Ion Beam Assisted Formation of Magnetic Nano-particles

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The creation of Ni and Co nano-particles in silica epilayers using ion-implantation followed by heat treatment is explored. The processes are verified and optimized via the use of transmission electron microscopy and Rutherford backscattering to analyze specimens.

1. Introduction

Magnetic nano-particles embedded in semiconductor and insulating matrices are of interest in the study of fundamental properties such as superparamagnetism, magneto-resistivity and magneto-optics, as well as for technological applications like data storage and spintronics [1-4]. In the cases of magnetic data storage and magneto-logic devices, topographic patterning of magnetic materials is of importance. The use of ion beam techniques, easily adapted to patterning, as part of the fabrication process in such cases is clearly a sensible approach. Ion implantation of metallic ions into an insulating matrix is a powerful technique for the elaboration of nanosized metal particles. These nanosized particles are characterized by novel properties that are significantly different from those of the corresponding bulk phase [1]. With magnetic applications in mind, ion implantation has been used to synthesize Ni, Co and Ni-Co alloy nano-clusters. In this paper we report the preparation and effect of dose on the formation of these magnetic nano-clusters. Three different implanted doses were used in order to study the effect of dose on the formation of these nano-particles.

2. Sample preparation

A 100 nm SiO_2 layer was grown on a Si substrate by sputtering. The samples were then implanted at room temperature by 50 keV Ni and Co ions. Three different doses (1×10^{16}, 3×10^{16} and 6×10^{16} ions/cm^2) of each element were implanted to study the effects of different concentrations in the formation of nano-particles. One sample was implanted with both Ni and Co ions with a total dose of 3×10^{16} ions/cm^2. According to the Monte-Carlo ion-range simulation code SRIM 2007 [5], the average range of Ni and Co ions of energy 50 keV in SiO_2 is 43 nm.

After implantation the samples were annealed at 900 ^oC in nitrogen ambient for one hour. Implantation doses and depth was confirmed using Rutherford back scattering (RBS) using 2 MeV He^+ ions. Cross-sectional transmission electron microscopy was carried out to confirm the production of nano-particles.

3. Results

The net doses obtained from the RBS measurements were similar to those implanted. The RBS data for the Ni (6×10^{16} ion/cm^2) implanted sample is compared to a simulated spectrum in Fig. 1 which also shows that the depth profile of Ni implanted in SiO_2 is Gaussian.

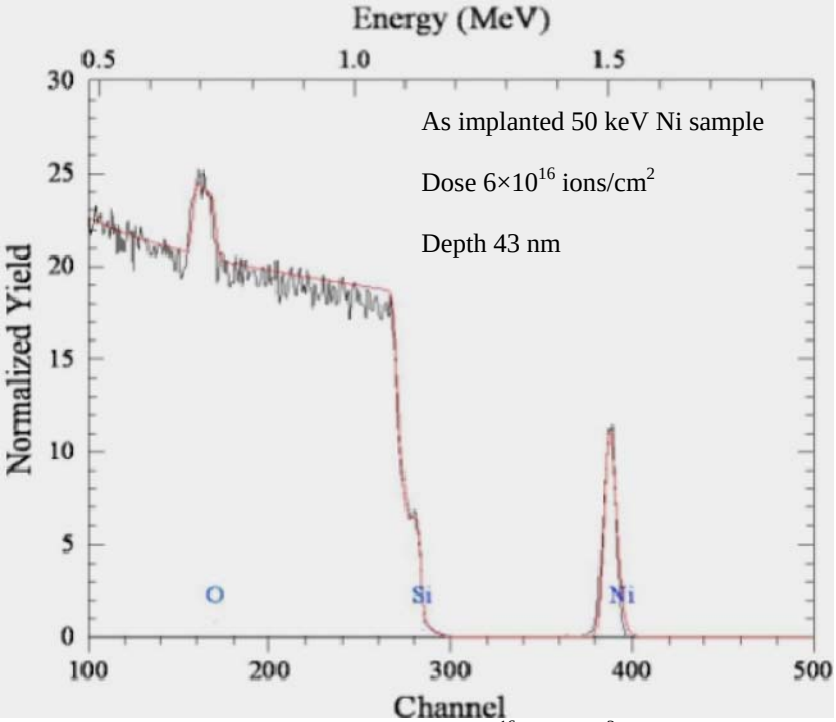


Fig. 1. RBS data and simulated spectrum for Ni (6 x 10^{16} ions/cm^2) implanted into SiO_2.

A systematic structural and compositional investigation was carried out on the samples by TEM. Fig. 2 shows the bright-field, cross-sectional, TEM images for the Ni and Ni-Co implanted samples after one hour annealing in N_2 ambient. In all of the cases investigated, well-defined spherical particles with dimensions in the nanometer range can be observed. These nanoparticles are distributed randomly throughout the sample with the majority of the particles in the centre of the sample. The brightness of the nano-particles varies for different viewing points. That is, by changing the tilt angle, there is a variation in the brightness of these crystals. Such nano-particles are produced as the result of diffusion and nucleation processes which occur due to the annealing. The increased particle size also suggests that the nucleation of particles is occurring [2]. It has been reported earlier [6] that the surface layer is transferred into a highly supersaturated, far from equilibrium state after implantation which relaxes towards equilibrium during annealing. Nano-crystal formation happens by phase separation, where self-organization may occur, therefore they nucleate and grow during ion implantation and subsequent annealing.

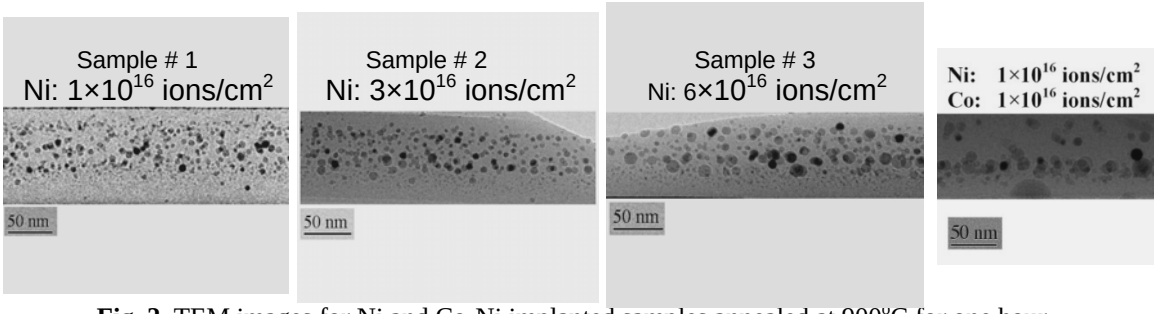
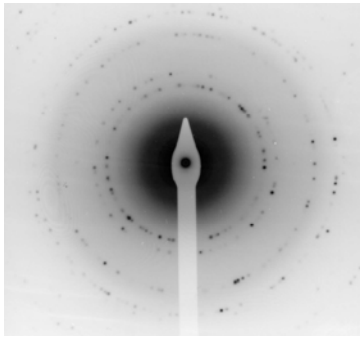
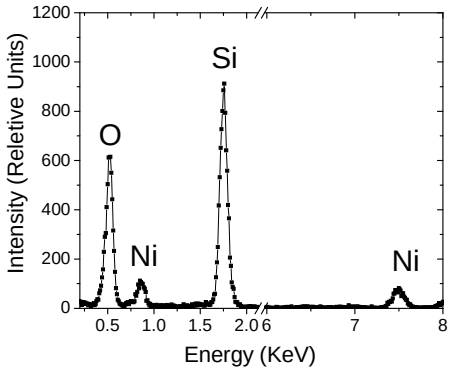


Fig. 2. TEM images for Ni and Co-Ni implanted samples annealed at 900^{o}C for one hour.

We also analyzed the samples with selected area electron diffraction (SAED) to investigate the crystalline structure of the clusters. For all the analyzed compositions, the SAED pattern exhibits Debye-Scherrer rings [2] of a single fcc phase (Fig. 3a). Electron diffraction spectra were also recorded for the confirmation of the composition of nano-

particles. Fig. 3b shows a typical EDX spectrum for the Ni (3×10^{16} ions/cm^2) implanted sample.





(a) SED pattern (b) Ni EDS spectrum

Fig. 3(a). SAED pattern of Ni (3×10^{16} ions/cm^2) implanted sample. Fig. 3(b) Typical EDS spectrum for Ni similar dose sample.

The sizes and size distributions of the nano-particles were obtained by using Image J software. However the particles in a selected area were counted manually, since the large numbers of overlapping nano-particles were not counted accurately by the software automatically. An area from each sample was selected and particles were counted. Fig. 4 shows the sizes and size distributions of all the three different Ni-implanted samples. These sizes and size distributions are strongly dependent on the implant dose. Sample 3, with the highest doses has the largest nano-particles.

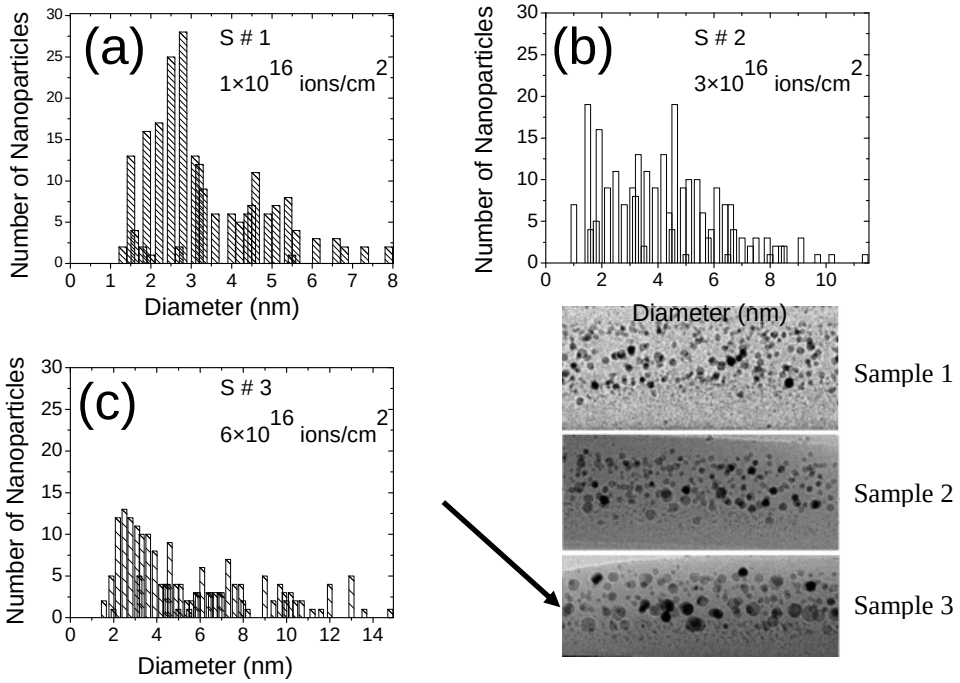


Fig. 4. Size distributions of Ni nano-particles as a function of implanted dose (ions/cm^2): (a) 1×10^{16}, (b) 3×10^{16}, (c) 6×10^{16}.

In order to study the distribution of nano-particles in individual samples, each sample was divided into three equal layers. The total number of nano-particles in each layer was

counted and their diameters measured. Fig. 5 shows the size distributions of Ni nano-particles for a dose of 6×10^{16} ions/cm^2. It was found that these nano-particles are distributed throughout the sample but mainly concentrated in the centre of the sample which, also has the highest number of larger nano-particles. Both effects are because the average depth of the implanted ions was nearly at the centre of the sample (43 nm) therefore the concentration of Ni was highest in the middle of the sample. This also resulted in ripening of larger clusters towards the centre.

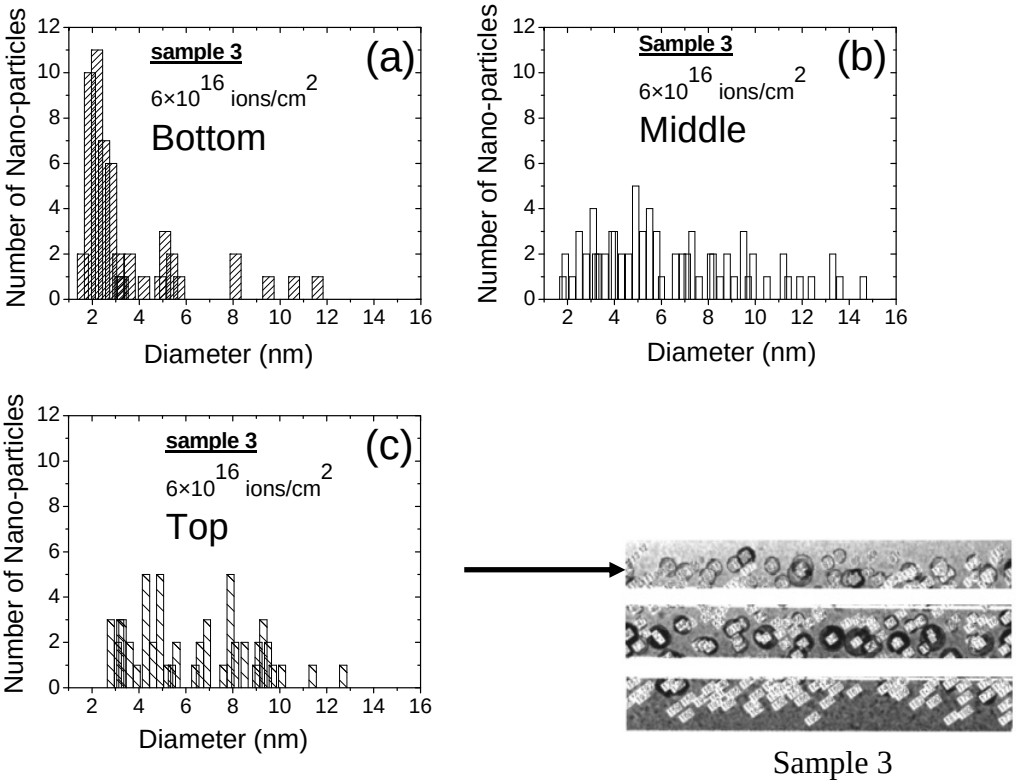


Fig. 5. Ni nano-particle size distribution in three regions (a) bottom, (b) middle and (c) top of the sample 3 6×10^{16} ions/cm^2**.**

4. Conclusions:

Ni, Co and Ni-Co alloy nano-particles were successfully produced using ion implantation. It was found that the size of the nano-particles increased with the implanted dose. The maximum number, and also the largest nano-particles, were found to be in the centre of the sample, which in turn revealed that maximum nucleation occurred at the centre of the sample with less in the top and bottom layers of the sample.

References

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