High Throughput Nanoscale Crystal Orientation and Phase Mapping of Heat Assisted Magnetic Recording (HAMR) and Bit Patterned Magnetic Recording (BPMR) Media

1. Front matter
   a. Date: April 29, 2011
   b. Abstract - The objective of the proposed work is to further develop and use high throughput electron diffraction based nanoscale crystal orientation and phase mapping methods in the transmission electron microscope (TEM) to characterize statistically significant populations of microstructural features in heat assisted magnetic recording (HAMR) and bit patterned magnetic recording (BPMR) media. The media features that will be mapped are grain size and grain size distribution, c-axis distribution, and crystalline phase distribution in case of multiphase materials. The nanoscale mapping will be carried out in the newly (March 2011) installed NanoMEGAS ASTAR hardware/software system in the Tecnai F20 TEM at Carnegie Mellon. In the ASTAR system, a nanosized beam is scanned over the field of view and at each point in the scan a spot diffraction pattern is collected. The diffraction patterns are matched against pre-calculated templates for all possible crystal orientations of a given phase, allowing the grains, the crystal orientations and the phases to be mapped with nanoscale resolution. The maps can be acquired in as little as a few minutes (<10 min) thereby allowing high throughput data collection. The samples for the proposed studies will be supplied by other researchers including university researchers funded by ASTC or by ASTC member companies.

In HAMR media (Topic No. 2), the mapping results are expected to (i) assist the development of media structures and processes for tailoring grain size in the range of 3-6 nm, (ii) further the understanding of the underlying phenomena in grain size control, microstructure development, and correlation to magnetic properties, (iii) help improve grain size distribution and dispersion of grain-to-grain anisotropy energies, and (iv) help develop processes with improved c-axis out-of-plane crystallographic texture and aid in the study of the underlying causes for undesirable c-axis in-plane growth. In BPMR media (Topic No. 10), the mapping results are expected to (i) help elucidate the origins of the switching field distribution (SFD) and thus aid the development of BPMR material with narrow SFD, and (ii) help establish a nanoscale metrology method for correlation with magnetic behavior of the medium.

c. Proponent(s) and affiliation(s)
   Media: Dr. Olav Hellwig, HGST (olav.hellwig@hitachigst.com)
          Dr. Timothy Klemmer, Seagate (timothy.j.klemmer@seagate.com)
   HAMR:  Dr. Matt Gibbons, WD (matt.gibbons@wdc.com)
   BPMR:  Dr. Thomas Albrecht, HGST (Thomas.albrecht@hitachigst.com)

d. Designated contact person.
   Technical: Prof. Katayun Barmak
              katayun@andrew.cmu.edu
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            Data Storage Systems Center
            ECE Department
            Roberts Engineering Hall
2. **Subject of research and relevance to issue(s) to be solved.**

   a. The following Research Topics identified by the ASTC sponsor companies will be addressed by the proposed work:

   **HAMR No. 2: Fabrication and Characterization of small grain size HAMR media**

   The proposed automated nanoscale crystal orientation and phase mapping of HAMR media will provide **statistically significant grain size data** (for populations of $10^3-10^4$ grains per sample), **the local crystal orientation as well as the spatial distribution of crystal orientations** for the mapped grains, and the **spatial distribution of the A1 disordered and L1$_0$ ordered phases of FePt**. Such automated, high throughput nanoscale crystal orientation and phase mapping information obtained in the TEM will be critical to the following 4 needs listed under this Research Topic:

   - The development of media structures and processes for tailoring grain size in the range of 3-6 nm with thermal stability at process temperatures of $<600$ °C
   - Further the understanding of the underlying phenomena in grain size control, microstructure development, and correlation to magnetic properties
   - Improve grain size distribution and dispersion of grain-to-grain anisotropy energies
   - Develop processes with improved c-axis out-of-plane crystallographic texture and study the underlying causes for undesirable c-axis in-plane growth

   **BPMR No. 10: Magnetic materials and properties for BPM**

   The proposed automated nanoscale crystal orientation and phase mapping will provide **the local crystal orientation as well as the spatial distribution of crystal orientations** for the mapped bits, and the **spatial distribution of phases present in the bits, if multiphase**. This automated, high throughput nanoscale metrology in the TEM will be critical to the following 2 needs listed under this Research Topic:

   - Understanding origin and control of switching field distribution
   - Magnetometry and metrology approaches
b. Proposed research approach(es)

Over the past decade, my microscopy efforts at CMU have focused on implementing instruments and methodologies for high throughput grain and crystal orientation mapping of nanocrystalline films in the transmission electron microscope. Until March 2011 our efforts had focused on the automated crystallography in the TEM (ACT) system by TSL. In early March 2011, we installed an alternative (and far superior) electron diffraction based phase and crystal orientation mapping system in the TEM, known as ASTAR by NanoMEGAS. The ASTAR hardware/software combination makes use of a nanosized beam and a precession technique to achieve diffraction intensities that are closer to the kinematical approximation. As will be discussed below, this system allows (i) nanoscale grain mapping (readily), (ii) orientation mapping (some development of sample preparation and data collection and analysis methodologies still needed), and (iii) phase mapping/determination of long range order parameter (significant development needed).

An example of the results we have obtained in the past month is shown below for a FePt sample prepared by S. Granz (CMU doctoral student of Prof. M. Kryder), deposited from an alloy target onto a room-temperature deposited MgO. The FePt layer was deposited at a nominal thickness of 10 nm from an alloy target with a composition of 52.5:47.5 at.%:at.% Fe:Pt at a temperature of 575 °C. The grain (clusters) of FePt on the background MgO layer are clearly visible in the bright field transmission electron micrograph shown in Fig. 1. However, bright field images do not permit ready identification of grains within these islands, nor do they permit grain/crystal orientation or the phases within the islands to be easily determined. Dark field images formed at a given electron beam tilt do allow identification of some grains in the field of view, but they are still not suitable for high throughput metrology of media.

Using the recently installed ASTAR system, however, we have been able to obtain spatial mapping of grains, their orientation and crystallographic phase. As mentioned above, the ASTAR system uses a nanosized electron beam. This beam is scanned at a pre-determined step size over the field of view, and, at each point in the scan, the spot diffraction pattern is recorded. An example of the diffraction pattern collected at a point in the FePt film of Fig. 1 is shown in the left panel of Fig. 2.

During the data analysis step, each and every one of the recorded spot diffraction patterns is matched against a set of pre-calculated templates for all possible orientations of given phases (within the fundamental zone for the given crystal symmetry). As an example, Fig. 2 shows a spot diffraction pattern (left) and its match to the template (right). Depending on the required spatial resolution, which then dictates the step size and the spot size in the scan, the thousands of

Fig. 1 – Bright field transmission electron micrograph of a plan-view sample of FePt film deposited at a nominal thickness of 10 nm from an alloy target with a composition of 52.5:47.5 at%:at.% Fe:Pt at a temperature of 575 °C. The FePt was deposited onto a room temperature deposited MgO layer deposited on a Si substrate. (Image and sample courtesy of S. Granz)
diffraction patterns needed for mapping a given field of view can take as little as a few minutes (<10 min.) to collect. The final results are images and maps as shown in Figs. 3 and 4.

Fig. 3a shows the index map, which is a measure of goodness of fit to the calculated template(s), for the FePt sample of Fig. 1. Fig. 3b is a corresponding orientation map of the same field of view with the index map superimposed. In contrast to Fig. 1, Figs. 3a and 3b make the presence of clusters of grains within the FePt islands readily apparent. Even without any further analysis to determine the actual orientation, images such as Figs. 3a and 3b can be easily used to obtain the average grain size and the grain size distribution (and thus grain size dispersion, i.e., the width of the distribution) for statistically significant populations ($10^3\text{-}10^4$) of grains by rapidly mapping the requisite number of fields of view.

As was noted, Fig. 3b is a crystal orientation map, or inverse pole figure (IPF) map, in which the color represents the crystal direction parallel to the sample direction of interest, which here is the
film normal ([001] sample). The red color over most of the map indicates that the normal to (001)/(002) crystal planes is pointing out of the page, in keeping with the fact that the FePt layer was deposited on the (002) oriented under-layer of MgO. The map also reveals that not all grains have their c-axis normal to the film plane. The c-axis in-plane variants appear green in Fig. 3b. Thus, maps such as Fig. 3b can be used to determine the percentage and the origin of c-axis in-plane variants.

The spot diffraction patterns (e.g., Fig. 2) can also be used to determine the phases present at each location in the sample. As an example, Fig. 2 right panel reveals the presence of L1₀ ordered FePt at the given position. However, owing to the (001)/(002) texture of the film, distinguishing between disordered A1 and ordered L1₀ poses some difficulty as can be seen in Fig. 4, where the color red now indicates the A1 phase, and the color blue the L1₀ phase. The sample appears to be mostly A1. Given that the coercivity of the film was 15 kOe and that x-ray diffraction indicated a highly ordered L1₀ film, there is clearly a need to further refine the sample preparation procedures and the data collection and analysis methods to obtain the correct measure of L1₀ phase fraction and the long range order parameter of the L1₀FePt grains.

To emphasize, in harnessing the capabilities of the ASTAR system in the proposed work, a critically important part of the effort is the preparation of suitable TEM samples for mapping. This effort, along with the TEM characterization, constitutes a significant part of the doctoral work of Xuan Liu (the current student) and the student who is expected to join the group in the fall 2011 semester.

In summary, the proposed tasks in order of increasing difficulty A-C and expected outcomes are:

**Task A**: Grain mapping

**Task B**: Crystal orientation mapping, c-axis variant/c-axis dispersion mapping

**Task C**: Phase mapping, long range order mapping

The grain maps, the crystal orientation maps (from which magnetic easy axis orientation distribution/c-axis dispersion can be obtained with associated local, spatial information), and A1 vs. L1₀ phase/L1₀ FePt ordered domain/L1₀ FePt long range order parameter maps obtained by using the ASTAR system recently installed at CMU will provide a more detailed characterization of HAMR and BPMR media than has been done to date. Where necessary, x-ray diffraction data as well as compositional data will be obtained, the latter using energy dispersive spectrometry (EDS) or electron energy loss spectrometry (EELS). The microstructural, x-ray diffraction and compositional data obtained for statistically significant populations of grains, domains, bits, etc. will enable more detailed structure-property correlations and will aid process development for production of HAMR and BPMR recording media with 1-4 Tb/in² recording densities.
c. Likely outcome of research

For HAMR and BPMR, we expect the following methodologies and results:

- Methodologies for sample preparation and for automated grain mapping, crystal orientation mapping and phase/long range order parameter mapping of HAMR and BPMR media samples.
- Grain size for statistically significant grain populations
- Spatial distributions of crystal orientations (c-axis dispersion)
- Nanoscale phase and long range order parameter maps

The mapping in the ASTAR system is expected be the underpinnings of a viable high throughput metrology that will further our understanding of the origin of switching field distribution in BPMR, as well as our understanding of grain and microstructure development, c-axis distribution and c-axis in-plane growth in HAMR media. This understanding will be critical to process development for production of media with 1-4 Tb/in² recording densities. In other words, the ASTAR maps will allow an unprecedented level of structure-magnetic property correlation for media development. Since the ASTAR system and its mapping techniques have only recently become available at CMU, the proposed work is also timely.

Please also see section 2a for more detailed expected outcomes under HAMR Topic No. 2 and BPMR Topic No. 10.

3. Resources required to perform project

a. Personnel:

Katayun Barmak, Professor of Materials Science and Engineering

Xuan Liu, doctoral student in the Department of Materials Science and Engineering, Carnegie Mellon University

New doctoral student in the Department of Materials Science and Engineering, Carnegie Mellon University

b. Equipment:

The equipment to be used for the proposed studies will be the FEI Tecnai F20 Field Emission Transmission Electron Microscope equipped with the NanoMEGAS ASTAR (hardware/software) system for nanoscale phase and crystal orientation mapping. See Section 7 for more detail.

c. Computational

Use will be made of computational facilities in Barmak’s lab and in the Materials Characterization Facility to run the requisite data analysis and visualization software from NanoMEGAS and from TSL, both of which are available at CMU.

4. Resources other than ASTC funding dedicated to perform project

None.
5. Resources requested from ASTC and how they will be utilized

   a. Funding

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<td>i. Overhead</td>
<td><strong>Facilities and Administration:</strong> Overhead on this proposal has been calculated at our current proposed or negotiated non-federal rate for all fiscal years. The modified total direct cost base (MTDC) amount used in calculating the indirect costs is the total direct costs, excluding equipment, capital expenditures, charges for tuition remission, rental costs, scholarships and fellowships, internally charged telephone, internally charged copying, and individual subcontract costs in excess of $25,000. The current non-federal rate is 63%.</td>
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<td><strong>ii. Direct Project Cost</strong></td>
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<td><strong>Senior Personnel:</strong> A modest amount of senior faculty salary support is requested to support project management and student advisement. Since the ASTAR system and its use are very new at CMU, significant involvement of the PI, Barmak, for development of sample preparation and data collection and analysis methodologies is required. <strong>Fringe Benefits:</strong> Fringe benefits for faculty and staff are calculated at the current proposed or negotiated non-federal rate for all fiscal years. The current non-federal rate is 29%. <strong>Technical Supplies and Services:</strong> Supplies include all technical items that are considered to be expendable. Items in this category include materials and supplies for the preparation of transmission electron microscopy samples (grids, etchants, grinding and polishing supplies, ion milling supplies, ion milling holders), supplies for x-ray diffraction studies (holders, etc.), sputter coating materials for samples, data storage and printing supplies, etc. Research services include the operation of sample preparation equipment and sample characterization. In detail: transmission electron microscopy facilities in the materials science and engineering department (Philips CM12 $50/hr, JEOL 2000 $50/hr, FEI Technai F20 $300/day), Gatan PIPS ($20/hr), and the x-ray diffractometers (Rigaku $35/unit, Philips Expert $35/unit).</td>
<td>63% of $34,406 MTDC</td>
<td>$21,675</td>
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<td><strong>Senior Research Effort = $6,357</strong></td>
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<td>Fringe benefits: $6,357 x 29% = $1,844</td>
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<td>Technical Supplies: $2,000</td>
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<td>Research Services: $7,000</td>
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### iii. Facility fees
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### iv. Materials
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### v. Student Stipend/ Tuition

**Graduate Student Support:** Stipend and tuition amounts are based on FY 2012 proposed rates. Graduate support is increased effective September 1. The colleges each set their graduate support rates in consultation with their faculty, department heads, deans and the Provost taking into account an evaluation of our historical market position in comparison to our peer institutions. Salaries for graduate students are based on a nine month academic year and three summer months. We are requesting 50% support of tuition and stipend for one graduate student researcher.

- **Stipend:**
  - $2,118 x 6 mos = $12,705
- **Tuition:**
  - Partial Academic Year support (4.5 mos) = $18,901

**Stipend:** $31,606

### vi. Travel

**Domestic Travel:** Travel will include attendance by the PI and/or graduate student to attend the ASTC sponsored meetings and relevant conference(s) from among Intermag, MMM, Microscopy and Microanalysis and other magnetic or materials related conferences. Educational Institutions are not subject to the Federal Civilian Employee and Contractor Travel Expense Act of 1985 (Pub. L. 99-234) at this time. Costs incurred for travel, including lodging and other subsistence, will be considered reasonable and allowable to the extent that they do not exceed charges normally allowed by the University in its regular operations according to institutional policy. In the absence on institutional policy, the GSA rates shall apply.

- **4 domestic trips @ $1,125 per trip**

**TOTAL REQUESTED:** $74,982
b. Expected technical cooperation with sponsor(s): materials to be provided by sponsor(s) (e.g., targets, devices, engineering support, etc.)

ASTC sponsors are asked to provide experimental HAMR and BPMR media films. TEM samples will be made at CMU from the films provided by the ASTC sponsors for characterization by the ASTAR system.

c. Sponsors’ facility utilization

Magnetic characterization facilities and x-ray diffraction facilities for measurement of magnetic properties, phase identification and determination of long range order parameter of L1₀ FePt of samples from the sponsor companies that will then be provided to us for characterization using the TEM ASTAR system at CMU.

d. Expected students’ internships

Xuan Liu, and the new student will be encouraged to intern at any of the sponsor companies that are willing to host them in order to carry out x-ray diffraction studies or to characterize the magnetic properties of HAMR and BPMR media samples. Additionally, the students would benefit from preparation of media films at sponsor companies during their internships. Such processing and characterization efforts at host companies will complement the TEM and ASTAR expertise that the students gain at CMU.

6. Time line

In the proposed timeline, we will begin with the simplest set of samples to be prepared and the simplest data to be collected and analyzed, namely samples and data for Task A - Grain Mapping. For crystal orientation and phase mapping under Tasks B and C more time is needed to develop the methodologies for preparation of suitable samples. Data collection and analysis for these two types of mappings will thus begin later in the year and will also require methodology development.

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<th>A. Grain Mapping</th>
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<td>Sample Preparation</td>
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<td>Data Collection</td>
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<th>B. Crystal Orientation Mapping</th>
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<td>Sample Preparation</td>
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<td>Data Collection</td>
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<th>C. Phase Mapping</th>
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<td>Sample Preparation</td>
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<td>Data Collection</td>
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<td>Data Analysis</td>
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7. Carnegie Mellon University and Data Storage Systems Center Facilities and Resources

The proposed project will primarily make use of the Materials Characterization Facility in the Department of Materials Science and Engineering at Carnegie Mellon University, as detailed below. The samples for study will be prepared by other groups, specifically by S. Granz under the guidance of Prof. M. Kryder and prepared in CMU Nanofabrication facility, or by ASTC sponsors such as HGST, Seagate and WD.

Materials Characterization Facility

The Materials Science and Engineering department maintains a state-of-the-art facility in electron microscopy, which is operated for the benefit of the university community at large. The Central Electron Microscopy operates four Transmission Electron Microscopes (TEM) and four Scanning Electron Microscopes (SEM).

The TEM that will be used for the proposed work is the FEI Tecnai F20 Field Emission Transmission Electron Microscope with a point resolution of 0.24 nm and Scanning Transmission Electron with resolution of 0.2 nm. The Tecnai is equipped with a Gatan Imaging Filter and an Energy Dispersive X-ray Spectrometer. All the imaging and spectroscopy software are interfaced on the microscope central computer. The microscope is equipped for Lorentz for magnetic imaging Fersnel and Foucault modes. In March 2011, the microscope was equipped with the NanoMEGAS ASTAR (hardware/software) system for phase and crystal orientation mapping.

The ASTAR system includes the following: (1) Precession Unit “Digistar”; Digistar unit controls the electron beam by forming a small beam (spot), and precessing and translating the beam on the sample. At each location, an electron diffraction pattern is obtained. (2) External optical CCD camera and PC: The external camera allows significantly faster data acquisition times with up to 100 electron diffraction patterns collected per second. The faster acquisition times allow for higher throughput. (3) Acquisition, visualization and orientation mapping software for phases with known crystal structures: The acquired diffraction patterns are compared with pre-generated diffraction patterns to determine the crystal orientation. The orientations are then mapped as inverse pole figure maps (i.e., map of sample direction of interest in the frame of reference of the crystal). (4) The extract, simulator and 3D-electron tomography software packages: Allow the intensities of spots to be measured for crystal structure determination, and allow other types of diffraction patterns to be generated thereby allowing us to obtain a more complete dataset for samples with unknown phases or where defects give rise to additional diffraction spots and thus complex diffraction patterns.

The x-ray analysis facility has a number of diffractometers, a Siemens fluorescence spectrometer, and a Laue back-reflection and Debye-Scherrer camera. The facility also houses a Philips X’pert diffractometer for pole figure collection and in-plane x-ray scans. In short, the accessories available with the various x-ray machines enable pole figure and texture analysis, thin film analysis, high and low temperature studies, stress measurements, and x-ray topography. The high resolution Philips diffractometer produces fine rocking curves and reciprocal space maps for epitaxial films and single crystals. A second Philips X’pert diffractometer has been purchased and has been added to the facilities in the coming year.

Carnegie Mellon Nanofabrication Facility (CMNF)

The Carnegie Mellon Nanofabrication Facility (CMNF), a premier research laboratory in the College of Engineering, is one of the most well equipped university based facilities for data storage thin film and device development in the United States. The facilities include a 4,000 square feet class 100 clean room, three thin film labs, and a photo reduction darkroom. The equipment list can be found at http://www.nanofab.ece.cmu.edu/equipment/index.html.
8. Contact information and biographical sketch

Katayun (Katy) Barmak, Professor
Department of Materials Science and Engineering and the Data Storage Systems Center
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EDUCATION
University of Cambridge, England  Natural Sciences  B.A. 1983
Massachusetts Institute of Technology  Metallurgy  M.S. 1985
Massachusetts Institute of Technology  Materials Science and Eng.  Ph.D. 1989

My research effort since the early 1990s has addressed the formation of the L1_0 ordered phase in CoPt, FePt and related ternary alloys using transmission electron microscopy (TEM) and differential scanning calorimetry (DSC).

INSIC EHDR Program funding in 2010: Quantitative Kinetic Experiments and Models of L1_0 Formation in FePt, CoPt and Related Alloy Films
- Experimental data obtained via DSC and used in model development

ASTC continuation of funding through June 2011: High Throughput Electron Diffraction Based Metrology of Magnetic Materials for Development of Bit Patterned Media (BPM) with Narrow Switching Field Distribution. The current proposal is an extension of the high throughput mapping to HAMR while continuing the planned work with BPMR.
- Experimental data obtained in the TEM

RELEVANT PUBLICATIONS
Supplemental Information

Summary of Prior Accomplishments: My research effort since the early 1990s has addressed the formation of the L1₀ ordered phase in CoPt, FePt and related ternary alloys. The studies in 1990s focused on the characterization of L1₀ formation in 10 nm-thick films using transmission electron microscopy (TEM). The studies since 2000 have focused on the measurement of thermodynamic and kinetic parameters of the transformation using differential scanning calorimetry (DSC). The salient findings are summarized below:

A. L₁₀ Formation Using Dark Field Imaging in the TEM
In this work, the ordered L₁₀ and disordered A₁ regions in post-deposition annealed 10 nm-thick, continuous CoPt and FePt thin films were imaged using the superlattice and fundamental reflections. The ordered fraction was quantified from the dark field images. The studies showed the:
1. The transformation of the A₁ phase to the L₁₀ phase during post-deposition annealing to occur by nucleation and growth of ordered domains.
2. The coercivity of the films to be a linear function of the ordered fraction, providing a simple link between the transformation extent and the magnetic behavior of the films.

B. L₁₀ Formation Using Differential Scanning Calorimetry (DSC)
In this work, the formation of the L₁₀ phase from the A₁ phase was studied in CoPt, and FePt; the latter with a series of ternary alloying additions. The studies made use of micrometer-thick, but nanocrystalline films (as deposited grain size 7-10 nm). The use of micron thick films is required for adequate signal in the DSC. The summary of the results are as follows:
1. In agreement with indirect measurements such as coercivity of annealed films, FePt has a lower (kinetic) ordering temperature (430 vs. 520 °C), and a lower activation energy for L₁₀ phase formation (1.7 vs. 2.8 eV) than CoPt.
2. The Fe content in FePt has a large effect on the kinetic ordering temperature. The lowest kinetic ordering temperature was reported at lowest at 359 °C for a 54.4 at% Fe binary alloy film. However, more recent composition measurements using energy dispersive X-ray fluorescence (EDXRF) with thin film standards and energy dispersive X-ray spectrometry (EDS) have shown the composition to be ~49 at.%.
3. For films deposited at room temperature and ordered by post deposition annealing, no ternary alloying addition (Mg, V, Mn, Cu, Ni, Ag, Au, B, or C studied to date) results in lowering the kinetic ordering temperature (KOT). The only alloying addition that does not negatively impact the formation kinetics of the L₁₀ phase is Cu. Cu additions also have the desirable effect of lowering the Curie temperature.
4. Using the Johnson-Mehl-Avrami-Kolmogorov (JMAK) nucleation and growth model, time-temperature-transformation (TTT) diagrams for the L₁₀ ordered fraction in post-deposition annealed films were calculated in order to aid in the choice of times and temperatures for full transformation to L₁₀. Using geometric corrections, the TTT curves for 10 nm-thick, continuous films were also calculated.