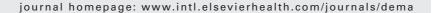


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## A study of the adhesion between dental cement and dentin using a nondestructive acoustic microscopy approach

Liudmila A. Denisova<sup>a</sup>, Roman Gr. Maev<sup>b</sup>, Valery K. Leontjev<sup>c</sup>, Alexandre F. Denisov<sup>a</sup>, George G. Grayson<sup>e</sup>, Fedor S. Rusanov<sup>c</sup>, Eugeny Yu. Bakulin<sup>b,\*</sup>, Dmitry Yu. Gavrilov<sup>b</sup>, Tatiana V. Grineva<sup>d</sup>

- <sup>a</sup> International Center for Advanced Material Characterization, Emanuel Institute for Bio-chemical Physics RAS, Moscow, Russia
- <sup>b</sup> Department of Physics, University of Windsor, 401 Sunset Avenue, Windsor, Ontario, Canada N9B 3P4
- <sup>c</sup> Material Research Laboratory of the Central Research Dental Institution, Moscow, Russia
- <sup>d</sup> Stomadent Ltd., Tomilino, Moscow Region, Russia
- <sup>e</sup> Ultradent Clinical Research Ltd., Windsor, Ontario, Canada

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#### ABSTRACT

Objectives. The goal of the present study was to investigate the potential for acoustic microscopy techniques to characterize the cement–dentin interface in restored teeth.

Methods. Special flat-parallel specimens and whole extracted teeth with restorations were scanned using a high-frequency (50 MHz) focused ultrasonic transducer. Visual acoustic images (B- and C-scans) of the cement-dentin interface were obtained nondestructively, analyzed and compared with optical images taken after the samples were cut along the scanning axis. The shear bonding strength of a subsection of specimens was tested in a Lloyd material testing machine.

Results. An essential distinction between the acoustical properties associated with good and failed bonding has been shown. In the case of failed adhesion, the ultrasound signal reflection from the cement–dentin interface is up to four to seven times higher in magnitude than in the case of good bonding. The comparison of the ultrasound imaging data with the data obtained using an optical microscope revealed a strong correspondence with the acoustical and optical results with respect to the presence, position and dimensions of the defects. The specimens showing higher ultrasound reflection from cement/dentin interface have also shown lower shear bonding strength.

Significance. The results demonstrate that acoustic scanning with a high-frequency focused ultrasonic probe is a valuable method for nondestructive morpho-mechanical analysis of cement/dentin interface for either experimental models or whole restored teeth. An appropriately expanded approach can be widely used for the pre-clinical evaluation of dental materials. Further, this method may prove beneficial in the design of new diagnostic ultrasound devices and techniques for use within clinical dentistry.

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<sup>\*</sup> Corresponding author. Tel.: +1 519 253 3000x2661; fax: +1 519 973 7075. E-mail address: bakulin@uwindsor.ca (E.Yu. Bakulin).

#### 1. Introduction

In many ways, the degree of the adhesion between restoration materials and dentin determines the overall effectiveness of dental care. Therefore, it is of paramount importance in the field of dental materials research and, ultimately in clinical practice, to quantify the bonding strength. Typically, light or electron microscopy as well as other specialized methods are used for the evaluation of restoration/dentin adhesion. However, most of these techniques are destructive in nature, and, as such, can be applied only after preparing experimental samples by cutting the tooth into halves or a series of thin sections. For this reason, these existing methods do not permit the dynamic investigation of peculiarities which develop at the restoration/dentin interface. In short, these methods are not transferable to in vivo evaluations of cement/dentin bonding and therefore cannot be applied in the diagnosis of such disorders as, for example, secondary caries that may develop beneath the restoration. For this reason, a novel approach to the nondestructive evaluation of cement-dentin adhesion is

The physical properties and technology of dental cements' are closely related to those of construction cements, for which a great number of nondestructive ultrasonic methods have been designed to evaluate their condition, physical properties and structure, as well as to characterize their hardening, aging and adhesion to other materials [1,2]. However these methods, as a rule, employ ultrasound probes with wide flat contact surfaces that are not suitable for the inspection of restored teeth. Also, the characterization of construction cements requires ultrasound frequencies in the range of 20-200 kHz, providing resolutions not higher than 1-2 cm. Unfortunately, due to its geometrical shape and dimensions, this is not sufficient for the evaluation of human teeth. Acoustic microscopy, on the other hand, uses a high-frequency (25-2000 MHz) acoustic wave, which is focused by means of a special acoustic lens into a narrow beam, whose diameter (1-100 µm) determines the precision of measurement and ultimately the resolution of the acoustic image [3]. Also beneficial is the fact that with focused ultrasound there is no need to ensure good acoustic contact between the probe and tooth surface – this is because the acoustic signal travels to the object's surface through an immersion media (ultrasound gel or water).

The first attempts to use high-frequency focused ultrasound to visualize the cement/dentin interface were undertaken in the investigation of the flat surfaces of the sections performed through the middle of restored teeth [4,5]. Here it has been shown that scanning with a focused ultrasonic probe (acoustic lens) enables one to obtain valuable information concerning the bonding conditions at the filling-tooth interface, as well as the condition and spatial distribution of the restorative material itself at the tooth tissue interface. In an earlier study [6], a scanning acoustic microscope was used to evaluate the microstructure and acoustic properties of a hard glass-ionomer cement. In this work a strong correlation was established between the acoustical parameters and the mechanical strength of the material. In another study [7], one tooth with amalgam restoration was examined in vitro using focused ultrasound (frequency 30 MHz) in A-scan and B-scan

modes, demonstrating the capacity to measure restoration thicknesses. Another example where focused ultrasound is used to determine the thickness of amalgam restoration in an extracted tooth is shown in an article [8].

The aim of the present study was to study and compare the peculiarities of ultrasound reflections from the restoration–dentin interface in the case of both good and failed bonding. In this work special flat-parallel layered models and whole extracted teeth with experimentally performed restorations were used.

#### 2. Materials and methods

This study was carried out using human teeth, which were collected through a local dentist from participating extramural private dental practices without concern to the patients' age, sex, health condition. Samples included pre-molar and molar teeth from consenting patient volunteers undergoing scheduled tooth extractions for orthodontic, orthopedic or other reasons. The samples were rinsed thoroughly and then sterilized by gamma irradiation and stored in the Thymol solution or in wet refrigerated chambers at 4°C. Before the model preparation or investigation with acoustic and light microscopes, all samples were stored in their own vial, anonymized and assigned an identification number. All samples were studied with short-pulse, high-frequency focused ultrasound using an acoustic microscope "Tessonics 1103" (Tessonics Corp., Canada). An electric pulse was applied to the piezoelement on the top of a fused quartz rod, in turn generating a short-pulse acoustic signal. This signal then propagated through the rod and, due to refraction by a spherical recession (called acoustic lens) at the end of the rod, converged at a small focal point (Fig. 1). Water was used as a coupling media between the probe and the object. In all the experiments an acoustic lens having a working frequency of 50 MHz and a halfaperture angle of  $15^{\circ}$  was used – this corresponded to a lateral resolution of approximately 0.07 mm. The lens was focused on the object's surface and the ultrasonic pulses reflected from the object's surface as well as from inhomogeneities in the bulk material were received by the piezoelement and recorded as a voltage vs. time delay (this produces an oscillogram showing the succession of reflections called A-scan). By scanning the lens horizontally in one direction, the A-scans were recorded at each position along the scanning line, and combined into a two-dimensional image called B-scan, where the horizontal axis corresponds to the scanning direction and the vertical axis corresponds to the time delay. By scanning the lens in two perpendicular horizontal directions and selecting a particular time interval (the so-called "C-scan gate") corresponding to a reflection of interest, images of an acoustical cross-section under the object's surface are obtained, which are called C-scans. A C-scan consists of several thousand pixels; the gray level (brightness) of each pixel is linearly proportional to the average of the absolute value of the signal within the thickness of the C-scan gate. A more detailed description of acoustic microscopy and, in particular, the technique used in the study can be found in [9].

In the first experimental series, the cement-dentin interface was imaged and evaluated using special models. The

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