

Effective Interdiffusion of Co/Pd multilayers

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An artificially modulated magnetic Co/Pd multilayer is one of the promising candidates for high density magneto-optic (MO) recording media in the wavelength of a blue laser beam, due to large Kerr rotation angle. However, since the Co/Pd multilayer is a non-equilibrium state in terms of free energy and MO recording is a kind of thermal recording which is conducted around Curie temperature (T_C) of the recording media, the assessment of the thermal stability in the Co/Pd multilayer is crucially important both for basic research and applications. As the parameter of the thermal stability in this research, effective interdiffusion coefficients (D_{eff}) perpendicular to the interface of the Co/Pd multilayers are measured in terms of Ar sputtering pressure and heat treatment temperature. From the results of the research, we find out that the magnetic exchange energy between Co and Pd sublayers strongly affects D_{eff} of the Co/Pd multilayers. This discovery will provide the understanding of the magnetic exchange energy in the effective interdiffusion process of a magnetic multilayer structure and suggest the operating temperature range for MO recording in the Co/Pd multilayer for the basic research and applications, respectively.

I. Introduction

It is well recognized that an artificially modulated magnetic Co/Pd multilayer is one of the promising materials for high density magneto-optic (MO) recording in the short wavelength of a laser beam (~ 400 nm)[1-4], due to large Kerr rotation angle. Since multilayer structure, as well as amorphous structure, is a non-equilibrium state in terms of free energy[5-6] and MO recording is a kind of thermal recording which is conducted around Curie temperature (T_C)[7] of the recording materials, when the multilayer is used for the MO recording media, it is deduced that changes in the multilayer structure are produced by stress release and interdiffusion during repeated radiation of a laser beam in the MO recording process. Hence, these inevitably affect magnetic and magneto-optic properties[8-9] as do structure relaxation and crystallization in the amorphous structure. Since the outstanding magnetic properties of the Co/Pd multilayer for the MO recording are induced by the artificially modulated multilayer structure[1-4], the changes of the multilayer structure during the MO recording process

affect structural and magnetic properties of the Co/Pd multilayer. However, despite the superiority of the magnetic and magneto-optic properties of the Co/Pd multilayer for the short wavelength of the laser beam, research into the thermal stability, i. e. interdiffusion process both for basic research and application has hardly been conducted on it.

In this research, the interdiffusion process of the Co/Pd multilayer will be discussed in terms of Ar sputtering pressure and heat treatment temperature which dramatically affect the structural and magnetic properties of the multilayer, for example coercive force[10], and is the characteristic parameter of MO recording, for example Curie temperature, respectively. As the parameter of the thermal stability in the multilayer structure, interdiffusion coefficients in the Co/Pd multilayers deposited at low and high pressures are measured below and above T_C . From the practical point of view, only the Co/Pd multilayers with a modulation wavelength of less than a few Å in Co sublayer are appreciable for MO recording materials [1-4], so that effective interdiffusion coefficient (D_{eff}) in the small modulation wavelength limit is considered in this

experiment. In studying the D_{eff} in the Co/Pd multilayers, extremely low of the order of $10^{-27} \text{ m}^2/\text{sec}$ can be measured using a X-ray diffractometer[11].

II. Experimental Procedure

The Co/Pd multilayers were deposited on R-plane sapphire substrates ($2\theta = 25.60^\circ$ and 52.50°) in a two-target UHV (Ultra High Vacuum) dc magnetron sputtering system which was gettered with liquid nitrogen. The configuration of the sputtering system is described elsewhere[12-13]. The substrates were sat on a rotating holder and passed alternately beneath the two magnetrons on which the targets of Co and Pd were attached. A fixed shield was positioned between the level of the magnetrons and that of the substrate holder, with slots cut beneath the magnetrons. The target-substrate distances were 30 mm and 40 mm for Co and Pd, respectively. The vacuum system was pumped down to 10^{-10} Torr by a liquid nitrogen trapped diffusion pump. The sputtering gas was Ar (99.999%), processed with an Ar purifier. The sputtering pressures were at 0.7 Pa and 2.0 Pa in low and high pressure runs, respectively. The Co sublayer thickness was designed to obtain both perpendicular magnetic anisotropy (less than 8 \AA)[14] and maximum magnetostriction (30 at. % of Co)[3].

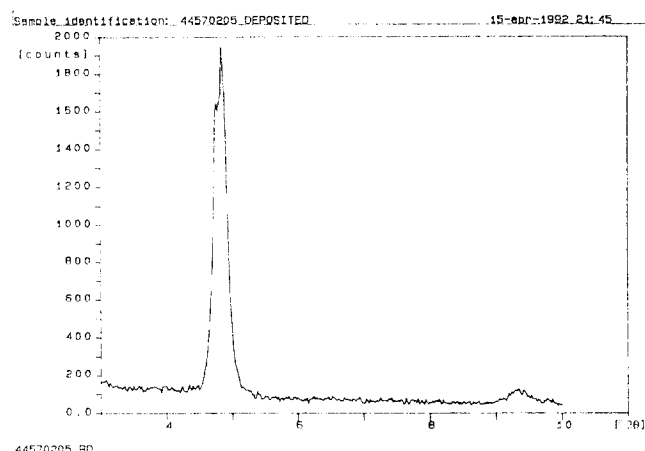
Compositions of the Co/Pd multilayers were determined using an EDS (Energy Disperse Spectrometer). T_c of the multilayers was determined by a DSC (Differential Scanning Calorimeter) with a heating rate of $40^\circ\text{C}/\text{min}$ and was $368 \pm 5^\circ\text{C}$. The X-ray intensities of the satellite peaks in the as-deposited and heat-treated Co/Pd multilayers were measured by a XRD (X-ray Diffractometer) with $\text{Cu K}\alpha$ ($\lambda = 1.5406 \text{ \AA}$) radiation in small ($2\theta=3^\circ-10^\circ$) angle regions with θ - 2θ scanning. In order to measure the X-ray intensities precisely, scans of rocking curve and calibration of 2θ -off method were adapted.

The as-deposited multilayers with a getter material (Zr-V-Fe) were inserted in a quartz ampoule for heat treatment. The ampoules were sealed after being pumped down to 10^{-6} Torr and back filled with Ar to 200 Torr to ensure good thermal contact between the multilayers and the surrounding during the heat treatment. Experiment to investigate the interdiffusion process was conducted via two methods to verify the precision of the experimental result. One was by a step method, i.e. the heat treatment of the Co/Pd multilayers was repeatedly carried out at fixed temperature and for a fixed time, and they were removed from the furnace to measure the changes in the X-ray intensities of the first satellite peaks in the small angle region. The other method used was a sequence method, which means that after taking a long enough pre-heat treatment for stress release of the Co/Pd multilayers, only one heat treatment for a fixed time was conducted on them

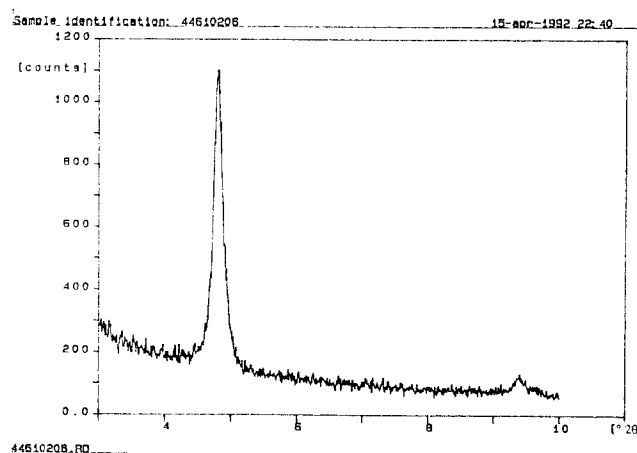
for the interdiffusion measurement. The former were heat-treated at 250°C , 350°C , 390°C and 400°C , and the latter at 300°C and 380°C .

III. Result and Discussions

The Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa by the magnetron sputtering apparatus formed well defined artificially modulated multilayer structures which displayed the first and the second satellite peaks in the small angle region of X-ray diffraction as shown in Fig. 1 (a) and (b), respectively. The compositions of the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa were 29.9 ± 0.2 at. % of Co and 29.2 ± 0.4 at. % of Co, respectively. The estimated nominal thicknesses of Co and Pd sublayers with each of bulk densities were 4.4 \AA and 14.1 \AA , respectively. The multilayers had 200 periods and total approximate effective layer thicknesses were about 3700 \AA for both the multilayers. The difference in the compositions between



(a)



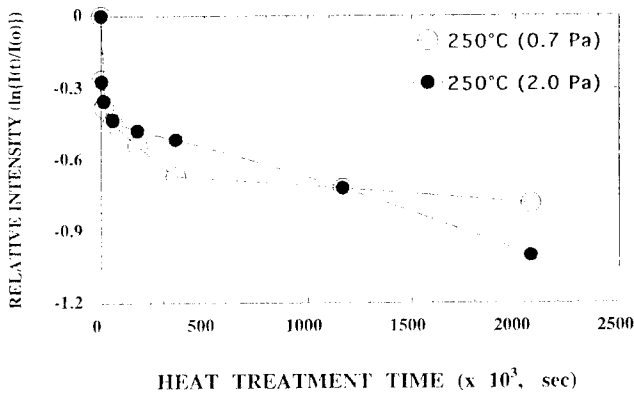
(b)

Fig. 1 (a) and (b): X-ray diffraction satellite peaks of the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa, respectively in small angle region

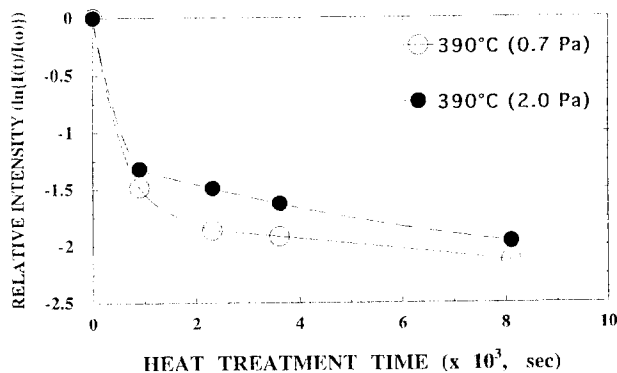
the Co/Pd multilayers showed less than 3% deviation from each other. Furthermore, they did not represent any intermetallic Co-Pd compound, which could have an effect on the interdiffusion process, on the phase diagram of Co-Pd alloy[15]. According to the calculation[16] of the modulation amplitudes of the Co/Pd multilayers, the modulation waves of the multilayers were sinusoidal with about 60% of modulation amplitude. Conclusively, the Co/Pd multilayers were suitable for investigating the interdiffusion process in terms of the Ar sputtering pressure and the heat treatment temperature.

1. Effective interdiffusion coefficients and activation energy of Co/Pd multilayers

Fig. 2 (a) and (b) show the logarithm of relative X-ray intensities ($\ln \frac{I_t}{I_0}$) versus heat treatment time for the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa, and heat treated below (250°C) and above (390°C) T_c , respectively. The continuum and discrete models, which are suitable for multilayers involving incoherent and coherent strain energy⁽¹⁷⁾, respectively are used to estimate effective



(a)



(b)

Fig. 2 (a) and (b): Changes of the relative X-ray intensities of the first satellite peaks in the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa, and heat-treated at 250°C and 390°C, respectively with respect to heat treatment time

interdiffusion coefficients (D_{eff}). When the relative X-ray intensities are linear on Fig. 2(a) and (b), the slope of the $\ln(I_t/I_0)$ is as follows for the continuum model,

$$\frac{d[\ln \frac{I_t}{I_0}]}{dt} = -D_{eff} \frac{8\pi^2}{\lambda x^2} \tag{1}$$

I_0 : X-ray intensity of satellite peak before heat treatment

I_t : X-ray intensity of satellite peak after heat treatment for (t) seconds

D_{eff} : Effective interdiffusion coefficient (m^2/sec)

λ_x : Wavelength of the X-ray (m)

t : Interdiffusion time (sec)

In the discrete approach, this becomes

$$\frac{d[\ln \frac{I_t}{I_0}]}{dt} = - \frac{4D_{eff}}{d^2} [1 - \cos(2\pi md/\lambda x)] \tag{2}$$

d: Spacing between the atomic planes perpendicular to the diffusion direction.

Since the compositional gradient in the continuum model and both the compositional gradient and strain effects in the discrete model are included in both of the above equations, the D_{eff} is determined from the slope of the best straight line fit through the linear portion of the ($\ln \frac{I_t}{I_0}$) versus heat-treated time (t) plot of the first satellite peak. The values of D_{eff} for both of the models are listed on Table 1.

Table 1: Effective interdiffusion coefficients of the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa by continuum and discrete models

Co/Pd multilayer	Continuum model (m^2/sec)	Discrete model (m^2/sec)
250°C (Lower pressure)	5×10^{-27}	5.3×10^{-27}
(Higher pressure)	15×10^{-27}	15.4×10^{-27}
300°C (Lower pressure)	130×10^{-27}	125×10^{-27}
(Higher pressure)	210×10^{-27}	210×10^{-27}
350°C (Lower pressure)	549×10^{-27}	570×10^{-27}
(Higher pressure)	797×10^{-27}	830×10^{-27}
380°C (Lower pressure)	3350×10^{-27}	5810×10^{-27}
(Higher pressure)	5675×10^{-27}	8250×10^{-27}
390°C (Lower pressure)	6465×10^{-27}	6430×10^{-27}
(Higher pressure)	8415×10^{-27}	12930×10^{-27}
400°C (Lower pressure)	13200×10^{-27}	13800×10^{-27}
(Higher pressure)	16600×10^{-27}	17300×10^{-27}

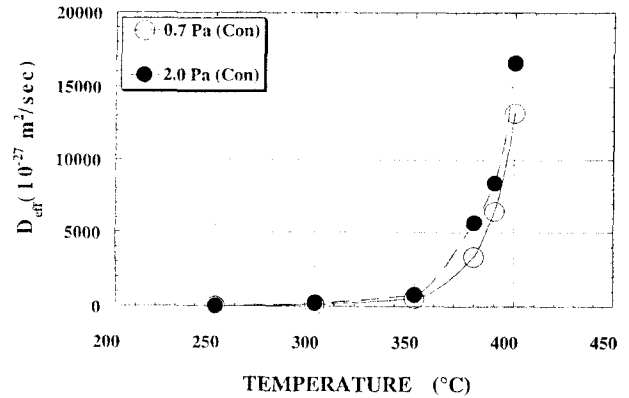
In theory, because of CI (Commensurate-Incommensurate) transition in the multilayer structure, no difference in D_{eff} should show between the two models below the critical value of $\frac{d}{\lambda} = \frac{1}{6}$ [17-18] from which the coherent strain energy is released to the incoherent one at the interface of the multilayer (d and λ represent lattice parameter and modulation wavelength of the multilayer, respectively). In this experiment, since the average value of the Co/Pd multilayer for $\frac{d}{\lambda}$ is 0.12 (2.15/18.52) which is less than the condition for the divergence in D_{eff} by the two models[17], no difference in the D_{eff} between the two models is shown under the assumption that any coherent strain energy is not involved at the interface due to the CI transition. Therefore, the difference in D_{eff} between the two models is experimentally small as listed on Table 1 and the Co/Pd multilayers involves only incoherent strain energy at the interface[11].

On the figures, the relative intensities decrease in two steps; fast and non linear in the initial stage, and slow and linear in the later stage during the heat treatment. Previous research pointed out that the non linearity in the relative intensity was mainly caused by micro structural change as the texture increases[19-20] or by densification of artificially modulated structure[21] during the stress release process, and the linear slope region was originated by interdiffusion process[19-21] in the multilayer structure. Therefore, the stress release and the interdiffusion in the Co/Pd multilayers sequently occur in non linear and linear regions, respectively.

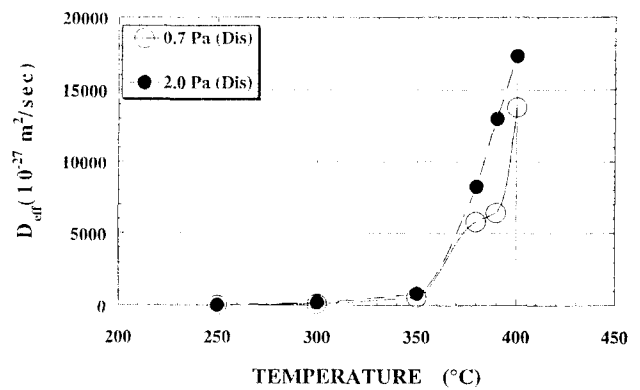
As shown in Fig. 2 (a) and (b), the Co/Pd multilayers deposited at 0.7 Pa shows the longer heat treatment time than those deposited at 2.0 Pa for the transition from the stress release process to the interdiffusion process. Because the Co/Pd multilayers deposited at 0.7 Pa are more compressive and dense by high energetic bombardment of Ar atoms during sputtering deposition than those deposited at 2.0 Pa, the former needs longer heat treatment time for the stress release than the latter. After the stress release process, the gradients in the linear stage deposited at 2.0 Pa are steeper than those deposited at 0.7 Pa.

The $\ln \frac{I}{I_0}$ in the linear slopes heat treated above T_c (390°C) are quite rapidly reduced compared with those heat treated below T_c (250°C) with respect to the heat treatment time, even though the tendencies of the relative intensity curves below and above T_c are quite similar in shape each other. The satellite peaks for the Co/Pd multilayers below and above T_c disappear within 2500×10^3 and 10×10^3 seconds, respectively. This means that the thermal stability of the Co/Pd multilayers is strongly dependent on the heat treatment temperature, which is directly related to operating temperature of MO recording.

Fig. 3 (a) and (b) display the effective interdiffusion coefficients (D_{eff}) of the Co/Pd multilayers calculated by continuum and discrete models[11], respectively in terms



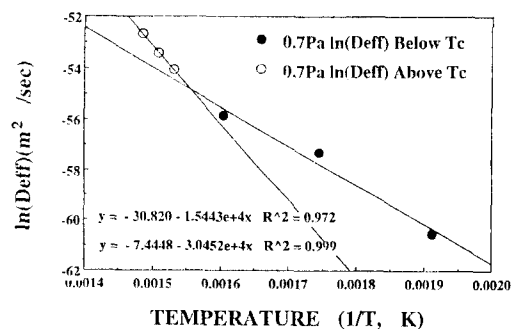
(a)



(b)

Fig. 3 (a) and (b): Effective interdiffusion coefficients (D_{eff}) of the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa calculated by continuum and discrete models, respectively

of heat treatment temperature. The differences in D_{eff} between the multilayers at 0.7 Pa and 2.0 Pa are small, less than a factor of 2. However, the differences in D_{eff} between the multilayers below and above T_c are dramatically large; at 250°C and 400°C the values are 2640 and 1110 times in the multilayers deposited at 0.7 Pa and 2.0 Pa, respectively. Even though the difference in the heat treatment temperature is small, the difference in D_{eff} is more than one order between 350°C and 390°C for below and above T_c , respectively. Because the difference in D_{eff} by the Ar



(a)

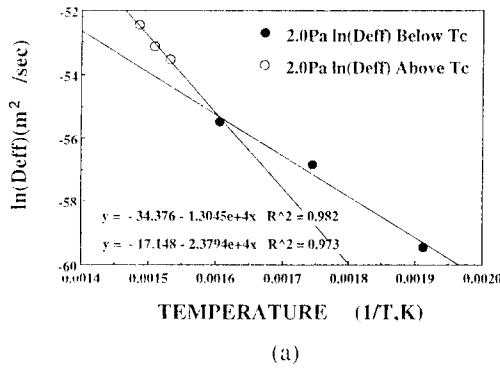


Fig. 4 (a) and (b): Logarithm of effective interdiffusion coefficients (Deff) of the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa, respectively as a function of reciprocal heat treatment temperature

sputtering pressure is negligible comparing with the difference in Deff by the heat treatment temperature, an important governing factor for the interdiffusion process, i.e. thermal stability, in the Co/Pd multilayers is not the former which represents stress state and crystalline quality of the multilayer structure, but the latter which represents operating temperature range of MO recording.

Fig. 4 (a) and (b) show the logarithm of the effective interdiffusion coefficients {ln (D_{eff})} of the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa, respectively, with respect to the reciprocal heat treatment temperature (1/T). On both of the figures, 3 points heat treated above T_c lie on linear slope, but the other 3 points heat treated below T_c lie on another linear line. Activation energy (Q) for the interdiffusion process of the Co/Pd multilayers is calculated by slope of Arrhenius equation as follows and listed on Table 2,

$$D_{eff} = D_0 \exp(-Q/RT) \quad (3)$$

D_{eff}: Effective interdiffusion coefficient (m²/sec)

D₀: Pre exponential factor

Q: Activation energy (Joule/atom)

R: Ideal gas constant(= 8.3170 Joule/mole)

Comparing with the previous researches in D_{eff} which found activation energies of 115 kJ/mole in Mo/Ge[19], 105 kJ/mole in Mo/Si[19], 103 kJ/mole in Pb/Mg[22], 141 kJ/mole in Cu/Au[23] and 106 kJ/mole in Cu/Ni[24] multilayers, the activation energy of the Co/Pd multilayers above T_c are comparable to these values. However, the activation energy below T_c are almost as twice as that above T_c. This means that anomalous interdiffusion process of the Co/Pd multilayers below T_c affects not only D_{eff} but also Q. Hindrance against the interdiffusion process below T_c presumably exists in the Co/Pd multilayers. The hindrance energy can be calculated by the

difference in Q between below and above T_c as follows.

a) Deposition at 0.7 Pa

$$Q_{Below} - Q_{Above} = (253 - 128) \text{ kJoule/mole} = 208 \times 10^{-21} \text{ Joule/atom}$$

b) Deposition at 2.0 Pa

$$Q_{Below} - Q_{Above} = (200 - 108) \text{ kJoule/mole} = 153 \times 10^{-21} \text{ Joule/atom.}$$

In section 3.2, the above experimental difference in Q will be theoretically compared to magnetic exchange energy of Co/Pd multilayers

2. Modeling of anomalous effective interdiffusion process

In bulk materials, deviations of diffusion coefficients from Arrhenius behavior in the magnetic phase transition between ferromagnetic and paramagnetic were observed in the self-diffusion of a-Fe[25-27] and the interdiffusion of Co in a-Fe[28]. The diffusion coefficients in ferromagnetic region were lower than those in the paramagnetic one. Moreover, the difference in the diffusion coefficients[25-28] between the ferromagnetic and the paramagnetic in the bulk materials were smaller by one order than those in the Co/Pd multilayers of this research. An anomaly in the elastic strain energy was also found in a-Fe around its magnetic transition, but not in the phase transition[29]. Since magnetic spins at lattice points display large magnetostatic energy in saturation magnetization state and try to expand their distance to reduce the magnetoelastic energy, as far as elastic strain energy is allowed in the ferromagnetic phase[30-31], it can be deduced that the lack of the magnetostatic energy in the paramagnetic phase causes the change in the elastic strain energy above T_c.

The basic concept for the modeling of the anomalous interdiffusion between below and above T_c in this research is the contribution of the magnetic bonding force (magnetic exchange energy) to the chemical bonding force (elastic strain energy) at the interface in the Co/Pd multilayers. In bulk materials, where all three dimensions are significant, the magnetic bonding force is generally negligible, compared with chemical bonding forces. However, in the multilayer structure, in which only two dimensions need to be considered, the chemical bonding force is weakened due to the loss of symmetry in the crystalline structure, but the magnetic bonding force is significantly enhanced due to the increment of the interfaces per unit volume. When the magnetic multilayer is heat-treated below T_c, this enhanced magnetic exchange energy at the interfaces can predominantly contribute to the total bonding force in the multilayer, and a large Q and a low D_{eff} are obtained. Even though the effect of the magnetic exchange energy (E_{ex}) on the diffusion process occurs in both of the multilayer structure and bulk

materials, the effect in the former is more predominant than in the latter, due to the increased interfaces per unit volume. Therefore, the differences in D_{eff} of the Co/Pd multilayers were larger than those in the bulk materials by one order. In the absence of E_{ex} above T_C , only the weakened chemical bonding force exists at the interface and D_{eff} is large in the multilayer.

In the case of the Co/Pd multilayers, the different interdiffusion processes for those heat treated below and above T_C are related to the particular magnetism of Co/Pd multilayers. It is well recognized that the Co/Pd multilayer has a larger saturation magnetization[1-4] than pure cobalt (1422 emu/cm^3)^[2], due to induced magnetization in the polarized Pd sublayer. Considering this magnetic phenomenon, the anomalous interdiffusion process can be established as follows; when the interdiffusion process is carried out in the Co/Pd multilayer below T_C , the enhanced ferro-magnetic exchange energy exists at the interface between the Co and Pd sublayers. This energy can prevent from the interdiffusion at the interface by increasing the total bonding energy, so D_{eff} and Q are reduced and increased, respectively. On the other hand, in the case of the interdiffusion above T_C , there is no E_{ex} at the interface as a barrier against the interdiffusion process. This can promote the diffusion process at the interface of the Co/Pd multilayers, so D_{eff} and Q are increased and decreased, respectively. E_{ex} between the Co sublayer and polarized Pd sublayer at the interface is described in detail as follows; E_{ex} between the two sublayers can be calculated under the assumptions. One is that magnetic exchange forces decrease rapidly with distance, so that E_{ex} are effective only between the 1st nearest neighbor pairs. The other is that the polarization of Pd occurs in all Pd sublayers, not in those near to the interfaces of the multilayer. Then, E_{ex} is described as below[32],

$$E_{\text{ex}} = Z(-2 J_{\text{ex}} J_{\text{Co}} J_{\text{Pd}} \cos\theta) \quad (4)$$

Z : Co-ordination number in Co/Pd multilayer (=12)

J_{ex} : Exchange integral (Joule/mole)

$J_{\text{Co}}, J_{\text{Pd}}$: Total angular momentum of Co and Pd, respectively

θ : Angle between two spin moments

$$J_{\text{ex}} = \frac{3kT_C}{2ZJ(J+1)} \quad (5)$$

k : Boltzman constant (1.38×10^{-23} Joule/K)

T_C : Curie temperature (641 K in the Co/Pd multilayer)

E_{ex} of the Co/Pd multilayers are as follows.

1) $J = J_{\text{Co}} = J_{\text{Pd}} = 1/2$ (all pure spin angular momentum)

$$J_{\text{ex}} = 0.167kT_C = 1.475 \times 10^{-21} \text{ Joule/atom}$$

$$E_{\text{ex}} = -8.851 \times 10^{-21} \text{ Joule/atom}$$

2) $J_{\text{Co}} = 1/2, J_{\text{Pd}} = 1$ (additional orbital momentum due to induced magnetization)

$$\text{a) } J_{\text{ex}} = 0.125kT_C = 1.106 \times 10^{-21} \text{ Joule/atom}$$

$$E_{\text{ex}} = -13.274 \times 10^{-21} \text{ Joule/atom}$$

$$\text{b) } J_{\text{ex}} = 0.083kT_C = 0.737 \times 10^{-21} \text{ Joule/atom}$$

$$E_{\text{ex}} = -8.846 \times 10^{-21} \text{ Joule/atom}$$

The negative sign in E_{ex} means that when the angle between the magnetic moments is reduced, the exchange energy is minimized. Comparing the difference in Q between below and above T_C as calculated in Fig. 4 and listed on Table 2 with E_{ex} , the measured former is larger than the calculated latter by one order. The difference between the observed energy and the calculated energy can be accounted for increased E_{ex} due to the effects of beyond the 1st nearest neighbor pairs and of the expanded polarization of Pd atoms near to the interfaces of the multilayer, which were excluded in the assumptions.

Table 2: Activation energy of the Co/Pd multilayers deposited at 0.7 Pa and 2.0 Pa, and heat-treated below and above Curie temperature

(kJ/mole = 0.01 eV/atom)			
Co/Pd multilayers	Activation energy (below T_C)	Activation energy (above T_C)	Difference in Activation energy
Deposition at 0.7 Pa	253 kJ/mole	128 kJ/mole	125 kJ/mole (208×10^{-21} J/atom)
Deposition at 2.0 Pa	200 kJ/mole	108 kJ/mole	92 kJ/mole (153×10^{-21} J/atom)

IV. Conclusion

Effective interdiffusion coefficients were measured in terms of Ar sputtering pressure and heat treatment temperature with respect to heat treatment time as the assessment of the thermal stability for the Co/Pd multilayers. Dependence of the Ar sputtering pressure on the thermal stability was not seriously at each heat treatment temperature. The anomalous interdiffusion process was observed in the multilayers heat treated below T_C . The anomaly was attributed to the magnetic exchange energy which played a hindering role for the interdiffusion process below T_C . The comparison between the magnetic exchange energy in the Co/Pd multilayer and the difference in the activation energy between below and above T_C presents a good coincidence under the assumptions. When the Co/Pd multilayer is used for MO recording material, temperature range near below T_C , for example 350°C is recommendable for thermal MO recording. This discovery of the anomalous interdiffusion process in the Co/Pd multilayers will provide the clue to understand the effect of magnetic exchange energy on interdiffusion in artificially modulated magnetic multilayers and suggest the temperature range to operate

magneto-optic recording in Co/Pd multilayers for basic research and application, respectively.

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