Development of L1o FePt (001) thin films for perpendicular magnetic recording media

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Development of $L1_0$ FePt (001) thin films for perpendicular magnetic recording media

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Abstract

Chemically ordered $L1_0$ (CuAu-I type structure) FePt films with an fct (face centered tetragonal) structure possess a high K_u (magnetic anisotropy constant) value ($\sim 6-10 \times 10^7$ erg/cm³), which allows them to have very small thermally stable magnetic grains (~ 2.6 nm) and makes them the most promising candidates for ultra-high areal density recording media. Usually, as-deposited FePt alloy thin films are a disordered fcc (face-centered-cubic) phase and tend to show a (111) texture. A high temperature (above 550°C) post annealing is needed to form an ordered $L1_0$ phase. In order to develop FePt films as perpendicular magnetic recording media, the $L1_0$ FePt ordering temperature has to be reduced and the easy axis of the films should be perpendicular to the film surface. Moreover, the grain size and exchange coupling between grains should be decreased. Thus, in this thesis, the research has been focused on two main issues (1) how to lower the $L1_0$ FePt ordering temperature when getting the crystallographic texture controlled at the same time and, (2) how to decrease the grain size and inter-granular exchange coupling.

The first part of the thesis focuses on the study of the intrinsic relationships between the chemical ordering, lattice constant, and anisotropy energy of the FePt films. The effect of the lattice mismatch between underlayer and FePt film on the chemical ordering of the FePt films was studied. A critical lattice mismatch near 6.33% was found to be the most favorable for chemical ordering. It was demonstrated that the residual stress helped to expand the a-axis and shrink the c-axis of the FePt films and thus favored the chemical

ordering of the FePt films at relatively low temperatures. An epitaxial stress-induced ordering and textures control mechanism was proposed to lower the ordering temperature of the $L1_0$ FePt thin films.

Based on the proposed and demonstrated ordering temperature reduction and orientation control mechanism, the second part of the thesis focuses on the study of the structural and magnetic properties of the FePt films grown on the CrX alloy (X=Mo, Ru, Ti, W) underlayers. It was found that the texture of the FePt films strongly depended on the texture of the CrX underlayers. Cr (200) texture and substrate temperature (T_s) were the key parameters for the growth of the FePt (001) films.

To decrease the grain size and keep the (001) preferred orientation in the FePt films, the effect of the Carbon additive on the structural and magnetic properties of the FePt films was also studied. A good FePt (001) texture was maintained even after incorporation of C up to 20 vol.% by a fine control of the sputter process. It was also found that a small amount of C doping could slightly enhance the ordering degree, while a large amount of C doping could prohibit the ordering degree. Well-defined grain boundaries and relatively uniform grains with a mean diameter of 5.6 nm and a standard deviation of 1.6 nm were obtained by doping 20 vol.% CFlyable disks using the developed FePt:C perpendicular media were fabricated, and read-write tests were carried out by a Guzik spin-stand. The signal to noise (SNR) ratios of the FePt:C media doped with 20 vol.% C were about 10 dB better than that of the pure FePt media.

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Table of Contents

Abstract 1

Acknowledgements iii

Table of Contents iv

List of Figures ix

List of Tables xv

Terminology ad symbols xvi

Chapter 1 Introduction 1

1.1 Background.....1

1.2 Research status and problem statements of FePt media 2

1.3 Objective.....5

1.4 Scope and Methodology 5

1.4.1 Orientation and ordering temperature control 5

1.4.2 Grain size and exchange coupling control.....7

1.4.3 Methodology 8

1.4.4 Project approaches 9

1.5 Novelty 10

1.6 Organization of the thesis 11

Chapter 2 Literature review 13

2.1 Longitudinal Magnetic Recording.....13

Thesis	Table of Contents
2.1.1	Characteristics of Longitudinal Magnetic Recording.....13
2.1.2	Longitudinal Magnetic Recording Media 15
2.1.3	Issues for Longitudinal Recording 16
2.1.4	Potential Solutions 17
2.2	Perpendicular Magnetic Recording 18
2.2.1	Characteristics of Perpendicular Magnetic Recording 18
2.2.2	Basic Process in Perpendicular Recording 21
2.2.3	Comparison with Longitudinal Recording 25
2.2.4	Advantages of Perpendicular Magnetic Recording 28
2.2.5	Challenges for Perpendicular Magnetic Recording 29
2.2.6	Requirements for Perpendicular Recording Media 34
2.2.7	Candidate Materials for Perpendicular Recording Media 35
2.2.7.1	CoCr-based alloys.....36
2.2.7.2	Co/Pd and Co/Pt multilayers.....37
2.2.7.3	FePt thin films 39
2.3	FePt alloys 40
2.3.1	Physical properties 40
2.3.2	Crystallography: 41
2.3.3	Magnetic Properties.....41
2.3.4	The current status as a recording media 42
2.3.5	Methods for producing $L1_0$ FePt (001) films 42
2.3.6	Methods for lowering the ordering temperature of $L1_0$ FePt films 44
2.3.7	Methods for controlling the grain size in $L1_0$ FePt films 45
2.3.7.1	Nano-layer engineering 45
2.3.7.2	Self-Organized-Magnetic-Arrays 46
2.3.7.3	Nano-cluster.....48
2.4	Model for formation of crystallographic texture of Cr thin films 49
Chapter 3 Experimental Details 52	
3.1	Sputter deposition 52
3.1.1	Introduction to sputter deposition.....52
3.1.2	Experimental procedure 54
3.2	Structure Characterization 57

Thesis	Table of Contents
3.2.1 X-Ray Diffractometer	57
3.2.1.1 Measurement of crystal orientation	57
3.2.1.2 Order-disorder transformations characterization	60
3.2.2 Transmission Electron Microscopy	62
3.2.3 Experimental procedure	64
3.3 Magnetic characterization.....	68
3.3.1 Vibrating Sample Magnetometer measurement	68
3.3.2 Alternating Gradient Force Magnetometer	72
3.3.3 Magnetic Recording Properties Characterization: Read-write testing	73
3.3.4 Magnetic Force Microscope.....	74
Chapter 4 Effects of lattice mismatch on chemical ordering in epitaxial L1₀ FePt films	77
4.1 Introduction	77
4.2 Sample structure	78
4.3 Results and discussion	80
4.3.1 XRD spectra.....	80
4.3.2 Epitaxial relationship demonstration with high resolution X-ray diffraction	88
4.3.3 Microstructural properties	90
4.3.4 Magnetic properties.....	95
4.3.5 Influence of lattice mismatch on chemical ordering	97
4.3.6 Residual stress in epitaxially grown FePt thin films	100
4.4 Conclusion	104
Chapter 5 FePt films deposited on chromium intermetallic underlayers	106
5.1 Introduction	106
5.2 Sample structure	107
5.3 Results and discussion	107
5.3.1 Influence of Mo content	107
5.3.2 Influence of Cr ₉₀ Mo ₁₀ underlayer thickness.....	109

Thesis	Table of Contents
5.3.3	Influence of substrate temperature 112
5.3.4	Influence of FePt film thickness..... 119
5.3.5	Microstructure of FePt films with different underlayer and magnetic layer thicknesses 124
5.3.6	Influence of different underlayers 126
5.3.7	Initial layer in FePt films grown on Cr ₉₀ Ru ₁₀ (200) underlayers..... 130
5.4	Conclusion 136
Chapter 6 FePt:C (001) nanocomposite films with ultrafine and uniform grains... 139	
6.1	Introduction 139
6.2	Sample structure 141
6.3	Influence of C additive on FePt films..... 142
6.3.1	Structural properties of FePt:C films..... 142
6.3.2	Magnetic properties..... 158
6.3.2.1	Hysteresis loops 158
6.3.2.2	Magnetization reversal mechanism..... 160
6.3.2.3	Switching field distribution 162
6.3.2.4	Read-Write test on FePt:C perpendicular media..... 165
6.4	Influence of substrate temperature 167
6.4.1	Structural properties 168
6.4.2	Magnetic properties..... 169
6.4.3	Microstructural properties 171
6.5	Conclusion 174
Chapter 7 Summary and conclusions 176	
7.1	Summary..... 176
7.2	Conclusions 177
Appendix A: Standard crystallographic XRD data 181	

<u>Thesis</u>	<u>Table o f Contents</u>
Appendix B: Phase diagram of Fe-C alloy	182
Appendix C: Phase diagram of Cr-Mo alloy.....	183
Appendix D: Phase diagram of Cr-W alloy	184
List of Publications	185

List of Figures

Figure 1-1 Schematic of film strain induced by lattice mismatch.	6
Figure 1-2 Schematic epitaxial relationship between Cr underlayer and FePt film.	7
Figure 2-1 Schematic of a longitudinal recording system. B is the bit length, W is the track width, t is the medium thickness, and d is the flying height of the head above the medium. ³⁰	14
Figure 2-2 (a) Magnetization of two transitions at $x = 0$ and 200 nm. Medium thickness is 15 nm and the transition parameter is 17 nm. (b) Perpendicular magnetic field, which is detected by the read head, at 10 nm distance from the surface of (a) longitudinal (—) and (b) type 1 perpendicular (----) media. PW_{50} is shown for a read head with zero gap. PW_{50} is larger for heads with finite gaps. ³¹	15
Figure 2-3 TEM image of a CoCrPtB recording medium, where the amorphous non-magnetic Cr rich boundaries magnetically separating the Co rich grains are visible as light gray areas. The inset schematically shows a magnetic transition meandering between the grains. ³²	16
Figure 2-4 Schematic process in perpendicular magnetic recording	22
Figure 2-5 Schematic type 1 perpendicular recording system with SUL and a single pole head. ⁵⁰	25
Figure 2-6 Transition regions: (a) perpendicular recording and (b) longitudinal recording	26
Figure 2-7 A simple model for magnetization regions in perpendicular and longitudinal recording. ⁵⁶	28
Figure 2-8 Iron-platinum phase diagram for alloys (from Watanabe et al. 1983)	40
Figure 2-9 Crystal structure for the disordered and ordered phases of FePt equiatomic alloy. ⁷⁶	41
Figure 3-1 Schematic cross-section image of a typical sputtering system ¹⁰²	53
Figure 3-2 DSI home-designed UHV sputter system	55
Figure 3-3 Schematic diagram of the UHV sputter system	56
Figure 3-4 Schematic illustration of the θ -2 θ scanning mode.	59

Figure 3-5 Illustrations of different off-spectral scan models.	59
Figure 3-6 Schematic ray diagrams of transmission electron microscope (TEM).	66
Figure 3-7 Schematic TEM work modes (a) bright field image, (b) low resolution dark field image, (c) high resolution dark field image, and (d) Electron diffraction pattern.	67
Figure 3-8 A schematic of a VSM.....	69
Figure 3-9 Typical hysteresis loop and parameters measured by VSM. ¹⁰⁸	70
Figure 3-10 A typical schematic DCD curve.	72
Figure 3-11 (a) Basic concepts of MFM (b) Geometry used in calculating the magnetic interaction (c) principle of AC force microscopy.....	75
Figure 3-12 Schematic of AFM with tapping mode.....	76
Figure 4-1 Illustration of sample structure.	78
Figure 4-2 Epitaxial relationship models used in this thesis.	79
Figure 4-3 XRD spectra of samples: (a) A, (b) B, (c) C, (d) D, and (e) E.	82
Figure 4-4 Lattice parameter of $\text{Cr}_{1-x}\text{Mo}_x$ with respect to Mo content.....	83
Figure 4-5 FePt (001) rocking curves of samples A, C, D and E.....	83
Figure 4-6 Off-specular $\theta/2\theta$ scans and rocking curves of samples A, B, C, D, and E.....	87
Figure 4-7 Lattice constants of FePt films with respect to different buffer layers.....	87
Figure 4-8 Off –specular phi scans of samples (a) E, (b) A, and (c) B.	90
Figure 4-9 TEM images (a) low magnification cross-sectional image of sample with the structure MgO/Pt (30 nm)/FePt (50 nm); (b) corresponding SAED pattern of (a) ; (c) simulated EDP of MgO (200) [electron zone axis [001]]; (d) simulated EDP of Pt (200) [electron zone axis [001]]; (e) simulated EDP of FePt (001) [zone axis [001]]; (f) high magnification interface between MgO substrate and Pt layer; and (g) interface between FePt and Pt layers.	93
Figure 4-10 (a) low magnification cross-sectional image of sample C; (b) interface between $\text{Cr}_{95}\text{Mo}_5$ and MgO; (c) interface between $\text{Cr}_{95}\text{Mo}_5$ and FePt; and (d) interface between FePt and MgO (sample E).	94
Figure 4-11 Hysteresis loops of samples A, B, C, D, and E.....	96
Figure 4-12 MFM images of samples A, C, and E. [Scale: $5\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$]	97

Figure 4-13 Lattice constants of a and c , tetragonality represented by c/a , $(I_{001}/I_{002})^{1/2}$, and K_u as a function of lattice mismatch.	99
Figure 4-4-14 $d(\psi)-\sin^2\psi$ curves of FePt films deposited on MgO/X (X= Pt, Cr) with different FePt film thicknesses. The unit of the Y axis of all the figures is Angstrom.	103
Figure 4-15 Thickness dependence of residual stress in FePt films.	104
Figure 4-16 Lattice mismatch dependence of residual stress in FePt films.	104
Figure 5-1 Illustration of sample structure.	107
Figure 5-2 XRD spectra of FePt films deposited at 350°C on Cr underlayer (a), Cr ₉₀ Mo ₁₀ underlayer (b), and Cr ₈₀ Mo ₂₀ underlayer (c).	108
Figure 5-3 Hysteresis loops of FePt films deposited at 350°C on Cr underlayer (a), Cr ₉₀ Mo ₁₀ underlayer (b), and Cr ₈₀ Mo ₂₀ underlayer (c).	110
Figure 5-4 XRD spectra of FePt films deposited at 350°C on Cr ₉₀ Mo ₁₀ underlayers with different Cr ₉₀ Mo ₁₀ layer thicknesses.	112
Figure 5-5 XRD spectra of FePt films deposited on Cr ₉₀ Mo ₁₀ underlayers at various substrate temperatures (150°C ~ 350°C). The reference spectrum is from a 4 nm thick Pt film deposited on Cr ₉₀ Mo ₁₀ underlayer at 350°C.	113
Figure 5-6 SAED patterns of FePt films deposited on Cr ₉₀ Mo ₁₀ underlayers at 150°C, 200°C, 300°C, and 350°C, respectively. [Note: 1: FePt (001); 2: FePt (110); 3: FePt (111); 4: Cr (110); and 5: FePt (200), (002)].	115
Figure 5-7 TEM micrographs of FePt films deposited on Cr ₉₀ Mo ₁₀ underlayers at 150°C (a) and (b), and 350°C (c) and (d). Here (a) and (c) are the bright-field micrographs and (b) [taken from FePt (200)] and (d) [taken from FePt (110)] are the dark-field micrographs.	116
Figure 5-8 Out-of-plane and in-plane hysteresis loops of FePt films deposited on Cr ₉₀ Mo ₁₀ underlayers at various substrate temperatures. FePt film deposited on glass substrate is included as a reference.	117
Figure 5-9 $(I_{001}/I_{002})^{1/2}$, lattice parameter, c , out-of-plane coercivity, H_c , and magnetic anisotropy, K_u , as a function of substrate temperature.	118
Figure 5-10 XRD spectra of FePt films with different thicknesses deposited on Cr ₉₀ Mo ₁₀ underlayers at 350°C.	120

Figure 5-11 Out-of-plane and in-plane hysteresis loops of FePt films with different thicknesses.	121
Figure 5-12 $(I_{001}/I_{002})^{1/2}$ and out-of-plane H_c ($H_c(\perp)$) as a function of FePt film thickness.	122
Figure 5-13 Angular dependence of coercivity with respect to FePt film thickness.....	123
Figure 5-14 TEM images of (a) 10 nm and (b) 80 nm thick FePt films deposited on 30 nm thick $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at 350°C; and 20 nm thick FePt films deposited on (c) 5 nm and (d) 20 nm thick $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at 350°C. The marked rings are FePt (110).....	125
Figure 5-15 TEM cross-sectional images of (a) 15 nm and (b) 60 nm thick FePt films deposited on 25 nm thick $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at 350°C.	126
Figure 5-16 XRD spectra of 20 nm thick FePt films grown on 20 nm thick CrX underlayers.....	127
Figure 5-17 Hysteresis loops of FePt films grown on (a) $\text{Cr}_{90}\text{Ru}_{10}$, (b) $\text{Cr}_{90}\text{Mo}_{10}$, (c) $\text{Cr}_{90}\text{W}_{10}$, and (d) $\text{Cr}_{90}\text{Ti}_{10}$ underlayers.	129
Figure 5-18 XRD spectra of FePt films with different thicknesses on the $\text{Cr}_{90}\text{Ru}_{10}$ underlayers: (a) without Pt buffer layers and (b) with Pt buffer layers.	132
Figure 5-19 TEM electron diffraction patterns of $\text{Cr}_{90}\text{Ru}_{10}$ underlayer and 3 nm thick FePt film grown on the $\text{Cr}_{90}\text{Ru}_{10}$ underlayer.	132
Figure 5-20 Hysteresis loops of FePt films of different thicknesses grown on $\text{Cr}_{90}\text{Ru}_{10}$ underlayers without a Pt buffer layer.	133
Figure 5-21 Hysteresis loops of FePt films of different thicknesses grown on $\text{Cr}_{90}\text{Ru}_{10}$ underlayers with a Pt buffer layer.....	134
Figure 5-22 Out-of-plane H_c as a function of FePt film thickness.	134
Figure 5-23 Depth dependence of M_s distribution.	136
Figure 6-1 Structures of FePt:C films (a) and media (b).....	141
Figure 6-2 XRD spectra of FePt films with different C volume fractions at 350°C.	143
Figure 6-3 Rocking curves of FePt (001) peaks with different C contents.	144
Figure 6-4 Long-range chemical ordering degree as a function of C content.....	146

Figure 6-5 TEM plane-view images of FePt:C films with (a) 10 vol.% and (b) 20 vol.% C doping. (a)* and (b)* are the corresponding grain size distributions of (a) and (b), respectively.....	147
Figure 6-6 TEM cross-sectional images of the 15 nm thick FePt:C film doped with 10 vol.% C: (a) low magnification and (b) high resolution.....	150
Figure 6-7 TEM cross-sectional images of the 10 nm thick FePt:C film doped with 20 vol.% C: (a) low magnification and (b) high resolution.....	151
Figure 6-8 TEM cross-sectional images of about 10 nm thick FePt:C film doped with 30 vol.% C: (a) low magnification and (b) high magnification.....	152
Figure 6-9 Proposed two-step growth model for FePt:C films. (a) Step 1: FePt is epitaxially grown on CrRu/Pt and carbon is squeezed onto the top surface of FePt film. (b) Step 2: With increased film thickness, carbon is accumulated to some extent, and the epitaxial stress is released. Fe and Pt atoms nucleate on some sites and then the nuclei grow larger.	153
Figure 6-10 Typical TEM cross-sectional images of (a) 5 nm thick, low magnification, (b) 5 nm thick, high resolution, (c) 15 nm thick, and (d) 30 nm thick FePt:C films (15 vol.% C) grown on 30 nm thick Cr ₉₀ Ru ₁₀ underlayers with 2 nm thick Pt buffer layers deposited at 350°C.	156
Figure 6-11 Typical XPS profiles of (a) Fe (2p), (b) C (1s), and (c) Pt (4f).	157
Figure 6-12 Out-of-plane hysteresis loops of FePt:C films with different C contents....	159
Figure 6-13 Angular dependence of the coercivity profiles of the FePt:C films doped with 10, 20 and 30 vol.% C. (0° refers to the film normal direction).....	161
Figure 6-14 MFM images of FePt:C media doped with 5, 10 and 20 vol.% C, respectively. [Scale: 3 μm $\times$ 3 μm].....	162
Figure 6-15 DCD curves and corresponding differential curves of the FePt:C media doped with 0, 5, 10 and 20 vol.% C.	164
Figure 6-16 SFD calculated from Fig. 6.14 with respect to C content.....	164
Figure 6-17 Reproduced waveforms of individual track at various densities for the FePt:C media doped with 20 vol.% C.	166
Figure 6-18 SNR with respect to linear density for pure FePt media and FePt:C media doped with 20 vol.% C.	167

Thesis

List of Figures

Figure 6-19 XRD spectra of FePt:C (15 vol.% C) films grown on Cr₉₀Ru₁₀ underlayers at different substrate temperatures..... 169

Figure 6-20 Hysteresis loops of FePt:C (15 vol.% C) films deposited at different substrate temperatures..... 170

Figure 6-21 Coercivity with respect to substrate temperature for FePt:C films with 15 vol.% C. 171

Figure 6-22 TEM cross-sectional images of FePt:C (15 vol.% C) films deposited at (a) 200°C, low magnification, (b) 200°C, high resolution, (c) 250°C, (d) 300°C, and (e) 350°C, low magnification, and (f) 350°C, high resolution. 174

List of Tables

Table 2-1 UHDR roadmap for PMR from 100 to 1000 Gb/in².....33

Table 2-2 Magnetic properties and theoretical minimal grain diameters of various
perpendicular media candidates.....35

Table 4-1 Lattice constants, epitaxial relationships, and lattice mismatches.....81

Table 4-2 Lattice mismatch and K_u of all the samples.....95

Table 5-1 Structural properties of both the underlayers and FePt films.....127

Table 5-2 H_c (⊥) K_u and S (⊥) of FePt films grown on different underlayers.....129

Table 6-1 Compositions of FePt:C films 140

Table 6-2 H_{c⊥}, H_n, S and S* of the FePt:C media.....159

Terminology ad symbols

α	Loop slope parameter
$\langle D \rangle$	Average grain size or grain diameter (nm)
AFM	Atomic Force Microscope
AGFM	Alternation Gradient Force Magnetometer
bcc	Body-centred Cubic
D_p	Grain Diameter
DSI	Data Storage Institute
Easy axis	Axis along which a ferromagnetic material can be magnetized easily
ΔE	Uniaxial energy barrier
f_0	Frequency
fcc	Face-centered Cubic
fct	Face-centered Tetragonal
FWHM	Full-width at half maximum (degree)
H_c	Coercivity (Oe)
$H_{c(\perp)}$	Perpendicular Coercivity (Oe)
$H_{c(\parallel)}$	In-plane Coercivity (Oe)
H_d	Demagnetization Field (Oe)
H_o	Media Switching Field (Oe)
HRXRD	High-resolution X-ray Diffractometer
HRTEM	High-resolution Transmission Electron Microscopy
k	Boltzmann's constant

Thesis	Terminology and symbols
K_u	Magnetic Anisotropy Energy (erg/cc)
kfc	kilo flux change per inch
ktpi	kilo track per inch
$L1_0$	Cu-Au I type Structure (one layer A atoms followed by one layer B atoms)
LMR	Longitudinal Magnetic Recording
M_r	Remanent Magnetization (emu/cc)
M_s	Saturation Magnetization (emu/cc)
MFM	Magnetic Force Microscope
MOKE	Magnetic Optical Kerr Effect
NTU	Nanyang Technological University
OSA	Optical Surface Analyzer
PMR	Perpendicular Magnetic Recording
S	Squareness (M_r/M_s)
SFD	Switching Field Distribution (Oe)
SPM	Scanning Probe Microscopy
S^*	Coercivity squareness
SOMA	Self Organized Magnetic Arrays
SUL	Soft underlayer
t_{FePt}	FePt layer Thickness (nm)
VSM	Vibrating Sample Magnetometer
XRD	X-Ray Diffractometer
SNR	Signal-to-noise ratio
SPT	Single Pole Head

Thesis

Terminology and symbols

T	Absolute temperature (K)
Tc	Critical temperature (°C)
Ts	Substrate temperature (°C)
UHDR	Ultra-high density recording (Gb/in ²)
V	Magnetic volume
w	Read track width

Chapter 1 Introduction

1.1 Background

The storage density of hard disk drives increased by over 100% per annum during the late 1990s,¹ 100 Gb/in² laboratory demonstrations were reported in the year 2002,² and 230 Gb/in² laboratory demonstrations were reported recently by Hitachi. The increases in data density, data rate, and other performance metrics have generally been achieved by scaling to make the read-write head smaller in size, the media thinner in thickness and higher in coercivity, and the head-medium spacing smaller. The performance of a thin film medium is limited by the noise originated from the granular microstructure of the film, so there has been a trend to decrease the grain size of the recording layer. However, for currently used Co-based longitudinal media, when the grain size decreases down to below 8 nm, the thermal energy stored in the grains will be sufficient to allow their magnetization to reverse spontaneously, with a consequent loss of the recorded signals (so called superparamagnetism). In order to overcome the thermal instability, high K_u (uniaxial anisotropy constant) materials are needed. Increasing K_u , however, also increases the media switching field, H_0 , which is proportional to the ratio K_u/M_s (M_s = saturation magnetization), and will exceed the write field capability of the head (the write field capability is believed to be near 5 kOe for longitudinal recording). Thus, an alternative is needed to achieve ultra-high data storage and a new recording medium with high K_u is needed to achieve a higher recording density.

Perpendicular recording has long been advocated as a means of achieving ultra high areal densities. In particular, in the context of the “superparamagnetic limit”, perpendicular recording with a soft underlayer promises several key advantages. These advantages include a higher coercivity and a thicker media that should be able to permit smaller diameter grains and a higher signal- to- noise ratio (SNR). In addition, the sharper edge-writing will facilitate recording at very high track densities (lower bit aspect ratio). More details will be discussed in Chapter 2. Simulation work also showed that the recording density could be beyond 1 Tbits/in² with perpendicular recording media.⁴ During this project, a high recording density of about 230 Gb/in² on CoCr-based alloy perpendicular media has been demonstrated by Hitachi in 2005. In addition, hard disk drive products with a recording density of about 170 Gb/in² based on perpendicular media and technology will move into market at the end of 2005 by Seagate.

As for the candidate perpendicular media materials, chemically ordered L1₀ (CuAu-I) FePt films with an fct (face centered tetragonal) structure possess a high K_u value ($\sim 6 \cdot 10 \times 10^7$ erg/cm³) which allows them to have very small magnetically stable grains (~ 2.6 nm) and makes them the most promising candidates for the ultra-high areal density recording media. More details will be introduced in Chapter 2.

1.2 Research status and problem statements of FePt media

Usually, as-deposited FePt alloy thin films are a disordered fcc (face-centered-cubic) phase and tend to show a (111) texture. A high temperature (above 550°C) post annealing

is needed to form an ordered $L1_0$ structure ($L1_0$: Cu-Au I structure).^{3,4} In order to develop FePt films as perpendicular magnetic recording media, the $L1_0$ FePt ordering temperature has to be lower than 400 °C and the easy axis should be perpendicular to the film surface. Moreover, the grain size and exchange coupling between grains should be decreased.

Much effort has been made to synthesize the ordered $L1_0$ FePt (001) films using various techniques, which can be roughly sorted as follows: (a) molecular beam epitaxial (MBE) deposition on a MgO (001) single crystal substrate at an elevated temperature (>200 °C);⁵ (b) in-situ sputtering deposition or laser ablation on a MgO (001) single crystal substrate at an elevated temperature or post annealing of the FePt films on a MgO substrate;^{6,7} (c) in-situ sputtering deposition on Ag (100)/Si(100) or Cr(100)/MgO/substrate at an elevated temperature (>400 °C);^{8,9,10} and (d) direct deposition of FePt alloy films or FePt-B₂O₃ nanocomposite films on a Si substrate with a layer of SiO₂, and then followed by post-annealing.¹¹ For methods (a) and (b), though fct-FePt films with (001) texture can be obtained at about 200°C, a complex MBE technique and expensive MgO (001) single crystal substrates have limited their practical applications. With method (c), a very thick underlayer such as Ag (100), Cr (100)/MgO and a high temperature (above 450°C) are required to obtain a good (001) texture. Although the $L1_0$ ordered FePt (001) films can be obtained by the non-epitaxial method (d), it needs a high post-annealing temperature (above 550°C) and a long annealing time, which are not compatible with the industrial processes. Though the films could become ordered at 300°C by incorporation of 15 vol.% Cu into FePt films, the crystallographic orientation was (111).¹² Methods (a), (b) and (c) show that the texture of the FePt film can be well controlled and the chemical ordering

temperature can be reduced by using an appropriate underlayer. It is deduced that the lattice mismatch at the interface between FePt film and underlayer may expand the a axis and shrink the c axis of the FePt film and thus favors the ordering at relatively low temperatures. Thus, a further understanding of the relationship between lattice mismatch and chemical ordering is of great scientific and technological interest, which is also one of the objectives of this research.

The decreases of the grain size and exchange coupling are usually achieved by adding a third material into the films. Materials such as AlO_x ,¹³ Al_2O_3 ,¹⁴ AlN ,¹⁵ TaN ,¹⁶ Ag ,¹⁷ W , and Ti ¹⁸ have shown to be promising in controlling the FePt grain size in ordered films. Other studies involving FePt with Ni , B , BN , or C ^{19,20} as additives have demonstrated that these can reduce the magnetic coupling. Oxide addition to the FePt films can provide a better grain size control and reduce magnetic coupling in the films. Luo et al. reported well-separated grains with a strong perpendicular orientation in nanostructured $\text{FePt/B}_2\text{O}_3$ multilayers.¹¹ FePt/SiO_2 multilayers showed a limited grain size of 10 nm but a poor orientation.⁴ HfO_2 and MnO additives were studied by Platt et al.,²¹ where the HfO_2 additive reduced the grain size with a random texture, while the MnO additive affected neither grain size nor L1_0 ordering of the annealed FePt films. However, the above experiments also suffered from a high temperature post-annealing treatment.

The current key issues for the application of FePt films for recording media are how to obtain a good fct-FePt (001) orientation at relatively low temperatures and, at the same time, how to decrease the grain size and exchange coupling of the FePt films.

1.3 Objective

The objectives of this research are as follows: To investigate the relationship between the lattice mismatch and the chemical ordering of epitaxial FePt films grown on MgO (100) single crystal substrates with different buffer layers; To control the easy axis of the FePt films to be perpendicular to the film surface; To lower the ordering temperature for the chemical ordering down to 350°C; To decrease the grain size of the magnetic layers to several nanometers on the glass substrates with nano-layer engineering methods; and to fabricate flyable disks based on the FePt (001) thin films developed in this study.

1.4 Scope and Methodology

1.4.1 Orientation and ordering temperature control

The idea for orientation control and lowering the ordering temperature is based on the following principle. When a thin film is deposited on a substrate with a lattice constant different from that of the film, there exists a strain between the substrate and the film. If the lattice constant of the substrate is larger than that of the film, the strain will expand the lattice of the film along the interface between the film and substrate, and compress the film lattice along the normal to the interface and thus make the film fitted to the substrate [Fig.1.1]. The c axis of fct-FePt is slightly smaller than that of the a axis ($c/a=0.968$), so it is expected that an underlayer with a lattice constant larger than that of the FePt may be able to aid the phase transformation from fcc-FePt to fct-FePt at relatively low temperatures.

In this study, Cr based alloy underlayers are to be developed to induce the texture of the FePt films based on the proposed epitaxial relationship, FePt (001)[100]||Cr (002)[110], as shown in Fig.1.2. The lattice misfit between FePt (001)[100] and Cr (002)[110] is about 5.8%. In addition, it was reported that by incorporating some elements into Cr films during deposition the lattice constant of Cr increased and thus the strain between the Cr underlayers and FePt layers became adjustable.²² Thus, it is expected that the fct-FePt films with (001) texture would be obtained at low temperatures with CrX underlayers by conventional magnetron sputtering.

Figure 1-1 Schematic of film strain induced by lattice mismatch.

Figure 1-2 Schematic epitaxial relationship between Cr underlayer and FePt film.

1.4.2 Grain size and exchange coupling control

As introduced in section 1.2, the decreases of grain size and exchange coupling are usually achieved by adding a third material into the films. The goal of this project is to develop FePt thin films as perpendicular recording media. Thus, one basic requirement for choosing a third element or alloy as an additive is to keep the (001) preferred orientation in the FePt films after doping. In our previous work, the easy axis of the FePt films grown on the CrRu/Mo underlayers changed from perpendicular to longitudinal by Ag²³ and Cu²⁴ doping. Though the reasons why the orientation changed after Ag and Cu doping are still not very clear yet, the suppressed mobility of the Fe and Pt adatoms should be one important reason. In this thesis, C (carbon) additive is selected to control the grain size and exchange coupling in the FePt (001) films based on the following considerations.

- a) The atomic radius of Carbon (0.077 nm) is much smaller than that of Pt (0.138 nm) and Fe (0.126 nm). Therefore, it is very likely that C atoms can diffuse through interstitial sites as in the case of diffusion in CoCr based alloys,²⁵ and the FePt films can epitaxially grown on the underlayers.
- b) Though the atomic radius of Boron (0.097 nm) is also much smaller than that of Pt and Fe, FePtB longitudinal media showed improved SNR and nonlinear transition shift (NLTS) than FePt media due to a reduced inter-granular exchange coupling,¹⁹ Boron can form alloys with Fe, which is not favorable for grain size reduction. In addition, a Boron additive in sputter target may cause some additional defects in the films during sputtering, since it is not electrically conductive.
- c) Previous studies revealed that most Carbon could remain as a pure element matrix rather than forming any carbide even after annealing FePt/C multilayers at 800°C for 1 hour.²⁶ Thus, it is expected that doping carbon into FePt films during deposition can suppress the FePt grain growth and, at the same time, the non-magnetic C matrix can reduce the exchange coupling between FePt grains. It is worth noting that carbon can be sputtered using DC power, which is preferred by industrial applications.

1.4.3 Methodology

The FePt perpendicular recording media were prepared with magnetron sputtering. Vibrating sample magnetometer (VSM), alternative gradient force magnetometer (AGM) and polar kerr were used to characterize the magnetic properties of the films. The microstructure of the films were characterized by high-resolution x-ray diffraction

(HRXRD) and transmission electron microscope (TEM). Atomic force microscopy (AFM) and magnetic force microscopy (MFM) were used to characterize the grain size and magnetic domain size. Actual disks with the FePt media to be developed in this research will be prepared. The disk surface topography was checked with optical surface analyzer (OSA), and the read-write test was carried out using a Guzik spin-stand.

1.4.4 Project approaches

- (a) It is deduced that a lattice mismatch (ϵ) at the interface between FePt and substrate and or/ underlayer may expand the a axis and shrink the c axis of the FePt film and thus favors the ordering at low temperatures. A series of experiments were designed to clarify the relationship between the lattice mismatch and chemical ordering of FePt films. In this thesis, 30 nm thick Pt, Cr, Cr₉₅Mo₅ or Cr₉₀Mo₁₀ intermediate layers were deposited on MgO (200) single crystal substrates prior to the deposition of the FePt films at 350°C to control the lattice mismatch. The effect of the lattice mismatch on the chemical ordering of the epitaxial FePt films were studied.
- (b) From application point of view, it is necessary to develop the FePt perpendicular media on commercial disk substrates, such as glass substrate, AlMg alloy substrate, etc. Cr and Cr based alloy (CrMo, CrRu, etc.) underlayers have been extensively used in commercial recording media. Based on the epitaxial relationship proposed above, the Cr or Cr based alloy underlayer should have a (200) preferred orientation to induce the growth of FePt (001) films. Effort was made to deposit L1₀ FePt films on CrMo seeded glass substrates at low temperatures, but the crystallographic

orientation of the FePt films was random.²⁷ In this project, CrX (X = Mo, Ru, Ti, W) (200) underlayers are to be developed. The structural and magnetic properties of the FePt films deposited on the $\text{Cr}_{1-x}\text{Mo}_x$ underlayers at relatively low temperatures were investigated.

- (c) There always exists an initial layer between underlayer and magnetic layer due to inter-diffusion during film deposition. It was suggested that an initial growth region in a recording layer be as thin as possible, since it could deteriorate H_c and M_s and recording performance.²⁸ In this thesis, the initial layers of the FePt films grown on the CrRu underlayers were studied.
- (d) To reduce the grain size and the exchange coupling between grains and to keep the (001) preferred orientation in the FePt films, the C doping effects on the microstructural and magnetic properties of the FePt films are to be investigated. The recording properties of the FePt:C perpendicular media were studied.

1.5 Novelty

- (1) A strain-induced low-temperature ordering mechanism was proposed and demonstrated. The relationship between the lattice mismatch and chemical ordering of the FePt films was first studied in this work. A critical lattice mismatch of $\sim 6.33\%$ between CrMo and FePt was found to be the most favorable for decreasing the ordering temperature of the $L1_0$ FePt thin films.
- (2) $L1_0$ FePt thin films with (001) preferred orientation can be obtained by DC magnetron sputtering at relatively low temperatures ($< 350^\circ\text{C}$).

- (3) By applying appropriate buffer layers and intermediate layers, the crystallographic orientation and the easy axis of magnetic anisotropy of the $L1_0$ FePt films can be controlled.
- (4) By doping a third element into the FePt films, the grain size and exchange coupling of the films can be well controlled.

1.6 Organization of the thesis

Chapter 2 reviews state-of-the-art magnetic recording and FePt materials as recording media in detail.

Chapter 3 deals with the experimental procedure and techniques used in this study.

In Chapter 4, the effects of lattice mismatch on the chemical ordering of FePt films are studied.

Chapter 5 presents the experimental results of the structure and magnetic properties of FePt films grown on CrX (X = Mo, Ru, Ti, W) underlayers. The effects of Mo content in CrMo underlayer, substrate temperature, CrMo underlayer and FePt magnetic layer thicknesses, and different underlayers on the structural and magnetic properties of the FePt films as well as the initial layers of the FePt films grown on the CrRu underlayers are investigated in this chapter.

The structural and magnetic properties of nanocomposite FePt:C films are addressed in Chapter 6.

The main achievements of this research are summarized in Chapter 7.

Chapter 2 Literature review

2.1 Longitudinal Magnetic Recording

2.1.1 Characteristics of Longitudinal Magnetic Recording

Present magnetic disk drives are based on longitudinal magnetic recording (LMR) systems where the magnetization of the recorded bits lies in the plane of the disk. These systems contain a recording head composed of a separate read and write element, which flies in close proximity to a granular recording medium, as illustrated in Fig. 2.1. The inductive write element records the data in horizontal magnetization patterns. The information is then read back with the giant magnetoresistive (GMR) read element by measuring the stray magnetic field from the transitions between regions of opposite magnetization. Finally, a signal processing unit transforms the analog readback signal into a stream of data bits.

In longitudinal recording, the readback signal is roughly proportional to the magnetic thickness of the media $M_r t$, where M_r is the remanent magnetization and t is the physical thickness. The magnetic width of the transitions is characterized by a transition parameter a , which depends both on the write head characteristics as well as media parameters, such as the demagnetization length $M_r t / H_c$ of the recording layer, where H_c is the coercivity of the medium. The transition width proves to be particularly important, as it ultimately limits the linear density of bits that can be written on the disk. A related and equally important attribute of the recording system is the ability to resolve transitions written at high linear density. This is investigated by measuring the dependence of the readback

amplitude as a function of the bit density, also known as the roll-off curve. Assuming linear superposition of the fields, the roll-off curve can be reconstructed from the readback signal of a single isolated transition. Such a pulse is characterized by the full width at 50% signal amplitude PW_{50} (Fig 2.2) which depends both on the a parameter of the transition and the resolution of the read head. Typically, modern hard disk drives operate up to a maximum normalized linear bit density of $PW_{50}/B = 3$, where B is the bit length.²⁹ The useable ratio PW_{50}/B strongly depends on system parameters such as the amount of precompensation,³⁰ which corrects undesired shifts of the transition position. This so-called ‘non-linear transition shift’ (NLTS) is a result of the fields from previously written transitions adding or subtracting to the write field and becomes increasingly a problem at high linear densities.

Figure 2-1 Schematic of a longitudinal recording system. B is the bit length, W is the track width, t is the medium thickness, and d is the flying height of the head above the medium.³⁰

Figure 2-2 (a) Magnetization of two transitions at $x = 0$ and 200 nm. Medium thickness is 15 nm and the transition parameter is 17 nm. (b) Perpendicular magnetic field, which is detected by the read head, at 10 nm distance from the surface of (a) longitudinal (—) and (b) type 1 perpendicular (----) media. PW_{50} is shown for a read head with zero gap. PW_{50} is larger for heads with finite gaps.³¹

2.1.2 Longitudinal Magnetic Recording Media

In general, the structure of a thin-film recording medium consists of a lubricant, a protective overcoat or overlayer, a magnetic layer, an underlayer, and a substrate. However, additional layer may be added between the magnetic layer and the underlayer for the purpose of controlling the magnetic properties of the recording layer. Most magnetic films are cobalt (Co) based metal alloys. CoCrPtB and CoCrPtTa were the most frequently used plated alloys for longitudinal media. There is a necessity to ensure strong bonding between layers, because mechanical wear of thin-film structures often occurs by adhesion failure. An adhesion-promoting interlayer has sometimes been used between

the magnetic coating and the overcoat, or between the substrate and the first deposited layer.

Recording media traditionally have a single magnetic storage layer and consist of weekly couple magnetic grains as shown in the transmission electron microscope (TEM) image [Fig 2.3].³¹

Figure 2-3 TEM image of a CoCrPtB recording medium, where the amorphous non-magnetic Cr rich boundaries magnetically separating the Co rich grains are visible as light gray areas. The inset schematically shows a magnetic transition meandering between the grains.³²

2.1.3 Issues for Longitudinal Recording

It has been predicted and is widely accepted that the practical limit of longitudinal recording will be around 200 Gbits/in².³² As bit density further increases, the grain size has to be decreased simultaneously, however, in longitudinal recording, for currently used Co-based media when decrease down to below 8 nm the grains will become

thermally unstable (also called superparamagnetism). In order to overcome superparamagnetic, anisotropy or coercivity need to be increased, but the increase in coercivity also requires very high head field which is not available for the currently used ring writing head.

2.1.4 Potential Solutions

It is becoming increasingly clear that to avoid limitations to areal growth rates arising from thermal instabilities in future high-areal-density products, significant improvements of the recording medium must be achieved. New thin film materials or architectures are needed that ease the write field requirements at high areal densities and are more thermally stable than conventional media. Oriented media and antiferromagnetically coupled (AFC) media are good examples for novel recording media.^{33,34} AFC media has been used for both areal density demonstrations^{35,36} at $> 100\text{Gbit/in}^2$ and has been integrated in products for more than two years. To avoiding the superparamagnetic limit, there are two types of magnetic recording technologies being researched extensively for extremely high-density recording: Perpendicular magnetic recording (PMR) and patterned recording. Patterned media is a very promising means for achieving high recording density near 500 Gbits/in^2 . However, since the paramagnetic limit for longitudinal recording is a current problem, an alternative is needed now. Perpendicular magnetic recording has shown promising results by extending the superparamagnetic limit to 1 Tb/in^2 .³⁷ In the following part, we will introduce the perpendicular magnetic recording.

2.2 Perpendicular Magnetic Recording

2.2.1 Characteristics of Perpendicular Magnetic Recording

In 1968, Iwasaki and Suzuki³⁸ reported making self-consistent iterative calculations for the first time by taking into account the vector demagnetization fields and the nonlinear field-dependence of magnetization components in standstill LMR. Their calculations of the spatial distributions of both longitudinal and perpendicular remanent magnetization confirmed the Tjaden and Leyten³⁹ large-scale LMR experimental model. In 1975, Iwasaki and Takemura⁴⁰ presented a dramatic demonstration of small-scale LMR using a single thick layer of standard longitudinal particulate tape media and a ring head. Bitter patterns of cross-sections of sinusoidal recordings provided confirmation of the vector nature and circular distribution of the remanent magnetization in true size LMR media. The pure circular mode of magnetization was shown to correspond to a null in the output as a function of write current in short wavelength sinusoidal longitudinal recording. By applying a strong magnetic field nearly normal to the media, they showed that the circular mode could be decoupled into a perpendicular mode. This demonstration constituted a major advancement in the understanding of magnetic recording because it resulted in the first proposal of using the PMR mode for data storage at very short wavelengths. Perpendicular magnetic recording initially received considerable attention when Iwasaki and Nakamura⁴¹ first demonstrated the high-density recording potential of PMR at 50 kfcpi by writing with an auxiliary-driven single-pole probe head on a CoCr single layer thin film having perpendicular magnetic anisotropy and reading with a ring head. This first demonstration of PMR marked the beginning of an extraordinary new era in the

evolution of magnetic recording. In 1978, Iwasaki and Ouchi⁴² reported that RF sputtered CoCr films exhibit a hexagonal-close-packed (hcp) perpendicular oriented columnar structure. A single pole type (SPT) head with a 3.5 μm thick main pole was used to write through a 1 μm thick CoCr film and a ferrite ring head with a gap of 1 μm was used to recover signal near 50 kfcpi. In 1979, Iwasaki, Nakamura, and Ouchi⁴³ reported RF sputtering of a double-layer PMR media on a flexible plastic substrate consisting of 0.5 μm CoCr on a 0.5 μm soft NiFe underlayer. A 1 μm thick SPT head was used both as the write head and the read head near 100 kfcpi. The NiFe underlayer enhanced both the writing field amplitude and gradient and the readback output by a factor 10 times by decreasing the demagnetizing fields at the pole tip of the head and by increasing the flux density for reading. In 1987, Yamamoto, Nakamura, and Iwasaki⁴⁴ reported contact writing and reading of an all "ones" pattern on a 0.1/0.5 μm CoCr/NiFe double-layer media at an incredible high density of 680 kfcpi using a single 0.4 μm thick SPT head for both writing and reading. However, the PMR has not yet been commercialized today because LMR has been improved significantly since the first demonstration of PMR and SPT heads have not been available.

A new approach to PMR media having unity squareness without correction for demagnetization fields utilizing multilayer superlattice perpendicular media has been pursued in recent years.^{45,46,47,48} The nanostructure, grain size, texture, and magnetic properties of the interfaces of the multilayer superlattices are extremely sensitive to the surface roughness, grain size, and crystallinity of the interfaces, and the smoothness of the ultra-thin underlayers. The nanostructures of PMR superlattice media must have

magnetically-isolated grains for high-SNR at high densities and thickness equal to several grain diameters for high-thermal stability. These new PMR superlattice media being fabricated are modeled by micromagnetic simulations using Voronoi cells and are interface-induced high-magnetic anisotropy hetero-epitaxial multilayer superlattices of CoB/Pt, CoB/Pd, CoB/Pt/Pd, CoBCrTaPt/Pt/Pd on super-smooth hard disk glass or alternative substrates. Double-layer PMR media is being deposited on optimized ultra-thin amorphous underlayers such as InSn oxide (ITO), CoZrTa, Ta, or SiN, deposited on magnetically soft high-saturation flux-density NiFe, CoFe, FeN, CoZr, or FeTaN/IrMn underlayers. PMR superlattice media must have saturation flux-densities of 200–900 G, 10–4 nm grain diameter, 10–24 nm thickness, 5–15 kOe remanent coercivity, loop side slope of unity, unity magnetization squareness, and have an extremely smooth (0.2–0.1 nm) surface.

There are potentially three different PMR writing modes: (1) single-layer media using a thin film ring write head (TFH), (2) double-layer media having a soft magnetic keeper underlayer using a single pole head (SPH) or a pseudo-pole head (PPH) constructed from a standard TF write head by focused-ion-beam etching, and (3) triple-layer media having a soft magnetic keeper overlayer and underlayer using a single pole head (SPH) or a pseudo-pole head (PPH) constructed from a standard TF write head by focused-ion-beam etching. In the 1st mode, the PMR media must be highly oriented with unity perpendicular squareness. In the 2nd and 3rd modes, the media must also have unity perpendicular squareness, but the soft underlayer and overlayer enhance the magnitude of the writing field and reduce the demagnetizing fields. However, the main advantages of

single and double-layer PMR media are that PMR media can support extremely high densities and can be significantly thicker than LMR media for the same density so that the resistance of PMR to thermally decay is increased. An SPH or PPH with a keeper underlayer provides a 2 times larger write field and higher head field gradient so that PMR media with coercivity higher than LMR can be used to give sharper transitions and sharper track edges suitable for achieving densities of 200–1000 Gb/in².

2.2.2 Basic Process in Perpendicular Recording

Perpendicular recording has been frequently proposed as a technique for surpassing the limits placed on longitudinal recording by thermal fluctuations. Fig.2.4 shows the basic process in perpendicular magnetic recording. Generally a single-pole head is being used for recording on the perpendicular media but of course, a ring-shaped head has also been used with some design changes. In longitudinal recording, when a transition is made from one magnetic orientation to another, the N and S poles of the neighboring cells are adjacent. Just as the poles of two magnets laid end to end in this manner weaken each other, so the cells tend to demagnetize each other, and the tendency gets worse with smaller cells, that is, an increase in recording density which causes the bit cell or size to shrink. However, in perpendicular recording, the opposing poles of the two cells lie next to each other, so the magnetic fields reinforce each other and thus favor sharp transition.

Figure 2-4 Schematic process in perpendicular magnetic recording

In perpendicular recording, the transition region width, a , is narrower than that in longitudinal mode and hence, a higher and more stable bit density is possible. In addition, the magnetic structure is resistant to demagnetization at higher packing densities. The columnar form of the crystal grains in perpendicular media helps to act against demagnetization and to avoid thermal instability of the media.

Two different types of recording media have been considered for the use in perpendicular recording systems. Type 1 consists of a recording media with the easy-axis oriented out-of plane deposited on a high permeable, soft magnetic underlayer. Type 2 is similar to type 1 without the soft underlayer (SUL) and utilizes a conventional ring head, as used in

longitudinal recording. Only little work is in process using type 2 recording media, as it does not possess some of the advantages of type 1 media. Therefore, a recording system based upon type 1 media is generally considered to be the main candidate for replacing longitudinal recording media.

A recording system based on type 1 media is depicted in Fig. 2.6 with a conventional GMR read head for detection of transitions. A write head with a wide gap between the write and collector pole is preferred to prevent flux leakage between both poles.⁴⁹ The transitions are written with the trailing edge of the write pole. The SUL guides the magnetic flux from the write pole to the collector pole and essentially becomes part of the write head. In simple models, it is often assumed that the SUL generates a mirror image of the write pole, therefore effectively placing the recording media in the 'gap' of the 'write head'. On the one hand, this arrangement has the advantage of theoretically almost doubling the write field, allowing writing transitions in recording media with a higher anisotropy, which in turn are thermally more stable. More complex micromagnetic modelings⁵⁰ still predict a magnetic field increase of up to 70%.⁵¹ On the other hand, the combination of head and SUL is a highly efficient magnetic flux guide and may focus external fields, such that degradation of previously written tracks and even data loss might occur. To avoid writing transitions with the collection pole, the magnetic field must be much smaller at the collection pole (~10%) than at the write pole. Simple surface areal scaling allows adjusting the field ratio of the collection pole and write pole.

Demagnetization field, which favors opposite magnetization of neighboring grains, also improves the thermal stability for high-density patterns (a small amount of inter-granular exchange interaction may be utilized to counteract some effects of the demagnetization field), but enhances decay rate at low linear densities, in particular if H_c is close to or smaller than $4\pi M_s$. Thus, high-density bit pattern decay can lower than low-density bit pattern, in which decay rate is plotted as a function of linear transition density for different types of perpendicular recording media. This is in contrast to longitudinal media, where demagnetization field destabilizes bit pattern at higher linear densities. Besides the enhanced thermal stability, perpendicular recording also benefits from the uniform easy-axis orientation as a result of its naturally high orientation ratio. Media noise is reduced, while the readback signal is increased. As of today, areal density demonstrations, however, could not yet take full advantage of the potential of perpendicular recording. Some of the critical components, such as perpendicular recording media and SULs, need further improvement. Also different shapes of the write pole of the head are investigated in order to optimize the magnetic write field,⁵² and the large readback signal amplitude at low densities (Fig.2.5) might require different read head designs. Finally, special modulation codes, which on average result in zero magnetization of the track, might be required to reduce the maximum demagnetization field.⁵³

Figure 2-5 Schematic type 1 perpendicular recording system with SUL and a single pole head.⁵⁰

2.2.3 Comparison with Longitudinal Recording

The differences between perpendicular recording and longitudinal recording are given in subsequent paragraphs of this section.

Fig 2.6 shows the transition regions of media in perpendicular and longitudinal recording.⁵⁴ With regards to direction of magnetization, the perpendicular media have closely packed fine grains, which grow perpendicular to the film surface, and those of longitudinal media are grown parallel to the plane of the film surface. In other words, the easy axis for perpendicular media stands perpendicular to its surface and that in longitudinal media lays in plane. Furthermore, in longitudinal media, the magnetic flux tends to close upon itself in the media so that flux availability for reproduction is reduced.

Whereas, in perpendicular media, the adjacent magnetized regions are in anti-parallel manner and their external magnetic flux assist each other in achieving stability. In addition, the width of the transition region in perpendicular media is usually smaller and is about the size of a grain diameter. For that of longitudinal media, it is usually much wider and saw-tooth like pattern can be observed at the transition region. This zigzag transition region is presumed to be the origin of noise in longitudinal media.

Figure 2-6 Transition regions: (a) perpendicular recording and (b) longitudinal recording

55

Fig 2.7 shows that both types of media are also suffered from demagnetizing effect but the regions where the strongest demagnetizing fields exist are rather different. For perpendicular media, the demagnetizing field is oriented perpendicularly to the film surface and is more pronounced away from the transition region, thus, magnetization decays more rapidly at distance further away from transition region. However, in

longitudinal media, demagnetization reaches its maximum at the transition region, hence, the magnetization decays more at this location. In addition, another source of demagnetization occurs between oppositely magnetized regions in longitudinal media. The field from one magnetized region tends to demagnetize its neighbors with the consequence of spreading the magnetization transition region and decreasing signal output and resolution.⁵² Another difference between the two is that perpendicular recording is more suited for recording digital signal as opposed to analog signal for longitudinal media. As the density increases, the reproduction waveforms of perpendicular and longitudinal media approach that of square wave and sinusoidal wave respectively. Demagnetizing effect in perpendicular media disappears at a higher recording density but that in longitudinal media become more severe as density increases. As a result, researchers are looking towards perpendicular recording for ultra-high density recording.

In terms of media thickness, perpendicular media are usually much thicker as compared to longitudinal media at a given recording density. In longitudinal recording, the higher the recording density, the thinner the medium thickness must be. Thicker perpendicular media can help to minimize demagnetizing field effects and the addition of a soft magnetic underlayer also assist in closing the flux line during writing process if SPT head is used.⁵⁶

Figure 2-7 A simple model for magnetization regions in perpendicular and longitudinal recording.⁵⁶

2.2.4 Advantages of Perpendicular Magnetic Recording

Perpendicular recording is thought to offer several advantages relative to longitudinal recording. Several advantages stem from the nature of the destabilizing field generated by neighboring bits. In longitudinal recording, a bit boundary forms a charged domain wall. This stored charge generates strong destabilizing fields that work to widen the transition and switch the magnetization of grains into unintended directions. The strength of these fields increases with media thickness. As density is increased, the number of charged domain walls and the requirement for sharp transitions increase, thus aggravating the problem. In perpendicular recording, a bit boundary places plus and negative magnetostatic charges of grains on opposite sides of the boundary in close alignment.

This is magnetostatically favorable. In other words, magnetostatics favors transitions and prefers high density. Furthermore, thicker media exhibit ever more favorable magnetostatics. Thus, the granular volume can be increased without affecting the areal density. These two effects account for most of the perceived benefit of single layer perpendicular media. However, it is generally argued that addition of a magnetically soft underlayer generates the largest benefits for perpendicular recording. This soft underlayer is placed between the recording layer and the substrate and serves to image the magnetic recording head. This effectively doubles the field generated, thus allowing the coercivity and anisotropy field to be doubled. This, of course, stabilizes the grain.

There are also several secondary advantages to perpendicular recording. Longitudinal recording for disk drives employs 2-d isotropic media meaning that the anisotropy axes of individual grains are not aligned. This produces noise that would be eliminated by the highly aligned perpendicular materials. This also implies that track edges are sharper in perpendicular recording. This is a consequence of the range of coercivities found in the disoriented longitudinal grains that may be written by the stray side fields generated by the recording head. In perpendicular recording, the coercivities will be more uniform owing to the better orientation and the track edge will be sharper.

2.2.5 Challenges for Perpendicular Magnetic Recording

The challenges facing perpendicular magnetic recording are to simultaneously achieve high thermal stability with high signal amplitude and high media signal-to-noise for low

bit-error-rate. Meeting these challenges will require extremely uniform average magnetic cluster diameters of 10–5 nm, double-layer 50–25 nm thick media with a high-saturation 10–20 kG low-noise magnetic underlayer or overlayer with permeability of 100–50, average surface roughness of 0.2–0.1 nm, remanent magnetization of 200–900 emu/cc, unity squareness without any demagnetization field correction, remanent coercivities of 5–15 kOe which can be written with sub-nanosecond rise times using a microstrip single-turn perpendicular single pole head and read with a 10–20% GMR spin-valve read head.

The basic theory of thermally activated time-decay of remanent magnetization or the ultimate superparamagnetic limit is based on the assumption that for a single magnetic energy barrier, the exponential relaxation time constant τ is given by the Arrhenius-Néel law as:

$$\tau = (1/f_0)\exp(\Delta E/kT) \quad (2-1)$$

where ΔE is the uniaxial energy barrier, T is the absolute temperature, k is the Boltzmann's constant, and f_0 is the frequency of approach to the energy barrier which is taken as 10^9 s^{-1} . For most magnetic materials, this exponential decay is not observed and the time-decay of remanent magnetization M_R follows logarithmic time dependence for a wide uniform distribution of energy barriers as follows:

$$M_R(t, H_{\text{int}}) = M_R(t_0, H_{\text{int}}) - S \ln(t/t_0) \quad (2-2)$$

where the initial time is t_0 and S is the magnetic viscosity or decay rate which is taken to be in units of %/decade (s), and H_{int} is the magnetic interaction field acting to reverse the M_R . For a system of magnetic particles having a uniaxial anisotropy energy density K_u

and magnetic volume V , the effective energy barrier to kT ratio can be shown to be given by:

$$\Delta E/kT = (KV/kT)_{\text{eff}} = (KV/kT)_0 [1 - (H_{\text{int}}/H_K)]^2 \quad (2-3)$$

where $H_K = 2 K_u/M_s$ is the uniaxial anisotropy field, and M_s is the saturation magnetization. Since the term $(KV/kT)_0$ is the energy barrier without magnetic interactions, the interaction field H_{int} tends to reduce the energy and increase the magnetic viscosity or time-decay rate. A ratio of about 42.543 corresponds to an M_r or signal loss of only 10% in 10 years at 65 °C.

In order to better understand the subtle dependence of the thermal-activated time decay brick wall facing ultra-high-density recording (UHDR) densities beyond 100 Gbits/in², the constraints and trends can be predicted by an UHDR areal storage roadmap. The results of Tarnopolsky⁵⁷ are used to generate the constraint to first achieve an on-track bit-error rate (BER) for an assumed media SNR_{peak} for a given bit length (B) and a user density $U = \text{PW50}/B$ is

$$(\text{SNR})_{\text{peak}} = (2/\pi) U (B/\sigma_{\text{jitter}})^2 \times [(\pi U/4)/\sinh(\pi U/4)]^2 \quad (2-4)$$

by calculating the timing position jitter (σ_{jitter}) which is directly related to the cross-track correlation length (s) and the transition length parameter (a) according to Bertram⁵⁸ by the following simple formula :

$$\sigma_{\text{jitter}}^2 = (\pi^4/48) \times s \times a^2/w \quad (2-5)$$

where (w) is the read track width. For perpendicular recording media with $H_c = H_k$ and $S = 1$, the average grain diameter D is assumed to be equal to the average magnetic cluster diameter and equal to the cross-track correlation length s , and the transition length parameter $a = 3D/4$, the SNR is then determined to take the simple form given by Bertram⁵⁹ as

$$(\text{SNR})_{\text{peak}} = 0.314 w \times \text{PW}_{50} \times B/s a^2 \quad (2-6)$$

However, a simpler method is to use the Poisson statistics of additive noise for the magnetic grains in the media to find the $(\text{SNR})_{\text{rms/rms}}$ as given by

$$(\text{SNR})_{\text{rms/rms}} = -10 \log_{10}(N) \quad (2-7)$$

where N is the number of magnetic grains per bit. This simpler approach has been followed to determine the average grain diameters assuming hexagonal close packing of the grains as shown in Table 2.1. Once the grain diameter has been determined and the media thickness has been chosen to insure sufficient thermal stability and resolution, the uniaxial crystalline anisotropy can be determined. Taking into account the demagnetizing fields of three adjacent recorded bits and using the optimum coercivity as a constant between 1 and 3 times the saturation magnetization $4\pi M_s$ to maximize the height of the energy barrier assuming $H_c = H_k$, the saturation magnetization M_s and remanent coercivity were determined as given in Table 2.1.

Areal density (Gbit/in ²)	100	250	500	1000
Bit density (Kbit/in)	635	1000	1400	2000
Rack density (Kbit/in)	160	250	354	500
BI aspect ratio = BAR	4	4	4	4
BIT length = <i>B</i> (nm)	40	255	18	13
Read track width = <i>W</i> (nm)	140	89	63	45
FLY height = <i>h</i> (nm)	8	7	6	5
Pulse Width = PW50 (nm)	100	64	45	32
log on-track bit error rate	-8	-7	-6	-5
Signal-to-noise SNR (rms/rms)	18	17	16	15
Number grains per bit = 10 ³ SNR/10	63	50	40	32
Average grain diameter = <i>D</i> (nm)	9	7	5	4
Magnetization squareness = <i>S</i>	1	1	1	1
Media thickness = <i>δ</i> (nm)	18	20	22	24
Anisotropy constant <i>K_u</i> (10 ⁶ erg/cc)	2	3	4	6
Media saturation = <i>M_s</i> (emu/cc)	400	550	700	875
Remanent coercivity = <i>H_c</i> (Oe)	8500	11000	12500	1430

Table 2-1. UHDR roadmap for perpendicular magnetic recording from 100 to 1000 Gb/in².⁵⁶

This resulting ultra-high density roadmap for perpendicular magnetic recording given in Table 2.1 shows that PMR is capable of achieving 100 Gbits/in² easily with on-track BER of 10⁻⁸ and 10 nm grain size with *K_u* = 1.7×10⁶ ergs/cc and a remanent coercivity of 8.5 kOe. Furthermore, it appears that 1000 Gb/in² may be possible with *D* = 4 nm *K_u* = 6×10⁶ and a coercivity of 14300 Oe even though the on-track BER is only 10⁻⁵. Either the SNR of the media will have to be increased or the required SNR will have to be provided by improving signal processing techniques. If SNR = 18 is assumed for 1000 Gb/in², *D* = 3 nm and *K_u* = 1.25×10⁷ erg/cc and the remanent coercivity required would be about 20 kOe. The future of perpendicular magnetic recording will be extremely challenging.^{60,61,62,63} The goals are to reduce grain size and dispersion significantly, enhance magnetic isolation so that the cluster size is a single grain, reduce transition and DC noise, optimize design a single-turn single-pole head for high-speed writing, and achieve durable contact recording.

2.2.6 Requirements for Perpendicular Recording Media

The promise of perpendicular recording can only be met if the media meets some stringent requirements as follows:

- 1) Large H_c . The advantage offered by a large write field is the ability to record on high coercivity materials that will be able to resist thermal fluctuations.
- 2) Small magnetic layer thickness t . Though perpendicular media are, in general, thicker and henceforth, more thermally stable than longitudinal media but from a commercial point of view, it should not be too thick.
- 3) Large M_r but should only be just enough for good playback signals by choosing materials with a large M_s by increasing volume fraction of magnetic particles in the recording layer, that is to pack many particles together.
- 4) Moderate S^* or narrow switching field distribution (SFD) by ensuring a narrow distribution of magnetic switching units. This can be achieved by controlling the magnetic film thickness and grain size and by reducing the demagnetizing field through introduction of a soft magnetic underlayer.⁶⁴
- 5) High perpendicular anisotropy, that is to produce thin film media with a good plane orientation of the easy-axis perpendicular to the media surface.
- 6) Well-controlled crystalline texture and small grain size. In other words, it is to fabricate media with uniform and small grains with a good orientation.
- 7) Low media noise by decoupling the interactions between grains.

2.2.7 Candidate Materials for Perpendicular Recording Media

Table 2.2 shows a compilation of candidate materials for perpendicular media. The last two columns show minimal thermally stable single grain diameters D_p calculated based on the stability criterion outlined above with $r_K \sim \ln(\tau f_0) \sim 50$. (a) for cylindrical grains of constant height $\delta = 10$ nm, (b) for cubic grains with $D = \delta$ and (c) for spherical grains.

Table 2-2: Magnetic properties and theoretical minimal grain diameters of various perpendicular media candidates. The minimal physical grain diameter is estimated using maximum demagnetization $4\pi M_s$ and constant thickness of $\delta = 10$ nm (a), cubic grains D_p (b) and spherical grains (c).⁶⁵

alloy system	Material	K_u (10^7 erg/cm ³)	M_s (emu/cm ³)	H_K (kOe)	T_c (K)	$D_p^{(a)}$ (nm)	$D_p^{(b)}$ (nm)	$D_p^{(c)}$ (nm)
Co-alloys	CoCr ₂₀ Pt ₁₅	0.25	330	15.2	--	15.5	12.4	15.4
	Co ₃ Pt	2.0	1100	36.4	--	6.4	6.9	8.5
	(CoCr) ₃ Pt	0.39	410	19.0		12.4	10.6	13.2
	CoPt ₃	0.5	300	33.3	600	9.0	8.6	10.7
CoX/Pt(Pd) multilayers	Co ₂ /Pt ₉	1	360	55.6	500	6.1	6.7	8.3
	Co ₂ /Pd ₉	0.6	360	33.3	500	8.4	8.2	10.2
L1 ₀ phases	FePd	1.8	1100	32.7	760	7.3	7.5	9.3
	FePt	7	1140	122.8	750	2.4	3.6	4.4
	CoPt	4.9	800	122.5	840	2.8	3.9	4.9
	MnAl	1.7	560	60.7	650	4.9	5.7	7.1
Rare-earth transition m.	Fe ₁₄ Nd ₂ B	4.6	1270	72.4	585	3.4	4.5	5.5
	SmCo ₅	20	910	219.8	1000	1.9	3.0	3.8

- $H_K = 2K_1/M_S$ anisotropy field; K_1 : intrinsic first order anisotropy constant;
- D_p = minimal stable grain size for $\delta=10\text{nm}$ tall cylindrical grains ($\tau=10$ years; $T=350\text{K}$);
- T_C =Curie Temperature;

^(a) Cylindrical grains of constant height $\delta=10$ nm

^(b) Cubic grains with

^(c) Spherical grains

2.2.7.1 CoCr-based alloys

In 2000, H. takano and co-workers⁶⁶ reported a laboratory demonstration of hard disk storage with 52.5 Gbits/in^2 using CoCrPt atop a soft underlayer. This demonstration illustrated a high signal-to-noise ratio afforded by Co based alloys. In particular, such materials can be produced with small grain size. Weak intergranular exchange coupling can be induced by annealing Cr to the grain boundaries. Both effects are essential for low noise. The demonstration also illustrated the weakness of these materials: low coercivities that make the media susceptible to the thermal decay. Although these materials could, in principle, attain the same coercivities as the longitudinally oriented Co-based alloys, it is thought that the lack of a seed layer along the c-axis allows excess stacking faults to appear and thus reduces the observed anisotropy and coercivity.

Other interesting results reported by the Hitachi group concern the role of dopants and sputtering deposition pressure. T. P. Nolan *et al.*⁶⁷ show that grain nucleation is best accomplished at low argon pressures, even though the majority of the deposition is

undertaken at high pressures, at least for the specific case of CoCrPt atop TiCr seeds. In particular, they advocate depositing the temperate layer (top of the seed) and the nucleation layer (bottom of the recording layer) at a low pressure in order to preserve good orientation. Granular media profit from the enormous amount of expertise in grain size control, which has been obtained for longitudinal media. As discussed above, grain size control is extremely important, as it directly influences media SNR (equation (1)). However, the anisotropy in the perpendicular granular layer (GL) is found to be substantially lower than in similar longitudinal media, leading to high thermal decay rates. High stacking fault density or FCC lattice formation present in the HCP grains might be the reason for low anisotropy.^{68,69} It is vital for the success of granular media to overcome this problem. The maximum recording density of this kind of media is about 300 Gbits/in².⁷⁰ During this project, 170 Gb/in² and 240 Gb/in² has been demonstrated by Seagate in the end of 2003 and Hitachi in 2005 on CoCr-based alloy perpendicular media. And Seagate company will ship the product with a recording density of 170 Gb/in² based on CoCr-based alloy perpendicular media.

2.2.7.2 Co/Pd and Co/Pt multilayers

Superlattice media offer performance very different than that provided by alloy media. Many superlattices generate large perpendicular anisotropies from their numerous interfaces. For example, Co/Pt and CoPd superlattices employing monolayers of Co have been shown experimentally and theoretically to exhibit uniaxial anisotropies of 6.5-8.5×10⁷ ergs/(cm³ of Co) when forming lattices of high structural and chemical uniformity. The corresponding magnetization ranges from 2000-2500 emu/(cm³ of Co).

The resulting anisotropy fields of 60-80 kOe offer the potential of enormous coercivities. In reality, the same experimental conditions that provide high crystal quality, e.g., Molecular Beam Epitaxy, also generate excessively large grains that are exchange coupled. This reduces the coercivity. Nonetheless, several groups have obtained coercivities beyond 10 kOe. Furthermore, it has been found both experimentally and theoretically that the anisotropy can be approximately tripled by employing a repeating quadrilayer of Co/Pt/Pd/Pt where the Pt layers are approximately one monolayer thick. Essentially the extra interfaces introduction between the spin polarized Pt and Pd make additional large contributions to the anisotropy owing to the large spin orbit energies of the Pt and Pd. In short, it is not expected that the obtaining of adequate coercivity will be a problem in these materials.⁶¹

In contrast, the hysteresis loops typically measured in these materials exhibit a very sharp drop of magnetization versus applied field as the loop crosses through the coercivity. This is indicative of strong inter-granular exchange coupling. It is also associated with excessive transition noise that is, of course, undesirable. To date, it is unclear whether this exchange coupling is a consequence of the history of Co/Pt and Co/Pd (they were studied extensively for use as magneto-optical media where this loop shapes is considered to be desirable) or whether it is intrinsic. Possible solutions include the addition of dopants such as boron that migrate to the grain boundaries or the deposition of physically separated grains. Superlattices also offer some useful secondary advantages. Proper choice of the nonmagnetic Pt or Pd layer makes the magnetization easily adjustable to meet the system needs. The high Pt and Pd content make the materials

highly corrosion resistant. This is an important consideration in view of the very thin protective carbon layers necessary for future recording densities. They can be deposited at relatively poor vacuum pressures. A negative feature is the requirement of many layers. Typically, this is accomplished by rotating a substrate beneath two distinct sources, e.g., Co and Pt. This technique would require modification of most current media manufacturing equipments. These multilayer media have large intrinsic exchange coupling in the film planes, which induces transition noise. High-pressure deposition and addition of segregants such as B or Cr, or impurities, such as O, during deposition reduce the in-plane exchange coupling. The exchange interaction between multilayer and SUL is reduced by an exchange break layer (EBL). It is believed that the $[\text{CoX/Pd}]_n$ multilayer perpendicular recording media with a granular seed layer and a soft magnetic underlayer using a single pole write head is the solution for 500 Gbits/in² magnetic recording application.

2.2.7.3 FePt thin films

Among the candidate perpendicular magnetic media materials as shown in Table 2.2, chemically ordered FePt with $L1_0$ (CuAu-I) structure (face centered tetragonal-fct) possesses very high magnetic anisotropy constant ($6.6\text{-}10 \times 10^7$ erg/cm³), large M_s , high corrosion resistance and small thermally stable grain size, all of which make it the most promising candidate for the ultra-high areal density (>1000 Gbit/in²) recording media.

2.3 FePt alloys

2.3.1 Physical properties

The properties of the FePt alloy system were first studied systematically in 1907.⁷¹ A transformation between ordered and disordered phase was observed in the equiatomic composition range (the transformation temperature is about 1300 °C), which was confirmed by measurements of X-ray spectra,^{72, 73} magnetic,^{74, 75} electrical^{75, 76} and mechanical⁷⁷ properties. Kussman & Rittberg found three stable crystal structures exist in Fe-Pt system: FePt₃, FePt, Fe₃Pt. The phases and properties of these have been documented by Hansen and Bozorth.^{79, 78}

Figure 2-8 Iron-platinum phase diagram for alloys (from Watanabe et al. 1983)

2.3.2 Crystallography:

As shown in Fig 2.9, the disordered γ -phase exists above the transformation temperature (1300 °C). It has a face-centered cubic (fcc) crystal structure.^{75,76} In 1941, Lipson et al. determined that the ordered r_1 -phase of the FePt alloy has a CuAu-I type structure with lattice parameters $a = 3.838 \text{ \AA}$ and $c = 3.751 \text{ \AA}$. The structure is shown in Fig 2.10.

Figure 2-9 Crystal structure for the disordered and ordered phases of FePt equiatomic alloy.⁷⁶

2.3.3 Magnetic Properties

The magnetic properties of iron-platinum alloys have been studied since the 1930's. Fallot determined that equiatomic alloy is a ferromagnet with a Curie temperature of 670K.⁷⁹ Kussman and Rittberg found that the saturation magnetization is greater for the disordered alloy than for the ordered alloy.⁷⁵ The FePt $L1_0$ alloy has uniaxial magnetocrystalline anisotropy and K_1 has been measured as $7.0 \times 10^6 \text{ J/m}^3$ for bulk alloy.⁸⁰ A similar value, $K_1 = 6 \times 10^6 \text{ J/m}^3$, was measured for thin film.⁸¹ In comparison, the disordered alloy has cubic anisotropy and $K_1 = 6 \times 10^3 \text{ J/m}^3$ ⁸³ the ordered alloy has a

saturation magnetization at 298K of 1150G.⁸² The critical diameter for a single domain FePt particle is around 600 nm.⁸² The thickness of a domain wall in the FePt bulk alloy is around 3.9 nm.⁸³

2.3.4 The current status as a recording media

In order to develop FePt as recording media, the $L1_0$ FePt ordering temperature has to be reduced and the easy axis should be either perpendicular or parallel to the thin film plane according to the recording mode.

2.3.5 Methods for producing $L1_0$ FePt (001) films

Many researchers made much effort to produce the $L1_0$ ordered FePt films with (001) preferred orientation by a variety of methods. So far, the methods for producing $L1_0$ ordered FePt films with (001) preferred orientation can be roughly sorted as follows:

(a) Molecular beam epitaxial (MBE) deposition on MgO (100) single crystal substrate at elevated temperature.

Lairson *et al.* reported formation of c-axis oriented epitaxial FePt with perpendicular magnetic anisotropy by annealing (001) oriented multiplayer precursors and obtained magnetic properties far superior to those of the as-deposited films or comparable disordered alloys.^{84,85} This processing, while resulting in films with desirable properties, is somewhat complicated, involving both multiplayer deposition and post annealing. A

simplicaiton of this was achieved by using MBE to directly synthesize c-axis oriented ordered FePt from element sources.

(b) Co-sputtering Fe and Pt or FePt on heated single crystal MgO (200) substrate at high deposition temperature

Visokay *et al.* reported a further processing simplicaion by directly synthesize of epitaxial c-axis oriented ordered FePt by co-sputtering Fe and Pt on single crystal MgO (200) at a high deposition temperature ($\geq 400^\circ\text{C}$), with resulting properties comparable to those of the previously mention authors.⁸ As of today, this method is widely used to study the structure and magnetic properties of FePt films for fundamental purpose, but the use of crystal MgO substrate is not accepted by industry.

(c) Deposition FePt on MgO underlayer or Ag underlayer

Y. N. Hsu *et al.* reported that a thick (175-nm) Ag underlayer on silicon substrate was found not only to induce epitaxial growth of FePt films, but also to reduce the FePt ordering temperature.¹¹ Recently, K. Kang *et al.* obtained (001)-textured nanogranular FePt composite films on amorphous quartz substrate using an MgO seedlayer and an Ag underlayer prior to the FePt/Ag multilayer followed by annealing at 550°C for 1 h. They found that a 20 nm thick Ag underlayer played a significant role in improving (001) texture and also in decreasing the fct FePt ordering temperature.⁸⁶

(d). Non-epitaxial method: Fe/Pt, FePt/B₂O₃, FePt/SiO₂, Fe/Pt/C, Fe/Pt/Ag

H. Zeng *et al.* reported that highly oriented, face-centered tetragonal ordered FePt thin films grown nonepitaxially by directly depositing (Fe/Pt)_n multilayer on thermally oxidized Si substrates and subsequent annealing.⁸⁴ Although this process results in films with desired magnetic properties, it involves both multilayer deposition and long time high temperature post annealing ($\geq 550^{\circ}\text{C}$). The orientation mechanism of this method is still not quite clear. C. P. Luo *et al.* obtained nanostructured FePt:B₂O₃ and FePt:SiO₂ thin films with perpendicular magnetic anisotropy by annealing FePt/B₂O₃ and FePt/SiO₂ multilayers.^{13,87} This method needs rf-sputtering and long time high temperature post annealing, which are not accepted by industry.

2.3.6 Methods for lowering the ordering temperature of L1₀ FePt films

As discussed above, it is desirable to find a way to produce L1₀ FePt magnetic thin films at low temperature. For this purpose, several attempts were made to fabricate the L1₀ FePt films at low temperatures by MBE⁷ and multiplayer film sputtering^{88,89} However, both methods require a rigorous control for film processing and are low fabrication processes. The development of a cheaper and a rapid low-temperature processing of L1₀-ordered thin films by the sputtering process is more desirable for successful industrial application. One method of fabrication the L1₀-ordered FePt films at low temperature is to grow a L1₀ film in situ at a heated substrate. For example, Suzuki *et al.*⁹⁰ reported an in situ growth of an L1₀ FePt film under a high Ar pressure at a substrate temperature of 400°C. More recently, Y. K. Takahashi *et al.* reported that fully ordered L1₀ FePt film can be growth in situ at a heated substrate only at 300°C.⁹¹ Hsu *et al.* also reported the

fabrication of an epitaxial L1₀ FePt sputtered film using an Ag underlayer.¹⁰ These in-situ annealing processes are among the most promising methods for preparing L1₀-ordered FePt film. Another interesting approach is to reduce the temperature for ordering by adding ternary elements to the FePt binary alloy. For example, Seong-Rae Lee *et al* reported that Zr-doped FePt alloy films showed accelerated ordering transformation kinetics accompanying smaller grains than pure FePt alloy.⁹² Tomoyuki Maeda *et al.* found that the addition of Cu to an FePt alloy film is an effective approach for reducing the ordering temperature of FePt.¹⁴ Y. K. Takahashi *et al.* reported that the temperature that A1 phase orders to the L1₀ structure decreased from 500°C to 400°C by alloying 4 at% Cu.⁹³ Ravelosona *et al.* reported that the long-range order parameter S of sputtered FePt (001) films may be improved by using post growth He ion irradiation.⁹⁴

2.3.7 Methods for controlling the grain size in L1₀ FePt films

2.3.7.1 Nano-layer engineering

In order to control grain size of the magnetic particles as well as to achieve magnetic isolation among grains, various substitutions of nonmagnetic elements (in multiplayer fashion, introduction of buffer layer, and/or exchange-decoupling layer between the FePt layer and soft-magnetic underlayer) have investigated in FePt system. This substitution obviously determines the mechanism of grain growth, inter-grain interactions.⁹⁵ Most of the studies on the earlier systems are followed by annealing the (001) oriented multilayer precursors and obtaining the magnetic properties superior to those of the as-deposited films. Although this process may result in some desired magnetic properties, it involve

both multilayer deposition and long time post-deposition annealing. Moreover, this method has disadvantages as a manufacturing process because such a high temperature post-deposition annealing can cause irreversible changes in substrates for magnetic media and enhances the particle growth considerably. H.S. Ko *et al.* reported that the ordering and grain growth kinetics of FePt films could be fine controlled by C doping.⁹⁶ However, the use of MgO single crystal substrate limits its practical applications.

Despite C doping, materials such as AlO_x , Al_2O_3 , AlN, TaN, Ag, W and Ti¹⁵⁻²⁰ also were widely studied and have shown promise in controlling FePt grain size in ordered films. Other studies involving FePt with Ni, B, BN^{21,22} as additives have demonstrated that these can reduce magnetic coupling. Oxide additions to FePt can provide a better grain size control and reduce magnetic coupling in the FePt film. Luo *et al.* reported well-separated grains with strong perpendicular orientation in nanostructured FePt/ B_2O_3 multilayers¹³ FePt/ SiO_2 multilayers showed a limited grain size of 10 nm but poor orientation.⁶ HfO_2 and MnO additives were studied by C.L. Platt *et al.*²³ where the HfO_2 additive reduced the grain size with a random texture. MnO additive affected neither grain size nor L1_0 ordering of annealed FePt films. T. Suzuki *et al.* reported that the MgO additive reduced the domain size of FePt film.⁹⁷

2.3.7.2 Self-Organized-Magnetic-Arrays

In 2000, Sun and co-workers reported the formation of FePt nano-particles with self-organized-magnetic-arrays (SOMA).⁹⁸ SOMA shows promising to fabricate particle nucleation and growth processes are separated from each other during chemical synthesis.

This allows for the formation of nearly mono-disperse magnetic nanoparticles of e.g. FePt and Co. These nanoparticles are encapsulated in a surfactant molecule shell, which prevents agglomeration in solution as well as during subsequent self-assembly and drying processes. So-formed SOMA media may serve as (i) conventional media with reduced dispersions (ii) as bit-patterned media with bit-transitions defined by rows of particles and (iii) they may eventually be scaled to single-particle-per-bit recording. In this last scenario, the ultimate areal density capability of SOMA media is governed by the minimal thermally stable size and center-to-center spacing of neighboring particles. Under most optimistic conditions, i.e. assuming that writability, signal retrieval, bit-addressability and spacing issues can be solved areal densities of up to 40 Terabit per square inch may be reached. However, to use such SOMA media for either conventional (longitudinal or perpendicular) or bit-patterned media, a number of obstacles have to be overcome. First, while self-organization and size distributions of better than 5% have been found in as-deposited FePt SOMA structures, annealing, as required to convert FePt material into the desired magnetically hard fct ($L1_0$ -) phase, has been found to destroy the array order and size distribution via metallurgical sintering processes.⁹⁹ Proposals to avoid sintering include synthesis of core-shell structures with harder shell (diffusion barrier) and enhanced adhesion of the particles to the substrate surface via chemical modification of substrates as well as particle legend shell. It has been shown that by sandwiching FePt nanoparticle layers between carbon films, sintering can be avoided even after heating to 700°C. Second, all FePt SOMA structures reported to-date have more or less random easy-axis orientation, which is not acceptable for future high density media as it will result in extra media noise and reduced the signal strength. This appears

to be one of the most difficult unsolved problems today. Attempts to anneal SOMA structures in large fields or deposit in the presence of a magnetic field so far, to our knowledge, have failed to produce oriented magnetic structures. Third, FePt SOMA nanoparticles today are approximately spherical (truncated octahedral before anneal). The surfactant shell coating is between 1-2 nm thick (oleic acid, oleylamine). Scaling, under these conditions is much more unfavorable as e.g. for cylindrical grains as advocated in conjunction with PMR or HAMR media. Finally, self-organization is limited to dimensions of order one to ten micrometer. To overcome these limitations two-step processes involving lithographic pre-patterning of substrates and subsequent nanoparticle assembly on the smaller scale of 100 nm – 10 nm have been proposed.^{100,101} This new concept could be used to push out the superparamagnetic limit. In rotating storage one would align the lithographically pre-patterned regions into a circumferential structure. For alternative probe storage devices one could choose an x-y pattern. Both topographic as well as chemical pre-patterning schemes have been proposed.

2.3.7.3 Nano-cluster

Another approach for controlling of FePt grain size is nano-cluster. In 2003, Stappert and co-workers reported a technique to prepare FePt nanoparticles from the gas-phase which aimed to obtain $L1_0$ ordered FePt nanoparticles by thermal sintering in the gas-phase. The particles in the size range 3–20 nm are generated via inert-gas condensation using DC-sputtering at very high pressures in the mbar range.¹⁰² This method shows great promising for controlling of the FePt grain size. However, to use such media for either

conventional (longitudinal or perpendicular) or bit-patterned media, similar obstacles as SOMA media have to be overcome.

2.4 Model for formation of crystallographic texture of Cr thin films

In this section, a model for the formation of crystallographic texture of Cr thin films is reviewed. The experiment design for the CrX underlayers deposition will be based on this model.

Cr thin films are commonly used as underlayers for thin film magnetic media. The role of such an underlayer is to induce the orientation of the c-axis of the upper magnetic layer. It is well known that sputtered Cr thin films have either (110) or (002) crystallographic texture or a mixture of the two, depending on the type of substrate and deposition conditions. To understand the dependence of the texture of Cr films on the sputtering conditions, Feng et al. proposed a model for the formation of the crystallographic texture of Cr thin films based on minimized surface energy.¹⁰³ Two mechanisms of the texture formation are proposed in their model. In the first mechanism (**Mechanism I**), the crystallographic texture originates from the preferential orientation of islands, which are nucleated on the substrate surface before a continuous film forms. During the film growth, if the islands can reach their equilibrium shape before they impinge into each other to form a continuous film, the islands prefer a (200) orientation and eventually the (200) texture forms. This mechanism occurs under the following circumstances:

- a) when substrate temperature is high ($>250\text{ }^{\circ}\text{C}$), which makes the adatoms more mobile and thus the equilibrium state is created more easily and faster;
- b) when the sputter power is low ($<25\text{ W}$), which the islands have more time to reach their equilibrium state; and
- c) when the Ar pressure is relatively low ($<1.33\text{ Pa}$), and the Ar ions have high a kinetic energy, which also makes the adatoms more mobile and thus the equilibrium state is created more easily.

In the second mechanism (**Mechanism II**), the crystallographic texture develops by the faster growth of grains with certain orientation after the film becomes a continuous film. If the islands cannot obtain their equilibrium shape before impingement, the (110) texture will develop due to the faster growth mechanism because the {110} planes for bcc Cr, have the lowest surface energy. During the development of the (110) texture after impingement, the smaller the impingement island size (higher concentration of islands), the easier the (110) texture develops. The mechanism takes place in the following conditions:

- a). When the substrate temperature is low (room temperature), in contrast to the case (a) of Mechanism I. In this case, the atoms are “quenched” onto the substrate. The islands are small and their concentration is high. They cannot reach their equilibrium orientation before impingement, therefore, after a continuous film is formed, the (110) texture will develop by faster growth mechanism.
- b). When the sputter power is high ($>100\text{ W}$), however this case will also create high surface temperature, which makes the formation of the (110) texture not obvious.

- c). When the Ar pressure is very low (<0.67 Pa), it means that the Ar ions have very high kinetic energy. This causes sputtered particles to have very high energy. When arriving on the growing islands, they create a lot of defects. Hence, the concentration of islands is increased, and therefore the (110) texture can develop easily.
- d). When substrate bias is applied. In this case, Ar ions bombard the film surface, having the same effect as in the case (c).¹⁰⁴

Chapter 3 Experimental Details

This chapter gives experimental details of this project and some descriptions and important remarks on experimental methods and the equipments.

3.1 Sputter deposition

3.1.1 Introduction to sputter deposition

All the thin films samples were prepared by an ultra-high vacuum magnetron sputter system. It is well known that the sputtering deposition method has been used to fabricate various kinds of the microelectronic devices and systems in many industries.¹⁰⁵ It is one of the most important methods in preparing the media and head thin films for magnetic recording purpose.^{106,107} The significant advantages of the sputtering deposition method, compared with other physical deposition methods, are its high yielding rates, good adhesion and controllability of structural and magnetic properties of the films.^{102,108}

The sputtering process is accomplished by applying a certain voltage of potential across the paralleled electrodes, which is bonded with a metal target and a selected substrate in a vacuum chamber that contains a sputtering gas. A glow discharge (also called plasma) is then ignited and maintained, where the sputtering gas particles continuously collide with the metal targets and generate the sputtered particles, which are later deposited on the substrate. Fig. 3.1 shows a greatly simplified cross-section image of a typical sputtering system.

Figure 3-1 Schematic cross-section image of a typical sputtering system¹⁰²

Usually, the target is connected to a negative power supply (either DC or RF power) and the substrate holder faces the target. The substrate holder may be grounded, floating, biased, heated, cooled, or some combination of these. A gas (such as argon) is introduced into the vacuum chamber to provide a medium in which a glow discharge can be initiated and maintained.

In the presence of the negative potential, both free electrons and nucleus are accelerated. However, due to the mass difference between the electrons and the nucleus, the gas atoms are dissociated and ionized, and the glow discharge is thus started. The accelerated positive ions strike the target plate and remove the neutral target atoms by their momentum gained from the electrical field. Those target atom clusters travel back into the plasma then condense on the top surface of the substrate to form a thin film.

Some other particles and radiation are produced during the sputtering deposition process: desorbed gases, x-rays, photons, secondary electrons and ions etc. All of them may affect film properties. In some cases, a bias potential (usually negative) is applied to the substrate, so that the growing film is subject to positive ion bombardment. The sputtered film properties will change greatly by this method. This is widely known as the bias sputtering or ion implanting.

3.1.2 Experimental procedure

All the thin films samples were prepared by a DSI home-designed ultra-high vacuum magnetron sputter system. A picture of this system is shown in Fig.3.2. The schematic diagram of this system is shown in Fig.3.3. This system has three chambers, and the main chamber is a four-target magnetron co-sputtering system with a heater behind the substrate holder. The temperature of the substrate could vary from room temperature to 400 °C, and the substrate temperature was measured by a thermocouple attached onto the backside of the substrate holder. Next to the main chamber is used to deposit carbon coating thin films. The base vacuum of the sputter chamber can reach about 5×10^{-9} Torr (6.65×10^{-7} Pa) at room temperature. The substrate holder rotated at approximately 40 rpm in order to ensure uniformity during deposition. The sputtering rate was adjusted by the sputtering power and the working Ar gas pressure. The film thickness was controlled by the sputtering time and was calibrated by TEM cross-sectional image. Fe₅₀Pt₅₀ alloy target was used to deposit FePt films in this thesis.

In order to study the effect of lattice mismatch on chemical ordering in epitaxial $L1_0$ FePt films, 30 nm thick (in order to minimize the strain from MgO substrate) Pt, Cr, $\text{Cr}_{95}\text{Mo}_5$ and $\text{Cr}_{90}\text{Mo}_{10}$ intermediate layers were used to adjust the lattice mismatch. FePt films were sputtered on MgO (200) single crystal substrates with or without intermediate layers at 350°C . The base pressure of the sputtering chamber was less than 1×10^{-8} Torr (1.33×10^{-6} Pa). The Pt and FePt films were deposited under an Ar gas pressure of 10 mTorr (1.3 Pa), while the Cr, $\text{Cr}_{95}\text{Mo}_5$ and $\text{Cr}_{90}\text{Mo}_{10}$ buffer layers were deposited with an Ar gas pressure of 3 mTorr (0.39 Pa). The experiment results were discussed in chapter 4.

Figure 3-2 DSI home-designed UHV sputter system

Figure 3-3 Schematic diagram of the UHV sputter system.

The FePt thin films with a structure of Glass / CrX (X = Mo, Ru, W, Ti) / Pt (4 nm) / FePt were prepared to study the structural and magnetic properties of the FePt films grown on the CrX alloy (X=Mo, Ru, Ti, W) underlayers. The targets used were Cr, Cr₉₀Mo₁₀, Cr₈₀Mo₂₀, Cr₉₀Ru₁₀, Cr₉₀Ti₁₀, Cr₉₀W₁₀, Fe₅₀Pt₅₀, and Pt. The deposition temperature for the CrX underlayers was fixed at 350°C and that for the Pt and FePt layers was varied from 150°C to 350°C. A reference sample was deposited directly on glass substrate at 350°C. The working pressures were 3mTorr (0.39 Pa), 10 mTorr (1.33 Pa, and 10 mTorr (1.33 Pa) for CrX, Pt, and FePt layers, respectively. The experimental results were discussed in chapter 5.

FePt:C samples were prepared by co-sputtering FePt alloy target and C target. The deposition temperature for the Cr₉₀Ru₁₀ underlayers was fixed at 350°C, while for the Pt and FePt:C layers varied from 200°C to 350°C. The working Ar pressures were 2 mTorr (0.26 Pa), 2 mTorr (0.26 Pa) and 10 mTorr (1.33 Pa) for the Cr₉₀Ru₁₀, Pt and FePt:C

layers, respectively. The volume concentration of C was varied up to 30 % in the FePt films. For the media used for read-write testing, the FePt:C films were deposited on commercial HOYA 2.5 inch glass substrates. The deposition temperature for the Cr₉₀Ru₁₀, Pt, and FePt:C layers was fixed at 350°C. Finally, 5 nm thick carbon overcoat was deposited on the top of the FePt:C films at room temperature when the samples were cooled down to room temperature in vacuum. The results of this part were discussed in chapter 6.

3.2 Structure Characterization

3.2.1 X-Ray Diffractometer

In this project, X-Ray Diffractometer (XRD) is used to characterize the structure properties of the FePt thin films, including the crystal orientation, epitaxial growth relationship, and chemical ordering degree.

3.2.1.1 Measurement of crystal orientation

The basic principle of x-ray diffraction is the Bragg's Law, which is given by:

$$n\lambda = 2d\sin\theta \quad (3-1)$$

where λ is the wavelength of the x-ray, d is the lattice spacing of the diffracting planes, θ is the angle between the x-ray beam and the diffraction planes, and n is an integer index.

Lattice parameters (also called lattice spacing) of (hkl) plane of the materials could be calculated by the following equations for different lattice group:

$$\text{Cubic: } \frac{1}{d^2} = \frac{(h^2 + k^2 + l^2)}{a^2} \quad (3-2)$$

$$\text{Hexagonal: } \frac{1}{d^2} = \frac{4}{3} \frac{(h^2 + hk + k^2)}{a^2} + \frac{l^2}{c^2} \quad (3-3)$$

Different scanning modes in x-ray diffraction have been used to obtain information of the thin film structure, in which the θ - 2θ scanning mode and the grazing scanning mode are most popular. In addition to these two scanning modes, the rocking curve mode is also popular in determining the texture quality and the off-spectral scans are used to further investigation the texture quality and epitaxial relationship between substrate underlayer and top layer.

(a). θ - 2θ scanning

A schematic illustration of the θ - 2θ scanning is shown in Fig. 3.4. In θ - 2θ scans, the sample stage and the detector are rotated simultaneously during the scans and the angle between the x-ray source and the films surface is always equal to the angle between the detector and the film surface. The dependence of the x-ray diffraction intensity on the direction of the incoming x-ray beam is commonly presented by a spectrum of the diffraction intensity vs. 2θ angle. In this mode, only the crystal planes, which are parallel to the thin film surface, diffract the x-rays. This scanning mode is the most convenient method to determine the crystallographic axis of fiber texture of thin films. However, it takes too long time to make a scan on the media thin film. This is because the thickness of the concerned layer is very thin and the intensity of diffraction x-ray is weak. Therefore, grazing scanning mode was generally used for thin film measurement.

(b) Off-spectral θ - 2θ scanning, phi (ϕ), psi (Ψ) scans

The physics mechanical of the off-spectral θ - 2θ scanning, phi (ϕ), psi (Ψ) scans are same with normal θ - 2θ scan. The only difference is the rotation mode during measurement. Fig. 3.5 show the rotation modes of a D8 discover HRXRD for different types off-spectral scans.

Figure 3-4 Schematic illustration of the θ - 2θ scanning mode.

Figure 3-5 Illustrations of different off-spectral scan models.

3.2.1.2 Order-disorder transformations characterization

In most substitution solid solutions, the two kinds of atoms A and B are arranged more or less at random on the atomic sites of the lattice. In solutions of this kind the only major effect of a change in temperature is to increase or decrease the amplitude of the thermal vibration. But, there are some solutions which have this random structure only at elevated temperatures. When these solutions are cooled below a certain critical temperature T_c , the A atoms arrange themselves in an orderly, periodic manner on one set of atomic sites, and the B atoms do likewise on another set. The solution is then said to be ordered or to possess a superlattice. When this periodic arrangement of A and B atoms persists over very large distances in the crystal, it is known as long-range order. If the ordered solution is heated above T_c , the atomic arrangement becomes more or less random again and the solution is disordered.

Since there is only a very slight change in the size of the unit cell and its shape on ordering, there will be practically very small change in the positions of the diffraction lines. But the change in the positions of the atoms must necessarily cause a change in line intensities. We can determine the nature of these changes by calculating the structure factor F for each atom arrangement:

a) *Complete disorder*. The atomic scattering factor of the “average” Fe-Pt atom is given by

$$f_{av} = (\text{atomic fraction Fe}) \times f_{Fe} + (\text{atomic fraction Pt}) \times f_{Pt} \quad (3-4)$$

There are four "average" atoms per unit cell, at 000 , $\frac{1}{2} \frac{1}{2} 0$, $\frac{1}{2} 0 \frac{1}{2}$ and $0 \frac{1}{2} \frac{1}{2}$. Therefore the structure factor is given by

$$F = f e^{2\pi i (hu+kv+lw)}, \quad (3-5)$$

$$F = f_{av} [1 + e^{\pi i(h+k)} + e^{\pi i(h+l)} + e^{\pi i(k+l)}] \quad (3-6)$$

$$\text{For hkl unmixed, } F = 4 f_{av} = (2 f_{Fe} + 2 f_{Pt}) \quad (3-7)$$

$$\text{For hkl mixed. } F = 0. \quad (3-8)$$

b) *Complete order*. Each unit cell now contains two iron atoms and two platinum atoms.

$$F = (f_{Fe} + f_{Pt}), \text{ for hkl unmixed,} \quad (3-9)$$

$$F = (f_{Pt} - f_{Fe}), \text{ for hkl mixed.} \quad (3-10)$$

Any departure from perfect long-range order in a superlattice causes the superlattice lines to become weaker. This departure from perfect order can be described by means of the *long-range order parameter* S . The structure factors of partially ordered FePt are given by

$$F = (2f_{Fe} + 2 f_{Pt}), \text{ for hkl unmixed,} \quad (3-11)$$

$$F = S (f_{Pt} - f_{Fe}), \text{ for hkl mixed.} \quad (3-12)$$

Comparing these equations with Eqs. (3-9, 3-10), we note that only the superlattice lines are affected. But the effect is a strong one, because the intensity of a superlattice line is proportional to $|F|^2$ and therefore to S^2 . Thus, by comparing the integrated intensity ratio of a superlattice and fundamental line, we can determine s experimentally.

For FePt systems, the long range ordering parameter S is given by¹⁰⁹

$$S = \left[\left(\frac{I_{001}}{I_{002}} \right) \times \left(\frac{F_f}{F_s} \right) \times \frac{(L \times A \times D)_f}{(L \times A \times D)_s} \right]^{1/2} \quad (3-13)$$

where I_{001} and I_{002} are the integrated intensities of the (001) and (002) peaks, respectively; F is the structure factor; L is the Lorentz polarization factor; A is the absorption factor; D is the temperature factor; and the subscripts f and s refer to the fundamental peak and superlattice peak, respectively.

3.2.2 Transmission Electron Microscopy

The Transmission Electron Microscope (TEM) is used to study the microstructure and morphology of the samples prepared in this project. In a TEM machine as shown in Fig. 3.6, a beam of collimated high-energy electrons (100 keV ~ 300 keV) pass through a specimen under vacuum. Electrons are diffracted by the atomic layer stacks inside the specimen and generate a pattern with black and white contrast. Fig. 3.7 shows the schematic TEM work modes. When we form images in TEM, we either form an image using the central spot, or we use some or all of the scattered electrons. The way we choose which electrons form the image is to insert an aperture into the back focal plane of the objective lens, thus blocking out most of the diffraction pattern except that which is visible through the aperture. If the direct beam is selected we call the resultant image a bright-field (BF) image [Fig. 3.72 (a)], and if we select scattered electrons of any form, we call it a low-resolution dark-field (DF) image [Fig.3.7 (b)]. If we tilt the incident

electron beam to form a diffraction image, we call it a high-resolution dark-field image [Fig.3.7 (c)]. If we pull out the aperture, both the direct electrons and the scattered electrons go through it, we can take a electron diffraction pattern [Fig.3.7 (d)]. To obtain lattice images, a larger objective aperture has to be selected that allows many beams including the direct beam to pass. The image is formed by the interference of the diffracted beams with the direct beam (phase contrast). If the point resolution of the microscope is sufficiently high and a suitable sample oriented along a zone axis, then high-resolution TEM (HRTEM) images are obtained. In many cases, the atomic structure of the specimen can directly be investigated by HRTEM. In the bright field mode of the TEM, mass-thickness and diffraction contrast contribute to image formation: thick areas, areas in which heavy atoms are enriched, and crystalline areas appear with dark contrast. In spite of the useful information obtainable from BF images, it should be mentioned that the interpretation of contrast is often impeded since these phenomena occur simultaneously. In the dark field mode of the TEM, the diffracted beam has interacted strongly with the specimen, and often very useful information is present in DF images, e.g., about planar defects, stacking faults or particle size. Electron diffraction is a collective elastic scattering phenomenon with electrons being scattered by atoms in a regular array (crystal). The incoming plane electron wave interacts with the atoms, and secondary waves are generated which interfere with each other (analogous to the Huygens principle for diffraction of light). This occurs either constructively (reinforcement at certain scattering angles generating diffracted beams) or destructively (extinguishing of beams). As in X-ray diffraction (XRD), the scattering event can be

described as a reflection of the beams at planes of atoms (lattice planes). The Bragg law gives the relation between inter-planar distance d and diffraction angle θ :

$$n \frac{h}{\sqrt{2meV}} = 2d \sin \theta \quad (3-14)$$

where

- $\sin \theta$ is proportional to $1/d$: diffraction angle gets bigger as d becomes smaller
- $\sin \theta$ is proportional to $1/\sqrt{eV}$: diffraction angle becomes bigger as electron kinetic energy becomes smaller
- Diffraction has same probability with $n = 1$ and $n = -1$: diffraction pattern is symmetric.

Since the wavelength λ of the electrons is known, inter-planar distances can be calculated from ED patterns. Furthermore, information about crystal symmetry can be obtained. Consequently, electron diffraction represents a valuable tool in crystallography.

3.2.3 Experimental procedure

A Philips X'pert HRXRD and a D8 discover HRXRD (both system with Cu K_α radiation) are used to characterize the crystallographic texture and order-disorder transformation of the films. θ - 2θ scan, omega (ω) scan, and Psi (ψ) scan are used to detect the texture, rocking curve, and epitaxial growth relationship of the samples, respectively. The typical 2θ scan angle range used in this project is from 20° to 80° . The rocking scan range is 20° . The Psi (ψ) scan angle range is 360° .

All the TEM specimens (plan view and cross-sectional samples) were prepared using traditional method: by mechanical lapping and dimpling followed by the ion milling. The grain size distributions were calculated by measuring the grain diameters of about 100 grains in the central areas of the TEM plan-view images, and the EDP simulations were performed with the free EDP simulation software (EMS)

There equipments were used in this project: JEM-2010, JEM-3010 and Philips CM 300-FEG-EM HRTEM, which operate at an accelerating voltage of 200kV, 300 kV and 300kV, respectively.

Figure 3-6 Schematic ray diagrams of transmission electron microscope (TEM).

Figure 3-7 Schematic TEM work modes (a) bright field image, (b) low resolution dark field image, (c) high resolution dark field image, and (d) Electron diffraction pattern.

3.3 Magnetic characterization

3.3.1 Vibrating Sample Magnetometer measurement

In this project, VSM is used to characterize the magnetic properties of the material used in magnetic recording in order to obtain its hysteresis loops, for example, M-H loop, major and minor hysteresis loops, initial magnetization curves and remanent magnetization curves. Vibrating Sample Magnetometer (VSM) is the most popular instrument used for accurately measuring the magnetic properties of recording media.¹¹⁰

In a VSM, the sample is suspended on a nonmagnetic rod which vibrates vertically at a frequency of about 60 Hz. The rate of change of magnetic flux in the pick-up coils is produced when the magnetized sample is mechanically vibrated. This vibration due to the resulting magnetization induced in the sample, induces a voltage across the terminals of the pick-up coils which is proportional to the magnetization of the sample by the following relationship:

$$V(t) = C \frac{df_i(t)}{dt} \quad (3-15)$$

where $f_i(t)$ represents the (changing) flux in the pick-up coils caused by the moving magnetic sample. Thereafter, the coil output voltage is combined with the output from the displacement transducer to produce a magnetization signal. Variations in vibration amplitude and frequency are then cancelled out. The signal is then detected by a high gain but narrow bandwidth “lock-in amplifier” and fed along with the applied field signal

to an x-y plotter to generate a hysteresis loop. The schematic of a VSM is shown in Fig.3.8.

Figure 3-8 A schematic of a VSM

Hysteresis loop measurement

For hysteresis loop measurements, a DC magnetic field (H_{max}) which is strong enough to saturate the magnetic sample was applied and then decreased step by step to $-H_{max}$. Similarly, for the reverse direction, the applied field was changed from $-H_{max}$ to H_{max} . At each step, the magnetic moment was measured and a hysteresis loop was thus obtained as shown in Fig 3.9.

Figure 3-9 Typical hysteresis loop and parameters measured by VSM.¹⁰⁸

The common parameters that characterize a hysteresis loop are:

Saturation Magnetization, M_s

Remanence magnetization, M_r

Coercivity, H_c

Squareness, $S = M_r/M_s$

Coercive squareness, $S^* = 1 - (M_r/H_c)(dM/dH)_{H=H_c}$

Switching field distribution, $SFD = (dM/dH)/H_c = \Delta H/H_c$

These parameters are extremely important in characterizing the recording performance of the thin film media. For example, the transition length, a , which is widely used to estimate the linear recording density of the recording media, is closely related with the M_r and H_c property. According to William-Comstock model mentioned in chapter one, the

transition length is: $a = \sqrt{\frac{M_r t d}{\pi H_c}}$, where t is the thickness of the magnetic layer and d is the flying height.¹⁰⁷ From this equation, it is easily found that ultra-high-density recording (smaller transition length) requires low $M_r t$ and high H_c .

The switching field of a particular grain in a material is not identical. Therefore, there is a distribution of the switching field, which is denoted by SFD. Theoretically, a magnetic material which is composed of uniform and well-segregated magnetic unit (with similar grain shape, grain size and crystal orientation) will give a narrow SFD that is desirable for narrow, stable magnetic transition in the medium. Generally, high S and S^* from the hysteresis loop will contribute to narrow SFD.

DC-demagnetization curve

The dc-demagnetization curve (DCD) is obtained by starting with an initially positively saturated sample. A small negative dc field ($-H$) is then applied and removed so that the remaining remanence $M_d(H)$ can be measured. This is again repeated for increasing values of H until negative saturation is reached. As shown in Fig. 3.10, the DCD curve is a plot of $M_d(H)$ against H .

Figure 3-10 A typical schematic DCD curve.

3.3.2 Alternating Gradient Force Magnetometer

The alternating gradient force magnetometer (AGFM) is used for the determination of hysteresis loop, DCD curves and time dependence measurements in this project.

In an AGFM, the sample is mounted on an extension rod attached to a piezoelectric element. An alternating gradient field produces a periodic force on a sample placed in a DC field of an electromagnet. The alternating field gradient exerts an alternating force on the sample proportional to the magnitude of the gradient field, the magnetic moment of the sample and the intensity of the applied field. The resulting deflection of the extension is transmitted to the piezoelectric sensing element. The output signal from the piezoelectric element is synchronously detected at the operating frequency of the gradient field. The signal developed by the piezoelectric element is greatly enhanced by operating at or near the mechanical resonant frequency of the assembly. A built-in software

function automatically determines the mechanical resonance and sets the appropriate operating frequency for the sample under study.

Alternating Gradient Force Magnetometer has enjoyed significant acceptance in present-day magnetic metrology because of its high sensitivity, easy operation and fast measurement. The AGFM has a noise floor of 10^{-8} emu compared to 10^{-6} emu for the VSM. The AGFM used in this project is Model 2900 MicroMag™ System (Princeton Measurement) with a maximum field of 20 kOe.

3.3.3 Magnetic Recording Properties Characterization: Read-write testing

In this project, Signal-to-Noise Ratio (SNR) and Noise level were used to characterize the FePt and FePt:C media recording performance. The read-write tests were carried out on an air bearing spin-stand (GUZIK S-1701) with a commercial 60 Gb/in² ring head.

The Noise Test measured a ratio between the amplitude of an HF signal written on a given track and the RMS (root mean square) value of the signal after the track was erased. The SNR was calculated using the following formula:

$$\text{SNR} = -20 \times \text{Log} (V_{\text{noiseRMS}}/\text{TAA}) \quad (3-16)$$

where TAA is the track average amplitude of an HF signal, and V_{noiseRMS} is the RMS value of a signal on an erased track (DC erased noise).

The test also measured the Crest Factor, which was the peak amplitude of the erased signal normalized to the envelope amplitude of a previously written HF signal (in percentage). For an extra pulse on the track, the Crest Factor was the ratio of the extra pulse amplitude to the HF signal envelope amplitude. This test was sensitive to the peak detector time constant, charge mode, and peak detection calibration.

3.3.4 Magnetic Force Microscope

The magnetic domain structure was measured by an MFM (Veeco-Dimension 3100). Magnetic force microscopy (MFM) is a form of AFM non-contact scanning using a magnetized tip to map magnetic field strength across the sample surface.¹¹¹ In MFM, a tapping cantilever equipped with a special tip is first scanned over the surface of the sample to obtain topographic information. Using Lift Mode, as described in Fig 3.11, the tip is then raised just above the sample surface. The surface topography was scanned while being monitored for the influence of magnetic forces, which are measured using the principle of force gradient detection. In the absence of magnetic forces, the cantilever has a resonant frequency f_0 . This frequency is shifted by an amount Δf proportional to vertical gradients in the magnetic forces on the tip, which can be detected in three ways: phase detection, frequency modulation and amplitude detection. During scanning, Lift-mode allows the imaging of relatively weak but long-range magnetic interactions while minimizing the influence of topography. Therefore, topographic features and MFM data obtained during the same scan, but can be processed and analyzed separately.

Figure 3-11 (a) Basic concepts of MFM (b) Geometry used in calculating the magnetic interaction (c) principle of AC force microscopy.

The magnetic domain structure was measured by an Veeco MFM (Dimension 3100) in this project. The schematic of the MFM is shown in Fig.3.12. During scanning, the amplitude ranged from 20 to 100 nm.

Figure 3-12 Schematic of AFM with tapping mode.

Chapter 4 Effects of lattice mismatch on chemical ordering in epitaxial L1₀ FePt films

4.1 Introduction

The growth of magnetic layers can be controlled by appropriate underlayers or seed layers. An adequate tensile stress, due to lattice misfit induced by the CrRu underlayer has been proposed to accelerate the phase transformation.²² Furthermore, the strain energy arising from lattice misfit between Ag/FePt¹¹ and Cu/FePt¹¹² has been found to favorably reduce the ordering temperature. These previous studies strongly suggest that the tensile stress in the FePt films originating from a lattice misfit between underlayers and FePt films may significantly reduce the ordering temperature of FePt. Now it is generally accepted that the tensile stress due to lattice mismatch at the interface between FePt film and underlayer may expand/shrink the a axis and shrink/expand the c axis of the FePt film and thus favors the ordering at relatively low temperatures. It is deduced that there should be a critical lattice mismatch which is most favorable for chemical ordering. A further understanding of the relationship between the lattice mismatch and the chemical ordering is of great scientific and technological interest. This study is to identify the relationships between the lattice mismatch, stress and the chemical ordering of the FePt films and their magnetic anisotropy constant.

4.2 Sample structure

In this work, 30 nm thick Pt, Cr, Cr₉₅Mo₅ and Cr₉₀Mo₁₀ intermediate layers were used to adjust the lattice mismatch, details can be found in Chapter 3. The sample structure is shown in Fig 4.1. The epitaxial growth models of this work are shown in Fig. 4.2.

FePt (5~60 nm)
Pt, Cr ₉₅ Mo ₅ , Cr ₉₀ Mo ₁₀ (30 nm)
MgO (200)

Figure 4-1 Illustration of sample structure.

(a) Schematic diagram of MgO (100)<001>|| FePt (001)<100>.

(b) Schematic diagram of $\text{MgO (100)}\langle 001 \rangle \parallel \text{Pt (001)}\langle 100 \rangle \parallel \text{FePt (001)}\langle 100 \rangle$.

(c) Schematic diagram of $\text{MgO (100)}\langle 001 \rangle \parallel \text{Cr (200)}\langle 110 \rangle \parallel \text{FePt (001)}\langle 100 \rangle$.

Figure 4-2 Epitaxial relationship models used in this thesis.

4.3 Results and discussion

4.3.1 XRD spectra

Fig. 4.3 shows the XRD spectra of 20 nm thick FePt films grown on the MgO substrates with different buffer layers. Since only FePt (00 n) diffraction peaks are observed, the films are strongly textured to the (001) planes. The intense FePt (001) peak and the appearance of the FePt (003) peak in all scans indicate a high degree of chemical ordering with the tetragonal c axis along the film normal. The Pt (200) and Cr (200) peaks from the intermediate layers are clearly observed. The unlabeled peaks (near 38° and 39°) are from the MgO substrates that have been confirmed by the XRD spectra of the pure MgO substrates (Figures not shown). The calculated lattice constant values of the intermediate layers grown on both the MgO and the glass substrates (Figures not shown) are listed in Table 4.1. The lattice constants of the intermediate layers grown on the MgO substrates are only slightly larger than that grown on the glass substrates, which illustrates that a 30 nm thick intermediate layer is enough for relaxing the strain from the MgO substrates. Fig. 4.4 shows the lattice parameter of Cr_{1-x}Mo_x as a function of Mo atomic ratio. As can be seen, the lattice parameters of Cr intermediate layers increase with the addition of Mo, which appears to follow the Vegard's law.¹¹³

Sample	Intermediate layer	a_1 (Å)	a_2 (Å)	Epitaxial relationship	ϵ (%)
A	Pt	3.928	3.923	MgO (100)<001> Pt (100)<001> FePt (001)<100>	2.23
B	Cr	2.884	2.878	MgO (100)<001> Cr (100)<110> FePt (001)<100>	5.88
C	Cr ₉₅ Mo ₅	2.898	2.891	MgO (100)<001> Cr (100)<110> FePt (001)<100>	6.33
D	Cr ₉₀ Mo ₁₀	2.915	2.911	MgO (100)<001> Cr (100)<110> FePt (001)<100>	6.89
E	MgO	4.211		MgO (100)<001> FePt (001)<100>	8.86

Notes:

a_1 : Lattice constant of intermediate layer (grown on MgO)

a_2 : Lattice constant of intermediate layer (grown on glass)

ϵ : Lattice mismatch

Table 4-1 Lattice constants of intermediate layers, epitaxial relationships, and lattice mismatches.

Figure 4-3 XRD spectra of samples: (a) A, (b) B, (c) C, (d) D, and (e) E.

The degree of the FePt (001) texture of the samples is characterized by their rocking curves. Fig. 4.5 shows the FePt (001) rocking curves of sample A, C, D and E, where the FWHMs (full width at half maximum) of all the FePt (001) rocking curves are smaller than 6° indicating a good FePt (001) texture of all the samples.

Figure 4-4 Lattice parameter of $\text{Cr}_{1-x}\text{Mo}_x$ with respect to Mo content.

Figure 4-5 FePt (001) rocking curves of samples A, C, D and E.

It is well understood that normal XRD $\theta/2\theta$ and ω scans can only get the structural information of the planes which are parallel to the film surface. In order to further investigate the structural properties of the current samples and to calculate the lattice constant of the FePt films, XRD off-specular $\theta/2\theta$ and ω scans were performed by a D8-discover HRXRD. Fig. 4.6 shows typical off-specular FePt (202) or (111) $\theta/2\theta$ scans and corresponding rocking curves of sample A, B, C, D, and E. The small FWHM values of FePt (202)/(111) rocking curves confirm that the FePt films grown on different buffer layers have a good texture. The lattice constant c of the FePt films was calculated from the FePt (003) peaks rather than (001) peaks, since the systematic errors were smaller at higher angles and tended to vanish at $\theta = 90^\circ$. The ratio c/a of the FePt films was calculated from the FePt (202) peaks. The ratio c/a of the FePt films was calculated from the FePt (202) peaks. The c/a of FePt films was calculated from FePt FePt (202) peaks. The calculated results are plotted in Fig 4.7 with respect to different buffer layer. As can be seen, the c holds the minimum while a holds maximum when $\text{Cr}_{95}\text{Mo}_5$ underlayer is used. The results also show that the c axes of the FePt films have been indeed shrunked and a axes have been expanded with different buffer layers.

Figure 4-6 Off-specular $\theta/2\theta$ scans and rocking curves of samples A, B, C, D, and E.

Figure 4-7 Lattice constants of FePt films with respect to different buffer layers.

4.3.2 Epitaxial relationship demonstration with high resolution X-ray diffraction

The epitaxial relationships between the FePt films and/or the intermediate layers and MgO substrates are investigated by off-specular phi scan.

The off-specular MgO (202) and FePt (202) phi (ψ) scans (45° and 44.1° away from the specular rod, respectively), MgO (202), Pt (202) and FePt (202) ψ scans (45° , 45° and 44.1° away from the specular rod, respectively) and MgO (111), Cr (101) and FePt (111) phi scans (45° , 45° and 44.1° away from the specular rod, respectively) are used to demonstrate the epitaxial relationships between FePt/MgO, FePt/Pt/MgO and FePt/Cr/MgO, respectively. The typical phi scan results are shown in Fig. 4.8. For sample E, four MgO {202} diffraction peaks with an even spacing of 90° are observed in the phi scan spectra and four FePt {202} peaks with the same spacing are also found at the same positions [Fig. 4.8(a)]. This indicates that the FePt (202) reflection follows the MgO (202) reflection, which confirms that the $L1_0$ FePt film is in fact epitaxially grown on the MgO (200) substrate. For sample A, four MgO {202}, four Pt {202} and four FePt {202} diffraction peaks are observed at the same locations [Fig. 4.8 (b)], which implies that the Pt buffer layer first epitaxially grows on the MgO substrate and then the FePt film turns to epitaxially grow on the Pt buffer layer. For sample B, four MgO {111}, four Cr {101} and four FePt {111} diffraction peaks are observed at the same locations. Fig. 4.8 (c) demonstrates that the Cr buffer layer epitaxially grows on the MgO substrate and then the FePt film turns to epitaxially grow on the Cr buffer layer.

The epitaxial relationships can be revealed by the direction of the momentum transfer at the peak projected to the film plane relative to the in-plane orientation of the MgO substrate. From the relative directions of the FePt films, intermediate layers and MgO substrates crystalline axes, the epitaxial relationships for samples E, A, and B can be derived as FePt (001)<100>||MgO (100)<001>, FePt (001)<100>||Pt (100)<001>||MgO (100)<001> and FePt (001)<100>||Cr (100)<110> || MgO (100)<001>, respectively.

Figure 4-8 Off-specular phi scans of samples (a) E, (b) A, and (c) B.

4.3.3 Microstructural properties

In order to get the direct proof of the epitaxial growth and to investigate the microstructure of the samples used in this chapter, HRTEM cross-sectional view of the samples was performed. Fig. 4.9 shows the typical TEM images. Figs. 4.9 (a), (b), (f) and (g) were taken with Philips CM300-FEG-EM HRTEM from a sample with the structure of MgO/ Pt (30 nm)/FePt (50 nm), which was fabricated under the same conditions as for sample A, and Fig. 4.10 was taken from Joel JEM-3010 HRTEM. Figs. 4.9 (c), (d) and (e) are simulated electron diffraction patterns for the MgO (200), Pt (200) and L_{10} FePt (001) single crystals with zone axis of [001], respectively. Fig. 4.9 (a) is a low magnification cross-sectional image, from which one can see that both FePt and Pt layers show a continuous microstructure and the thicknesses of the FePt and Pt layers are about 51 nm and 30 nm, respectively. This indicates that the actual thickness of the sputtered films has been well controlled with the method as introduced in chapter 3. In addition, the interface

between the FePt and Pt layers can be distinguished due to the different contrasts of the two layers which result from different atomic masses. Fig. 4.9 (b) is the corresponding selected area electron diffraction (SAED) pattern taken from Fig. 4.9 (a). The zone axis is FePt [001]. As revealed in Fig. 4.9 (b), only the FePt 0,0,1 and FePt 0,0,-1 diffraction spots appear, which is well consistent with the simulated electron diffraction pattern of the $L1_0$ FePt (001) single crystal, suggesting that the FePt film has a single crystal $L1_0$ structure. For the Pt film, the observed diffraction pattern is also consistent with the simulated diffraction pattern of the Pt (200) single crystal. This means that the crystal Pt (200) film has first grown on the MgO (200) single crystal substrate, and then the crystal FePt (001) film grown on the Pt (200) film. As shown in Fig. 4.9 (b), the FePt 0,2,2; 0,2,-2; 0,-2,2; 0,-2,-2 diffraction spots follow the Pt 0,2,2, 0,2,-2; 0,-2,2; 0,-2,-2 diffraction spots and the Pt 0,2,2 0,2,-2; 0,-2,2; 0,-2,-2 diffraction spots follow the MgO 0,2,2; 0,2,-2; 0,-2,2; 0,-2,-2 diffraction spots. This depicts that the FePt {022} planes follow the Pt {022} planes in the FePt [001] direction, and the Pt {022} planes follow the MgO {022} planes. Thus, the epitaxial growth is again confirmed. Figs. 4.9 (c), (d) show the MgO/Pt interface and FePt/Pt interface, respectively. From Fig. 4.9 (c), continuous lattice images from the MgO to Pt confirm the epitaxial growth. In Fig 4.9 (d), a sharp interface between FePt and Pt can be observed.

Figure 4-9 TEM images (a) low magnification cross-sectional image of sample with the structure MgO/Pt (30 nm)/FePt (50 nm); (b) corresponding SAED pattern of (a) ; (c) simulated EDP of MgO (200) [electron zone axis [001]]; (d) simulated EDP of Pt (200) [electron zone axis [001]]; (e) simulated EDP of FePt (001) [zone axis [001]]; (f) high magnification interface between MgO substrate and Pt layer; and (g) interface between FePt and Pt layers.

Fig. 4.10 (a) is the low magnification cross-sectional image of sample C. The image reveals that the thicknesses of the FePt and $\text{Cr}_{95}\text{Mo}_5$ layers are about 20 and 30 nm, respectively. Fig. 4.10 (b) is the high-resolution image of the interface between MgO and $\text{Cr}_{95}\text{Mo}_5$. As figure shows, the $\text{Cr}_{95}\text{Mo}_5$ is indeed epitaxially grown on the MgO. Fig. 4.10 (c) shows the interface between FePt and $\text{Cr}_{95}\text{Mo}_5$. A small area with a very poor texture is observed near the interface, most likely be due to the inter diffusion between FePt and $\text{Cr}_{95}\text{Mo}_5$. Fig. 4.10 (d) shows the interface between MgO and FePt of sample E. The epitaxial growth of the FePt film on the MgO substrate can be observed. In addition, some line dislocations are observed both at the interface and in the FePt film due to a

large lattice mismatch between FePt and MgO. This indicates that the lattice mismatch induced line dislocations occur not only at the interface but also in the FePt films during growth.

Figure 4-10 (a) low magnification cross-sectional image of sample C; (b) interface between Cr₉₅Mo₅ and MgO; (c) interface between Cr₉₅Mo₅ and FePt; and (d) interface between FePt and MgO (sample E).

4.3.4 Magnetic properties

Fig. 4.11 shows the hysteresis loops of samples A, B, C, D, and E. As figures show, the easy magnetization axes of all the samples are perpendicular to their film planes. These results confirm that for all the samples the tetragonal c axis is along the film normal as reflected by the XRD spectra.

Figure 4-11 Hysteresis loops of samples A, B, C, D, and E.

K_u of the samples was measured by torque. The measured K_u values of all the samples are listed in Table 4.2. The K_u first increases with increased lattice mismatch from 2.23% to 6.33%, then decreases with further increased lattice mismatch.

Sample	ϵ (%)	$K_u (\times 10^7 \text{ erg/cc})$
A	2.23	1.8
B	5.88	2.4
C	6.33	3
D	6.89	2.6
E	8.86	1

Table 4-2 Lattice mismatch and K_u of all the samples.

Fig 4.12 shows the typical MFM images of samples A, C and E. The FePt film grown on the Cr underlayer (sample C) shows the smallest domain size, which should be due to the inter-diffusion between Cr and FePt as revealed by TEM cross sectional image [Fig. 4.10 (c)]. These results are similar to that of the FePt films grown on the glass substrates with CrRu underlayers.¹¹⁴ For sample E, a smaller domain size compared to sample A may be due to a larger lattice misfit at the FePt/MgO interface than that between FePt and Pt, and thus more defects are formed at the MgO/FePt interface and/or in the FePt films and may act as the pinning sites.

Figure 4-12 MFM images of samples A, C, and E. [Scale: 5 $\mu\text{m} \times 5 \mu\text{m}$]

4.3.5 Influence of lattice mismatch on chemical ordering

Fig. 4.13 summarizes the lattice constants a and c , tetragonality represented by c/a , $(I_{001}/I_{002})^{1/2}$ (I_{001} and I_{002} are the integrated intensities of FePt (001) and (002) peaks, respectively), and K_u with respect to the lattice mismatch. The lattice constants of the FePt films are calculated from the FePt (001) and (202) peaks. For the long range ordering parameter is proportional to $(I_{001}/I_{002})^{1/2}$, $(I_{001}/I_{002})^{1/2}$ is used to characterize the variation of the chemical ordering degree in this thesis. The lattice constants of the

intermediate layers are also calculated from the XRD spectra. The lattice mismatches are calculated from the following equation

$$\varepsilon = (a_s - a_f) / a_s \quad (5-1)$$

where ε is the lattice mismatch, a_s is the lattice constant of the substrate or intermediate layer, and a_f is the lattice constant of the FePt film. In calculation, the a_f value of the FePt films is fixed at 3.838 Å. The a_s values for the MgO substrates and Pt intermediate layers are equal to the calculated lattice constants, while for Cr, Cr₉₅Mo₅ and Cr₉₀Mo₁₀ layers, a_s is equal to $\sqrt{2}a$ rather than the calculated lattice constant a , according to the epitaxial relationships. The calculated lattice mismatches are also listed in Table 4.1. The results manifest that the c values decrease with an increase in lattice mismatch from about 2.23% to 6.33%. Upon further increase of ε , c increases in turn. On the other hand, the variation of a illustrates a controversial behavior to that of c with increased lattice mismatch. The ratio of c/a holds a minimum about 0.9466, while both $(I_{001}/I_{002})^{1/2}$ and K_u hold maxima at $\varepsilon \cong 6.33\%$.

The present results depict that there is a critical lattice mismatch at $\varepsilon \cong 6.33\%$, which is the most favorable for improving the chemical ordering of the FePt films. It has been generally accepted that an adequate tensile stress due to a lattice misfit is considered to play a significant role in the formation of the L1₀ ordered structure.¹¹⁵ In this study, the strain may increase with an increase in lattice mismatch from about 2.23% to 6.33%, which can help expand the a axis and compress the c axis of the FePt films, and thus favors the chemical ordering of the FePt films. Upon further increase of the lattice mismatch, the strain might be relaxed due to the formation of dislocations at the

interfaces, which results in a lower degree of chemical ordering. In order to confirm the above suggestion, the residual stresses in the epitaxially grown FePt films are measured by high resolution XRD and will be discussed in next section.

Figure 4-13 Lattice constants of a and c , tetragonality represented by c/a , $(I_{001}/I_{002})^{1/2}$, and K_u as a function of lattice mismatch.

4.3.6 Residual stress in epitaxially grown FePt thin films

In this section, the relationship between stress and lattice mismatch is studied. For the samples cut for TEM observation, two series of samples were re-fabricated under the same sputter condition i.e. with a structure of MgO/Pt (30 nm)/FePt (5 ~ 60 nm) to study the influence of FePt film thickness on the residual stress and another with a structure of MgO/FePt (40 nm) and MgO/X (X= Pt, Cr) (30 nm)/FePt (40 nm) to study the influence of the lattice mismatch on the residual stress.

Normally, the origin of residual stresses in thin films includes epitaxial stresses, thermal stresses, and intrinsic or growth stresses.

- a). Epitaxial stress:** Epitaxial stresses arise when films have perfectly coherent interfaces with their substrates, i.e., when the crystal lattices in the film and substrates line up perfectly.
- b). Thermal stress:** When a film on a substrate is subjected to a temperature change, a differential thermal expansion will result in thermal stresses in the film and substrate.
- c). Intrinsic or growth stress:** It is often observed that stresses developed in films during deposition or growth. These stresses are not due to lattice mismatch or thermal mismatch strains and are therefore called intrinsic or growth stresses. These stresses arise because the films are generally deposited under non-equilibrium conditions. In general, any redistribution of matter will result in film stresses, since the film is constrained by the substrate. In this study, the epitaxial stress should be the dominant residual stress in the epitaxial growth FePt films.

In this study, X-ray diffraction is used to measure the residual stress in the FePt films. To calculate the strain and stress from x-ray data, the “ $\sin^2\psi$ method” is used:¹¹⁶

$$(d-d_0)/d_0 = [(1+\nu)/E] \sigma_\phi \sin^2\psi - (\nu/E) (\sigma_{11} + \sigma_{22}) \quad (4-2)$$

where d is the measured inter-planar spacing at different ψ , d_0 is the measured inter-planar spacing at $\psi = 0^\circ$. ϕ is the incident angle, ψ is the inclination angle ($\psi = \theta - \phi$), ν is poisson's ratio, and E is Young's modulus

- (i) Measure d with varying ϕ for a specific set of crystallographic planes.
- (ii) Plot $d(\psi)$ vs. $\sin^2\psi$
- (iii) Fit data to a straight line and find the line slope.
- (iv) Calculate stresses using the stiffness matrix.

During calculation, the d used in this project was the measured c value of the FePt films. Poisson's ratio (ν) and Young's modulus (E) values of pure bulk Fe and Pt bulks are used to evaluate the variation of the residual stresses in the FePt films due to the lack of the data for the FePt alloy. For the pure bulk Fe, Young's modulus and Poisson's ratio are 211 GPa and 0.29,¹¹⁷ respectively, and for the pure Pt, Young's modulus and Poisson's ratio are 168 GPa and 0.38, respectively.¹¹⁵

Fig 4.14 shows the $d(\psi)$ - $\sin^2\psi$ curves of the FePt films epitaxially grown on the MgO substrates. The linear plots of d vs. $\sin^2\psi$ are observed in all the samples, suggesting that the residual stresses be biaxial or uniaxial. Fig. 4.15 shows the thickness dependence of

the residual stress in the FePt films with Pt buffer layers. As can be seen from the figure, the stress decreases sharply with increasing FePt film thickness from 5 nm to 10 nm. With further increased FePt film thickness, the stress decreases slightly. We did not have the stress data for the 20 nm thick FePt film grown on the Pt buffer layer, so we just compare the stress value of the 10 nm thick FePt film on the Pt buffer layer with that of the 20 nm thick FePt films on the MgO and MgO/Cr. According to the above stress variation with film thickness, the 20 nm thick FePt film on MgO/Pt should have a stress value between 30 and 37 MPa. Fig. 4.16 shows the lattice mismatch dependence of stress in the FePt films. The variation of stress with respect to on lattice mismatch is well consistent with the trend of the chemical ordering degree, confirming that the residual stress offers a driving force for chemical ordering. Recently, Chih-huang Lai and co-works also demonstrated that a dynamic stress could induce low-temperature ordering of FePt.¹¹⁸ These results suggest that stress (static and dynamic) induced ordering mechanism be one of the most promising methods to overcome the barrier for the FePt media application.

Figure 4-4-14 $d(\psi)$ - $\sin^2\psi$ curves of FePt films deposited on MgO/X (X= Pt, Cr) with different FePt film thicknesses. The unit of the Y axis of all the figures is Angstrom.

Figure 4-15 Thickness dependence of residual stress in FePt films.

Figure 4-16 Lattice mismatch dependence of residual stress in FePt films.

4.4 Conclusion

A series of FePt films were sputter-deposited on MgO (200) substrates with or without intermediate layers. The epitaxial relationships between FePt/intermediate layer/MgO were identified with HRXRD, and the epitaxial growth was confirmed with HRTEM. The relationships between lattice mismatch, residual stress, chemical ordering, and Ku for the epitaxial FePt films were investigated and summarized as follows:

- 1) The lattice parameters of Cr intermediate layers increase with the addition of Mo, which appears to follow Vegard’s law.

- 2) With different buffer layers, the lattice mismatch between FePt and buffer layer could be adjusted from 2.23% to 8.66%.
- 3) The epitaxial relationship between MgO/Pt/FePt, MgO/Cr_xMo_{1-x}/FePt, and MgO/FePt were identified with HRXRD as MgO(100)<001>||Pt(100)<001>|| FePt(001)<100>, MgO(100)<001>||Cr(100)<110>||FePt(001)<100>, and MgO(100)<001>||FePt(001)<100>, respectively.
- 4) The c value decreased with an increase in lattice mismatch from about 2.23% to 6.33%. Upon further increase of ϵ , c increased in turn. On the other hand, the variation of a illustrated a controversial behavior to that of c with increased lattice mismatch. The ratio of c/a held a minimum about 0.9466, while both $(I_{001}/I_{002})^{1/2}$ and K_u held maxima at $\epsilon \cong 6.33\%$. Thus, a critical lattice mismatch near 6.33% was found to be the most suitable for improving the chemical ordering of the FePt films.
- 5) The residual stress decreased with increased FePt film thickness. The variation of residual stress with lattice mismatch was consistent with that of chemical ordering, suggesting that a static tensile stress from lattice mismatch favored to the phase transformation.

Chapter 5 FePt films deposited on chromium intermetallic underlayers

5.1 Introduction

In Chapter 4, a strain induced ordering mechanism was demonstrated, and a critical lattice mismatch around 6.33% was found to be the most suitable for improving the chemical ordering and keeping the epitaxial stress in FePt films. However, the work introduced in Chapter 4 was carried out on MgO single crystal substrates. As far as the industrial application is concerned, it is essential to deposit magnetic recording media on commercial disk substrates, such as glass disks, Al alloy disks, etc. Thus, to apply the proposed idea to actual applications, it is necessary to develop CrMo(200) underlayers on glass and/or Al alloy substrates. It is well known that the Cr based alloy (CrX) (X is Mo, Ru, W, V, or Ti, etc.) underlayers and/or seedlayers have been widely used in commercial longitudinal media. Effort was made to deposit the FePt films on the CrMo seeded glass substrates at low temperatures,¹¹⁹ but the crystallographic orientation of the FePt films was random. Thus, the development of the $\text{Cr}_{1-x}\text{X}_x$ (200) underlayers on the glass substrates is of both scientific and technological interests. In this chapter, the structural and magnetic properties of the FePt films deposited on the $\text{Cr}_{1-x}\text{Mo}_x$ underlayers at relatively low temperatures are mainly introduced. A brief comparison of the FePt films grown on the CrX (X = Mo, Ru, W, Ti) underlayers is also introduced. Finally, the study of the initial layers for the FePt films is addressed.

5.2 Sample structure

The samples used in this work has a structure of Glass / CrX (X = Mo, Ru, W, Ti) / Pt (4 nm) / FePt (Fig 5.1), details can be found in Chapter 3.

FePt (1~80 nm)
Pt (0 or 4 nm)
CrX (0~80 nm)
Glass

Figure 5-1 Illustration of sample structure.

5.3 Results and discussion

5.3.1 Influence of Mo content

In this series of experiments, the deposition temperature for all the films is fixed at 350°C and the thicknesses of the FePt, Pt and Cr_{1-x}Mo_x layers are fixed at 20 nm, 4 nm and 30 nm, respectively. The Mo content in the underlayers varies from 0 to 20%.

Fig. 5.2 shows the XRD spectra of the FePt films deposited on the CrMo underlayers containing various contents of Mo. For the FePt film grown on the pure Cr underlayer, in addition to an intense (001) peak, a FePt (111) peak is also observed [Fig. 5.2 (a)], which indicates that the (001) preferred orientation of the film is still not strong. When the Cr₉₀Mo₁₀ underlayer is used, the FePt (111) peak disappears, which implies an improved (001) preferred orientation [Fig. 5.2 (b)]. While for the FePt film grown on the Cr₈₀Mo₂₀

underlayer, the FePt (001) peak disappears and only very weak FePt (111) peak appears [Fig. 5.2 (c)]. It is expected that a (001) preferred FePt film can hetero-epitaxially grow on a Cr (200) underlayer with a crystallographic relationship of Cr (200)[110] $\parallel$ FePt (001)[100]. Thus, the texture evolution of the FePt films should be mainly governed by the texture of the $\text{Cr}_{1-x}\text{Mo}_x$ underlayers in this study. For the pure Cr underlayer, both the Cr (110) and Cr (200) peaks appear [Fig. 5.2 (a)], while for the $\text{Cr}_{90}\text{Mo}_{10}$ film only the Cr (200) peak appears [Fig. 5.2 (b)]. In addition, the Cr (200) peak shifts from 64.5° to 63.86° , which corresponds to the expansion of the lattice constant a when Mo is doped into the Cr underlayer. However, only a weak Cr (110) for the $\text{Cr}_{80}\text{Mo}_{20}$ layer appears in this deposition condition [Fig. 5.2 (c)].

Figure 5-2 XRD spectra of FePt films deposited at 350°C on Cr underlayer (a), $\text{Cr}_{90}\text{Mo}_{10}$ underlayer (b), and $\text{Cr}_{80}\text{Mo}_{20}$ underlayer (c).

The growth mechanism of the Cr and Cr₉₀Mo₁₀ underlayers with (200) preferred orientation in this study can be explained by Mechanism I as introduced in section 2.4. However, the growth mechanism for Cr₈₀Mo₂₀ underlayer deviates from the above model, which may be due to the change of the melting point and surface energy after too much Mo is doped.

The hysteresis loops of the FePt films deposited on the Cr, Cr₉₀Mo₁₀, and Cr₈₀Mo₂₀ underlayers at 350°C were measured by VSM as shown in Fig. 5.3. The loops for the FePt films grown on the Cr and Cr₉₀Mo₁₀ underlayers show a strong perpendicular magnetic anisotropy, which confirms that the *c* axis is perpendicular to the film surface, while for the FePt film grown on the Cr₈₀Mo₂₀ underlayer, the loop reveals a random anisotropy distribution. The ratios of the out-of-plane H_c to the in-plane H_c for the FePt films grown on the Cr₉₀Mo₁₀ and Cr underlayers are 0.5 and 1, respectively, indicating that the FePt films grown on the Cr₉₀Mo₁₀ underlayers have a stronger perpendicular anisotropy than those grown on the pure Cr underlayers. The nucleation fields for the FePt films grown on the Cr and Cr₉₀Mo₁₀ underlayers are -1450 Oe and -1890 Oe, respectively. The above results depict that the Cr₉₀Mo₁₀ underlayers are more suitable for growing the FePt films with (001) preferred orientation.

5.3.2 Influence of Cr₉₀Mo₁₀ underlayer thickness

In this section, the thicknesses of the FePt and Pt layers are 20 nm and 4 nm, respectively, and those of the Cr₉₀Mo₁₀ underlayers vary from 0 nm to 20 nm. The deposition

temperature for all the layers is 350°C. The effect of the Cr₉₀Mo₁₀ underlayer thickness on the structural properties of the FePt films is presented.

Figure 5-3 Hysteresis loops of FePt films deposited at 350°C on Cr underlayer (a), Cr₉₀Mo₁₀ underlayer (b), and Cr₈₀Mo₂₀ underlayer (c).

The evolution of the crystallographic structure of the FePt films with respect to the Cr₉₀Mo₁₀ underlayer thickness is shown in Fig. 5.4. Without a Cr₉₀Mo₁₀ underlayer, the FePt film shows a (111) preferred orientation, which is a chemically disordered face centered cubic (fcc) phase reflected by its soft magnetic properties. Upon introduction of a 5 nm thick Cr₉₀Mo₁₀ underlayer, the FePt (001) peak appears, indicating the occurrence of the chemical ordering. The intensity of the FePt (001) peak increases with an increase in underlayer thickness, which implies an improved degree of crystallization degree and a (001) preferred orientation. The full width at half maximum of the FePt (001) peak

decreases from about 1.3° to 0.9° with increased underlayer thickness from 5 nm to 20 nm, suggesting an increase of FePt (001) oriented grain size. In addition, the peak between 45° and 50° shifts from about 47.3° to 48.4° with increased $\text{Cr}_{90}\text{Mo}_{10}$ underlayer thickness. This peak is indexed as Pt (200)+FePt (200)+(002) in this study. It is known that the diffraction peaks Pt (200), L1_0 FePt (200), fcc FePt (200) and L1_0 FePt (002) are located at 46.4° , 47.305° , 47.66° and 48.929° [Appendix A], respectively. The Pt (200) peak for the 4 nm Pt layer is very weak and the proportion is very small compared with that of the FePt (200)+(002) peak as shown in the reference XRD spectrum [Fig 5.4]. Though the fcc FePt (200) peak locates at a slight higher angle than the L1_0 FePt (200) peak, the ordering degree of the FePt films increases with the underlayer thickness as discussed below. Thus, the proportion of the fcc FePt (200) peak should not increase. Based on the above analysis, the shift of the peak to a higher angle should be mainly due to the increase of the proportion of the L1_0 FePt (002) peak.

For the long range ordering parameter is proportional to $(I_{001}/I_{002})^{1/2}$ (I_{001} and I_{002} are the integrated intensities of FePt (001) and (002) peaks, respectively),¹²⁰ $(I_{001}/I_{002})^{1/2}$ is used to characterize the variation of the chemical ordering degree in this thesis. The calculated $(I_{001}/I_{002})^{1/2}$ values increase from about 0.7 to 1.04 with increased $\text{Cr}_{90}\text{Mo}_{10}$ underlayer thickness from 5 nm to 20 nm. It is well understood that the texture quality of a thin film in epitaxial growth is largely affected by the texture quality of its underlayer and/or substrate. Therefore, the increase of the ordering degree with increased underlayer thickness may be due to that the orientation and crystallinity of the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers are improved, and thus the improved interface quality between FePt films and

underlayers is more favorable for the strain-induced chemical ordering in the FePt films.¹²¹

Figure 5-4 XRD spectra of FePt films deposited at 350°C on $\text{Cr}_{90}\text{Mo}_{10}$ underlayers with different $\text{Cr}_{90}\text{Mo}_{10}$ layer thicknesses.

5.3.3 Influence of substrate temperature

In this section, the thicknesses of the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers, Pt buffer layers and FePt films are 20 nm, 4 nm and 20 nm, respectively. The deposition temperature for the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers is 350°C and that for the Pt and FePt layers varies from 150°C to 350°C. The effects of the substrate temperature on the structural and magnetic properties of the FePt films are discussed.

Fig. 5.5 shows the XRD spectra of the FePt films deposited on the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at various substrate temperatures. As mentioned in the previous section, the FePt film without a $\text{Cr}_{90}\text{Mo}_{10}$ underlayer has a (111) preferred orientation [Fig. 5.4]. For the FePt film grown on the $\text{Cr}_{90}\text{Mo}_{10}$ underlayer at 150°C , a FePt (200) peak dominates. A weak FePt (001) superlattice peak observed at 200°C implies that the chemical ordering has occurred. With an increase of the substrate temperature, the intensity of the FePt (001) peak increases, while the FePt (111) peak weakens and eventually disappears at 350°C , which implies an improved (001) preferred orientation. In addition, the peak between 45° and 50° shifts to a higher angle with increased substrate temperature due to the increase of the ordering degree.

Figure 5-5 XRD spectra of FePt films deposited on $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at various substrate temperatures ($150^\circ\text{C} \sim 350^\circ\text{C}$). The reference spectrum is from a 4 nm thick Pt film deposited on $\text{Cr}_{90}\text{Mo}_{10}$ underlayer at 350°C .

The selected area electron diffraction (SAED) patterns of the FePt films produced at different substrate temperatures are shown in Fig. 5.6. As the substrate temperature is 150°C, only some fundamental diffraction rings appear. At 200°C, weak (110) reflections confirm the occurrence of the chemical ordering and a low degree of chemical ordering of the FePt film. At 300°C, a weak (001) reflection indicates the presence of (100) and/or (010) oriented grains. The (001) reflection is not observed from the FePt films grown at 150°C and 200°C, because of a low ordering degree and/or a weaker (001) reflection compared to some other reflections. At 350°C, the (001) reflection disappears again, which may be due to the improved FePt (001) preferred orientation. Typical TEM bright-field and dark-field images of the FePt films deposited at different temperatures shown in Fig. 5.7 reveal that the FePt films grown on the Cr₉₀Mo₁₀ underlayers have a continuous microstructure.

Fig. 5.8 shows both the out-of-plane and the in-plane hysteresis loops of the FePt films deposited on the Cr₉₀Mo₁₀ underlayers at different substrate temperatures. In order to highlight the effect of the Cr₉₀Mo₁₀ underlayer, the hysteresis loops of a reference FePt film deposited directly on the glass at 350°C are also included in the graph. The FePt film grown on the glass at 350°C is magnetically soft (both in-plane and out-of-plane $H_c < 100$ Oe) and has an in-plane anisotropy. Both the in-plane and the out-of-plane coercivities of the FePt film deposited on the Cr₉₀Mo₁₀ underlayer at 150°C are larger than 550 Oe, which is much larger than that of the references sample. This might suggest that a very weak chemical ordering process has occurred. As the substrate temperature is 200°C, both the in-plane and the out-of-plane coercivities of the FePt film are larger than 1000

Oe, which should be mainly attributed to the formation of the ordered $L1_0$ FePt phase as reflected from the XRD spectrum [Fig. 5.5] and SAED pattern [Fig. 5.6]. When the temperature is higher than 250°C, the films show the out-of-plane anisotropy. This further confirms that the c-axis is perpendicular to the film surface. When the substrate temperature is $\geq 300^\circ\text{C}$, a high squareness (M_r/M_s) value (~ 1) is obtained.

Figure 5-6 SAED patterns of FePt films deposited on $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at 150°C, 200°C, 300°C, and 350°C, respectively. [Note: 1: FePt (001); 2: FePt (110); 3: FePt (111); 4: Cr (110); and 5: FePt (200), (002)].

Figure 5-7 TEM micrographs of FePt films deposited on $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at 150°C (a) and (b), and 350°C (c) and (d). Here (a) and (c) are the bright-field micrographs and (b) [taken from FePt (200)] and (d) [taken from FePt (110)] are the dark-field micrographs.

$(I_{001}/I_{002})^{1/2}$, lattice constant c , out-of-plane H_c ($H_c(\perp)$), and magnetic anisotropy K_u as a function of substrate temperature are summarized in Fig. 5.9. $(I_{001}/I_{002})^{1/2}$ increases and c decreases with increased substrate temperature, which confirms the improved ordering degree. The out-of plane coercivity shows a similar tendency to that of the $(I_{001}/I_{002})^{1/2}$ with the increased substrate temperature. K_u is calculated by $K_u = H_k \bullet M_s / 2$ with the anisotropy field H_k being estimated by extrapolating the magnetization curves. The error bar is estimated from the area enclosed by the in-plane hysteresis loop. The estimated K_u

increases from about 9.3×10^6 to 1.31×10^7 erg/cm³ with increased substrate temperature from 250°C to 350°C.

Figure 5-8 Out-of-plane and in-plane hysteresis loops of FePt films deposited on Cr₉₀Mo₁₀ underlayers at various substrate temperatures. FePt film deposited on glass substrate is included as a reference.

Figure 5-9 $(I_{001}/I_{002})^{1/2}$, lattice parameter, c , out-of-plane coercivity, H_c , and magnetic anisotropy, K_u , as a function of substrate temperature.

The FePt thin film deposited directly on the glass at 350°C is a disordered fcc phase and has a (111) preferred orientation, while the FePt films grown on the Cr₉₀Mo₁₀ underlayers show a preferred orientation changing from FePt (111) to FePt (200) or FePt (001). It is well known that the evolution of the film texture during the film growth should obey the minimum energy principle. For (111) plane is the most close-packed plane of the fcc structure and has the smallest surface energy, the (111) textured FePt film on the glass substrate indicates that the texture of the FePt films is governed by the surface energy during growth. For the FePt films grown on the (200) textured Cr₉₀Mo₁₀ underlayers,

from the epitaxial growth point of view, the texture of the FePt films should have the following orientation relationships: $L1_0$ FePt (001)[100] $\parallel$ Cr (200)[110] for the $L1_0$ FePt films and fcc FePt (200)[001] $\parallel$ Cr (200)[110] for the fcc FePt films. Thus, the (200) or (001) textured FePt films on the (200) textured $Cr_{90}Mo_{10}$ underlayers are due to the occurrence of epitaxial growth. With the $Cr_{90}Mo_{10}$ underlayers, the ordering process of the FePt films can start at a relatively low temperature (about 200°C), which is ascribed to the lattice mismatch between FePt film and $Cr_{90}Mo_{10}$ underlayer, can expand the a axis and shrink the c axis of the FePt film, and thus favors the ordering at low temperatures.

5.3.4 Influence of FePt film thickness

In this section, the thicknesses of the $Cr_{90}Mo_{10}$ underlayers and Pt buffer layers are 20 nm and 4 nm, respectively, while the FePt film thickness varies from 10 nm to 80 nm. The deposition temperature for all the layers is about 350°C. The dependence of the structural and magnetic properties of the FePt films on the film thickness is discussed.

Fig. 5.10 shows the XRD spectra of the FePt films of different thicknesses. When the FePt film thickness is 10 nm, broad and weak FePt (001) and FePt (200)+(002) peaks appear, indicating small grain sizes of the films. With an increase in FePt film thickness, the FePt (001) and FePt (200)+(002) peaks become stronger and sharper, which suggests an increase in grain size. When the film thickness is 40 nm, a weak FePt (111) peak appears. Upon further increase of the FePt film thickness up to 80 nm, the FePt (111)

peak becomes stronger. The appearance of the FePt (111) peak indicates the deterioration of the (001) preferred orientation in the FePt films.

Figure 5-10 XRD spectra of FePt films with different thicknesses deposited on $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at 350°C .

Fig. 5.11 shows the in-plane and out-of-plane hysteresis loops of the FePt films with various thicknesses. A perpendicular anisotropy is observed from all the FePt films. When the FePt film thickness increases from 10 nm to 40 nm, the squareness S ($S = M_r/M_s$) of the films shows a value about 1. With the further increase of the FePt film thickness up to 80 nm, the squareness decreases to about 0.5.

Figure 5-11 Out-of-plane and in-plane hysteresis loops of FePt films with different thicknesses.

$(I_{001}/I_{002})^{1/2}$ and $H_c(\perp)$ as a function of the FePt film thickness are summarized in Fig. 5.12. $(I_{001}/I_{002})^{1/2}$ increases while $H_c(\perp)$ decreases with increased FePt film thickness. Usually, the coercivity is strongly dependent on the chemical ordering due to the dependence of the magnetic anisotropy on the chemical ordering. In addition, the coercivity of a magnetic film is also an extrinsic property which is closely related to the microstructure of the film and the magnetization reversal mechanism.¹²²

Figure 5-12 $(I_{001}/I_{002})^{1/2}$ and out-of-plane H_c ($H_c (\perp)$) as a function of FePt film thickness.

To understand the coercivity mechanism of the FePt films used in this study, the angular dependence of the **coercivity** (not remanence coercivity) of the samples is measured at various angles from 0° (out-of-plane) to 90° (in-plane) as shown in Fig. 5.13. An ideal domain wall motion mode follows:

$$\frac{H_c(\theta)}{H_c(\perp)} = \frac{1}{\cos(\theta)} \quad (0^\circ \leq \theta < 90^\circ) \quad (5-1)$$

where θ is the angle between a magnetic field applied and a magnetic easy axis, while an ideal rotation mode follows:

$$\frac{H_c(\theta)}{H_c(\perp)} = \left(\sin^{\frac{2}{3}} \theta + \cos^{\frac{2}{3}} \theta \right)^{\frac{3}{2}} \quad (\theta \leq 45^\circ) \quad (5-2)$$

$$\frac{H_c(\theta)}{H_c(\perp)} = \frac{1}{2} \sin 2\theta \quad (\theta \geq 45^\circ) \quad (5-3)$$

The profile of the angular dependence of the coercivity for the 80 nm thick FePt film shows a typical domain-wall motion predominant model. The magnetic reversal mechanism deviates from the ideal domain wall motion mode and approaches closely a rotational mode with decreased FePt film thickness. The profile of the angular dependence of the coercivity for the 10 nm thick FePt film shows a rotation predominant model. Therefore, the discrepancy of the coercivity with respect to ordering degree may be due to the variation of the magnetic reversal mechanism from domain-wall motion predominant model to rotation predominant model.

Figure 5-13 Angular dependence of coercivity with respect to FePt film thickness.

5.3.5 Microstructure of FePt films with different underlayer and magnetic layer thicknesses

In this section, the effects of both FePt and Cr₉₀Mo₁₀ layer thicknesses on the microstructure of FePt films are introduced.

Fig. 5.14 shows the typical TEM images of the FePt films with different thicknesses. The images illustrate that the FePt films with different thicknesses grown on the Cr₉₀Mo₁₀ underlayers with various thicknesses also show a continuous microstructure as discussed in section 5.4.3. The insets are the SAED patterns. In all cases, a (110) reflection appears, confirming the occurrence of the chemical ordering and the presence of (001) oriented grains in all the films.

In order to further study the microstructures of both the Cr₉₀Mo₁₀ underlayers and the FePt films, TEM cross-sectional view is performed. Figs 5.15 (a) and (b) are the TEM cross-sectional images of the 15 and 60 nm thick FePt films grown on the Cr₉₀Mo₁₀ (25 nm)/Pt (4 nm) underlayers, respectively. The images reveal that both the Cr₉₀Mo₁₀ underlayers and the FePt layers have a continuous microstructure, which is consistent with the above analysis.

Figure 5-14 TEM images of (a) 10 nm and (b) 80 nm thick FePt films deposited on 30 nm thick Cr₉₀Mo₁₀ underlayers at 350°C; and 20 nm thick FePt films deposited on (c) 5 nm and (d) 20 nm thick Cr₉₀Mo₁₀ underlayers at 350°C. The marked rings are FePt (110).

Figure 5-15 TEM cross-sectional images of (a) 15 nm and (b) 60 nm thick FePt films deposited on 25 nm thick Cr₉₀Mo₁₀ underlayers at 350°C.

5.3.6 Influence of different underlayers

In this series of experiments, the deposition temperature for all the films is fixed at 350°C and the thicknesses of the FePt, Pt and Cr₉₀X₁₀ (X= Mo, Ru, Ti, W) layers are fixed at 20 nm, 4 nm, and 30 nm, respectively.

Fig 5.16 shows the XRD spectra of the FePt films grown on the different underlayers. The inserted spectra are the rocking curves of the FePt (001) peaks. As can be seen from the graphs, the FePt films grown on the Cr₉₀Ru₁₀, Cr₉₀Mo₁₀ and Cr₉₀W₁₀ (200) underlayers show a (001) preferred orientation due to the epitaxial growth, while the FePt film grown on the Cr₉₀Ti₁₀ underlayer shows a (111) preferred orientation. The inserted rocking curves of the FePt (001) peaks reveal that the FWHM of the FePt film grown on

the Cr₉₀Ru₁₀ underlayer is the narrowest among all the samples, depicting a good FePt (001) texture.

Figure 5-16 XRD spectra of 20 nm thick FePt films grown on 20 nm thick CrX underlayers.

According to the proposed epitaxial relationship between Cr (200) and FePt (001) films, the FePt films grown on the CrX (200) underlayers should have a (001) preferred orientation, and the texture quality of the FePt films mainly depends on the texture

quality of the underlayers. However, the FePt film grown on the Cr₉₀Ti₁₀ (200) underlayer shows a (111) preferred orientation. To understand the mechanism, the atomic radii of different additives, lattice constants of different underlayers and FePt films, FWHM of Cr (200) peak, and lattice mismatches between underlayer and FePt layers are analyzed and summarized in Table 6.1. The lattice parameter of the Cr intermediate layers increases with the increase of atomic radius of X, which appears to follow the Vegard's law. The lattice mismatch between FePt (001) and CrX (200) increase with the increase of lattice parameters of CrX. The lattice constant *c* of FePt films increase with increased lattice mismatch, indicating a decreased ordering degree. This result is consistent with our previous findings (Chapter 5): The critical lattice mismatch which is the most favorable for chemical ordering is about 6.33%. With a further increase in lattice mismatch, the chemical ordering degree decreases. In Chapter 5, we also found that the residual stress decreased with increased lattice mismatch from 6% to 8%. Thus, a possible reason for the FePt film grown on the Cr₉₀Ti₁₀ (200) underlayer showing a (111) preferred orientation is due to the released residual epitaxial stress caused by a larger lattice mismatch.

Underlayer (CrX)	Atomic Radius of X (Å)	CrX $\sqrt{2} a$ (Å)	Cr (200) FWHM (°)	FePt <i>c</i> (Å)	Mismatch * (%)
CrRu	1.34	4.09	0.7121	3.73	6.5
CrMo	1.39	4.12	0.7549	3.76	7.1
CrW	1.41	4.14	0.8917	3.78	7.6
CrTi	1.47	4.15	1.3769		7.9

Table 5-1 Structural properties of both underlayers and FePt films.

* Refers to the lattice mismatch between FePt (001)[100]||CrX (200)[110].

Both the in-plane and the out-of-plane hysteresis loops are shown in Fig 5.17, and the magnetic parameters are summarized in Table 5.2. The FePt films grown on the $\text{Cr}_{90}\text{Ru}_{10}$, $\text{Cr}_{90}\text{Mo}_{10}$ and $\text{Cr}_{90}\text{W}_{10}$ underlayers show a perpendicular magnetic anisotropy, while the FePt films grown on the $\text{Cr}_{90}\text{Ti}_{10}$ underlayer shows a longitudinal anisotropy. The easy axis variation is consistent with that reflected by the XRD spectra. As seen from Table 5.2, the FePt film grown on the $\text{Cr}_{90}\text{Ru}_{10}$ underlayer shows higher perpendicular coercivity, magnetic anisotropy and squareness, which appears to be the most suitable for the perpendicular magnetic recording media among the samples used in this study. The improved magnetic properties are mainly due to the improved texture quality when the $\text{Cr}_{90}\text{Ru}_{10}$ underlayer is used.

Figure 5-17 Hysteresis loops of FePt films grown on (a) $\text{Cr}_{90}\text{Ru}_{10}$, (b) $\text{Cr}_{90}\text{Mo}_{10}$, (c) $\text{Cr}_{90}\text{W}_{10}$, and (d) $\text{Cr}_{90}\text{Ti}_{10}$ underlayers.

	$H_{c\perp}$ (Oe)	K_u (10^7 erg/cc)	$S_{\perp}$ (Mr/Ms)
CrRu	3160	1.50	0.96
CrMo	2950	1.2	0.95
CrW	2960	1.0	0.93
CrTi	1881		0.33

Table 5-2 H_c ($\perp$), K_u and S ($\perp$) of FePt films grown on different underlayers.

5.3.7 Initial layer in FePt films grown on Cr₉₀Ru₁₀ (200) underlayers

It was reported that an initial layer in a recording layer should be as thin as possible, since it could deteriorate some magnetic properties.¹²³ In Chapter 4, HRTEM cross-sectional images [Fig 4.9 and Fig 4.10] revealed that the inter-diffusion between Pt and FePt layers was much weaker than that between FePt and CrMo. Thus, a Pt buffer layer between FePt and CrRu may suppress the inter-diffusion between the two layers and the initial layer thickness can be reduced. In this section, the initial layers of the FePt films with and without Pt buffer layers are studied. The FePt films with thickness, t_{FePt} , varied from 1 to 15 nm are sputtered on the 15 nm thick Cr₉₀Ru₁₀ underlayers with and without a 4 nm thick Pt buffer layer at 350°C.

Figs 5.18 (a) and (b) show the XRD spectra of the FePt films of different thicknesses on the Cr₉₀Ru₁₀ underlayers without and with Pt buffer layers, respectively. As seen from Fig. 5.18 (a), no peak from the FePt film of 3 nm thick is observed except the Cr (200) peak from underlayer without a Pt buffer layer. This may be due to the following two reasons. The first one reason is that the texture is very poor due to the diffusion at the FePt/Cr₉₀Ru₁₀ interface, and thus the diffracted signals from the FePt thin film are too

weak to be detected by the XRD detector; The second possible reason is that the texture of the thin FePt film is amorphous. As t increases to 5 nm, weak FePt (001) and FePt (002) peaks appear. The intensities of the FePt (001) and FePt (002) peaks increase with the increase of the FePt film thickness. The long-range chemical ordering degree estimated from the intensity of the superlattice peaks versus the fundamental peak increases with increasing FePt film thickness. This suggests that the initial layer has a very low chemical ordering degree. With a Pt buffer layer, at $t = 3\text{nm}$, a peak near 45° is observed, which is believed to be an overlap between Pt (200) peak and weak FePt (200) according to the 2θ location [Appendix A] and TEM electron diffraction pattern [Fig.5.19 (b)]. At $t = 5\text{ nm}$, an obvious FePt (001) peak is observed, and the intensity of the FePt (001) peak is much stronger than that without a Pt buffer layer, indicating a better texture quality.

In order to further study the microstructure of very thin FePt films ($t \leq 3\text{ nm}$), TEM electron diffraction patterns are acquired. Fig. 5.19 shows a selected area electron diffraction pattern (SAEDP) of the 3nm FePt film grown on the $\text{Cr}_{90}\text{Ru}_{10}$ underlayer without a Pt buffer layer and an SAEDP of the pure $\text{Cr}_{90}\text{Ru}_{10}$ underlayer as a reference. Except the reflection rings from the $\text{Cr}_{90}\text{Ru}_{10}$ underlayer, weak FePt (200) and FePt (220) reflection rings appear [Fig .5.19 (b)], indicating that the initial FePt film has a poor (200) texture, but not an amorphous structure.

Figure 5-18 XRD spectra of FePt films with different thicknesses on the Cr₉₀Ru₁₀ underlayers: (a) without Pt buffer layers and (b) with Pt buffer layers.

Figure 5-19 TEM electron diffraction patterns of Cr₉₀Ru₁₀ underlayer and 3 nm thick FePt film grown on the Cr₉₀Ru₁₀ underlayer.

Fig. 5.20 and Fig. 5.21 show the hysteresis loops of the FePt films with different thicknesses grown on the $\text{Cr}_{90}\text{Ru}_{10}$ underlayers without and with Pt buffer layers, respectively. The out-of-plane H_c of the FePt films as a function of the film thickness is shown in Fig. 5.22. Without a Pt buffer layer, as $t \leq 5$ nm, the FePt films show soft magnetic properties ($H_c < 500$ Oe) and random easy axis distribution as revealed by the similar in-plane and out-of-plane loops. With a Pt buffer layer, all the FePt films show perpendicular anisotropy. The out-of-plane H_c and M_s increase with increased FePt film thickness for both cases. These results depict that the initial layer has soft magnetic properties, low M_s and random easy axis distribution.

Figure 5-20 Hysteresis loops of FePt films of different thicknesses grown on $\text{Cr}_{90}\text{Ru}_{10}$ underlayers without a Pt buffer layer.

Figure 5-21 Hysteresis loops of FePt films of different thicknesses grown on Cr₉₀Ru₁₀ underlayers with a Pt buffer layer.

Figure 5-22 Out-of-plane H_c as a function of FePt film thickness.

In a previous study, a mode based on anisotropy has been developed to estimate the initial layer thickness of FePt films.¹²⁴ However, a Ku based model takes into account the contributions of both structure defects and diffusion, since Ku is related to the structure of materials. In addition, it is very difficult to estimate the demagnetization factor of films. Thus, this model is not suitable for the films with many structural defects. In this chapter, a method based on M_s is proposed to estimate the initial layer thickness, since M_s is mainly dominated by composition. With this model, an FePt film is divided into slices and each slice is assumed to have a uniform M_s when the thickness of the slice is small enough. M_s of each slice is calculated according to the formula:

$$M_s \times t_{total} = \sum_i M_s(i) \times t(i) \quad (5-4)$$

where M_s is the average M_s of the whole film, and $M_s(i)$ and $t(i)$ are the M_s and thickness of the i th slice, respectively. The depth dependence of M_s for the FePt films is shown in Fig. 5.22, where M_s first increases sharply with increased height from the FePt/CrRu and FePt/Pt interfaces and then keeps constant at about 930 emu/cc with further increased height. From the M_s distribution, the estimated initial layer thickness is about 2.5 nm and 7.5 nm for the FePt films with and without Pt buffer layers, respectively [Fig. 5.23].

The above results depict that the initial layer thickness can be reduced from about 7.5 nm to 2.5 nm with a 4 nm thick Pt buffer layer. In addition, the texture quality and magnetic properties are improved with Pt buffer layer.

Figure 5-23 Depth dependence of M_s distribution.

5.4 Conclusion

FePt films were sputtered on CrX underlayers at substrate temperatures near 350°C. The effects of the Mo content in the $\text{Cr}_{1-x}\text{Mo}_x$ underlayers, underlayer thickness, substrate temperature, FePt film thickness, and different underlayers on the structural and magnetic properties of the FePt films were investigated. The initial layers of the FePt films were also studied. The main conclusions are drawn as follows:

- (1) The crystallographic orientation of the FePt films showed strong dependence on the Mo content in the $\text{Cr}_x\text{Mo}_{1-x}$ underlayers. The FePt films with Cr and $\text{Cr}_{90}\text{Mo}_{10}$ underlayers had a good fct FePt (001) preferred orientation, while for

the FePt films grown on the $\text{Cr}_{80}\text{Mo}_{20}$ underlayers, FePt (001) and Cr (200) peaks disappeared and very weak FePt (112) superlattice peak, (111) peak and Cr (110) peak were found. The results indicated that the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers were suitable for growing the FePt films with (001) preferred orientation.

- (2) For the FePt films directly grown on the glass substrates, only an FePt (111) peak was observed, which was an fcc FePt (111) confirmed by their soft magnetic properties. Upon introduction of a 5 nm $\text{Cr}_{90}\text{Mo}_{10}$ underlayer, an fct FePt (001) peak appeared. The ordering degree increased with increased $\text{Cr}_{90}\text{Mo}_{10}$ underlayer thickness from 5 nm to 20 nm.
- (3) With the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers, the ordering process of the FePt films could start at as low as 200°C. Both the ordering degree and the out-of-plane coercivity (H_c) of the FePt films increased with an increase in substrate temperature. As the substrate temperature was $\geq 250^\circ\text{C}$, the FePt films grown on the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers could have the (001) preferred orientation. The FePt films grown on the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers at different temperatures showed a continuous microstructure. Large uniaxial anisotropy energy ($K_u \geq 1 \times 10^7$ erg/cc), high ordering degree, good magnetic squareness (~ 1) and large negative nucleation field (-2500 Oe) were obtained at $T_s \geq 300^\circ\text{C}$ when using the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers with thickness above 10 nm.
- (4) The out-of-plane coercivity (H_c) decreased while the ordering degree increased with increased FePt film thickness. The discrepancy of the coercivity with regard to the ordering degree could be due to the variation of the magnetic reversal mechanism.

- (5) The FePt films grown on the $\text{Cr}_{90}\text{Mo}_{10}$ underlayers with different thicknesses also showed a continuous microstructure.
- (6) The experimental results showed that the (200) textured $\text{Cr}_{90}\text{Mo}_{10}$ underlayers were promising for growing the FePt (001) films at relatively low temperatures.
- (7) The FePt films grown on the $\text{Cr}_{90}\text{Ru}_{10}$ underlayers showed better (001) texture quality and magnetic properties than that grown on $\text{Cr}_{90}\text{Mo}_{10}$, $\text{Cr}_{90}\text{W}_{10}$ and $\text{Cr}_{90}\text{Ti}_{10}$ underlayers. The chemical ordering degree indicated by the lattice constant c of the FePt film decreased with increased lattice mismatch from 6.5% to 7.9%, which was consistent with the results in Chapter 4.
- (8) The initial layer in FePt films was studied. A model based on M_s was suggested to estimate the initial layer thickness in FePt films. With a 4 nm Pt buffer layer, the initial layer thickness could be reduced from about 7.5 nm to 2.5 nm due to suppressed inter diffusion.

Chapter 6 FePt:C (001) nanocomposite films with ultrafine and uniform grains

6.1 Introduction

As introduced in Chapter 5, FePt (001) films grown on CrX (200) underlayers showed a continuous microstructure. As far as the recording media requirement is concerned, a continuous microstructure is not suitable for magnetic recording due to a strong intergranular exchange coupling between magnetic grains, which will result in a high transition noise. Thus, to improve the recording performance of the FePt thin films, it is essential to decrease the grain size and exchange coupling. As reviewed in Chapter 1 (Section 1.2), the decreases of grain size and exchange coupling were usually achieved by adding a third material into the films. The target of this project is to develop the FePt thin films as perpendicular recording media, so a basic requirement for choosing a third element or alloy as an additive is to keep a (001) preferred orientation in the FePt films after doping.

Previous studies revealed that most of the carbon could remain as a pure element matrix rather than forming any carbide phases even after annealing FePt/C multi-layers at 800°C for 1 hour.²⁷ Thus, it is expected that doping carbon into the FePt films during deposition at low temperatures can suppress the FePt grain growth and at the same time the non-magnetic C matrix can reduce the exchange coupling between the FePt grains. It is worth noting that carbon can be sputtered using DC power, which is preferred by industrial

applications. In 2003, Ko et al. reported that the $L1_0$ ordering and grain growth kinetics of FePt films could be finely controlled by C doping.¹²⁵ However, the use of MgO single crystal substrates limits the practical applications of FePt:C films. In addition, the magnetic properties of FePt:C films were poor for perpendicular magnetic recording. In 2004, Yan and co-workers reported a synthesis of $L1_0$ FePt:C (001) composite films by multilayer deposition plus a subsequent thermal annealing,¹²⁶ but a high temperature (550°C) long time post annealing was not compatible with the current manufacturing processes for hard disks. In Chapter 5, CrX (200) underlayers were developed to induce an ordered FePt (001) texture on glass substrates at temperatures below 350°C . However, pure FePt films grown on CrX underlayers show a continuous microstructure [Chapter 5].¹²⁷

This chapter studies the effects of C additive and substrate temperature on the microstructure and magnetic properties of the FePt films. The main objective of this work is to control the FePt (001) grain size at relatively low substrate temperatures. A direct synthesis of the ordered FePt:C (001) nanocomposite films with ultra small and uniform grain size, adjustable out-of-plane coercivity and narrow c -axis distribution on glass substrates by magnetron sputtering at 350°C , is introduced.

6.2 Sample structure

Figure 6.1 shows the samples and media structures used in this chapter. The atomic ratio of the fabricated FePt:C films was measured by XPS with the results shown in Table 6.1. The experimental details can be found in chapter 3.

Figure 6-1 Structures of FePt:C films (a) and media (b).

C content (vol.%)	Atomic ratio Fe: Pt: C (by XPS)
5	40: 48: 12
10	38: 40: 22
15	30: 33: 37
20	26: 30: 44
25	21: 26: 53
30	20: 23: 57

Table 6-1 Compositions of FePt:C films.

6.3 Influence of C additive on FePt films

6.3.1 Structural properties of FePt:C films

In this section, the thicknesses of the $\text{Cr}_{90}\text{Ru}_{10}$ underlayers, Pt buffer layers and FePt:C films are 30 nm, 2 nm and 10 nm, respectively. The deposition temperature for all the layers is 350°C. The C content is varied from 0 to about 30 vol.%. The microstructural properties of the FePt:C films are discussed.

Fig. 6.2 shows the XRD spectra of the FePt:C films with various C volume fractions deposited at 350 °C. From the XRD spectra, one can see that all the peaks are attributed to the $\text{Cr}_{90}\text{Ru}_{10}$ underlayers and FePt films, while no peaks are from carbide phases, which indicates that most C remains as a pure element matrix. With increasing C fraction from 0 to 30 vol.%, the FePt (001) peak locations remain almost unchanged, illustrating that the lattice constant c has no change. The FePt (002)+(200) peaks of the samples doped with 5, 10 and 15 vol.% C locate at a slightly higher angle than that of the pure FePt film, indicating an increase of the FePt (002) content. With further increase of the C content from 15 to 30 vol.%, the FePt (002)+(200) peak shifts to a lower angle, indicating an increase of the fcc FePt (200) content.

Figure 6-2 XRD spectra of FePt films with different C volume fractions at 350°C.

The XRD results depict that the FePt films can keep a (001) preferred orientation after incorporating C up to 20 vol.%, which is different from those doped with Ag²⁴ or Cu²⁵. In previous reports, the easy axis of FePt films grown on CrRu or CrMo underlayers changed from perpendicular to longitudinal by Ag²⁴ and Cu²⁵ doping, which the suppressed mobility of Fe and Pt adatoms by Cu and Ag doping was believed to be a main reason. The atomic radii of Cu and Ag atoms are comparable with those of Fe and Pt atoms, and thus some Fe and Pt atoms have to overcome the energy barriers caused by the Cu and Ag atoms before reaching their minimum energy sites. While for the C additive, the atomic radius of carbon atom (0.077 nm) is much smaller than those of Pt (0.138 nm) and Fe (0.126 nm) atoms. Thus, it is very likely that the C atoms can diffuse

through interstitial sites as in the case of CoCr based alloys and the mobilities of the Fe and Pt adatoms are not restricted so much. Thus, most of the Fe and Pt adatoms can move to their minimum energy sites and align with the atomic arrangement of the underlayer.

Fig. 6.3 shows the texture quality of the FePt:C films characterized by the FePt (001) peak rocking curves. The FWHM of the curves increases slightly from about 7.2° to 9.2° with increased C content from 0 to 30 vol.%, which implies that a relatively good (001) texture can be maintained even after incorporating C up to 30 vol. %.

Figure 6-3 Rocking curves of FePt (001) peaks with different C contents.

The chemical ordering degree, $(I_{001}/I_{002})^{1/2}$ (I_{001} and I_{002} are the integrated intensities of FePt (001) and (002) peaks, respectively), as a function of C content is shown in Fig. 6.4. $(I_{001}/I_{002})^{1/2}$ first increases with increasing C content from 0 to 15 vol.%, and then

decreases with further increasing C volume fraction. This signifies that a small amount of C doping can slightly enhance the ordering degree, while a large amount of C doping can decrease the ordering degree. These results are similar to those reported previously.¹²⁵ The variation of the ordering degree of the FePt films with increased C content may be explained as follows. When the C content is small, C atoms may diffuse through FePt interstitial sites during growth; the diffusion of C atoms may provide a driving force for FePt phase transformation from fcc to fct. As the C content is large, the epitaxial growth of the FePt on Pt buffer layer may be suppressed, and the FePt/Pt interface may be destroyed by C adatoms, and the lattice mismatch induced epitaxial stress may release, thus the FePt films have a lower ordering degree. The diffusion of the C during deposition has been verified by TEM images and will be discussed in the following section.

The microstructural properties of the FePt:C films are investigated by transmission electron microscopy (TEM). Figs. 6.5 (a) and (b) show the plane-view TEM images of the FePt:C films with 10 and 20 vol.% C, respectively. As introduced in Chapter 5, pure FePt films grown on Cr₉₀Ru₁₀ underlayers show a continuous microstructure. When 10 vol.% C is doped, a granular structured film is formed though some grains aggregate together and not all grain boundaries can be distinguished, and the grain size roughly follows a Gaussian distribution with a mean diameter of about 9.9 nm and a standard deviation of 2.3 nm. As the C content increases to 20 vol.%, the TEM image shows well-defined grain boundaries and relatively uniform grain sizes. The mean grain size and standard deviation are about 5.6 nm and 1.6 nm, respectively. In the both cases, the grain

size distribution of the FePt:C films is relatively narrow and encouraging with reference to the results reported previously.¹²⁵

Figure 6-4 Long-range chemical ordering degree as a function of C content.

Figure 6-5 TEM plane-view images of FePt:C films with (a) 10 vol.% and (b) 20 vol.% C doping. (a)* and (b)* are the corresponding grain size distributions of (a) and (b), respectively.

Figures. 6.6-8 show both the low magnification and the high-resolution cross-sectional images of the FePt:C media doped with 10, 20 and 30 vol.% C, respectively. Fig. 6.6 shows the TEM images of a 15 nm thick FePt:C films instead of the 11 nm thick one. All the images in Figs. 6.6-8 reveal that the films have three layers, according to different contrast. The top FePt:C layers show a granular microstructure with clear grain boundaries, confirming that the FePt:C films have well-defined grain boundaries and uniform grains, while the $\text{Cr}_{90}\text{Ru}_{10}$ and Pt layers show a continuous microstructure. In all the images, the middle continuous layers are about 7 nm thick which is much thicker than

2 nm from the design, while the top FePt:C layer are thinner than the designed thickness. It has been reported that the Cr diffusion could be effectively blocked by a thin Pt buffer layer.^{128, 129} Thus, the middle continuous layers consist of a 2 nm thick Pt layer and a 5 nm thick FePt continuous layer. This may be explained as follows. It is well known that an epitaxial stress is very strong when a film is very thin. Thus, at the initial growth stage of the FePt:C film, a strong epitaxial stress governs the thin film growth mechanism: i.e. the Fe and Pt adatoms can follow the atomic arrangement of the Pt layer and then a continuous microstructure is formed, and the C adatoms may diffuse onto the surface of the FePt layer at the same time. With the increase of the FePt:C film thickness, the epitaxial stress releases. More C atoms on the FePt film surface may inhibit the mobility of the Fe and Pt adatoms and reduce the surface bonding of the FePt film, thus, some Fe and Pt adatoms cannot reach their epitaxial sites, but instead form three-dimensional islands. The ball-like shape of the FePt grains should be mainly governed by the film surface tension. That the FePt grains can have a (001) preferred orientation is mainly due to the epitaxial growth as observed in high-resolution images [Fig. 6.6 (b), Fig. 6.7 (b) and Fig. 6.8 (b)]. A proposed two-step growth mode of the FePt:C films used in this study is shown in Fig. 6.9.

There exists a light gray contrast layer (~ 2 nm) between middle continuous and top granular layers as seen in the low magnification images [Fig. 6.6 (a), Fig. 6.7 (a) and Fig. 6.8 (a)]. This layer has been generally considered “initial layer” caused by inter-diffusion between two layers¹³⁰ and/or by elastic strain due to a lattice misfit between two layers in Co-based recording media.¹³¹ In this study, the continuous lattice images from the Pt to

the FePt continuous layers and then to the FePt:C granular layers are observed [Fig. 6.6 (b), Fig. 6.7 (b), and Fig. 6.8 (b)], which indicates the epitaxial growth between Pt and FePt/FePt:C layers. In addition, some amorphous areas are also observed between Pt/FePt layer and FePt:C layers [Fig. 6.6 (b), Fig. 6.7 (b)], which should be carbon atoms. Thus, the regions with light contrast between the continuous FePt layer and overlying granular FePt grains should be mainly due to the carbon. For the FePt:C film doped with 30 vol.% C, the high resolution image does not show a clear continuous lattice from the Pt/FePt layer to the FePt:C layer, but a rather poor texture which is believed to be caused by the C additive [marked with circle in Fig. 6.8 (b)]. An interesting phenomenon is that the morphology of the FePt layers changes from column-like to ball-like with increased C content, which should be due to the surface tension and deteriorated epitaxial stress resulted from the lattice misfit at the Pt/FePt interface.

Figure 6-6 TEM cross-sectional images of the 15 nm thick FePt:C film doped with 10 vol.% C: (a) low magnification and (b) high resolution.

Figure 6-7 TEM cross-sectional images of the 10 nm thick FePt:C film doped with 20 vol.% C: (a) low magnification and (b) high resolution.

Figure 6-8 TEM cross-sectional images of about 10 nm thick FePt:C film doped with 30 vol.% C: (a) low magnification and (b) high magnification.

Figure 6-9 Proposed two-step growth model for FePt:C films. (a) Step 1: FePt is epitaxially grown on CrRu/Pt and carbon is squeezed onto the top surface of FePt film. (b) Step 2: With increased film thickness, carbon is accumulated to some extent, and the epitaxial stress is released. Fe and Pt atoms nucleate on some sites and then the nuclei grow larger.

In order to confirm the suggested growth mode, the FePt:C (15 vol.% C) films with different thicknesses from 5 nm to 30 nm were deposited on the Glass/Cr₉₀Ru₁₀ (30 nm)/Pt (2 nm) under the same condition with above samples. The thickness dependence of the microstructure is investigated by taking TEM cross sectional images as shown in Fig. 6.10. As seen from Fig. 6.10 (a), when the FePt:C film thickness is 5 nm, the cross-sectional image does not reveal a clear FePt grain boundary, and the grains are found to have formed a continuous microstructure rather than a granular one. In addition, the contrast of the top areas of the FePt:C film [marked with arrows in Figs. 6.10 (a) and (b)] is obviously lighter than other areas, suggesting the existence of a carbon rich area on the top of the FePt:C film, which has been confirmed by X-ray photoelectron spectroscopy (XPS). This confirms the above suggestion: in the initial stage of the FePt:C film growth the carbon adatoms tend to diffuse onto the surface of the FePt layer. As the thickness of

the FePt:C film increases to 15 nm, the top of the FePt:C layer shows a ball-like microstructure with a 7~8 nm thick continuous intermediate layer which consists of 2 nm Pt and about 5 nm thick continuous FePt layer. With a further increase of thickness up to 30 nm, a columnar microstructure appears, while the thickness of the middle continuous layer with dark contrast does not change, with the growth of FePt grains only in the film normal direction. The evolution of the microstructure with increased film thickness should obey the system energy minimum principle. In our case, the thickness dependence of the microstructure should be mainly attributed to the competition between the epitaxial stress, surface tension and surface energy.

Figure 6-10 Typical TEM cross-sectional images of (a) 5 nm thick, low magnification, (b) 5 nm thick, high resolution, (c) 15 nm thick, and (d) 30 nm thick FePt:C films (15 vol.% C) grown on 30 nm thick Cr₉₀Ru₁₀ underlayers with 2 nm thick Pt buffer layers deposited at 350°C.

Both XRD and TEM results suggest that most of the carbon remain as a pure element matrix rather than forming any carbide phases. In order to further confirm this suggestion, XPS was used to verify the chemical binding of Fe, Pt and C in the FePt:C films. Fig 6-11 shows the typical XPS profiles of Fe (2p), C (1s) and Pt (4f) in FePt and FePt:C (30 vol.% C) films. The XPS profiles don't reveal any binding energy shift for Fe 2p and Pt 4f, and C 1s. In addition, the binding energies of the Fe 2p and Pt 4f, and C 1s in the FePt:C films are same with those of the pure Fe, Pt and C, respectively.¹³² This may be due to the XPS resolution limitation and a very small amount of carbide (if has some).

Figure 6-11 Typical XPS profiles of (a) Fe (2p), (b) C (1s) (b), and (c) Pt (4f).

6.3.2 Magnetic properties

In this section, the magnetic properties including hysteresis loops, H_c , H_n , magnetization reversal mechanism, magnetic domain structure, switching field distribution and time dependent effect of the FePt:C films are discussed

6.3.2.1 Hysteresis loops

Figure. 6.12 shows the out-of-plane hysteresis loops of the FePt:C films doped with various C contents as measured by polar kerr. The loops illustrate that all the FePt:C films have a perpendicular magnetic anisotropy, confirming that the easy axis is perpendicular to the film plane, which is consistent with the preceding XRD analysis.

Figure 6-12 Out-of-plane hysteresis loops of FePt:C films with different C contents.

Table 6.2 summarizes the out-of-plane coercivity $H_{c\perp}$, nucleation field H_n , magnetization squareness S ($S = M_r/M_s$), and S^* ($S^* = [(1-M_r/H_c)(1/\text{Slope at } H_c)]$) of all the FePt:C films. From Table 6.2, one can see that $H_{c\perp}$ increases from about 2.5 kOe to 3.9 kOe with increasing C content from 0 to 15 vol.%, and then decreases with further increased C content, showing a similar trend to that of the ordering degree. When the C content is ≤ 20 vol.%, relatively large negative H_n and high S are obtained, both of which (large negative H_n and high S) are required for lowering the DC noise in perpendicular media. The S^* , which is usually used to reflect the exchange coupling in perpendicular magnetic recording media, does not change much with increased C content, indicating that the exchange coupling does not change much with increased C content either. For all the samples, S^* is higher than 0.85, indicating a strong exchange coupling in all the FePt:C films. These results are similar to those reported by Yan and co-workers.¹²⁵

It is well understood that inter-granular exchange coupling depends on the microstructure of the magnetic films. Normally, a continuous magnetic film always shows a strong

exchange coupling and an isolated granular magnetic film shows a weak exchange coupling. In our case, the strong exchange coupling in the FePt:C films should be due to a continuous FePt film between Pt layer and FePt:C granular layer. Because the FePt grains in the continuous layer have a strong exchange coupling, this continuous layer also strongly couples with the overlying granular FePt grains. Thus, the magnetization of granular FePt grains switches together with the continuous FePt layer when an external magnetic field is applied.

C content (vol.%)	$H_{c\perp}$ (Oe)	H_n (Oe)	S	S*
0	2510	-2350	1	0.981
5	3153	-3169	1	0.962
10	3237	-2980	1	0.964
15	3908	-2590	1	0.851
20	3659	-3439	1	0.95
25	2839	3300	0.9	0.904
30	1832	3697	0.86	0.920

Table 6-2 Out-of-plane coercivity ($H_{c\perp}$), nucleation field (H_n), magnetization squareness (S) and S* of all the FePt:C films.

6.3.2.2 Magnetization reversal mechanism

In order to study the magnetization reversal mechanism of the FePt:C films, the angular dependence of coercivity is measured by VSM. Fig. 6.13 shows the angular dependence of coercivity profiles of the FePt:C films with 0, 10, 20 and 30 vol.% C doped. The magnetization reversal of pure FePt films grown on the Cr₉₀Ru₁₀ underlayers show

typical domain-wall motion model. The angular dependence of the H_c profiles of the FePt:C films show that the magnetic reversal mechanism deviates from the domain wall motion predominant model and tends to approach to the rotation predominant model with increasing C content [Fig. 6.13]. The variation of the magnetic reversal mechanism should be due to the variation of the microstructures of the FePt:C films. As discussed above, pure FePt films have a continuous microstructure, while the FePt films doped with carbon consist of an FePt:C granular layer and a thin continuous FePt layer. Thus, pure FePt films show a domain wall motion dominant magnetic reversal mechanism while the FePt:C films show a combined domain wall motion and rotation mode.

Figure 6-13 Angular dependence of the coercivity profiles of the FePt:C films doped with 10, 20 and 30 vol.% C. (0° refers to the film normal direction)

Fig. 6.14 shows typical MFM images of the FePt:C media doped with 5, 10 and 20 vol.% C. As can be seen from the figure, the domain size decreases with increased C content, which should be due to increased pinning sites such as grain boundaries, interface defects etc, caused by carbon additive.

Figure 6-14 MFM images of FePt:C media doped with 5, 10 and 20 vol.% C, respectively. [Scale: $3\ \mu\text{m} \times 3\ \mu\text{m}$]

6.3.2.3 Switching field distribution

The width of the switching field distribution (SFD) is known to influence the recording behavior of media,^{133,134} and a narrow SFD is required to achieve a sharp transition at a high recording density. The ideal situation would be a perfect square loop with all particles switching at the coercivity. In particulate media this is never even approximately the case. In thin film media very square loops are often obtained but these are produced due to a co-operative switching of grains due to strong magnetic interactions, which can lead to transition broadening and hence noise.¹³⁵ Generally, an SFD can be determined simply from the differential of a hysteresis loop in the second and third quadrants.

However, a hysteresis loop contains components of magnetization derived from both reversible and irreversible changes. Hence, the DCD curves are measured to examine the switching field distribution in this study, and the SFD is roughly estimated from the DCD curves, where the reversible components of the magnetization do not contribute. As well understood that in perpendicular recording media, it is very difficult to measure the real SFD value due to the demagnetization effect. Thus, in this research, the effect of the carbon additive on the SFD of the FePt:C films is only roughly studied without taking demagnetization effect into account. The DCD curves for the FePt:C media are shown in Fig. 6.15 together with their differential forms which give a measure of the SFD and provide a definition of the remanence coercivity H_{cr} . Normally, the SFD is defined as a ratio of $\Delta H_c/H_{cr}$, where ΔH_c is the full width at half maximum of differential DCD curve which is generally used to characterize the irreversible susceptibility χ_{irr} . In this research, the H_{cr} has a big variation with increased C content, thus we define ΔH_c as SFD.

Fig. 6.16 shows the SFD with respect to C content. In all the FePt:C films, a relatively narrow range of SFD values is obtained (from 200 to 577 Oe). As discussed above, the FePt:C films used in this research show a strong exchange coupling, thus the narrow SFD should be due to a co-operative switching of grains caused by strong magnetic interactions.

Figure 6-15 DCD curves and corresponding differential curves of the FePt:C media doped with 0, 5, 10 and 20 vol.% C.

Figure 6-16 SFD calculated from Fig. 6.14 with respect to C content.

6.3.2.4 Read-Write test on FePt:C perpendicular media

In this section, a Guzik spin-stand (1701B) with a commercial ring head (for longitudinal media) was used to roughly characterize the recording properties of the fabricated FePt:C media. Only selected interesting media are measured.

The reproduced waveforms of the individual tracks of the FePt:C perpendicular media doped with 20 vol.% C at various densities are shown in Fig. 6.17. The square waveforms are observed at a linear density of 59 kfc/i, indicating that it is a perpendicular medium, though the peaks are not smooth and there exist some small fluctuations. It is well understood that the demagnetization field is quite large at a low linear density for a perpendicular medium, and a large demagnetization field will result in the reverse of magnetic domains with a small magnetic anisotropy. Thus, the fluctuations at the peaks should be mainly due to the demagnetization field and the inter-granular exchange coupling. As the linear density increases to 118 kfc/i, the read-back signals can still be distinguished. However, as the linear density is further increased to 221 kfc/i, some signals overlap in addition to the amplitude fluctuations, which should also be mainly due to the demagnetization field and exchange coupling from the media point of view, in addition to the effects from the head, head-media spacing (HMS), etc.

The SNR with respect to the linear density for the pure FePt media and the FePt:C media doped with 20 vol.% C are shown in Fig. 6.18. As seen from the graph, the SNR ratios of the FePt:C media doped with 20 vol.% C are about 10 dB better than that of the pure FePt media. It is worth noting that the SNR measurement is not absolute, but only relative.

This is because the signal and noise are determined by the type of the head (e.g. sensitivity, writing capability) used, head-media spacing, etc. As far as the recording media are concerned, for given track width, smaller de-coupled grains will produce a higher SNR. In this study, the decreased grain size in the FePt:C media should be one of the main reasons for the improved SNR.

Figure 6-17 Reproduced waveforms of individual track at various densities for the FePt:C media doped with 20 vol.% C.

Figure 6-18 SNR with respect to linear density for pure FePt media and FePt:C media doped with 20 vol.% C.

6.4 Influence of substrate temperature

Substrate temperature T_s is always considered one of the most important parameters in film deposition. For practice applications, the substrate temperature should be as low as possible, because a high substrate temperature may cause many side effects, such as large grains, longer time for heating and cooling, substrate distortion, and thermal stress, etc.

In order to study the influence of T_s on the microstructure and magnetic properties of the FePt:C films, a series of samples with a structure of Glass/Cr₉₀Ru₁₀ (30 nm)/Pt (2 nm)/FePt:C (15 vol.% C) (~10 nm) at different temperatures was fabricated. T_s for the growing Cr₉₀Ru₁₀ underlayers was fixed at 350°C, while for the Pt and FePt:C layers

varied from 200°C to 350°C. The influence of the substrate temperature on the structural and magnetic properties of the FePt: C films are discussed below.

6.4.1 Structural properties

The crystalline orientation of the FePt:C media deposited at different substrate temperatures is shown in Fig. 6.19. At 200°C, an FePt (200) peak is dominant. The soft magnetic properties indicate that it is an fcc FePt (200) peak [Fig. 6.20]. In addition, a weak FePt (111) peak near 40° appears, indicating a poor texture in the FePt:C film. As the substrate temperature increases to 250°C, a very weak FePt (001) peak appears, indicating a weak chemical ordering degree in the FePt:C film. In addition, the peak between 45° and 50° shifts to a higher angle, due to the appearance of a small amount of fct FePt (002) phase. The hysteresis loops and coercivity values illustrate that it is still an fcc FePt (200) predominant peak [Fig. 6.19]. With a further increase in substrate temperature, the intensity of the FePt (001) peak becomes stronger. In addition, the peak between 45° and 50° shifts to a higher angle again, depicting an improved fct FePt (002) phase [Chapter 5]. The TEM cross-sectional images of the FePt:C films reveal that the grain size does not rise with increased substrate temperature (from 200 to 350°C) [Fig. 6.22], suggesting that the disorder-order transformation in these FePt:C films is a continuous type.

Figure 6-19 XRD spectra of FePt:C (15 vol.% C) films grown on Cr₉₀Ru₁₀ underlayers at different substrate temperatures.

6.4.2 Magnetic properties

The magnetic properties of the FePt:C (15 vol.% C) films deposited at different substrate temperatures were measured by VSM. Fig. 6.20 shows both the in-plane and the out-of-plane hysteresis loops of the FePt:C (15 vol.% C) films grown on the Cr₉₀Ru₁₀ underlayers with different substrate temperatures. At 200°C and 250°C, the FePt:C films show the soft magnetic properties with a coercivity smaller than 300 Oe. At 300°C and

350°C, the films show a perpendicular anisotropy. The out-of-plane coercivity increases monotonously with increased substrate temperature [Fig. 6.21], which should be mainly due to the increased ordering degree. For the FePt:C (15 vol.% C) film deposited at 350°C, there exists a “shoulder” in the second quadrant of the hysteresis loop [Fig. 6. 20], indicating that there exists some magnetic soft phase in the film. A thing worth noting is that the out-of-plane coercivity of the FePt:C (15 vol.% C) film measured by VSM (3.5 kOe) is smaller than that measured by polar kerr (3.9 kOe), which should be due to a slower field sweep rate during the VSM measurement, in addition to a different system sensitivity.

Figure 6-20 Hysteresis loops of FePt:C (15 vol.% C) films deposited at different substrate temperatures.

Figure 6-21 Coercivity with respect to substrate temperature for FePt:C films with 15 vol.% C.

6.4.3 Microstructural properties

The microstructural properties of the FePt:C (15 vol.% C) films deposited at different substrate temperatures are investigated by TEM as shown in Fig. 6.22. The TEM cross-sectional images of the FePt:C films reveal that the average grain size of the FePt:C films is about 6 nm with increased substrate temperature from 200°C to 350°C. The high resolution images show a clear continuous lattice from the Cr₉₀Ru₁₀ underlayers to the Pt buffer layers and further to the FePt:C layers, depicting that the Pt buffer layers first epitaxially grew on the Cr₉₀Ru₁₀ underlayers and then the FePt:C layers epitaxially grew on the Pt buffer layers during film deposition [Fig. 6.22 (b), (f)]. It is worth highlighting that the epitaxial growth has indeed occurred at 200°C [Fig. 6.22].

Figure 6-22 TEM cross-sectional images of FePt:C (15 vol.% C) films deposited at (a) 200°C, low magnification, (b) 200°C, high resolution, (c) 250°C, (d) 300°C, and (e) 350°C, low magnification, and (f) 350°C, high resolution.

6.5 Conclusion

FePt:C (001) nanocomposite films with ultrafine and uniform grains were prepared on glass substrates by co-sputtering at 350°C. The influences of C content and substrate

temperature on the FePt:C media were studied. The following are the main conclusions drawn:

- (1) The (001) oriented FePt:C films with perpendicular magnetic anisotropy and relatively narrow orientation distribution (rocking curve of FePt (001) $< 10^\circ$) were obtained with C doping.
- (2) A small amount of C doping could slightly enhance the ordering degree, while a large amount of C doping could prohibit the ordering degree.
- (3) The FePt:C films had a well controlled grain size (from about 5 to 10 nm) with a narrow grain size distribution.
- (4) A strong exchange coupling in the FePt:C media fabricated was believed to be due to the existence of a thin continuous FePt layer caused by the carbon diffusion during the initial stage of the FePt:C film growth. A two-step growth model for the FePt:C films was proposed.
- (5) The SNR ratio of the FePt:C media doped with 20 vol.% C was about 10 dB better than that of the pure FePt media.
- (6) The epitaxial growth of the FePt:C films on the Pt layers was observed from high resolution TEM cross sectional images even for the films grown at about 200°C. The TEM images did not show an obvious change in the morphology of the FePt:C films deposited at different temperatures (from 200°C to 350°C), though the ordering degree and coercivity of the films increased with increased substrate temperature.

Chapter 7 Summary and conclusions

7.1 Summary

This thesis is devoted to development of high anisotropy $L1_0$ FePt magnetic thin films for perpendicular magnetic recording media. The main challenges of the FePt thin films as recording media are: how to lower the $L1_0$ FePt ordering temperature with controlled crystallographic texture and, how to decrease the grain size and exchange coupling between grains. Thus, this research has been always focused around the main challenges. The stress-induced chemical ordering mechanism and nano-layer engineering were proposed to lower the $L1_0$ FePt ordering temperature and to control the crystallographic texture. By doping a third element and developed process, the grain size in FePt films has been well controlled, and the (001) preferred orientation could be maintained. In Chapter 4, the influence of lattice mismatch on intrinsic properties of FePt films has been studied on MgO (200) single crystal substrates. Next, in Chapter 5, the stress-induced chemical ordering mechanism demonstrated in Chapter 4 was applied to the development of the FePt (001) films on the glass substrates. Based on the results of Chapter 4, CrX (X=Mo, Ru, Ti, W) (200) underlayers have been developed on glass substrates to induce the (001) preferred orientation in FePt films. However, the FePt (001) films developed on CrX underlayers showed a continuous microstructure. To decrease the grain size in FePt (001) films, the effects of C additive on microstructure and magnetic properties of FePt films has been investigated in Chapter 6.

7.2 Conclusions

The main findings out of this research are summarized as follows:

- 1) With different buffer layers, the lattice mismatch between FePt and buffer layer could be adjusted from 2.23% to 8.66%.
- 2) The epitaxial relationships between MgO/Pt/FePt, MgO/Cr_xMo_{1-x}/FePt, and MgO/FePt were identified as MgO (100)<001> || Pt (100)<001> || FePt (001)<100>, MgO (100)<001> || Cr (100)<110> || FePt (001)<100>, and MgO (100)<001>|| FePt (001) <100>, respectively.
- 3) The c value decreased with an increase in lattice mismatch from about 2.23% to 6.33%. Upon further increase of ε , c increased in turn. On the other hand, the variation of a illustrated a controversial behavior to that of c with increased lattice mismatch. The ratio of c/a held a minimum about 0.9466, while both $(I_{001}/I_{002})^{1/2}$ and K_u held maxima at $\varepsilon \cong 6.33\%$. A critical lattice mismatch near 6.33% was believed to be the most suitable for improving the chemical ordering of the FePt films.
- 4) The residual stress decreased with increased FePt film thickness. The variation of the residual stress with the lattice mismatch was consistent with that of the chemical ordering, suggesting that the static tensile stress from the lattice mismatch favored the phase transformation of the FePt films from fcc to fct.
- 5) The crystallographic orientation of the FePt films showed strong dependence on the Mo content in the Cr_xMo_{1-x} underlayers. The results depicted that the Cr₉₀Mo₁₀ underlayers were suitable for inducing the growth of the FePt films with (001) preferred orientation.

- 6) For the FePt films grown on the glass substrates, only the FePt (111) peak was observed, which was an fcc FePt (111) confirmed by their soft magnetic properties. Upon the introduction of a 5 nm CrMo underlayer, the fct FePt (001) peak appeared. The ordering degree increased with increased Cr₉₀Mo₁₀ underlayer thickness from 5 nm to 20 nm.
- 7) With the Cr₉₀Mo₁₀ underlayers, the ordering process of the FePt films could start at as low as 200°C. Both the ordering degree and the out-of-plane coercivity (H_c) of the FePt films increased with an increase in substrate temperature. As the substrate temperature was $\geq 250^\circ\text{C}$, the FePt films grown on the Cr₉₀Mo₁₀ underlayers could have the (001) preferred orientation. The FePt films grown on the Cr₉₀Mo₁₀ underlayers at different temperatures showed a continuous microstructure. Large uniaxial anisotropy energy $K_u \geq 1 \times 10^7$ erg/cc, good magnetic squareness (~ 1) and large negative nucleation field (-2.5 kOe) were obtained at $T_s \geq 300^\circ\text{C}$ when using the Cr₉₀Mo₁₀ underlayers with the thickness above 10 nm.
- (8) The out-of-plane coercivity (H_c) decreased while the ordering degree increased with increased FePt film thickness. The discrepancy of the coercivity with regard to the ordering degree could be due to the variation of the magnetic reversal mechanism.
- (9) The FePt films grown on the Cr₉₀Ru₁₀ underlayer showed better (001) texture quality and magnetic properties than those grown on Cr₉₀Mo₁₀, Cr₉₀W₁₀ and Cr₉₀Ti₁₀. The chemical ordering degree indicated by the lattice constant c of the FePt films decreased with increased lattice mismatch from 6.5% to 7.9%.
- (10) The initial layers of the FePt films were also studied. A method based on M_s was suggested to estimate the initial layer thickness. With a 4 nm Pt buffer layer, the

initial layer thickness could be reduced from about 7.5 nm to 2.5 nm due to suppressed inter-diffusion.

- (11) FePt:C (001) nanocomposite films with ultrafine and uniform grains were prepared on the glass substrates by co-sputtering at 350°C. The (001) oriented FePt:C films with perpendicular magnetic anisotropy and relatively narrow orientation distribution (rocking curve of FePt(001) < 10°) were obtained with C doping.
- (12) Light C doping could slightly enhance the ordering degree, while heavy C doping could prohibit the ordering degree.
- (13) With 10 vol.% C doped, a granular structured FePt film was formed with a mean diameter of about 9.9 nm and a standard deviation of 2.3 nm. As the C content increased to 20 vol.%, the mean grain size and standard deviation were about 5.6 nm and 1.6 nm, respectively.
- (14) A strong exchange coupling in the FePt:C media fabricated was believed to be due to the existence of a thin continuous FePt layer caused by the carbon diffusion during the initial growth of the FePt:C films.
- (15) The SNR ratios of the FePt:C media doped with 20 vol.% C were about 10 dB better than that of the pure FePt media.
- (16) The epitaxial growth of the FePt:C films on the Pt layers was observed from the HRTEM cross-sectional images even for the films grown at about 200°C. The TEM images did not show an obvious change in the morphology of the FePt:C films deposited at different temperatures (from 200°C to 350°C), though the ordering degree and coercivity of the films increased with increased substrate temperature.

- (17) Flyable disks were fabricated based on the FePt and FePt:C perpendicular magnetic media developed in this research, suggesting that the FePt and FePt:C media had a very smooth surface. The fabrication procedure for the disks developed in this research was compatible with the current commercial disk processes.

Appendix A: Standard crystallographic XRD data

The angle θ is the Bragg angle. Values of 2θ are angles at which the peaks corresponding to the lattice planes appear. The data are calculated for Cu-K α radiation, $\lambda = 1.54056\text{\AA}$.

2 θ (°)	d-spacing (Å)	h k l	2 θ (°)	d-spacing (Å)	h k l	2 θ (°)	d-spacing (Å)	h k l
Fct FePt ¹			Fcc FePt ²			Cr ³		
23.901	3.72	001	40.320		111	44.392	2.039	110
33.026	2.71	110	46.916		200	64.581	1.4419	200
41.186	2.19	111	68.423		220	81.721	1.1774	211
47.305	1.92	200	82.521		311			
48.929	1.86	002	Pt ⁴			MgO ⁵		
53.866	1.70	201	39.763	2.265	111	39.63	2.4316	111
60.457	1.53	112	46.243	1.9616	200	42.917	2.1056	200
68.997	1.36	220	67.454	1.3873	220	62.304	1.4890	220
70.783	1.33	202						
74.677	1.27	221						
79.077	1.21	301						

Reference:

[JCPDS-97] JCPDS- International Centre for Diffraction Data.

- 1. No. 02-1167
- 2. No.04-0717
- 3. No.06-0694
- 4. No.04-0802
- 5. No.45-0946

Appendix B: Phase diagram of Fe-C alloy

Fe-Fe₃C Phase Diagram ¹³⁶

Appendix C: Phase diagram of Cr-Mo alloy

Appendix D: Phase diagram of Cr-W alloy

List of Publications

Scientific Journal papers

1. L1₀ FePt films epitaxially grown on MgO substrates with or without a Cr underlayer

Journal of Magnetism and Magnetic Materials. **303**, 238 (2006).

Y.F. Ding, J.S. Chen, and E. Liu

2. Effect of lattice mismatch on chemical ordering of epitaxial FePt films

Journal of Applied Physics. **97**, 10H303 (2005).

Y.F. Ding, J.S. Chen, E. Liu, C.J. Sun and G.M. Chow

3. Dependence of microstructure and magnetic properties of FePt films on Cr₉₀Ru₁₀.

Journal of Magnetism and Magnetic Materials. **285**, 443 (2005)

Y.F. Ding, J.S. Chen, E. Liu and J.P. Wang

4. Structural and magnetic properties of FePt films grown on Cr_{1-x}Mo_x underlayers

Applied Physics A: Materials science & processing. **81**, 1485 (2005).

Y.F. Ding, J.S. Chen and E. Liu

5. Epitaxial L1₀ FePt films on SrTiO₃ (1 0 0) by sputtering

Journal of Crystal Growth. **276**, 111 (2005)

Y.F. Ding, J.S. Chen and E. Liu

6. Structural and magnetic properties of nanostructured FePt/MgO granular films

Thin Solid Films. **474**, 141 (2005)

Y.F. Ding, J.S. Chen and E. Liu

7. Controlling the crystallographic orientation and easy axis of magnetic anisotropy in L1₀ FePt films with Cu additive

Thesis

Publications

Surface and Coatings Technology. **198**, 270 (2005).

Y.F. Ding, J.S. Chen and E. Liu

8. Modulation of preferred orientation and easy axis of magnetic anisotropy in L1₀ FePt films with Cu buffer layers

Surface and Coatings Technology. **198**, 262 (2005).

Y.F. Ding, J.S. Chen, and E. Liu

9. Microstructure and magnetic properties of rapidly annealed FePt (001) and FePt-MgO (001) films

Journal of Applied Physics, **97**, 10N108, (2005).

J.S. Chen, T.Z. Zhou, **Y.F. Ding**, B.C. Lim, and B. Liu

10. Low temperature deposition of L1₀ FePt(001) textured thin film for ultra high density magnetic recording

Journal of Magnetism and Magnetic Materials, **303**, 309 (2006).

J.S. Chen, B.C. Lim, **Y.F. Ding**, and G.M. Chow.

11. L1₀ FePt:C perpendicular media with ultra small and uniform grains

IEEE Trans. Magn. (in press)

Y.F. Ding, J.S. Chen. B.C. Lim, E. Liu and S.Y. Chow

12. Influence of Humidity on the Friction of Diamond and Diamond-like Carbon Materials.

Tribology International. 40 (2007) 216-219.

E. Liu, **Y.F. Ding**, L. Li, B. Blanpain, J.-P. Celis

13. L1₀ FePt and FePtB perpendicular magnetic recording media

Journal of Applied Physics (Submitted)

Y.F. Ding, J.S. Chen, B.C. Lim, J.F. Hu, and E. Liu

14. Initial layers in FePt films grown on CrRu underlayers.

Journal of crystal growth (Submitted)

Y.F. Ding, J.S. Chen, E. Liu

15. Understanding of strong exchange coupling in FePt:C (001) textured granular film.

Applied Physics letters (Submitted)

J.S. Chen, **Y.F. Ding**, B.C. Lim, S.Y. Chow, and E. Liu

International Conference papers

1. Controlling the crystallographic orientation and easy axis of magnetic anisotropy in L1₀ FePt films with Cu buffer layers.

Thin films 2004 & Nanotech 2004 international conference. Jul, 2004, Singapore (Presentation)

Y.F. Ding, J.S. Chen and E. Liu.

2. Controlling the crystallographic orientation and easy axis of magnetic anisotropy in L1₀ FePt films with Cu additive

Thin films 2004 & Nanotech 2004 international conference. Jul, 2004, Singapore. (Presentation)

Y.F. Ding, J.S. Chen and E. Liu.

3. Effect of lattice mismatch on chemical ordering of epitaxial FePt films

The 49th annual conference on magnetism and magnetic materials, No. CE-04, (Jacksonville, Florida, USA, 7-11, Nov, 2004). (Presentation)

Y.F. Ding, J.S. Chen, E. Liu.

Thesis

Publications

4. L₁₀ FePt films epitaxially grown on MgO substrates with or without Cr underlayers with magnetron sputtering

International symposium on physics of magnetic materials, Sep. 13-15, 2005. Singapore. (Poster)

Yinfeng Ding, Jingsheng Chen, **Erjia Liu**

5. Microstructure and magnetic properties of rapidly annealed FePt (001) and FePt-MgO (001) films

The 49th annual conference on magnetism and magnetic materials. (Jacksonville, Florida, USA, 7-11, Nov, 2004). (Poster)

T. Zhou, J. Chen, **Y.F. Ding**, B. Lim, and B. Liu.

6. Low temperature deposition of L₁₀ FePt (001) textured thin film for ultra-high density magnetic recording

Invited Talk on 2005 international symposium on physics of magnetic materials, Sep. 13-15, 2005. Singapore. (Presentation)

J.S. Chen, B.C. Lim, and **Y.F. Ding**.

7. Influence of Humidity on the Friction of Diamond and Diamond-like Carbon Materials

1st International Conference on Advanced Tribology (iCAT), Dec, 1-3, 2004, Singapore. (Presentation)

E. Liu, **Y.F. Ding**, L. Li, B. Blanpain, J.-P. Celis

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