FePt微粒子および集積膜の磁気特性

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あらまし 水溶液中での反応をベースとした逆ミセル法、有機溶液内で有機金属化合物を熱分解および還元する方法の2種類の溶液プロセスによりFePtナノ粒子を作製した。どちらの方法においても直径約3nmの大さきの粒子が生成した。これらのFePtナノ粒子を用いて自己集積膜の作製を試みたところ、前者の方法で作製した粒子は自己集積が見られたが、後者の方法で作製した粒子については規則的な配列は見られなかった。自己集積した粒子も熱処理により粒子同士の焼き方が起こった。しかしながら、粒径は最大でも10nm程度であった。集積膜の磁気特性評価はその磁化量が小さいことから更なる実験精度の向上が必要であるが、粉末での評価においてはL1₀相におけるFeとPtの規則配列が保磁力や磁気緩和に大きな影響を及ぼすことがわかった。

キーワード FePt、ナノ粒子、液相合成、自己集積、磁気緩和

Magnetic Properties of FePt particles and their assembly-films

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Abstract Nanoparticles of FePt were fabricated by two types of solution process. One is thermal decomposition and reduction of organometallic compounds in an organic solvent, and the other is reduction of metal ions in reversed micelles based on an aqueous solution process. In either way, monodisperse FePt particles of about 3nm are formed. Self-assembly was observed in case of the particles fabricated by the former method, while in case of the latter method, self-assembly was not observed. The maximum diameter of particles in assembled film increased up to 10nm by annealing. Magnetic evaluation in the form of powder showed that atomic ordering of Fe and Pt in L1₀ structure strongly influenced coercivity and magnetization decay. It is necessary to improve the accuracy for magnetic measurements of very thin assembly-films since their magnetization is too small.

Keyword FePt, Nanoparticle, Solution synthesis, Self-assembly, Magnetization decay

1. Introduction

An ordered alloy FePt is attracting as a solution to a problem of thermal decay in high-density magnetic recording media because of its great magnetocrystalline anisotropy. In particular, its nanoparticle-assembly is aggressively studied for a future magnetic recording media. The present report describes preparation and characterization of the FePt nanoparticles and their assembly-films. The particles were fabricated by wet processes and their array-films were done by self-assembling.

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process. Two wet processes for preparing the FePt particles were compared in this study. One is based on an aqueous solution process called "reverse micellar solution method\(^2\)," and the other is based on an organic reaction in solutions using metal-organic materials\(^1\). Characterization was performed mainly by X-ray diffractometry, transmission electron microscopy, and magnetic evaluation including thermal magnetization decay.

2. Experimental

An aqueous solution of FeCl\(_2\) and H\(_2\)PtCl\(_6\) were used in the reverse micellar method. Fe\(^{2+}\) and Pt\(^{4+}\) ions were reduced by NaBH\(_4\), and disordered FePt alloy particles of about 3 nm in diameter were obtained at room temperature. In the case of the metal-organic process, Fe(CO)\(_5\) was decomposed to Fe and Pa(acac)\(_2\) was reduced into Pt in the reaction at 282°C, resulting in the formation of disordered FePt alloy particles of about 3 nm in diameter. The composition of the particles was almost Fe\(_{59}\)Pt\(_{37}\).

The particle-assemblies were fabricated by dropping an FePt particle-dispersed solution on SiO\(_2\)/Si substrates or a collodion membrane. Annealing for both powder and film samples was performed at 300-650°C in Ar+H\(_2\) atmosphere to obtain L\(_{10}\) type FePt.

Particle size and their assembly were observed by a transmission electron microscope (TEM), crystal structure, in particular, atomic ordering of Fe and Pt in L\(_{10}\) superlattice was determined by an X-ray diffractometer (XRD), and magnetic properties of the particles and array-films were investigated by a SQUID and a Kerr magnetometer. Magnetization decay in the form of powder was investigated using a vibrating sample magnetometer (VSM) and PPMS (Quantum Design Corp.)\(^3\).

3. Results & discussion

3.1. Powder sample

FePt As-synthesized FePt particles of 3 nm in average diameter were synthesized for both wet processes. Figure 1 shows the particles fabricated by the reverse micellar solution process. The particles exhibited disordered fcc phase, and lattice image is observed in a TEM image of a single particle as shown in Fig. 1(b) and superparamagnetism. Annealing above 400°C changed the structure of the particles from disordered fcc to ordered phase called L\(_{10}\) structure as shown in Fig. 2. The atomic ordering of Fe and Pt in L\(_{10}\) structure was evaluated for the powder samples annealed at 450 and 550°C from the XRD patterns. The XRD patterns were fitted as a superposition of disordered fcc phase and ordered L\(_{10}\) phase, and the ordering x was defined as the molar fraction of the ordered phase. x of the samples annealed at 450 (sample 1) and 550 (sample 2) °C was 0.66 and 0.9, respectively. After annealing, the particle diameter increased to 10-15 nm in average.

![TEM images of as-synthesized FePt nanoparticles by reverse micellar solution process.](image)

![Annealing temperature dependence of XRD patterns of Fe\(_{53}\)Pt\(_{47}\) particles fabricated by reverse micellar solution process.](image)
3.2. Particle-assembled film

Regarding the preparation of a particle-array by self-assembling, while the FePt particles synthesized by the metal-organic process were partly arrayed as shown in Fig. 5, the particles by the reverse micellar process were randomly deposited on a substrate. The difference in the assembling status is probably originated in the difference in surfactant and solvent. To obtain the array of the particles, it is necessary to optimize the distance between the particles and the potential of the particle surface by selecting the surfactant and solvent.

After annealing FePt particle-assemblies, their particle size was increased by aggregating together. The particle size increased with the annealing temperature Ta as shown in Figs. 6 (a) and (b), and the size increased up to 10 nm in diameter at 650°C with increasing the annealing temperature. A TEM cross sectional view revealed that a single-layered particle-assembly was obtained in this study as shown in Fig. 7. Figures 8 and 9 show a Kerr hysteresis loop and an M-H loop of the assembly after annealing at 550°C. Since the maximum field is different between the two measurements, it is difficult to discuss the difference in the shape of the loops and the magnetization process, in particular, coercivity in Figs 8 and 9. Moreover, it is necessary to improve evaluation methods of magnetic properties for assembly-films, in particular, for a single or a few layer(s) since the detected signal in the magnetic measurements is too low regarding such films.

![Fig. 3 M-H loops of highly (sample 1) and poorly (sample 2) ordered FePt particles measured in the form of powder.](image1)

![Fig. 4 Magnetization decay of highly (sample 1) and poorly (sample 2) ordered FePt particles measured in the form of powder. H is a demagnetizing field.](image2)

![Fig. 5 TEM image of FePt particle-assembly on collodion film. FePt particles were fabricated by metal-organic process.](image3)
FePt particles were prepared by two wet processes. Ordered phase of FePt was obtained by annealing at 400-650°C, resulting in exhibiting ferromagnetism and increasing coercivity. Coercivity was increased and magnetization decay was decreased with increasing the annealing temperature since the atomic ordering of Fe and Pt in L1₀ structure was improved. Aggregated particles of 10 nm in the maximum size were observed in a single-layer of the particle-assembly after annealing.

References

