Abrasive-free polishing of hard disk substrate with H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry

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Abstract: The effect of *tert*-butyl hydroperoxide-sodium pyrosulfite ((CH₃)₃COOH-Na₂S₂O₅) as an initiator system in H_2O_2 -based slurry was investigated for the abrasive-free polishing (AFP) of a hard disk substrate. The polishing results show that the H_2O_2 -C₄ $H_{10}O_2$ -Na₂S₂O₅ slurry exhibits a material removal rate (MRR) that is nearly 5 times higher than that of the H_2O_2 slurry in the AFP of the hard disk substrate. In addition, the surface polished by the slurry containing the initiator exhibits a lower surface roughness and has fewer nano-asperity peaks than that of the H_2O_2 slurry. Further, we investigate the polishing mechanism of H_2O_2 -C₄ $H_{10}O_2$ -Na₂S₂O₅ slurry. Electron spin-resonance spectroscopy and auger electron spectrometer analyses show that the oxidizing ability of the H_2O_2 -C₄ $H_{10}O_2$ -Na₂S₂O₅ slurry is much greater than that of the H_2O_2 slurry. The results of potentiodynamic polarization measurements show that the hard disk substrate in the H_2O_2 -C₄ $H_{10}O_2$ -Na₂S₂O₅ slurry can be rapidly etched, and electrochemical impedance spectroscopy analysis indicates that the oxide film of the hard disk substrate formed in the H_2O_2 -C₄ $H_{10}O_2$ -Na₂S₂O₅ slurry may be loose, and can be removed easily during polishing. The better oxidizing and etching ability of H_2O_2 -C₄ $H_{10}O_2$ -Na₂S₂O₅ slurry leads to a higher MRR in AFP for hard disk substrates.

Keywords: abrasive-free polishing; material removal rate; initiator; hard disk substrate

1 Introduction

Chemical mechanical polishing (CMP) has been used as a global planarization technique since the 1990s, and it is currently widely used in the manufacture of ultra-precision surfaces, such as Si wafers and the Damascus interconnection structures of Cu in integrated circuits, computer disks and heads, etc. [1, 2]. Due to the rapid development of information technology, the amount of information that is transmitted has also exponentially increased, leading to much higher storage requirements for computer disk systems, which is an important aspect of information processing. For example, the physical size of hard drives continues to decrease, while the memory capacity of computer hard drives has increased rapidly, meaning that the

head-disk spacing has become narrower. It is believed that a flying height as small as 2.5–3.5 nm is necessary for an areal recording density of 1 Tbit/in², which is the next target in industry [3]. In this case, there is a need to satisfy significantly higher requirements for the machining accuracy and the surface quality of the heads and hard disk substrates. As a result, the CMP process for hard disk substrates should also be improved.

In the traditional CMP process, there are chemical reactions between the slurry and the material surface to be polished, which form a layer of oxidation film. Then, with the mechanical wear of the abrasive in the slurry and the polishing pad, the oxidation film will be removed, and the above behavior will happen again and again [4, 5]. Due to the combined chemical and mechanical processes, the material surface can be planarized. However, this leads to depression, erosion, and mechanical damage because of the abrasives [6–9].

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Further, these damages are difficult to remove using the CMP technique, resulting in faulty products. For the above reasons, an alternative process is to reduce or eliminate the abrasives in the slurry. Compared with the CMP process, in the abrasive-free polishing (AFP) process, the oxidation film is removed by the soft polishing pad without the abrasives. Therefore, it will not cause defects in the AFP process. In addition, there is a simpler post-clean process for materials polished by AFP [10], which enables significant reductions in the cost of the product.

In 2000, a Japanese researcher [10] developed a completely abrasive-free process for Cu damascene metallization, which provided a very clean, scratch-free, and anticorrosive surface. Since then, AFP has attracted the interest of many researchers, and it has been successfully applied to the surface planarization of materials such as Al metal film [11], GaN [12], Si, and SiC [13]. To the best of our knowledge, there have been few reports of surface machining of hard disk substrates that use the AFP process.

Oxidizers play a key role in the AFP process. Up to the present, due to its very strong oxidation capacity, and the fact that decomposed product is water (which is environmentally friendly), H₂O₂ has been chosen as the preferred oxidizer for use in the AFP process. However, the decomposition energy of H_2O_2 is 54 kcal/mol, which causes it to decompose too slowly at room temperature to be completely effective. In our previous studies, the use of Cu (II) [14] or potassium peroxydisulfate-sodium hydrogensulfite (K₂S₂O₈-NaHSO₃) as catalyzers [15] in the H₂O₂ slurry promoted the decomposition of H₂O₂, and caused it to exhibit a much higher material removal rate (MRR); a better substrate surface was obtained compared to that of the H₂O₂ slurry under the same conditions after the AFP process. In this paper, we investigated the effect of tert-butyl hydroperoxide-sodium pyrosulfite ((CH₃)₃COOH-Na₂S₂O₅) as the free radical initiator system for H₂O₂ slurry on the hard disk substrate AFP.

2 Experiment

2.1 Preparation of the abrasive-free slurry

A series of abrasive-free slurries was prepared, and we used 5 wt% H₂O₂ as the oxidant and 6 wt% dispersing

agent added into deionizer (DI) water to obtain the H_2O_2 slurry. 0.7 wt%–4.2 wt% (CH₃)₃COOH-Na₂S₂O₅ was used as the initiator to obtain H_2O_2 -C₄ $H_{10}O_2$ -Na₂S₂O₅ slurry. The molar ratio of (CH₃)₃COOH: Na₂S₂O₅ was 1:1.

2.2 Abrasive-free polishing tests

Polishing tests were conducted using a UNIPOL-1502 polishing equipment (Shenyang Kejing instrument, Co. LTD, China). The down force was 0.80 psi and the plate rotating speed was 80 rpm. The polishing time was 30 min. Work pieces were φ95 mm × 1.25 mm aluminum alloy disk substrates that were NiP plated; the plated layer consists of about 85 wt% nickel and 15 wt% phosphorus elements. The polishing pad was a Rodel porous polyurethane pad. The supply rate of the slurry was 160 mL/min. After polishing, the hard disk substrates were washed by ultrasonic cleaning in a cleaning solution containing 0.5 wt% surfactant in DI water. Finally, they were dried by a multifunctional drying system. The mass of the hard disk substrate was measured by an analytical balance both before and after the AFP process.

2.3 Observation of the surface morphology of the substrates

The surface roughness is a parameter that is most commonly used to evaluate the quality of a surface. A white light interferometer (NanoMap WLI, Caep Technology Corp., U.S.A) with a solution of 0.1 Å was used to measure the average roughness (Ra) and morphology of the polished substrate surface. The measurement area was $100~\mu m \times 100~\mu m$.

2.4 Electrochemical measurement of the substrates

All of the electrochemical experiments were carried out using a Solartron electrochemical workstation in a conventional three electrode system. The electrolyte solutions were prepared from the slurries. One of the slurries was the H_2O_2 slurry, and the other was the H_2O_2 slurry containing 3.5 wt% of initiator. The round hard disk sheet sealed with epoxy was used as the working electrode, and its diameter was 10 mm. The counter electrode was a Pt electrode and the reference electrode was a saturated calomel electrode (SCE). The potentiodynamic polarization plots were acquired by scanning the working electrode in the potential range

from -0.60 V to +0.20 V at a scan rate of 1 mV/s, and the electrochemical impedance spectroscopy (EIS) spectrum was acquired in the frequency range of 10^6 Hz to 0.1 Hz with a potential amplitude of 10 mV rms.

2.5 Electron spin-resonance spectroscopy (EPR) test of the slurries

The concentration of free radicals in the H_2O_2 slurry and the H_2O_2 slurry containing 3.5 wt% of initiator was measured by EMX EPR (Center field: 3518.07 G, scan width: 100 G, microwave power: 20 mW, scan time: 5.24 s, scanning frequency: 6 times). Dimethyl pyridine-N-oxide (DMPO) was added as trapping agent.

2.6 Element examination of hard disk substrate surface

To study the chemical reaction between the slurry and the disk substrate, we conducted a static immersion test of the disk substrate in the slurries. One of the slurries was the H_2O_2 slurry, and the other was the H_2O_2 slurry containing 3.5 wt% of initiator. After 48 h immersion at room temperature, the disk substrates were cleaned and dried. Then, the contents of the elements and their deep distribution in the polished surfaces were analyzed using PHI 680-Auger electron spectroscopy (AES, beam voltage: 5 kV, beam current: 10 nA, Ar ion beam: $2 \text{ kV } 1 \text{ mm} \times 1 \text{ mm}$, sputter rate (relative to SiO_2) is 6 nm/min).

3 Results and Discussion

3.1 AFP performances of the slurries

MRR can be calculated as in Eq. (1).

$$MRR = \frac{4 \times 10^9 \, m}{\pi d^2 t \, \rho} \tag{1}$$

where: MRR- material removal rate, nm/min

m- mass of material removed, g

d- diameter of work piece, mm

t- polishing time, min (in the test, t = 30)

 ρ - density, g/cm³ (plated NiP layer consisting of about 85 wt% nickel and 15 wt% phosphorus elements, ρ = 7.9)

Figure 1 clearly shows that as the concentration of the $C_4H_{10}O_2$ - $Na_2S_2O_5$ initiator increases, the MRR increases gradually, but it begins to decrease when the initiator's concentration is above 3.5 wt%. The maximum MRR (H_2O_2 slurry containing 3.5 wt% of $C_4H_{10}O_2$ - $Na_2S_2O_5$ initiator) is almost 5 times that of the minimum MRR (H_2O_2 slurry), which indicates that the $C_4H_{10}O_2$ - $Na_2S_2O_5$ initiator enhances the MRR of the hard disk substrate in the H_2O_2 -based slurry at the same polishing condition. It may be inferred that the chemical effect of the slurry is improved dramatically by the $C_4H_{10}O_2$ - $Na_2S_2O_5$ initiator.

In addition, the maximum MRR at 3.5 wt% of the initiator may be due to the moderate ratio of the oxidant to the initiator, while the amount of initiator is 3.5 wt% and the oxidant is 5 wt%. This means that there is an ideal rate of oxidant decomposition due to free radicals promoted by the initiator. In this situation, the oxidation reaction rate of the free radical is much higher than its quenching rate, so the disk substrate has a maximal MRR. However, when the initiator concentration increases to 4.2 wt%, the decomposition of the oxidant promoted by the initiator is too quick, and the quenching rate of the free radical may be higher than the oxidation reaction rate, so the disk substrate has a smaller MRR.

Further, 3D images of the surface morphology of the disk substrates are shown in Fig. 2, which clearly shows that the protuberance on the non-polished substrate surface can be partly removed after polishing with the H_2O_2 slurry. However, it still failed to achieve an adequate global flat surface (Fig. 2(b)). However,

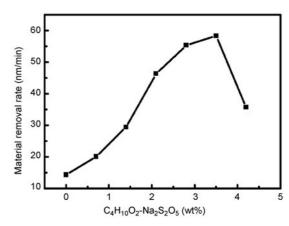
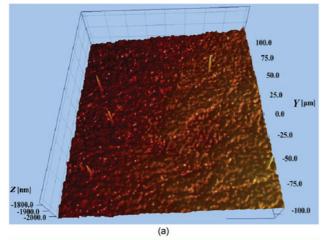
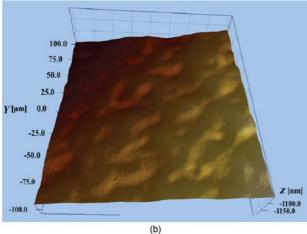


Fig. 1 The MRR of the disk substrates polished with the H_2O_2 slurry added with different weights of the $C_4H_{10}O_2$ - $Na_2S_2O_5$ initiator.





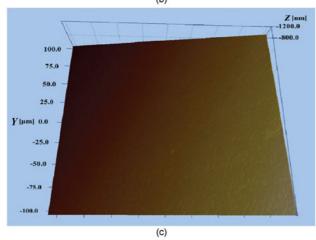


Fig. 2 3D images of the disk substrate surfaces: (a) before polishing, Ra = 33.4 nm; (b) polished with the H_2O_2 slurry, Ra = 25.6 nm; (c) polished with the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry, Ra = 17.8 nm.

it's the global flat surface (Fig. 2(c)) after polishing with the $\rm H_2O_2\text{-}C_4H_{10}O_2\text{-}Na_2S_2O_5$ slurry and the nano-asperity peaks are hardly observed. Besides, the $\rm H_2O_2\text{-}C_4H_{10}O_2\text{-}Na_2S_2O_5$ slurry has a Ra of 17.8 nm, which is nearly

half of that of the non-polished surface (Ra = 33.4 nm). In other words, the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry can obviously improve the surface planarization of hard disk substrates.

3.2 Mechanism discussion

3.2.1 Electrochemical analysis of hard disk substrate

The electrochemical test can be carried out to investigate the corrosion action of the hard disk substrate in AFP slurry [16–18]. It can reflect the kinetic action and thermodynamic action of the metal dissolution and the surface film formation of metal in the AFP slurry, which can be used to predict the MRR. Herein, the potentiodynamic polarization plots were used to analyze the corrosion of the hard disk substrate in the slurries, as shown in Fig. 3. The corrosion current (I_{corr}) values are estimated from these plots using the Tafel extrapolation method.

In particular, by comparing the behavior of the anodic polarization curve between the H_2O_2 slurry and the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry, the different slopes of the anodic polarization curve can be observed. In the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry, the slope of the anodic polarization is decreased. This indicates that the corrosion reaction of the oxide film on the substrate surface occurs easily in the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry. Besides, The I_{corr} of the hard disk in the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry is 0.684 mA, which is much larger than that in the H_2O_2 slurry (I_{corr} = 0.201 mA). The increase of the corrosion current indicates that

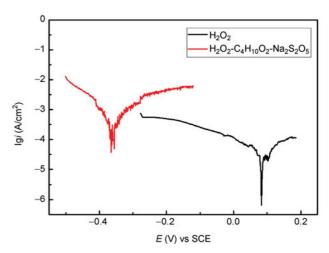


Fig. 3 Potentiodynamic polarization plots of hard disk substrates in the H_2O_2 slurry and the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry.

the $C_4H_{10}O_2$ -Na $_2S_2O_5$ initiator promotes the corrosion of the disk surface in the kinetics. In this case, the hard disk substrate in the H_2O_2 - $C_4H_{10}O_2$ -Na $_2S_2O_5$ slurry can be etched faster than that with the H_2O_2 slurry. Therefore, the enhancement of the electrochemical corrosion can help to increase the MRR of the hard disk substrate in AFP processes with the H_2O_2 - $C_4H_{10}O_2$ -Na $_2S_2O_5$ slurry.

In the previous CMP studies, He et al. [19] analyzed the oxide film thickness of Cu surfaces in different media by using the impedance spectra, and reported that the magnitude of the impedance can be used to characterize the oxide film formed on the Cu surface. Here, the impedance spectra were used to characterize the oxide film of the hard disk substrate in the slurries. Fig. 4 shows the impedance spectra of the hard disk substrate in the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry (Fig. 4(b)) and the H_2O_2 slurry (Fig. 4(a)), respectively. All of the spectra show the same pattern, i.e., two linked depressed semicircle. A comparison of the two spectra shows that the magnitude of the impedance at low frequencies is significantly smaller for the surface on a hard disk substrate in the H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry. This indicates that the polarization resistance of the surface is effectively decreasing. Accordingly, it can be inferred that the oxide film formed on the substrate surface in the H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry may be very loose, and can be removed easily and quickly by the polishing pad in AFP. Consequently, the MRR of the hard disk substrate in the AFP process with the H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry becomes large, which is consistent with the results of polishing tests.

3.2.2 Comparison of the oxidizing ability of the slurries

It is known that the oxidation performance of H_2O_2 solution is very high because H_2O_2 can be decomposed into hydroxyl free radicals, which shows a stronger oxidizing ability. It may be inferred that the solution's oxidizing ability can be determined by the concentration of free radicals in it. To investigate the free radical's change caused by the $C_4H_{10}O_2$ -Na₂S₂O₅ initiator, we conducted EPR tests of the two slurries, as shown in Fig. 5.

There are four obvious spectral peaks in the EPR spectrum of H₂O₂ slurry (Fig. 5(a)), and the relative intensity of the peaks is 1:2:2:1. This indicates a typical EPR spectrum of hydroxyl free radical [20]. The EPR spectrum of the H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry is shown in Fig. 5(b), and shows that the four spectral peaks of hydroxyl free radical in Fig. 5(a) also exist in the spectra of Fig. 5(b). In addition, the peak intensity of these four spectral peaks of the spectrum in Fig. 5(b) is even 150 times that shown in Fig. 5(a). It can be concluded that the concentration of hydroxyl free radicals in the H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry is much larger than that in the H_2O_2 slurry. The reason may be that the $C_4H_{10}O_2$ -Na₂S₂O₅ initiator can strongly induce the generation of the hydroxyl free radical (as seen in Eq. (2), Eq. (4), and Eq. (5)). In Fig. 5(b), it is also noted that there are also three small spectral peaks with an intensity of about 2.5×10^5 , which may be alkoxy free radicals (as in Eq. (2)). The possible reaction is shown as Eqs. (2)–(5). The $C_4H_{10}O_2$ -Na₂S₂O₅ initiator in the H_2O_2 slurry may greatly enhance the oxidation ability of the slurry since

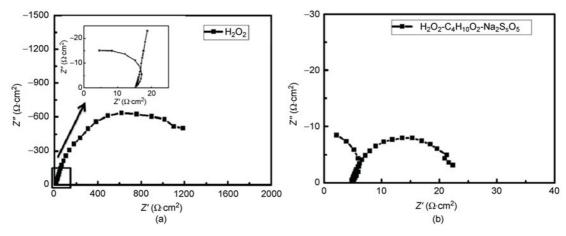
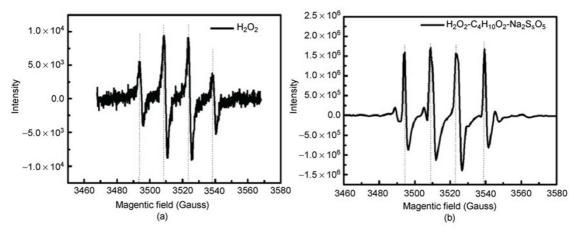


Fig. 4 The EIS spectra of hard disk substrate in the H₂O₂ slurry (a) and the H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry (b).



(4)

Fig. 5 EPR analysis of two slurries: (a) the H₂O₂ slurry; (b) the H₂O₂-C₄H₁₀O₂-Na₂S₂O₅ slurry.

it can strongly induce the generation of the hydroxyl free radical and the alkoxy free radical.

$$ROOH \rightarrow RO \cdot + \cdot OH$$
 (2)

$$HO-OH \rightarrow HO \cdot + \cdot OH$$
 (3)

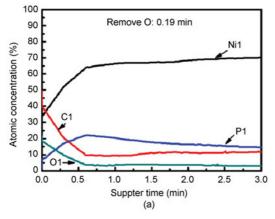
$$4ROOH + S_2O_5^{2-}$$
 → $4RO \cdot + 2SO_4^{2-} + 2H^+ + H_2O (R: (CH_3)_3C-)$

$$RO+HO-OH \Rightarrow HO+RO-OH$$
 (5)

Next, AES was conducted to observe the element components and their deep distribution in the surface of hard disk substrate. Figure 6 shows the element components in the surface of the hard disk substrate after the immersion test in the H_2O_2 slurry (Fig. 6(a)) and the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry (Fig. 6(b)). A comparison of the two slurries' spectra shows that

the atomic concentrations of the elements Ni and P present a similar increasing trend, that is, it first increases and then fluctuates slightly as the sputter time increases. At the same time, the atomic concentrations of the elements O and C present a similar decreasing trend and are then stabilized. In addition, from the spectra of the two slurries before sputtering, we found that the atomic concentration of the elements Ni, P, and O were 15 wt%–40 wt%, 0 wt%–10 wt%, and 20 wt%, respectively. In comparison with that consisting of 85 wt% Ni and 15 wt% P for the plated NiP substrate, the introduction of the element O and the reduction in the atomic concentration of Ni and P imply that an oxidization reaction may occur between the substrate and the two slurries.

Further, the thickness of the oxide film formed on the substrate surface in the slurries can be estimated by the sputter time taken to remove O. The sputter



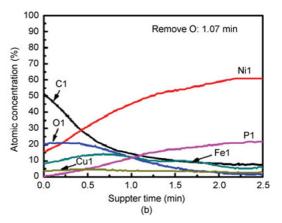


Fig. 6 AES analysis of the hard disk substrate soaked in (a) the H_2O_2 slurry and (b) the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry.

time for the removal of O is 0.19 min and 1.07 min for the H_2O_2 slurry and the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry, respectively. The sputter rate (relative to SiO₂) is 6 nm/min. Therefore, we can estimated that the thickness of the oxide film formed in the H_2O_2 slurry and the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry is about 1.14 nm and 6.42 nm, respectively. This means that the oxidizing ability of the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry is much greater than that of the H_2O_2 slurry, which is consistent with the results of the EPR analysis.

In summary, the electrochemical analysis of the hard disk substrate shows that the $H_2O_2\text{-}C_4H_{10}O_2\text{-}Na_2S_2O_5$ slurry possesses stronger electrochemical corrosion ability than the H_2O_2 slurry. At the same time, EPR and AES analyses show that the $H_2O_2\text{-}C_4H_{10}O_2\text{-}Na_2S_2O_5$ slurry has a stronger oxidizing ability than the H_2O_2 slurry because of the large number of hydroxyl free radicals induced by the $C_4H_{10}O_2\text{-}Na_2S_2O_5$ initiator. Consequently, the MRR of the substrate polished with the $H_2O_2\text{-}C_4H_{10}O_2\text{-}Na_2S_2O_5$ slurry is much larger than that with the H_2O_2 slurry.

4 Conclusion

The addition of the $C_4H_{10}O_2$ - $Na_2S_2O_5$ initiator into H_2O_2 -based slurry can greatly improve the MRR and surface planarization of the hard disk substrate in the AFP process. This improvement may be attributed to the effect of the $C_4H_{10}O_2$ - $Na_2S_2O_5$ initiator. The rate of the corrosion reaction and the rate of the oxidation reaction of the hard disk substrate were all accelerated in the H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ slurry. Also, due to the combined effect of these factors, the MRR of the hard disk substrate increased dramatically. The results imply that H_2O_2 - $C_4H_{10}O_2$ - $Na_2S_2O_5$ abrasive-free slurry possesses promising prospects in AFP.

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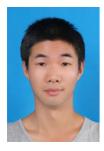
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