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ESR study of thermal demagnetization processes in ferromagnetic nanoparticles with Curie temperatures between 40 and 60 °C

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Abstract

Thermal demagnetization in the vicinity of the Curie temperature of silver and sodium manganite nanoparticles, as well as copper—nickel and palladium—nickel alloy nanoparticles were studied by both static magnetic measurements and by electron spin resonance (ESR). ESR data indicate that some magnetic ordering remains even above the Curie temperature, determined by static magnetometry. Mechanisms of thermal demagnetization in alloy nanoparticles appear to be different from that in manganites. © 2006 Elsevier B.V. All rights reserved.

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0. Introduction

Nanoparticles made from various ferromagnetic materials (alloys, ferrites, manganites) with Curie temperatures ($T_{\rm c}$) between 40 and 60 °C are intensively studied as potential mediators for magnetic fluid hyperthermia treatment of cancer with parametric feedback temperature control [1–8]. During the procedure, the particles are introduced into the tumor and heated by alternating magnetic field (AMF) with a frequency in the hundreds of kilohertz range. The particles gradually demagnetize and lose the ability to absorb energy from AMF as their temperature approaches $T_{\rm c}$. Therefore, temperature in the tissue remains within the therapeutic limits despite non-uniform distribution of the particles throughout the tissue and heat losses [1].

Ideal mediator particles for magnetic fluid hyperthermia with parametric feedback should be biocompatible, have

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the Curie temperature in the medically suitable range, and efficiently absorb AMF energy (i.e., have high specific power absorption rate) below T_c . They should also rapidly loose the ability to absorb AMF energy in the vicinity of T_c , thus, their thermal demagnetization rate (f = dM/dT, where M is the relative magnetization of the sample, and Tis temperature) should be high near T_c . The thermal demagnetization rate of the mediator particles is the main feedback parameter for high-frequency magnetic heating. Thermal demagnetization occurs over a range of temperature even in metals; for example, the last 50% of demagnetization of an ideal single domain nickel monocrystal occurs over $3-5^{\circ}$ interval [9]. The value of ffor ultradisperse particles is determined both by peculiarities of the demagnetization process in the bulk material, and by variations in chemical composition and phase content within individual particles, as well as by variability of characteristics between different particles (size, shape, chemical composition, phase content, condition of the surface, etc.). An overview of the effects of these parameters on magnetic properties of nanoparticles can be found in Ref. [10].

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We studied thermal demagnetization of ultradisperse powders of copper–nickel and palladium–nickel alloys and that of several perovskite manganites with magnetic transition temperatures from 40 to 60 °C using static magnetic measurements and electron spin resonance (ESR) measurements. ESR provides information about the state of the spin system, which changes during thermal demagnetization and determines the ability of the mediator particles to absorb AMF energy [9].

1. Materials and methods

Ultradisperse powders of $La_{1-x}Ag_xMnO_{3-\delta}$ and $La_{1-y}Na_yMnO_{3-\delta}$ were prepared by the cryochemical method [1]. Solutions of nitrates and acetates of the corresponding elements, mixed in the stoichiometric ratio, were sprayed into liquid nitrogen and freeze-dried at 5×10^{-2} mbar. Thermal decomposition of the freeze-dried salt precursors at $500-750\,^{\circ}\text{C}$ in air was followed by several de-agglomeration procedures.

CuNi (28 atomic% of Cu) alloy nanoparticles were prepared by co-precipitation of salts from solution followed by reduction in hydrogen and thermal treatment [8]. PdNi (22 atomic % of Ni) alloy nanoparticles were manufactured by a similar technique with reduction by carbon monoxide.

Sodium manganite particles were estimated from TEM images to be 40–100 nm in diameter, while silver manganite particles ranged from 25 to 350 nm. Cu–Ni alloy particles varied from 5 to 50 nm.

Static magnetic measurements of dry powders were performed using a Faraday balance (Bruker) with a custom temperature control unit.

ESR measurements of dry powders were done using Bruker EMX-8/2.7 spectrometer, working in the X range (9.43 GHz). The temperature was varied from 293 to 373 K, and spectra were recorded at 5° intervals. The spectra were analyzed using the WinEPR software package.

2. Results and discussion

According to the Faraday balance measurements (Fig. 1), Curie temperature of La_{1-x}Ag_xMnO_{3- δ} nanoparticles is 318 K (45 °C) and for La_{1-y}Na_yMnO_{3- δ} particles $T_c = 323$ K (50 °C). For CuNi nanoparticles, T_c was determined to be 333 K (60 °C), and for PdNi particles 348 K (75 °C).

Fig. 2 shows ESR adsorption spectra of La_{1-y} $Na_yMnO_{3-\delta}$ nanoparticles during thermal demagnetization. At low temperatures, the spectra are wide and asymmetric, which is typical for ferromagnetic resonance (FMR) [9,11]. The lines have Gaussian shape, also characteristic for FMR. As the temperature increases, the spectra are gradually transformed into narrower symmetric curves with Lorentzian shape, typical for paramagnetics. The area under the RF absorption curve (S), which is proportional to the sample magnetization M, also decreased systematically as the temperature increased. On

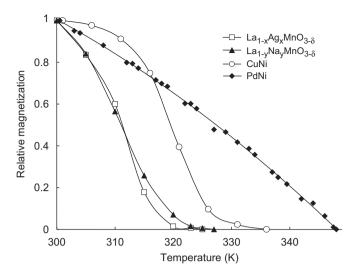


Fig. 1. Thermal demagnetization of mediator nanoparticles (static measurements, Faraday balance).

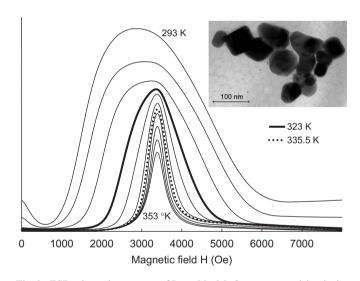


Fig. 2. ESR adsorption spectra of $La_{1-y}Na_yMnO_{3-\delta}$ nanoparticles during thermal demagnetization at temperatures between 293 and 373 K. The bold line indicates the specter taken at the Curie temperature measured by the Faraday balance. Insert: TEM image of the nanoparticles.

this and the following figures, the spectrum corresponding to the Curie temperature determined by the static measurements is shown as a bold line. This spectrum is clearly still a wide asymmetric FMR-type spectrum, suggesting that some magnetic ordering remains in the sample.

The dotted line represents the lowest temperature spectrum shaped as a Lorentz line according to the computer analysis. Such ESR absorption spectrum indicates paramagnetism of the sample, although the line is still wider than that of typical paramagnetics. Thus, this is the magnetic transition temperature (or Curie temperature) determined by ESR. For sodium manganite, it is $335 \pm 2.5 \, \text{K}$, which is 12° higher, than the Curie temperature determined by the Faraday balance measurements.

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