Evaluation of The Mechanical Properties of The Multilayered Films by Nanoindentation at a Penetration Depth as small as 10nm

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Abstract - In nanometer order nanoindentation test, roundness or truncation of the indenter tip cannot be avoided. In this paper, we analyzed the indentation problem of a rounded triangular indentation into a layered elastic half-space by a finite element analysis, and then established a method to estimate the intrinsic elastic modulus of the film from the nanoindentation data. The method was demonstrated by the nanoindentation test with a less than 10nm penetration depth on a 10nm thick diamond-like carbon film deposited on a 50nm thick magnetic layer.

Keywords : Berkovich indenter, tip truncation, composite Young's modulus, hard disk, diamond-like-carbon thin film.

1. INTRODUCTION

The media of hard disk drives are composed of multilayered films. The magnetic layer is protected by carbon overcoat, of which thickness in recent media becomes less than 30nm for improving the recording density of hard disks. In parallel with this, the flying height of the head slider on the disk decreases to less than 50nm, and the proximity contact recording will become reality in very near future. The tribology at the Head/Disk Interface (HDI) will be crucial in designing the future hard disk drives. Thus, the establishment of the method to evaluate the mechanical properties of 10nm order thickness carbon films on a multilayer structure is urgently needed.

Nanoindentation is a simple and effective means for evaluating the mechanical properties of thin films. In the method, the applied load, P , and the penetration depth, h , of the indenter are continuously monitored, and the mechanical properties are determined from the obtained P - h relationship. In common sense, for evaluating the intrinsic mechanical properties of a thin film, the penetration depth should not exceed one tenth of the film thickness.

Doerner and Nix [1] suggested that the effective composite Young's modulus, E_{eff}^* , in a layered solid can be described as a function of plain strain Young's moduli, $1/E^* = E/(1 - \nu^2)$, of the relevant materials;

$$\frac{1}{E_{eff}^*} = \frac{1}{E_f} (1 - e^{-\beta t/h}) + \frac{1}{E_s} e^{-\beta t/h} + \frac{1}{E_I} \quad (1)$$

Figure 1: Geometrical illustration of a truncated Berkovich indenter.

where β is an unknown parameter, t the film thickness, E the Young's modulus, and ν Poisson's ratio. Subscripts f , s and I refer to the film, substrate, and indenter properties, respectively. They determined β based on measurements of tungsten films on silicon substrates. King [2] analyzed the problem of triangular punches indenting a layered solid via a singular integral equation, and numerically obtained β as a function of normalized contact radius by t .

A triangular indenter is commonly used for nanoindentation measurement for the reason that the three facets theoretically converge to a single point. However, in practice, the truncation or roundness of the indenter tip may not be avoided during machining and testing, and the truncation may have a critical influence on nanometer order indentation testing. Namely, the problem in a nanoindentation test is not only the performance of a nanoindentation instrument but also the effect of the roundness or truncation of the indenter tip.

Shimamoto *et al.* [3] analyzed the effect of the tip truncation of a Berkovich indenter when indenting an elastic halfspace by a three-dimensional elastic finite el-

Figure 2: The finite element mesh of the layered solid.

ement method (FEM). They modeled the geometry of the tip truncation as shown in Fig. 1. The radius of curvature at the apex, R , is defined as represented in the G-G' section, where the profile of indenter is tangential to the edge OG and its center is situated on the indentation axis (z direction). They suggested that the P - h relationship can be approximately written by

$$P + \Delta P = C(\tan \alpha)E^*(h + \Delta h)^2, \quad (2)$$

where C is a geometrical factor and α the apical angle of the indenter. They determined that the factor C is 0.95 for the Berkovich indenter with $\alpha = 65^\circ$. Δh and ΔP are related to the truncation length, $\Delta h_T = (R/8) \cot^2 \alpha$, and expressed by

$$\begin{aligned} \Delta h &= 1.10\Delta h_T, \\ \Delta P &= 1.16C(\tan \alpha)E^*\Delta h_T. \end{aligned} \quad (3)$$

In this paper, we analyze the process of a rounded Berkovich indenter pressing into an elastic layered solid by FEM, and then demonstrate a method which can be utilized to calculate the intrinsic elastic modulus of a diamond-like carbon film with a thickness of 10 nm.

2. FINITE ELEMENT ANALYSIS

Figure 2 shows the finite element mesh of an elastic layered solid. For treating the coating, six mesh layers with a minimum thickness of 2nm are placed onto the previous mesh of a non-layered solid [3][4]. The elements are ten-noded isoparametric blocks (three-dimensional tetrahedral). The total number of nodes and elements are 6396 and 4014, respectively. The dimension of the analytical region is a tetrahedron with 2347 nm in sides along to the x , y and z axes. The minimum-sized elements are placed along to x , y and

Figure 3: Schematic block diagram of the depth controlling nanoindentation tester.

z axes beneath the origin O shown in Fig. 2. The displacements at the outer boundaries are fixed to be zero. The FEM analyses were computed for the indentation in a 30nm-thickness film by using a rigid indenter ($1/E_I^* = 0$). Young's modulus of the substrate E_s was fixed to be 206 GPa and that of the film was varied into four levels as $E_f/E_s = 1/4, 1/2, 1, 2, 4$, equating Poisson's ratio of all the materials to 0.3. The radius curvature of the indenter was varied into four levels, $R = 0, 100, 200$ and 300 nm.

3. NANOINDENTATION EXPERIMENT

A 10 nm thick diamond-like carbon (DLC) thin film was deposited on a 50 nm thick CoCrTa coating pre-sputtered on a toughened glass substrate.

The nanoindentation tests were performed by a depth controlling nanoindentation tester [5] as shown in Fig. 3 under a penetration speed of 0.25nm using a Berkovich indenter [5]. The applied load of the indenter is measured by an electronic microbalance with a $0.1\mu\text{N}$ resolution and the relative displacement between the indenter and the sample surface is monitored by an optical fiber displacement sensor [6] with a 0.1 nm resolution and 100Hz bandwidth. The indenter and the displacement sensor probe are fixed to an aluminum block, and the block is driven by a piezo-electric actuator with a $15\mu\text{m}$ travel length. The extension of the actuator is servo-controlled by a power amplifier via a proportional-integral-derivative (PID) controller to compensate difference between output signal from the displacement sensor, V_h , and reference signal, V_{h-ref} , from a digital-to-analog converter (DAC). The computer in Fig. 3 controls V_{h-ref} and stores P and V_h continuously through the RS-232C interface and an analog-to-digital converter (ADC), respectively.

4. NUMERICAL RESULTS

Figure 4 shows E_{eff}^*/E_s^* vs h/t relationships obtained by the FEM calculation for the films with $E_f/E_s=4$ and $1/4$; the results for differently truncated indenters are indicated by using different symbols, while those in the case of indenting on films with relative stiffnesses other than 4 and $1/4$ are not shown, but behave similar on the semi-log plot. The result of a non-truncated indenter ($R = 0$) indenting on films with four different stiffnesses are represented in Fig. 4 by the curves originated from E_f/E_s at $h/t = 0$, which were obtained by the regression of the calculated data; the corresponding result at $E_f/E_s = 1$ converges to a straight line at $E_{eff}^*/E_s^* = 1$. These curves for the non-truncated indenter were approximated to eqn (1) when $E_f < E_s$, while to the following equation when $E_f > E_s$,

$$\frac{1}{E_{eff}^*} = \frac{1}{E_s^*}(1 - e^{-\gamma(h/t)}) + \frac{1}{E_f^*}e^{-\gamma(h/t)} + \frac{1}{E_I^*}, \quad (4)$$

where γ is an unknown parameter. Both β and γ are weakly dependent on the h/t and the regressed curves gave the best fitting to the approximation when

$$\begin{aligned} \beta &= 0.257 + 0.022(h/t) \text{ (for } E_f/E_s = 1/4), \\ \beta &= 0.261 + 0.094(h/t) \text{ (for } E_f/E_s = 1/2), \\ \gamma &= 1.277 - 0.293(h/t) \text{ (for } E_f/E_s = 2), \\ \gamma &= 1.013 - 0.180(h/t) \text{ (for } E_f/E_s = 4). \end{aligned} \quad (5)$$

The E_{eff}^*/E_s^* values for the truncated indenter become higher than those for the non-truncated indenter and the discrepancy of the values between the two indenters increases with decreasing in h/t . Particularly, a striking discrepancy appears when $E_f < E_s$; the values for the truncated indenter increase with decreasing in h/t , while the trend appears converse for the non-truncated indenter. These facts suggest the opposite of the commonsense view that the intrinsic mechanical properties of a thin film are measured with the penetration depth less than one tenth of the film thickness.

Figure 5 shows the replot of Fig. 4 replacing h by $h + \Delta h$ and P by $P + \Delta P$, where Δh and ΔP are given in eqns (3). It is obvious that effect of truncation on E_{eff}^*/E_s^* is corrected by this replot based on the modified $P-h$ relation in eqn (2).

5. EXPERIMENTAL RESULTS AND DISCUSSION

In this study, making use of Fig. 5 on the basis of the relations in eqns (4) and (5), we estimate the elastic modulus of a 10 nm thick DLC layer on top of a 50 nm thick CoCrTa magnetic coating deposited on a

Figure 4: Relationship between E_{eff}^*/E_s^* and h/t for truncated Berkovich indenters calculated by FEM.

Figure 5: Replot of Fig. 4 replacing h by $h + \Delta h$ and P by $P + \Delta P$.

glass substrate. For this, we should determine Δh_T for the indenter to be used and E_s^* for CoCrTa magnetic coating/glass system. For the determination of Δh_T for the indenter, we follow the procedure mentioned in a previous paper [3]. We performed nanoindentation up to $h = 8$ nm on a substrate glass (Corning code 7059F) having $E = 67.5$ GPa and $\nu = 0.28$. We obtained a loading-unloading curve with no hysteresis and confirmed its elastic behavior by reloading. Then, we analyzed the elastic loading curve thus obtained as to give a best-fitting to the $P-h$ relationship in eqn (2) and determined at $\Delta h_T = 2.2$ nm by taking eqn (3) into account.

Figure 6 shows $P-h$ curves for the CoCrTa/glass coating system with and without a DLC coating. We can determine E_s^* by analysing the stiffness-measuring technique [1] to the unloading curve of the CoCrTa/glass coating system without a DLC coating, of which load-

Figure 6: P - h curves for a CoCrTa/glass system with and without a DLC coating. The thicknesses of the DLC and CoCrTa layers were 10 and 50 nm, respectively.

ing cycle shows a hysteresis indicating the occurrence of plastic deformation. Assuming that the contact area remains constant during initial unloading stage, an approximated elastic contact solution can be obtained by using the flat-ended punch model with equivalent contact area [1]. The solution can be written by

$$S = C_A E^* \sqrt{A_r}, \quad (6)$$

where C_A is a geometrical factor and A_r the real projected contact area. C_A for Berkovich indenter is evaluated at 1.46 for elastic contact [3] and 1.17 for elastic-plastic or plastic contact [2].

The indentation curve of the CoCrTa layer shows elastic-plastic behavior but not far from elastic. Thus we assume that the behavior represents a transient phase from elastic to elastic-plastic and that C_A becomes the arithmetic mean of the pertinent values; $(1.46 + 1.17)/2 = 1.32$. According to the assumption by Doerner and Nix [1], A_r may be related to the plastic depth h_p indicated in Fig. 6 and calculated by considering the geometry of the tip-truncated Berkovich indenter;

$$A_r = 3\sqrt{3}(\tan^2 \alpha)(h_p + \Delta h_T)^2. \quad (7)$$

The contact stiffness, S , and the plastic depth, h_p , at the maximum load for the CoCrTa layer are estimated at 11.5 kN/m and 6.0 nm, respectively. Substituting eqn (7) into eqn (6), E^* yields 219 GPa. This value may be reasonable because it is close to the composite elastic modulus calculated from a bulk cobalt.

The P - h curve for the DLC film behaves more elastic because the hysteresis is not as clear as the previous case. Figure 7 shows the E_{eff}^*/E_s^* vs h/t relationship for the experimental data of the DLC layer. E_{eff}^* is calculated from eqn (2) by assuming that $E_I = 1050$ GPa

Figure 7: Experimental E_{eff}^*/E_s^* vs $(h + \Delta h)/t$ relationship for a DLC film of 10 nm thickness onto CoCrTa layer of 50 nm thickness.

and $\nu = 0.1$. The experimental data agree with the curve for $E_{eff}^*/E_s^* = 1/2$, and the intrinsic composite elastic modulus of the DLC film can be estimated at 114 GPa. This value is comparable order of the elastic modulus prepared by a plasma decomposition method obtained by Jiang *et al.* [7]. The Young's moduli which they obtained decreased from 220 GPa to 90 MPa with the increase in C_2H_2 gas pressure.

In conclusion, this method is useful to evaluate the elastic moduli of films as thin as 10nm which will be used as a protective layer for information storage systems in the near future.

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