Fabrication and characterization of cobalt particles by ion-beam induced chemical vapor deposition (IBICVD)

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ABSTRACT

The dot and ring shape of Co particles have been made using IBICVD technique. Results have shown that the shape and size of Co particles depend on the precursor vapor pressure, intensity and dwell time of ion-beam, designed pattern sizes, and nature of substrates. The Co dot shows a mean diameter ~ 3.9 μm and the width of the Co ring is around 1.8 μm. The effective Co thickness is in the order of 10 nm with a low aspect ratio (< 0.1), as revealed by AFM sectional analysis. Fresnel mode Lorentz TEM studies suggest that inside each dot, the Co particles (sizes ranging from 100 nm to 300 nm) exhibit single domain behavior, which has great potentials in high density magnetic recording applications.

Keywords: Magnetic dot arrays, ion-beam induced chemical vapor deposition (IBICVD), AFM, Fresnel image, Lorentz TEM.

I. INTRODUCTION

Nanometer-sized patterned media has been of great interest in high density data storage applications [1,2]. Different fabrication techniques have been studied with its unique physical properties of nanoscale features. Among these, ion-beam induced chemical vapor deposition (IBICVD) technique, combining three independently established techniques (computer-controlled, ion-beam, and CVD) in a single procedure has been successfully demonstrated on fabrication of cobalt particles [3]. Our previous work [4] has shown that these IBICVD dots exhibit ferromagnetic properties. The shape and size of these Co particles depends on the precursor vapor pressure, intensity and dwell time of ion-beam, designed pattern size, and nature of substrates. The aim of this work is to explore the size limitation and to study the morphology and the magnetic structure of submicron cobalt particles by this novel technique.

II. EXPRIMENTAL

The submicron-sized Co particles were deposited on different substrates by focused ion beam system (Hitachi FB 2000A) equipped with a deposition nozzle filled with precursor of octacarbonyl dicobalt, $Co_2(CO)_8$ powders, as shown in Figure 1. The base pressure of the deposition chamber was about 2 × 10^{-5} Pa. The partial pressure of $Co_2(CO)_8$ can be controlled by the heating stage in the deposition nozzle. The unique design of this state-of-the-art IBICVD system [5] can be used for patterning samples or for depositing Co by close or open the deposition nozzle, respectively. The ion current and the pressure during deposition was ~8 pA and ~8 × 10^{-5} Pa, respectively. The in-situ image of as-deposited cobalt particles was taken by secondary electron microscopy of the FIB system. The Digital Instruments (Nanoscope III) atomic force microscopy (AFM) was performed to study the morphology of these Co particles. The Fresnel mode of Lorentz TEM (JEOL 4000FXII, operated at 400 keV) was used to characterize the magnetic structure of these Co particles.

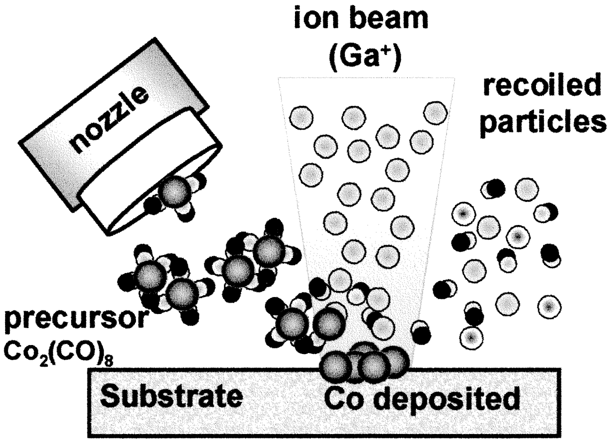

Figure 1. A schematic illustration of fabrication of Co particles by IBICVD technique.

III. RESULTS AND DISCUSSION

Figure 2 shows the SEM image of a periodic submicron-sized Co particles deposited on Si_3N_4 substrate (diameter~ 3 μm) by IBICVD technique. These cobalt particles appear to be uniform with regular shapes. The morphology of Co particles depends on the precursor vapor pressure, intensity

(~ 8 pA) and dwell time (5 ~ 120 μs) of ion-beam, designed pattern size, the nature of substrates, and the scan cycles (1000 ~ 20000) of ion beam. The inset of Figure 2 shows that the Co size can be as small as ~150 nm by optimization of deposition parameters.

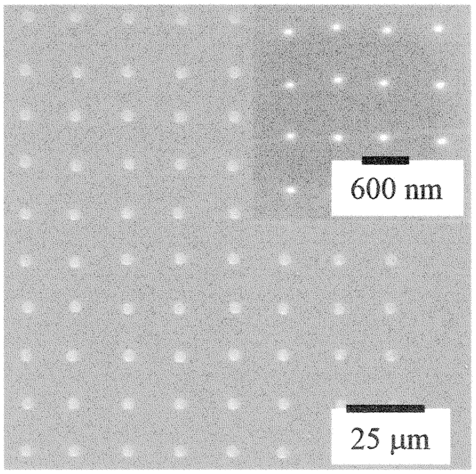

Figure 2 The SEM image of cobalt dots array. Inset shows the smaller Co particles deposited on MgO-coated TEM grid.

To distinguish the fabrication process between deposition and patterning/etching, the valve to the deposition nozzle was closed to stop supplying $Co_2(CO)_8$ precursor. The dark contrast in Figure 3(a) corresponds to the hole (diameter~ 2 μm) etched by energetic (30 keV), focused ion-beam whereas the bright contrast in Figure 3(b) corresponds to the Co particles by the decomposition process of $Co_2(CO)_8$ precursor by the Gallium ions. The dark and light contrast in Figure 3(d) implies the formation of three-dimensional structure. This is likely due to the imaging technique being provided in the FIB system. Compared to the dots structure in Figure 3(b), ring shape of Co particles results from longer scan cycles (20000) and dwell time (T_d~ 120 μs) of ion-beam, as shown in Figure 3(c). Further, the elliptical shape of traces of resputtering effects by ion-beam is seen clearly in this intensive ion-beam deposition condition. This is due to the dominant ion-beam etching over cobalt supply from $Co_2(CO)_8$ vapor. It is effectively improved by increasing the $Co_2(CO)_8$ partial pressure and by reducing the ion-beam current, as shown in Figure 3(d).

Our previous work [4] has shown that these Co particles are not expected to exhibit single domain properties due to their relatively large size (diameter~ 500 nm). In stead, these Co particles reveal multidomain features, as obtained by alternating dark and light contrast by MFM image, with in-plane magnetization (Hc~ 100 Oe, Ms~ 1000 emu/cm^3). The randomly oriented magnetic directions of each Co particle are the consequences of the competition between shape and magnetocrystalline anisotropy [6].

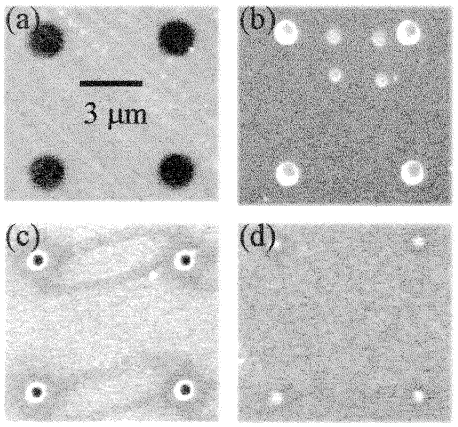

Figure 3. The SEM images of (a) etched holes, (b) Co dots, (c) Co rings, and (d) smaller size of Co dots (d~ 500 nm) on glassy carbon substrates.

AFM observations were carried out in order to study the morphology of these Co particles. Compared to the topographic images, AFM sectional analysis clearly shows the difference between Co particles (inset of Figure 4(a)) and etched patterns (inset of Figure 4(b)) on Si_3N_4 substrates. The corresponding SEM image of Co particles gives little information in the detailed features inside the dot, as shown in Figure 4(c). These Co particles exhibit a 3.9 μm mean diameter (peak-to-peak distance) with height of ~20 nm. It is likely that the width of the ring (~ 1.8 μm) consists of both Co from decomposition process of $Co_2(CO)_8$ precursor as well as from resputtering impurities from Si_3N_4 substrate. After background subtraction (by

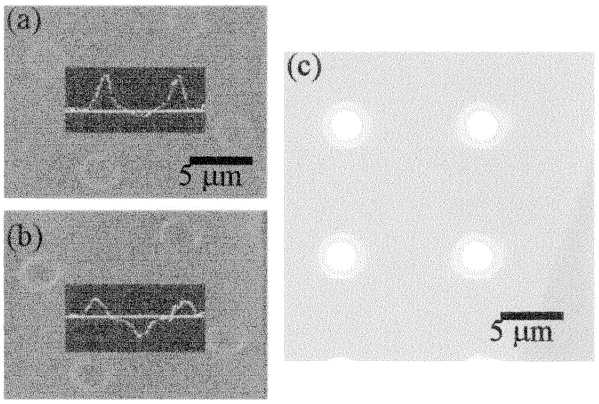

Figure 4. The AFM images of (a) Co particles and (b) etched patterns on Si_3N_4 substrate. Figure 4(c) is corresponding SEM image in Figure 4(a).

comparing the difference in shape and size of these two curves in inset of Figure 4), it seems that the major Co signal (in height) is in the range of ~10 nm and ~8 nm for the ring and dot regions, respectively. This indicates that the dot and ring shape of Co particles formed with this experimental condition by this unique IBICVD technique, although the distribution of Co particles and its mixing with Si_3N_4 substrate remain unclear due to the limited resolution of MFM, which is about 100 nm [7], depending on the sample preparation. Therefore, it is not surprised that the measured MFM image (not shown here) exhibits the featureless contrast. Lorentz TEM, which yields higher depth resolution, helps to identify the detailed features inside the dot.

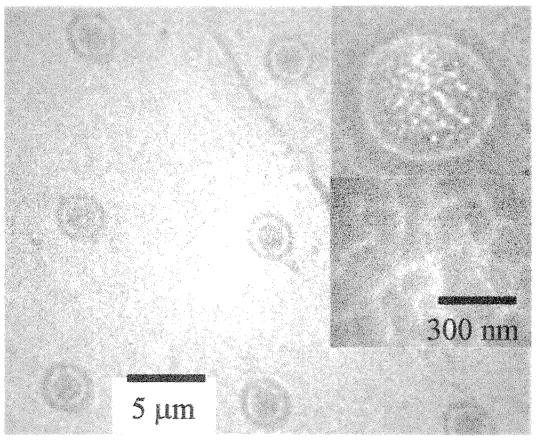

Figure 5. The Fresnel Lorentz TEM image of Co particles. Inset at upper and lower part shows the enlarged image of one particle and detailed features in the center, respectively.

While the MFM is sensitive to the second derivative of the stray field, the Lorentz TEM is sensitive to the magnetization inside the samples [8]. Figure 5 shows the LTEM Fresnel image (defocused distance~ 100 μm) of Co particles fabricated on TEM grid. The Co particles consist of ring and dot (diameter ~ 2.6 μm) in the outer and center region, respectively. The dark and light contrast of ring shape of Co with roughly equal width of ~ 370 nm (not resolvable by SEM or MFM techniques) were clearly revealed by utilizing the Fresnel imaging technique (inset of Figure 5 at upper right part). In Fresnel mode of LTEM, the domain walls appears as dark or light contrast due to the spatially varying component of the in-plane magnetic induction [9]. Further, the Fresnel contrast of the center region consists of Co particles with sizes ranging from 100 nm to 300 nm (inset of Figure 5 at lower right part). Chou et al. [10] have shown by nanolithography technique that ring shape of Co particles with diameters ≤ 500 nm exhibits single domain behavior. Further, Cowburn [11] has systematically studied the particle size as a function of thickness and found that the diameter where single domain particles forms increases as thickness decreases from 3-D (bulk) approaching to 2-D. The bulk Co has a critical diameter ($2R_c$) ~ 54 nm, estimated from $R_c \leq 9\sigma_w/4\pi M_s^2$, where σ_w (8 erg/cm^2) and M_s (1440 emu/cm^3) are domain wall energy ($\sim 4(Ak_u)^{1/2}$) and saturation magnetization of Co, respectively. The reduced M_s due to defects or impurities by IBICVD also gives rise to the bigger particle size, which still exhibits single domain behavior, as measured by TEM. Recently, Ross et al. [12] have reported the calculated relation between in-plane/out-of-plane single domain and vortex (or multidomain) behaviors as a function of aspect ratio (height/diameter) and diameter. The low aspect ratio (< 0.1) and small particle size (~ 200 nm) lead us to conclude that these Co particles exhibit single domain behavior. This further implies that the fabricated Co particles with diameter ~ 150 nm (inset of Figure 2) serve as great potentials in high density magnetic recording applications. Further studies on switching behavior of these Co particles by utilizing synchrotron radiation technique are in progress.

IV. CONCLUSIONS

The submicron-sized Co particles (diameters ranging from 100 nm to 4 μm) have been successfully fabricated by IBICVD technique. Under optimization of fabrication parameters, the dot and ring shape of Co particles were made, as revealed by AFM and LTEM. The low aspect ratio (< 0.1) as well as small particle size (~ 200 nm) suggest that within each dot, these Co particles exhibit single domain behavior.

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REFERENCES

[1] M. Hehn et. al., Science 272, 1782, 1996.

[2] B. D. Terris et. al., Appl. Phys. Lett. 75, 403, 1999.

[3] A. Lapicki, E. Ahmad, and T. Suzuki, J. Magn. Magn. Mat. 240, 47, 2002.

[4] A. Lapicki, K. Kang, and T. Suzuki, IEEE Trans. Magn., in press.

[5] T. Suzuki and A. Lapicki, Jpn. Patent # 2001-142476, 2001.

[6] E. F. Wassermann, M. Thielen, S. Kirsch, A. Pollman, H. Weinforth, and A. Carl, J. Appl. Phys. 83, 1753, 1998.

[7] A. Hubert and R. Schafer, Magnetic Domains: The Analysis of Magnetic Microstructures, 105, Springer-Verlag Berlin-Heidelberg, 2000.

[8] E. N. Abarra, I. Okamoto, and T. Suzuki, J. Appl. Phys. 85, 5015, 1999.

[9] J. N. Chapman, J. Phys. D: Appl. Phys. 17, 623, 1984.

[10] S. Y. Chou, P. R. Krauss, and L. Kong, J. Appl. Phys. 79, 6101, 1996.

[11] R. P. Cowburn, J. Phys. D: Appl. Phys. 33, R1, 2000.

[12] C. A. Ross, M. Huang, M. Shima, J. Y. Cheng, M. Farhoud, T. A. Savas, H. I. Smith, W. Schwarzacher, F. M. Ross, M. Redjdal, and F. B. Humphrey, Phys. Rev. B. 65, 144417, 2002.