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Ion beam induced chemical vapor deposition (IBICVD) of cobalt particles

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Abstract

A novel method aiming at fabricating submicron-scale particles utilizing dicobalt octacarbonyl as a precursor for cobalt, formed upon localized ion beam induced decomposition, is presented. Patterns of deposited particles are fabricated through vector scan rastering. Measurements of cumulative magnetic properties (arrays of $2 \mu m$ -size dots) show coercivity of about 100 Oe and the saturation magnetization of approximately 1000 emu/cm³. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

Patterned media for high density data storage with nanoscale dots have recently attracted a lot of attention and a "one dot-one bit" scheme has been suggested [1-3]. However, before a mass scale patterning technology can be successfully implemented, all possible prototypes should be studied with respect to both physical properties of nanoscale features and the effects introduced by chosen fabrication techniques [4-7]. Consequentially, it is advisable to choose a research method for generating such patterns that would allow easy variation of some fabricating parameters corresponding to different properties of the material. The technique, hereby, proposed and investigated, is ion beam induced chemical vapor deposition (IBICVD) which is a variation of the ion beam-assisted deposition. Novelty of this method has been constituted by combining three independently established techniques in a single procedure. Octacarbonyl dicobalt $(Co_2(CO)_8)$ has been successfully used for the deposition of metallic cobalt via thermal activation [8,9]. Ion beams have been used for depositions of other

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metals [10] and computer-controlled ion beam processing has been a known scheme in ion beam lithography [11,12]. The combination of those three, in a single procedure, has not been reported yet.

2. Experimental

All deposition experiments are performed by means of a commercial focused ion beam (FIB) system featuring liquid gallium ion source (Hitachi FB2000A). Co₂(CO)₈ is used as a precursor for metallic cobalt particles. The vapor pressure required for the deposition process is obtainable at $28-30^{\circ}$ C. It corresponds to the chamber pressure of $5-7 \times 10^{-5}$ Pa measured by the ion pump, but its value at the deposition spot must be 2–5 times higher. Higher pressure could be beneficial for the reaction efficiency, but it is not acceptable for both the ion microscopy detector and the pumping system used for all experiments. Almost all carbonyl and some organometallic complexes can be accommodated for such deposition schemes.

The energy necessary for activation of the decomposition process is provided by means of the gallium ion beam and defined by the acceleration voltage of 30 kV. This value is required for the operational feasibility of the liquid metal ion source and the conditions of

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ultimate focusing. The focusability is also the factor determining the selection of possibly lowest controllable ion current (2–4 pA). Such conditions are important if a high resolution of deposited patterns is desired. Using argon or other noble gas ion beams might be advisable if gallium contamination becomes critical. Any pattern (although limited in size) of deposited particles can be fabricated if properly designed and processed through the vector scan rastering scheme supported by software provided with the apparatus.

The deposition occurs at a region of overlapping molecular and ion beams. It has been suggested that mechanisms of such IBICVD processes [10] are better described in terms of synergetic effects of several phenomena than as a simple model. So far nobody has been able to quantitatively model even individual aspects of the problem as, for example, the deposition rate. The first step of the process is energy exchange between highly energetic gallium ions and almost stationary (thermal energy distribution) carbonyl complex molecules. The energy of 30 keV is rather excessive in terms of thermodynamics so it is rational to look for other mechanisms than those modeling thermal decomposition. Among several energy exchange pathways like electromagnetic radiation, electronic excitation and atomic collisions, the latter one should be dominating in the experimental conditions under considerations. For the same reason (high energy, heavy species), it may be sufficiently adequate to describe the collision interactions between the gallium ions and precursor molecules in terms of a hard sphere model. Dynamics of such approximation can be presented as an instant and complete momentum transfer from the ion moving with a velocity of about 300 000 m/s to a heavier molecule. After the collision, the gallium ion/atom is furnished with only thermal-scale velocity oriented in a random direction. The precursor molecule will gain a significant momentum towards the surface and an explosive decomposition will be initiated. It is rather hard to predict the exact time sequence and velocity distribution among cobalt atoms and CO molecules (some may also undergo decomposition). The recoiled species will probably need several nanoseconds to reach the surface which is significantly a longer time than the periodicity of intramolecular vibrations, and therefore, it is rather safe to assume that the decomposition process of the precursor molecule is quite advanced at the moment of the second collision, this time with the surface. However, the presence of even unbound carbonyl radicals still moving almost together with cobalt atoms has probably a cushioning effect and subsurface penetration of cobalt is rather limited. The impact energy is being dissipated over neighboring atoms through enhanced lattice vibrations (local heating) and this will certainly finalize the dissociation process. The lighter carbonyl species as well as carbon or oxygen atoms have a higher probability of

backscattering than cobalt. In addition, those carbonyls that do get trapped in surface layers still have a chance to desorb due to intensive thermal activation. The explosive character of decomposition certainly contributes to a lower ultimate resolution of deposited patterns in comparison with the FIB focusing limit. Secondary processes such as thermally activated surface and bulk diffusion (resulting either in enhanced segregation or mixing), possible defect cascades within the substrate lattice and formation of complex defects, all work against the sharply defined patterns. Charging effects and mechanical vibrations are also important factors but can be minimized. Problems of local overheating (promoting some undesirable secondary effects) can be minimized when the fabrication procedure is performed as a series of rather quick scanning cycles over a larger pattern instead of sequential (serial) deposition of individual dots. The satisfactory results were obtained by a combination of 5-point dot definition, 128 µs dwell time and 60 000 cycles for an array of 100 dots. The total time of such an experiment was about 40 min. The deposited particles can be as small as about 150-200 nm when deposited on a smooth surface like MgO(100) substrate (it has been confirmed for the deposition of tungsten), but were larger in the presented case of Collodion membranes (1.5–2.5 µm).

3. Results and discussion

General topographic appearance of deposited islands was verified in situ by ion beam microscopy (secondary electron imaging, SEM) as shown in Fig. 1. Magnetic



Fig. 1. Ion beam microscopy (SEM) of cobalt dots deposited by IBICVD onto Collodion membrane. The inset shows remanent states after saturated magnetization in two opposite directions parallel to the surface plane, measured by MFM. Magnetic field strength applied is 2000 Oe.



Fig. 2. Hysteresis loops measured by AGFM with the magnetic field applied in two directions with respect to the substrate surface plane: parallel and perpendicular. Magnetic field strength applied ranged from -15 kOe to 15 kOe, but only the range from -6000 to 6000 Oe is shown.

properties of samples featuring multiple arrays of particles deposited onto Collodion membranes coating copper mesh were examined by alternate gradient force magnetometer. Measurements were conducted both in perpendicular and parallel directions with respect to the surface plane. Results show in both cases low coercivity (approximately 100 Oe) as presented in Fig. 2, and saturation magnetization of about 1000 emu/cm³. The latter was estimated taking into account the shape of the deposited islands determined from subsequent AFM (atomic force microscopy) measurements (diameter of $2.0\pm0.5\,\mu\text{m}$, height of $0.10\pm0.05\,\mu\text{m}$) and the total number of successfully fabricated particles (about 1000). The magnetization in the surface plane appears saturated at a slightly lower value of the magnetic field, as shown in the hysteresis curves in Fig. 2.

The obtained patterns were also examined by magnetic force microscopy (MFM). In order to clarify the magnetic behavior, experiments attempting switching (a permanent magnet with the field strength of 2 kOe was employed) were performed for the perpendicular and parallel directions of the magnetic field with respect to the sample plane. Switching in the surface plane can be observed for at least some dots, as demonstrated in the inset of Fig. 1, where the contrast is reversed with the polarity of the applied field.

It is concluded that IBICVD can produce magnetic dots and patterns valuable for research purposes. Many aspects of the fabrication procedure need further optimization that will be a subject of future investigation. The principles of the method may be potentially expanded towards more technologically oriented applications if appropriate instrumentation is developed.

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