DOI: 10.1017/S1431927605502538
Microsc Microanal 11(Suppl 2), 2005

IN-SITU TEM OF ION IRRADIATED FE-PT ALLOY NANOSTRUCTURES

G.B. Thompson[1], N.W. Morgan[1], and R. Birtcher[2]

[1]University of Alabama, Department of Metallurgical and Materials Engineering, Box 870202, Tuscaloosa, AL 35487
[2]Argonne National Laboratory, Materials Science Division, 9700 S. Cass Ave MSD/212, Argonne, IL 60439

Considerable interest has been generated in Fe-Pt alloys because of their potential use in a variety of magnetic related applications. For example, the $L1_2$ phase of Fe_3Pt has potential uses in GMR devices or as spring magnets[1]. The $L1_0$ phase of FePt, because of its high magnetocrystalline anisotropy, is being thoroughly researched as the potential storage media for next-generation ultra-high-density bits[2]. In each application, controlled grain sizes of the intermetallic phase are essential. Unfortunately, the as-deposited material adopts a metastable face-centered-cubic (*fcc*) structure, wither synthesized as a nanoparticle or deposited as a thin film[2,3]. A subsequent anneal at 500+^{o}C is required to chemically order the phase into the intermetallic structure with the desired magnetic properties. During annealing, the granular dimension of the as-prepared material are lost as a result of grain growth or sintering reactions. This is a particular concern for $L1_0$ FePt because the ultimate bit cell size is determined by the grain size of the film or the self-assembled dimensions of individual nanoparticles.

In our study, we have irradiated Fe-Pt nanostructures with Kr^+ ions at energies of 500keV. The Fe-Pt films were sputtered deposited with nominal compositions of $Fe_{75}Pt_{25}$ or $Fe_{50}Pt_{50}$ determined by deposition rates and verified by Energy Dispersive Spectroscopy. The FePt nanoparticles were synthesized by a metal-salt decomposition reaction[3]. Ion penetration within a lattice will displace atoms from their nominal lattice sites generating lattice strains as well as vacancy formations. The use of ion irradiation allows a controlled processing of defects to be generated in the as-prepared *fcc* Fe-Pt alloy structure. These defects could facilitate ordering reactions at a lower temperatures, hence a potential reduction of grain growth or sintering. A limited number of studies have been reported on ion irradiation of stoichiometric FePt[4-5]. In these reports[4-5], the films were in-situ annealed at 280^{o}C during which He ion irradiation at energies of 30-130 keV facilitated chemical ordering. The ion and energy choice of our study would result in nuclear collisions with a larger cascade collision envelope than the electronic collision associated with He, thus providing a unique comparison. Using in-situ TEM, we are able to monitor real-time evolution of the granular morphology with phase for a complete characterization of the nanostructure under the ion beam. Argonne National Laboratory has a unique 300keV TEM that is connected directly into an ion accelerator beam line to perform these types of studies.

Ion irradiation at dosages greater than 10^{12} ions/cm^2 resulted in a texture evolution from the predominate {111} to other orientations, as indicated in the diffraction patterns and the diffraction contrast in Fig. 1 (a) and (b). No grain growth or change in crystallographic phase was observed with these changes. At 300^{o}C, the Fe-Pt25% film commenced the $L1_2$ transformation, as evident with the presence of the superlattice {110} reflection shown in Fig. 2 (note the lack of large grain growth). As the temperature increased beyond 400^{o}C, the film's grains grew rapidly and prevalent twinning in the grains was observed.

The FePt films exhibited similar microstructural changes to the Fe-Pt25at.% films under the ion beam at ambient temperature, including the texture evolution of the grains at dosages up to 10^{15} ions/cm^2 and the requirement for an anneal to promote the ordering transformation. *Unlike Fe_3Pt, we did not observe the reduction in the ordering transformation temperature*. The $L1_0$ ordering commenced at temperatures near 500^{o}C. This was a very surprising result since the ordering temperature was shown to be ~300^{o}C with the simultaneous irradiation of He ions[4-5] and ordering was lowered for Fe_3Pt with the Kr^+ ion. This difference between ordering is suspected to be a

result of the free mobility of defects. The lowering of the order temperature appears to be a strong function of the composition of Fe-Pt.

Finally, a series of 3 nm and 6 nm FePt nanoparticles were ion irradiated under similar conditions. The irradiation did not promote discernible ordering in the nanoparticle arrays as determined by the diffraction pattern of pre- and post-irradiation up to 10^{14} ions/cm^2 (no anneal). The 6 nm nanoparticles appeared to be irregular in shape, as compared to the spherical shaped 2 nm nanoparticles. This suggests that during the nanoparticle synthesis, the larger particles are formed from the coalescence of smaller nanoparticles. By using ion irradiation, at ambient temperature, we were able to cause the irregular-shaped nanoparticles to evolve into a spherical shaped morphology. For particularly closely spaced nanoparticles, the imparted ion energy allowed surface migration and coalescence of these particles to form larger, spherical particles.

In conclusion, ion irradiation with Kr^+ at 500keV at ambient temperature was insufficient to promote ordering and annealing is still required. The ordering temperature with ion dosage appears to be a strong function of composition, as demonstrated between the Fe_3Pt (300°C) and FePt films (500°C). Ion irradiation appears as a potential processing mechanism in controlling and refining the shape of larger nanoparticles.

References:

1. M.A. Nahid and T. Suzuki "Magnetic anisotropy of Fe3Pt alloy thin films" *Applied Physics Letters* **85**(18) 4100-4102.
2. Barmak, K. J. Kim, S. Shell, E.B. Svedberg, and J.K. Howard "Calorimetric studies of the A1 to $L1_0$ transformation in FePt and CoPt thin films" *Applied Physics Letters* **80**(22) (2002) 4268-4270.
3. Shouheng Sun, C.B. Murray, Dieter Weller, Liesl Folks, Andreas Moser, "Monodisperse FePt nanoparticles and ferromagnetic FePt nanocrystal Superlattices," *Science* **287**, 1989 (2000).
4. Bernas, H., J.-Ph. Attane, K.-H. Heinig, D. Halley, D. Ravelosona, A. Marty, P. Auric, C. Chappert, and Y. Samson "Ordering intermetallic alloys by ion irradiation: a way to tailor magnetic media" *Physical Review Letters* **91**(7) (2003) 77203-1 -77203-4.
5. Ravelosna, D., C. Chappert, and V. Mathet "Chemical order induced He+ ion irradiation in FePt (001) films" *Journal of Applied Physics* **87**(9) (2000) 5771-5773.
6. G.B Thompson and N.W. Morgan gratefully acknowledge NSF-MRSEC-DMR-0213985 in support of this work.

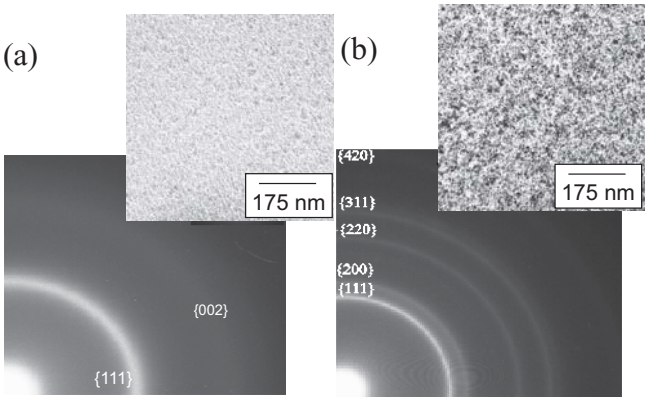

Fig. 1: Bright-field micrographs of Fe-Pt25at% film. Note the change in texture as evident by the increase diffraction contrast and appearance of additional diffraction rings in the pattern for each film. (a) as-prepared (b) 2.5x10^{14} ions/cm^2

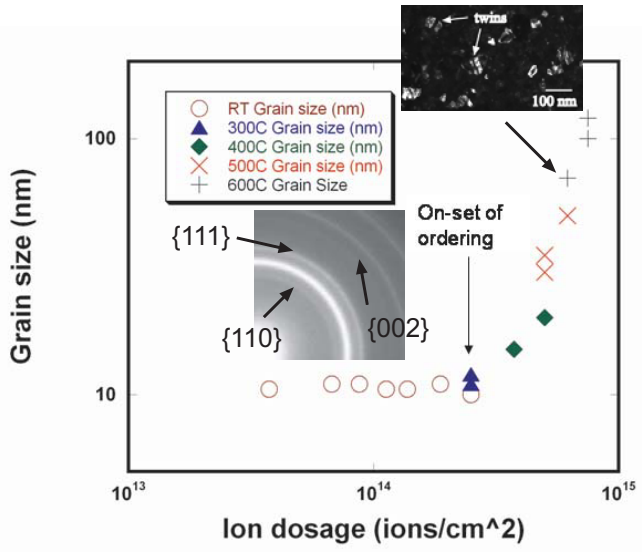

Fig. 2: A plot of grain size with ion dosage and temperature for Fe_3Pt.

https://doi.org/10.1017/S1431927605502538 Published online by Cambridge University Press