]

7. Tay FR, Pashley DH, Suh BI, Hiraishi N, Yiu CKY. Water treeing in simplified dentin adhesives - Deja Vu? *Oper Dent*. 2005; 30:275–296. [PubMed: 15986945]
8. Van Meerbeek B, Willems G, Celis JP, Roos JR, Bream M, Lambrechts P, Vanherle G. Assessment by nano-indentation of the hardness and elasticity of the resin-dentin bonding area. *J Dent Res*. 1993; 72:1434–1442. [PubMed: 8408887]
9. Oliviera SSA, Marshall SJ, Habelitz S, Gansky SA, Wilson RS, Marshall GW. The effect of a self-etching primer on the continuous demineralization of dentin. *Eur J Oral Sci*. 2004; 112:376–383. [PubMed: 15279658]
10. Katz JL, Spencer P, Nomura T, Wagh A, Wang Y. Micromechanical properties of demineralized dentin collagen with and without adhesive infiltration. *J Biomed Mater Res*. 2003; 66A:120–128.
11. Tay FR, Carvalho RM, Yiu CKY, King NM, Zhang Y, Agee K, Pashley DH. Mechanical disruption of dentin collagen fibrils during resin-dentin bond testing. *J Adhes Dent*. 2000; 2:175–192. [PubMed: 11317391]
12. Frankenberger R, Pashley DH, Reich SM, Petschelt A, Tay FR. Characterization of resin-dentin interfaces by compressive cycling loading. *Biomaterials*. 2005; 26:2043–2052. [PubMed: 15576178]
13. Wood JD, Wang R, Weiner S, Pashley DH. Mapping of tooth deformation caused by moisture change using moiré interferometry. *Dent Mater*. 2003; 19:159–166. [PubMed: 12628426]
14. Kishen A, Asundi A. Experimental investigation on the role of water in the mechanical behavior of structural dentin. *J Biomed Mater Res A*. 2005; 73:192–200. [PubMed: 15761819]
15. Post, D.; Han, B.; Ifju, P. High Sensitivity Moiré: Experimental Analysis for Mechanics and Materials. New York: Springer; 1994. p. 293-312.
16. Örtengren U, Andersson F, Elgh U, Terselius B, Karlsson S. Influence of pH and storage time on the sorption and solubility behavior of three composite resin materials. *J Dent*. 2001; 29:35–41. [PubMed: 11137637]
17. Kinney JH, Marshall SJ, Marshall GW. The mechanical properties of human dentin: A critical review and re-evaluation of the dental literature. *Crit Rev Oral Biol Med*. 2003; 14:13–29. [PubMed: 12764017]
18. Pashley DH, Agee KA, Wataha JC, Rueggeberg F, Ceballos L, Ito K, Yoshiyama M, Carvalho RM, Tay FR. Viscoelastic properties of demineralized dentin matrix. *Dent Mater*. 2003; 19:700–706. [PubMed: 14511727]
19. Ito S, Hashimoto M, Wadgaonkar B, Svizero N, Carvalho RM, Yiu C, Rueggeberg FA, Foulger S, Saito T, Nishitani Y, Yoshiyama M, Tay FR, Pashley DH. Effects of resin hydrophilicity on water sorption and changes in modulus of elasticity. *Biomaterials*. 2005; 26:6449–6459. [PubMed: 15949841]
20. Carrilho MRO, Geraldini S, Tay F, deGoes MF, Carvalho RM, Tjäderhane L, Reis AF, Hebling J, Mazzoni A, Breschi L, Pashley D. *In vivo* preservation of the hybrid layer by chlorhexidine. *J Dent Res*. 2007; 86:529–533. [PubMed: 17525352]
21. Pashley DH. Clinical correlations of dentin structure and function. *J Prosthet Dent*. 1991; 66:777–781. [PubMed: 1805028]
22. Mjör IA, Nordahl I. The density and branching of dentinal tubules in human teeth. *Arch Oral Biol*. 1996; 41:401–406. [PubMed: 8809302]
23. Lee S-Y, Chiang H-C, Huang H-M, Shih Y-H, Chen H-C, Dong D-R, Lin C-T. Thermo-debonding mechanisms in dentin bonding systems using finite element analysis. *Biomaterials*. 2001; 22:113–123. [PubMed: 11101156]
24. Misra A, Spencer P, Marangos O, Wang Y, Katz JL. Micromechanical analysis of dentin/adhesive interface by the finite element method. *J Biomed Mater Res Part B (Appl Biomater)*. 2004; 70B(1):56–65. [PubMed: 15199584]
25. Tay FR, Pashley DH, Suh BI, Hiraishi N, Yiu CKY. Water treeing in simplified dentin adhesives - Deja Vu? *Buonocore Memorial Lecture*, 2005. *Oper Dent*. 2005; 30:561–579. [PubMed: 16268390]
26. Miyazaki M, Onose H, Moore BK. Analysis of the dentin-resin interface by use of laser Raman spectroscopy. *Dent Mater*. 2002; 18:576–580. [PubMed: 12385898]

27. Okudo M, Pereira PNR, Nakajima M, Tagami J. Relationship between nanoleakage and long-term durability of dentin bonds. *Oper Dent*. 2001; 26:482–490. [PubMed: 11551013]
28. DeMunck J, Van Meebeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, Lambrechts P, Vanherle G. Four-year water degradation of total-etch adhesives bonded to dentin. *J Dent res*. 2003; 82:136–140. [PubMed: 12562888]
29. Carrilho MRO, Carvalho RM, Tay FR, Yiu C, Pashley DH. Durability of resin-dentin bonds related to water and oil storage. *Am J Dent*. 2005; 18:315–319. [PubMed: 16433398]

Fig.1.

Schematic showing mineralized dentin (bottom), the hybrid layer (cross-hatched) at the top of mineralized dentin consisting of 5 μm of demineralized collagen matrix infiltrated by adhesive resin that also fill open tubules as resin tags. The excess adhesive resin (grey zone) couples filled-composite (irregular particles) to the hybrid layer. Values are approximate moduli of elasticity and layer thicknesses.

Fig. 2. Composite schematics of the microscopic moiré interferometry system. **a)** overview of system mounted on an air table; **b)** general layout of the interferometry; **c)** simultaneous two-field phase acquisition assembly using polarizing beam splitter and example of a decomposed interferogram; **d)** optical fringe-shifting subsystem driven by a piezoelectric transducer (PZT); **e)** loading jig configuration with sponge slot; **f)** water trapped between wet sponge and specimen with grating replica on its surface.

Fig. 3. Schematic of how an aluminum replica of a diffraction grating is transferred via cyanoacrylate into a specimen.

Fig. 4.

a) Schematic showing tooth preparation, 1 mm thick slab of resin composite-tooth buildup, and preparation of resin composite-dentine beam specimen. The replica of the diffraction grating covered the entire resin-dentine beam surface. **b)** shows progressive changes in the fringe patterns across resin-dentine interfaces in response to step-wise increases in compressive strains up to 18 MPa. Note that as the compressive stress increased, the distance between the fringes decreased indicating increased strain in the x or U -field. Also, note the lack of fringes and hence strain in the y direction or V -field.

Figure 5.

Two-dimensional profile plots of the U-field strain distribution across three regions indicated by dotted lines on a rectangular specimen of resin composite bonded to dentin. **a)** experimental strain compared with results from the FEM using elastic moduli of resin composite, adhesive, hybrid layer and dentin of 18, 4, 5 and 20 GPa, respectively. **b)** experimental strains compared with results from the FEM using elastic moduli of resin composite, adhesive, hybrid layer and dentin of 13.5, 3, 3.75 and 16 GPa, respectively. The strains shown in the interfaces were the strains exhibited by the adhesive/hybrid layer complex, not the hybrid layer alone.

Table

Validation of microscopic moiré interferometry system

Material	Elastic modulus, E	Poisson's ratio, ν
Aluminum 3000 series	Expected 68.9 GPa	0.33
	Measured 70.4 GPa	0.32
Aluminum 6061-T6	Expected 70.2 GPa	0.33
	Measured 71.3 GPa	0.33
Nylon 6,6	Expected 1-6 GPa	--
	Measured 3.7 GPa	--
Pure Lead	Expected 14.1 GPa	--
	Measured 14.0 GPa	--
Dentine	Expected 20.0 GPa	0.30
0,45,90° tubule orientation*	Measured 16, 20, 27* GPa	0.26, 0.22, 0.20*

* When measured parallel to the long axis of the dentinal tubules (i.e. 0° difference between direction of loading and direction of dentinal tubules), E = 16 GPa, ν = 0.26. When the load was applied at a direction that was 45° to the tubule direction, E = 20 GPa, ν = 0.22. When the compressive load was applied normal to the tubule direction, E = 27 GPa, ν = 0.20.