

Microstructural Analysis of Fe₃₅Pt₄₉Cu₁₆ Thin Films

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The $L1_0$ FePt phase is a candidate material for achieving ultra high magnetic storage density because of its high magnetocrystalline anisotropy K_u [1]. When FePt is sputter-deposited as a thin film, it adopts the magnetically soft, solid solution, face-centered cubic phase denoted as A1. The $L1_0$ phase can be obtained through subsequent annealing at temperatures greater than 500°C. However, annealing FePt results in detrimental grain growth. Takahashi *et al.* [1] have shown that Cu in FePt can reduce the A1 to $L1_0$ phase transformation temperature to approximately 300°C. Nonetheless, the addition of Cu accelerates the grain growth of FePt. To date, there have been few atomic scale characterization studies to understand the interrelationship of metal additives on the ordering and grain growth in FePt thin films. In this paper, the grain boundary composition in a Fe₃₅Pt₄₉Cu₁₆ thin film has been characterized using an Imago Scientific Instrument Local Electrode Atom Probe (LEAP).

A 100 nm Fe₃₅Pt₄₉Cu₁₆ film was DC-magnetron sputter-deposited onto a silicon substrate from elemental Fe, Pt, and Cu targets. A 10 nm Si₃N₄ underlayer and 10 nm Si₃N₄ capping layer were used to prevent undesired reaction with the Si substrate or atmosphere during subsequent annealing studies. Prior to deposition, the base pressure in the sputtering chamber was $\sim 1.2 \times 10^{-7}$ Torr where upon ultra-high purity Ar was flowed at 10 sccm into the chamber to a pressure of 2 mTorr. Post-deposition, the film was diced into $\sim 5 \times 5$ mm squares. These samples were then annealed at 350°C, 400°C, 600°C and 800°C for 1 minute, 30 minutes and 100 minutes in a laboratory tube furnace. The quartz furnace tube was evacuated to a base pressure of $\leq 1 \times 10^{-4}$ and back filled to a pressure of 30 Torr with Ar/5%H₂ prior to heating.

X-ray diffractometry (XRD) was conducted using a Bruker D8 diffractometer using CoK α radiation for crystal structure determination. An alternating gradient magnetometer (AGM) was used to measure the magnetic coercivity, H_c , for each sample. The Fe₃₅Pt₄₉Cu₁₆ annealed at 600°C for 30 minutes sample was fabricated into atom probe tip using a focus ion beam lift-out technique [2]. The atom probe tip was analyzed in a Local Electrode Atom Probe (LEAP) with a field evaporation rate of 1.0%, a pulse energy of 0.4nJ, a pulse rate of 250 kHz, and a base temperature of 50K. Transmission electron microscopy (TEM) plan-view foils were prepared and characterized in a FEI Tecnai F20 TEM for phase and grain size analysis.

The XRD scan, Fig. 1, of the Fe₃₅Pt₄₉Cu₁₆ film annealed at 600°C for 30 minutes was indexed as the $L1_0$ phase. AGM results indicated that this sample had an H_c of 7.09 kOe, consistent that the phase was magnetically hard. This sample exhibited a distribution in grain sizes, Fig. 2, with the mean grain size being ~ 23 nm. The atom probe reconstruction, Fig. 3a, indicates a density fluctuation which is believed to be a grain boundary based on previous TEM and LEAP correlation studies [3]. Unlike these former studies [3], which indicated Pt segregation to the boundaries, this annealed Fe₃₅Pt₄₉Cu₁₆ showed a significant increase in Fe content and slight increase in Cu content at boundary as compared to the nominal bulk composition, Fig. 3(b). The role of preferential segregation and grain growth will be discussed.

References:

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- [2] K. Thompson, D. Lawrence, D.J. Larson, J.D. Olson, T.F. Kelly, and B. Gorman *Ultramicroscopy* **107** (2007) 131.
- [3] K.L. Torres and G.B. Thompson, *Materials Research Society Proceedings* Vol **1032** (2008) I14-16
- [4] The authors gratefully acknowledge NSF-DMR-CAREER-0547445 for support of this work. In addition, B.C. Hornbuckle acknowledges the Microscopy Society of America for the Undergraduate Research Fellowship. The TEM was acquired from NSF-DMR-MRI-0421376.

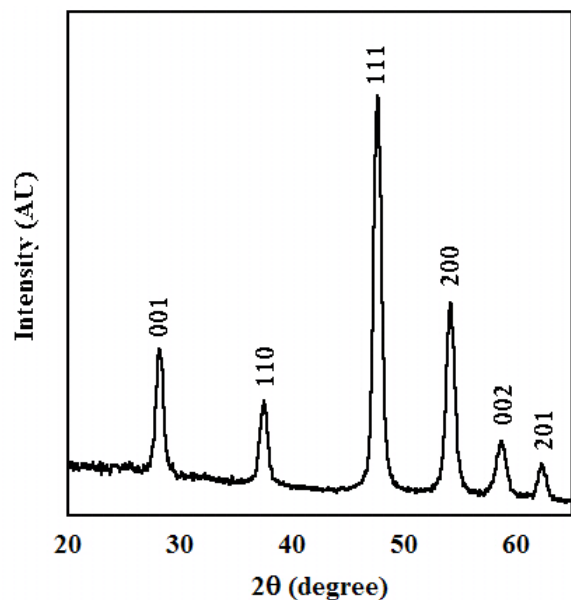


Fig. 1: XRD measurement, using Co K_{α} radiation, of the $\text{Fe}_{35}\text{Pt}_{49}\text{Cu}_{16}$ film annealed at $600^{\circ}\text{C}/30$ minutes.

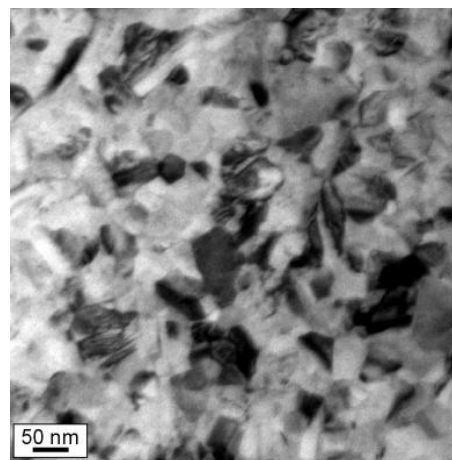


Fig. 2: TEM bright field image of $\text{Fe}_{35}\text{Pt}_{49}\text{Cu}_{16}$ film annealed at $600^{\circ}\text{C}/30$ microstructure.

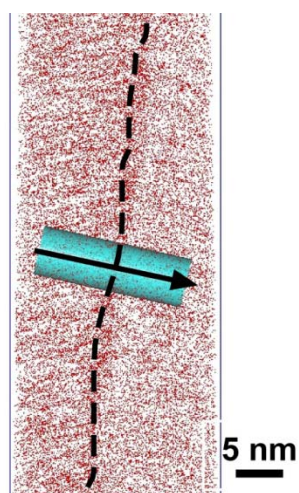


Fig. 3a: Atom probe reconstruction of the $\text{Fe}_{35}\text{Pt}_{49}\text{Cu}_{16}$ film annealed at $600^{\circ}\text{C}/30$ minutes. Dotted lines indicate significant Fe diffusion to the grain boundary.

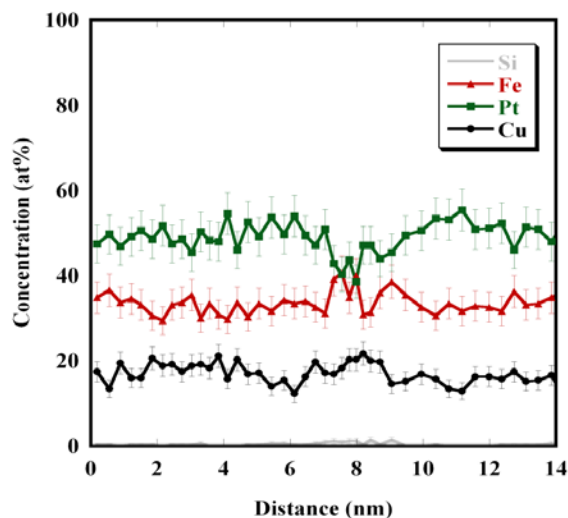


Fig. 3b: Compositional profile through the grain boundary. Slight increase in Fe and Cu content observed at grain boundary.