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Three-Dimensional Characterization of Dental Bonded Interface Degradation Using Serial Ion-Ablation Scanning Electron Microscopy

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In order to produce esthetic dental restorations, human dental tissues must be modified with acidic solutions to produce micromechanical and chemical bonding. The dissolution of the hydroxyapatite exposes the most superficial dentin collagen fibers; interdiffusion of resin monomers then follows to fill the interstices within the collagen network. The formation of this hybrid layer between the adhesive and dental tissues is responsible for bonding to the dental tissues. However, the collagen network is often not fully infiltrated, and nanometer scale pores and channels can cause hydrolytic degradation. Porosity within the hybrid layer is a serious problem for long-term quality of teeth restorations [1]. Studies of resin-impregnated layer degradation allow static interpretation of hydrolytic degradation [2]. A dynamic reconstruction of these water channels would allow in-depth understanding of the problem.

The aim of this study was to evaluate the application of serial ion-ablation scanning electron microscopy (SIA-SEM) to evaluate the nanoleakage of resin-bonded dental interfaces [3,4]. Human dentin was exposed and restored with ethanol- and water-based dentin adhesive (Clearfil SE Bond, Kuraray) followed by resin composite restoration (Filtek Z250, 3M ESPE). The specimens were immersed in 50% ammoniacal silver nitrate dye to trace possible voids within the hybrid layer. The specimens were fixed in 2.0% paraformaldehyde/2.5%glutaraldehyde in 0.2M sodium cacodylate buffer, dehydrated in ascendant degrees of ethanol and dried in hexamethyldisilazane. All specimens were mounted on aluminum stubs and sputter-coated with palladium.

The SIA-SEM specimen analysis was carried out using a dual beam focused ion beam (FIB), Nova Nano-lab 200, FEI Company. Figure 1a shows 3 slices taken through the restoration and dental tissue. Figure 1b shows a resliced section taken from the 3D stack across the hybrid and adhesive layer. Using the electron imaging, a specific area of the hybrid layer was identified and selected for investigation. To avoid damaging the surface by the ion beam, an initial Pt layer was deposited using a Pt organometallic source. This involves initial deposition using the electron beam to "crack" the Pt source, followed by deposition of a thicker layer using the ion beam. Stacks of several hundred sections, 20nm thick, were obtained and combined into a 3-D image (Fig 2). The tissue nanostructure and nm scale silver deposits were traced and quantified. The results showed that adhesive interface underwent hydrolytic degradation as soon as the bonded interfaces were produced. The 3-dimensional reconstruction of the adhesive interface allowed, for the first time, visualization of the dynamics of nanoporosities within the hybrid layer.

The present work shows that the use of SIA-SEM is a new and remarkably effective tool for investigation of the bonded interface in human teeth. Specifically, 3-D reconstruction of resin-

bonded interfaces yields a novel approach for the understanding of the degradation of bonded restorations.

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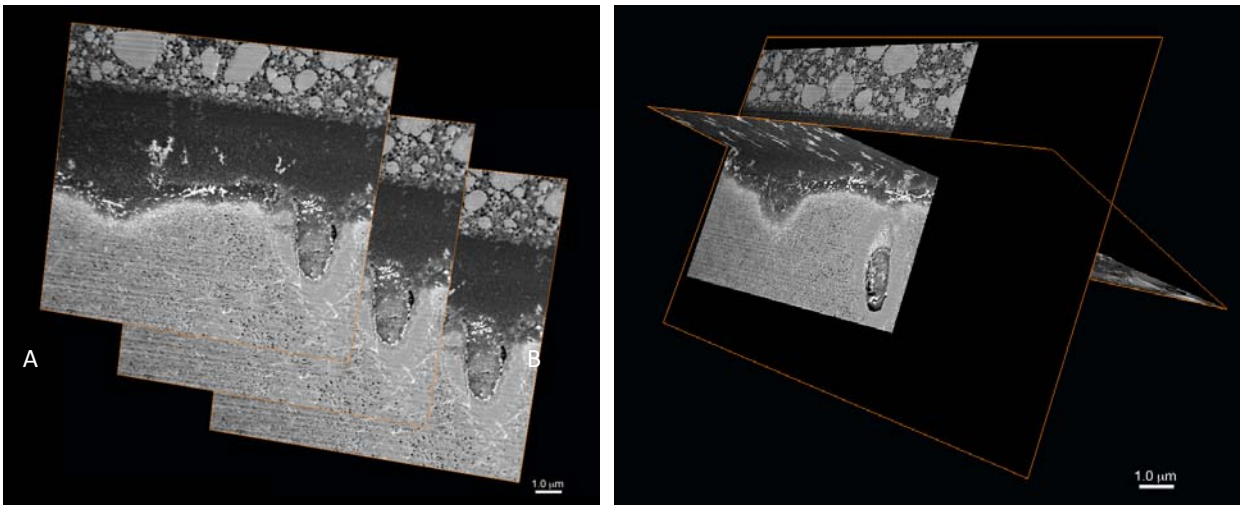

Fig 1: A) Three slices from a stack containing 545 slices at 20nm thickness, B) Three-dimensional view of the nanoleakage within the hybrid and adhesive layer.

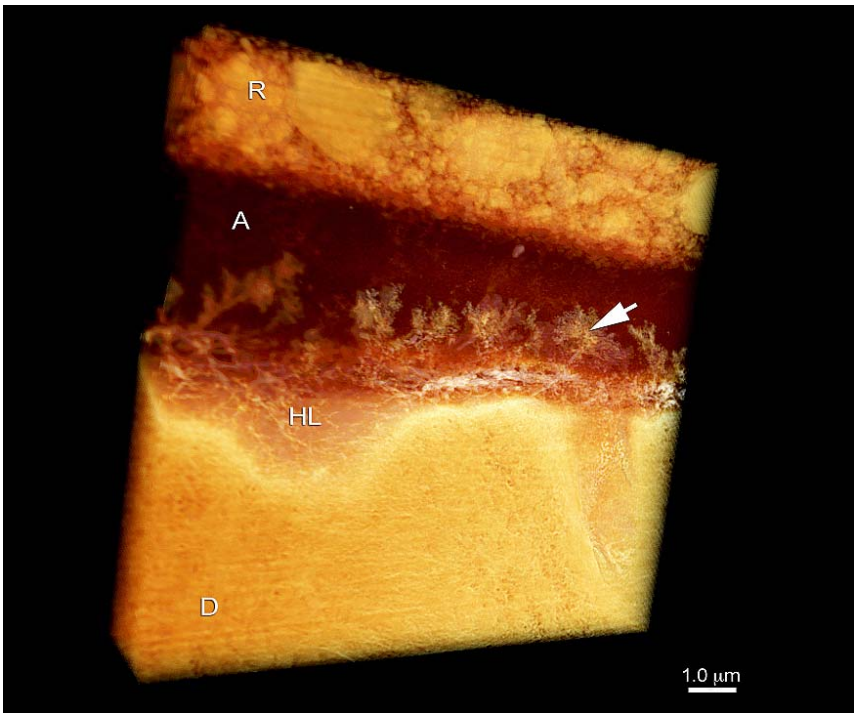

Fig 2: Three-dimensional reconstruction of the resin-bonded interface showing the nanoleakage within the hybrid and adhesive layer. (D: dentin, HL: hybrid layer, A: adhesive layer, R: resin composite, White Arrow: nanoleakage).

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