**FERROMAGNETIC RESONANCE STUDIES
OF DC MAGNETRON SPUTTERED CO-CR FILMS**

by

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ABSTRACT

The X-band FMR has been employed to investigate the angular variation of resonance fields of DC sputtered Co-Cr films with different substrate temperatures. This angular variation has been fitted with the classical uniaxial anisotropy crystal model and yields the values of $2K_1/M - 4\pi M = -4 \sim -7$ KOe, $4K_2 = -0.8 \sim 0.8$ KOe and $g\text{-factor} = 2.3 \sim 2.8$. The FMR measurements of the first anisotropy constant are quite different from the counterparts measured with VSM. This discrepancy is interpreted as a result of the formation of two ferromagnetic phases. With this simple model, the substrate temperature dependence of first anisotropy is explained and it is predicted that a lower substrate temperature will improve the Co-Cr films for their potential application in perpendicular magnetic recording devices. The angular dependence of the FMR linewidth is discussed.

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CHAPTER 1. INTRODUCTION

1.1. GENERAL INTRODUCTION

Magnetic materials have figured heavily in the spectacular growth of the electronic industry. The reason is that the preeminent method for storing information in computers—ranging from personal computers to large mainframes—is magnetic recording. In one form of the technology, a rigid 14-inch aluminum disk coated with iron oxide is spun about its axis at 3,000 revolutions per minute. Such a rate of spin corresponds to a speed of more than 100 miles per hour at the edge of the disk. As the disk rotates, a ring-type "head" with which to "read" and "write" data is brought near it. The head consists of a coil of wire wrapped around a magnetic core, which is typically nickel-iron alloy.

By passing an electric current through the head coil, one can record data on the disk. The current generates a magnetic field in the coil, which magnetizes a particular area of iron oxide on the disk parallel to the disk surface. That area retains its magnetization and so can "remember" information. The process by which data are encoded on the disk is known as the write cycle. Information on the disk is read out with the same head by reversing the procedure. As the head moves over the disk, magnetized regions on the disk induce a current in the coil. By measuring the current as a function of time, the stored information is retrieved.

The system described above is dominant today and is called longitudinal

magnetic recording in the sense that the magnetization of the stored information is parallel to the recording medium surface.

Even though the processes of reading and writing are based on simple principles, attempts to pack more information onto disks have proved somewhat problematical with the present system. It has been demonstrated that, in the high density recording of the present system, demagnetization in the medium not only decreases the remanent magnetization, but also rotates the magnetization vector to establish a circular magnetization mode, resulting in a significant decrease of the strength of reproduced signals (*Iwasaki and Takemura, 1975*). Even though it is understood that the merit of using a thin film is to prevent the formation of this circular magnetization, there are a few unavoidable obstacles if a future higher density recording system is to be pursued in the same manner. The obstacles all stem from the property of the longitudinal magnetization mode recorded by the ring-type head, that is, the demagnetizing field in the mode increases and approaches a maximum value of $4\pi M_s$, as the recording density increases.

Iwasaki et al (*Iwasaki and Ouchi, 1978*) first published their work in 1978, describing the interesting magnetic properties of cobalt-chromium films and their potential use as media for perpendicular recording. The proposed perpendicular magnetic system, in which the magnetization is perpendicular to the medium surface, has the unique property that the demagnetizing field basically approaches zero in higher recording densities. It was also pointed out (*Iwasaki and Nakamura, 1977*) that a high saturation magnetization and a high coercive force

as well as perpendicular anisotropy of the medium are necessary to obtain high output voltage and high recording resolution. Furthermore, mechanical and chemical stability and high productivity are desired. These properties being taken into account, the cobalt-chromium film is the best candidate.

1.2. THE MOTIVATION TO CHOOSE COBALT-CHROMIUM FILMS

At room temperature, a cobalt single crystal has an hexagonal close-packed lattice (hcp) (See, for example, *Allibert et al.*, 1978). In addition cobalt has a large magnetocrystalline uniaxial anisotropy energy (*Paige et al.*, 1984), and the easy direction is along the c-axis. To meet the requirement for perpendicular magnetic recording, the film must have an anisotropy energy which surpasses the demagnetization energy $2\pi M_s^2$. For pure single crystal cobalt, unfortunately, the anisotropy energy $K(4 \times 10^6 \text{ erg/cc})$ is less than the demagnetization energy $2\pi M_s^2$ ($12.6 \times 10^6 \text{ erg/cc}$). Therefore it is necessary to add other metals to reduce M_s while keeping the c-axis oriented perpendicular to the film surface. When another nonmagnetic metal is added, the magnetization M_s will decrease. Roughly speaking, the anisotropy energy is proportional to M_s while the demagnetization energy $2\pi M_s^2$ is proportional to M_s^2 as long as the film has at least a cluster structure with the c-axis along the normal to the film. As a result, the anisotropy energy will eventually exceed the demagnetization energy when the magnetization is sufficiently small.

There are two obvious reasons to choose chromium as the added metal. One is that the Co-Cr alloy has a relatively stable hcp phase at a low content

of Cr. Another one is that the saturation magnetization is expected to decrease dramatically when a small amount of Cr is added, since Co is antiferromagnetic at room temperature. Other metals such as Ti, V, Mo, Rh, Pd and W have been investigated experimentally (*Kobayashi and Ishida, 1981; Iwasaki et al., 1980*), but it was found out that Cr was the most promising candidate.

1.3. THESIS OUTLINE

Chapter 2 generally reviews the recent development in the Co-Cr films including the preparation techniques, the structure and the magnetic properties. Also a brief review over the effect of sputtering parameters, particularly of the substrate temperature, is presented.

Chapter 3 contains the properties of our samples as well as the experimental procedure. A general description of the ESR apparatus is included.

Chapter 4 gives an account of detailed ferromagnetic resonance theory with first and second anisotropy constants in uniaxial crystals. Special attention is paid to the polycrystallinity of the samples. The linewidth formula in FMR due to the spatial dispersion of the crystallites is also discussed.

Chapter 5 describes the procedure of numerical fitting and how the magnetic properties are derived. A new phase segregation model is proposed to interpret the discrepancy between FMR and VSM. The angular dependence of linewidth is also discussed.

Chapter 6 summarizes the main conclusions of this work and further investigations are proposed.

CHAPTER 2. PREVIOUS INVESTIGATION ON CO-CR FILMS

After the pioneering work of Iwasaki et al. in 1978, an ever increasing number of papers are appearing on this topic. However it is not the intention to review this topic comprehensively here. Therefore only pertinent works will be discussed.

2.1. PREPARATION TECHNIQUES OF CO-CR FILMS

Almost all of the Co-Cr films reported in the literature have been prepared by sputtering techniques. Other methods have been attempted, such as vacuum evaporation. Sugita et al.(1981) have pointed out that evaporation in vacuum can be used for very high rate depositions. However these authors have used a single source of Co-Cr alloy for vacuum deposition which indeed was not an ideal situation. Co and Cr have different evaporation temperatures and evaporation rates, resulting in a deposit of different composition than that of the original alloy target, and even worse inhomogeneities could result. To overcome this disadvantage, Krishnan et al.(1985) employed an two E-beams vacuum co-evaporation technique to prepare the films, which yielded the remarkable result that the crystalline orientation in the initial deposited layer in the co-evaporated Co-Cr films was the same as in the bulk of the film. However, since the two E-beams vacuum co-evaporation method is not sufficiently developed and the available films were sputtered, this thesis will concentrate on sputtering methods.

RF sputtering has been widely used in the preparation of Co-Cr films,

since this method is suitable to prepare films of a high melting point alloy such as Co-Cr, and is superior to the other methods for the adhesion of the deposited magnetic layer to the substrate, and in addition it is reproducible. Another important factor is that Cr has the same sputtering yield as Co in the RF sputtering process. It has been found that the RF sputtered Co-Cr film has a large perpendicular magnetic anisotropy, a high coercivity, and other favorable properties for high density magnetic recording. In the rest of this chapter the samples are assumed to be RF sputtered unless otherwise indicated.

In the past few years, Hoffmann et al.(1985), Ouchi and Iwasaki(1985), and more recently Ravipati et al.(1986) and Li et al.(1986) have studied films deposited by high rate DC Magnetron sputtering. This will be discussed in more detail later.

2.2. STRUCTURE OF CO-CR FILMS

2.2.1. Texture Structure and Morphology

The film microstructure is usually described by its two important properties: the crystallographic(preferred) orientation(or texture) and the morphology(crystal size and shape). The structure properties are experimentally determined by Scanning Electron Microscopy(SEM), Transmission Electron Microscopy(TEM), X-ray diffraction and rocking curve methods. For example, SEM, TEM and X-ray are used to determine the morphology properties of the crystals. The rocking curve represents the angular distribution of the intensity of

X-rays diffracted from a certain plane of the crystal, and consequently, expresses the axis dispersion corresponding to that plane. In all studies of Co-Cr films, $\Delta\theta_{50}$ has been defined as the half angle width of the rocking curve: the larger the $\Delta\theta_{50}$, the broader the angular dispersion. Thus the rocking curve method is mainly devoted to determining the preferred orientation of crystal.

The microstructure was first reported by Iwasaki et al.(1979). In that paper, and subsequently most of all other papers, only hexagonal close-packed structure has been identified by electron diffraction and X-ray diffraction with (002) peaks. The $\Delta\theta_{50}$ are usually a few degrees. So it is safe to conclude that most of all sputtered Co-Cr films exhibit only hcp crystallization. A possible exception is the reported appearance of the fcc phase due to the presence of nitrogen(*Coughlin et al., 1982*) in the RF sputtering chamber.

The morphology of Co-Cr films is not clear at present time. There are two contrasting points of view about this. Iwasaki et al.(1979) determined that the films had a columnar structure from a cross-sectional SEM view. Since then, a columnar structure viewed by a cross-sectional SEM has been accepted as one of the basic properties for perpendicular anisotropic films and this has accounted for a large part of literature on Co-Cr films. (*Iwasaki et al., 1980a; 1980b; Kabayadshi and Ishida, 1981; Honda et al., 1983; Haines, 1984b*). In addition it should be noted that electroplated Co films with perpendicular anisotropy also exhibited clear columnar structure in a cross-sectional SEM view(*Chen and Cavclotti, 1982*).

On the other hand, in studies of Co-Cr films deposited by opposing target cathode sputtering (Kadokura and Naoe, 1982), round grain structure was observed on the cross-sectional SEM of the film which is suitable for perpendicular magnetic recording whereas films with a columnar structure showed undesirable characteristics. A conically shaped grain crystal also has been observed (Lodder *et al.*, 1983; Hwang *et al.*, 1986). However a cross-sectional structure viewed by a SEM is not always an indication of film structure, that is, SEM is believed only to be able to give evidence of intergranular fracture in films. In another words, in the case of the existence of both transgranular fracture and intergranular fracture in the film with hcp phase only, TEM is capable of viewing the columnar structure, which would be missed by cross-sectional SEM due to its inherent insensitivity. This was the result reported by Sagoi *et al.* (1984), which also was strongly supported by tensile test measurements. It can be concluded that almost all the samples with favorable properties of perpendicular magnetic recording are believed to consist of columnar structure whether they can be viewed in cross section SEM or not. Another point is that columnar structure in cross section SEM can not rule out poor c-axis orientation. (Kadokura and Naoe, 1982; Honda *et al.*, 1983).

2.2.2. Phase Segregation and Grain Boundaries

Experimental results regarding the presence of various phases in the films, which are thought to be responsible for the high recording density, can be summarized as follows. (1) The M_s and the T_c are higher than those in the bulk alloy with the same average composition as the films. (Fisher *et al.*, 1984)

(2) The M_s of the film changes depending on preparation conditions such as substrate temperature (Smits *et al.*, 1984). (3) The M_s of the film changes drastically following annealing (Maeda *et al.*, 1982). This behavior suggests that a phase segregation at temperatures below 400°C. (4) The films exhibit a particle-like rotation magnetization reversal mechanism in most samples. (Iwasaki, 1980a; Wuori and Judy, 1984)

Noting from the Co-Cr phase diagram (Moffatt, 1978) that an intermetallic compound Co_3Cr can coexist with an $\epsilon\text{-Co(hcp)}$ phase under a condition of thermodynamic equilibrium in the bulk alloy, Chen and Charlen (1983) reported that the film could have a two phase segregation with possible Co_3Cr precipitation around the grain boundary. The substrate temperature was around 600°C when this phenomenon appeared in the TEM micrographs.

Another paper was published by Jhingan (1986) with a chemical analysis from a nanoprobe. For the as-deposited samples with 95°C substrate temperature, three types of grain boundary segregation were observed: higher Cr/Co ratio, lower Co/Cr ratio and non-segregated. However after annealing treatment with temperatures over 400°C, only the last type was observed. Furthermore, there was no segregation within the grains at all temperatures.

The results from all these experiments seem to suggest a segregated microstructure model whereby the ferromagnetic Co-rich region inside a columnar particle is surrounded by a nonmagnetic Cr-rich region near the grain boundary and the pseudocircular Co-rich particles show a rotational magnetization reversal

mechanism. This model probably can be safely used below 400°C substrate temperature or annealing below 400°C.

A rather powerful TEM study(*Maeda and Asahi, 1987*) has suggested a finer microstructure in crystallites than the grains. This study revealed by selective wet etching shows a definite striped pattern in each crystallite. These stripes, identified by SEM, were caused by dissolution of the Co-rich ferromagnetic regions.

2.2.3. Initial Layer and Substrate Material

Several recent experiments have shown that the films are often composed of two layers—an initial transition layer deposited on the top of a substrate with the thickness 10 nm to 100 nm in a 1000 nm thick film, followed by a structurally oriented bulk layer having the intended magnetic properties. (*Haines, 1984b*)

The first magnetization hysteresis loops presented by Iwasaki and Nakamre(1977) exhibited an abrupt range or anomalous jump in the in-plane loops. This effect was attributed to a fine grained transition layer separating the substrate and the columnar structure(*Iwasaki, 1980a; Ouchi and Iwasaki, 1982; Byun, 1985*).

A series of reports have been published recently. (*Mitchell et al., 1985; 1986; Hwang et al., 1986*) A second resonance peak in FMR was identified as

the transition layer with the magnetocrystalline anisotropy averaged to zero. The result was correlated to TEM and the transition layer was thought to be composed of small, randomly oriented grains. The vacuum annealing at a temperature of 365°C after 45 h effectively turned the transition layer into the well oriented bulk film. Application of a magnetic field during the annealing process improved this result, particularly when the as-deposited film only consisted of a transition layer.

Such a initial transition layer is not desirable in magnetic recording so that the control of this layer could optimize the performance of this media.

The nucleation of Co-Cr crystals in various underlayers which are formed on a substrate prior to Co-Cr alloy deposition has been studied. (*Futamoto, 1985*) An amorphous Ge was the best in a large number of sublayer materials including hcp structure metals such as Ti and Cr. The reason for this was explained by a new growth mechanism. However it was noted by Lodder et al.(1983) that the electrical charge in the electrically insulating substrate from the sputter plasma should be taken into consideration and furthermore the clearness of the substrate had a strong influence on the crystal habit(*Coughlin et al., 1981; Leu et al., 1985*). Hence it is most likely that substrate materials are not very critical as long as they generally satisfy the sputtering conditions.

2.3. MAGNETIC PROPERTIES

A large positive effective anisotropy and a high perpendicular coercive field are the favourable properties for perpendicular high density magnetic recording. Magnetic characterization commonly involves Vibration Sample Magnetometer(VSM) and Torque(balance) Magnetometer(TM). Each of these instruments can be used to determine both the magnetization and the anisotropy. However, VSM is preferred when finding the magnetization while TM is preferred when determining the anisotropy.

2.3.1. Magnetization

Basically, the magnetization loop of ferromagnetic films like Co-Cr is rectangular along the easy axis. Almost all saturation magnetizations are derived from the VSM measured magnetization loops. The saturation magnetization of the films decreases almost linearly with an increase of the Cr content and agrees with that of bulk Co-Cr(*Bozorth, 1951*) when the Cr content is less than 15 at.%. Between 15% and 30% atomic content, discrepancies in the magnetization begin to show between the bulk alloy and the films, and also among different authors(*Iwasaki and Ouchi, 1978; Kobayashi and Ishida, 1981; Bolzoni et al., 1983; Fisher et al., 1984*). This latter inconsistency could be explained by the different phase segregations in the samples deposited under different conditions. (*Hains, 1984a*) These discrepancies are not large and almost all the samples have saturation magnetizations around 400 emu/cc at about 20 at.% Cr.

The microstructure inhomogeneities do not influence the saturation magnetization but rather magnetization process. The known coercive fields vary considerably from 2 KOe to 400 Oe. Through careful adjustment of the sputtering conditions, reasonably large coercive fields can be achieved. The optimum parameters of sputtering will be discussed in the section 2.4.

2.3.2. Anisotropy

There are two kinds of anisotropy in the Co-Cr films: one is magnetocrystalline anisotropy, another is shape anisotropy. Of these, the magnetocrystalline anisotropy is intrinsic to the films. This is due to the spin-orbit-lattice interaction. The shape anisotropy comes from the demagnetizing effect. These two anisotropies cannot be separated in all measurements owing to the fact that they possess the same angular dependence so that sometimes an effective anisotropy is used to represent their combination. The anisotropy energy may be measured by torque magnetometer, magnetization curve or magnetic resonance, although the common method in this case is torque magnetometer.

The hcp uniaxial anisotropy energy

$$E = K_0 + K_1 \sin^2 \theta + K_2 \sin^4 \theta + \text{higher order} \quad (2.1)$$

in which θ is the angle between the M vector and the c-axis. The shape anisotropy is $-2\pi M^2 \sin^2 \theta$ so that

$$E = K_0 + (K_1 - 2\pi M^2) \sin^2 \theta + K_2 \sin^4 \theta + \text{higher order} \quad (2.1a)$$

$K_1 - 2\pi M^2$ is called the effective anisotropy constant in most cases. More accurately, $K_U = K_1 + K_2 - 2\pi M^2$ should be used.

The anisotropy constants depend strongly on the sputtering conditions. The K_1 and K_2 of pure bulk cobalt have been investigated extensively (*Paige, 1984* and references therein). At room temperature, K_1 is about 5.0×10^6 emu/cc, K_2 , 1.0×10^6 emu/cc. The effective anisotropy constant is negative for pure Co films. The experiments confirmed that the effective anisotropy become positive within the range of Cr 15-30 at.%(*Iwasaki, 1978; Fisher, 1984*). Experimentally the K_1 are distributed among $1.1 \sim 1.9 \times 10^6$ ergs/cc(*Iwasaki, 1978; Honda et al., 1983; Bolzoni et al., 1983; Fisher et al., 1984; Sagoi, 1984*) and the only K_2 values were obtained by Fisher et al.(1984) with a careful torque curve analysis ($-0.05 \sim 0.6 \times 10^6$ ergs/cc). Both values are dramatically different from the pure bulk Co values, and the reason for that remains undetermined at present.

2.4. EFFECTS OF SPUTTERING PARAMETERS

It is well known that the magnetic properties in RF-sputtered Co-Cr films vary considerably depending on the substrate material, substrate temperature and argon gas pressure, etc. To prevent the oxidation and nitrogen induced fcc nonmagnetic phase from occurring, the initial background pressure should be reduced to not less than 10^{-7} τ (*Coughlin et al., 1982; Honda et al., 1984*).

2.4.1. Argon Gas Pressure

Ar pressure has significant effects not only on c-axis orientation, but also on mechanical strength for Co-Cr sputtered films. Two articles (*Honda et al., 1983; Sagoi et al., 1984*) presented similar results. They found that as the Ar pressure decreases (lowerst 1 m τ), the films deposited have superior c-axis orientations, the anisotropy constants become large due to the reduction of the demagnetizing fields caused by the change of the magnetic domain structure and, in addition, the films have larger coercive fields and a very high mechanical strength. Lower Ar pressure was tested by Niimura and Naoe(1986) with the result that 0.5-1 m τ was deemed the best range for a favorable Co-Cr film.

2.4.2. Substrate Temperature

The most important parameter, but unfortunately also the most elusive parameter in the case of sputtering, is the substrate surface temperature. In RF-sputtering, the surface temperature, *i.e.*, the temperature at which the film formation takes place, is predominantly determined by three factors which are of same order of magnitude: (1) the temperature of the substrate holder, (2) the temperature produced by heat from the condensing atoms and (3) the temperature produced by heating from the sputter plasma, mainly by the electrons. This complex mechanism not only makes it difficult to determine an accurate surface temperature but also implies that this temperature will not be stationary during the sputter run. The surface temperature in RF-sputtering was determined by measuring the hcp-fcc transition temperature and comparing this

with bulk data for Co-Cr. The temperature was found to be 550°C higher than the measured substrate temperature (*Iwasaki and Ouchi, 1980b*), though such a comparison is questionable. Despite all these difficulties, the dependence of substrate temperature has been examined and found to be ambiguous(*Coughlin et al., 1981; Wieldinga and Lodder, 1981*). It is not easy to control the surface temperature or even to be able to compare the different results.

2.5. DC MAGNETRON SPUTTERED FILMS

Although the RF sputtered Co-Cr films are highly satisfactory for the perpendicular recording medium, its deposition rate is usually less than 1 nm per second. The industrial production needs a higher deposit rate to be viable. DC Magnetron Sputtering may be introduced to meet such a requirement.

The general features of these high rate depositions are similar to the RF sputtering deposition. But the high rate deposition seems to result in a more inhomogeneous film, which in turn has a lower anisotropy constant(*Ouchi and Iwasaki, 1985*). In these films more phase segregation can be identified and the effective anisotropy constant turns negative at a smaller percentage content of Cr than that in RF sputtered films.

2.6. FERROMAGNETIC RESONANCE AND OUR MOTIVATION

2.6.1. FMR Measurements

At the present time, there are only a few ferromagnetic resonance(FMR) reports on Co-Cr films. VSM and TM are static measurements so that only the volume average magnetic quantities can be retrieved. FMR, on the other hand, is a sensitive dynamic technique which can provide detailed information about the magnetic inhomogeneities and, ultimately, the different regions.

One advantage was shown by Krishnan et al.(1985) that when the film is too thin to be measured with VSM, FMR still can faithfully be trusted. Other situations have also been successfully handled, such as the detection of the transition layer(*Mitchell et al., 1986*) and the surface effect(*Cofield et al, 1986*).

An extensive comparison between FMR and VSM/TM measurements was made by Mitchell et al.(1985). They found that when two layers existed in the film, there was a discrepancy, while when the film had only single layer there was agreement.

Resonances at different microwave frequency were compared explicitly(*Cofield et al., 1986*). The resonance features changed little, except that the broad resonance observed in the 9.8 GHz spectra had a more symmetrical lineshape and a smaller linewidth at 35 GHz. This broadened resonance was attributed to the domain wall resonance. As long as the sample was saturated, there seemed excellent agreement between the results of these two frequencies.

2.6.2. Our Motivation

This work was done at X-band(9 GHz) with a conventional Electron Spin Resonance(ESR) spectrometer. Certainly Q-band(35 GHz) FMR has some advantages over X-band, among others, Q-band is more sensitive, the analysis is easier owing to the fact the the sample is most likely to be saturated at the resonance fields(10 KOe). But, on the other hand, we believe that X-band can be related more to the inhomogeneities of the samples since the resonances happen at relatively low magnetic fields. The numerical analysis is manageable with the help of a computer.

Our objective was to study some characteristics of the DC Magnetron Sputtered Co-Cr films and to compare with the results of VSM on the same films made by Li et al.(1986). The variation of the magnetic properties with the change of substrate temperature are especially emphasized. Such a investigation should be quite valuable to the characterization of these inhomogeneous films. Furthermore, there is a lack of information about the angular dependence of the resonance for these Co-Cr films. This angular dependence should allow determination of the second anisotropy constant, which is an important parameter in magnetization studies.

CHAPTER 3. SAMPLES AND EXPERIMENTAL TECHNIQUES

3.1. THE PREPARATION OF THE SAMPLES

All our samples are courtesy of Prof. R. R. Parsons and Mr. Z. Li in Physics Dept., UBC. The samples were prepared with DC Planar Magnetron Sputtering with floating glass as the substrates.

A composition target CoCr(20 at.%) was used. The chamber was pumped down to less than 10^{-6} τ . Then 3 m τ Argon gas was flushed into the chamber. The target-substrate distance was 9.5 cm while the substrates were biased at $-200\sim 0$ V. The sputtering power was driven high (350 W) so that a high deposition rate of 100 nm per minute was obtained. The substrates were heated by a halogen lamp and as a result of this a series of samples with substrate temperatures from 150°C to 320°C were achieved.

3.2. THE PROPERTIES OF THE SAMPLES

The thickness was controlled to about 500 nm. The structure of the samples were investigated with a SEM and an X-ray spectroscope. The X-ray diffraction results indicate clear (001) peaks so that the films consist of equiaxed grains rather than columnars. The diameters of the grains range from 10 nm to 100 nm and the grains seemed to "stick" together according to the SEM pictures.

The magnetization curves have been characterized by a VSM, and a typical example is illustrated in *Fig3.1*. The coercive fields and the ratios of the remanent magnetization to the saturation magnetization are plotted in *Fig3.2* and *3.3*, respectively, against the substrate temperature. The effective anisotropy constants K_u in *Fig3.4* were determined by measuring, on the $M \sim H$ graph, the area included between the magnetization curves for easy and hard crystal directions. The interesting thing here is that there are some anomalous behaviors, particularly of the anisotropy constants, at a substrate temperature around 200°C.

3.3. GENERAL DESCRIPTION OF THE APPARATUS

The block diagram of ESR X-band spectrometer which was used in this work is shown in *Fig3.5*. The microwave bridge utilized a circulator. A reflex klystron(Varian VA-273), a one way isolator, and a flap attenuator were connected to the input arm, a crystal detector with a slide-screw tuner to the output. The third arm ended in a TE_{102} resonance cavity coupled through an adjustable iris.

The klystron was frequency locked to the resonance cavity by modulating the reflector voltage with a 10 KHz signal and using the corresponding phase sensitive detected output from the crystal detector as an error signal. The magnetic field was modulated at 2.3 KHz through a pair of modulation coil attached to the pole caps.

Fig3.1 A typical $M \sim H$ curve. This curve corresponds to the sample with $T_s = 199^\circ\text{C}$.

Fig3.2 The substrate temperature dependence of the coercivity measured with VSM.

Fig3.3 The substrate temperature dependence of the remanent magnetization measured with VSM.

Fig3.4 The substrate temperature dependence of the effective anisotropy constant measured with VSM.

Fig3.5 The block diagram of the X-band ESR spectrometer.

The pre-amplified output from the crystal detector was phase sensitive detected at 2.3 KHz (Ithaco 391A Dynatrac 391A Lock-in Amplifier). The output was then connected to a chart recorder.

The frequency of the microwaves was measured with a digital frequency meter (Hewlett-Packard 5245L Electronic Counter with plug-in 5255A Frequency Converter). The measurement range is 3~12.4 GHz with an accuracy 1 KHz.

The experiments were performed on a 12" Magnion Magnet with HS-1365B Precision Magnet Supply with the FFC-4 Field Control Regulator. The maximum output current is 65 Amperes which gives about 17 KOe dc magnetic field. The set accuracy is 0.1 Oe with the stability 1×10^{-5} after three hours warm up. The sweep linearity is within 0.1%.

3.4. EXPERIMENTAL PROCEDURES

The spectrometer was operated at 9.46 GHz. The microwave power was low and estimated to be ~10 mW. The field modulation frequency was 2.3 KHz. The time constant of lock-in was usually set at 1.25 msec.

The scanning rate of DC field was 50 Oe/Min. The accuracy requirement for the dc field was easily met without the calibration sample. However, $\text{CuSO}_4 \cdot 6\text{H}_2\text{O}$ was employed as a standard sample and simultaneously placed on the bottom of the cavity to permit estimation of the relative resonance amplitudes.

The resonance cavity operated in the TE_{102} mode and was made of brass. The measurements were conducted at room temperature. The samples with the size $1.0 \times 0.5 \text{ cm}^2$ were placed on a rotatable lucite holder, so that the angular dependence of resonance could be measured.

CHAPTER 4. FERROMAGNETIC RESONANCE THEORY

4.1. JUSTIFICATION OF CLASSICAL RESONANCE THEORY

The specific features of resonance phenomena in ferromagnetics are determined primarily by the fact that in these materials we deal not with individual isolated atoms or with the comparatively weakly coupled moments of paramagnetic systems, but with a complicated system of strong interacting electrons. The exchange nature of this interaction causes the unpaired spin magnetic moments of the ions in the ferromagnetic crystal lattice to assume parallel orientation below the Curie temperature. The exchange interaction creates a substantial total magnetization and consequently a large internal magnetic field, and this internal field may alter the usual paramagnetic resonance condition considerably.

A macroscopic description can be used for ferromagnetic resonance since the quantum numbers corresponding to the energy levels are of the order of 10^{15} and higher. (See, for instance, *Luttinger and Kittel, 1948*). It follows from the correspondence principle that classical and quantum-mechanical treatments of the problem yield identical results. This conclusion has been improved by *J.H. Vleck (1950)* to the extent that his Hamiltonian includes the exchange interaction and the ordinary dipolar interaction as well as the crystalline anisotropy though minor approximations should be taken.

4.2. PROPAGATION OF ELECTROMAGNETIC WAVE IN A FERROMAGNETIC METAL

The question to be addressed is that of a ferromagnetic metal in a microwave cavity as used in ESR which interacts with the microwave field while it is in an applied dc magnetic field $\vec{H}_0$. It is a formidable task to have a complete analytic solution of such a problem, which fundamentally differs from the usual situation in that the induced magnetization is not linearly proportional to the applied field. To elucidate the specific features of the phenomenon in its pure form, we should first consider an idealized problem, namely 1) the damping processes should not be allowed, and 2) we shall neglect the crystalline and the shape anisotropies. In this case the equations describing the propagation of electromagnetic waves in a ferromagnetic conductor (Ament and Rador, 1955) include an equation of motion for the magnetization $\vec{M}(\vec{r})$

$$\frac{1}{\gamma} \frac{d\vec{M}}{dt} = -\vec{M} \times \vec{H} + \frac{2A}{M_0^2} \vec{M} \times \nabla \vec{M} \quad (4.1)$$

and Maxwell's equations:

$$\nabla \times \vec{E} = \frac{1}{c} \frac{\partial}{\partial t} (\vec{H} + 4\pi \vec{M}) \quad (4.2)$$

$$\nabla \times \vec{H} = \frac{4\pi}{c} \vec{j} \quad (4.3)$$

the first and the second terms in the right-hand side of eq4.1 are, respectively, the moment densities of the force exerted by the magnetic fields and the effective exchange field

$$\vec{H}_{ex} = \frac{\gamma A}{M_0^2} \nabla^2 \vec{M} \quad (4.4)$$

the latter can be obtained from the expression for the exchange energy, which is proportional to the square of the magnetization gradients.

In eq4.3, the displacement current has been neglected since at the microwave frequency it is small compared with the conduction current in a metal. When the penetration depth δ of the electromagnetic field into the metal is large in comparison with the mean free path l of an electron, then the $\vec{j}$ is related to the electron field intensity $\vec{E}$ by Ohm's law(normal skin effect):

$$\vec{j} = \sigma \vec{E} \quad (4.5)$$

We shall assume that inside the sample

$$\vec{H} = \vec{H}_0 + \vec{h}, \quad \vec{M} = \vec{M}_0 + \vec{m} \quad (4.6)$$

here $\vec{M}_0$ is the induced saturation magnetization caused by $\vec{H}_0$ and the microwave components $|\vec{m}| \ll M_0$ and $|\vec{h}| \ll H_0$. Certainly the boundary condition for the fields are the continuity of the tangential componets of $\vec{e}$ and $\vec{h}$:

$$\vec{e}_\perp|_+ = \vec{e}_\perp|_-, \quad \vec{h}_\perp|_+ = \vec{h}_\perp|_- \quad (4.7)$$

The boundary condition for $\vec{m}$ can be obtained from the condition that the total moment of the exchange force should vanish over the entire sample, i.e.,

$$\int_{\text{sample}} \vec{m} \times \nabla^2 \vec{m} d\tau = 0 \quad (4.8)$$

Obviously, all of these equations have to be solved simultaneously with Maxwell's equations outside the sample but within the cavity and under the

corresponding boundary conditions.

Stronger approximations must be adopted before the equations can be solved. To get a flavor for this kind of electromagnetic propagation, a plane incident microwave and an infinitive cavity are assumed. Let an electromagnetic wave be incident on a plane plate perpendicular to its surface, and the applied field H_o parallel to the surface of the plate. If the skin effect is neglected ($\sigma=0$), then

$$\mu_1 = \frac{1}{\frac{H_o}{4\pi M_o} - \left(\frac{\omega}{\gamma 4\pi M_o}\right)^2}, \quad \mu_2 = 0 \quad (4.9)$$

so the resonance condition is:

$$H_o = \frac{1}{4\pi M_o} \left(\frac{\omega}{\gamma}\right)^2 \quad (4.10)$$

where $\mu_{eq} = \mu_1 - i\mu_2$, ω angular frequency of microwave.

With $\sigma \neq 0$, $\mu_2 \neq 0$ and therefore the electromagnetic field energy is absorbed. In this case the resonance field H_o , determined from the maximum of μ_2 , is equal to

$$H_o = \frac{1}{4\pi M_o} \left(\frac{\omega}{\gamma}\right)^2 - \frac{4}{3} \sqrt{\frac{4\pi A}{\delta}} \quad (4.11)$$

where δ is the classical skin depth (with $\mu=1$). Thus, as a result of the exchange interaction, the skin effect causes a resonance line shift in ferromagnetic metals towards weaker fields by

$$\Delta H_o = \frac{8\pi}{3} \sqrt{\frac{A\sigma\omega}{c}} \quad (4.12)$$

Practically such a rigorous derivation in most cases has never been done and usually is unnecessary. For our case, the skin effect can be ignored since the skin effect basically comes from the microwave tangential electric field, which is almost zero at the middle of the cavity where the samples are placed. The cobalt at room temperature has a skin depth of about $1.5\mu\text{m}$ at 10 GHz. The tangential microwave electric field $E \sim E_0 l/a$, where E_0 is the maximum electric field in the cavity. In our case, $l \sim 0.4$ cm the dimension of the sample and $a \sim 2$ cm the cavity size. Approximately this can give a factor of 5 increase to the skin depth, which is then about $7.5\mu\text{m}$ whereas the sample thickness is $0.5\mu\text{m}$. Both theoretical and experimental work have confirmed that a solution without the consideration of Maxwell's equations and the boundary conditions is justified as far as just the ferromagnetic resonance frequency is concerned.

4.3. THEORETICAL FORMULA FOR THE RESONANCE FIELD

Suppose we are dealing with a single domain ferromagnetic crystal with the magnetic crystalline anisotropy and the shape anisotropy, but free from the stress. The various interactions can be considered phenomenologically, provided it is assumed that the spins responsible for the ferromagnetism process with the frequency ω , not in the external field $\mathbf{H}$, but rather in a certain effective field $\mathbf{H}_{eff}$, whose effect is equivalent to that of an external field. In this case, the equation of motion of the magnetization vector $\mathbf{M}$ becomes

$$\frac{1}{\gamma} \frac{d\vec{M}}{dt} = -\vec{M} \times \vec{H}_{eff} \quad (4.1a)$$

Another method was independently proposed by Smit and Beljers(1955) and

Suhl(1955), which is more convenient for our present purpose and also permits extensive generalizations. We shall dwell on it in some detail.

The coordinate system used in our calculation of the magnetostatics and the fields for resonance is shown in *Fig4.1*.

$$\begin{aligned} M_x &= M \sin \theta \cos \phi \\ M_y &= M \sin \theta \sin \phi \\ M_z &= M \cos \theta \end{aligned} \tag{4.13}$$

hence the radial H_M , polar H_θ and azimuthal H_ϕ components are

$$\begin{aligned} H_M &= H_x \sin \theta \sin \phi + H_y \sin \theta \cos \phi + H_z \cos \theta \\ H_\theta &= H_x \cos \theta \cos \phi + H_y \cos \theta \sin \phi - H_z \sin \theta \\ H_\phi &= -H_x \sin \phi + H_y \cos \phi \end{aligned} \tag{4.14}$$

and the eq4.1a in our spherical coordinate system assume the form

$$\dot{\theta} = \gamma H_\phi, \quad \dot{\phi} \sin \theta = -\gamma H_\theta \tag{4.1b}$$

where we take into account that $M=\text{constant}$.

In the state of thermodynamic equilibrium, the components H_θ and H_ϕ of H_{eff} vanish, i.e., at the equilibrium position θ_{eq} , ϕ_{eq}

$$F_\theta = \frac{\partial F}{\partial \theta} = 0, \quad F_\phi = \frac{\partial F}{\partial \phi} = 0 \tag{4.15}$$

Fig4.1 Orientation of the dc magnetic field $\vec{H}$ and of the magnetization $\vec{M}$ with respect to the coordinate system used the in the calculations.

If a small perturbation exists, such as a microwave field, the orientation of the vector $\vec{M}$ varies due to the influence of the nonvanishing field componets

$$H_\theta = -F_\theta/M, \quad H_\phi = -F_\phi/M \sin \theta \quad (4.16)$$

If the deflection from equilibrium is

$$\delta\theta(t) = \theta(t) - \theta_{eq}, \quad \delta\phi(t) = \phi(t) - \phi_{eq} \quad (4.17)$$

then we may limit ourselves to the linear terms in the expansion of F_θ and F_ϕ

$$F_\theta = F_{\theta\theta}\delta\theta + F_{\theta\phi}\delta\phi, \quad F_\phi = F_{\phi\theta}\delta\theta + F_{\phi\phi}\delta\phi \quad (4.18)$$

where the second derivative of the free energy with respect to the angles $F_{\theta\theta}$,

$F_{\phi\phi}$ and $F_{\theta\phi}$ are calculated from the equilibrium position. Now, using eqs 4.1b, 4.16, 4.17 and 4.18, the small free oscillations of M about the equilibrium position are obtained

$$\begin{aligned} -\gamma^{-1} M \sin \theta_{eq} \delta \dot{\theta} &= F_{\phi\theta} \delta \theta + F_{\phi\phi} \delta \phi \\ \gamma^{-1} M \sin \theta_{eq} \delta \dot{\phi} &= F_{\theta\theta} \delta \theta + F_{\theta\phi} \delta \phi \end{aligned} \quad (4.19)$$

which have the periodic solutions $\delta\theta, \delta\phi \sim \exp(i\omega t)$ if the determinant of eq 4.19 vanishes:

$$F_{\theta\phi}^2 - F_{\theta\theta} F_{\phi\phi} + \omega^2 \gamma^2 \sin^2 \theta_{eq} M^2 = 0 \quad (4.20)$$

whence for the eigen- or resonance- frequency of oscillation we obtain

$$\omega_{res} = \frac{\gamma}{M \sin \theta_{eq}} \{F_{\theta\theta} F_{\phi\phi} - F_{\phi\theta}^2\}^{1/2} \quad (4.21)$$

The free energy density function appropriate to a crystal with hexagonal structure, up to the second-order term in magnetic anisotropy, is given by the expression

$$\begin{aligned} F &= -HM \sin \theta \cos(\phi_H - \phi) + \frac{1}{2} (4\pi M^2) \sin^2 \theta \sin^2 \phi \\ &\quad - (K_1 + 2K_2) \sin^2 \theta \sin^2 \phi + K_2 \sin^4 \theta \sin^4 \phi \end{aligned} \quad (4.22)$$

where the first term represents the Zeeman energy, the second the demagnetization energy, and the last two terms the axial anisotropy energy with

the c-axis parallel to the y axis. K_1 and K_2 are the first- and second- order anisotropy constants. For $K_1 < 0$, the easy magnetization direction is in the x-z film plane.

Written in the units of M ,

$$\frac{F}{M} = -H \sin \theta \cos(\phi_H - \phi) - \left(\frac{1}{2}(H_1 - 4\pi M) + \frac{H_2}{2}\right) \sin^2 \theta \sin^2 \phi + \frac{1}{2} H_2 \sin^4 \theta \sin^4 \phi \quad (4.23)$$

where $H_1 = 2K_1/M$, $H_2 = 2K_2/M$. From the eq4.15

$$\begin{aligned} F_\theta &= \cos \theta_{eq} \{ -H \cos(\phi_H - \phi_{eq}) - ((H_1 - 4\pi M) + 2H_2) \sin \theta_{eq} \cos \theta_{eq} \\ &\quad + 2H_2 \sin^3 \theta_{eq} \sin^4 \phi_{eq} \\ &= 0 \end{aligned}$$

$$\begin{aligned} F_\phi &= -H \sin \theta_{eq} \sin(\phi_H - \phi_{eq}) - ((H_1 - 4\pi M) + H_2) \sin^2 \theta_{eq} \sin \phi_{eq} \cos \phi_{eq} \\ &\quad + H_2 \sin^4 \theta_{eq} \sin^3 \phi_{eq} \\ &= 0 \end{aligned} \quad (4.24)$$

since the dc field H is applied in the x-y plane, $\theta_{eq} = \pi/2$ and substituting eq4.24, we obtain the equation for ϕ_{eq} :

$$H \sin(\phi_H - \phi_{eq}) = -((H_1 - 4\pi M) + H_2) \cos \phi_{eq} \sin \phi_{eq} + H_2 \sin^3 \phi_{eq} \cos \phi_{eq} \quad (4.25)$$

The resonance fields can be calculated with the aid of eq4.21,

$$\begin{aligned} \left(\frac{\omega}{\gamma}\right)^2 &= \{ H_{res} \cos(\phi_H - \phi_{eq}) + ((H_1 - 4\pi M) + H_2) \cos \phi_{eq} \sin \phi_{eq} + 2H_2 \sin^3 \phi_{eq} \cos \phi_{eq} \} \\ &\times \{ H_{res} \cos(\phi_H - \phi_{eq}) - ((H_1 - 4\pi M) + H_2) \cos 2\phi_{eq} + 2H_2 (3 \sin^2 \phi_{eq} \cos^2 \phi_{eq} - \sin^4 \phi_{eq}) \} \end{aligned} \quad (4.26)$$

and the g-factors are related to γ by

$$g = \gamma \mu_B \quad (4.27)$$

where $\mu_B = 2\pi \times 1.40 \text{ GHz/KOe}$.

4.4. FMR WIDTH OF CONDUCTING SINGLE CRYSTALS

For pure single crystal, single domain ferromagnetic conductors the relaxation processes are due to the spin-spin, spin-lattice and spin-conduction electrons interactions. These relaxation processes can be taken into consideration formally by introducing a damping term in eq4.1a:

$$\frac{d\vec{M}}{dt} = -\gamma \vec{M} \times \vec{H} - \frac{\alpha}{M} \gamma \vec{M} \times (\vec{M} \times \vec{H}) \quad (4.1c)$$

Where α is a dimensionless factor, and in most cases it is quite small. Repeating the procedure in section4.3 with the additional damping term, yields out the resonance frequency

$$\left(\frac{\omega_{res}}{\gamma}\right)^2 = \frac{(1 + \alpha^2)}{M^2 \sin^2 \theta_{eq}} \{F_{\theta\theta} F_{\phi\phi} - F_{\theta\phi}^2\} \quad (4.21a)$$

and the resonance absorption width

$$\left(\frac{\Delta\omega}{\gamma}\right)^2 = \frac{\alpha^2}{M^2} \{F_{\theta\theta} + F_{\phi\phi} / \sin^2 \theta_{eq}\} \quad (4.28)$$

Finite relaxation ($\alpha \neq 0$) leads to, for small α , an insignificant shift in the resonance frequency ω_{res} defined by eq4.21, and a few Oersted width which usually results from the effective anisotropy field $\sim 10^5 \text{ Oe}$.

Moreover, the line width and shape may change due to the finite penetration depth and the resulting spatial nonuniform magnetization. But in our case, this effect could be totally ignored, as has been discussed earlier.

In spin-spin relaxation the energy of the uniform procession of the magnetization excited at resonance is transmitted to other spin waves with $k \neq 0$, and in spin-lattice relaxation it is transmitted to lattice vibrations, and in spin-conduction electron relaxation it is transmitted to the nonmagnetic electrons. These relaxation processes will give an intrinsic linewidth, which can be assumed to be of the order of tens Oe and possess some symmetric lineshape such as Gaussian or Lorentzian. The detail is not quite meaningful here, since the inhomogeneities would broaden this intrinsic width greatly and cause the true lineshape to have little connection with the intrinsic lineshape.

4.5. FMR WIDTH OF POLYCRYSTALS

Certainly in real crystals the various structural inhomogeneities would influence the lineshape dramatically. This implies that the line width usually is much larger than the intrinsic width, particularly in polycrystals. Hence in this case unless a be taken into account, an erroneous g value could be concluded.

Frequently one has to include the polycrystallinity of samples, surface defects and domain structure. The influence of these inhomogeneities on the line width can be described in many cases with the aid of a nonuniform internal effective anisotropy field, whose spatial fluctuation δH_i determines the line

broadening.

In ferromagnets with relatively low anisotropy, $H_i \ll M$, strong dipole-dipole interactions between the various crystallites may lower the effect of the nonuniformity of H_i appreciably by coupling the crystallites together. In strong anisotropic crystals, $H_i \gg M$, the resonance conditions in individual crystallites appear to be independent, and the line width is determined by the dispersion of the resonance frequency in the various crystallites due to the different orientations of the anisotropy field H_i . Here the resonance curve is generally asymmetric and even possesses a definite structure (two or more resonance peaks).

It seems reasonable to employ the rough estimation of the line width resulting from the inhomogeneities of the anisotropy field due to the spatial dispersion of the c-axis and the magnitude fluctuation of the internal field, or in another words the internal field is treated as a vector. In our case we can denote $H_i = H_1 - 4\pi M + H_2$ and treat H_2 as constant since H_2 is relatively small. So the half width at resonance

$$\Delta H = \Delta H_o + \Delta \phi_H \left(\frac{\partial H_{res}}{\partial \phi_H} \right) + \Delta H_i \left(\frac{\partial H_{res}}{\partial H_i} \right) \quad (4.29)$$

where the first and second terms are, respectively, due the spatial dispersion of the c-axis and the effective internal field fluctuation.

Our case is closer to the strong anisotropic crystal one, and with the help of eqs 4.25 and 4.26, and remembering that the H_{res} and ϕ_{eq} are the dependent variables of H_i and ϕ_H :

$$\frac{\partial H_{res}}{\partial \phi_H} = H_{res} \frac{\{(F_1 + F_2) \sin(\phi_H - \phi_{eq}) - (\frac{F_2}{F_1} \frac{\partial F_1}{\partial \phi_{eq}} + \frac{\partial F_2}{\partial \phi_{eq}}) \cos(\phi_H - \phi_{eq})\}}{\{(F_1 + F_2) \cos(\phi_H - \phi_{eq}) + (\frac{F_2}{F_1} \frac{\partial F_1}{\partial \phi_{eq}} + \frac{\partial F_2}{\partial \phi_{eq}}) \sin(\phi_H - \phi_{eq})\}} \quad (4.30)$$

$$\frac{\partial H_{res}}{\partial H_i} = \frac{\{(F_2 \cos \phi_{eq} - F_1 \sin^2 \phi_{eq}) - (\frac{F_2}{F_1} \frac{\partial F_1}{\partial \phi_{eq}} + \frac{\partial F_2}{\partial \phi_{eq}}) \cos(\phi_H - \phi_{eq})\}}{\{(F_1 + F_2) \cos(\phi_H - \phi_{eq}) + (\frac{F_2}{F_1} \frac{\partial F_1}{\partial \phi_{eq}} + \frac{\partial F_2}{\partial \phi_{eq}}) \sin(\phi_H - \phi_{eq})\}} \quad (4.31)$$

here

$$F_1 = H_{res} \cos(\phi_H - \phi_{eq}) - (H_1 - 4\pi M + H_2) \cos 2\phi_{eq} + H_2(3 \sin^2 \phi_{eq} \cos^2 \phi_{eq} - \sin^4 \phi_{eq}) \quad (4.32)$$

$$F_2 = H_{res} \cos(\phi_H - \phi_{eq}) + (H_1 + H_2) \sin^2 \phi_{eq} - H_2 \sin^4 \phi_{eq} \quad (4.33)$$

and noting that symbolically

$$\frac{\partial F_{1,2}}{\partial \phi_{eq}} = \frac{\partial F_{1,2}}{\partial \phi_{eq}} \bigg|_{H_1, H_2, H_{res}, \phi_H} \quad (4.34)$$

Such a complicated calculation only can be attacked numerically by the computer, which will sort out the best fitting parameters $\Delta\phi_H$ and ΔH_i . Qualitatively these two fitting parameters may reveal some microscopic structure of the films and can be compared with the data of other measurements such as θ_{50} .

CHAPTER 5. RESULTS AND DISCUSSION

The general features of the resonance are complicated. Generally there are more than one peak but always one of these is much stronger than the rest. The resonances are broad with the linewidths of about 1 KOe and are also unsymmetrical.

There are some variations between the different samples. In parallel positions, the samples with $T_s = 157$ and 192 °C have three obvious resonance peaks(*Fig5.1a*) while the other samples have only two obvious peaks with an additional weak peak(*Fig5.1b*). As for the perpendicular positions, all these samples have two peaks with the second one not quite visible(*Fig5.1c*).

5.1. NUMERICAL ANALYSIS OF ANGULAR DEPENDENCE OF RESONANCE FIELDS

The spectrum of each film was investigated as a function of the orientation of the applied dc field H in a plane perpendicular to the plane(angle ϕ_H in *Fig4.1*). The signal detected corresponds to the field derivative of the absorbed power.

In the case of more than one peak, the strongest one was considered first. The resonance field was chosen as the cross over point between the spectrum and the estimated baseline.

Fig5.1a The FMR spectrum of the sample with $T_s=157^\circ\text{C}$ at $\phi_H=0^\circ$. The narrow resonance is due to the calibration sample.

Fig5.1b The FMR spectrum of the sample with $T_s=217^\circ\text{C}$ at $\phi_H=0^\circ$. The narrow resonance is due to the calibration sample.

Fig5.1c The FMR spectrum of the sample with $T_s = 217^\circ\text{C}$ at $\phi_H = 90^\circ$. The narrow resonance is due to the calibration sample.

The angular dependence of the resonance fields were obtained for each sample with different substrate temperature T_s . One of them is illustrated in Fig5.2 with $T_s = 192^\circ\text{C}$. The uncertainty of the resonance field is ~ 0.1 KOe.

It is seen from eqs 4.25, 4.26, and 4.27 that the resonance field H_{res} depends on three parameters, i.e., the first and second anisotropy field H_1 , H_2 , and the g factor. The saturation magnetization M was obtained from the VSM measurements as 400 emu/cc for all the samples.

In the numerical fitting computation, however, an additional parameter needed to be considered, i.e., the misalignment angle. The sample was mounted with the parallel position adjusted by hand, which unavoidably introduced a

Fig5.2 The angular dependence of resonance field for the sample with $T_s=217^\circ\text{C}$. The solid line represents the fitted curve with $H_1=-4.5$ KOe $H_2=-0.05$ KOe, and $g=2.36$.

misalignment angle. The fitting computation with four parameters simultaneously turned out to be rather lengthy and tedious, consequently these parameters were treated separately.

First of all, the misalignment angle was determined by the curve fitting method around the perpendicular position. The angle corresponding to the maximum of the curve fitting represented the true perpendicular position because of the symmetry of the resonance fields with respect to the y axis. Then all of the data points including the resonance fields and the linewidth were shifted by this angle.

Secondly, the adjusted resonance fields in parallel and perpendicular positions H_{pa} and H_{pe} , respectively, were combined to further reduce the number of the dependent fitting parameters. When $\phi_H=0^\circ$, $\phi_{eq}=0^\circ$ under the condition $(H_{pa}+H_1-4\pi M+H_2)>0$, so the resonance equation 4.26 becomes

$$\left(\frac{\omega}{\gamma}\right)^2 = H_{pa}(H_{pa} - (H_1 - 4\pi M) - H_2) \quad (5.1)$$

Similarly $\phi_H=90^\circ$, $\phi_{eq}=90^\circ$ when $(H_{pe}+H_1-4\pi M)>0$, hence

$$\frac{\omega}{\gamma} = H_{pe} + H_1 - 4\pi M \quad (5.2)$$

These two inequalities were forced to be satisfied through the fitting procedure. The eqs 5.1 and 5.2 would reduce the number of the dependent parameters into one, which was chosen as H_1 thereafter.

A special programme was written to solve the eqs 4.25 and 4.26

simultaneously. For a given H_1 (and consequently H_2 and g factor) and given angle ϕ_H , there are usually two solutions, which can be understood in the sense that the number of the stable equilibrium positions from *eq4.25* could be more than one. In the procedure of calculating the second resonance peak, it should be noted that the stable condition of the magnetization vector $\mathbf{M}$ is not the usual one, i.e., the free energy is minimum. For a general case, the stable condition may be complicated. But one can have some feeling of this from a simple example. Assuming $\delta\phi = \text{constant} = 0^\circ$, the stable condition from *eq4.19* is clearly seen as $F_{\theta\phi} < 0$ if γ is positive. Fortunately for our case, this term is zero and there are not explicit conditions except for the case in *eq4.26* that $F_{\theta\theta}F_{\phi\phi} > 0$.

Thereafter the curve fitting procedures could be accomplished by varying the parameter H_1 . The fitted parameters of all the samples are illustrated in *Fig5.3*, *Fig5.4*, and *Fig5.5*, respectively. The H_2 is one tenth to one fifth of H_1 , which indicates in some instances the second anisotropy is important in Co-Cr films.

5.2. LINEWIDTH AND LINESHAPE

As has been pointed out before, the g -factor determined from *eqs4.26* and *4.27*, where the influence of relaxation was not taken into consideration, may prove to be anomalously large (Belson and Kriessman, 1959). According to *eq4.28*, which was assumed to be valid only for single crystal, the linewidth should be much larger for parallel directions than for perpendicular directions. This is not consistent with our data. The angular dependence of the linewidth is similar to

Fig5.3 The substrate temperature dependence of the first effective anisotropy constant measured with FMR.

Fig5.4 The substrate temperature dependence of the g-factor measured with FMR.

Fig5.5 The substrate temperature dependence of the second anisotropy constant measured with FMR.

some FMR data before: it is greatest for the easy magnetization axis and smallest for the hard magnetization axis(Yager *et al*, 1955).

Obviously such a Landau-Lifshitz relaxation model is not suitable to polycrystals. However qualitatively the influence of the broadening to the g factor can still be estimated with this model. To minimize this estimate from the inhomogeneous broadening, the smallest linewidth should be chosen which in this case is the perpendicular directions.

From eqs4.28 and 5.2,

$$\alpha = \frac{\Delta H_{pe}}{2(H_{pe} + H_1 - 4\pi M)} \quad (5.3)$$

where δH_{pe} is the linewidth in the perpendicular direction and the true g factor

$$g = g_{uncorrected} / \sqrt{1 + \alpha^2} \quad (5.4)$$

In our case, the α is too small to make any significant shift in the g factor. For example, for the sample with substrate temperature 192°C , $\alpha=0.07$, which does not alter g from 2.76. Here and below, the linewidth is defined as the peak to peak difference in magnetic field as usually defined in ESR, and referred to as peak-peak linewidth.

Generally the signal is unsymmetrical and the low side of the absorption line is more prominent as shown in *Fig5.6*. This is even true for the case when the sample has been saturated completely, which certainly excludes the possibility that this unsymmetry is due to the domain walls. The large anisotropy may account for this phenomenon. A large anisotropy usually can have two strong impacts on FMR. One is that this can lead to a second resonance peak, and the other is that in most cases a unsymmetrical peak is characteristic. It is known that under the condition of weak coupling between the crystallites, the absorption as a function of magnetic field should be essentially proportional to the number of crystallites that go through resonance at a given applied field. Because of the finite linewidth of a crystallite the observed absorption linewidth would not be exact but rather a smeared-out image of the calculated distribution. However, such an approximation is certainly valid for a qualitative comparison. So for large anisotropic polycrystals, the magnetization vector will deviate appreciably from the dc field with an inclination towards the nearest easy direction. As a consequence, the anisotropy field preferentially assists the dc field such that the

Fig5.6 A typical unsymmetrical FMR spectrum. This corresponds to the sample with $T_s = 192^\circ\text{C}$, measured at $\phi_H = 84^\circ$. The narrow resonance is due to the calibration sample.

low field side of the absorption curve becomes more prominent. Very similar absorption peaks have been detected by *Schlomann and Jones*(1959) as depicted in *Fig5.7*.

The next step is to study the angular dependence of linewidth. From the model of independent grains, this function can be generated according to *eq4.31* and compared to the experiment. This procedure is not completely satisfactory as shown in *Fig5.8*. Some relaxation mechanisms other than pure inhomogeneous broadening must exist and it is only possible to do a rough fit. This, in turn, makes it impossible to distinguish between the second and third terms in *eq4.29*. Since there is not available any information about the third term, this was neglected. Only the data around the perpendicular direction were attempted to be

Fig5.7 Measured and calculated absorption lines for $\text{Ba}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}(\text{Mg}_2\text{Y})$ measured at 15 967 Mc. The theoretical curve is valid for $g=2$, $2K_1/M=-3800$ Oe, $2K_2/M=-420$ Oe, $2K_4=120$ Oe, and $K_3=0$ Oe. (Schloman and Jones, 1959)

fit(Fig5.8), and a series of values of $\delta\phi_H$ are plotted in Fig5.9. The δH_0 were found to be zero for all the samples.

Let us now consider the discrepancies as seen in Fig5.8. To further clarify the problem of the eddy current loss, two sets of particular experiments were arranged. First, a small piece of the film cut from the previous film, which was small enough to eliminate eddy current loss if any, was tested under the same experimental conditions as before with the result that no appreciable change in linewidth was found. Second, the spectrum was taken where the film was placed on the bottom of the cavity with the substrate touching the bottom and also upside down with the film touching the bottom. The film was thus exposed to two different strengths of the microwave electric field, and if the eddy

Fig5.8 The measured and calculated angular dependence of peak-peak linewidth for the sample with $T_s=232^\circ\text{C}$. The solid curve is valid for $\delta H_o=0.0$ KOe, $\delta H_1=0.0$ KOe, and $\delta\phi_H=3^\circ$.

Fig5.9 The substrate temperature dependence of the c-axis dispersion angles measured with FMR.

current loss was responsible for part of the linewidth, the linewidths should have been significantly different. The results were found to be the same. Therefore, it is safe to conclude that the skin effect is not important in our case.

Maybe the only possible consistent explanation is due to the scattering by the domain walls. Principally when the resonance occurs before saturation is reached, the domain walls, if they existed, could broaden the FMR peaks significantly. At the angle ϕ_H far away from 90° , the resonance fields are relatively low; whereas as one approaches 90° , the resonance fields move to rather high field values. There are more domain structures at low fields and consequently more domain scattering. This certainly results in broader resonance peaks as is evident in our case. A quantitative analysis must be associated with

the detailed domain structure which is not available at the present time. A serious explanation of this angular dependence of the linewidth needs to be examined further, particularly Q band FMR should be employed to eliminate the effect of domain walls.

5.3. DOMAIN WALL

The magnetization process in crystals with domain structure can be classified into three parts as shown in *Fig5.10*.

The existence of domain walls may have a few implications on FMR. Certainly the first is that they can behave like a relaxation mechanism as already discussed above.

Secondly, the phenomenon known as domain resonance could happen. In general, one resonance peak is expected for each type of domain. Any component of the static magnetization perpendicular to the domain wall is continuous. However, the time-varying magnetization perpendicular to the wall may be out of phase and thus leads to uncompensated poles at the wall. These poles produce a demagnetization field with some demagnetization factor which couples the two magnetization vectors together (*Polder and Smit, 1953*) (*Fig5.11*). These resonance peaks as indicated in *Fig5.1* are believed to correspond to the domain wall resonances.

The irreversible domain wall movement can lead to irreversible FMR

Fig5.10 Magnetization process.

signals. To determine decisively the existence of domains, the scanning direction of dc magnetic field has been reversed, *i.e.*, the dc field was scanned from higher value to lower value. The reversibility at small resonance fields was generally high. This can be easily understood since these fields correspond to the reversible magnetization procedure in Fig5.10. But when the resonance fields were close to the fields in the range of irreversible domain wall movement, the large difference of the two lineshapes took place (Fig5.12a). This can be readily understood since the film should be in a state of more order in the domain structure when the applied field is scanned from high to low values than when the applied field is increased from low to high. It is well known that the FMR signals should be narrower and more symmetrical in the ordered state. Another consistent fact is that when the dc field was set even higher to reach the range

Fig5.11 The influence of domain structure on resonance.

a- the alternating magnetic field $\mathbf{h}$ is perpendicular to the static magnetization and the boundary layers.

b- the alternating magnetic field $\mathbf{h}$ is perpendicular to the static magnetization and parallel to the boundary layer.

of reversible rotation processes, the signal was reproducible again in the procedure described above as shown in Fig5.12b.

In conclusion, the films are most likely to have some kind of domain structure. Additional information is needed to be more specific in order to deal with this problem.

5.4. THE INTERPRETATION OF OTHER RESONANCE PEAKS

There is one more peak unexplained in the parallel direction. This still can be possibly attributed to the domain wall resonance. But, on the other hand, it is almost certain that all the Co-Cr films have some kind of transition layer,

Fig5.12a The irreversible resonance due to the irreversible domain wall movement. The arrows indicate the direction of dc field scanning. The narrow resonance is due to the calibration sample.

Fig5.12b The reversible resonance after the complete saturation. The arrows indicate the direction of dc field scanning. The narrow resonance is due to the calibration sample.

which has been identified by FMR(*Mitchell et al., 1985*).

Suppose the transition layer is thin and its magnetocrystal anisotropy is averaged to vanish as is usually done. Then it turns out that the resonance at a field of about 6.3 KOe can not be accounted for this layer unless the magnetization of this layer is assumed to be negative. However, if the assumption that the magnetocrystal anisotropy averages to be zero does not hold, then these resonances can be perfectly interpreted as due to the transition layer. (The concept of transition layer will be elaborated later). As an example, consider the analysis of the film with $T_s = 217^\circ\text{C}$. From *Fig5.1b*, $H_{pa} = 6.3$ KOe, and according to *eq5.1* with the neglect of H_2 and the assumption $g = 2.76$ as the bulk layer, $H_1 = 5.3$ KOe. This in turn gives $H_{pe} = -2.8$ KOe, which explains why the corresponding resonance is not detected in the perpendicular direction as shown in *Fig5.1c*. The relative volume of this transition layer can be estimated from the FMR curve. The area underneath a j constituent absorption curve is proportional to $4\pi M_j V_j$, where V_j is the relative volume. The derivative absorption curve may be roughly calculated by multiplying the vertical separation of the absorption derivative peaks by the square of the linewidth δH . The magnetization is reasonably assumed to be uniform. Then from the resonance curve, the volume of the transition layer is one third of the whole film.

5.5. DISCUSSION

5.5.1. "Transition Layer"

The primary question which arises is why there is such a huge inconsistency between the measurements of K_1 in VSM and FMR as can be compared from *Figs3.4* and 5.3. The saturation magnetization M_s can be determined fairly accurately and the volume averaged value is about 400 emu/cc. Then the effective anisotropy energy as defined in *Fig3.4* can be converted into the unit of magnetic fields by dividing $0.5M_s$, which would be $-2 \sim 2.2$ KOe while the counterparts in FMR are $-7 \sim -3.5$ KOe. In order to understand this discrepancy, a few possibilities will be discussed.

One of these derived from the so-called anhysteretic magnetization. As defined by Li et al(1986), the K_u in VSM is the area between the two curves, each of which is an average of the corresponding magnetization loop in the parallel and perpendicular directions, respectively. When the samples show considerable hysteresis as in our case when T_s is beyond 200°C (In *Figs3.2* and 3.3, the coercivity H_c and the ratio M_r/M_s become larger), the method of taking an average field becomes unreliable. It is then preferable to use anhysteretic magnetization which eliminates the contribution of irreversible processes to the magnetization energy. By the application of the alternating field the film is allowed to choose the state of lowest potential energy from the many metastable states that cause the hysteresis. Certainly this can not account for all of the discrepancy. This correction is estimated to be at most 1 KOe.

Secondly, the VSM tends to measure the whole averaged magnetization

and anisotropy while FMR can literally distinguish the different constituents. For example, the effective anisotropy is reduced from -6.0 KOe in FMR to -4.0 KOe in VSM/TM owing to this transition layer (Mitchell *et al*, 1985). In our case the film with $T_s=217^\circ\text{C}$ is a good example. The volume of the transition layer was estimated to be one third of the whole film, and the magnetization is considered to be uniform. The K_u from FMR is -4.5 KOe for the bulk layer and 5.3 KOe for the transition layer. Then the volume averaged anisotropy is calculated to be -1.6 KOe, fairly close to the value in VSM. This agreement with such a crude calculation is gratifying. The distorted weak signals prevent us from proceeding along this direction to treat other films.

5.5.2. A Phase Segregation Model

Now we should summarize the physical picture of phase segregation and the transition layer.

As discussed in chapter 2, it is generally believed that some phase segregation can exist in the Co-Cr films. From the structure analysis (sec 2.2.1) it is accepted that the films consist of columnar structure surrounded by a nonferromagnetic Co-rich region. The columnar shape is a very important factor which partially causes the positive effective anisotropy. The TEM investigation suggested a similar model but a finer microstructure (sec 2.2.2). In that study, it was suggested that phase segregation into ferromagnetic and nonferromagnetic phases in the hcp crystallites causes the compositional fluctuations along the grain boundaries as film growth proceeds. Finally, coherent ferromagnetic Co-rich

regions are formed in a crystallite with a wall-like structure which is perpendicular to the substrate. This model is different from above in that the phase segregation occurred in each crystallite having a single phase and results in a finer structure.

In order to interpret some previous results, an additional constituent was introduced, namely the transition layer. The existence of a transition layer located between the substrate and the bulk layer is certain from such results as SEM fracture cross section(*Ouchi and Iwasaki, 1982*) and electron diffraction patterns(*Hwang et al., 1986*). However, the correlation between this layer and the magnetic properties, particularly the anisotropy, are not well proved, although a number of papers have been devoted to this subject. The most frequently used methods are VSM and TM, which is not quite suitable to this problem. This insufficiency was noticed by Mitchell et al.(1985) and a series FMR reports were published which were mentioned briefly already in sec2.6.1.

They found that most of their samples had two resonances. One of them was attributed to the transition layer, which could occupy as much as half of the whole volume of the films. Two points are of interest in their study. They described that some films with poor polycrystalline alignment could demonstrate as many as four unexplained resonances, and they believed that their research represented the first known confirmation by FMR of positive anisotropy(0.7 KOe) in Co-Cr films. Before that time there were already several reports which claimed a large positive effective anisotropy(For example, 4 KOe by Fisher et al., 1984).

This investigation also presents some surprising results. Primarily the large positive effective anisotropy of the thick (150nm out of 500nm) "transition" layer is contrary to previous results. Usually, it is believed that the transition layer should only have negative shape anisotropy and the thickness is less than 50 nm for a film with 500 nm thickness. Also the failure of the fitting for the angular dependence of linewidth under the independent grain model seems to suggest a much more complicated microstructure associated with the film, one of which is phase segregation. Furthermore, the SEM graphs have not shown any thick transition layer.

Although in reality there may be a number of phases, it is useful to pursue a simple model such as a two phase model. Both phases are supposed to be ferromagnetic rather than only one of them as before so that two resonances in FMR should be detected. The scale of each magnetic unit may vary from film to film, and in some cases it can be much smaller than the grain size viewed through SEM. One of the phases has positive effective anisotropy(named positive phase) and the other, negative(negative phase). The different anisotropies in two phases are realized from the compositional fluctuation or the different shapes, which may not be universal. A transition layer could develop but in most cases, the influence of this layer is considered to be much weaker than that of these two phases.

This model can be applied to the discussion in last section and the results of Mitchell et al(1985) with the only substitution that the so-called "transition layer" actually now is a new phase. Moreover, this model allows the possibility

that a number of phases could form, which of course could explain the several resonances reported by Mitchell. The reason for the variety of results among different authors is understandable now since any phase segregation is quite sensitive to the preparation conditions.

5.5.3. Substrate Temperature Dependence

The most important deposition parameter to influence the phase segregation is surface temperature as explained in sec2.4.2. The variation of surface temperature should be intimately related to a) the substrate temperature, and b) the deposit rate. A higher deposit rate would lead to a higher surface temperature.

The surface temperature should be classified into three ranges according to the phase segregation model:

- [1] low temperature: the phase segregation should not happen and the temperature is too low to allow Co/Cr atoms to form hcp structure with positive anisotropy;
- [2] medium temperature: the film begins to form some hcp structure and at the same time phase segregation is starting to take place. As the temperature goes higher, more negative phase would be formed;
- [3] high temperature: if the temperature is too high, then Cr/Co diffuse out of alloy causing a reduction in positive phase. Finally, even the atomic hcp lattice structure could be destroyed and fcc lattice would show up.

There is a suitable temperature range in which films with positive effective anisotropy could be made.

In our case, $T_s = 200^\circ\text{C}$ seems to represent the transition from [2] to [3]. The K_u of one phase corresponding to the "bulk" layer increases monotonically in *Fig5.3*, and the resultant anisotropy goes from positive to negative when the substrate temperature is increased. The tail in *Fig3.4*, which turns positive again, is believed to be due to some other causes and one possibility could be anhysteretic magnetization. The anomalies of K_2 and g factors may also be attributed to this but not enough information is available to make any definite conclusions. Also this model predicts a lower substrate temperature would improve the properties of these kinds of films.

5.5.4. Comparison With Other Studies

The coercivity has a maximum in the study of Coughlin et al.(1981) within the range of 200 to 300 $^\circ\text{C}$ substrate temperature while the coercivity goes up as the T_s is increased from 0 to 200 $^\circ\text{C}$ as shown by Wieldingn and Lodder(1981). The former is consistent with our result generally, although a precise comparison is hardly possible. The first anisotropy constant spreads over a very large range while our K_2 is quite close to the only available value at the same composition obtained by Fisher et al.(1984) with TM: at Cr 20.5 at. %, 0.44×10^5 ergs/cc. Our K_2 are distributed from -0.8 to $+0.8 \times 10^5$ ergs/cc.

CHAPTER 6. CONCLUSIONS AND SUGGESTIONS

The Co-Cr films are quite inhomogeneous with possibly two ferromagnetic phases. FMR can potentially sort out different constituents in the films. The angular dependence investigation gives the possibility of determining the second anisotropy and g factor. The discrepancy between VSM and FMR, which may be quite common, is qualitatively explained with the proposed two ferromagnetic phases. This model also is consistent with the magnetic properties of substrate temperature dependence, which is very sensitive to preparation parameters. Moreover, this may help understand the diverse properties occurring under different deposition conditions.

As for the future, Q band FMR could be employed to investigate properties such as the existence of domain wall resonance and the transition layer. The angular dependence of the linewidth could also be studied further.

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