コアー‐シェル構造 CoPt‐$CoFe_2O_4$ 粒子の作製とその還元雰囲気熱処理による結晶構造および磁気特性の変化

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Structural and magnetic property changes of core-shell structured CoPt-$CoFe_2O_4$ particle during heat treatment under reducing atmosphere

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1. Introduction

Energy product (BH)max, which is a function of saturation magnetization(M_s) and magnetic anisotropy of the material, is the decisive factor in the development of strong magnets. Though record energy product has been obtained by the invention of $Nd_2Fe_{14}B$, the use of rare-earth elements such as Nd has prompted the researchers to look for (a) new compounds composed of earth abundant materials with high magnetization and high anisotropy on one hand and (b) exchange-coupling between hard-magnetic compound and a soft phase with high Ms. We attempted the preparation of exchange-coupled magnet through the formation of CoPt(core)-FeCo(shell) using solution technique and subsequent heat treatment. Here, the synthesis of core-shell structured CoPt-$CoFe_2O_4$ particle and structural and magnetic property changes during heat treatment under reducing atmosphere are reported.

2. Experimental

CoPt nanoparticles with varying composition and size ranging between few nm to few tens of nm were synthesized by heating in an alcoholic solution containing cobalt acetate, dihydrogen hexachloroplatinate and surface protecting agent, oleylamine. Then, these particles were introduced into a mixture of oleylamine and oleic acid, containing cobalt and iron acetylacetonate. Finally, the suspension was heated to temperatures above 250 °C to obtain the $CoFe_2O_4$-coated CoPt particles. The core-shell structured CoPt-$CoFe_2O_4$ particles were heat treated under reducing atmosphere to obtain a soft-hard magnetic composite. Morphological, structural and magnetic properties of the products obtained at various heat treatment temperatures were analysed using TEM, XRD and VSM, respectively.

3. Results and Discussion

A typical result obtained from the morphological analysis of the samples synthesized using the above technique is shown in Fig. 1(a). The presence of the CoPt core surrounded by the cobalt ferrite shell was confirmed from the observation under TEM and EDS mapping. Though the heat treatment under hydrogen atmosphere was carried out to reduce the $CoFe_2O_4$ shell and to obtain high Ms and magnetically soft FeCo alloy shell, the presence of FeCo was not determined at none of the heat-treatment temperatures. In contrast, $CoFe_2O_4$ crystals grew with higher heat treatment temperatures. Then, at 600 °C underwent reduction and diffused into the CoPt core. This transition was clearly observed in the XRD profiles (Fig. 1(b)) and in the transmission micrograph (Fig. 1(c)). On the other hand, the coercivity of the particles exhibited a maximum of 1.1 kOe, when shell was partially reduced. The details of the structural transformation process and the magnetic properties obtained thereof will be discussed.

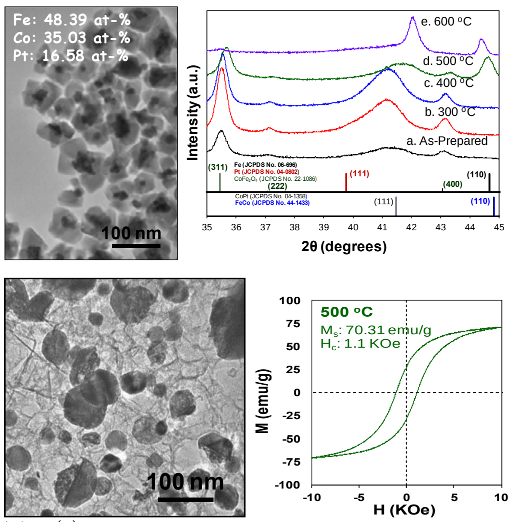

図 1 (a) TEM photograph of core-shell structured CoPt/$CoFe_2O_4$, (b) XRD patterns of the product heat-treated at different temperatures, (c) micrograph of the product annealed at 600℃, (d) magnetic loop of the product annealed at 500℃.