INVESTIGATION INTO PERPENDICULAR ANISOTROPY
COBALT-CHROMIUM FILMS WITH APPLICATION TO
WINCHESTER COMPUTER DISCS

by

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requirements for that degree.

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DECLARATION

I declare that this thesis is the result of my own investigations only, and has not been submitted either in full or in part for the award of any other degree. During the research program I was not a registered candidate or enrolled student for another award of the C.N.A.A or any other academic or professional institution.
During the research program I undertook a course of advanced studies. These included the extensive reading of literature relevant to the research project; the attending of several one day, and one week, long conferences related to my studies; and undergoing a course of tuition on the use of both transmission and scanning electron microscopy for use in microscopy and micrography and film analysis.
ABSTRACT

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RF-sputtered CoCr films have been characterized for a variety of deposition conditions, and the most effective parameters which bring about perpendicular anisotropy have been evaluated. In particular bias sputtering has been noted for its role in improving the crystallographic orientation and magnetic properties when applied to the substrate at certain values, hence the root mechanism of the observed improvement has been, as far as possible, investigated. These investigations have, in particular, focused on the extent to which impurity gases are likely to effect the magnetic properties of sputtered films, as well as quantifying the gas contents of the films using as yet unexploited (in this area) method of thermal desorption experiments.

In a further attempt to quantify tolerable level of impurity species in the sputtering environment, impurity gases of nitrogen, oxygen and hydrogen, which are commonly present even in high vacuum systems, were intentionally introduced in the sputtering chamber and their effects on both magnetic and crystallographic properties of CoCr were noted.

To measure the perpendicular and in-plane magnetic properties of CoCr, a combined polar-transverse Kerr magneto-optic system was constructed, through which direct magnetic measurement of CoCr/NiFe becomes feasible. This method was further exploited to compare volume and surface magnetic properties of CoCr, as measured using a V.S.M and this M-O system.

Finally, a CoCr/NiFe Winchester disc was fabricated on which a recording experiment was successfully performed.
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"To my parents"
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CHAPTER "1"

Introduction to perpendicular recording
1.1. Introductory Remarks

Since the first demonstration that a magnetic medium can be used to record information by Valdemer Poulsen in 1898, researchers have been striving to achieve higher and higher recording densities. This search has manifested itself in many varied and vivid forms - from the first steel wire recording to ubiquitous magnetic tape and finally to the present high-tech Winchester Discs.

In figure (1.1) a conventional recording system is shown, consisting of a recording medium with "in-plane" magnetization and a "ring head" for writing and reading the information.

In the write mode the head acts as a transducer that converts electrical signals into time dependent magnetization patterns which are then transferred onto the recording medium by means of fringing magnetic fields. Its requirements are, therefore, low remanence in order to avoid unwanted writing, followed by small coercivity for exclusion of hysteresis losses. Such low coercivity materials are said to be "soft".

For its part, the magnetic medium retains this field after it has been removed through its remanence and protects the data against stray fields if a material with the large coercivity is selected. However, if the
coercivity is too high the medium cannot be written upon.

When the data are being read out, i.e. in the read mode, the magnetic field lines (flux) permeate the core and produce an induced voltage.

In the digital world, the need for a data storage system that offers rapid access to data at any location in the file has meant moving to magnetic discs, and the desire for smaller systems has necessitated drastic increase in the capacity of the existing ones. For the latter, however, four limiting cases have to be considered:

a) The dimension of the head

It is obvious that reducing the dimensions of the head in general and that of the head gap in particular can lead to an increase in bit density. The minimum distance between the head and the medium which is set by the medium's and the head's surface roughness imposes a limit to this extreme scaling. Yet, presently, head gap dimension and head-to-medium distance of 0.15 micron and 0.1 micron are easily obtainable.

b) "Demagnetization" in the medium

In the conventional longitudinally magnetized medium, the most limiting constraint to high bit density is effected due to the phenomenon of demagnetization which can be categorized as;
i) Recording demagnetization

The finite gradient of the horizontal component of the writing head field tends to demagnetize asymmetrically the previously recorded magnetization transitions at high bit densities. The signal versus write current response of all longitudinal media exhibits a peak at high bit densities which becomes sharper and shifts to lower write currents as the bit density is increased (figure(1.2) [1].

ii) Self and adjacent bit demagnetization

As the bit density is increased, self demagnetization increases sharply because the aspect ratio of the bit becomes increasingly less and less favourable.

Due to negative interaction between adjacent bits in longitudinal recording, the field from each bit tends to demagnetize its neighbors, with the consequence of spreading the magnetization transition region and decreasing signal output and resolution.

In the early 70's, Iwasaki discovered that high density recording is inhibited by the nucleation of flux-closure inside the medium. He named this flux-closure phenomenon the "Circular Magnetization Mode", [1.5], which is indeed another aspect of recording demagnetization. An obvious disadvantage is that the flux available for
Figure (1.1). A conventional recording system, employing a "ring head", which exploits the longitudinal mode of magnetization.

Figure (1.2). A typical output Vs. record current for a longitudinal recording mode, showing a decrease as the recording frequency is increased (After Iwasaki [1]).
reproduction is reduced.

In order to suppress this detrimental magnetization mode, three steps can be taken:

1) Use a medium with strong in-plane anisotropy. This is in response to the increase in "magnetostatic energy" caused by the demagnetization and, in effect, acts to counterbalance the detrimental effects of the demagnetization.

2) Use a medium with high coercivity. However, in the limit, we are led to a medium on which we cannot write because of its extremely high coercivity.

3) Scale down the medium thickness to a minimum. This use of extremely thin films is equivalent to providing the medium with strong "shape anisotropy" so that the magnetization easy axis is in-plane.

However, this imposes severe constraints in the fabrication of the medium because of the impact on the media imperfections, defects, yields, durability, and cost. It will also mean a reduction in the signal resulting from the decrease in magnetization.
1.2 Perpendicular Recording

An alternative approach is to develop a medium that will support perpendicular magnetization.

Several research efforts towards perpendicular recording took place during the late 50's [2, 3]. None of those analyses was successful in demonstrating the superiority of perpendicular recording over conventional recording. In 1977 Professor Iwasaki and co-workers proposed a perpendicular recording system using a new recording medium and a magnetic head and demonstrated that it was capable of realizing high storage density. He had earlier described how the circular mode of magnetization could be transformed to a perpendicular mode, and the amplitude of the reproduced voltage recovered [4].

Figure (1.3) shows the magnetization transition between two adjacent bit sizes for both the longitudinal and the perpendicular modes of magnetization. Ideally the transition should be step-like for M1 and M2, with their respective demagnetizations, Hd1 and Hd2. Regions where the demagnetizing field exceeds the medium coercivity will be subject to demagnetization. For the longitudinal case the region immediately around the transition centre is demagnetized, leading to a broad transition, whereas, for the perpendicular case the region around the transition centre remains unchanged, resulting in a sharp transition.
Figure (1.3). A model normally used to illustrate the magnetization transitions in longitudinal and perpendicular recording.
1.3 The perpendicular Anisotropy Film

The question as to the kind of material that can be utilized which possesses suitable properties now arises. In deciding this, we need to consider that the medium of our choice should possess a high Ms with correspondingly high value of Hc to support Ms. It should also have a strong adhesion to the base material as well as having high producibility, especially in mass production. Finally, cost effectiveness of the material also plays a real part in this choice. Taking into account these properties, Iwasaki [5] suggested Co-Cr perpendicular anisotropy films.

Cobalt has a large magnetocrystalline anisotropy energy which it derives from its hexagonal close-packed (hcp) crystal structure, which exhibits a strong uniaxial anisotropy along the crystallographic c-axis. Hence it may be used to develop perpendicular anisotropy films. To develop the films in a relatively thin thickness, the anisotropy field, Hk, must satisfy the relation

\[ H_k > 4\pi M_s \text{, or } K_u > 2\pi M_s^2 \]

where Ku is the magnetocrystalline anisotropy constant, i.e., the films must have anisotropy field surpassing the maximum demagnetizing field, \( 4\pi M_s \). Therefore, It is effective to add another material to cobalt so as to reduce Ms, keeping the c-axis oriented perpendicular to film surface [6].
In developing the Co-M alloy, it was expected of the alloy to have a relatively stable hcp structure and that their uniaxial magnetocrystalline anisotropy does not decrease as rapidly as their Ms decreases by the addition of another metal M to cobalt [7].

Figure (1.4) shows the relationship between Ms and anisotropy field Hk of Co-M films. Hk increases and reaches a maximum and then decreases with increasing the amount of V, Cr, Mo and W added to Co. It is evident that Co-Cr film exhibits the largest Hk among the Co-M films, with a maximum value of 6.4 KOe. It was thus concluded that Co-Cr films possess the best magnetic properties among the Co-M films in realizing the perpendicular mode.

It was further discovered [8] that the addition of Cr reduces the size of the crystallites in Co-Cr film and some precipitation of the Cr takes place at the crystalite boundaries. This enhancement of the "particulate" character of the recording medium could reduce the material noise and improve its switching characteristics. Smaller grain size could also imply an improvement in surface roughness, and therefore, a better head-medium interface.

To improve head-medium interaction, it was later proposed [9] that a magnetically soft underlayer can be used as an efficient flux-guiding medium. They noted an improvement by an order of magnitude by backing the Co-Cr
Figure (1.4). The $M_s$ dependence of the perpendicular anisotropy field, $H_k$, of Co-M alloy films.
with Ni-Fe underlayer.

In the case of single pole head, figure(1.5a), which is a magnetic open structure, the induced magnetic charges at the tip of the pole head interact with magnetic charges induced on the Co-Cr surface. As a result, the demagnetizing field acting on the head tip is reduced and, therefore, the head produces a stronger magnetic field. Now, if a double layer is used the magnetic charges on the Co-Cr far surface disappear and only weak magnetic charges are dispersively induced on the Ni-Fe far surface, resulting in a much stronger magnetic interaction between the head and the magnetic medium. In the case of our thin-film heads, figure(1.5b), therefore, the magnetic flux flows from the tip of the main pole directly to the back layer, instead of spreading thinly, resulting in a much enhanced flux density. The back layer for its part acts as a magnetic path which leads the flux to the auxiliary pole. This is equivalent to reducing the magnetic reluctance of the air gap between the Co-Cr layer and the auxiliary pole.

In figure (1.6), a schematic diagram of a perpendicularly thin film head is presented.
Figure (1.5). Representation of head-media interaction, showing, a) the induced magnetic charges and, b) the field lines for medium with and without NiFe.

Figure (1.6). A schematic representation of perpendicular thin-film head.
CHAPTER "2"

Models for thin-film growth
2.1 - Zone models for thin-film growth

The formation of thin films takes place via, first, nucleation and then growth.

On arriving at the substrate, the "adatoms" are physically adsorbed onto the substrate, and since they are not initially in thermal equilibrium with it, they move around and interact, forming clusters. These nuclei, or clusters grow in number as well as in size until a saturation nucleation density is reached. A nucleus can grow both parallel to the substrate, by surface diffusion of the adsorbed species, as well as perpendicular to it by direct impingement of the incident atoms. In general, however, the rate of lateral growth at the early stages is much higher than the perpendicular growth. The next stage for these clusters which are now grown into "islands" is to coalesce with each other in an attempt to reduce the surface area in order to reduce their surface tension [10]. Larger islands grow together, and if these islands have a slightly different orientations, a "grain boundary" will occur between the crystals where they meet, figure (2.1).

Of all the crystallographic planes, figure (2.2), by which an h.c.p crystal can be bounded to the substrate, thermodynamically, the one with the lowest free energy will be dominant [11], i.e (00.1). alternatively, the most densely populated planes will be parallel to the substrate.
Figure (2.1). After formation of the grain boundary in (b), it cannot move laterally since its area would increase increasing its interfacial energy. Deposition occurs until stage (c) is reached, when the boundary moves.

Figure (2.2). equilibrium form of a free growing h.c.p crystal.
However, residual gases and, where relevant, deviations from stoichiometry may change the ideal equilibrium.

As to which crystallographic direction is the one of the fastest growth, the survival-of-the-fastest regime will apply [13], i.e., the densest axes will grow out of existence, for they require large amount of flux for a given growth, figure (2.3). For h.c.p crystals c-axes is the least dense direction.

The orientation during the nucleation process can be further enhanced by preferred growth of the oriented nuclei. However, even if the nuclei themselves have a random orientation, a preferred orientation can develop from them in the course of the development of initial stages of the film growth due to preferred growth of crystals with certain orientations [14]. This seems to be the growth pattern for CoCr films, where the initial layer, as will shortly be seen, does not exhibit a strong preferred orientation, whereas the thicker films are well oriented.

If during the growth process the whole of the crystal surface is not supplied with sufficient atom flux, the growth form can deviate from the equilibrium form. Therefore, if atoms (or molecules) are not supplied to the crystal from all directions, and the surface diffusion is
Figure (2.3). The direction of fastest growth of a free growing h.c.p crystal. This will determine the preferred orientation of the growing film.
not sufficient to compensate for this uni-directional adatom supply by redistribution, there will be a memory of the angle of incidence of the adatoms on the crystallographic growth direction. In the case of perpendicular molecular flux, these deviations will be such as to promote a perpendicular columnar habit [15].

More generally, the coating condensation process in sputtering occurs in three steps. The kinetic energy of the incident atoms is transferred to the lattice and, therefore, becomes a loosely bound adatom. These adatoms then migrate over the surface exchanging energies with the adsorbed species as well as the lattice until they are either desorbed by the impact of high energy ions (particularly in bias sputtering), or, more commonly, become trapped at low energy lattice sites. It was further proposed [16] that the development of texture in the deposited films is not only a function of the angle of incidence of the molecular flux and the crystallographic orientation, it is also strongly related to the roughness of the growing thermodynamic probability of adsorbed molecules becoming part of the crystal and all data relevant to the activation energies for the surface and bulk diffusion, and the sublimation energies. For metals these energies are related to the melting point [17].

Thornton [18] presented a zone classification model, figure 2.4a, where it was proposed that various basic
Figure (2.4). Structural zone model for coating growth. (a) Model proposed by Thornton [14] following the model (b) first proposed by Movchan [15].
processes could be expected to be explained in terms of $T/T_m$, where $T$ is the deposition temperature and $T_m$ the melting temperature. This was based on what already existed for evaporated films, figure (2.4 b) but with an extra axis to account for the sputtering gas (Ar). In addition to the three zones already identified by Movchan et. al. [19], Thornton proposed a fourth zone (zone T) consisting of an array of fibrous grains without apparent voided boundaries.

Iwasaki [20] measured an h.c.p to f.c.c transition temperature of the sputtered films 550 K lower than the bulk data. This implies that the surface temperature is that much higher than that measured through the substrate. The substrate temperature is commonly measured to be around 450 K. Therefore, the actual surface temperature could be as high as 1000 K. Considering that the melting point for Co and Cr is around 1750 K, the ratio of $T/T_m \sim 0.6$, and an argon sputtering pressure of 5 m Torr should, according to the zone 2 in the above classification, result in columnar growth. This columnar growth is attributed to the presence of $T/T_m$ sufficient to reduce the adatom surface mobility (and so new crystal nucleation) so that existing crystallites grow as columns, figure (2.5), by the localized epitaxial process [21].
Figure (2.5). A two dimensional growth structure.
CHAPTER "3"

Magnetization in films with perpendicular anisotropy
3 - Anisotropies in perpendicular magnetic films

It was mentioned that to counteract the strong in-plane demagnetizing fields present in thin films, strong anisotropies perpendicular to the film are needed. A knowledge of the kinds of anisotropies present will help in the understanding of the magnetic processes involved.

There are three kinds of uniaxial anisotropies associated with thin ferromagnetic films;

a) Uniaxial crystal anisotropy
b) Shape anisotropy
c) Strain anisotropy

3.1 Uniaxial crystal anisotropy

Figure (3.1) shows the magnetisation curve for a single crystal of cobalt where the c-axis is the direction of easy magnetization. Therefore, energy has to be spent if the magnetization is to be rotated by 90 degrees from this minimum energy magnetization direction to that of the "hard axis". This energy is expressed by expanding it in a series of powers of \( \sin \theta \), where \( \theta \) is the angle between the saturation magnetization and the c-axis;

\[
E_u = K_1 \sin^2 \theta + K_2 \sin^4 \theta + \text{higher terms} ---(3.1)
\]

The first term is sufficient to express the actual anisotropy energy, with \( K \) being the uniaxial anisotropy
Figure (3.1). The magnetization curve for a single crystal of cobalt [78].
constant.

It should be noted that when the grains of a polycrystalline film are oriented randomly in space the net anisotropy of the grains will be zero. It is, therefore, only when they exhibit preferred orientation (texture) that the crystal anisotropies of the individual grains determine the anisotropy of the film.

3.2 - Shape anisotropy

This is the result of differential demagnetization energies (magnetostatic energies) associated with the principle axes of a given sample. In the case of a thin film with uniform magnetization \( M_s \) the total demagnetizing energy density is given in SI units as

\[
\text{Ed} = -\left( \frac{\mu_0}{2\gamma} \right) \left( \int H_d \perp M_s \perp dv + \int H_d \parallel M_s \parallel dv \right) \quad \text{(3.2)}
\]

But the demagnetizing field along the film plane is very nearly zero. Therefore, integrating we obtain

\[
\text{Ed} = \frac{1}{2} \mu_0 M_s^2 \cos^2 \theta \quad \text{(3.3)}
\]

we can substitute \( (1 - \sin^2 \theta) \) for \( \cos^2 \theta \), drop the constant term and write the energy as

\[
\text{Ed} = \frac{1}{2} \mu_0 M_s^2 - \frac{1}{2} \mu_0 M_s \sin^2 \theta \quad \text{(3.4)}
\]
In the case when the aspect ratio of the columns favours perpendicular orientation and there is minimal magnetostatic interaction between the columns (i.e. there is appreciable boundary separation), then the in-plane demagnetization energy may no longer be negligible and the film may even exhibit favourable perpendicular shape anisotropy. In actual fact, the contribution of the columnar shape anisotropy to the total perpendicular anisotropy of the film is negligible [22], with the perpendicular demagnetization energy being considerably greater than that of in-plane.

### 3.3 Strain anisotropy

This uniaxial anisotropy is the result of magnetostrictive stresses set in the magnetic film when it is magnetized. The energy associated with the lattice strain is called the magnetoelastic energy and is given by

\[
E_s = \frac{3}{2} \tau \lambda_s \sin^2 \theta
\]  

where \( \lambda_s \) is the fractional change in length and is called the saturation magnetostriction coefficient and \( \tau \) the in-plane stress.

Therefore the total magnetic anisotropy of a thin cobalt film is given by

\[
E = Eu + Ed + E_s
\]
Although the magnetoelastic contribution to the perpendicular anisotropy can be as much as -20% [23], this term is usually omitted. The remaining part of the equation is then solved for the condition of stable magnetization \( \frac{\partial E_k}{\partial \theta} = 0 \) for perpendicular magnetization, provided \( \frac{\partial E_k}{\partial \theta} > 0 \). Therefore for a film to possess perpendicular anisotropy, i.e. \( \theta = 0 \) degrees, the condition that

\[
K_1 > 0.5\mu_0 M_s^2
\]  

must be satisfied. It also follows that for in-plane anisotropy to prevail, i.e. \( \theta = 90 \) degrees

\[
K_1 < 0.5\mu_0 M_s^2
\]  

There is a quality factor \( Q \) given by

\[
Q = K_1/(\mu_0 M_s/2)
\]  

which, when positive, indicates that the film possesses perpendicular anisotropy. This is thought to be by the precondition of perpendicular anisotropy [24, 25, 26]. On the other hand many authors find this condition too stringent in view of the fact that in the remanent state
the film is divided into domains with antiparallel magnetization, or when the layer is in a recorded state, together reducing the demagnetizing field [27]. Nevertheless, this factor remains a means of quantitative characterization of the film properties.
Figure (4.1) shows typical perpendicular and in-plane hysteresis loops for Co-Cr films measured using a V.S.M. To the nearest approximation they can be represented by the simplified loops of figure (4.2), where the in-plane coercivity and the curvature of the loops are ignored.

The fact that there is shear at all rules out single domain switching of the film. This shearing implies that the saturation magnetization can acquire any value which is inconsistent with a single domain saturation magnetization model, where it would switch abruptly without any shear. Therefore the magnetic microstructure must be multidomain, if the domains are to be held responsible for the switching mechanism of the film. However, similar sheared loops can also exist when magnetization reversal is brought about by the numerous particles "switching" their magnetization, which then average out to the total magnetization. This average magnetization can take any value (depending on how many particles complete switching), leading to shearing.

Therefore, two models are generally presented to account for the shape of the hysteresis loops, a): the model of the isolated particles magnetostatically coupled together through the sheet magnetic charge (Stoner-Wolfarth switching). This model is termed the "particulate" model,
Figure (4.1). Typical perpendicular and in-plane hysteresis loops of CoCr with perpendicular anisotropy.
figure (4.2). The hysteresis loops of a magnetic film with uniaxial anisotropy both with and without demagnetizing field. (a) Field applied perpendicular to the film and (b) field applied in the plane of the film.
and b): the "continuum" model, where magnetization reversal occurs by stripe domain motion as observed by Kooy and Enz [28] for barium ferrite layers and the domains run continuously throughout the film interrupted only by the column boundaries.

4.1 - Particulate model

Figure (4.3) is an SEM micrograph of the fractured cross-section of Co-Cr sputtered film in which the columnar microstructure can clearly be seen. In fact many authors have observed the same kind of columnar morphology in thin magnetic films [28, 5], in general, and sputtered Co-Cr films [29, 30, 31, 57] in particular. It is argued that these columns have a ferromagnetic core with the segregated chromium forming an anti-ferromagnetic shell around them, i.e., they are magnetically separated columns. Iwasaki [32] revealed a honeycomb-shaped grain boundary after chemically etching his Co-Cr films proving the Cr segregation theory.

The above implies that the exchange forces (which decrease very rapidly with atomic spacing) are regularly disrupted by the antiferromagnetic shell, reducing the exchange coupling between the columns, if not destroying them altogether. Therefore, conventional domain wall motion would be difficult to envisage, and "interaction domains" would then be the best we could hope for.
Figure (4.3): A S.E.M fracture cross-section of a 3 micron thick film showing the columnar structure right through the thickness of the film.
4.2 - Coercivity of the particulate model

Consider an aggregate of single domain particles (from which, by definition, domain walls are excluded) in the shape of prolate spheroids, figure (4.4a) whose easy axes are aligned. Stoner and Wohlfarth [33] examined the magnetization rotation of such a system, where it was assumed that Ms vector of the single domain rotates out of the easy direction against some kind of restoring force of some anisotropy, usually shape, stress or crystal anisotropy. They calculated the hysteresis loops of figure (4.4b) for the case when the magnetic field made an angle \( \Theta \)-ranging from 0-90 degrees. It can be seen that only when the field is parallel to the easy axes that the rotation is completely irreversible. At other angles the curved parts correspond to the reversible parts of the hysteresis.

Consider now that the shape anisotropy is the relevant restoring force, the anisotropy field (and therefore the coercivity) is given by

\[
H = (N_a - N_c) M_s
\]  

when \( c/a = 10 \) (reasonable ratio for Co-Cr particle) \((N_a - N_c)\) for an elongated particle is equal to \( 2\pi \), and if we take a typical value of \( M_s \) for CoCr ~700 emu/cc, we will obtain for coercivity a value of around 4300 Oe, which
Figure (4.4). (a) Rotation of magnetization in an ellipsoid, and (b) the resulting hysteresis loops as the angle between the field and easy axes is varied [78].
is evidently much too high for the values obtained experimentally.

Should we take the crystal anisotropy alone accountable for the restoring force, the anisotropy field is then given by

\[ H = \left( \frac{2K}{Ms} \right) \]

for pure cobalt yields

\[ = 2 \times 45 \times 10^5 \div 1422 \]
\[ = 6300 \text{ Oe} \]

These rather high values of coercivity suggest that anisotropy-reducing modes of rotation must be in action. "Curling" is proposed [78] as a possible mode of rotation, figure (4.5), in which shape anisotropy is effectively eliminated if the c/a, the aspect ratio of the columns, is large, as the spins are then parallel to the surface and no free poles are formed, drastically reducing the magnetostatic energy involved. The energy barrier to curling reversal is then entirely exchange energy. In the case of intermediate values of c/a, the energy barrier includes exchange and magnetostatic energy.
Figure (4.5). A schematic diagram of "curling" and "coherent rotation" modes [72].
4.3 - Magnetization reversal by wall motion

There are many authors who believe that the presence of columns, although favourable, is not a necessary condition for CoCr films with perpendicular anisotropy [33.5]. Some have gone as far as discounting altogether any role that the columnar morphology may have in effecting the magnetization reversal of CoCr films [34, 35].

In this mode of reversal domains are nucleated in the particle; movement of the domain wall inside the particle effects the magnetization reversal in Barkhausen jumps. The wall nucleation has its roots in spin rotation on a few atoms at the surface against the crystal anisotropy forces of magnitudes given by, as in the case of coherent rotation, by

\[ H = \frac{2K}{Ms} \]

which is too high compared to the magnitude of the external fields normally applied when magnetizing a film.

Real crystals, however, suffering from imperfections such as interstitials, dislocations, etc, produce local fields which act to demagnetize the particle. Advocates of this mode of reversal [34, 35] contend that their measured domain nucleation fields are compatible with measured coercivity results. They argue that the exchange forces do
extend through the crystal. Maximum coercivity then occurs when the domain wall thickness is equal to the pinning sites (column boundaries) which are in theory the most major sources of crystal imperfections.

Although recent observations with digitally enhanced Kerr Microscope [36] have gone a long way towards justifying stripe-domain behaviour, it is thought that both models act together in bringing about the rather intricate magnetic behaviour of Co-Cr film in different composition range and thickness region.
CHAPTER "5"

Experimental techniques for determination of structure, preparation and deposition of CoCr films
5 - Experimental techniques for determination of structure in CoCr films

5.1 X-ray diffraction

Figure (5.1) is a schematic diagram of the X-ray diffractometer used in these experiments. Here the specimen is mounted in the centre of the diffractometer and is rotated by an angle around an axis in the film plane. The arm holding the counter moves around $2\theta$, i.e. twice as large as the specimen rotation. Focusing is achieved by making the specimen a part of the circumference of a circle, the focusing circle. It is noticeable that only the (hkl) planes parallel to the film plane contribute to the diffracted intensity.

The diffracted radiation was detected by a counter tubes which move through the angular range of the radiation. The intensities were then recorded on a chart recorder for processing.

When using a monochromatic radiation, the interplanar d spacing between the neighbouring $(h,k,l)$ planes as well as other lattice parameters for h.c.p crystallographic orientation are given by [36.5]

$$
\frac{1}{d^2} = \frac{4}{3} \left( \frac{2}{h^2} + \frac{2}{hk} + \frac{2}{k^2} \right) / a^2 \left( 1 / c \right)
$$
Figure (5.1). A schematic diagram of a Bragg-Brentano X-ray diffractometer.
also the Bragg angle is calculated through the Bragg Law

\[ \lambda = 2d \sin \theta \]

Hence, for \((h,k,l) = (00.2)\)

\[ \frac{1}{d} = \frac{4}{c} , c = 2d = \lambda / \sin \theta \quad (00.2) \]

and for \((h,k,l) = (10.0)\)

\[ \frac{1}{d} = \frac{4}{3a^2} , a = 2d/a = \left(\frac{1}{3}\right)\lambda / \sin \theta \quad (10.0) \]

5.2 - Electron diffraction technique

Although X-ray diffraction benefits from the advantage that separation of the films from the substrates is not needed, the rather low scattering power of atoms for X-rays implies that only films of sufficient thickness (over 1000 angstroms) would result in adequate diffraction intensities. On the other hand, the intensity of scattered electrons is a hundred million time that of X-rays, making the diffraction of films as thin as 50 angstroms possible.

Figure (5.2) is a schematic illustration of a transmission electron microscope in the diffraction mode. As the beam of the electron passes through the polycrystalline material, peaks of intensity occur in the diffraction pattern in the form of concentric rings. These diffraction maxima appear because only those electron
Figure (5.2). Schematic representation of the electron scattering and final diffraction pattern from a polycrystalline specimen.
"waves" which are scattered from successive parallel lattice planes lying at particular orientations, through the depth of the crystal, are in phase and combine constructively. The particular orientations at which sets of lattice planes with certain d spacings will diffract the electron beam to form rings. As in the case of the X-ray diffraction, the Bragg law governs the diffraction, i.e.

\[ \lambda = 2d \sin \theta \]

Now, consider figure (5.3), where the electron microscope is reduced to a simple diffraction camera, forming a ring at a distance R from the centre of the diffraction pattern. The distance between the specimen and the plate, the camera length, is designated L. By simple geometry

\[ \tan 2\theta = \frac{R}{L} \]

for small angles

\[ \tan 2\theta = 2 \sin \theta \]

then, using the Bragg law

\[ \frac{R}{L} = \frac{\lambda}{d} \]

or
Figure (5.3). A simple representation of electron diffraction using an electron microscope.
\[ Rd = \lambda L \]

So if values of \( R, L \) and \( \lambda \) for a particular diffraction ring can be measured, then the \( d \) spacings can be determined.

The value of \( \lambda L \) was determined by evaporating a thin film of gold whose \( d \) spacings are given in the ASTM file 4-0784. By measuring the radius of any one of diffraction rings and taking its corresponding \( d \) spacing (for instance the (111) diffraction ring for gold has a \( d \)-spacing of 2.355 angstroms), the electron microscope was calibrated. Without altering the voltage and the value of \( L \), diffraction rings for each CoCr films were obtained, whose \( d \) spacing could then be measured using the above relationship. These \( d \) values were then compared with those given for Cobalt in ASTM file 5-0727 for crystal structure determination. Table \( \chi \) shows the results of one of the bias sputtered films whose electron diffraction is compared with ASTM file 5-0727. The measured lattice spacings for the 300 angstrom film are in close agreement with the hcp cobalt.

The accelerating voltage of the T.E.M unit used was limited to 100 KV, and therefore diffraction of films thicker than ~ 300 angstroms could not be achieved. Nevertheless, this order of resolution was thought to be sufficient to reveal the crystal structure of the very
Table (1). Comparison between the lattice spacings as given by ASTM for Alfa Co and that calculated by electron diffraction method.

<table>
<thead>
<tr>
<th>hk.l</th>
<th>(d(\AA))</th>
<th>(\delta(%))</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.0</td>
<td>2.165</td>
<td>2.19</td>
</tr>
<tr>
<td>00.2</td>
<td>2.023</td>
<td>2.07</td>
</tr>
<tr>
<td>10.1</td>
<td>1.910</td>
<td>1.96</td>
</tr>
<tr>
<td>10.2</td>
<td>1.48</td>
<td>1.46</td>
</tr>
<tr>
<td>11.0</td>
<td>1.252</td>
<td>1.26</td>
</tr>
</tbody>
</table>
important initial layers of the growing film.

For films thicker than 1000 angstroms, however, the X-ray diffraction technique proved very useful, especially when considering that it is the preferential orientation of 0.3-0.5 micron thick films which is desired.

5.3 -X-ray microanalysis (Electron-probe microanalysis)

When an area of a solid is bombarded with high energy electrons, the resulting emission includes backscattered primary electrons, low energy photo-electrons and Auger electrons together with characteristic X-ray emission, whose wavelengths depend on the nature of the atoms in the specimen, superimposed on a background of continuous X-radiation (Bremsstrahlung). It is the characteristic X-ray spectra which are utilized in electron-probe microanalysis. Measurement of the wavelength (or energy) of each characteristic X-ray enables us to find which elements are present in the specimens; measurement of rate of emission of a particular X-ray should also tell us how much of the element is present, i.e. its concentration. The latter quantitative measurement was of particular interest in this work.
5.4 Rutherford Backscattering Technique (R.B.S)

R.B.S is a technique of structural and interfacial analysis of thin epitaxial or non-epitaxial films, which was used in this work to complement X-ray microanalysis. Its importance is primarily because of its ability to analyze in depth and because of its intrinsic mass dispersion.

R.B.S is basically an ion scattering phenomenon and as such rests on the fundamental relationships that govern ion scattering in solids. Briefly, as an energetic ion traverses a solid, figure (5.4), it gradually loses its energy when encountering atomic monolayers, coming to rest tens of microns below the surface. However, a small fraction of the particles undergo energetic nuclear encounters before coming to rest. It is these nuclear backscattering that represent the signal in ion scattering solids [37]. The final energy of the particle determines the depth at which the scattering event occurred, since the particle loses energy to the electrons in penetrating the solid.

When analysing the various elements of the film, to ensure that the peaks from different elements in the substrate do not swamp the signal from the various elements of the film itself, the films for R.B.S analysis were intentionally sputtered on single crystal silicon.
Figure (5.4). A schematic representation of R.B.S. and energy spectrum of the scattered particles corresponding to scattering from various depths of substrate [37].
substrate which, unlike glass, does not contain the especially troublesome element of Barium whose R.B.S signal is uncomfortably close to those of cobalt and chromium.
Co\(_{1-X}\) Cr\(_X\) and Ni\(_{75}\) Fe\(_{21}\) under investigation were prepared by r.f-sputtering technique.

### 6.1 - Sputter deposition

In sputtering, the material of interest is passed into the vapour phase by ion bombardment of the "target", the source of the coating material. These ions are provided by the inert gas (argon in the range of 2-20*10\(^{-3}\) torr) which is introduced to the sputtering chamber to form a plasma with the aid of an r.f power supply, once the chamber is evacuated to background pressures as low as 10\(^{-7}\) torr. The power supply maintains the target at a negative potential (1500-2500 V) relative to the plasma. The argon ions are drawn from the plasma and hence accelerated towards the target surface, whereupon they strike with sufficient energy to dislodge the atoms resulting in their ejection to all directions. The substrate is suitably positioned to intercept these atoms.

A Nordiko NM2000 r.f sputtering unit was used to deposite the Co-Cr films, figure (6.1). The planar diode geometry employed by this unit is schematically represented by figure (6.3). The discharge is operated at 13.56 MHz. At such high frequencies the massive ions are unable to follow the temporal variation of the applied potential. Now, if one of the electrodes is coupled with
Figure-6.1 Schematic of pumping system
Figure (6.2): The "NordiKo Rig"
Figure (6.3). Electron, ion and voltage distribution for a planar-diode sputtering.
the r.f generator through a capacitor a negative voltage will present itself on that electrode alone, therefore sputtering is obtained from only one electrode [41].

6.1.1 - The choice of deposition process

Sputtering as a deposition process has valuable advantages over other deposition methods that could have been utilized. Because of the much higher kinetic of the ejected atom compared to evaporation for example (50-100 times), the sputtered atoms reach the substrate with sufficient energy to surmount those activation energies required for reacting with the substrate and for reforming the compound from which they originate [38]. The energy of the sputtered atom may also contribute to improved adhesion by permitting these atoms to become the bombarding species in removing weakly bonded contamination from the substrate surface [39]. Furthermore, the most energetic of the sputtered atoms may promote adhesion by penetrating into the substrate [40]. The sputtering rate is also much less dependent on the specific properties of the coating material. For example, the sputtering yields of some materials may be fractionally off, whereas, the evaporation rates of the same materials can be very different [41].
6.2 - Sputtering chamber

The sputtering chamber consists of a 24 inch diameter stainless-steel cylinder within which three target electrodes sit on the target work table, and a substrate holder electrode (anode), directly facing the target, sits on the substrate work table. At any deposition run, therefore, three different layers could be deposited.

6.3 - The targets

The targets were in thermal contact with the water-cold electrodes through copper backing plates. A cobalt disc, of 99.99% purity and 8 inches in diameter, on which 1cm pieces of chromium were hexagonally arranged [42] was used (in the form of a composite target) to give the desired composition with maximum uniformity throughout the film.

The Ni-Fe target was also 8 inches in diameter and was an alloy target, and therefore, of fixed composition.

6.4 - The substrate holder

The substrate holder was also of sufficient thermal mass and surface finish to realize a fine uniformity in the temperature over the surface of the deposited film, as any temperature difference would alter the film composition and
lead to localized magnetic properties. There was an optional substrate water-cooling facility through the substrate holder electrode which was employed, except when substrate heating was undertaken. To achieve maximum contact the substrates were firmly clamped on the holder.

6.4.1 - The sample heater
-----------------------------

When sample heating was required, to avoid outgassing of the entire substrate work table by using the heating system already incorporated in the electrode, a separate substrate heater was constructed which was then mounted 2 inches away from the electrode, figure (6.4). It comprised of two stainless steel plates (for minimum out gasing) between which a flat heating element was sandwiched. A "radiation shield" was mounted half way up the spindle connecting the heater to the electrode to increase the efficiency of the heater as well as preventing the heating of the electrodes. When using this arrangement, the electrode's height was subsequently adjusted to compensate for the heater's protrusion.

The temperature of the samples was measured by placing a chromel-Alumel thermocouple of a very low thermal mass (for rapid response) on the surface of a glass substrate identical to the one on which Co-Cr was deposited for experimentation. Hence the temperature measured was as close to that experienced by the growing film as possible.
Figure (6.4). A schematic diagram of the home-built substrate heater.
Figure (6.5): The substrate heater and temp. controller
Since the thermocouple was at high voltages (especially when bias sputtering was performed), the meter was placed in an insulated cage and the meter's reading was optically interfaced for temperature control purposes.

6.5 - Sputtering procedure

After evacuating the sputtering rig to better than $2 \times 10^{-7}$ torr (normally achieved after three hours), the target shutter was placed in between the target and the substrate and, using the power divider, 100 watts of power was applied to both the target and the substrate electrodes. The throttle was then closed above the diffusion pump (to reduce its pumping capacity) and controlled amounts of argon were introduced into the chamber via a micro-flow valve. In this way, a plasma at both the target electrode and the substrate electrode was established. Constant tuning of the matching unit was found to be necessary—at least for the initial twenty minutes of sputtering—mainly due to the thermal stabilisation of the plasma, reflecting itself on the capacitance and the inductance of the plasma.

Further to sputter cleaning for appropriate interval (normally ten minutes), the required deposition power was set on the target with the substrate bias adjusted to the proper value, or removed altogether when not called for, and the deposition was commenced by removing the shutter.
6.6 - The substrates

The substrates used for our investigation were silicon wafers with 001 preferred orientation; 2 inches square Boro-Silicate glass wafers of 0.5 mm thickness; 0.1 mm thick molybdenum sheet of 99.99% purity cut into 2 by 1/2 inch strips; 5.25 inch diameter aluminium discs, and finally, mica wafers on which 100 angstrom thick carbon layer was evaporated. The last substrate was used for analysis of films under Transmission Electron Microscope.

6.7 - Substrate cleaning

All the substrates were cleaned in hot Decon solution using an ultrasonic bath for a period of 20 minutes. They were then immersed in deionized water and subsequently dried (using 99.9% pure nitrogen jet). They were further bathed in acetone and I.P.A for approximately ten minutes. The last two procedures help to reduce the hydrocarbon contamination prior to evacuation of the rig, as well as acting as dehydrating agents.

Although the liquid nitrogen trap between the chamber and the diffusion pump prevents most of the oil vapours from back-streaming into the chamber, some hydrocarbon contamination of the substrates is inevitable. Therefore sputter cleaning of the substrates was routinely performed [43] to reduce this contamination, in addition to
removing the ubiquitous oxide layers on the substrates. Low sputtering powers (of the order of 100 Watts) was applied to reduce the possibility of surface damage caused by ion bombardment to the substrates.
CHAPTER "7"

Characterization of sputtered CoCr films

-66-
CoCr films of different sputtering characteristics were sputtered on glass, silicon, molybdenum and mica (covered with a 100 angstrom layer of carbon) substrates. Unless otherwise specified, the base pressure was \( <2 \times 10^{-7} \) torr, and the target to substrate distance was 7 cm, and the film thickness was 0.5 micron, deposited at an argon pressure of 5 m-torr.

A few trial films were first sputtered and the perpendicularity of the hexagonal C-axis, which is the main criterium of a good perpendicular anisotropy, was measured using X-ray diffraction intensity technique. It was thus realized that only the films which were bias-sputtered exhibited good perpendicular orientation. Furthermore, there is evidence that negative substrate biasing leads to an improvement in the surface smoothness of the films [44] which is of prime importance especially when head flying height of less than 1000 angstroms is required in order to ensure adequate read/write signal.

7.1 Effect of biasing on the orientation of the films

Figure (7.1) shows the variation in (00.2) X-ray orientation against the negative substrate bias for films containing 16% Cr, sputtered at an argon pressure of 5 m-
Figure (7.1). (00.2) and (10.0) X-ray intensity counts, Ms and Cr content dependence of the films on negative substrate bias voltage.
torr, and sputtering power of 0.8 KW. A (00.2) intensity peak at around -80 V can be seen, while the (10.0) X-ray diffraction intensity, which represents the orientation in the plane of the film, remains small and constant. Therefore, an appreciable improvement in the orientation of the C-axis is made when a bias of around -80 volts is applied to the substrates while sputtering. There have, indeed, been reports before of improved preferred orientation as a direct result of application of negative bias [45].

Figure (7.2) is the TEM micrograph of biased and unbiased films, approximately 300 angstrom in thickness, with the inset showing the corresponding TEM diffraction pattern. Although the diffraction images are similar, i.e. all the reflections are present in both cases, and are typical of h.c.p. polycrystalline samples of very small grain size, the micrographs are distinctly different. In the case of the films with bias there is more order in the topography than there is in the unbiased films. This could be due to the stacking faults introduced in the films, probably resulting from the gas contents of the films. This will be later discussed when considering the impurity contents of the films. It has also been shown that presence of negative bias brings about some resputtering of the film material [46], and high values of the so called "re-emission coefficient" during film growth is essential in obtaining good quality films. This is said to be the
Figure (7.2): T.E.M. micrograph of unbiased and biased films with their corresponding electron diffraction. Although the diffraction rings for both are the same, there is a distinct difference in their texture.
Figure (7.3): S.E.M. micrographs of a 2 micron thick CoCr films sputtered under different bias voltages. The film "b" shows the best texture.
consequence of the fact that many molecules that would otherwise be trapped in non-optimum positions (e.g. interstitials) are returned to the vapor phase leaving behind material of high quality. Vossen et al. [47] in fact showed that the density of thin films increases and then decreases with increasing bias, reflecting possibly on the improvement of the crystal structure in the high density region of the films.

To understand at which stage of the columnar growth biasing is more effective in advancing (00.2) orientation, a simple experiment was carried out in which the bias was switched on at various stages of the film growth. In so doing, it was learned that biasing was more effective when applied during the initial stages. For example, two films were sputtered with identical sputtering parameters each for 20 minutes except that in one of the films a bias field of -80V was applied to the substrate during the first 8 minutes of the film growth, and in the other, it was switched on after 10 minutes of the sputtering. A (00.2) X-ray intensity count of 38500 was obtained for the former, whereas that of the latter was a mere 1280 counts.

7.2 Physical effects at the target and the substrate

In order to explain the effect of the negative substrate bias on the topography and crystallographical orientation of the films, a brief review of some effects
that occur at the target, in the gas discharge, and at the substrate is necessary.

When the solid surface of the target is bombarded by energetic ions, several effects may occur some of which are illustrated in figure(7.4): (1) emission of the sputtered neutral particles; (2) emission of secondary electrons; (3) emission of positive and/or negative ions; (4) emission of radiation; (5) reflection of incident particles; (6) desorption of gases; (7) implantation of incident particles; (8) heating; (9) chemical dissociation; (10) bulk diffusion; (11) crystallographic changes; (12) reflection of some emitted species back to the target surface either by collision in the gas discharge or electrostatic attraction [48].

However, the fraction of the positive ions that reach the substrate which originate from the target is very small (<1%) [49], and do so through elastic collision with the target. As will be seen in a latter chapter, this is the main mechanism of argon entrapment in the films. Nevertheless, most of these "primary" bombarding particle are neutralized upon reflection to atoms again. In any case, these particles are a source of high energy bombardment of the substrate, which can noticeably effect film properties [50]. The role of these bombardments, being especially prevalent in sputter deposition process, in promoting for example certain kinds of lattice defects will now be discussed.
Figure (7.4). A schematic representation of the target bombardment phenomenon.
7.3 - Defect production in crystals

The impact of an ion or an atom with a solid and its subsequent movement through the solid will result in its gradual loss of energy in a succession of collisions. This energy is dissipated to the surrounding atoms leading to an increase in their kinetic and potential energies and possibly resulting in ionization, when the excitation is to the atomic electrons. If in a collision an atom can receive enough kinetic energy, it will be ejected from the equilibrium lattice position to which it is normally bound by the forces of interaction, creating a vacancy in the atomic array and repositioning an extra lattice atom to some distance from the vacancy. The extra atom could itself promote further displacements if it is in possession of sufficient energy. The atoms thus displaced could occupy vacant lattice positions or may come to rest at "non-normal" positions in the lattice known as interstitials. The injected atoms or ions themselves having come to a halt may occupy an interstitial position and occupy a vacant lattice site and become a substitutional impurity. These are a class of defects known as simple, or point, defects.

In addition to these point defects, extended defects in solids may also occur which are in effect caused by the point defects agglomerating in the form of clusters. The strain in the lattice caused by these clusters may be
relaxed either by the presence of impurity atoms in the matrix, or the occurrence of dislocations.

A dislocation, which is an example of extended defects, is the collapse of the vacancies and/or interstitials, which represent a discontinuity in the lattice perfection over an extended area, into a plane or sheet as a result of lattice relaxation in the vicinity of the clusters. Generally, dislocations are not formed by thermal means but by mechanical stress which cause deformation, radiation damage which produces super-thermal defect concentrations, and non-uniformity of the crystal growth process [51].

An impurity atom, on the other hand, can accommodate into a host lattice in many ways. If it occupies a lattice site it is known as substitutional impurity, or if in isolation in a non-normal lattice site, an interstitial impurity. It often turns out that impurities act as effective "sinks" for lattice defects, in which case it can promote defect clustering process. Example of this is formation of gas bubbles by the agglomeration of inert gas atoms and vacancies in sputtering systems [52]. Although inert gases are quite insoluble in most solid, in many cases they condense into the "host" material at free surfaces to minimize the energy of the system [53].

The role of inert gas entrapment on the structural
and magnetic properties of the films will be studied in a later chapter. What is certain is that the particular vulnerability of crystal formation to ion bombardment and its possible subsequent defect formation makes it necessary for us to have a closer look at various occurrences at the substrate.

7.4 - Further occurrences at the substrate

The secondary electrons that are emitted from the target are accelerated towards the substrate due to its being at a high negative potential. After suffering some inevitable collisions their remaining energy is transferred to the substrate in the form of heat [54].

At the substrate three interactive effects take place. Material from the target arrives at the substrate surface and condenses from an "excited" state; the substrate is subjected to bombardment by various energetic particles, as can be seen from figure (7.5). Apart from the effect that these can have on defect formation discussed above, they can be a major source of heat transfer through their kinetic energy to the substrates. Condensation too can produce heat, leading to an interactive state between these two heat generating phenomena. For instance, both bombardment and condensation produce heat. Heat, in turn, effects condensation phenomena, and so on. What determines the film properties is the totality
Figure (7.5). The particles bombarding the substrate.
of events occurring at the substrate [55]. It is possible to control all these effects, and so the film properties can be adjusted. It is difficult to directly modify the behaviour of the neutral particles. However, the charged particles can be controlled by changing the local electric field, and that is exactly what bias-sputtering does.

By applying a negative potential to the substrate, it can be arranged [56] for the substrate platform to become a secondary sputtering target, thus the flux and energy of all charged particles bombarding the substrate can be modified.

The role of bias sputtering in affecting the impurity content of our Co-Cr films will first have to be studied before a judgement is made regarding the process responsible for the appreciable improvement in the orientation of the films around certain value of bias field. This will be undertaken elsewhere in this work.

7.5 - Chromium segregation

The saturation magnetization in figure (7.1), as measured by VSM, shows a monotonic decrease with negative substrate-bias, while the Cr content, as measured by the x-ray microanalysis, remains approximately constant. This is attributed to the temperature-dependent segregation of
Cr atoms at column boundaries [57]. It is thought that in the absence of bias Cr segregation is promoted, figure (7.6a). In this way the Cr gather around the columns, leaving the cores rich in Co. Evidence of the segregation was presented by Iwasaki [58]. A CP (chrysanthemum-like pattern) structure has recently been observed at elevated substrate temperatures [59], figure (7.6b), which is claimed to explain the continuous transition of the magnetization between the continuous and particulate modes. Haines [60] presented a mathematical model for the atomic distribution of Cr in CoCr films in which temperature dependent Cr segregation to the grain boundaries is predicted.

Although addition of negative bias aids to repel the energetic electrons from the substrate, it helps to attract an equivalent current of positive ions which then dissipate their energy [61] leading to an increase in the temperature of the substrate. This could provide additional mobility for the adatoms [62], altering the microstructure of the films. Conversely, the same in-flux of ions can also decrease the adatom mobility as they are capable of creating radiation damage which can lead to an increase in the density of the nucleating sites. It is likely that the observed effect of biasing on the Cr content of the films is a combination of the above. These factors could then effect the mobility of the Cobalt and Chromium differentially.
Figure (7.6), a) a conventional Cr segregation model and b) a chrysanthemum pattern (CP) recently observed by Maeda et al. [59].
An attempt to measure the increase in the surface temperature using a thin-film thermocouple (of a very low thermal mass) posing as a substrate was not too successful. It is thought that this increase in temperature is localised lattice heating and limited to a radius of the order of 10 angstroms [63]. This discrepancy between the bulk and the surface temperature was indeed demonstrated by Iwasaki [20] where he measured an hcp to fcc transition temperature 550 K lower for the CoCr films (measured from the substrate during sputtering) compared to that obtained from the bulk data, as was mentioned earlier.

7.6 Magnetic data for films with bias

Figure (7.7) shows some corresponding magnetic properties for the films of figure (7.1). The results show, within graphical error, perpendicular coercivity and remanence ratio reaching maxima with the orientation at the same time as the in-plane values are minima. The peaks are slightly broader and shifted indicating that orientation is not the dominant and the only factor influencing the coercivity. To take account of this a best bias voltage of -80V to -120V was chosen for the rest of the experiments. The decrease in Hk beyond around -120V is a breakdown of the hcp structure and possibly the loss of shape anisotropy due to too much decrease in the amount of Cr segregation.

-82-
Figure (7.7). Some in-plane and perpendicular magnetic properties Vs. negative substrate bias voltage.
Figure (7.8) shows the variation of perpendicular and in-plane coercivity and remanence as measured by VSM, with figure (7.9) showing the variation of (00.2) X-ray diffraction intensity and the anisotropy field (Hk) as the Cr content of the films is altered. It can be seen that the collapse of the in-plane remanence is followed by an increase in the perpendicular orientation and, hence, perpendicular remanence and coercivity, as the chromium concentration is increased. A maximum in the perpendicular remanence and coercivity is found for 16 atomic % Cr. This is a good illustration of the way in which addition of Cr enhances the degree of the C-axis orientation normal to the film plane, as was mentioned in the introduction. The increase in the Cr concentration has lead to an increase in Hk, which when exceeding $4\pi M_s$ (i.e. the maximum demagnetizing field) will give the CoCr film an easy axis of magnetization normal to the film plane.

In figure (7.10) the variation of saturation magnetization, as measured from the hysteresis loops obtained from VSM calibrated using Ni-Fe foil, together with the data for the bulk [64] is shown. This data is similar to that obtained by Iwasaki [65] and Kobayashi [66].
Figure (7.8): Variation of perpendicular and in-plane coercivities and remanence ratios as a function of Cr content.
Figure (7.9). Variation of anisotropy field, $H_k$, and X-ray orientation against Cr content. Although the X-ray intensity count shows a monotonic increase, $H_k$ starts to decrease above 14 at.% Cr.
Figure (7.10). Variation of saturation magnetization of CoCr against Cr content.
7.8 - Argon pressure dependence of CoCr films

In figure (7.11) the variation of (00.2) diffraction intensity against the argon pressure for both the biased and unbiased films is shown. The surprisingly large increase in the orientation for films sputtered at about 1-3 m torr was found to be the result of a negative floating potential $V_f$ set up on the substrate. This potential arises because of the higher mobility of electrons, as opposed to ions, in the discharge so that more electrons reach the surface than ions [67]. In effect, the substrate has set up its own negative bias. Indeed, a voltage reading of around 40 Volts was obtained when a voltmeter was connected between the substrate and the earth when sputtering at those values of argon pressures.

Application of an external negative bias to the substrate leads to a clear maximum in (00.2) intensity count at about 3 m-torr Ar pressure, with figure(7.12) showing the corresponding magnetic results. A peak in the perpendicular remanence and coercivity the same Ar pressure can be seen. These effects could only be explained in the light of the influence that sputtering pressure has on the heating of the deposited film, namely, a high gas pressure can aid in slowing the numerous energetic particles (electrons, ions, and neutrals) which tend to bombard the
Figure (7.11). Variation of (00.2) X-ray intensity against argon sputtering pressure for both biased and unbiased films.
Figure (7.12). Variation of perpendicular and in-plane coercivities and remanence ratios of the films against argon sputtering pressure.
substrate, and therefore minimize substrate heating [68].

The effect of argon pressure on the argon content of the films will be described elsewhere.

7.9 - Sputtering power dependence of CoCr films

Figure (7.13) shows the variation of (00.2) and (10.0) of biased and unbiased films as the sputtering power is varied in the range of 0.2-1.2 KW. No appreciable difference in the intensity of the biased films between 0.2-1.0 Kw can be observed.

However, the magnetic measurements of the same films, figure (7.14), indicate that the utilization of the two extreme powers yields films of poor magnetic quality. This could be due to the fact that when sputtering at very low rates the heat flux to the growing layer may not be sufficient to cause adequate desorption of harmful impurities. If we consider that the deposition rate is actually the net accumulation rate of the film material which includes the effect of the condensation and re-sputtering (due to bias and other ion bombardment effects) rates, the behaviour at high sputtering rate could most likely be the result of the condensation rate being so high that the re-emission rate cannot remove the material arriving in non-ideal positions before new film material arrives and covers it up [69]. This implies that a balance
Figure (7.13). Graph of (00.2) and (10.0) X-ray intensities against sputtering power.
Figure (7.14). Variation of perpendicular and in-plane coercivities and remanence ratios against sputtering power.
between condensation rate and re-emission rate must be established to produce films of high quality. Also, at much higher sputtering rates, the heat flux and the intensity of ion bombardment may be such that it may bring about breakdown of the lattice structure. A sputtering power of between 0.6-1.0 KW, where a plateau in the magnetic properties is observed, is recommended.

7.10 - The effect of use of various substrates

It has been claimed by some authors [70] that the use of silicon, as opposed to glass, as a substrate should lead to an improvement in the c-axis orientation being attributed to the charges set up on the insulating glass which are otherwise conducted away by silicon. No such an orientational improvement, as measured by (00.2) X-ray intensity was observed when sputtering on silicon. The coercivity and perpendicular remanence of the films sputtered on silicon was in general lower, with its corresponding in-plane remanence being larger which again reflects adversely on the perpendicular orientation of the CoCr sputtered on silicon.

At least one other author [71] has observed the same kind of properties for their CoCr film sputtered on Si substrates. It is believed that glass presents a good starting base for the early development of polycrystalline structures since, being amorphous, it does not impose its
own -what could be unfavourable- crystal structure on the growing film.

The CoCr films sputtered on NiFe (sputtered on glass substrates), however, presented a better picture than Si, yet they were not an improvement on glass substrates.
Figure (7.15) shows the way in which the development of (00.2) orientation takes place together with the variation of perpendicular coercivity of the films as measured by V.S.M. against thickness for films biased at -120V. The maximum orientation is reached for film thickness of the order of 6000 angstroms and beyond. This is in agreement with the development of the fiber texture as the film thickness increases. Coughlin et. al. found a similar value for their films [72].

Although the diameter of individual CoCr crystallites show a lot of dispersion [73, 74, 75], and the average diameter is not exactly uniform throughout the film thickness [76], it can be assumed that the crystallite diameter is approximately one order of magnitude smaller than the film thickness, figure (7.16), after Coughlin [77]. With this assumption, an understanding of the shape of the coercivity curve may be acquired by referring to figure (7.17a), in which the variation of coercivity is given against particle diameter [78], where it is said that the mechanism by which the magnetization of a particle changes differs from one part of the size range to another as follows:
Figure (7.15). Variation of coercivity and (00.2) X-ray intensity against the film thickness.
Fig. 7.16-Grain diameter VS film thickness
(After Coughlin [77])
Figure (7.17a). Variation of coercivity with particle diameter.

Figure (7.17b). Relation between the energy $E$ of a crystal and its linear dimensions $D$ for two kind of magnetic state.
Multidomain. Where magnetization changes by domain wall motion. A crystal will spontaneously break up into a number of domains in order to reduce the magnetostatic energy, figure (7.17b), it would have if it were a single domain. Although the demagnetizing field along the long axis of CoCr is small (long particle of aspect ratio 10 to 1), it may still reverse its magnetization by domain wall motion owing to lower switching fields. The size dependence of the coercivity is experimentally found to be

\[
H_c = a + \frac{b}{D^2}
\]

where "a" and "b" are constants.

Single-domain. Below a critical Ds diameter the particle becomes single domain. Figure (7.17b) shows why the crystallite may prefer to remain in the single domain state. Magnetization reversal can therefore only take place by rotation of whatever anisotropy forces (crystal, shape, etc.) happen to be present. As the particle size decreases further the coercivity decreases because of thermal vibrations according to

\[
H_c = g - \frac{h}{D}
\]

where "g" and "h" are constants.
7.12 - Effect of heating the substrates

Using the substrate heater in conjunction with the temperature controller, the substrates could be heated to temperatures up to 320 degrees C, as recorded by the sensor mounted on the substrates. Since even in the absence of an external heater, substrate temperatures as high as 170 degrees C were recorded, it was decided to start the temperature measurements from 170 degrees C, and not the substrate holder's (usually just above the room temperature) as is customary, since, in my opinion, this will not constitute a true reading. The temperature measurements were based on the readings obtained directly from the surface of a dummy substrate making it more indicative of the actual temperature of the film, due to a faster sensor response time and a reduction in the temperature gradient between the sensor and the surface. This is especially necessary in the case of glass, rather than silicon, owing to their respective thermal conductivities.

Due to the high temperatures involved, there was an appreciable release of occluded gases from both the substrate and the surroundings (this will be discussed in much detail in the following chapters) which were mostly excluded after some patient pumping.
However, the results obtained from substrate heating were not so encouraging in that the orientation of all the films thus heated, as measured by (00.2) X-ray intensity count, was very poor. Figure (7.18) shows a T.E.M micrograph of a 250 angstrom film next to the S.E.M picture of a sputter etched (for better resolution), 2 micron thick film, both heated to 200 degrees C, in which a distinct lack of texture is evident.
Figure (7.18): (a) A T.E.M. micrograph with its corresponding diffraction pattern and, (b) an S.E.M micrograph of a 3 micron thick film, both of a film heated to 200 degrees C.
CHAPTER "8"

Analysis of films' gas contents
As was noted in a previous chapter, physical properties of thin films can be correlated with the environment in which they are grown. Partial pressures of various gases in the deposition chamber have been shown by some authors to affect nucleation and growth [79], resistivity [80, 81] and magnetic properties [82]. It was noted that this factor becomes especially acute in the case of sputter deposition where a film is grown in a relatively high pressure glow discharge environment such as sputtering environment. Oblas and Hoda [83] reported that as much as 12% argon may be present in dc sputtered tungsten films. Chemically active gases which are desorbed from the surrounding surfaces, as a result of sputtering chamber's temperature increase, itself resulting from thermal processes, may also be adsorbed at the film surface. It may therefore be a good practice to measure the gas contents of the films and, when possible, to relate the result to the particular deposition condition, such as bias sputtering. As was discussed earlier, one option in measuring the impurity contents of the films is to take advantage of R.B.S. technique.

To measure the gas contents of the films against negative substrate bias using this technique, several 2000 angstrom thick films at various bias values were sputtered
on Si substrates for examination. It was confirmed that
the gas content of the films goes through a minimum at
substrate bias values of around -150 V, table (2),
however the nature of the gases in the films could not be
determined with any reasonable accuracy, as the signals
associated with each element were either too small or
overwhelmed by that of their neighbouring elements. This
prompted the use of other possible techniques of measuring
gas contents.

The purpose of this section is to describe a new
method for measuring the impurity contents of CoCr films
against different sputtering parameters.

8.1 - Thermal evolution study of gas contents of film

Thermal evolution mass-spectroscopy T.E.S. (in order
not to be confused by T.E.M) has frequently been used to
investigate gas entrapment in films [84]. It is simply a
process by which the specimen is placed in a u.h.v envi­
ronment and the species of gases released as a result of
application of heat are analysed using a mass-spectrometer.
There are however two approaches to this technique. Either
the volume into which release proceeds contains effectively
no pumps for the gas of interest ("static" experiment), or,
alternatively, the gas is pumped with a large pumping speed
("dynamic"experiments) [85]. In the former case, provided
that the conductance between the release volume and the
Table (2). Variation of film to substrate yield with negative substrate bias, showing an increase followed by a decrease in value as the bias is increased. (Note that any contamination would result in a decrease in the yield ratio).

<table>
<thead>
<tr>
<th>Substrate Bias Voltage (V)</th>
<th>Metal-Substrate Yield (Ratio)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.49</td>
</tr>
<tr>
<td>-50</td>
<td>2.51</td>
</tr>
<tr>
<td>-100</td>
<td>2.67</td>
</tr>
<tr>
<td>-150</td>
<td>2.88</td>
</tr>
<tr>
<td>-200</td>
<td>2.69</td>
</tr>
</tbody>
</table>
detector is large, the rate of change of pressure in the system is proportional to the release rate of gas at the specimen's surface:

\[ \frac{dp}{dt} \propto \frac{dN}{dt} \]

In the latter case, provided that the pumping speed for the gas of interest is large so that the vacuum system time constant is small compared to the time constant associated with the release transient, i.e.

\[ \frac{\partial p}{\partial t} + \frac{p}{\tau} = \sigma / \nu \]

where \( p \) = pressure; \( t \) = time; \( \tau \) = characteristic pumping time; \( \sigma \) = evolution rate; \( \nu \) = chamber volume, then the pressure signal itself is proportional to the release rate:

\[ p \propto \frac{dN}{dt} \]

This is the technique used in the experiments reported here, as the characteristic pumping time of the chamber used is of the order of 0.2s. To facilitate our calculations, a linearly increasing temperature ramp was utilized.
8.2 - Experimental equipment and technique

The apparatus used here was developed by Donnelly et. al. [86], and is schematically represented in figure - (11.1). To achieve u.h.v the entire vacuum system including the cold trap is baked for minimum 10 h at 500 K. It has been shown that bakeout of upto 600 K does not significantly release the entrapped gas species [87].

Three micron thick CoCr films were sputtered on 0.1 mm thick molybdenum substrates. Five millimeter diameter discs, figure (8.3), were punched out of the substrates which were then used in the desorption experiment.

A four target carousel was where four different samples were placed for analysis, figure(8.4a). The targets were made of tungsten in the form of boats, figure (8.5) and were held in position using very fine tungsten wire (in order to minimize thermal conduction to the surrounding environment during the temperature ramp). To monitor the temperature, four separate thermocouples were used, each made of Pt:Pt/13%Rh which has a maximum operating temperature well in excess of melting point of CoCr. To obtain maximum accuracy, they thermocouples were positioned such that good contact with the targets was achieved.

An electron gun was employed to heat the samples during the temperature ramps, figure (8.4b). With the aid of a focusing tube, the beam area was reduced to 10 mm in diameter into which upto 300 W of power (3 KV at 100 mA
Figure (8.1). T.E.S. apparatus vacuum schematic. Dashed lines represent bakeable sections.
Figure (8.2): T.E.S. Equipment
Figure (8.3): CoCr sputtered on Molybdenum for T.E.S. experiments.
Figure (8.4). A schematic diagram of the T.E.S. apparatus.
Figure (8.6): A picture of T.E.S. sample holders and electron gun.
emission) was supplied.

Figure (8.7) is a schematic representation of the TES evolution cycle instrumentation.

8.3 - Mass spectrometer Calibration

In order to determine the fraction of the gas trapped, it is necessary to accurately calibrate the mass spectrometer for various gases. This is achieved by admitting gas into the vacuum system at a known rate via a previously calibrated SiC leak. The pressure behind the leak is measured directly by a capacitance bridge micromanometer and a mercury/oil manometer. Knowledge of the conductance of the leak and the gas pressure allow the calculation of the gas flow rate into the system. Using this technique, gas flow rates in the region of $10^{12} - 10^{16}$ atoms/s can be accurately produced [88]. The u.h.v gas handling plant used in these experiments is schematically depicted in figure (8.8).

with GV1-3, V1-4, UHV1-4 all open, and with UHV5, LV1-2 shut, the calibration plant is pumped to u.h.v. GV1, GV3 and V3&4 are then shut and by operating LV1 (mean while constantly monitoring either of the manometers, depending on the pressure of interest) known quantity of air is admitted into the system. Using LV2, the gas of interest (Argon and Nitrogen in this case) is now admitted into the
Figure (8.7). A schematic representation of the T.E.S evolution cycle.
Figure (8.8). Gas handling and calibration plant [84].
Figure (8.9): The gas handling plant.
u.h.v side of the plant, bearing in mind that V4 keeps the two sections of the plant isolated. The capacitance bridge micromanometer is now used to equalize the pressure of the argon (or nitrogen) admitted with that already known for the air in the glass manometers. In this way, the pressure of the gas is very accurately measured, before being admitted to the TES.

Figure (8.10, 8.11) shows that mass-spectrometer's signal is a linear function of the argon and nitrogen pressure.

8.4 - The evolution spectrums of argon and nitrogen

The average residence time $\bar{t}$ which an adatom spends on the surface is given by Frenkel equation

$$\bar{t} = \tau_0 \exp \left( \frac{E_d}{RT} \right)$$

where $\tau_0$ is the adatom's period of thermal vibration normal to the surface, and is of the order of Debye frequency $10^{-14}$ (10 s), $R$ is the gas constant and "$E_d$" is the binding energy of the trapping site, normally referred to as the activation energy of adsorption. If the equilibrium concentration of adatoms is "Na", the number of particles leaving the surface per unit time, using the above equation, is given by
Figure (8.10). Calibration graph of Ar pressure leaked into the T.E.S, against the mass-spectrometer's reading while it is being continuously pumped away.
Figure (8.11). Calibration graph of nitrogen pressure leaked into the T.E.S, against the mass-spectrometer's reading while it is being continuously pumped away.
\[
\frac{dN_a}{dt} = - \frac{N_a}{\tau} = \frac{N_a}{\tau_e} \exp \left( - \frac{E_d}{RT} \right)
\]

i.e.

\[- \frac{dN_a}{dt} = \left( \frac{N_a}{\tau_e} \right) \exp \left( - \frac{E_d}{RT} \right)\]

Since desorption rate depends exponentially on temperature, it is expected to increase drastically upon heating.

Figure (8.12) shows a typical evolution spectrum as a linear function of time for argon content one of the Co-Cr films, as observed by monitoring the signal intensity of the mass 40 on the mass-spectrometer. The minor desorption peak of around 400-500 degrees C is followed by a major peak around 600-700 degrees C. These peaks are attributed to the different trapping sites or "bands" with their own unique activation energy [89].

Figure (8.13) shows a typical desorption spectrum for nitrogen content of the films, when the mass 14 on the mass-spectrometer was monitored (monitoring mass 28 is impractical as ever-present hydrocarbons are liable to contribute significantly to the mass-spectrometer's output signal). Since nitrogen is abundantly present in the desorption environment (such as the boat, the substrate, etc.) even after long bakeout, we had to determine which portion of the spectrum was contributed by CoCr film alone. A desorption experiment was therefore performed on a molybdenum substrate on which no CoCr was deposited. It
Figure (8.12). A pen-recorder trace of Ar evolution as the temperature was being linearly ramped.
Figure (8.13). A pen-recorder trace of nitrogen evolution Vs. temperature.
can be seen from figure (8.13) that the initial two peaks are the result of nitrogen evolution by the boat and the substrate, and only the second peak is the result of nitrogen evolution by the film.

The areas under appropriate peaks were measured and the resulting values were converted to equivalent torr L/s of gas using respective calibration graph given in figures (8.11&12). These values were further converted to number of gas atoms released as follows:

The SiC leak conductivity $\sigma$ for helium is known to be $2 \times 10^{-6}$ torr L/s. Therefore the conductivity of the leak for other gases can be calculated taking into account the fact that the conductivity of any gas is proportional to $1/\sqrt{M}$, where $M$ is the atomic weight of the gas concerned. For argon therefore we have

$$\frac{\sigma(\text{He})}{\sigma(\text{Ar})} = \frac{\sqrt{M(\text{Ar})}}{M(\text{He})}$$

$$\sigma(\text{Ar}) = 2 \times 10^{-6} \sqrt[6]{\frac{1}{M(\text{Ar})}} \text{ torr L/s}$$

Ar density is given in the literature at one atmospheric pressure as 1.78 g/L, i.e 1.78/760 for one torr (760 is the number of torrs in one atmosphere), therefore the weight of one liter of gas at a pressure of one torr is given as

$$\sigma(\text{Ar}) = \left( \frac{6.3}{760} \right) \times 10^{-6} \times (1.78)$$
-9

= 1.475 \times 10^{-9} \text{ g/torr/liter}

To convert this into the number of released atoms, the above value is divided by 40 \times 1.66 \times 10^{-24} g, the weight of one argon atom. Note that the value 1.66 \times 10^{-24} is cancelled out when percentage of argon atoms to CoCr atoms are calculated.

The same procedure was followed when calculating the conversion factor for nitrogen and the value of 1.758 \times 10^{-9} g/torr/liter was obtained. For the number of atoms this is divided by 14 \times 1.66 \times 10^{-24}, the weight of one nitrogen atom.

To calculate the weight of a 3 micron thick CoCr film, on a disc 5 mm in diameter, the volume of the film was calculated and multiplied by the density of cobalt at 8.8 g/cm$^3$ alone for simplification (taking the atomic weight of Co alone is reasonable in view of the fact that over 80% of the film is cobalt and that the densities of cobalt and chromium are very similar, 7.2 g/cm$^3$ against 8.8 g/cm$^3$).

$$
(\pi) \times (0.25)^2 \times 3 \times 10^{-4} \times 8.8
$$

$$
= 5.18 \times 10^{-4} \text{ g}
$$

The number of CoCr atoms in the films was hence calculated (dividing by 59 \times 1.66 \times 10^{-24}). The ratio of the number
of atoms of the gas released over this value yields the percentage content of the gas concerned.
Before the results of the gas content measurements are presented, the numerous particle-solid interactions that are likely to take place when sputtering will be studied which may help to bring about an understanding of the physical processes dominant in sputtering in general -and reactive sputtering in particular- which aid impurity gas entrapment.

9.1 - Interaction between particles and solids

Gas-solid interactions can be split into three separate processes: collision, adsorption (with possible subsequent desorption), and diffusion [90]. The ability of certain reactants interacting with solid surfaces is given the general term of adsorption. The question arises as to what causes molecules to react with solid surfaces.

Any atom on the surface is surrounded by other atoms in the same plane or the planes below it to which it is attracted, figure (9.1), but there are none above it. There is thus a net force acting inwards and solids therefore possess a "surface energy" very similar, but much stronger than, the surface tension of a liquid. Hence, each surface atom has its valency requirement unsatisfied.

One form of adsorption occurs as a result of molecule interaction with these free valencies, and can
Figure (9.1). Pictorial representation of surface energy of a covalent solid.
involve so much exchange of energy that it can be regarded as chemical reaction. This type of adsorption is termed chemical adsorption or "chemisorption".

The other kind of adsorption that is recognized to occur at the surfaces of solids is due to Van Der Waals forces such as exists between molecules themselves in the liquid state. When these forces are exerted between an atom or a molecule and a surface, there is physical attraction, without chemical reaction. This is termed physical adsorption or "physisorption". Whereas chemisorption leads at most to one monolayer coverage of the available surface, with physical adsorption several adsorbed layers may be formed [91].

Figure (9.2) shows three common forms for the potential energy between a gas molecule and a solid [92]. Curve (I) gives the potential energy for physical adsorption; curve (II) gives the potential energy for chemical adsorption of a diatomic molecule which dissociates upon adsorption; curve (III) gives the potential energy for a dissociated molecule whose atom can penetrate the solid by diffusion. All the potentials are similar for large separations and a shallow minimum $E_p$ is present for all gas-solid combinations. However, at short separations the deep attractive minimum $E_c$ of chemisorption is different from the repulsive potential of physisorption. The surface plane of atoms presents an insuperable barrier to atoms.
Figure (9.2). Representation of potential energy between gas molecules and solid surfaces. (I) physisorption; (II) chemisorption and (III) diffusion [92].
which cannot diffuse into the solid. Which of these events will take place depends primarily upon which potential applies during the collision and upon the surface temperature.

An incoming molecule experiencing potential (I) may become trapped or physisorbed in the minimum $E_p$, [93]. After a certain time of stay on the surface the trapped molecule will desorb and return to the gas phase. An incoming molecule experiencing potential (II) will go through all the events of potential I, but in addition can cross the potential barrier between $E_p$ and $E_c$ and become chemisorbed and therefore trapped in the deep potential minimum $E_c$. After chemisorption atoms can penetrate into the solid by random-walk process or diffusion. This is the physical process by which impurities can be implanted in the growing film.

9.2 - Gas incorporation in the sputter deposition

The occurrence of adsorption in sputtering is aided in that whereas the endothermic adsorption sites of most metals are populated only by exposing their surface to an atomic gas, and thus being totally unaffected by exposure to the molecular gas [94], the same sites can be filled by particular mechanisms occurring in sputtering which produce free gas atoms at the surface, such as bombardment with molecules and molecular ions, or by decomposition of other
gases such as N2, NH3, CO2, etc. [95]. Apart from inducing such molecule-to-atom decompositions, gas particles with large kinetic energies (>75 eV) can themselves become trapped by embedding in the lattice [96].

Consequently, three sorption mechanisms are expected to make a major contribution to the gas incorporation into sputtered films when reactive sputtering: (1) direct chemisorption of the molecular gas, (2) the chemisorption of atomic gas at endothermic sites, and (3) the sorption of energetic ions or neutrals which are physically trapped or embedded in the lattice.

The gas-solid interaction thus far presented is made even more complicated by the heat flux of sputtering discharge and constant bombardment of the growing film with thermal electrons. The overall picture can therefore only be one of extremely complex thermodynamic and chemical interaction.

9.3 - Mechanism of argon entrapment

The flux of argon at a pressure of 10 m-torr on the substrate is 10 times higher than that of contaminants at 10 m-torr. Therefore there is inevitably some argon trapped in the growing film. Although argon, like all noble gases, is chemically inert, its atoms do become adsorbed on the surface of most metals, although at room
temperature the surface coverage is small [97].

The fraction $f_i$ of impurity of species $i$ trapped in a film is given by

$$f_i = \frac{\alpha_i N_i}{\alpha_i N_i + R}$$

where $N_i$ is the number of atoms of species $i$ bombarding unit area of film in a unit time during deposition, $\alpha_i$ is the effective sticking coefficient of the species $i$ during deposition, and $R$ is the deposition rate of the film. It has been shown [98] that for inert gases with thermal energies, $\alpha$ is zero. This is the result of - apart from the inertness of these gases - the fact that due to the high mobility of the adsorbed gases it is improbable for them to be trapped by the condensing film. Therefore, the amount of argon incorporated into the growing film should be very small. Winter and Key [99] proved this point by sputtering nickel film and, under similar conditions, evaporated an identical nickel film. They found that the argon content of their evaporated film was very small, less than 1% of that of sputtered film. Although for noble atoms chemisorption is discounted, there is a high probability of noble gas ions of significant kinetic energy, such as encountered in sputtering, being trapped when in collision with a surface [100]. However, it has also been argued that since the substrate is in the
relatively field free, negative glow region, there are very few mechanisms by which low energy argon ions could be given the minimum 100 eV required to penetrate into the substrate [99] unless they are a fast target argon neutrals and ions or thermal neutral argon atoms. Neutrals are liable to being trapped in the growing film as high energy argon ions, on impact with the target, are neutralized, and backscattered as fast neutrals towards the substrate [99].

Schwartz et. al. [102] found that during deposition, argon is partly removed from the surface by bombardment-induced re-emission and/or thermal release. The argon left is covered by adatom species and buried in the film.

9.4 - Argon content of CoCr versus sputtering parameters

9.4.1 - Ar content as a function of bias voltage

Figure (9.3) shows the argon concentration as a function of bias voltage. Here, the bias voltage is taken as an indication of the maximum energy with which Ar+ ions bombard the growing film. On this basis, the increase in the argon content of the films could be explained by simply relating the implantation rate of the argon into the growing film to the energy imparted to the argon ions by the negative substrate bias field. Hence, the higher the
Figure (9.3). Argon content of the films against negative substrate bias.
bias field, the greater the number of ions accelerated towards the substrate by the bias field. The small amount of argon in the films at zero bias is attributed to the high energy neutrals originating from the target, as there are very few argon ions in collision with the substrate at zero substrate bias [103].

Cuomo et al. [103] found a similar graph for the argon content of their Co-Gd-Mo, and derived the relationship

$$\% \text{ Ar} = K \frac{2}{\text{Ar} \text{Va}}$$

where $K$ is a constant and $\text{Va}$ is the sputtering voltage.

Winter and Key [105], however, measured for their dc sputtered Ni films a different functional dependence. They found a decrease in argon content between -50 to -100 V and attributed it to the fact that Ar+ ions could have a high probability of sputtering previously adsorbed argon, yet do not possess enough energy to be driven into the surface and themselves be trapped. This they verified by experiments [106] which indicated that Ar+ ions with energies between 30 and 100 eV cause appreciable sputtering of previously sorbed argon. At higher bias voltages, they argued, the Ar+ ions are of sufficient energy to be embedded.

No such a minimum in the concentration of argon was
observed for the series of CoCr films tested.

The work described above for argon illustrates that the simple generalization that bias sputtering will lead to the preferential resputtering of the previously sorbed gases is not necessarily true for all kinds of impurity gases. In the case of the CoCr films examined, it in fact led to an increase rather than a decrease in the impurity concentration.

9.4.2 - Ar content as a function of sputtering pressure

In order to expand on our data on the argon content of the films, further experiments where other sputtering parameters were altered were performed.

In figure (9.4) the argon concentration of the films against sputtering pressure in the case where no bias was applied is shown. It can be seen that raising the argon pressure actually reduces argon's tendency to accumulate in the coating. This graph is rather interesting, for it hints at the origin of the implanted neutrals such as argon as being the target. At higher pressures, due to numerous collisions, progressively fewer of the energetic argon atoms which are backscattered from the target arrive at the substrate with enough energy to drive into the surface having been reduced to mere low energy thermal neutrals.
Figure (9.4). Variation of Ar content of the films against argon sputtering pressure for unbiased films.
through their collisions with the sputtering gas molecules and ions.

Some authors [107, 108] believe that in bias sputtering the impurities preferentially sputtered from the films are still liable to return to the film surface by backscattering from the target and, therefore, suggest that bias sputtering may be more effective in reducing the film impurities at elevated sputtering pressures.

9.4.3 - Sputtering power dependence of CoCr's Ar content

The dependence of argon concentration on sputtering power for films biased at -100 V is shown in figure (9.5). This could easily be explained by arguing that the higher the sputtering power, the greater the energy of the backscattered neutrals, and therefore the higher the probability of their implantation into the growing film.

9.5 - Nitrogen concentration of bias sputtered CoCr films

The nitrogen concentration for films grown under negative substrate bias condition is shown in figure (9.6). A similar graph of nitrogen concentration in tungsten was obtained by Winter et. al. [107]. It is apparent that bias sputtering does have a beneficial effect in reducing the
Figure (9.5). Variation of argon content of the films biased at -100V against the sputtering power.
Figure (9.6). Variation of nitrogen content of a series of films reactively sputtered in an argon-nitrogen atmosphere.
nitrogen contents of the films. This fact will later be put into good use when trying to assess what values of base pressure can be reasonably tolerated when depositing CoCr films before adverse properties set in. Bias sputtering should help to raise this threshold.
CHAPTER "O"

Effect of addition of impurity gases in sputter deposition of CoCr
In addition to the common outgassing of the walls of the sputtering chamber and the target material, most commercially available substrates release finite amount of occluded impurity gases after evacuation through a degassing process, aggravated by the plasma heat flux, the combined effect of which is to contaminate the sputtering gas and the depositing film. Furthermore, there is a limit to the purity of sputtering gases commercially available, and these could therefore prove to be a source of contamination themselves.

An interesting example is given by Chapman [109] in which he has calculated that at a sputtering rate of 200 angstroms/s, the monolayer formation time to be only one 15 second (based on 10 atoms/cm/s, for a typical atom which is 3 angstroms in diameter). This rate corresponds to an equivalent gas pressure of 10⁻⁶ torr. Therefore, at a background pressure of only 10⁻⁶ torr, for every sputtered atom that arrives at the substrate, there will be one impurity atom arriving too. This amount of contamination could alone result from argon at a pressure of 20 m-torr which is 50 ppm pure. Hence even highly pure argon can introduce a considerable amount of impurities.

The impurities resulting from the surroundings could
be reduced if fast and efficient pumping, both before and during sputtering, of the chamber is effected. On a small, laboratory, scale the cost of pumping to high vacuums, as well as the time required for it, may not be of great concern, however, when considering a large scale industrial plant these aspects become vitally important if the final product is to be commercially viable.

Nevertheless, there is little that can be done about the possible presence of impurities arising from the deposition gas. One possible counter measure could be to selectively exclude the impurity species from being implanted into the growing film by, for example, utilizing bias-sputtering technique. However, this method has its limitations too. It was observed, in the previous chapter, that there was a limit in the reduction of nitrogen in the films at moderate bias voltages, as the increase in the argon content of the films when too high a substrate bias voltage was used, coupled to a corresponding collapse of orientation at elevated substrate bias, set a limit to the upper threshold of the value of the negative substrate bias permissible.

It may therefore be useful to know the extent to which common impurities can affect the quality of the films not withstanding any preventative measures that could be taken, such as biasing. In this way an estimation of tolerable quantities of each impurity gas may be made.
In the following experiments, the effect of common impurity gases such as N2, O2 and H2 on the magnetic and structural properties of CoCr is studied by intentionally adding controlled amounts of each individual contaminant to the sputtering chamber during sputtering, i.e by "reactive sputtering". Iwasaki et. al. [110] have already made a study of the effect of these gases on the properties of dc-magnetron sputtered CoCr films, and Coughlin [72] measured the effect of N2 on the quality of CoCr perpendicular recording media. Here, based on the data already presented in this work, it will be assumed that biasing will make a noticeable contribution in reducing the quantity of the particularly harmful species embedded in the films and, therefore, the role of those impurities on the magnetic and structural properties of the films in the presence of negative substrate bias will be studied.

10.1 - The effect of addition of nitrogen

The relationship between nitrogen concentration in sputtered films and partial pressure of nitrogen divides materials into three classes. The class 1 materials, such as W and Ta, are characterized by the fact that they have relatively high concentration of nitrogen for all values of partial pressures. Class 2 materials, such as Co and Cr, contain little nitrogen for small values of nitrogen partial pressure but they form nitrides at higher values, while the materials of class 3, Au and Ag, contain
relatively little nitrogen for all partial pressures [111].

Figure (10.1) shows the way in which presence of nitrogen destroys the (00.2) h.c.p. orientation above a partial pressure of about \(2 \times 10^{-6}\) torr. This also represents a minimum in Ms. As the nitrogen partial pressure is increased, the Cr which starts off at around 16 at% in the films sputtered in a relatively nitrogen free atmosphere, goes down to values below 14 at%. Ms could be expected to rise because of greater at% cobalt and not necessarily because of greater chromium segregation. The chromium decrease could be due to an increase in positive ion density from the additional nitrogen atoms preferentially re-sputtering chromium atoms in the negative bias field.

In figure (10.2) \(H_k\) reduces rapidly with increase in nitrogen content. At the same time an in-plane orientation is developed as indicated by increase in in-plane remanence ratio. There is a corresponding decrease in perpendicular remanence ratio and an expected decrease in perpendicular coercivity. Figure 10.2.5 is a TEM diffraction picture of a 300Å thick CoCr film deposited in an atmosphere containing as little as \(4 \times 10^{-5}\) torr partial pressure of nitrogen, in which the reflections of the h.c.p. texture are not as clear as those of a typical well-oriented film presented before.
Figure (10.1). The influence of nitrogen on the in-plane and perpendicular orientations, $M_s$ and Cr content of CoCr films, biased at -100 V.
Figure (10.2). The effect of nitrogen on the magnetic properties of CoCr biased at -100V.
Figure (10.2.5): T.E.M. micrograph and its corresponding diffraction pattern of a CoCr film reactively-sputtered in an atmosphere of nitrogen.
10.2 - The effect of addition of Oxygen

There are two processes by which oxygen can be incorporated in the films. The column boundaries are relatively open structures and can allow Cr, which surrounds the column, to getter oxygen without creating appreciable lattice distortion [112]. Worse still, it can take part in a process of "selective oxidation" of column boundaries, thus removing Cr from solution by formation of anti-ferromagnetic \( \text{Cr}_2\text{O}_3 \) and affecting the structure of the films [113, 114]. Yet, negative substrate bias can suppress the oxygen incorporation into the sputter-deposited films [115].

The effect of controlled amounts of oxygen on the orientation of bias-sputtered films is shown in figure (10.3). It is evident that the effect of oxygen on the perpendicular orientation of the films is quite severe. The disappearance of h.c.p. (00.2) orientation is rapidly followed by an enhancement of the (10.0) in-plane orientation. This in-plane component is itself destroyed as the oxygen partial pressure is further increased.

In figure (10.4), the disappearance of perpendicular coercivity and remanence is followed by the appearance of a strong , in-plane remanence. 
Figure (10.2.6): T.E.M micrograph and diffraction pattern of a 300 angstrom thick CoCr film reactively sputtered in an atmosphere of argon and 20 micro-torr of oxygen.
Figure (10.3). The influence of oxygen on the perpendicular and in-plane orientations of CoCr films, biased at -100 V.
Figure (10.4). The influence of oxygen on the magnetic properties of CoCr biased at -100V.
It is known that hydrogen [117, 118, 119] forms true solid solutions with large number of metals, table (3), including Co and Cr, but with a solubility of less than 1%. There is, however, very little data concerning the effect of H2 on the properties of CoCr films. Iwasaki et. al. [110] found that the presence of as much as 1 in 10000 partial pressure of hydrogen in the chamber did not drastically effect the magnetic properties of CoCr films.

The presence of small quantities of hydrogen actually brought about an improvement in the orientation of the films as is shown in figure(10.5). The results show a sharp peak for (00.2) orientation coupled with a decrease in the in-plane value. For this pumpdown, the initial values are lower because the quantity of impurity gases of N2 and O2 present in the chamber exceed that of H2 at such low partial pressures, but the effect of hydrogen is still to compensate for the additional impurity gases of N2 and O2 and produce optimum conditions at $10^{-6}$ torr partial pressure of hydrogen.

One possible explanation for this improvement is that hydrogen, itself easily evacuated by the diffusion
Table (3). The ability of Cr and Co to chemisorb common impurity gases, present in sputtering environment.
Figure (10.5) shows the influence of hydrogen on in-plane and perpendicular orientations for a bias voltage of -100V.
pump, could help the removal of other residual gases by reducing their mean free paths through collision. Alternatively it could combine with difficult gases like oxygen and nitrogen to form more manageable compounds.

Figure (10.6) shows the magnetic characteristics of films sputtered in hydrogen-argon plasma. The perpendicular coercivity and remanence-ratio peak at about the same region as the (00.2) orientation of figure (10.5). The in-plane remanence rises sharply as hydrogen partial pressure is increased. Iwasaki et. al. [110] have indicated that the precise role of hydrogen is not known because there was no evidence of hydrogen inclusion in their films. The mechanism by which hydrogen controls film behaviour is yet to be discovered.

The T.E.M diffraction when the hydrogen partial pressure was $4 \times 10^{-5}$ is presented in figure (10.6.5).

10.4 - X-ray diffraction intensity

A general summary of the effects of various gases and substrate bias on the crystallographic orientation of 0.5 micron thick films can be seen from figure (10.7). It is noted that films with no bias result in no intensity peaks. It was found that the f.c.c. phase is predominant at $6 \times 10^{-5}$ torr nitrogen partial pressure. Hydrogen and
Figure (10.6). The influence of hydrogen on magnetic properties of CoCr films, biased at -100V.
Figure (10.6.5): T.E.M. micrograph and its corresponding diffraction pattern of a CoCr film reactively-sputtered in an atmosphere of hydrogen.
Figure (10.7): X-ray diffraction intensities for a series of CoCr films sputtered under various conditions.
Oxygen behave in a similar manner with strong (10.0) and (10.1) orientation and a much reduced (00.2) orientation. f.c.c. phase was not apparent for this partial pressure for H2 and O2. The most significant peak is in the (00.2) orientation under -100V bias.
CHAPTER II

Application of Magneto-optic effect to CoCr films
In this section the three magneto-optic effects will be briefly outlined, and a semi-mathematical description of the effect will be given.

11.1 - Magneto-optical Effects

It was observed in the latter part of the last century that after a plane polarized beam of light has split into a transmitted and a reflected portion upon incidence on a ferro-magnetic body, on magnetization of the body in a direction parallel to the beam, the beams become elliptically polarized with their major axes rotated with respect to the plane of polarization of the incident beam. When referring to the transmitted beam, this phenomenon is called the "Faraday Effect"; it is known as the magneto-optic "Kerr Effect" when it refers to the reflected beam. The magnitude of the Kerr rotation is normally much smaller than Faraday rotation.

Three different kinds of Kerr effect can exist depending on the relative orientation of the sample magnetization with respect to the optical geometry, figure (11.1).

11.2 - Longitudinal effect

Longitudinal Kerr effect manifests itself in the following manner. A polished ferromagnetic surface is
Figure (11.1). Kerr effect configurations, a) Polar, b) Longitudinal and c) Transverse. The magnetization vector is represented by $M$, while the plane of incidence is shown dotted.
magnetized in a direction that is both parallel to the plane of incidence and to the surface of the material. If light that is plane polarized with its electric vector in the plane of incidence (i.e., "p" polarized) is reflected obliquely from the magnetized surface, there will appear a small "s" component in addition to the reflected "p" component. Therefore a rotation is introduced, which is zero at normal incidence, reaching maximum for angles of around 60 degrees.

Since the longitudinal Kerr rotation is minute for Co [120] and therefore hard to detect, this is the only effect which will not be explored in detail here and the transverse Kerr effect is used instead, in the following experiments for characterization of the in-plane magnetic properties of CoCr films.

11.3 - Polar Kerr effect

The polar Kerr effect is the only one of the three effects which is non-zero at normal incidence. A rotation is obtained in a similar way to the longitudinal case, i.e. a small, out of phase, "s" component is added to the "p" polarized reflected light, resulting in ellipticity and, hence, rotation. In this mode, the magnetization M must necessarily have a component normal to the reflecting surface and, therefore, only materials with high uniaxial anisotropy can exhibit such effect. Also, owing to the
large demagnetizing fields in ferromagnetic thin films, large applied fields are required before noticeable effects can take place. Yet this is the largest of the three Kerr effects.

11.4 - Transverse Kerr effect

In the transverse Kerr effect, however, there is no rotation of the plane of polarization. It is observed merely as a result of a change in the reflection coefficient of a ferro-magnetic surface, for an obliquely incident light which is "p" polarized. This effect can also be measured as an effective rotation and is of the same magnitude as the longitudinal effect [121].

\[ \delta = \frac{\rho_p(+) - \rho_p(-)}{\rho_p^0} \quad \text{(11.1)} \]

here \( \rho_p(+) - \rho_p(-) \) reflection coefficient for "p" polarized light and \( \rho_p^0 \) is the intensity reflection coefficient for a light reflected from the demagnetized specimen. The magnitude of \( \delta \) is dependent on \( \lambda \), the wavelength of the incident beam and also on the angle of incidence of the light, reaching a maximum around 85 degrees from normal [122].
11.5 - Macroscopic description of Kerr effect

Here, an outline of a phenomenological discussion of magneto-optical phenomena as given by Sokolov [123] will be presented. For a comprehensive description of this phenomenon the reader is referred to text books on the subject [124, 125].

The macroscopic description is based on general differential equations for the electromagnetic field

\[
- \frac{1}{c} \frac{\partial B}{\partial t} = \text{Curl} \ E
\]

\[
\frac{1}{c} \frac{\partial D}{\partial t} = \text{Curl} \ H
\]  \hspace{1cm} (11.2)

and the tensor equation

\[
D = \xi E
\]

\[
B = \mu H
\]  \hspace{1cm} (11.3)

where \( \xi \) is the permittivity tensor of the magnetized ferromagnetic metal. This tensor takes the same form as the permittivity tensor used in crystal optics, except that here the components of the tensor are complex (due to the medium being absorbing), and their imaginary parts are negative. Consider now the usual Fresnel reflection formulae
Also the generalised reflection formulae which allows for the effect of magnetisation

\[
\begin{pmatrix}
R_s \\
R_p
\end{pmatrix}
= 
\begin{pmatrix}
r_{11} & 0 \\
0 & r_{22}
\end{pmatrix}
\begin{pmatrix}
A_s \\
P_p
\end{pmatrix}
\quad \text{----- (11.4)}
\]

\[
\begin{pmatrix}
r_{11} & r_{12} \\
r_{21} & r_{22}
\end{pmatrix}
\begin{pmatrix}
A_s \\
P_p
\end{pmatrix}
\quad \text{----- (11.5)}
\]

Comparison of equations (11.4) and (11.5) shows directly that it is only the non-diagonal components of the reflection matrix which are functions of magnetization. In the absence of magnetization \(r_{21}\) and \(r_{12}\) vanish and the generalised reflection formulae (11.5) becomes identical with the Fresnel formulae (11.4). Similarly, in the permittivity tensor

\[
\xi' = \begin{pmatrix}
\xi' & -i \psi \xi' & 0 \\
& i \xi' \psi & \xi' & 0 \\
& 0 & 0 & \xi'_o
\end{pmatrix}
\quad \text{----- (11.6)}
\]

where

\[
\xi = \xi - i \frac{4 \pi \sigma}{\omega}
\]
is the complex permittivity and $\psi$ is the magneto-optical parameter, and $\sigma$ and $\omega$ are the conductivity and angular frequency of the vibration respectively. When the magnetization is zero, the non-diagonal elements of the tensor vanish and the diagonal elements become identical ($\xi' = \xi_o$), that is, the body becomes optically isotropic.

The diagonal components of the permittivity tensor of a ferromagnet $\xi'_{xx} = \xi'_{yy} = \xi'$ are functions of the magneto-optic parameter $\psi$. Experiments show [126, 127] that this parameter is small ($<< 1$) and therefore the function $\xi'(\psi)$ may be expanded into a series of increasing powers of $\psi$. Taking the most significant terms

$$\xi'(\psi) = \xi_o + \left( \frac{\partial \xi'}{\partial \psi} \right)_{\psi=0} \psi + \frac{1}{2} \left( \frac{\partial^2 \xi'}{\partial \psi^2} \right)_{\psi=0} \psi^2 \quad (11.7)$$

On reversal of the direction of magnetization the above expression becomes

$$\xi(-\psi) = \xi_o - \left( \frac{\partial \xi'}{\partial \psi} \right)_{\psi=0} \psi + \frac{1}{2} \left( \frac{\partial^2 \xi'}{\partial \psi^2} \right)_{\psi=0} \psi^2 \quad (11.8)$$

Further more, $\xi'(\psi) = \xi'(-\psi)$, since the physical properties of a body must be invariant under magnetization reversal. Comparison of equations (11.7) and (11.8) yields
\[ \xi'(\psi) = \xi_0' + \frac{1}{2} \left( \frac{\partial^2 \xi'}{\partial \psi^2} \right) \psi^2 \]  
\[ \text{---- (11.9)} \]

A vector \( \xi' \) can be defined such that

\[ \frac{1}{2} \left( \frac{\partial^2 \xi'}{\partial \psi^2} \right)_{\psi=0} = \xi_0' \xi_0 \xi' \]  
\[ \text{---- (11.10)} \]

and hence

\[ \xi = \xi_0 + \xi \xi' \psi^2 \]  
\[ \text{---- (11.11)} \]

i.e.

\[ \xi' = \xi_0' (1 + \xi \psi^2) \]  
\[ \text{---- (11.12)} \]

On substituting (11.12) into (11.6), we obtain

\[ \xi' = \xi_0 \begin{pmatrix} \left(1 + \xi \psi^2 \right) & i(\psi + \xi \psi^3) & 0 \\ i(\psi + \xi \psi^3) & (1 + \xi \psi^2) & 0 \\ 0 & 0 & 1 \end{pmatrix} \]  
\[ \text{---- (11.13)} \]

It will be assumed that the medium is not only electrically
but also magnetically anisotropic and that it has magnetic permeability tensor of the form

\[
\mu = \begin{pmatrix}
\mu & -i \mu \mathcal{J} & 0 \\
\mu \mathcal{J} & \mu & 0 \\
0 & 0 & \mu_0
\end{pmatrix}
\]

\[\text{---- (11.14)}\]

where \( \mathcal{J} \) is the second magneto-optical parameter, which is like \( \psi \) a function of magnetization \( M \) and permeability \( \mu \)

\[
\mu = \mu' - i \mu''
\]

\[\text{---- (11.15)}\]

Following the same procedure as permittivity, we obtain

\[
\mu(\mathcal{J}) = \mu_0(1 + \zeta_\mathcal{J} \mathcal{J}^2)
\]

\[\text{---- (11.16)}\]

Substituting (11.16) and (11.14) we obtain

\[
\mu = \mu_0 \begin{pmatrix}
(1 + \zeta_\mathcal{J} \mathcal{J}^2) & i(\mathcal{J} + \zeta_\mathcal{J} \mathcal{J}^3) & 0 \\
i(\mathcal{J} + \zeta_\mathcal{J} \mathcal{J}^3) & (1 + \zeta_\mathcal{J} \mathcal{J}^2) & 0 \\
0 & 0 & 1
\end{pmatrix}
\]

\[\text{---- (11.17)}\]
On substitution of equations (11.3) and (11.17) into (11.2-11.3), we are left with a set of simultaneous equations whose solutions, subject to boundary conditions, allow the magneto-optical scattering coefficients of equ. (11.5) to be expressed in terms of the phenomenological constants, \( \psi \), and \( \vartheta \).

An alternative description using the conductivity tensor is given by Bennet and Stern [128].

11.6 - Physical origins of magneto-optical effects

Early attempts to explain the above phenomena on a microscopic theory of matter consisted in using the Becquerel formula for the difference of the indices of refraction for right and left circularly polarized light under the action of an external magnetic field [129], where he had considered a system of harmonically bound electrons in an oscillating electric field and with a static magnetic field along the z axis, and derived the polarizability

\[
\chi = \frac{Ne^2}{\varepsilon_0 m} \left\{ \omega_0^2 - [\omega + \omega_L]^2 + i \omega \gamma \right\}
\]

where \( \omega_L = e\mu_B H / 2m \) is the Larmor precession frequency, \( \gamma \) the damping factor associated to electron motion, and \( m \) is the electron mass.
The above is related to the dielectric factor by

\[ \chi_\pm = \varepsilon_0 (\varepsilon_\pm - 1) \frac{4 \pi}{\varepsilon_0} \]  \hspace{1cm} (11.19)

the upper sign referring to right circularly polarized light (RCP), and the lower to left circularly polarized light (LCP) respectively. If the index of refraction in the absence of applied field is \( n(\omega) \), then the indices of refraction in the presence of the field are

\[ n_+(\omega) - n_-(\omega) = 2 \omega \frac{dn}{d\omega} \]

In the above treatment it was assumed that in ferromagnetics there is an effective magnetic field active of considerably higher order of magnitude than that of external field (10^7 Oersteds). To explain this, Hulme [130] introduced into the picture the spin-orbit interaction, which is a phenomenon associated with quantum mechanics. This is the energy of interaction of the magnetic moment of an electron, \( \mu \), with the magnetic field it "sees" as it moves through the electric field, \( \nabla V \) inside the medium with momentum, \( \rho \), and it has the form.

\[ \sim \mu \times \nabla V \cdot \rho \]
Such an interaction provides a relation between the motion \((\rho)\) and the magnetic moment \((\mu)\) of the electron (bearing in mind that it is the motion of the charges that is of importance here, since this motion gives rise to the electric current and thus affects the optical properties of the specimen). Therefore, spin-orbit interaction could be responsible for the optical and the ferromagnetic properties that Kerr effect indicates. In so doing, he postulated that the spin-orbit interaction can be thought of as an effective magnetic field, of vector potential, on the motion of the electrons.

One of the interesting points that comes out of Hulme's calculations is that the Kerr rotation vanishes unless there is a difference between the extinction coefficients of right- and left-circularly polarized light, that is, the medium must necessarily be absorptive as was noted before.
CHAPTER 12

Magneto-optical signal detection
Because of the finite amount of signal available in magneto-optical detection systems, optimization of the signal-to-noise ratio becomes essential if accurate and meaningful magnetic information is to be obtained.

In this section the nature of the various kinds of noise inherent in magneto-optic detection systems will be discussed so that efficient counter measures in the form of a noise reduction technique can be taken.

12.1 - The polar Kerr magneto-optical signal

The magnetic information in the polar Kerr magneto-optical effect is transferred to the reflected light beam essentially as a change in the direction of polarization. Let the difference of this direction for the two states of polarization be denoted by the Kerr angle $\phi$. If an analyzer is introduced in the path of the reflected beam, then the change in transmitted power for light reflected from two oppositely magnetized areas will be

$$\Delta W = W_0 [ \sin^2 (\theta + \phi) - \sin^2 \theta ]$$

where $\theta$ is the angle of the rotation of the analyzer from
its position of minimum tranmittance and $W_o$ is the light power reflected by the magnetic medium. The angle $\phi$ is very small (a few minutes of a degree) [130.6]. Normally $\theta \gg \phi$, therefore

$$\Delta W = W_0 \phi \sin 2\theta \quad (12.2)$$

and the average light power reaching the photodiode will be

$$W = W_0 \sin^2 \theta \quad (12.3)$$

### 12.2 - Noise

Having found the magnitude of the signal that one expects, it is now appropriate to deal with the various noise sources inherent in such a system. The three major ones are: shot noise in the photodetector due to quantized nature of light; noise due to depolarization as a result of surface imperfections; light level fluctuations due to arc instability, plasma noise in the gas laser and minute fluctuations in the laser's supply [131].

#### 1. Shot Noise

The shot noise current $I_n$ in photodetectors [132] is given by;
\[ I_n = (2e f P W_0)^{\frac{1}{2}} \sin \theta \] (12.4)

and the signal current, \( I_s \), is given by

\[ I_s = P W_0 \Phi \sin 2\theta \] (12.5)

where \( e = 1.6 \times 10^{-9} \), \( C \) is the electron charge, \( f \) is the bandwidth, and \( P \) is the conversion coefficient of the photodetector, a typical value of which is \( 3 \times 10^{-2} \) A/W.

From these two equations one finds the signal-to-noise ratio

\[ \frac{I_s}{I_n} = (2 P W_0 / e f)^{\frac{1}{2}} \Phi \cos \theta \] (12.6)

It is noted that this ratio is essentially independent of \( \theta \), the analyser angle, in the whole region of interest, which rarely will exceed 10 degrees, for reasons which will be obvious later. This ratio will increase with the light power, \( W_0 \); hence the superiority of a laser to a conventional light source. Also the laser beam has the advantage of being a well-collimated which is valuable when complicated optical paths are to be tackled.

2) Surface depolarization of the light

Observation of the film surface shows surface imperfections and mound-like features. Suppose that these
features have a dimension, d, of a few tenth of a micron; they scatter light roughly as if they were pinholes in the path of plane wave, and thus the diffracted light originating spreads with a cone angle, $\beta$, of the order of $\lambda/d$ where $\lambda$ is the wavelength of light. Although the incident light is polarized, the light diffracted by the imperfections is essentially depolarized, hence there will be a certain depolarized component in the light reflected of the surface.

Since the fraction of the depolarized light is independent of the analyzer angle, $\theta$, and the signal is proportional to $\sin 2\theta$, equ.(12.2), therefore by increasing $\theta$, one will increase the signal-to-noise ratio due to surface imperfections.

3) Laser Noise

On the other hand, as $\theta$ increases, the contrast decreases, and the effect of light level fluctuations become more and more pronounced. Following equ.(12.3), this fluctuation is proportional to $\sin^2 \theta$; thus the ratio of signal to this kind of noise is proportional to $\cot \theta$ and decreases with increasing $\theta$. The relative size of these two noise sources will determine the optimum angle $\theta$ for minimum total noise. This is the limiting factor that sets the size of $\theta$ to around 10 degrees, as was mentioned earlier.
12.3 - noise reduction techniques

Figure (12.1) shows the schematic diagram of the differential and the modulation schemes used to reduce the output noise of the polar Kerr magneto-optical system [130.5].

a) Differential scheme

This scheme exploits the fact that the laser noise and the signal are carried in a different way by the beam of light; the noise is essentially an amplitude modulation, while the signal is the modulation of the direction of polarization of the light. Because of this difference, the beam can be split into two information channels in which the noise will appear in common mode while the signal appears in opposite polarity so that the useful information can be added and the noise cancelled out by feeding the two channels to a differential amplifier.

There are two different optical paths that can be adopted in polar Kerr measurements where polarization modulation is concerned. One is to split the beam into a reference and a signal channel before the beam strikes the sample, and the other is to split the signal channel alone, i.e., after the beam strikes the sample. The former benefits from the advantage that the same arrangement has
to be used for transverse Kerr effect, and is therefore useful when a combined transverse-polar Kerr effect system is needed where the experimenter can obtain both measurements with minimum alterations to the instrument at the expense of some decrease in S/N ratio. However, the latter approach is more useful when polar Kerr detection alone is of interest, where a superior polar Kerr S/N ratio is obtained. This latter technique will be described below.

As explained above, the beam reflected by the Co-Cr film is amplitude split by a non-polarizing, non-absorbing beam splitter into two beams identical in every respect. Two analyzers are used for the two beams, set at angles $+\theta$ and $-\theta$ to the minimum transmittance position, respectively. This setting is made for the medium magnetized in "zero" state. The light is fed to two photodetectors and to a differential amplifier. Hence all the fluctuations that are common to both detectors (such as laser intensity fluctuations, sample depolarization) will be cancelled out. If the medium is now magnetized in one direction, one analyser will make an angle $\theta + \phi$ and the other $-\theta + \phi$ with the minimum transmittance position, and the difference in the light power that the two detectors will receive is given by Equ. (12.2). In this way one has complete freedom of choice in $\theta$.

Note that any intensity modulation of the beam by the sample be it stray or genuine signal in this polariza-
tion modulation mode will not present itself as a change in the output, as any such a change would be common to both detectors. Hence, this differential scheme, although ideally suited to polar Kerr signal detection which manifests itself through polarization modulation of the light, cannot be exploited in transverse Kerr signal detection.

**b) Modulation as a noise reduction technique**

The differential scheme, although very effective in reducing the laser noise, etc., does little to alleviate the problems caused by other kinds of noise, for instance, shot noise. This class of noise is more efficiently handled by the modulation techniques in the form of phase-sensitive detectors (PSD), commonly known as lock-in amplifiers. Therefore, a Lock-in amplifier was constructed and was placed at the output stage of the differential amplifier. The light beam was chopped at a frequency of 375 Hz, and a reference voltage derived from the same light was fed to the lock-in amplifier.

A lock-in amplifier shares the same property common to most ac indicating instruments in that it provides a dc output proportional to ac signal under investigation. The traditional rectifier, however, makes no distinction between signal and noise and produces an error voltage due to rectified noise components.
The noise elements in the input signal to the PSD is removed when it is multiplied by a reference voltage of a frequency chosen to make the noise signal intentionally incoherent.

In figure (12.2), the signal and the reference are expressed in terms of their r.m.s. values Vs and Vr

\[
\frac{V_o}{p} = V_s V_r \cos \left( (\omega_s + \omega_r)t + \phi_s + \phi_r \right) + V_s V_r \cos \left( (\omega_s - \omega_r)t + \phi_s - \phi_r \right) \tag{12.6}
\]

Given that the low-pass filter cuts off at frequencies much less than \( \omega_r \), the sum-frequency component is effectively eliminated from the output. Hence, the noise in a lock-in signal is not rectified but appears in the output as an ac fluctuation. This means that the desired dc response can be separated from the noise output, figure (12.3). The mean output is therefore not affected by the presence of noise in the case of a lock-in. The reference voltage therefore makes sure that the detection process is "locked" to the signal of interest and will track the frequency variation in the signal source automatically.

12.4 - Optimization of transverse Kerr effect signal

The problems associated with transverse Kerr effect are, however, more acute than polar effect in that this effect exhibits itself as a change in the reflection
Figure (12.1). A schematic diagram of the electronic detection system used for the Kerr loop plotter.

Figure (12.2). A mathematical representation of the multiplication of the input signal to a PSD with a sine wave reference frequency.
Figure (12.3). The detection process in a lock-in amplifier. a) The signal and reference 90 degrees out of phase hence the O/P is zero. b) here the phase difference is 180 degrees and the O/P is negative (when it is zero, the O/P is positive). c) The effect of spurious frequency component, still averaging to zero.
coefficient of the ferromagnetic surface and is, therefore, intrinsically independent of polarimetric measurements described before. More over, this fractional change in the intensity is very small (of the order of 1 in 10000), making the case for a high value of S/N ratio even stronger. This intensity modulating effect can lead to the undesirable laser fluctuation making its presence noticed on the signal in the following manner.

For the differential system to operate, with no magnetic field applied, the detectors' pre-amplifiers' gains (or for that matter the intensity of the beam to each detector) are adjusted until the output of the differential amplifier gives a zero reading. Then, on application of a magnetic field to the sample this balance is disturbed since the amplitude of the laser is modulated. (as one of the detectors will now receive more light). However, since this signal appearing on one of the detectors is necessarily not compensated by the other detector, the amplitude of the modulation, originating from a noisy beam, will appear noisy, although to much lesser extent than if a differential scheme were not used at all, as fluctuations on the unmodulated portion of the beams are still cancelled; Hence the differential scheme's limited use in eliminating noise such as laser fluctuations in transverse effect signal detection. Therefore, owing to this light-intensity dependence of the signal amplitude, the S/N ratio is particularly prone to laser fluctuations.
This factor helps to reduce the S/N ratio of the transverse Kerr detection to below that of its polar counterpart, figure (12.4). The in-plane hysteresis loop was therefore essentially noisy. Using a signal averaging method by going round the hysteresis loop several times, the S/N ratio was considerably improved. A hysteresis loop could then be plotted, from which coercivity values were obtained.

Owing to the particular sensitivity of signal detection technique used in transverse Kerr effect to laser intensity fluctuations, the use of ordinary filament lamps, being inherently less liable to fluctuate, assuming a stable power source, is probably more advantageous. However, collimating this kind of filament light source to small diameters and maintaining the collimation over adequate optical paths requires sophisticated optical apparatus and is not readily achieved, a problem from which laser beams do not suffer.

12.5 Experimental set-up of the magneto-optic system.

Figure (12.5) shows a schematic diagram of the magneto-optic system for both polar and transverse Kerr effects. A picture of the same set up presented in figure (12.6). A 3mW He-Ne laser (6328 angstroms) was used as the light source. Having passed through a 1:1 mark-space ratio
Figure (12.4). Typical perpendicular and in-plane magneto-optic loops.
Figure (12.5). A schematic diagram of the combined polar and transverse Kerr magneto-optic loop plotter.
Figure (12.6): The combined transverse-polar magneto-optic Kerr system.

Figure (12.9): The improved polar Kerr loop plotter.
chopper, the beams was then split into two beams of equal amplitudes by a non-polarizing beam splitter. The polarizers P3 and P4 serve to adjust the amplitude of the reference beams, with that of P1 setting the plane of polarization. The polarizer P1 was intentionally placed after the beam-splitter to minimize unavoidable depolarization of the light beam by the beam splitter.

The electro-magnet supplies a perpendicular field of up to 10 KOe in the critical region of the disc. The pole shaping for perpendicular fields was arranged to achieve maximum uniformity of field, figure (12.7a). The angle for measurements of the perpendicular properties was chosen at 4 degrees (2 degrees from normal), for which the polar Kerr effect hardly decreases [133].

The removal of the polarizer P2, centre pole C and the reversal of the current in one of the coils supplying the side poles A or B changes the magnetic field from polar to transverse Kerr effect. In this arrangement the fringe fields, figure (12.7b), were used to magnetize the film transverse to the plane of incidence of the laser. For the transverse mode, angle of incidence of the laser was set at 80 degrees (40 degrees from normal). To maximize the transverse gyro-electric effect (which is a bigger effect than transverse gyro-magnetic effect) [124], the light beam was p-polarized in the in-plane mode.

The whole apparatus was mounted on an anti-
Figure (12.7). (a) Schematic illustration of the pole pieces and the magnetic field lines for the perpendicular case and; (b) that of transverse case which exploits the fringe fields to magnetize the film.
vibration platform in the form of marble slab resting on an air cushion.

The detectors were of photo-voltaic kind which are inherently less noisy. The size of their active area was chosen to be 50 mm square - much larger than the diameter of the laser beam - in order to ensure that any physical vibration of the beam does not translate into noise. At a later stage in the experiments when only polar Kerr results were of interest, a new arrangement for the magnetic field and the optical lay out was used. It was hoped that by using this new set up the magnetic field could be made more homogeneous in the region occupied by the sample. Figure (12.8) shows this new set up where a bore was made in one of the centre pole of an electromagnet to allow the passage of the laser beam at an angle of 2 degrees from perpendicular. This was found to result in even more uniform magnetic field for the polar Kerr effect. Further more, the beam splitter was replaced by a mirror (gold coated for maximum efficiency) and Kerr rotation detection was effected using the lay out depicted in the same figure. The uniformity of the field was then checked by applying a small AC voltage to the magnet, and measuring the voltage induced in a loop 0.1 square mm in diameter when inserted in the region where the sample was placed. This technique yields a better resolution than using commercially available Hall probes, which at best resolve areas no smaller than 1 square mm.
Figure (12.8). Reconstructed polar Kerr loop plotter. The magnetic field is homogeneous over a much larger space. Note that any noise originating before the beam-splitter will be common to both detectors, and will therefore be cancelled out.
This new kind of differential scheme, where the beam splitter was placed after the beam hit the sample, as was explained earlier, resulted in an improved S/N ratio.

One of the analysers is set at an angle of +5 degrees from extinction, and the other analyser is set at -5 degrees. The detectors, D1 and D2, monitor the beam intensity, the output of which is fed to the differential amplifier eliminating most of the common mode fluctuations of the light intensity. With this polarizer setting, when no field is applied to the film, the differential amplifier gives a zero reading. The change in the amplifier's reading, on application of magnetic field to the sample, is then registered and plotted against the magnetic field.

12.6 - Magneto-optical measurement on CoCr/NiFe sputtered on an optically polished aluminium disk

Figure(12.10) shows the perpendicular hysteresis loop obtained from a CoCr sample, sputtered on NiFe, using a V.S.M. next to that obtained using the magneto-optical technique. The NiFe itself was sputtered on glass substrates. The V.S.M. results for double layer films give only an average assessment of both films together, whereas for magnetic recording applications it is necessary to know the properties of surface CoCr layer only. This demonstrates the advantage of the magneto-optical technique.
Figure (12.10) illustrates the way the V.S.M perpendicular signal becomes swamped by that of NiFe underlayer, whereas its M-O loop is unaffected.
Figures (12.11) and (12.12) illustrate the variation of perpendicular coercivity, in-plane coercivity and the saturation magnetization of the double layer Winchester disk both tangentially and radially. The uniformity in the deposition profile as offered by hexagonal arrangement of Cr pieces with comparatively small Cr spacing to target-substrate distance ratio, has indeed yielded a uniform $M_s$. The coercivity which is, as was shown before, the function of both the thickness of the film and Cr concentration also shows a good consistency throughout the surface of the disk.
Figure (12.11). The tangential variation of $H_c$ and $M_s$. 
Figure (12.12). The radial variation of $H_c$ and $M_s$. 
measurements of bias sputtered CoCr films

13.1 - Coercivity versus film thickness

Owing to the small skin depth of He-Ne laser beam (around 150 angstroms [133.5]), the magneto-optic technique provides a good medium for the measurement of surface magnetic properties, whereas the corresponding V.S.M measurements would yield the volume magnetic properties as a whole.

In figure (13.1) the variation of the surface coercivity, as measured by a magneto-optic instrument and volume coercivity measured using a VSM both against the film thickness is shown. That these measurements should be different at all could imply that there is a coercivity gradient through the film thickness. However, the squareness of the perpendicular loop would discount any such gradient. It is therefore reasonable to conclude that the origin of this discrepancy should lie at the surface of the CoCr film alone. A knowledge of the various possible coercivity lowering mechanisms in CoCr film may help to elucidate the cause of this anomalous behaviour.

The presence of an initial transition layer of a thickness of 1000-1500 angstroms in RF sputtered CoCr film
Figure (13.1). Variation of volume and surface $H_c$ as measured by V.S.M. and M-O loop plotter respectively, against the film thickness.
is noticeable on fracture cross-section. The columnar microstructure grows out of the initial layer that appears to be amorphous, but in actual fact it is polycrystalline, as the electron diffraction result of our 300 ångstrom films, and thicker films [77], shows. Furthermore, the presence of an abrupt change or anomalous jump in the in-plane loops as observed by some authors [134, 135] has been attributed to this transition layer. Wouri and Judy [136] reported that the transition layer in RF-sputtered CoCr films is magnetically soft and reverses by domain wall motion.

This layer could then simulate a soft magnetic underlayer which could bring about flux closure in the CoCr layer at the substrate interface. This flux closure could result in the reduced coercivity of the back layer, lowering the volume coercivity compared to that of the surface for films ranging in thickness from 300 to 4000 ångstroms.

At a critical thickness of around 4500 ångstroms the volume coercivity begins to exceed that of the surface coercivity. This deviation from homogeneous magnetization is thought to be due to the occurrence of reversed closure domains or "spikes" [137] at the surface of the films which become energetically favourable above certain film thickness. These spikes, figure (13.2), are the result of the phenomenon of minimization of total energy which is the
Figure (13.2) - (a), (b) and (c) show the occurrence of closure domains in thin magnetic films with perpendicular anisotropy as the film thickness increases.
sum of anisotropy field, wall, stray field and closure 
energies and were first observed by Takata [138] in a 
single crystal of cobalt.

13.2 - The effect of negative bias voltage variation

Figure (13.3) shows the variation of the surface coercivity and remanence, measured by magneto-optic technique, as a function of bias field. The same measurements carried out using a VSM are shown along side. The Cr content of these films was 16%, although Cr segregation as bias voltage increases will mean that a smaller fraction of the Cr will take part in the dilution of the saturation magnetization. At a critical voltage of around -100 V a reversal in the magnitude of the coercivity occurs. This is probably as a result of onset of processes which promote closure domains on the surface of the films. One such an occurrence could be the collapse of the columnar structure, possibly leading to a reduction in grain orientation (in the way indicated by (00.2) X-ray intensity measurements presented before) leading to a reduction in the magneto-crystalline uniaxial anisotropy. This weakening of the anisotropy could facilitate the formation of closure domains [137]. The other could simply be the extent of Cr dilution which, as was mentioned previously, could effect both the magnetic and microstructural properties of the films. It will be, therefore, interesting to observe the effect of Cr content of the
Figure (13.3). Variation of surface and volume coercivities against negative substrate bias.
films on the surface coercivity in the next section.

It is noted that there is a slight difference in the bias voltage where the two coercivities peak. This is again a possible effect of volume and surface inhomogeneities.

13.3 - Cr content dependence of surface magnetic properties

The variation of surface coercivity and remanence against Cr concentration for the case of -120 V bias is shown in figure(13.4), with the VSM measurements presented again for comparison. This graph shows that the occurrence of reversal is indeed Cr content dependent. This is a good representation of the way in which the magnetization is not uniform throughout the film. Where the increase of Cr content above 17% was seen in a previous chapter to lead to degradation of perpendicular orientation of the columns, and so to a reduction in volume coercivity and remanence (VSM measurements), the same magnetic parameters for the surface continue to increase.

13.4 - Comparison between the surface and the substrate interface properties of CoCr films

We have already seen that polar Kerr effect can be used effectively to obtain hysteresis loops of the surface
Figure (13.4). Comparison between the surface and the volume magnetic properties of CoCr, as measured by M-0 and V.S.M., respectively, against Cr content, for films biased at -120 V.
of the films. Application of the same technique to obtain hysteresis loops of the film-substrate interface is more complicated in that, even if the film can be physically separated from its substrate, it will inevitably lose its flatness, leading to a scattering and divergence of the laser beam, however much effort one makes to ensure that the film is adhered flatly on its supporting base. Furthermore, it is not at all evident that the film will retain its unique magnetic characteristics after such a separation.

However, these measurements can be extended to the film-substrate interface without a need for separating the films if a transparent substrate, such as glass, is utilized. The problem that will then arise is that the glass will introduce a Faraday rotation of its own to the polarized laser beam and therefore, has to be subtracted from the final signal to obtain the Kerr rotation due to the magnetic film alone. To reduce this extraneous Faraday rotation, CoCr films were sputtered on thin glass cover-slips, as the rotation here is proportional to the medium thickness.

13.5 - Measurement of saturation magnetisation of the film-glass interface

Figure (13.5) shows the dependence of Kerr rotation,
Figure (13.5). Variation of polar Kerr rotation angle, against Cr content for CoCr films. The variation of Ms is also shown for comparison.
\[ \phi_k \text{, and the saturation magnetization on the Cr content of} \]
\[ \text{the films. Here, } \phi_k \text{ was measured by placing a home built} \]
\[ \text{Faraday rotator, figure(13.6), between the reflected beam} \]
\[ \text{from the sample and the detector.} \]

**13.6 - The Faraday rotator**

A Faraday rotator, figure(13.6), works on the basis that when a plane-polarized light is sent through glass in a direction parallel to the applied magnetic field, the plane of vibration is rotated. The amount of rotation observed for any given substance is proportional to the field strength \( B \) and the distance the light travels through the medium. The rotation can be expressed by relation

\[ \theta = V_B \ell \]

where \( B \) is the magnetic induction in tesla, \( \ell \) is the length in meters, and \( \theta \) is the angle of rotation in minutes of arc, and \( V \) is the Verdet constant of the material, defined as the rotation per unit path per unit field strength. The material used was a 0.05 m long piece of Skan SF 57 glass with a Verdet constant of 69 min/Oe/m. If a solenoid is used to generate the magnetic field, then the above relationship can be expressed
Fig. 13.6  Faraday Rotator
\[ \theta = \mu I N V \ell \]

i.e. a linear relationship between the current in the coil and the rotation. The number of turns on the solenoid was 1000.

The application of a magnetic field to the sample will result in a Kerr rotation. The Faraday rotator is then employed to effect an equal and opposite rotation (this is done by increasing the current to the solenoid until a null point, or zero signal, is reached). The rotation thus registered will give the value of the Kerr rotation.

13.7 - Comparison between saturation magnetizations of the surface and the substrate-film interface

From the figure (13.5) it can be concluded that the polar Kerr rotation angle of CoCr films is an alternative method to the saturation magnetization measurements. The rotation signal can hence be used to compare the saturation magnetization of the films.

Figure (13.7) shows three kinds of rotation obtained from bias-sputtered CoCr on glass cover-slips. The film-glass interface rotation represented in (a) is the polar Kerr rotation of the film plus a small, negative, Faraday rotation contribution due to the glass substrate.
Figure (13.7). (a) The magneto-optic rotation against the applied field for the glass-film interface; (b) the polar Kerr rotation of the film surface and (c) the Faraday rotation of the glass cover-slip.
shown in (c). Despite this subtraction even, the Kerr rotation from the back of the film is still much greater than that of the front surface of the film represented by (b), hence the saturation magnetization of the film-glass interface is much greater than that of the surface, so much so that even a field of 10 K Oe has difficulty saturating the film, as depicted by (a). It should be pointed out that the rather noisy loops of (a) & (c) are due to the depolarization of the light as it goes through the glass. Here, the Faraday loop (c) was obtained when a thin layer of gold was evaporated on the cover-slip which would act as a reflector without having any magnetic rotation of its own. A similar difference in rotation was obtained for unbiased films. The coercivity of all the back layers was found to be smaller than 200 Oe.

To ascertain whether the difference between the front and the back of the film is due to the change in the Cr content of the two respective layers, small film flakes which had been separated from their substrates were analysed for their Cr content using X-ray microanalysis. Table (4) shows that there is no appreciable difference between the Cr content of the front surface and the back layer of the films each sputtered under different conditions. The difference in Ms between the surface and the back of these film can therefore only be attributed to the chromium segregation i.e. the lack of proper alloying of Co and Cr. Segregation around the column boundaries for
Table (4). Variation between the front and the glass-interface Cr content of CoCr for biased and unbiased films. (Measurements made by X-ray micro-analysis method)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Bias (V)</th>
<th>Front At.% Cr</th>
<th>Back At.% Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>CoCr</td>
<td>-100</td>
<td>16.23</td>
<td>16.80</td>
</tr>
<tr>
<td>CoCr</td>
<td>0</td>
<td>16.54</td>
<td>16.70</td>
</tr>
</tbody>
</table>
the glass-film interface is discounted as no columns could have yet developed in this initial layer. Besides, the process of segregation takes place through diffusion of the adatoms on the surface of the growing film, and this necessitates the provision of adequate thermal energy. It is doubtful that what is practically a cold substrate is able to supply this energy. It is more likely that in the initial stages of film growth, the adatoms, on arrival on the substrate, condense instantly, preventing the CoCr alloy from mixing adequately. It is only at later stage of film growth when the deposition environment has acquired sufficient thermal energy that segregation becomes a likely process.

This was confirmed when two films were sputtered with identical sputtering parameters except that one of the substrates was heated to 230 degrees Centigrade during sputtering, the heated substrate yielded the biggest difference between the front and the back layer, indicating that heating the substrates does indeed lead to Cr segregation, although not necessarily around the column boundaries.
CHAPTER 14

Optimization of the underlayer and the recording experiment
It was mentioned in the introduction that to reduce the magnetic reluctance of the recording-head's air gap, a composite anisotropy recording medium was invented [139]. In this way a marked decrease in the saturation recorded current was obtained after a 0.5\textsubscript{Ni-Fe} was r.f sputtered as an underlayer for Co-Cr films [140]. In the remanent state too, it is argued, the Ni-Fe, with its in-plane anisotropy, acts to increase the magnetization by decreasing the demagnetizing field through rotating the magnetization vector from the perpendicular to the plane of the film, hence helping to close the flux lines.

A Ni\textsubscript{77}Fe\textsubscript{21} alloy target was used for these experiments. At this composition a high permeability of the underlayer is expected since the magnetostriction and the crystal anisotropy constants are small, figure (14.1) [85]. A low value of \(K\) decreases domain-wall energy, and inclusions then become less effective hindrances to wall motion. A low value of \(\lambda\) means that microstress similarly becomes less effective.

Sputtered Ni-Fe possesses an f.c.c crystal structure with (111) preferred orientation [140.5]. This orientation is a favourable starting base for the subsequent Co-Cr base because it allows epitaxial growth.
Figure (14.1) (a) Variation of saturation magnetostriiction and (b) permeability of Ni-Fe with Ni content [78].
In fact evidence of heteroepitaxial growth of Co-Cr on permalloy was obtained by Uesaka [143] when he determined that the smallest atomic spacing in the f.c.c (111) plane of permalloy is only 0.02 angstrom less than that of the h.c.p (00.2) plane of Co-Cr.

14.1 - Results and discussion

Ni-Fe at a rate of 260 angstrom/min. was bias-sputtered on glass and silicon substrates in a unidirectional magnetic field of 400 Oe. Figure (14.2) shows the variation of the coercivity versus the sputtering bias for films sputtered on glass. A maximum coercivity of 800 mOe is obtained in the easy direction when bias sputtering at 80 V. In view of the fact that comparatively high values of coercivity are recommended as a means of reducing the underlayer's noise contribution [144, 145], this bias value was adopted for all our subsequent Ni-Fe sputterings.

It should be pointed out that this method of sputtering yields anisotropic magnetic parameters for the underlayer, whereas for a recording disc those parameters should be at least axially symmetric, if not totally isotropic. This problem is not undertaken in this work. One suggestion to achieve magnetic isotropy is balancing the magnetic field induced anisotropy with the angle of incidence anisotropy induced as the disc is made to rotate while Ni-Fe is being deposited [146].

-221-
Figure (14.2). Variation of coercivity (in the easy axes) of NiFe, against negative substrate bias.
In any magnetic recording system which employs a magnetic medium and magnetic head, the ability to record as well as replay is, in the first place, conditional on placing the recording head in very close proximity to, if not in contact with, the medium. This poses little problem for the kind of media which are on flexible substrates, as the head would then be able to make actual physical contact with the disc without any detrimental consequences. However, such an intimate contact for hard discs would lead to certain head-medium crash, especially when considering that discs are likely to rotate at speeds of up to 450 inches per second and beyond. A compromise solution for hard discs is to fly the head a few micro-inches over the medium. This would then set very stringent limits to the surface roughness of the disc, the medium and the head itself.

Figure (14.3) shows the extremely low surface roughness of the polished soda-lime glass discs used. This data was obtained using a "Tally Surface" equipment with a 0.5 micro-meter stylus.

The Winchester discs were fabricated in a class 1000 particles-per-cubic-feet clean room, since even the smallest dust particle embedded in the film would increase the risk of head crash. Considerable care was taken to
Figure (14.3). Surface roughness of glass discs as measured by a Tally-Surface with a 0.5 micron stylus.
ensure that no physical damage was inflicted on their surface during deposition process.

A 200 angstrom thick layer of SiO2 was sputtered on the discs which, in addition to lubricating the film, acts to protect the CoCr layer from atmospheric corrosion.

Figure (14.4) shows a Winchester disc comprising of bias sputtered CoCr (0.5 micron)/NiFe (0.5 micron) sputtered on a soda-lime glass substrate. Figure (14.5) the frequency response of the disc when a thin film head with a longitudinal field was used for recording and play back, as no suitable thin film perpendicular head is yet available. It was hoped that there would still be sufficient perpendicular field to effect the required flux reversals. The fall in the reproduced signal at high frequencies is attributed to this choice of head.

In figure (14.6) the variation of the reproduced signal against the radius of the disc when the recording frequency was 2.5 MHZ is shown. The reduction of the signal at reduced radii is not due to medium inhomogeneities, but more the result of effective reduction in the head-medium velocity.
Figure (14.4): The Winchester disc, with CoCr/NiFe media sputtered on a glass disc.
Figure (14.5). The output Vs. bit density (in K-flux change/inch) for Co-Cr/Ni-Fe medium using a thin-film head with a longitudinal field.
Figure (14.6). Variation of head output against the radius of the disc.
Conclusion

1) Since the start of this project in 1985, the interest in CoCr as a medium with perpendicular anisotropy has declined in many countries but many problems associated with the fabrication of a perpendicular head are now being overcome, and prototypes of working high density recording systems using Co-Cr Winchester discs and thin film heads have been on display in many exhibitions.

2) For my part, CoCr films with good perpendicular anisotropy were successfully deposited. The perpendicular anisotropy, as indicated by $H_k$, showed a maximum around 16 at.% Cr, with a corresponding minimum in in-plane remanence and perpendicular coercivity.

3) Bias sputtering led to a marked improvement in the perpendicular orientation of the films around -100V bias field. The magnetic properties as measured by perpendicular remanence and coercivity showed an improvement, with welcome detrimental effects on the in-plane parameters. The columnar morphology of the films, as depicted by the S.E.M. photographs of the sputter-etched films, showed an improvement at around that bias voltage too.

4) There are three schools of thought regarding the possible cause of the above improvement; a) supply of
additional thermal energy to the growing film, aiding diffusion of newly arrived adatoms on the surface of the growing film leading to a better crystal structure, b) increasing the re-emission coefficient and thereby removing atoms which are awkwardly positioned in the lattice, and c) preferentially removing contaminants, in particular gas impurities, from the growing film leading to a reduction in the gas contents of the films. Of these, (a) could not be proved as no noticeable temperature increase when bias sputtering could be measured, even though the temperature sensor was of a very low thermal mass and that it was placed directly on the surface of the substrate, therefore facing the glow discharge. Also (b) could not be experimentally proved, except that there was indeed a small difference in the sputtering rate of biased and unbiased films, indicating that there was some re-emission factor involved, but whether this is the cause of improvement in the quality of the films when bias sputtering cannot easily be proved. It leaves (c) as the most likely candidate for the observed improvements. Indeed there was a noticeable reduction in the harmful species of nitrogen as measured by T.E.S technique. Although this was followed by a corresponding increase in the argon contents of the films, argon, being an inert gas, is not thought to participate in the crystallographic structure of the growing film, as nitrogen does.
5) By intentionally introducing gas impurities of oxygen, nitrogen and hydrogen into the chamber, it was learned that oxygen, even in smallest quantities, had a drastic effect on the crystal structure and magnetic properties of the films. Nitrogen proved to be just as harmful for the perpendicular anisotropy and the orientation of the films when introduced in the deposition environment. However, there appears to be an improvement when small amounts of hydrogen is incorporated in the sputtering environment. This is possibly the result of hydrogen combining with more harmful species, such as oxygen and nitrogen, and forming more manageable compounds.

6) Substrate heating did not, in my case, prove beneficial, probably due to an increase in the amount of desorption of occluded gases in the substrate surroundings. Using silicon as a substrate, too, did not yield the magnetic and structural benefits that a conducting, crystalline, substrate is claimed to. This has also been observed by some other authors.

7) A polar Kerr effect system with a $45 \text{dB} \ S/N$ ratio, for measuring magnetic properties of CoCr films sputtered on NiFe underlayer was constructed. This instrument was further used to successfully compare the volume, surface and the back layer perpendicular magnetic properties of CoCr films. When used in the transverse mode, however, the $S/N$ ratio of the instrument was not too impressive,
although it was sufficient for approximate values of in-plane coercivities to be extracted.

8) Regarding the actual final product, the surface of the CoCr/NiFe films was of sufficient smoothness for a head to fly at a distance of 10 micro inches quite successfully. The surface texture of the glass disc was obviously equal in merit. The actual density of the recording experiment was not as high as one hoped, probably due to the tentative use of longitudinal head. This remains to be investigated by a series of recording experiments.
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PUBLICATIONS
The Influence of Nitrogen and Other Gases on the Magnetic Properties of Bias sputtered Co-Cr Perpendicular Films for Computer Memory Applications
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Abstract
This paper investigates the effect of negative bias on sputtered CoCr layers for hard-disc memory applications. Negative bias can significantly improve the C-axis orientation and the perpendicular magnetic properties. Oxygen and Nitrogen in particular degrade the desired properties but Hydrogen can have a beneficial effect at low concentrations.

Introduction
Following the interest in Co-Cr thin films as suitable media for perpendicular recording, results are presented showing the influence of impurity gases on films produced by R-F sputtering under negative bias conditions. This follows recent work by Iwanski et al. who emphasised the importance of impurity gases, especially nitrogen, in sputtering films on polyamide flexible base films. Nitrogen has previously been found to be detrimental by Coughlan et al. and its presence is given as one of the main reasons for the need to sputter from low base pressures. The role of Oxygen in hard-substrate films has been reported by Thompson and Stevenson who found a peak in in-plane orientation with oxygen and a sharp decline in (00.2) intensity above a concentration of about 10 at% atoms/cc.

The use of substrate negative bias voltage in magnetic film production is interesting because it may provide good perpendicular orientation films from higher base pressures as may be used in commercial sputtering equipment. The paper by Tamashita et al. for instance, shows a peak in (00.2) orientation at a bias in the region of -80 volts. This leads to the present work where we investigate how negative bias voltage affects the properties of CoCr films and report the effect of nitrogen, oxygen and hydrogen under negative bias conditions.

Experimental Results and Discussion

Figure 1 shows the variation in (00.2) orientation with substrate negative bias voltage. This reaches a sharp peak at about -80V. Over this range, the overall chromium content stays substantially constant at 16 at%. but the saturation magnetisation Hs falls in a similar way to that reported by Tamashita et al. This has been attributed to a temperature-dependent segregation of Cr at column boundaries when there is no bias, implying that biasing leads to a more uniform distribution of Co and Cr atoms. A significant temperature change for our films, however, was not detected so it is assumed that this effect is limited to the first few atomic layers.

If biasing does indeed lead to a temperature increase, it could cause the desorption of impurity gases, thereby improving the quality of the films. It has been shown that intense ion bombardment in bias-sputtering can suppress the development of inter-columnar, voids affecting the quality of entrapped gases. Biasing could also result in the preferential re-sputtering of the impurity atoms from the substrate, thus reducing the number of defects and stacking faults caused by these impurities.

Sputtering power was maintained at 0.8 kW giving a deposition rate 250 A/min in all cases to produce films 0.5 μm thick at an argon plasma pressure of 5 microns. Argon pressure was adjusted when impurity bases were introduced for the plasma pressure to total 5 microns. All gases used were high quality grade (99.99%). The target was 8º dia cobalt on which 1 cm pieces of Cr were hexagonally arranged to obtain a uniform composition. Utilisation of a large target also leads to uniform composition and thickness.

Film composition and magnetic properties were measured using X-ray microanalysis and vibrating sample magnetometer respectively. The deposition temperature was measured with a low thermal mass thermocouple on the surface of a glass substrate. This gave a direct measurement of surface temperature.

Figure 2. Some in-plane and perpendicular magnetic properties versus negative substrate bias voltages.
To understand at which stage of the columnar growth biasing is more important, a simple experiment was carried out in which the bias was switched on at various stages of the film growth. It was learned that biasing was more effective when applied in the initial stages. Figure 2 shows some corresponding magnetic properties for the films of figure 1. The results show, within graphical error, perpendicular coercivity and remanence ratio reaching maxima with orientation at the same time as the in-plane values are minima. The peaks are slightly broader and shifted slightly indicating that orientation is not the dominant and only factor influencing coercivity. To take account of this a best bias voltage value of -100V was chosen for the impurity gas experiments.

Figure 3 shows how the presence of nitrogen destroys the h.c.p. orientation above a partial pressure of about 1x10^-6 Torr. This also represents a minimum in M. The Cr was sputtered from a constant area source of 21% but this translated into an At% reduction as nitrogen partial pressure increased. H should be expected to rise because of greater At% cobalt and not necessarily because of greater chromium segregation. This Cr decrease could be due to an increase in positive ion density from the additional nitrogen atoms leading to preferential re-sputtering of chromium atoms in the negative bias field.

In Figure 4, H reduces rapidly with increase in nitrogen content. At the same time an in-plane orientation is developed as indicated by the increase in in-plane remanence ratio. There is a corresponding decrease in perpendicular remanence ratio and an expected decrease in perpendicular coercivity.

The effect of controlled amounts of oxygen on the orientation of bias-sputtered films is shown in Fig. 5. Although oxygen did not yield as much f.c.c. phase as nitrogen, its effect on the perpendicular orientation of the films was severe. The results tend to be in agreement with those of Thompson et al which show (in the X-ray intensity results) a peak in the in-plane orientation with increasing oxygen content.

In Figure 6, for nitrogen, the disappearance of perpendicular coercivity and remanence is followed by the appearance of a strong, in-plane remanence. Hydrogen improves the perpendicular orientation of the films when introduced in small quantities (Figure 7). The results show a sharp peak for (00.2) orientation coupled with a decrease in the in-plane value.
For this pumpdown, the initial values are lower because of the impurity gases of \( N_2 \) and \( O_2 \), introduced to the chamber exceed that of \( H_2 \) at such low partial pressures, but the effect of hydrogen is still to compensate for the additional impurity gases of \( N_2 \) and \( O_2 \) and produce optimum conditions at \( 10^{-6} \) Torr partial pressure of hydrogen.

One possible explanation is that hydrogen, itself easily removed by the diffusion pump, could help the removal of other residual gases by reducing their mean free paths through collision. Alternatively, it could combine with difficult gases like oxygen and nitrogen, forming manage able compounds.

At higher partial pressures, hydrogen caused improvement in the in-plane component of film orientation in a similar way to nitrogen and oxygen.

Figure 8 shows the magnetic characteristics of films sputtered in hydrogen-argon plasma. The perpendicular coercivity and remanence-ratio peak at about the same region as the (00.2) orientation in figure 7. The in-plane remanence risis sharply as hydrogen partial pressure is increased. Iwaseki et al. have indicated that the precise role of hydrogen is not known because there was no evidence of hydrogen inclusion in their films. The mechanism by which hydrogen controls film behaviour is yet to be discovered.

A general summary of the effects of various gases and substrate bias can be seen from figure 9. It is noted that films with no bias result in no intensity peaks. It was found that the f.c.c. phase is predominant at \( 8 \times 10^{-3} \) Torr nitrogen partial pressure. Hydrogen and oxygen, behave in a similar manner with strong (10.0) and (10.1) orientation and a much reduced (00.2) orientation. f.c.c. phase was not apparent for this partial pressure for \( N_2 \) and \( O_2 \). The most significant peak is in the (00.2) orientation under 100V negative bias.

Conclusions

Negative substrate bias can have a substantial beneficial effect on chrome-cobalt films for perpendicular recording. A bias of about -200V corresponds with a sharp peak in perpendicular orientation and a broad peak in perpendicular magnetic properties is seen around -100V. Nitrogen and Oxygen degrade the films but low concentrations of hydrogen can improve the properties, possibly as a result of combining with other impurities to produce more manageable compounds.

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Abstract

This paper describes a Kerr magneto-optic effect apparatus for measuring in-plane and perpendicular magnetic properties of Co-Cr coated computer discs. A combined differential amplifier, phase-sensitive detector technique was used to achieve a signal-to-noise ratio of 44 dB in the perpendicular system. Comparisons are made with v.s.m. measurements and results presented for 5.25" Winchester discs.

Introduction

Kerr-Magneto-optic systems have been suggested as a good substitute for conventional magnetometers when requiring H-N loops from a Co-Cr perpendicular recording medium. This can provide a direct magnetic field measurement of H-X-sputtered Co-Cr on an Ni-Pd underlayer without the Ni-Pd overwhelming the magnetic information from the Co-Cr. It has also the potential of being used as a non-destructive method for assessing the magnetic uniformity of thin-film coated floppy or Winchester-type computer discs. In addition, it is capable of measuring the magnetic properties of small areas of the medium, against the laser intensity and laser intensity fluctuation; a direct correlation with magnetic and optical parameters can be achieved. Film thickness was measured using a Talley-step.

A more complete analysis of properties is possible if in-plane as well as perpendicular magnetic properties are measured and this is possible if both polar and transverse Kerr-effect is used.

A method of obtaining the transverse and Kerr loops within the same apparatus is described in this paper.

Experimental Details

Initially, it was considered that driving the thin-film disc with an a.c. field would produce accurate results. This approach, however, suffers from induced eddy-currents in the aluminium leading to difficulty in calibration of the field and presenting the possibility of a phase-shift in the Kerr-effect leading to an inaccurate coercivity measurement. In addition, there is a phase-shift in the Kerr-effect due to the magneto-optic interaction. These problems were eliminated by using a near d.c. field but detection with d.c. is difficult because of sensor temperature effects, mechanical vibration and laser intensity fluctuation. For this reason it was decided to use a combined differential/phase-locked loop method for the detection system.

Thin-film Preparation

43/17 at% Co-Cr films, 0.5 μm thick were deposited on to 81/19 at% Ni-Pd films, 0.5 μm thick, on Aluminium discs with optically finished surfaces. The planar plane of Co-Cr was hexagonally arranged on an α* disc to give the maximum uniformity in the desired composition. A bias of -80 volts was applied to the substrates to improve the perpendicularly orientation of the Co-Cr (3), and the films were deposited at RF power and Ar pressure of 800 watts and 3 mtorr respectively, giving a deposition rate of approximately 250 Å/m. The composition of the films was analysed by electro-probe X-ray micro analysis, and the film thickness was measured using a Talley-step.

Films of the same Co-Cr but without an underlayer were also deposited for measurement by V.S.M.

Figure 1. The magneto-optic system.

Magnetic-Optic System

Figure 1 shows a schematic diagram of the magneto-optic system. A 3 mW He-Ne laser (λ = 6328Å) was used as our light source. Having passed through a 1 l/mk mark-space ratio chopper, the beam was then split into two beams of equal amplitudes by a non-polarizing beam splitter. The polarizers P1 and P2 serve to adjust the amplitude of the reference beams, whereas that of P1 and P2 detect any rotation of the plane of polarization in the polar case, and a means of adjusting the intensity of the beam in the in-plane mode. The electro magnet supplies a perpendicular field of up to 10 K Oe in the critical region of the disc. The polarizer P1 was intentionally placed after the beam-splitter to minimize depolarization of the light beam.

The pole shaping for perpendicular fields was arranged to achieve maximum uniformity of field (Figure 2). The removal of the central pole C and the reversal of the current in one of the coils supplying the side poles A or B changes the apparatus from polar to transverse Kerr-effect. In this arrangement the fringe fields were used to magnetize the film transverse to the plane of incidence of the laser. The field was calibrated and conforms in amplitude and spatial distribution to the Kariquist approximation (1). In this mode the laser angle of incidence was at 65°. To maximize the gyro-electric effect, the light beam was P-polarized for in-plane mode. The angle for measurement of perpendicular properties was chosen as 5°.
The Electronic Detection System

A large effort was made to optimize S/N ratio. Figure 3 illustrates the electronic detection system.

In order to reduce the laser noise a 'differential method' was employed in which the transmitted and the reflected beams are detected separately and the noises are cancelled electrically at a differential D.C. amplifier. However, this method alone is not effective enough for obtaining sufficient S/N ratio due to the ubiquitous presence of shot noise which is not cancelled by differential method. Therefore a modulation technique was utilised to efficiently reduce the shot noises. The phase sensitive detector (p.s.d.) measures the difference voltage of interest by using a synchronous reference voltage derived from the input modulator. Detection with respect to synchronous reference enables the use of very long averaging times for the purpose of S/N ratio improvement. Apart from the incidental pick-up and interference, the noises level due to thermal noise alone was several millivolts peak-to-peak while our signal of interest had an amplitude measured in microvolts making the employment of p.s.d. necessary. When the signal output from the differential amplifier and the reference signal of Figure 3 is precisely in phase, they change polarity in symmetrical fashion, and so bring about full-wave rectification of the signal at the switch output. The output of the switch is then applied to the low-pass filter which smooths out the ripple component and delivers a.d.c. voltage which is proportional to the amplitude of the signal. Using this method a final S/N ratio of 45 dB was achieved.

The reference signal was phase locked to the chopper which ran at 375 Hz.

Since this method requires a large time constant, the magnets were driven at a low frequency of 0.2 Hz.

Experimental Results

Several samples of Co-Cr films were measured using vibrating sample magnetometer and Kerr techniques. The results, shown in figures 4 and 5 indicate close correspondence between the measurement methods for films of Co-Cr only (single layer) but a divergence for double layer films as expected. The V.S.M. results for double layer films give only an average assessment of both films together, whereas for magnetic recording applications it is necessary to know the properties of surface Co-Cr layer only, hence demonstrating the advantage of this magneto-optic technique.
The uniformity in the deposition profile as offered by Hexagonal arrangement of Cr pieces with comparatively small Cr spacing to target substrate distance ratio, has indeed yielded a uniform $H_s$. The coercivity which is a function of both the thickness of the films and Cr concentration also shows a good consistency throughout the surface of the disc.

**Conclusions**

A combined transverse/perpendicular Kerr-effect system has been developed for analyzing the magnetic properties of CoCr on double-layer discs. The signal-to-noise ratio is 45 dB and the system has been used successfully to investigate the variation in magnetic properties over the surface of sputtered discs.

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ABSTRACT

INVESTIGATION INTO PERPENDICULAR ANISOTROPY COBALT-CHROMIUM FILMS WITH APPLICATION TO WINCHESTER COMPUTER DISCS

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RF-sputtered CoCr films have been characterized for a variety of deposition conditions, and the most effective parameters which bring about perpendicular anisotropy have been evaluated. In particular bias sputtering has been noted for its role in improving the crystallographic orientation and magnetic properties when applied to the substrate at certain values, hence the root mechanism of the observed improvement has been, as far as possible, investigated. These investigations have, in particular, focused on the extent to which impurity gases are likely to effect the magnetic properties of sputtered films, as well as quantifying the gas contents of the films using as yet unexploited (in this area) method of thermal desorption experiments.

In a further attempt to quantify tolerable level of impurity species in the sputtering environment, impurity gases of nitrogen, oxygen and hydrogen, which are commonly present even in high vacuum systems, were intentionally introduced in the sputtering chamber and their effects on both magnetic and crystallographic properties of CoCr were noted.

To measure the perpendicular and in-plane magnetic properties of CoCr, a combined polar-transverse Kerr magneto-optic system was constructed, through which direct magnetic measurement of CoCr/NiFe becomes feasible. This method was further exploited to compare volume and surface magnetic properties of CoCr, as measured using a V.S.M and this M-O system.

Finally, a CoCr/NiFe Winchester disc was fabricated on which a recording experiment was successfully performed.