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IN SITU STRESS MEASUREMENTS OF Co-BASED MULTILAYER FILMS

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Abstract - We have constructed an apparatus for *in situ* measurement of stress of the film prepared by sputtering using an optical non-contact displacement detector. A change of the gap distance between the detector and the substrate, caused by stress of a deposited film, was detected by a corresponding change of the reflectivity. The sensitivity of the displacement detector was 5.9 μV/Å and thus, it was turned out to be good enough to detect stress caused by deposition of a monoatomic layer. The apparatus was applied to *in situ* stress measurements of Co/X(X=Pd or Pt) multilayer thin films prepared on the glass substrates by dc magnetron sputtering. At the very beginning of the deposition, both Co and X sublayers have subjected to their own intrinsic stresses. However, when the film was thicker than about 100 Å, constant tensile stress in the Co sublayer and compressive stress in the X sublayer were observed, which is believed to be related to a lattice mismatch between the matching planes of Co and X.

I. INTRODUCTION

Stress in a film is known as a prime limitation to the growth of very thick films and an important factor that influences many physical properties of thin films. For instance, stress on the magnetic thin film affects magnetic anisotropy via inverse magnetostriction mechanism. In particular, the stress induced-anisotropy is often referred to one of the origins of perpendicular magnetic anisotropy in Co-based multilayer films[1].

Various methods have been suggested for stress measurements[2], among which the cantilever beam technique pioneered by Klockholm[3] is widely used. In this method, one side of the substrate is fixed by a substrate holder and the other side of the substrate is free to move. Stress of a film is determined by detecting the deflection of a thin substrate as the film is deposited on it. The cantilever capacitance methd [4] has been popularly adopted for *in situ* measurement of deflection. However, this method cannot be applied to a sputtering system, since the capacitance is largely influenced by plasma existed during sputtering process. Therefore, we have adopted an optical method, where a change of the reflectivity with deflection of a substrate was monitored.

In this paper, we describe an *in situ* stress-measurement apparatus using an optical displacement detector and report *in situ* stress measurements of Co/X (X=Pd, Pt) multilayer films prepared by dc magnetron sputtering.

II. EXPERIMENT

A. Description of an apparatus

In Fig.1, we depict a schematic diagram of an optical displacement-detection apparatus for *in situ* measurement of stress of a film. A displacement sensing probe, detecting a deflection of the substrate was composed of 38 multimode optical fibers of 50-μm core diameter and it was located

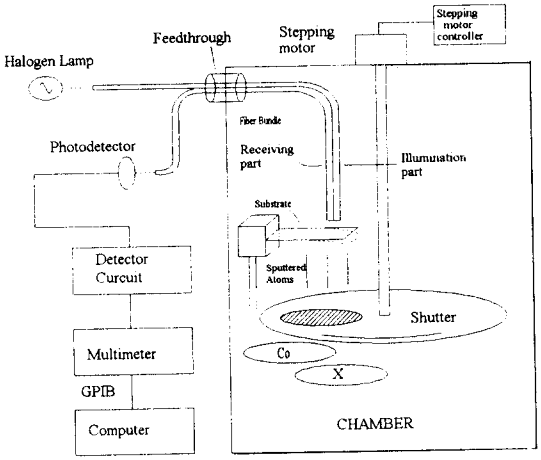

Fig. 1. A schematic diagram of an optical displacement-detection apparatus for *in situ* measurement of stress of a film prepared in a sputtering system (X=Pd or Pt).

behind the free end of the substrate. 38 optical fibers glued using a liquid epoxy were closely packed inside a 1-mm-dia. capillary tube. They were divided into 19 'sending fibers' where the light was transmitted to the substrate and 19 'receiving fibers' where the reflected light was guided to a photodetector. The sending and receiving fibers were randomly distributed to improve the sensitivity of the probe. The surface of the probe was polished with SeO_2 to achieve a maximum transmittance of the light from Halogen lamp (220 V, 650 W).

The initial gap distance between the probe and the substrate was adjusted using a Z-axis translator equipped with a differential micrometer. When the probe was in contact with a non-deflecting substrate, no light was transmitted to the receiving fibers. As the gap distance increased, the light transmitted to the receiving fibers increased linearly with the distance until a maximum intensity was reached when the entire surface of the receiving fibers was illuminated by the reflected light. This linear range was utilized for *in situ* measurement of the gap distance: A change in the gap distance between the probe and the substrate, caused by the stress of a film, was detected by measuring a corresponding change in the reflectivity from the substrate.

The probe was guided outside the vacuum chamber through a 3.5" ϕ del-seal fringe. Any ac noise received by a photodetector was filtered by an 8-pole chebyshev-type low-pass filter and only dc signal was fed to a computer using a GPIB interface. The displacement of the substrate with deposition of a film could be seen in a real time on the computer monitor. Fig. 2 shows a calibration curve of our detector for a 4-cm-long, 1.1-cm-wide, and 130-μm-thick glass substrate. The back side of the substrate was coated by 1000-Å-thick Al to enhance the sensitivity of the detector. As seen in the figure, a linear range exists in the gap distance from 360 μm to 490 μm with a sensitivity of 5.9 μ V/Å.

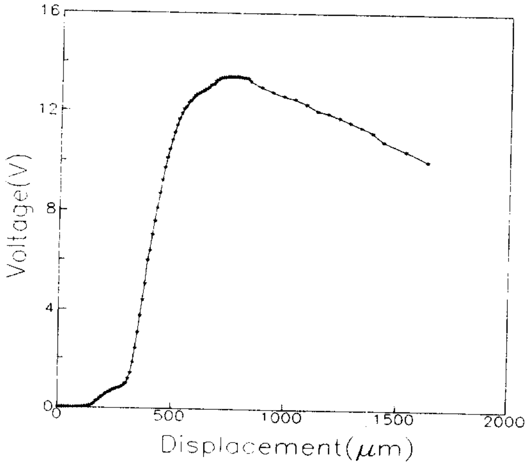

Fig. 2. A calibration curve of the displacement detector for a substrate coated by 1000-Å -thick Al.

B. Sample Preparation

The optical displacement detector was applied to the stress measurements of Co/Pd and Co/Pt multilayer films prepared by dc magnetron sputtering from 2-in.-diameter Co, Pd, and Pt targets on the glass substrates having the same dimensions as the one used for the calibration. The back side of the substrate was coated with 1000-Å-thick Al. The distance between the substrate and the sputtering source was 14.9 cm. An edge of the substrate was fixed by a cantilever holder. At the beginning of the deposition, the free end of the substrate was made to be deflected between 360 μm and 490 μm from the displacement probe, since our probe linearly responds with the displacement in this range. The base pressure below 4×10^{-6} Torr and the Ar sputtering pressure of 10 mTorr were used. Dc power of 50 W was applied to each sputtering target, which yielded the deposition rates of 0.53 Å/s for Co, 0.81 Å/s for Pd, and 0.94 Å/s for Pt, respectively. Pd and Pt sublayer thickness was fixed to 9 Å and Co sublayer thickness was varied for a series of Co/X multilayer films. The multilayer structure was achieved by alternately exposing the substrate to two 2sputtering sources via a reciprocating shutter.

For a cantilever beam of length L, stress of a film is determined by the gap distance of Δd using a well-known Stoney's formula [5] as follows:

$$\sigma = \frac{E_s \times t_s^2}{3(1-\nu_s) \times L^2} \frac{\Delta d}{\Delta h} \qquad (1)$$

where E_S, ν_S, and t_S are Young's modulus, Poisson's ratio, and the thickness of a substrate, respectively and Δh is the change of the film thickness.

III. RESULTS AND DISCUSSION

All samples in this study developed low-angle x-ray diffraction peaks irrespective of the sublayer thickness, which suggested the existence of the multilayer structure in those samples. High-angle x-ray diffraction studies revealed that the samples were polycrystalline grown along the [111] cubic orientation.

Fig. 3 shows a typical plot of the gap distance vs. the deposition time for Co/Pd multilayer composed of 8-Å -thick Co and 9-Å-thick Pd sublayers. Here, the positive and negative slopes indicate the tensile and compressive stresses, respectively. It can be seen in the figure that at the initial stage of the film growth large tensile stresses are existed in both Co and Pd sublayers. However, with progress of the deposition the stress in the Co sublayer become less tensile and the stress in the Pd sublayer is changed to be compressive. Fig. 4 shows the variation of the gap distance with the deposition time for Co/Pt multilayer composed of 4-Å -thick Co and 9-Å -thick Pt sublayers. Here, Co sublayer is subjected to tensile stress as Co/Pd multilayer, but stress in the Pt sublayer is compressive from the initial stage of the film growth in contrast to Co/Pd multilayer. To clarify this difference, we have performed *in situ* stress measurements of pure Pd and Pt thin films under the same deposition conditions. It was found that the stress in Pd was tensile at the initial stage, whereas Pt was compressive from the beginning of the film growth. This result means that at the beginning of Co/X multilayer formation the substrate yields a substantial effect on the stress of the film.

Stress in a multilayer is believed to be mainly caused by two origins: an adhesion of the film to the substrate and the lattice mismatch between two dissimilar adjacent sublayers. It is reasonable to conjecture that the effect of the substrate on stress of a film is monotonically diminished. Indeed, this effect was observed to be negligible when the film was thicker than about 100 Å in our samples. However, Co/Pt multilayers undergoes a less variation of this effect than Co/Pd multilayers, which is possibly due to a larger biaxial modulus of Pt than Pd.

After the effect of the substrate on the stress of the film was diminished, constant tensile and compressive stresses for the Co sublayer and the X sublayer were observed, respectively. We believe that at this stage, a lattice mismatch between the (111) matching planes of Co and X became the main origin of the stress in the sublayer. In Fig. 5, we show the measured stress in the Co sublayer for a series of the samples with different Co-sublayer thickness, but having a constant X-sublayer thickness of 9 Å. As seen in the figure, the stress in the Co sublayer is ranged from 1.8×10^{10} to 4.8×10^{10} dyne/cm^2 in our samples, which are larger than the stresses of e-beam evaporated samples[6].

Interestingly enough, we have observed that stress of the Co sublayer was suddenly dropped with increasing the Co-sublayer thickness. A distinct drop in stress can be seen when the Co-sublayer thickness is larger than about 5 Å in Co/Pd multilayer films and about 3 Å in Co/Pt multilayer films. We believe that the result is ascribed to a structural change from a coherent-to-incoherent interfacial matching [7] between Co and X. It should be noted that Co/Pt multilayer system has a smaller critical thickness and a larger Co-sublayer stress in the coherent range in

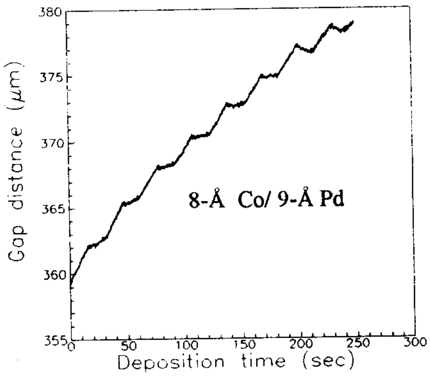

Fig. 3. A plot of the gap distance vs. the deposition time for 8-Å -thick Co and 9-Å -thick Pd.

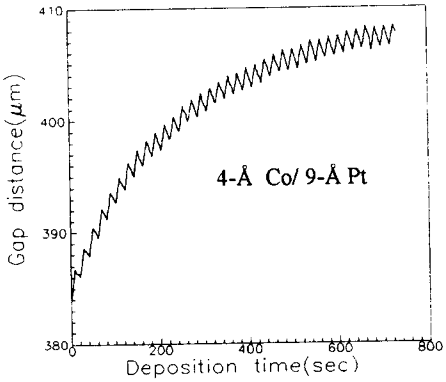

Fig. 4. A plot of the gap distance vs. the deposition time for 4-Å -thick Co and 9-Å -thick Pt.

comparison with Co/Pd multilayer system. The results are believed to be caused by a larger misfit of Co/Pt multilayers than Co/Pd multilayers.

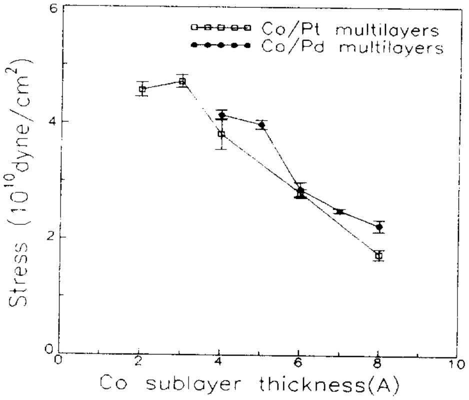

Fig. 5. The measured stress in the Co sublayer for a series of the samples with different Co sublayer thickness.

IV. CONCLUSIONS

We have constructed an optical non-contact displacement detector for *in situ* measurements of stress. Several Co/X(X=Pd or Pt) multilayer thin films prepared by dc magnetron sputtering have been examined to investigate the variation of stress during the deposition. The sensitivity of our displacement detector was 5.9 μV/Å and thus, it was good enough to detect stress caused by deposition of a monatomic-layer-thick Co or X. When the film was thicker than about 100 Å, constant tensile and compressive stresses were observed for the Co and X sublayers, respectively. The results are believed to be caused by a lattice mismatch between the matching planes of Co and X. An abrupt reduction of stress was observed when the Co sublayer thickness was larger than 5 Å in Co/Pd multilayers and 3 Å in Co/Pt multilayers, which is presumably related to a coherent-to-incoherent transition.

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