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Characterization of Polycrystalline Diamond Films by the LFB Ultrasonic Material Characterization System

LFB 超音波材料解析システムによる多結晶ダイヤモンド膜の評価

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1. Introduction

Polycrystalline diamond films have intensively been employed for surface acoustic wave (SAW) devices at GHz band because of the very high SAW velocity and their high quality fabricated [1, 2]. Performance of the SAW devices depends on acoustic properties of their constituent materials. Especially for high-frequency applications, device characteristics are significantly affected by not only the elastic properties but also SAW scattering at polycrystalline grain boundaries. Therefore, it is important to develop a method for evaluating the acoustic properties including scattering effects due to polycrystalline structure.

We have aimed to establish the evaluation method of thin films fabricated on bulk substrate by the line-focus-beam ultrasonic material characterization (LFB-UMC) system. This system can precisely measure the propagation characteristics (phase velocity and propagation attenuation) of leaky surface acoustic waves (LSAWs) propagating on a water-loaded specimen surface [3]. Although measurement values of LSAW propagation characteristics include not only elastic information but also scattering effects due to structural factor [3], the scattering effects have not yet been discussed in detail.

In this paper, we investigate an effect of structural difference in polycrystalline diamond films on their acoustic properties, through measurements of frequency dependence of LSAW propagation characteristics for polycrystalline diamond films fabricated by two methods with different grain sizes.

2. Specimens and numerical calculation

Several polycrystalline diamond films with different grain sizes were fabricated by the microwave plasma chemical vapor deposition (MPCVD) method and the hot filament CVD method as shown in **Table I**.

Before making experiments, we conducted numerical calculations of leaky acoustic wave (LSAW and leaky pseudo acoustic wave: LPSAW) propagation characteristics for specimen structure illustrated in **Fig. 1**. Substrates were taken as (001) Si single crystal and polycrystalline Si substrates (Table I). For calculation, we used elastic constants and density of Si single crystal in the literature [4] and elastic constants of polycrystalline diamond film and polycrystalline Si substrate, estimated from the constants of their single crystals [4, 5] according to Hill's approximation [6] for a model of homogeneous isotropic polycrystalline material, with the densities of the single crystals. **Fig. 2** shows the calculation results of *fH* (product of frequency *f* and film thickness *H*) dependence of the propagation characteristics ignoring the scattering effect; thick solid lines are for LSAW mode, thick dashed lines and dotted lines are for LPSAW mode. In Fig. 2, we also show the calculated values of longitudinal and shear velocities, LSAW velocity and normalized attenuation for (001)-[110] Si single crystal, and normalized attenuation of polycrystalline diamond.

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3. Experiments

The measurement principle of leaky acoustic waves using the LFB-UMC system was described in detail in the literature [3]. We measured the propagation characteristics of the leaky acoustic waves in a frequency range from 100 to 300 MHz for each specimen in Table I. The measured results were presented in Fig. 2. For the UNCD films, we plotted data of LSAW propagation direction along the [110] direction of (001) Si substrate. The measured velocities for the UNCD films were by 200-1200 m/s lower than the calculated ones of LSAW mode. The measured normalized attenuation was almost flat different from the calculated ones varying with *fH*. For the NCD film, although the measured values of velocities and attenuations were close to the calculated ones for the LPSAW mode, but the values decreased for velocity and increased for attenuation with increasing *fH*, as comparing to the calculated ones. The measured values for the MCD film were higher than the calculated ones by 300-1000 m/s for velocity and by 0.03-0.05 for attenuation.

4. Discussion

In general, the scattering effects of polycrystalline structure for bulk waves make velocity lower and attenuation higher. The changes depend on D/λ, which is ratio of grain size D to wavelength λ, and the higher D/λ makes the scattering effects greater. D/λ is 0.01-0.03 for the NCD film and 0.04-0.16 for the MCD film and so we can understand that scattering loss for the MCD film is greater than that for the NCD film in Fig. 2.

In the results of the NCD film, it might be considered to detect the effect of scattering in the film that the measured velocities decrease and the measured attenuations increase comparing to the calculated ones with *fH* increase. The attenuation for the MCD film is significantly greater than that for the NCD film, so the scattering effects in the MCD film is very large. However the velocities for the MCD film are larger than that for the NCD film. We observed velocity distribution due to film-thickness distribution on the MCD film (the maximum velocity difference of ±290 m/s at 225 MHz corresponding to ±1.5-μm difference in film thickness). We measured the velocity at the position exhibiting the maximum velocity (maximum thickness). The measured velocity is 190 m/s greater than the calculated one at 225 MHz even if we assumed the film thickness was 16.5 μm at this position. This velocity change corresponds to 10% increase in elastic constants comparing to those calculated using Hill's

Table I. Polycrystalline diamond film specimens.

Fabrication process	Microwave plasma CVD Ar / CH_4(1%)	Hot filament CVD H_2 / CH_4(1~3%)	
Sample	UNCD (Ultrananocrystalline)	NCD (Nanocrystalline)	MCD (Microcrystalline)
Grain size	~ 5 nm	~ 1 μm	3 ~ 5 μm
Thickness	0.5, 1, 2, 5 μm	15 μm	
Roughness	~ 2 nm (As grown)	Optically polished	
Substrate	(001) Si 35 mm × 43 mm × 4~5 mm^t	Polycrystalline Si 50 mm$^\phi$ × 0.8 mm^t	

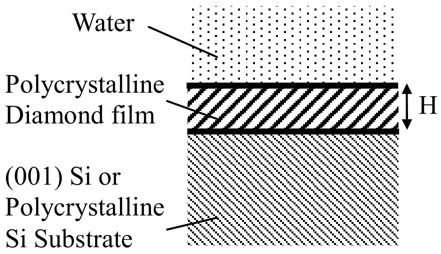

Fig. 1. Structural model for calculation.

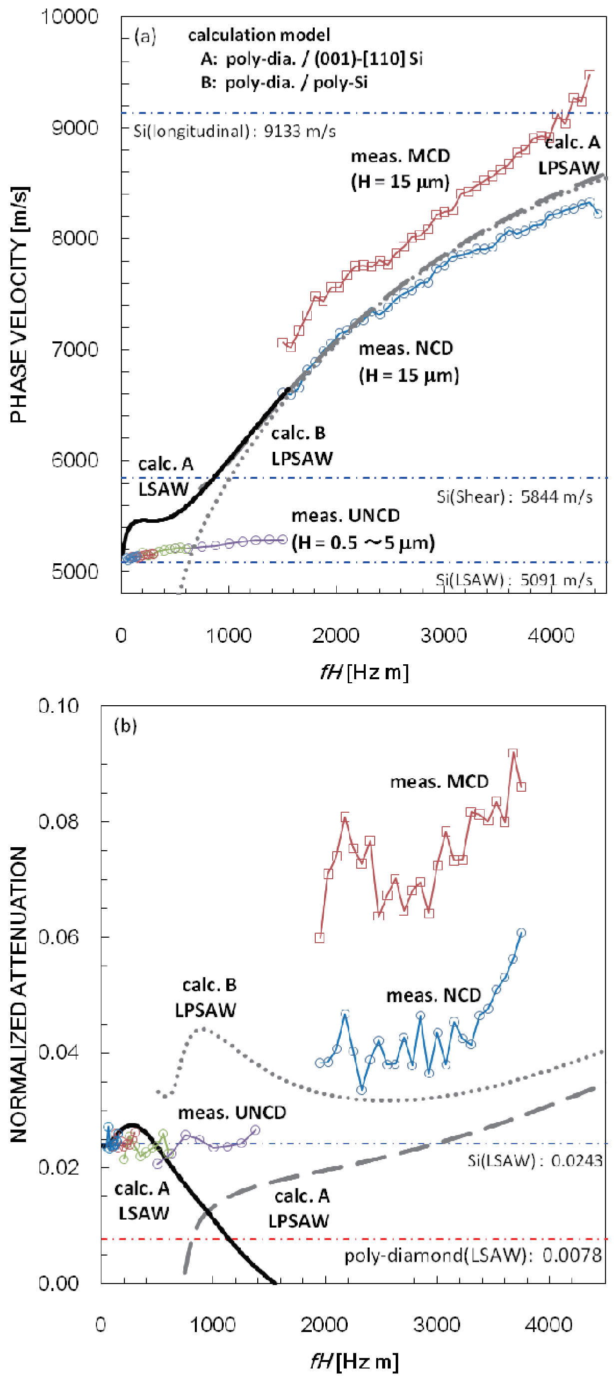

Fig. 2. Frequency dependences of LSAW propagation characteristics.
(a) Phase velocity. (b) Normalized attenuation.

approximation assuming that c_{11} and c_{44} change with an equal percentage. Therefore, it is considered that the increases of elastic constants cause increasing of measured velocity relative to the calculated one because the grain size of the MCD film become larger than that of the NCD film, then the MCD film become denser in structure. On the other hand, the calculated variation in the attenuation caused by the 10% increase in elastic constants described above is small and the situation that the measured value is greater than the calculated one does not change. From the discussion, we summarize that attenuation is insensitive to change in elastic constants but sensitive to change in scattering effects and the attenuation increases with D/λ. For the velocity, it might be considered that the increase amount caused by change in elastic constants is sufficiently larger than the decrease one caused by change in scattering effects.

Since D/λ for the UNCD film is 0.0001-0.0003, the scattering effects are sufficiently smaller than those for the NCD and MCD films. In the UNCD films, C-H bond is partially produced depending on the film fabrication condition, and it makes bonds among diamond particles weak [2]. Therefore, we consider that the velocity decrease for the results of the UNCD films is mainly caused by the decreasing elastic constants.

5. Summary

In this paper, we tried to characterize polycrystalline diamond films fabricated on Si substrates by two methods, through the measurements of the frequency dependences of LSAW velocity and attenuation by the LFB-UMC system. In the results, we observed significant differences in velocity and attenuation caused by the fabrication methods and conditions. In the near future, we will discuss the effects of polycrystalline structure on elastic constants and those of nano-grain size on scattering by preparing UNCD films with denser structure.

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