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Measuring Thickness of Thin Film Whose Sound Velocity is Unknown Utilizing Acoustic Resonant Spectroscopy

音響共鳴法を利用した音速が未知の薄膜の膜厚計測

Takamasa Saito[1†], Iwao Matsuya[1], Kiyoshi Ohnuma[1], Takashi Kuwahara[1], Mizuki Kondo[1], and Ikuo Ihara[1] ([1]Nagaoka Univ. of Tech.)

齊藤崇允[1†]，松谷 巌[1]，大沼 清[1]，桑原敬司[1]，近藤みずき[1]，井原郁夫[1]([1]長岡技科大)

1. Introduction

Recently, biofuel cells are received adequate attention from the view point of some environmental advantages. In a biofuel cell, sugars are broken down and electrons are transferred directly between enzyme and a electrode by an activity of the enzyme. Conductive polymers such as thiophene, aniline and pyrrol play an important roll in this energy conversion system as immobilized enzyme carrier between enzyme and a electrode. So, controlling electropolymerization of these conductive polymers is absolutely critical [1]. Above all, measuring thickness of the thin film formed by the polymers on a metal electrode during synthesis has highly engineering benefits. However, it is extremely difficult to measure thickness because a thin film of polymer is formed by randomly tangled fibers so that its mechanical properties and acoustic properties are inhomogeneous. In addition, thickness or even acoustic parameters will change if the polymers are taken out of the liquid solution after synthesis. For this reason, a novel method which can measure thickness of these materials during synthesis is strongly demanded.

Acoustic resonant spectroscopy is a potential candidate for measuring thickness of such a thin film because this method using ultrasound is characterized by a non-invasive inspection and its high accuracy [2-4]. Tohmyoh et. al. reported the technique which can measure the thickness of not only top side but also back side of coatings on a metal plate by using a resonant frequency of the transmitted ultrasound. When the coating thickness coincides with a quarter wavelength of the transmitted ultrasound, the thickness of the coating can be calculated precisely with a known value of a sound velocity [5, 6]. However, only the material whose acoustic property is homogenous inside the film can be measured by the above mentioned techniques because the known sound velocity information has to be utilized as the constant value to derive the thickness of the thin film.

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matsuya@mech.nagaokaut.ac.jp

Therefore, a simultaneous in-situ measurement method of both thickness and sound velocity is required for measuring thickness of thin film formed by these polymers.

In this paper, a novel method for measuring the thickness of thin film whose sound velocity is unknown by combining an acoustic resonant spectroscopy and an ultrasonic pulse-echo method is proposed. Using this method, both the thickness and the sound velocity of epoxy coating on metal plate are simultaneously measured experimentally and an availability of this method is verified.

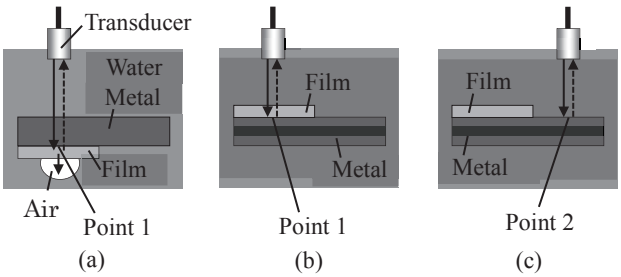

Fig. 1 Schematic of measurement theory. (a) Acoustic resonant spectroscopy, (b) time-domain measurement on thin film and (c) on metal plate. Solid arrows show transmitted ultrasound and dashed arrows show reflected ultrasound.

2. Measurement theory

Figure 1 shows a schematic of both thickness and sound velocity measurement method for a thin film on a metal plate. A metal plate is covered with a thin film not all over the plate but partially as a test specimen. An underwater type ultrasound transducer is used as an ultrasound transmitter and a receiver. A measurement procedure comprises following three steps. An acoustic resonant spectroscopy is conducted for a first step at point 1 as shown in **Fig. 1 (a)**. The test specimen is placed horizontally in the water and the surface of the thin film is arranged to be downward against the transducer. The air layer is attached on the thin film at point 1 to emphasis acoustic resonant phenomenon. When the acoustic resonant phenomenon is occurred, a following relationship is obtained by detecting the resonant frequency [5, 6],

$$f_r = \frac{v}{4d} \quad (1)$$

where f_r is resonant frequency, v and d are sound velocity and thickness of the thin film, respectively. As shown in **Fig. 1 (b)** and **Fig. 1 (c)**, time-domain measurements are carried out at point 1 for a second step and at point 2 where there is no thin film on the metal plate for a third step. The surface of the thin film is arranged to be upward. Because the point 1 is covered with the thin film and the sound velocity v is faster than that in the water, the reflected waves from the point 1 reach faster than that from the point 2. Consequently, this delayed time Δt is expressed as follows,

$$\Delta t = \frac{2d}{v_w} - \frac{2d}{v} \quad (2)$$

where v_w is the known sound velocity in the water (1451.4 m/s). Therefore, both the thickness d and the sound velocity v of the thin film are derived simultaneously by eq. (1) and eq. (2).

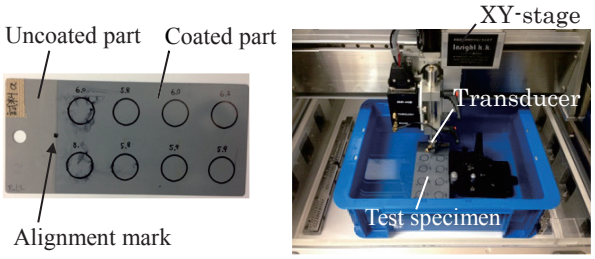

Fig. 2 Test specimen and experimental setup.

3. Experiment and results

Figure 2 shows a test specimen and an experimental setup. The ultrasound transducer whose nominal frequency is 75 MHz is fixed on the XY-stage. The 1 mm steel plates that are partially covered with the epoxy coatings are used as the test specimen. The coatings are 15 μm and 20 μm in nominal thickness. An aluminum tape of square, 2.5 mm on a side is pasted on the borderline between the uncoated part and the coated part as the alignment mark. Both time domain and frequency domain analyses are applied to the reflected waves.

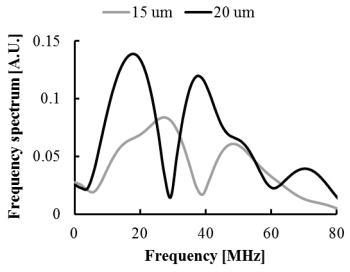

Fig. 3 Frequency spectrum of reflected waves.

Figure 3 shows frequency spectrums of reflected waves on which the typical acoustic resonant phenomena are seen. The resonant frequencies are obtained at the minimal value of the spectrum, which are 39.1 MHz for the 15 μm-coated specimen and 29.3 MHz for the 20 μm-coated specimen, respectively. **Figure 4** and **Figure 5** show waveforms of reflected ultrasound. The delayed times are 9.63 ns for the 15 μm-coated specimen and 10.00 ns for the 20 μm-coated specimen, respectively. The calculated sound velocities and the thicknesses are summarized in **Table I** including the results from conventional acoustic resonant spectroscopy with the known sound velocity (2300 m/s) for comparison. As compared with the conventional method, the sound velocities and the thicknesses of the coating are successfully obtained with high accuracy by the proposed method.

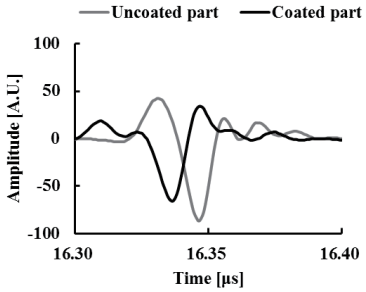

Fig. 4 Waveforms in 15 μm-coated test specimen.

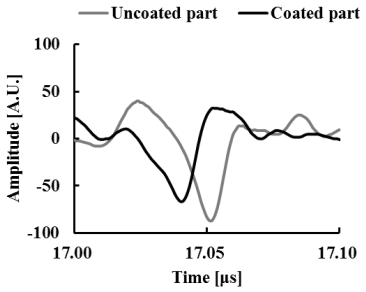

Fig. 5 Waveforms in 20 μm-coated test specimen.

Table I Experimental results.

Specimen	Proposed method		Conventional method	
	v m/s	d μm	v m/s	d μm
15 μm	2543	16.28	2300	14.72
20 μm	2301	19.64	(Const.)	19.63

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