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Multi-peak ferromagnetic resonance in Co nanowires array

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ABSTRACT

Ferromagnetic resonance in an array of Co nanowires electrolytically deposited into nanoporous alumina template is investigated at four microwave frequencies in the range from 9.3 to 69.7 GHz. The array consists of highly textured hcp Co with hexagonal axes perpendicular to the nanowires. The spectra measured at higher frequencies can be decomposed into four wide resonances peaks. Different mechanisms, which can lead to the multi-peak resonance, are discussed.

1. Introduction

Large attention has been recently paid to magnetic properties of nanowires embedded in insulating nonmagnetic matrices because for their potential use in microwave and other applications. Nanowire arrays exhibit larger saturation magnetization than conventional microwave ferrites, which allows achieving higher operating frequencies. Small diameters of nanowires compared to the skin depth and their mutual insulation by the dielectric matrix reduce the eddy current losses associated with the metallic nature of the magnetic component. Cobalt nanowire arrays are particularly suitable for some applications because of high natural ferromagnetic resonance frequency [1].

Ferromagnetic resonance (FMR) is the standard experimental technique, which can be used for investigation of ferromagnetic materials at microwave frequencies. Two different options can be used for FMR measurements. In the conventional FMR technique the excitation frequency is constant and the microwave absorption of the sample is measured as a function of applied magnetic field (field swept mode). The other method uses constant magnetic field and measures the response of the sample as a function of exciting signal frequency (frequency swept mode). This method is often used in the network analyzer FMR measurements. Nanowire arrays are generally complex assemblies of ferromagnetic particles. Their FMR spectrum is given by the superposition of the spectra of individual nanowires. The wires need not be identical (in the shape, structure or magnetic state) and the resulting FMR spectrum can be rather complicated.

Many authors have observed two peaks in the resonance spectra of nanowires arrays. Using the frequency swept FMR technique two resonance peaks were found in nonsaturated arrays of CoFeB [2,3], CoFe and NiFe [4,5] nanowires. In the field swept measurements the double-peak spectra were reported for Ni [6–9], Co [9–11] and CoFeB [12] nanowires arrays. Different explanations of the double-peak resonance have been proposed. In case of low field measurements the two oppositely magnetized wire populations in the nonsaturated state are responsible for two different resonance frequencies [3,12]. Arias and Mills [13] attributed the small splitting of resonance peak, observed by Ebels et al. [6] at 23.6 and 34.4 GHz on Ni nanowires, to the excitation of standing exchange/dipole mode. Ramos et al. [7] suggested that a week satellite on the high field side of main resonance in Ni nanowires could be explained by the spin-wave excitations. Li et al. [8] observed a double-peak resonance in Ni nanowire arrays at 9.7 GHz. The two resonances practically merged for magnetic field parallel to the wires but become well separated when the field diverged from the wire axis. The high-field resonance was explained by the uniform precession mode and the low-field one by the complicated domain structure in nonsaturated wires. Yalcin's group [9–11] investigated Co and Ni nanowires arrays at X-band frequency. They have shown that for some orientations of magnetic field well distinguished peak appears on the high field side of the main resonance. It was attributed to the surface spin-wave mode, which was expected to originate from the surface anisotropy of individual wires. Unfortunately no estimation of the surface anisotropy was given.

When interpreting the resonance measurements in the field

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swept mode one should take into account which microwave frequency is used. If the resonance appears at low fields, where the nanowires array is not saturated, the spectrum depends on the magnetic state of the sample, i.e. on its magnetic history. This can usually happen at X-band frequency. Then the resonance conditions derived for saturated array are not applicable. It was well demonstrated by Encinas et al. [14] on Co nanowire array. They used two different microwave frequencies and the full magnetic field cycle. At lower frequency (29 GHz) the maximum absorption appeared in the field range of nonsaturated state. The resonance curves were asymmetric and showed large hysteresis. For higher frequency measurements (37 GHz) the curves were symmetric and the hysteresis was negligible. When interpreting double-peak resonance measured at low microwave frequency in the field swept mode one should be careful.

In this work we investigate ferromagnetic resonance of an array of Co nanowires electrolytically deposited into nanoporous alumina template. Four microwave frequencies from 9.3 to 69 GHz are used for FMR measurements in the field swept mode. Four resonances peaks are observed at higher microwave frequencies. Several different mechanisms which can lead to the multi-peak resonance are discussed.CGS units are used for convenience.

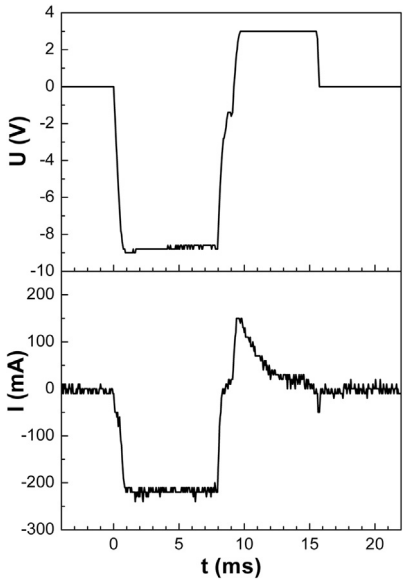

Fig. 1. The typical voltage and current profile of electrodeposition pulse.

2. Experimental

The porous anodic alumina (PAA) templates were used for the preparation of cobalt nanowire arrays. Porous alumina films were prepared by anodic oxidation of high purity (99.995%) aluminum disc 0.5 mm thick and 2.5 cm in diameter. The two-step anodization process in 0.3 M oxalic acid was used to obtain a hexagonal close packed array of pores with diameter about 40 nm and distance about 105 nm [15]. Prior anodization the Al target was electropolished for 2 min at voltage of 20 V and temperature about 10 °C in 1:4 vol solution of perchloric acid and ethanol. The anodization was carried out at constant voltage of 40 V and the temperature 2.5 °C. After the first 24 h anodization the aluminum oxide was removed by etching for 4 h at 60 °C in the aqueous solution of phosphoric and chromic acids. The second anodization was done under the same conditions for 4 h in order to obtain nanoporous alumina film about 10 μm thick. To thin the barrier at the bottom of the pores at the end of the second anodization the voltage was sequentially reduced by a factor $1/\sqrt{2}$ from 40 to 5 V in 6 steps each with duration about 6 min [16]. Finally the pores were slightly widened by etching for 5 min in 5% aqueous solution of H_3PO_4 at 35 °C.

The electrodeposition of Co nanowires was done in the Watts type electrolyte under the pulsed conditions [17]. The following composition of electrolyte was used: 250g/l $CoSO_4 \cdot 7H_2O$ and 40g/l H_3BO_3 in H_2O [18]. The pH of the solution was 3.6. The deposition cell with the Al disc as the cathode and a Pt wire mesh as the anode was used. The rectangular pulse signals (Fig. 1) are provided by the Keithley 2400C SourceMeter. The metal deposition at the pore tip occurs during the first negative 8ms pulse with constant current density of -70mA/cm^2. The second positive pulse of constant voltage +3V and 6ms duration is used to discharge the capacitance of the barrier layer at the pore bottom. The delay time of 0.7s between the pulses enables the recovery of ion concentration and the diffusion of generated hydrogen from the metal – electrolyte interface. A chain of 8000 pulses was used to fill about 50% of the pore length. The initial temperature of electrolyte 30°C increased by about 2° during the deposition. For the preparation of porous alumina templates and the electrodeposition of nanowires self-made equipment was built. The temperature of aluminum target and the electrolytic deposition were controlled by a LabVIEW program.

The samples were analyzed by the scanning electron microscopy (SEM) and X-ray diffraction (XRD). The scanning electron microscope Tescan FERA 3 was used to investigate microstructures of samples. The composition of selected areas was measured by the energy dispersive spectroscopy (EDS) using EDAX Octane 60 mm^2 detector with acceleration voltage 20 kV. The XRD was measured on PANalytical diffractometer X'Pert PRO with the Co radiation ($\lambda = 0.178901$ nm) equipped with texture cradle ATC-3. The point focus of the tube with slits of size $0.25 - 1$ mm was used. Two type of diffraction experiment were done. First, samples were measured in Brag-Brentano geometry for phase identification. Second, since the pole figures show cylindrical symmetry, the crystallite preferred orientation was studied by "psi"-scans.

Hysteresis loops with magnetic field parallel and perpendicular to the sample surface were measured by the vibrating sample magnetometer (VSM). The FMR experiments at frequencies of 9.4 and 34.4 GHz were performed in a Bruker Elexsys E 500 EPR spectrometer. The sample on a rotatable quartz sample holder was placed in the center of the cylindrical TE_{011} cavity. The resonance curves were taken for different angles θ_H of magnetic field to the wire axis. To avoid the heavy loading of the cavity due to eddy currents the Al substrate was removed in the supersaturated solution of Hg_2Cl_2. For the measurements at frequencies 49 and 69 GHz the homemade FMR spectrometer was used. The sample was placed on the sidewall of short-ended rectangular TE_{10} waveguide in the place of maximum RF magnetic field. Two orientations of static magnetic field were used: parallel and perpendicular to the nanowires.

3. Experimental results

3.1. Structure and hysteresis loops

For the scanning electron microscopy the porous alumina film filled with Co was removed from the Al substrate by immersing into the supersaturated solution of Hg_2Cl_2. Fig. 2a shows the top view of the film. An ordered hcp structure of the pores with average diameter about 40 nm and the distance of 108 nm can be seen. In Fig. 2b the cross-section picture observed on the fracture of the film is shown. The light lines correspond to the Co nanowires. The thickness of alumina film is about 8.6 μm. The pores are