APPLICATION OF ACOUSTIC MICROSCOPY TO DENTAL MATERIAL CHARACTERIZATION

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ABSTRACT

Acoustic microscopy, which has been established for the evaluation of the elastic properties of solid materials in imaging and in quantitative measurements, is introduced into the dental field as a research tool to investigate directly the change of visco-elastic properties in dental hard tissues (enamel and dentine) associated with the progress of caries. A point-focus-beam (PFB) acoustic microscope is employed to image primary caries lesions in human enamel with a spatial resolution of 3 μm at 400 MHz in both amplitude and phase. Destruction of structure and decrease of acoustic impedance, mainly due to the demineralization, are observed. A method of quantitative material characterization, using the V(z) curve analysis of line-focus-beam (LFB) acoustic microscopy, is applied to study changes in the visco-elastic properties associated with the demineralization. Preliminary acoustic measurements are reported for the hard tissues of human enamel and dentine, and also for a hydroxyapatite ceramic. It is found that in order to analyze the visco-elastic properties the propagation characteristics of leaky surface acoustic waves (LSAWs) for enamel and of leaky surface skimming compressional waves (LSSCWs) for dentine, as a function of frequency, should be made.

1. Introduction

In the dental research fields, the fundamental problem of the change of visco-elastic properties in the dental hard tissues of enamel and dentine associated with the progress of caries remains unsolved, although the therapeutic technology of treating decayed teeth has developed successfully. Some kinds of ultrasonic methods, such as the pulse interference method [1] and the critical angle reflection method [2], have been employed for dynamic testing of teeth and for determination of elastic constants of dental materials. However, ultrasonic instruments suitable for the research purpose do not exist. Recently, acoustic microscopy has been established as a most useful technique for material analyses in imaging and quantitative measurement in material science and nondestructive evaluation [3,4]. Peck and Briggs [5] have been trying to demonstrate the usefulness of acoustic microscopy in dental fields, especially imaging in comparison with conventional optical microscopy and microradiography. Since 1981, when we developed the quantitative material characterization method by means of the LFB acoustic microscope, studies on the quantitative analysis of materials have been conducted extensively and successfully made to exploit the practical applications [6-9].

In this paper, acoustic microscopy which combines LFB and PFB acoustic microscopes, is introduced to investigate directly the visco-elastic properties of the dental hard tissues of enamel and dentine associated with demineralization. A new LFB system, suitable for this study, is developed and the fundamental problems in applying the system to dental material characterization are discussed.

2. Composition and Structure of Tooth

Dental hard tissues of human enamel and dentine are composed of minerals (hydroxyapatite; HAP), organic materials (keratin in enamel and collagen in dentine) and water. The representative composition is given in Table 1. Hydroxyapatite, which is the chief component of dental hard tissues, has hexagonal-like crystalline symmetry characteristics [10], and enamel and dentine are described as a pseudo-hexagonal in which the c-axes are approximately in line with the enamel rods and dentinal tubules, respectively [2].

As far as we know, complete data of the elastic properties of human enamel and dentine are not

Table 1. Representative composition of human enamel and dentine*.

	mineral (%)	organic material (%)	water (%)
Enamel	92	2	6
Dentine	64.5	29	6.5

* S.N.Bhaskar, "Orban's oral histology and embryology", 8th Ed., C.B.Mosby Co.(1976).

Table 2. Bulk and LSAW velocities, V_{ij}(m/sec), and densities, ρ (Kg/m^3), in hydroxyapatite and bovine incisor.

	V_{11}	V_{33}	V_{44}	V_{66}	V_{1saw}^{*}	ρ	Ref.
HAP	6574	7366	3534	3863	3350	3200	a
Enamel	6297	6565	2804	3538	2747	2900	b
Dentine	4101	4210	1610	2153	1682	2200	b

* LSAW on the basal plane
a) J.L.Katz and K.Ukraincik, J.Biomechanics,Vol.4,pp.221-227(1971).
b) S.Lees and F.R.Rollins.Jr.,J.Biomechanics,Vol.5,pp.557-566(1972).

available. Table 2 comprises reference values to interpret our experimental results described later, viz., velocities for bovine hard tissues and hydroxyapatite, which are calculated from the data given by Lees and Rollins [2] and by Katz and Ukraincik [10], respectively.

Fig. 1 shows a typical optical micrograph of part of a human incisor in longitudinal section exhibiting the structure of enamel and dentine. It can be seen that the striae of Retzius run radially from the dentino-enamel junction to the surface of the tooth, and dentinal tubules start from pulp and appear like a highly striated columnar structure normal to the junction. Microscopically the structure of enamel appears as enamel rods approximately 5-7 µm in diameter stretching nearly normally to the surface of tooth, and transverse striations, produced by poor calcification associated with daily biorhythm, cross the enamel rods at intervals of approximately 3-5 µm. The striae of Retzius, crossing the enamel rods obliquely, are formed by period transverse striations with intervals of several tens of microns. It can be seen also from the figure that there is considerable variation in the orientation of enamel rods and dentinal tubules. In addition to such complex histological structure, defects such as cracks occurring in growth, are often observed.

Additionally, demineralization in hydroxyapatites and dissolution of organic materials in hard tissues results in structural destruction, so that significant changes in the visco-elastic properties are expected to appear.

3. Systems and Sample Preparation

3.1. Systems

A reflection-type PFB acoustic microscope system, which operates in both amplitude and phase modes, was employed. The amplitude mode is useful for morphological observation of the dental lesion and its progress. Interpretation of contrast in the images obtained by this mode is made with leaky wave propagation [3]. The phase mode is also useful for topographic evaluation of surface status of polished specimens, and the unevenness is readily calculated from the interferogram [11].

Fig. 1. Optical micrograph of part of human incisor in longitudinal section.

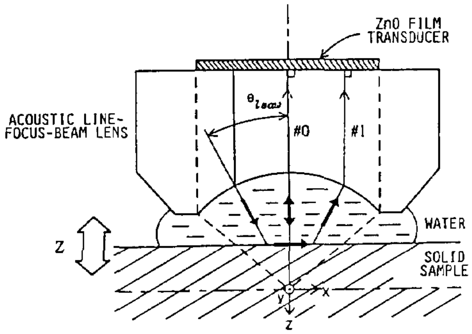

Fig. 2. Cross-sectional geometry of acoustic line-focus-beam lens for explaining the construction mechanism of V(z) curve.

The operating frequency used here is 400 MHz, using a PFB sapphire lens of 0.5 mm radius, and the spatial resolution is approximately 3 µm.

An LFB acoustic microscope was employed to investigate quantitatively the visco-elastic properties of dental materials. The measurements are made using the V(z) curve analysis. Fig. 2 illustrates the basic concept. The system and the measurement principle have been described in detail [4]. The LFB excites leaky waves on the boundary between the specimen and water at the critical angle

$$\theta_l = \sin^{-1}(V_w/V_l) \, , \qquad (1)$$

where V_l is the velocity of leaky waves, and V_w is the longitudinal velocity in water, respectively. By analyzing measured V(z) curves, the propagation characteristics, namely, the velocity V_l and the normalized attenuation factor α_l, of leaky waves can be determined. The complex wave number is defined as

$$k_l = (\omega/V_l)(1+j\alpha_l) \, . \qquad (2)$$

As described above, dental hard tissues are composed of mineral, organic materials, and water with complex histological and structural textures. Therefore, the attenuation in this case should be defined as

$$\alpha_l = \alpha_w + \alpha_a + \alpha_s \, , \qquad (3)$$

where α_w, α_a, and α_s are the normalized attenuation factors due to the water loading, absorption, and scattering losses, respectively. α_w is independent of the acoustic frequency, while α_a and α_s are dependent on frequency.

A new LFB acoustic microscope system combined with an optical microscope, for finding proper positions for measurements of such dental materials with complex textures, was developed. The system operates in the frequency range of 100-275 MHz using an LFB sapphire lens of 1.0 mm radius, with a half aperture angle of 60°.

3.2. Sample preparation

In applying the new technology of acoustic microscopy to the study of dental hard tissues, it is important to establish the technique of polishing dental tissues, which are composite materials consisting of the hard and soft components and including cracks in the enamel. The following procedure was developed: Each tooth specimen is embedded in a Pyrex-glass housing with an Araldite resin and supported with a dental composite resin which is actually used to repair human teeth, as shown in Fig. 3. The dental composite resin is very useful for obtaining a smooth surface with sharp edges, as it has almost the same hardness as dental hard tissues.

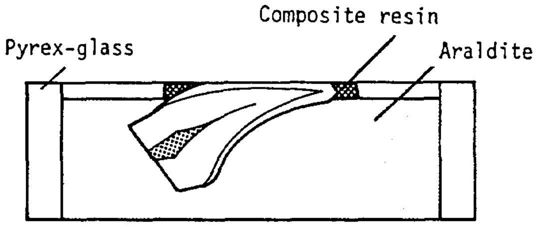

Fig. 3. Sample configuration in polishing process.

For imaging measurements of the lesions, a human incisor with primary dental caries was cut in transverse section perpendicular to the tooth axes, and for quantitative measurements, sound human incisors were ground in parallel to the front surface of teeth. All processes for producing the flat surfaces were carried out wet, and then preserved with saline in a refrigerator. The teeth were kept fresh, until the measurements were completed.

4. Results and Discussion

4.1. Acoustical image of primary dental lesions

Fig. 4 shows the amplitude image (a) and the interferogram (b) at 400 MHz for primary caries lesion of human incisor. As known from the interferogram, the unevenness is estimated to be less then 0.8 µm, because a fringe shift corresponds to the change of about 2 µm. Thus, the surface finish is considered to be adequate. The amplitude image was taken at a defocus of 15 µm. The caries lesion is contrasted well as a dark region in the amplitude image. It is clear that the subsurface enamel region in primary caries is strongly demineralized, softened, and the structure destroyed, although the external surface of the tooth is comparatively intact. Consequently, it should be interpreted that the contrast in the image is mainly caused by decrease of acoustic impedance and the sound scattering due to demineralization.

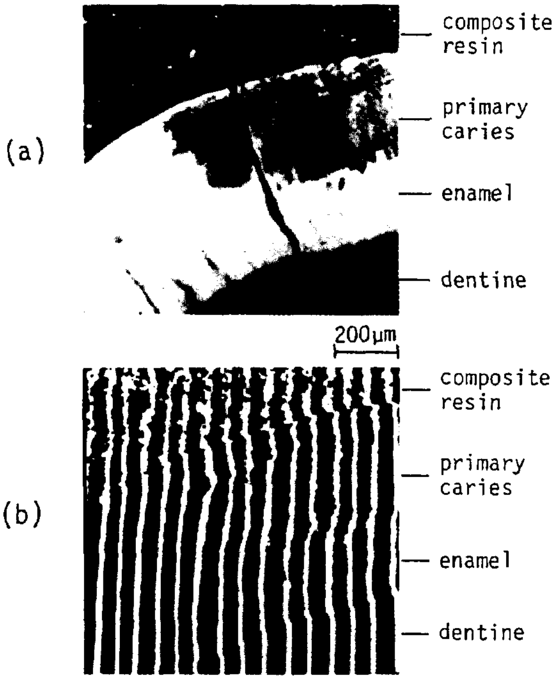

Fig. 4. Acoustic image of a primary dental lesion in human enamel observed at 400 MHz. (a) Amplitude image, (b) Interferogram

4.2. Quantitative measurements

To investigate the fundamental characteristics of dental materials on a microscopic scale, measurements were made on a hydroxyapatite ceramic (HAP: 99%), healthy human materials of enamel and dentine, using an LFB acoustic microscope at 225 MHz.

A. Modes for characterization

Fig. 5 shows typical V(z) curves of HAP ceramic, human enamel and dentine, together with the final results of propagation characteristics of leaky waves. According to the processing procedure of V(z) curves [4], it is easy to determine propagation characteristics, viz., velocity and attenuation, of leaky waves which are dominantly detected and recorded in the V(z) curves. To interpret measured results, we must first know the kind of leaky modes present. The modes, which contribute to the V(z) curves, and their propagation characteristics, can be understood in reference to the data of Tables 1 and 2. From the V(z) curves obtained for HAP ceramic and enamel specimens, LSAW characteristics can be measured to be V_{lsaw} = 3581 m/s and α_{lsaw} = 2.28×10^{-2} for HAP ceramic, and V_{lsaw} = 3153 m/s and α_{lsaw} = 2.73×10^{-2} for enamel, respectively. The LSAW velocity for enamel is slower than that for HAP ceramic, while the attenuation is higher than that for HAP ceramic. This is mainly due to the difference in HAP content. On the other hand, in a case of dentine, the calculated critical angle θ_{lsaw} of about 63° for LSAW mode is larger than the half aperture angle of 53.4° in water of the LFB acoustic lens, so that an LSAW mode cannot be excited on the boundary and information on an LSSCW mode is included in the V(z) curve. The velocity V_{lsscw} is determined to be 4198 m/s, and the attenuation α_{lsscw} is 4.75×10^{-2}. Comparing the values for the human materials with the data for the bovine dental materials, given in Table 2, it can be imagined that there are considerable differences between them in elasticity and density. This agrees with results reported previously [12]. From these experiments, it is believed that enamel and dentine materials should be characterized through LSAW and LSSCW modes, respectively, in applying the LFB acoustic microscope to characterization of dental hard tissues.

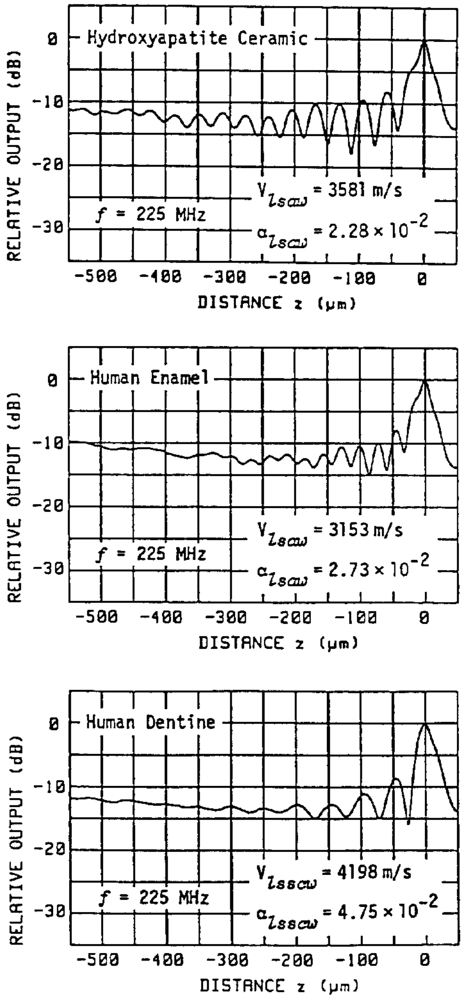

Fig. 5. Typical V(z) curves measured for dental materials.

B. Anisotropy

Experiments on acoustic anisotropies were carried out for the human enamel and dentine specimens, described above, as a function of the propagation directions. Fig. 6 shows typical anisotropies observed at 225 MHz. Velocity and attenuation vary remarkably with the directions. For the enamel specimen, the LSAW velocities range from about 3105 m/s to about 3155 m/s and the maximum is obtained in the direction (θ =0°) parallel to the tooth axis, while the maximum attenuation is obtained in the perpendicular direction (θ =90°). For the dentine specimen, similar dependences of the velocity and attenuation for the LSSCW mode are observed, although the values change considerably in comparison with those for enamel. It might be considered that these anisotropies are related to the following: (a) slight inclination of the c-axis, (b) spatial variation of the orientation of enamel prisms and dentinal tubules, and (c) structural anisotropies of striae of Retzius, transverse striations, and curved trajectory of dentinal tubules.

C. Inhomogeneity

The velocity variations on two other specimens of enamel and dentine were measured at 225 MHz by line-scanning along the direction parallel to the tooth axis. The results are given in Fig. 7. In the case of enamel, the LSAW velocities around the incisal portion are much faster than those around the cervical portion. In the case of dentine, the LSSCW velocities around the incisal part is about 200 m/s slower than those for the other part. In the central portion, the velocities were distributed throughout the range of 3850±50 m/s. These inhomogeneities in the velocities might also be related to the structural and compositional variations in materials depending on their positions. This argument may be supported by comparing the experimental results with the optical micrographs showing the structural variations close to the surface, as given in Fig. 8. For example, in a particular region corresponding to the slower velocities in the dentine specimen, low concentration of mineral (HAP) read from the picture results in the decrease of the LSSCW velocity.

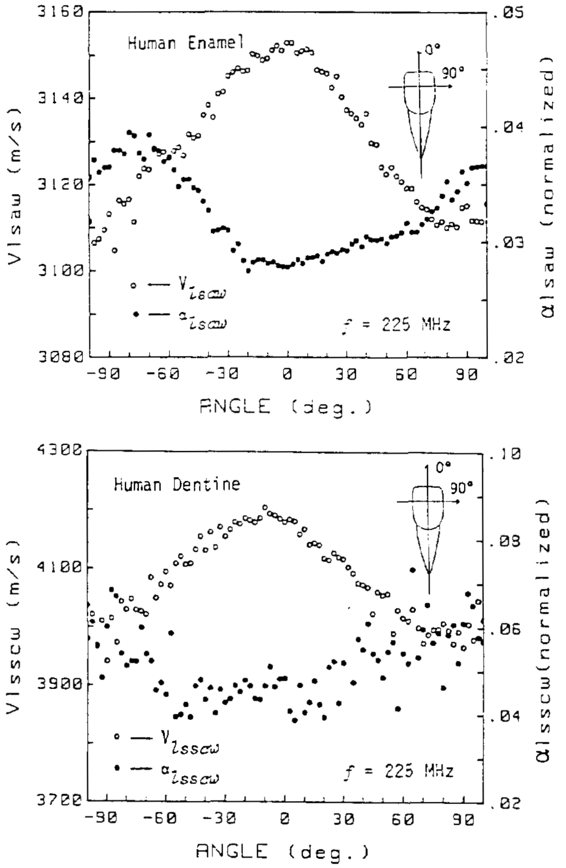

Fig. 6. Propagation characteristics of leaky waves in human enamel and dentine.

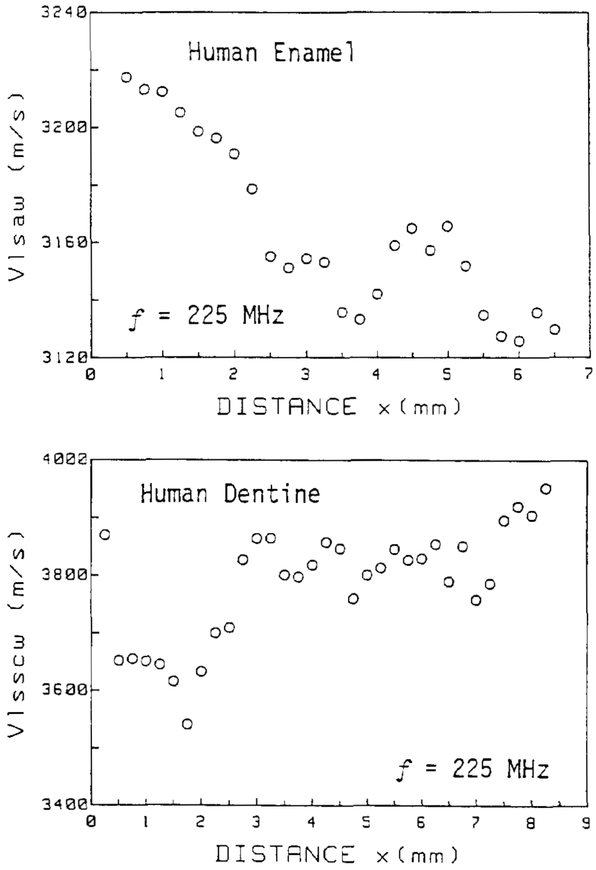

Fig. 7. Velocity variations in the parallel direction to tooth axis in human enamel and dentine.

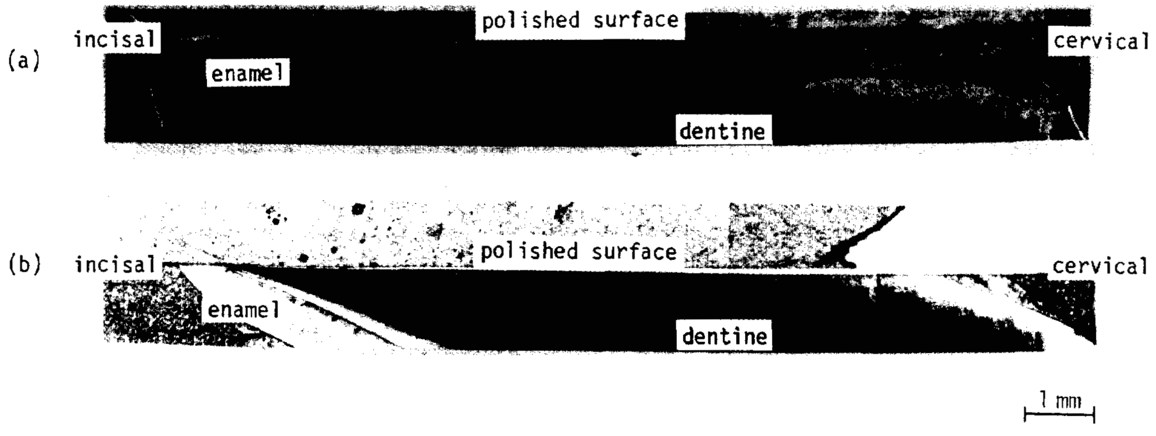

Fig. 8. Optical micrographs of structural variations in human incisor. (a) Human enamel sample, (b) Human dentine sample.

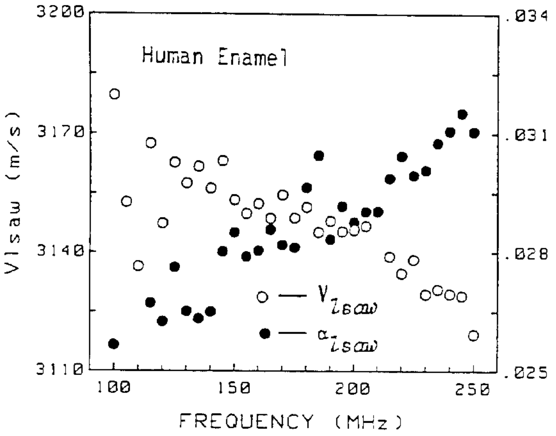

Fig. 9. Frequency dependence of LSAW velocity and attenuation in human enamel.

D. Frequency dependence

The frequency dependence for LSAW mode on human enamel was measured ranging from 100 to 250 MHz, where waves propagated in the direction parallel to the tooth axis. Fig. 9 shows the results. Both the velocity and attenuation vary significantly with the acoustic frequency. As the frequency increases, the velocities decrease and the attenuations increase.

As described previously, dental tissues are composed of mineral, organic materials and water, and have complicated histological structures. These factors influence the propagation characteristics of leaky waves and give rise to the dispersion. Therefore, it should be noted that it is necessary to measure the propagation characteristics of related leaky waves as a function of frequency, in order to be able to analyze completely the visco-elastic properties of dental materials.

5. Concluding Remarks

In this paper, acoustic microscopy has been discussed in the dental research fields as a new technology to investigate directly visco-elastic properties of dental materials, such as enamel and dentine. The quantitative applications of an LFB acoustic microscope, as well as the imaging applications of a PFB acoustic microscope, have satisfactorily demonstrated the possibilities. In particular, the LFB acoustic microscope can be expected to play an important role as a powerful tool to solve basic and practical problems in all kinds of dental materials, including dental hard tissues of enamel and dentine, and dental porcelains, metals and composites for repairing human teeth. To develop the method further in this field, it is necessary to conduct basic studies by combining the LFB acoustic microscope method with the conventional bulk method, or the critical angle reflection method [2], according to the way presented in the literature [6].

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