INFLUENCE OF R.F. SPUTTER PARAMETERS ON THE MAGNETIC ORIENTATION OF Co-Cr LAYERS.

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Abstract - Co-Cr layers for the perpendicular recording mode were deposited by means of RF-sputtering. The most important sputter parameters, i.e. the RF sputter high voltage V_{RF}, the argon pressure P_{ar} and the substrate holder temperature T_{sh}, gave an optimum value for perpendicular orientation of the magnetization. The crystal structure is always hcp within the ranges of varied parameters and no other magnetic phases were observed. If the sputter parameters do not have optimum values an additional hcp compound with in-plane orientation of the c-axis is observed. This orientation causes an increase of the in-plane remanence $S_{//} = (M_r/M_s)$. Measurements of the substrate temperature T_s as an function of the various sputter parameters lead to the conclusion that an exclusive perpendicular c-axis orientation is only obtained at $T_s \sim 150^{\circ}C$. At other T_s established either directly by changing T_{sh} or indirectly by changing the sputter conditions an additional hcp compound with in-plane c-axis orientation appears. We concluded that T_s is the dominant parameter for sputtering CoCr layers.

INTRODUCTION

RF-sputtered Co-Cr layers show suitable properties for perpendicular recording [1,2]. The layers only exhibit the hexagonal close-packed (hcp) structure with perpendicular c-axis orientation, thereby establishing large perpendicular crystal anisotropy. The optimum perpendicular orientation of the magnetization strongly depends on the sputter parameters used: V_{RF}, P_{ar} and T_{sh}. The aims of this investigation, were first to determine the optimal sputter parameters and then to clarify the causes of magnetic deterioration outside this set of optimal parameters. The quality of the perpendicular orientation of the magnetization is easily perceived by means of the hard-axis (in-plane) hysteresis loop. In the ideal case, i.e. the c-axis is perfectly perpendicularly oriented, the anisotropy axis has no dispersion and the hard-axis loop, exhibiting no in-plane remanence, should be observed. In fact Co-Cr films sputtered with optimal parameters have a very small $S_{//}$ of about 5%, as shown by the hard-axis loop in Fig.1a. However, $S_{//}$ is considerably larger at other values of the sputter parameters as shown in Fig.1b. We use $S_{//}$ as a measure of the perpendicular orientation of the magnetization and assume that a non-zero value of $S_{//}$ may have the following causes: a) Dispersion of the c-axis around the film normal. b) Orientations of the c-axis other than the film normal. c) Other magnetic phases with low magnetic anisotropy like for example fcc Co-Cr or magnetic oxides.

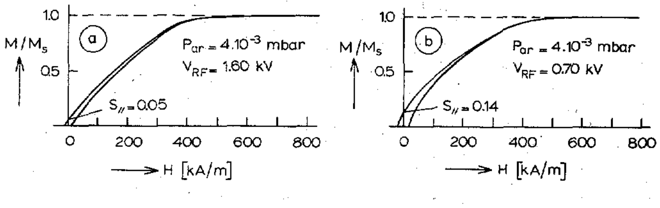

Fig. 1. The in-plane (VSM) hysteresis loops: a) under optimal conditions. b) under other sputter conditions, e.g. a different V_{RF}; $S_{//}$ is considerably increased

EXPERIMENTAL PROCEDURE

A commercial Leybold-Heraeus RF sputter unit (type Z400) was used for the deposition. The 19 at%Cr alloy target, had a diameter of 10 cm and a purity of 99,99%. The target-substrate distance was 5 cm. Monocrystalline (100)-oriented Si substrates (0.3 mm thick) were used for magnetic and X-ray measurements and freshly cleaved mica sheats covered with carbon for transmission electron microscopy (TEM). The substrate holder was water cooled, but for experiments with varying T_{sh} a heated one was used. Diffusion of the Co-Cr into the Si substrate takes place at temperatures higher than about $100^{\circ}C$. Therefore only mica-carbon substrates are used for sputtering on the heated substrate holder. The thickness of all layers is approx. 150 nm and has been so chosen because the c-axis dispersion is then no longer critically dependent on the film thickness [3] while TEM is still possible. After a background pressure of 2.10^{-7}mbar is reached by pumping 10 hours with a turbomolecular pump, the pressure is further reduced to below 10^{-7}mbar by means of a liquid N_2 Meissner trap and argon (purity 99.998 %) was admitted as sputter gas. Before deposition the target is sputter cleaned for 30 min. and the substrates cleaned by means of glow discharge. Both the film composition and thickness of the deposited films were determined by means of X-ray fluorescence [4]. The film composition appeared to be independent of the sputter parameters and close to the target composition of 19(±0,5) at%Cr. The in-plane hysteresis loop of the Co-Cr layers was measured with a vibrating sample magnetometer (VSM). The $S_{//}$ could be determined with a relative accuracy of about 5%. In order to analyse the crystallographic phases and texture, we used a 200kV Jeol microscope and a Philips X-ray diffraction apparatus, using Cu-K α radiation. Electron diffraction was primarily applied to investigate which alien crystal structures were present in the hcp Co-Cr layers, because its sensitivity to small amounts of mixed phases is higher than that of X-ray diffraction. Furthermore electron diffraction also reveals a possible in-plane orientation of the c-axis, in which case the (00.2) and {10.1} planes are parallel to the electron beam and cause diffraction. The diffraction patterns were optically read-out by means of a microdensitometer and recorded. We then only needed information on out-of-plane orientations of the c-axis which could readily be measured by means of the X-ray rocking curve method [3]. The (00.2) rocking curve enables registration of almost all c-axis orientations between the normal and the plane of the layer. Only an in-plane orientation of the c-axis cannot be detected, but this is obtained from the diffraction pattern. The dispersion of the c-axis is denoted by $\Delta\theta 50$ the half-width angle of the (00.2) rocking curve peak around the film normal. The T_{sh} was measured by means of a chromel-alumel thermocouple, glued in a piece of aluminum oxide. This and the substrates were laid on the substrate holder without further measures to improve the heat conductivity. The measured temperature was stationary only after about one hour. In principle the optimum value of one sputter parameter, V_{RF} for example, may depend on the values of other parameters such as P_{ar}. It is not known in advance how much the parameters are mutually dependent. This complicates the optimalization of the sputter process and forces us to vary each parameter at various values of the others. In order to reduce the number of runs needed for a systematic variation of all parameters, we first fixed V_{RF} at 1.60 kV when varying the P_{ar} and used the water-cooled substrate hol-

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der. At this V_{RF} a reasonable deposition rate R is obtained (R=0.2 nm/s) and furthermore other authors have found this voltage to be optimal [5]. We then varied the V_{RF} at the optimum P_{ar}. Finally the dependence of the magnetic orientation on T_{sh} was investigated using the optimized V_{RF} and R_{ar}.

EXPERIMENTAL RESULTS

Fig. 2 presents $S_{//}$ and $\Delta\Theta 50$ as a function of P_{ar}. The lower limit of P_{ar} is determined by the requirements for stable plasma operation, which is about 2.10^{-3}mbar for our equipment. The $S_{//}$ exhibits a faint minimum at $P_{ar}=4.10^{-3}$mbar and increases strongly at higher P_{ar}. Surprisingly the $\Delta\Theta 50$ shows the opposite behaviour, viz. an improving normal c-axis orientation at higher P_{ar}. Obviously $S_{//}$ originates from causes (b) and (c), mentioned above. The (00.2) rocking curve however exhibits no other orientations of the c-axis than the perpendicular one, but electron diffraction reveals the presence of the {10.1} and the (00.2) reflections in layers, sputtered at 2.10^{-3} and 1.5×10^{-2} mbar. These reflections are not observed in the layer, sputtered at 4.10^{-3}mbar (see Fig.3a.). No reflections of other magnetic phases are observed, like fcc Co-Cr as was concluded by the absence of the (200) fcc reflection. Furthermore, the diffraction rings of the layer sputtered at $P_{ar}=1.5\times10^{-2}$mbar, are more blurred, which indicates a defect-rich crystal structure. This is not accompanied by an increase of $\Delta\Theta 50$, which improves at higher P_{ar}. The increase of $S_{//}$ for argon pressures, which are different by 4.10^{-3} mbar, is apparently caused by the additional presence of an hcp compound with in-plane orientation of the c-axis. In the following runs the P_{ar} was therefore kept constant at 4.10^{-3}mbar. The V_{RF} was then varied between 0.70 and 2.0 kV at $P_{ar} = 4.10^{-3}$mbar, as depicted in Fig.4. The $S_{//}$ has a clear minimum at V_{RF}=1.60 kV but again there is no correlation between $S_{//}$ and the perpendicular c-axis dispersion. The $\Delta\Theta 50$ is almost constant in the entire voltage range, showing only a slight increase in the lower part, while no other orientations of the c-axis are revealed by the (00.2) rocking curve. Electron diffraction reveals the same behaviour of the crystal structure, as was found for P_{ar} variation. Co-Cr layers sputtered at an RF voltage, which is different from the optimal value of 1.60 kV, show the presence of a certain amount of a hcp phase with in-plane orientation of the c-axis, as indicated by the {10.1} and (00.2) diffraction rings. Also the diffraction rings of the high voltage layer are more blurred, like those of the layer sputtered at high P_{ar}. At an elevated T_{sh} the magnetic orientation is strongly deteriorated (see Fig.5). The increase of $S_{//}$ is in fact so steep, that the lowest possible and ambient T_{sh} of 20°C seems to be very critical. Unfortunately no substrate cooling, e.g. by means of liquid nitrogen, was possible in our equipment. Nevertheless it appears that the perpendicular magnetic orientation depends strongly on T_{sh}. At elevated temperature, $\Delta\Theta 50$ hardly changes (see Fig.5), but again the {10.1} and (00.2)diffraction rings become apparent and more blurred at increasing temperature (see Fig. 3b.). The very strong relation between the in-plane c-axis orientation and the temperature may imply, that the dependence of the in-plane c-axis orientation on the other parameters is indirectly caused by T_s variations. An increase of V_{RF} for example is accompanied by an increase of the heat flux to the film surface, not only by secondary electrons from the target, but also by the increased kinetic energy of the condensing atoms. In order to obtain an indication of the heating by the sputter process, T_s was measured with the same thermocouple used for measuring the heated substrate holder. This measured "bulk" temperature differs from the surface temperature where the actual layer grows [3]. Nevertheless it will correlate with the surface temperature and can therefore be used to examine the heating from the plasma. Fig.6. shows the measured T_s when P_{ar} is varied. Surprisingly T_s exhibits a minimum at $P_{ar}=4.10^{-3}$ mbar, as was also found for

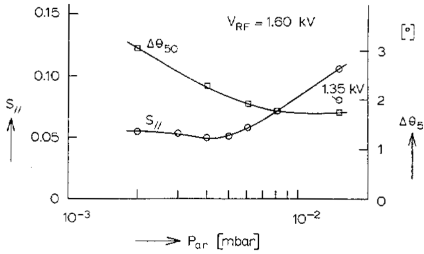

Fig. 2. The half width angle $\Delta\Theta 50$ and the in-plane remanence $S_{//}$ of 150 nm thick Co-Cr layers as a function of the argon pressure P_{ar}.

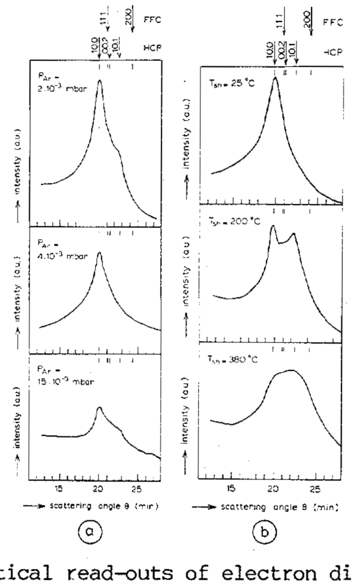

Fig. 3. Optical read-outs of electron diffraction patterns using a microdensitometer: a) at various argon pressures b) at increasing substrate holder temperature

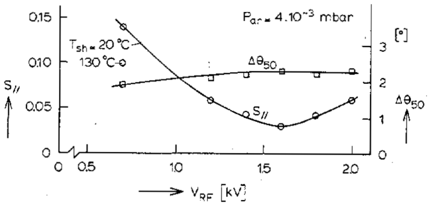

Fig. 4. The relation between the in-place remanence $S_{//}$, the half width angle $\Delta\Theta 50$ and the RF-sputter voltage V_{RF}.

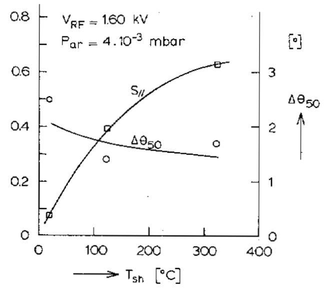

Fig. 5. The in-plane remanence $S_{//}$ and the half width angle $\Delta\Theta 50$ at elevated substrate holder temperature T_{sh}.

$S_{//}$ (see Fig.2). This minimum of T_s is probably caused by the competitive heating by the kinetic energy of the condensating atoms and the secondary electrons from the target. At lower P_{ar}, the heating by the secondary electrons is reduced, but the mean free path in the argon gas is simultaneously enlarged. As a result the sputtered atoms will arrive at the film surface with higher kinetic energy, less decelerated by collisions with argon atoms. As an indication we can calculate the mean free path F_{ar_o} of the argon gas at 4.10^{-3}mbar. At 1 bar and 20°C the mean free path F_{ar} is 10^{-7}m [6]. With $F_{ar}::1/P_{ar}$ we see that $F_{ar}=2.5$ cm at $P_{ar}=4.10^{-3}$mbar, half the target to substrate holder distance of 5 cm,and at $P_{ar}=2.10^{-3}$ mbar F_{ar} even equals this distance (corrections for the different collision diameters of the Co, Cr and Ar atoms, are disregarded). The minimum of T_s is not caused by an anomalous minimum in the sputter power, because R continuously increases in the entire argon range, as shown in Fig.6. Figure 7 shows T_s, as measured in the V_{RF} range from 0.7 to 2.0 kV. There is a continuous increase of T_s as there is of R, with the rate of increase of T_s largest in the lower voltage range. This corresponds qualitatively with the rate of decrease of $S_{//}$, (see Fig.4).

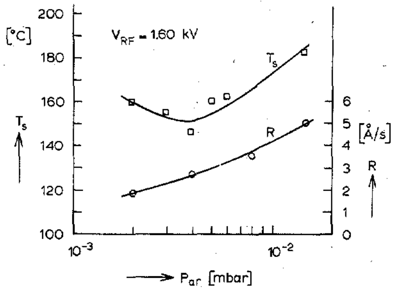

Fig. 6. The substrate temperaure T_s, established at various argon pressures by heating from the sputter process and the deposition rate R.

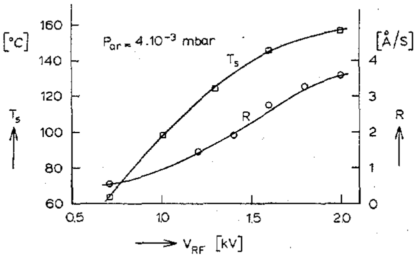

Fig. 7. The substrate temperature T_s and the deposition rate R as measured for different sputter voltages V_{RF}.

DISCUSSION AND CONCLUSIONS

The most important sputter parameters, i.e. V_{RF},P_{ar} and T_{sh}, were found to exhibit an optimum value for perpendicular orientation of the magnetization. Nevertheless, the crystal structure is always hcp within the ranges of the varied parameters and no other magnetic phases were observed. We only found that if sputtering is not carried out at the optimum values of the parameters, an additional hcp compound with in-plane orientation of the c-axis is observed. The {10.1} and (00.2) diffraction rings then become apparent in the electron diffraction pattern. This hcp compound with an in-plane orientation of the c-axis also causes some in-plane anisotropy and consequently an increase of $S_{//}$. Measurements of T_s as a function of the different sputter parameters lead to the conclusion, that an exclusive perpendicular c-axis orientation is only obtained at T_s~150°C. At other temperatures, established either directly by a change of T_{sh} or indirectly by changing a sputter parameter, an additional hcp compound with in-plane c-axis orientation is formed. The optimum value T_s~150°C is more or less in agreement with the temperature found by Ouchi and Iwasaki [7] for minimum dispersion of the perpendicular c-axis orientation. At a constant V_{RF} of 1.60 kV and varying P_{ar}, a minimum of $S_{//}$ was found at $P_{ar}=4.10^{-3}$ mbar, because the optimum T_s~150°C was reached at this argon pressure. This minimum can be considered as a coincidence because a different value of V_{RF}, for example 1.0kV, would not have given such a minimum. In order to verify our conclusions, we sputtered Co-Cr under modified conditions. First a Co-Cr layer was sputtered at V_{RF}= 0.70 kV and $P_{ar}=4.10^{-3}$mbar but at an elevated T_{sh}=130°C (see Fig.4). Actually $S_{//}$ shows a significant decrease. The same is observed if Co-Cr is sputtered at $P_{ar}=1.5\times10^{-2}$mbar on the water cooled substrate holder but at a lower V_{RF}=1.35 kV (see Fig.2). Both results make it plausible that T_{sh} is the dominant parameter, no matter whether it is determined by direct or indirect heating of the substrate. In literature [2,5,7] the deterioration of the perpendicular magnetic orientation is commonly marked by a strong increase of $\Delta\theta_{50}$. This was not observed in the actual Co-Cr layers, which always exhibited an excellent orientation of the perpendicular c-axis. Probably this discrepancy is caused by the different film thicknesses used. In literature, data are presented of about 1 micron thick Co-Cr layers, while ours were 0.15 micron thick. This paper emphasizes the perpendicular orientation of the magnetization but other magnetic parameters, like the coercivity, are no less important for the recording performance. Fortunately, the coercivity of the Co-Cr layers, sputtered with optimum parameters, has a reasonable magnitude of about 50 kA/m depending on the thickness [8]. Sputtering at a higher T_{sh} for example, yields coercivities in the order of 100 kA/m [2,7], which are too high for common recording head materials. Finally we must conclude, that the set of optimal parameters, as found for our sputter equipment, is not directly applicable for other systems. The heating of the substrate from the plasma plays an important role for the determination of the optimal value of these parameters. This heating strongly depends on the geometry of the sputter system used, e.g. the target to substrate distance and the target diameter are important dimensions in connection with the substrate heating by secondary electrons. An unsatisfactory conclusion is therefore that the optimal parameters have to be independently determined for each sputter system.

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