# Measurement of elastic stiffness of Fe/Cr multilayer by picosecond ultrasound

ピコ秒超音波法による Fe/Cr 多層膜の弾性率計測

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# 1. Abstract

Elastic stiffnesses of multilayered films are expected to be different from those calculated with bulk stiffnesses using the rule of mixture. This is because interfacial defects, alloy phases, and misfit strain at interfaces between dissimilar materials come into effect. They have been measured by several methods such as the micro-tensile testing<sup>1</sup>), vibration-reed method<sup>2)</sup>, surface-acoustic-wave method<sup>3)</sup>, and Brillouin scattering<sup>4)</sup>, to study relationship between elastic stiffnesses and multilayered structure. In these methods, forcedisplacement relaionships, resonance frequencies, and sound velocities in multilayer/substrate specimens are measured. Thickness of а multilayered film is generally much smaller than that of a substarte. Then, contributions of multilayer stiffnesses are small. Furthermore, these methods require dimensions and elsatic stiffness of a substrate. Therefore, it is not straightforward to measure elastic stiffness by the conventional methods. An unified model explaining elastic stiffness of multilayered films has not been obtained and unsderstanding elastic properties of multilayered films is still challenging.

We have established a method to measure the out-of-plane elastic stiffness  $C_{\perp}$  of thin films using picosecond ultrasound<sup>5</sup>). This method measures a sound velocity of longitudinal wave propagating in the thickness direction through a film, where elastic stiffness and dimesions of a substrate is not required for determining thin film's  $C_{\perp}$ . This method has been applied to multilayered films such as Fe/Pt<sup>6)</sup> and Co/Pt<sup>7)</sup>, and it was observed that lattice misfit affects  $C_{\perp}$  of multilayered films. However, only a couple of multilayered films have been investigated, and relationship between the lattice misfit and  $C_{\perp}$  has not been understood completely. For evaluating effect of misfit strain on  $C_{\perp}$  in detail, in this study, we measure  $C_{\perp}$  of Fe/Cr multilayered films. Its misfit strain is about 0.6%, which is much smaller than those of the above systems, e.g. 10% in Co/Pt multilayered films, and it enable us to evaluate misfit strain dependence of elastic property of multilayered films.

# 2. Specimen

Fe/Cr multilayered films were deposited on monocrystal (100) Si substrates covered with native oxide by RF magnetron sputtering at room temperature. The background pressure was 3.6- $4.5 \times 10^{-6}$  Pa, and the Ar pressure during deposition was 0.8 Pa. The thickness of the Fe layer was fixed to 1 nm, and that of the Cr layer was ranged from 0.3 nm to 2.5 nm. The number of bilayers was changed to keep the total film thickness of 60 nm for all specimens studied. Total thickness was determined by the x-ray reflectivity (XRR) measurement.

### 3. Picosecond ultrasound

Picosecond ultrasound<sup>8)</sup> is a technique for generating and detecting GHz-range longitudinal pulses propagating in the film-thickness direction, which uses ultrashort-pulse lights to measure the round-trip time  $\Delta t$  of an acoustic phonon multireflected at the film surface and the film-substrate interface<sup>8)</sup>.  $C_{\perp}$  is determined from  $\Delta t$ , the mass density  $\rho$ , and the film thickness d, through  $C_{\perp} = \rho(2d/\Delta t)^2$ . We used the pulse-laser beam of 800 nm wavelength with 140-fs duration. The source light was separated into the pump and probe beams, and the wavelength of the probe beam was changed to 400 nm by the second harmonic generator. Their powers are 50 and 25 mW, respectively.

### 4. Results and discussion

Figure 1 shows a typical XRR spectrum, in which two kinds of peaks appear: a broad peak at around  $2\theta$ =4.2° and many small peaks with small periodicities of  $1.0^{\circ}$ ~4.0°. The broad peak originates from the interference between X-rays reflected at the interfaces between the Fe and Cr layers. The appearance of this peak confirms high periodicity of fabricated specimens. The peaks of smaller period originate from the interference between X-rays reflected at the film surface and film-substrate interface. Total film thickness determines this periodicity, and *d* is given by fitting the theoretical XRR curve to the measurement.

Figure 2 shows the typical X-ray diffraction (XRD) spectrum, in which two diffraction peaks

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Fig. 1 XRR spectrum of [Fe (1 nm)/Cr (1.5 nm)]<sub>24</sub> by Co  $K\alpha$  radiation. The vertical axis is on a logarithmic scale.



Fig. 2 XRD spectrum of [Fe (1 nm)/Cr (1.5 nm)]<sub>24</sub>

appear. One is the diffraction peak originating from Si substrate and the other is the fundamental peak associated with the multilayered structure. The fundamental peak appeared between the diffraction angles of the Cr(110) planes and Fe(110) planes, which indicates that the <110> directions of Cr and <110> directions of Fe are oriented in the film thickness direction.

Figure 3 shows a typical reflectivity change measured by the picosecond ultrasound. The horizontal axis shows the delay time of the probe beam from the excitation, and the vertical axis shows the change ratio of reflectivity of the probe light. The reflectivity change shows the train of echo signals, and  $\Delta t$  is determined from the slope of the relationship between delay time and the index number of echo signal, *m*, as shown in the inset of Fig. 3.

Measured  $C_{\perp}$  was compared to elastic stiffness  $C_{\perp}^{\text{bulk}}$  calculated from the elastic constants



Fig. 3 Reflectivity change of [Fe (1 nm)/Cr (0.5 nm)]<sub>40</sub>. m is the index number of pulse-echo signals.  $\Delta t$  is determined from the relationship between m and the individual transit times (see the inset).

of single crystal Fe and Cr using the rule of mixture, assuming that the Fe(110) and Cr(110) planes are aligned parallel to the film surface, referring to the measured XRD spectra. As the result,  $C_{\perp}$  is almost the same with  $C_{\perp}^{\text{bulk}}$ . This trend is different from that observed in Co/Pt multilayered films<sup>7</sup>. In the Co/Pt multilayered films, smaller stiffness than  $C_{\perp}^{\text{bulk}}$  was observed, and it was explained by interfacial defects that release large interfacial stress. But, in Fe/Cr multilayered films, misfit strain is small. We consider that the smaller misfit strain causes less defective, and it makes  $C_{\perp}$ close to  $C_{\perp}^{\text{bulk}}$ .

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