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Corrosion Characterization of High Moment Magnetic Material Coated with Diamond-like Carbon

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Abstract. In order to increase data storage density of hard drive, DLC overcoat thickness was decreased to allow read/write head to come closer to magnetic alloy of the disk, andwith maintaining the main purpose of the overcoat to provide good corrosion and mechanical protection for the underlying magnetic recording film under unfavorably environmental conditions and occasional reactions. Base on that the edges of topography will be weak point for deposition films cause of shadow effect during the process, it's hard that deposition atomic will stay at the concave and convex of topography. DLC coverage performance at the edge of 3D topography becomes high sensitive with limited DLC films thickness. Currently the 3D profile of magnetic material that needs to protect with DLC, is about 2 nanometers with subnanometres DLC thickness. The enormous of data shrew corrosion pattern was usually happened at the edge of topography. Thus, the accelerated test or indirect methodology was applied for DLC integrity. This investigation was explore the methodology to verify the weakness of DLC films especially at the topography edges which is difficult for any direct metrology tools can pursue. With pore resistance, which related to the film structure, the electrochemical impedance indicated that DLC/Si3N4 is a suitable choice to against corrosion. However topography surface influence to DLC coverage. Controllable surface for DLC deposition also needed well defined. Two types of step height was created as 1 nm and 2 nm on (100) silicon substrate. The 20Å DLC film thickness was deposited on the silicon substrate with promised technique, Filtered Cathodic Vacuum Arch (FCVA). After this process the aluminum (Al) was deposited on the surface of DLC film by using evaporation technique. The silicon substrate was driven to Al surface thro' pin holes on the DLC film at 577OC. To detect the pin holes on the DLC film, the aluminum layer was removed by using wet etch chemical process. The SEM image indicates that the square pitting at the edge of DLC film obtained for 2nm step height was around 2nm depth. The latest experiment in this investigation to characterize 2.3 T magnetic moment material degradation with DLC/Si3N4 coating, was performed with electrochemical impedance spectroscopy and AFM. The material with DLC overcoat after exposed to H2SO4 for 30 min, polarization resistance was increased for 2 times from uncoated material. Alternative charge transfer capacitance was reduced as desirable charge current.

Introduction

Demands for inexpensive and small hard disk drives (HDD) have driven the drastic increase in recording density. Reduction of magnetic spacing, which is the distance between magnetic head and media, has been one of the most important contributions made to support the increase in recording density. To achieve a 1 Tb/inch^2 system, magnetic spacing must be as small as 6.5 nm, where overcoats, lubricant, surface roughness, and real physical clearance must be packed [1]. The reduction of each component of magnetic spacing, such as head and disk overcoats, is essential to achieve magnetic spacing of 6.5 nm. The thickness of head and disk overcoats must thus be less than 3 nm by scaling accordingly to achieve 1 Tb/inch^2. To reduce thickness of head and disk overcoat, several difficulties have to be overcome. Head and disk overcoats play an important role in corrosion-

and wear-resistance. The reduction of the overcoat thickness will degrade their corrosion- and wear-resistance. Corrosion resistance depends on the chemical stability of the overcoat material and its coverage on the surface. With decreasing thickness of hard overcoat material, effective coverage of a film-substrate composite system decreases, which may degrade corrosion resistance.

DLC films have long been employed as head and disk overcoats due to their excellent chemical stability, physical integrity and mechanical hardness. To reduce overcoat thickness without sacrificing corrosion- and wear-resistance, the efforts have been devoted to increase density [4] and coverage performance. To achieve a 1 Tb/inch^2 system, thickness of overcoats must be reduced down to as thin as less than 2 nm. However, there have been several studies to report that the density of DLC films becomes smaller when the thickness becomes as thin as a few nanometers even with FCVA [5], [6]. No promising technology candidates have been found so far to further increase the density of DLC films to replace filtered cathodic vacuum arc (FCVA). For 1 Tb/inch^2, it is highly required to assess how much influence the reduction of overcoat thickness will have on corrosion and wear-resistance. Regarding to the edges of topography are most likely the weak point for film deposition that causes shadow effect during the process, consequently it is difficult for atomic deposition to stay at the concave and convex of topography. The purpose of this paper is focus on DLC coverage performance on 2.3T high moment materials (CoNiFe) since corrosion at the edges always found. The evaluation is composed of two studied steps, surface profile generating and followed with thin DLC deposition to verify influence of surface topography to its coverage. The fixed DLC films thickness coated on substrate will use for electrochemical impedance spectroscopy (EIS) to define corrosion behavior is the latest step.

Material and Method

Surface topography generation. The investigation was developed for new thin DLC generation, especially to serve read-write magnetic industry. Surface profile on the study must match to the right profile of the industry. Atomic force microscope (AFM) is tool to evaluate the actual profile of read-write head. Fig. 1 shows step height of actual part before DLC coating. Clearly observed 1 nm and 2 nm are the profiles that need to protect with noble material, DLC.

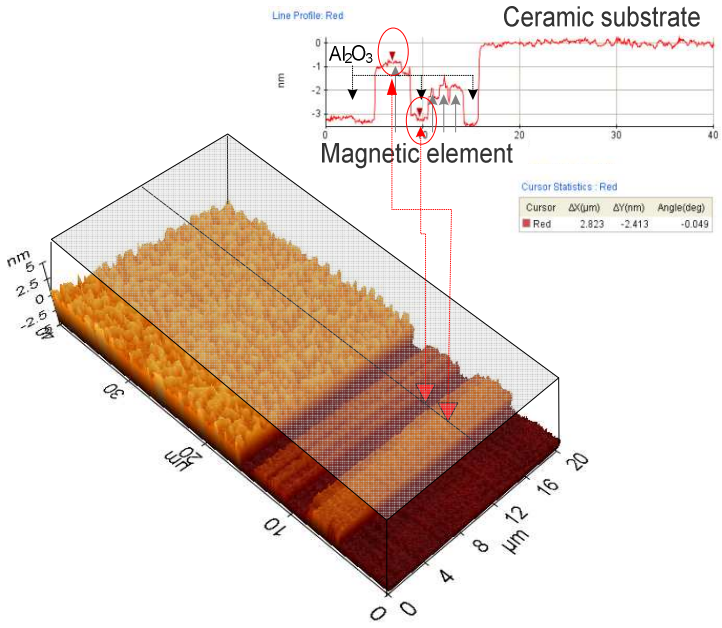

Fig. 1 Actual step profile of read-write magnetic head.

Due to magnetic element of read-write head was deposited over the alumina (Al_2O_3) on top of ceramic material (Al_2O_3 and TiC sintering composition). At starting point of investigation we will reduce the variation of complicated material by selected only one element to generate the surface profile followed the actual part in Fig. 1. The promising element for the evaluation is Si (100). Step

patterning is created on the cleaned silicon surface. Ion bombardment is the method to create step of silicon which have no protoresist protection by Ar+ plasma. Photoresist will be washed away after finishing step creating.

DLC deposition. Dense films were deposited with well-known technique [5, 6], filtered cathodic vacuum arc (FCVA) machine for 2 nm DLC thicknesses. Si_3N_4 target was sputtered and formed a-Si_3N_4 seed adhesion layer between substrate and final DLC films. The coating film is demonstrated as a single homogeneous layer with DLC embedded to Si_3N_4 seed layer from physical TEM image. [7]

Aluminum deposition and Diffusion driving. The sample was coated with aluminum by evaporation technique as shown in Fig. 2. With temperature 577^{O}C for sintering, silicon was driven to diffuse to aluminum layer as phase diagram in Fig. 3.

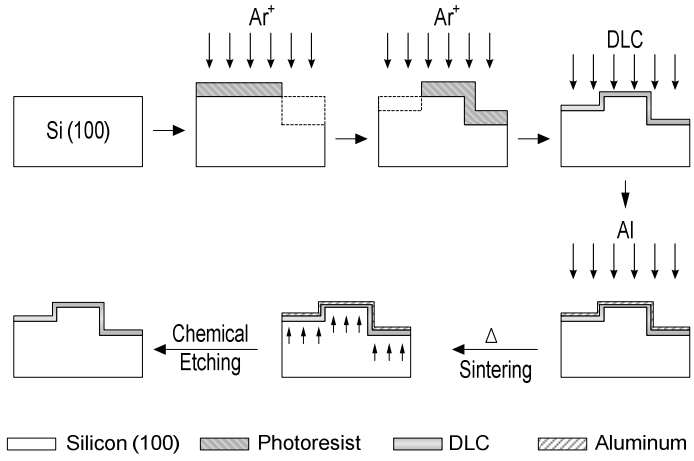

Fig. 2 Methodology for DLC coverage weak point verification

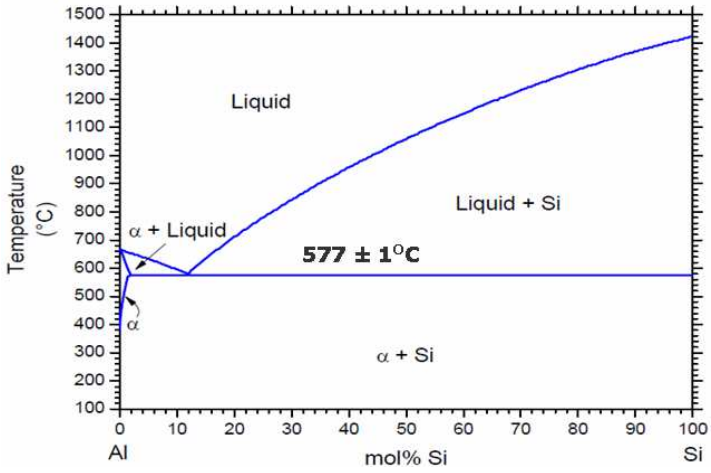

Fig. 3 Phase Diagram for sintering temperature selection [7]

Wet etching. Finally, aluminum was etched out with H_3PO_4 : DI water : HNO_3 : CH_3COOH (16:2:1:1) for 3 minutes. In this step if Si was diffused to Aluminum via DLC pin hole, it will be easily removed at the same time as aluminum.

Physical Analysis. Selected tools for experimental analysis were scanning electron microscope (SEM) Jeol, 6340 and atomic force microscope (AFM) Digital Instrument, model D5000. Surface morphology changing was detected with SEM and using AFM to analyze the depth of the holes.

Electrochemical Impedance Analysis. The general effectiveness of DLC on corrosion protection was determined by potentiostatic polarization measurements [1,2,4] with data interpretation by the Tafel extrapolation method. From this portion magnetic alloy as a real read-write head material was used for corrosion analysis to represent actual corrosion behavior in process. The electrochemical cell consists of 2.3T CoNiFe/ Si coated with fixed 2 nm DLC thickness as the working electrode, Ag/AgCl electrode as reference electrode and a platinum foil as counter electrode. Before the scan

was initiated, the sample was allowed to stabilized in 0.5M H_2SO_4 with N_2 de-aerated for 30 minutes. A potential scan rate of 0.002 $V.s^{-1}$ was used. The range of potential utilized in the corrosion tests ± 0.2 V with related to open circuit potential (OCP). EIS experiments were conducted at the OCP in AC current mode by using excitation signal amplitude 5 mV over frequency range 0.1 – 10^5 Hz. Potentiostat/Galvanostat instrument for the test is Autolab PGSTAT100.

Result

SEM and AFM at the intent 2nm step were as Fig.4. Square hole was clearly explicated along the step. However the holes don't appear at the edge exactly, seem like have some distance from the convex of the step because DLC films were precipitated along the wall of intent step more than flat area.The evidence can explain by following AFM image. The thicker DLC can cause the shadow effect at the top of the step and prevent the diffusion of Si to aluminum layer. AFM show the depth of hole is about 2 nm. For intent 1 nm step, the image of SEM and AFM were clear, surface still smooth without detected holes.

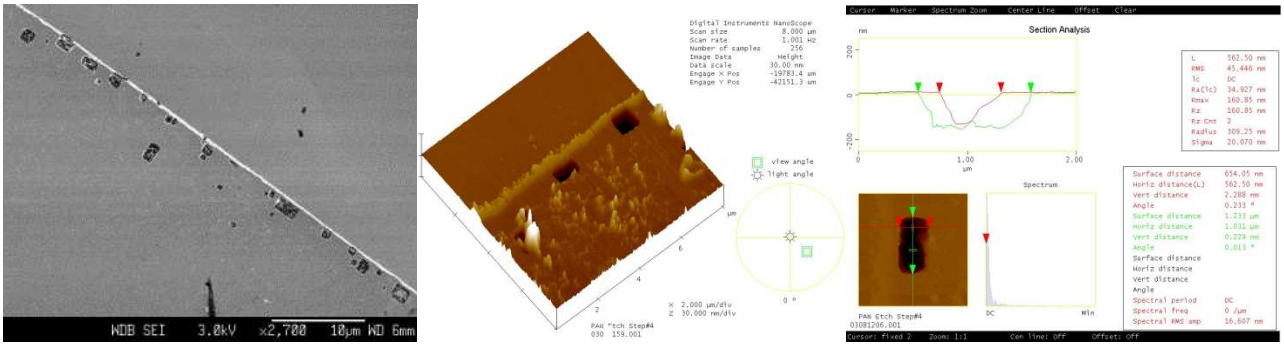

Fig.4SEM images andAFM images at edge of 2 nm step height with 2nm DLC thickness.

Fig.5presents potentiodynamic polarization curves of uncoated and DLC coated CoNiFe alloy in 0.5 M H_2SO_4 solution. The presence of DLC coating on CoNiFe lowered the current density and increased the corrosion potential. EIS Bode plots determined by the EIS technique, in Fig.6, represent the different corrosion behavior of the bared and DLC-Coated CoNiFe sample. DLC-Coated CoNiFe films present superior impedance in comparison to bared CoNiFe. The EIS response of the sample in H_2SO_4 solution is modeled by equivalent circuits as Fig.7The meaning of electronic elements in Fig.7 are as followed: R_s is ohmic resistance, associated with the electrolyte solution, was omitted from Table.1. RE and WE stand for reference and working electrode. C_{mf} and R_{mf}correspond to capacitance and resistance of metal/solution interface. C_c and C_{dl} are capacitance of film and double layer, respectively. R_{pore} and R_{ct}represent pore and charge transfer resistance, respectively.

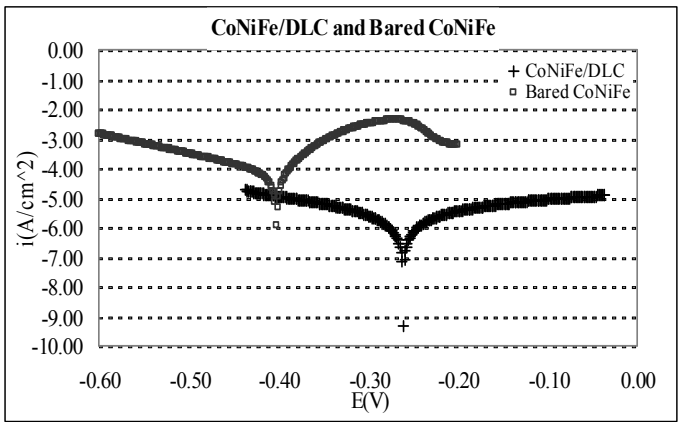

Fig.5Potentiodynamic polarization curves of bared and DLC coated CoNiFe alloy in 0.5 M H_2SO_4 solution

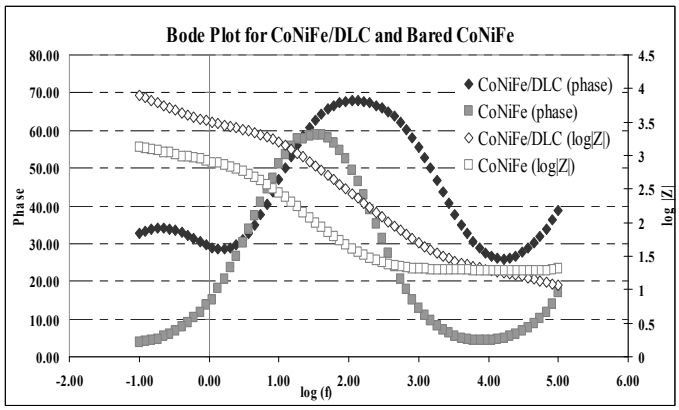

Fig.6Bode plot of bared and DLC coated CoNiFe alloy in 0.5 M H_2SO_4 solution

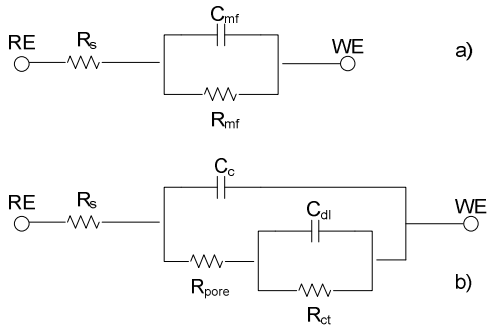

Fig.7Equivalent circuit model used to simulate a) the bared- CoNiFe b) CoNiFe/DLC

Table1 Electronic element values obtained from fitting EIS results with matching to equivalent circuit

Sample	R_{mf} (ohm)	C_{mf} (F/cm2)	R_{pore} (ohm)	C_c (F/cm2)	R_{ct} (Ohm)	C_{dl} (F/cm2)	Rp (Ohm)
Bared CoNiFe	968	2.4E-05					31084
CoNiFe/DLC			1769	5.8E-06	21560	9.8E-08	19560

Base on Bode plot (Fig.9), DLC-coated CoNiFe had prominent peak drifting from 10^{1-2} Hz of bared substrate to $10^{1.5-3}$Hz, indicated sample structure was changed. The capacitance and resistance values have been influenced by the DLC film properties. A low capacitance is desirable as it indicates that the film is less likely to generate a charge current, which could affect the magnetic properties of the device.

Conclusion and discussion

The square hole is Si grain size that diffused from substrate to aluminum layer as its indicies plane. If Si_3N_4/DLC films were good coverage, it will protect Si substrate underneath from diffusion. Indirect investigation to verify the DLC films weak point is powerful technique to provide the information for process engineer especially for the new process requirements that always continuously attempt to reduce DLC thickness for achieving better magnetic signal during data storage. Since the weak point area of thin DLC mostly occur along the edge and difficult to detect directly by metrology tools. According to the investigation we can conclude that 2nm DLC thickness is limited by the step of topography which shall not exceed 2nm. To prevent the corrosion with 2 nm DLC thickness, the underneath topography surface shall be less than 2 nm. Alternatively, if the step is fixed at 2nm, DLC layer should be thicker than 2nm. Another factor which influences weak point of DLC coverage is the shape of the step profile which causes the shadow effect, this factor shall be further characterized. By coating DLC films with Si_3N_4, the polarization resistance (R_p) values increased by two times of magnitude. Impedance spectra obtained from CoNiFe substrate with DLC coating exhibited two time constants. One peak in the Bode phase plot, observed at 100~10^{-1} Hz, is related to the AlTiC/solution

interface as determined by the bare substrate results. The other peak, comprising of R_{pore} and C_c, occurred at a higher frequency and is associated with the deposited film. R_{pore} represents the resistance of pores within the film and Cc is the coating capacitance. A high R_{pore} value implies that the film is effective in preventing the solution from penetrating to the substrate. A high R_{pore} value implies that the film is effective in preventing the solution from penetrating to the substrate.

CoNiFe high moment material in data storage device was protect from corrosion with Si_3N_4/DLC films. However its surface topography was influence to coverage performance. The investigation suggested 2 nm thickness was required for the coating. DLC/CoNiFe was improved corrosion resistance with charge transfer decreasing and pore resistance increasing

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