Granulated media for nanoelectronic applications

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Abstract. We report our experimental results on the structural and magnetic characteristics of nickel films electrodeposited on n-type (111) silicon substrates. Using scanning electron microscopy (SEM), magnetic force microscopy (MFM) and magnetic hysteresis loops, we indeed find a nominal thickness for transition from island to continuous film before which the magnetization of nanoparticles of Ni is represented by vortex states. The evidence of non-uniform ground states in Ni granules has been proven by micromagnetic simulations. After the formation of a continuous film, stripe magnetic domains are formed with spontaneous in-plane magnetization.

1. Introduction
An investigation of magnetic nanostructures is interested from both fundamental and practical points of view. The possible magnetic configurations in such structures are vortex state or single domain state which are stable for a long time in the wide range of applied magnetic fields. Unique physical properties of magnetic nanostructures make them suitable to be used as magnetic recording media [1,2] and logic elements [3,4]. It is well known that magnetic configuration in a nanostructure depends not only on material properties, but on its shape. A lot of studies focus on 2D structures with different geometry, for instance, discs, rings, ellipses and etc. However, one can modify micromagnetic structure and magnetization reversal with manipulating lateral parameters of 3D nanostructures [5,6].

A cheap and effective method for fabrication of magnetic nanostructures based on “bottom-up” approach is electrodeposition of metals in an appropriate electrolyte under an adequate electrical potential between two immersing electrodes. Advantages of the method include fast growth, highly homogeneous structure and good adhesion of material to substrate. Magnetic properties of thin nickel films have been studied on the metal substrates [7-9] and little information is present for electrodeposited films on silicon [10, 11] and GaAs [12, 13].

In this paper we report on an investigation of granular Ni films with different nominal thickness electrodeposited on Si(111) substrate. The deployment of silicon wafer as a substrate allowed excepting any seed layer. We show an evolution of micromagnetic structure and magnetic properties at the crossover from island-like to continuous Ni films.
2. Experimental procedures

Electrodeposition was performed in a conventional three-electrode cell connected to a computer-controlled potentiostat/galvanostat instrument. To fabricate nickel thin films on n-Si(111), the electrochemical deposition was performed in potentiostatic mode using the chronoamperometry technique. Electrodeposition of nickel was carried out in electrolyte with concentration (mole per 1 lit H2O): NiSO4.6H2O = 2.3, NiCl2.6H2O = 0.6, H3BO3 = 0.5 and pH = 2.

Scanning electron microscopy (SEM Supra, Carl Zeiss) and atomic force microscopy (Ntegra Aura, NT-MDT) were used to examine the morphology and structure of Ni electrodeposited on n-Si. Micromagnetic structure was investigated by magnetic force microscopy (MFM, Ntegra Aura, NT-MDT) with variable in-plane field (±1 kOe). MFM images were obtained using CoCr-coated tips (NSG01). Typical scan heights were about 100 nm. The magnetization reversal processes were studied using magneto-optical Kerr effect (NanoMOKE II system). Magnetic field (±3 kOe) was applied in-plane direction of samples. We employed MagPar software [14] for micromagnetic simulation work.

3. Results and discussions

In order to understand the growth behavior, we have examined the sequence of the electrodeposition of nickel from the Watts bath. Figure 1 illustrates SEM micrographs taken during different stages of the electrodeposition characterized by the nominal thickness $h_n$ of the film. It shows that the electrodeposition of nickel starts with the formation of individual Ni granules on silicon at the initial stages of growth, as shown in figure 1(a) for 8 nm thick nickel electrodeposited on Si. As the electrodeposition time increases, the individual Ni granules become bigger in diameter and tend to coalesce in the form of a continuous film (Figure 1b).

![Figure 1. SEM images of granular Ni films with the nominal thickness ($h_n$), of 8 nm (a) and 1000 nm (b).](image)

The AFM and MFM images are represented in figure 2. As seen for the film with $h_n$=8 nm, at zero field (figure 2(c)) and in demagnetization state (figure 2(d)) the individual granules or granules separated by a small gap have an uniform magnetic contrast seen with white or black areas. This confirms the out-of-plane magnetization. In case of chains consisting of a few adjoined granules, the single domain states are formed due to shape anisotropy. At magnetic fields near a saturation state the bipolar contrast is observed.

Provided that the density of granular (number per surface area) increases, they become in contact with each other, forming a cluster. For clusters the multidomain magnetic structure is more preferable. This argument is proofed by MFM image in figures 2(h) and 2(i). There is a stripe domain structure. The domain walls spread on a few granules adjoined in one cluster.

In micromagnetic simulation we took into account two cases: (i) when the granules are separated and (ii) after being adjoined to each other as is in a continuous film. We used standard material parameters for Nickel: $M_s$=484 emu/cm$^3$, $A$=9×10$^{-7}$ erg/cm, magnetocristalline anisotropy constant...
$K_i = -3.4 \times 10^4 \text{erg/cm}^3$ and the damping parameter $\alpha = 0.1$. Granules were represented by oblate spheroids based on the analysis of the SEM and AFM images. The mesh sizes were chosen to be equal to $\Delta x = \Delta y = \Delta z = 12 \text{ nm}$.

**Figure 2.** Surface topography (a, f) and magnetic structure in saturated states (b, g, e, j), without field (c, h) and in demagnetizing state (d, f) of Ni electrodeposits with $h_m = 8\text{nm}$ (a- e) and 500 nm (f - j).

**Figure 3.** Micromagnetic simulation of ground states for domain structure of isolated granules (a) and cluster (b) from top view. $M_{ij}$ is a vector of the in-plane magnetization direction.
Thus, we can conclude that at the $h_n < 50$ nm the magnetization reversal is defined by the displacement of the vortex core to granular edges. In samples with $h_n > 50$ nm the remagnetization process occurs due to domain wall movement. As result, the values of coercive force and remanent magnetization decrease. Further increase in $H_c$ at higher values of $h_n$ is due to the formation of continuous film with structural defects (inter-granular pores) playing role of domain wall pinning centers.

4. Conclusion

The electrodeposition of nickel from Watts bath undergoes two stages including (1) the formation of individual granules and (ii) coalescence of the them into clusters with following growth of a continuous film, below and above a critical nominal thickness 50 nm. The preferable micromagnetic pattern in isolated granules is the vortex state with high value of perpendicular magnetization induced by its spherical shapes. In films with the nominal thickness higher 50 nm we observed stripe domain structure similar to continuous films. Nickel films consisting of granules at vortex ground state are very promising for high density magnetic recording.

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