Ni-Nanostructures Within Porous Silicon

Investigation of the Morphology by SEM and TEM

A semiconducting/ferromagnetic nanocomposite, consisting of a porosified silicon wafer and deposited Ni-nanostructures, is presented. Porous silicon matrices achieved by anodization of a highly n doped wafer offer oriented pores with an average pore-diameter of 80 nm. Within these pores Ni-nanostructures are deposited resulting in a ferromagnetic system with specific magnetic properties. SEM and TEM investigations are performed to get a correlation between morphology and magnetic behavior.

Introduction

The fabrication of composite materials exhibiting specific magnetic properties which are tuneable by the process parameters are an emerging field in nanotechnology. Due to the nanostructuring of the involved materials the physical properties change drastically compared to their bulk behavior. A popular technique to produce nanometric structures is lithography but also self-assembled and self-organized systems are of interest due to the elementary fabrication process. Quite common are nanoparticles grown on a substrate by self-assembly [1]. But also 3-dimensional arrays of nanowires [2] or nanotubes have been produced without pre-structuring, whereas porous alumina templates, growing in a hexagonal arrangement are widely-used matrices [3]. Magnetic properties of metal filled membranes (e.g. porous alumina, polycarbonate) are under extensive investigation [4, 5]. In this work the electrodeposition of nickel-nanostructures into the pores of self-assembled porous silicon matrices is presented. Not only the investigation of the magnetic properties of the ferromagnetic/semiconducting nanocomposites is of interest but also the correlation with the morphology of the systems. Therefore a careful investigation by structural methods as electron microscopy (SEM, TEM) has been carried out.

Experiments

The fabrication of the composite material is performed electrochemically. The porous silicon matrices are achieved by anodization of a silicon wafer in aqueous hydrofluoric acid solution and subsequently the pores are filled with Ni in a
galvanic process step. The achieved system consists of Ni-nanostructures deposited within the pores, whereas the geometry of the deposits and also the spatial distribution within the pores can be tuned to modify the resulting magnetic properties as coercivity, magnetic remanence and anisotropy [6].

Discussion

Magnetization measurements on the nanocomposite samples are performed by SQUID-magnetometry. Samples prepared by different deposition conditions offer various magnetic properties which is caused by the different geometries and spatial distributions of the Ni-deposits. A correlation between the magnetic properties and the morphology of the nanocomposite can be shown by SEM and TEM. The characterization of the porous silicon matrices regarding the self-assembled pore-arrangement, porosity and pore-size as well as of the metal filled specimens with respect to the geometry and spatial distribution of the precipitated Ni-nanostructures is mainly carried out by SEM.

Figure 2a and 2b show a cross-sectional survey of the porous silicon layer exhibiting straight pores grown perpendicular to the surface as well as a top-view image of the same sample offering an average pore-diameter of 80 nm and a mean pore-distance of 40 nm. The porosity of the porous silicon structure has been estimated by analysis of the top-view image by image processing leading to a value of about 60% porosity. The precipitated Ni-structures are also investigated by SEM in using the back scattered electrons to get element-sensitive information.

Figure 3 shows two samples, one with precipitated sphere-like Ni-particles (a) and the other one with deposited Ni-wires (b). The magnetic behavior differs in the coercive fields HC which can be seen in figure 4. The sample containing mainly Ni-
particles as well as the sample with a high amount of deposited Ni-wires offer a temperature dependency of HC which decreases with increasing temperature (fig. 4). The coercivities of the samples with deposited Ni-wires exhibit always smaller coercivities due to demagnetizing effects of the elongated structures. Furthermore the samples are characterized by TEM, especially to get knowledge about the interface between the silicon skeleton and the deposited Ni-structures. Such investigations offer that the pore-walls are covered by a native oxide layer of about 3 nm which arises due to aging of the matrices in ambient air after their preparation. These results are evidenced by FTIR-spectroscopy measurements which also show the presence of silicon oxide in the case of aged porous silicon. HRTEM investigations of individual Ni-particles within the pores (fig. 5) also show the presence of oxide, whereas neither Raman-spectroscopy of the same samples show any NiO inside the pores, nor magnetization measurements show any exchange bias effects. Therefore we can assume that the oxidation of the Ni-structures inside the pores likely arises during the FIB preparation of the membrane for TEM-investigations.

Summary

In conclusion one can say that structural methods such as SEM and TEM are effective to investigate the morphology of the presented semiconducting/ferromagnetic nanocomposite. Together with these results a correlation between the structure and the magnetic behavior of the specimens can be figured out.

Acknowledgements

This work is funded by the Austrian Science Fund FWF under Project P21155.

References


Authors

Mag. Dr. Petra Granitzer
Dr. Klemens Rumpf
Karl Franzens University Graz
Graz, Austria
http://nanooptics.uni-graz.at/

Dr. Mihaela Albu
Univ.-Doz. Dr. Peter Pölt
University of Technology Graz
Institute for Electron Microscopy FELMI-ZFE
Graz, Austria
www.felmi-zfe.tugraz.at/

Contact

Universität Graz
Universitätsplatz 3 -5
8010 Graz
Austria