Atomic-Scale Segregation and Fluctuations in Chemical Ordering FePt Thin Films

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Instruments

During the past decade, the magnetic bit for areal storage density has decreased to a size that is rapidly approaching the superparamagnetic barrier, the thermal stability limit where magnetization randomly fluctuates due to thermal magnetic volume. The thermal stability of very small magnetic crystals or grains can be improved if the material has a large uniaxial magnetocrystalline anisotropy, K. The L1₀ phase of FePt has been identified as an attractive storage media because of its high K. [1] When this intermetallic is sputter-deposited as a polycrystalline thin film, a metastable solid solution facentered-cubic phase is formed [2]. By annealing temperatures in excess of 500°C, the crystalline lattice anisotropically orders into the desired hard magnetic L1₀ phase. There has been limited experimental studies on how subtle compositional fluctuations, at the nanoscale, evolve and contribute to this polymeric phase transformation. This poster provides atom probe tomography and transmission electron microscopy studies to elucidate and quantify how compositional segregation evolves during this phase transformation.

Experimental Details

Fe₃Pt thin films were DC magnetron co-sputter-deposited from commercial purity, elemental Fe and Pt targets in an AIX TEAT 1500 F system.

The thin films were simultaneously grown onto ambient temperature Si [001] wafers as well as pre-fabricated Si micro-tip arrays [3]. These arrays consist of Si pillars that are 100μm tall with each pillar narrowing to a tip radius of < 20 nm. These tips provide the geometry required for field evaporation in the atom probe. The Si wafer provided a coupon for representative X-ray diffraction (XRD) analysis of the film deposited onto the micro-tip array

Anomalist:
The tips, wafers and a Ti foil (used to getter residual oxygen) were encapsulated in a quartz tube that was evacuated to < 10⁻¹⁰ Torr then back-filled with Ar/4%H₂ gas mixture to 10 Torr for the annealing study at 600°C for 10 minutes. The evacuation and gas purging was done for five cycles. The annealing was done in a standard laboratory tube furnace.

Atom Probe Tomography:

An Image Scientific Instruments Local Electrode Atom Probe (LEAP®) 3000XSII was used to collect data in a voltage pulse mode with a target evaporation of 0.5%, a pulse fraction of 20% and a specimen temperature of 120K. Laser manipulation was performed in the LEAP® resulted in a propensity of fracture failures for this specimen.

Transmission Electron Microscopy - Focus Ion Beam “lift-out” of an Atom Probe tip:

To assist in the reconstruction of the atom probe data sets, Transmission Electron Microscopy (TEM) imaging was performed on a Focus Ion Beam (FIB) lift-out specimen of an atom probe tip, as shown in Fig.1. The TEM was performed using a 200kV FEI Tecnai F20. The FIB milling was performed using a FEI Quanta 3D dual beam FIB. The phase identification of the films was determined by XRD using a Bruker D8 diffractometer operating at 40kV and 35 mA with Cu Kα radiation as the source.

Results & Discussion

XRD of the as-deposited Fe₅₅Pt₄₅ film, shown in Fig. 2(a), indicates the A1 phase with a strong [111] fiber texture. The atom probe reconstruction of this specimens indicates high density regions, Fig 2(b), within the microstructure that is approximately 1 nm in width. To verify that these high density regions were not aberrations in the reconstruction, TEM Fresnel contrast images of an atom probe tip were taken. The thru-focus series of images, Δz= -1.1 μm, shown in Fig 3 indicated a change in contrast around the grains from dark (under-focus) to bright (over-focus). This change in contrast is consistent with a higher density in the grain boundaries [4]. The size and shape of the atom probe reconstruction of these high density regions are consistent with the TEM imaged grain boundaries. A one-dimensional concentration profile was performed through a reconstructed grain boundary, as shown in Fig. 4. An increase in the Pt concentration was observed at this boundary.

Conclusions

A series of atom probe studies were performed to investigate how compositional fluctuations in FePt contribute in the A1 to L1₀ phase transformation. The atom probe reconstructions showed small levels of Pt segregation at grain boundaries in the as-deposited film. TEM Fresnel-contrast imaging confirmed the presence of these high-density regions. These results provide initial experimental verification of modeling predictions of Pt surface segregation. XRD analysis of a 600°C for 10 minute anneal confirmed the A1 to L1₀ phase transformation. The atom probe reconstruction did indicate subtle compositional fluctuations at the boundaries that were Fe-enriched. This inversion of composition indicated a preference for Pt reintegretion into the grain to achieve an optimum Fe:Pt ratio in the L1₀ phase transformation.

References