



Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Scratch behavior of re-structured carbon coating by oxygen plasma etching technology for magnetic disk application

Meiling Guo^a, Dongfeng Diao^{b,*}, Xue Fan^a, Lei Yang^a, Liwei Yu^a^a Key Laboratory of Education Ministry for Modern Design and Rotor-Bearing System, School of Mechanical Engineering, Xi'an Jiaotong University, Xi'an 710049, China^b Institute of Nanosurface Science and Engineering (INSE), Shenzhen University, Shenzhen 518060, China

ARTICLE INFO

Article history:

Received 15 January 2014

Accepted in revised form 5 April 2014

Available online xxx

Keywords:

Oxygen plasma etching technology

Carbon coating

Re-structure

Scratch behavior

Magnetic disk application

ABSTRACT

An oxygen plasma etching technology to prepare ultrathin carbon coatings for magnetic disk in electron cyclotron resonance (ECR) plasma system was developed. In the preparation process, as-deposited carbon coatings were etched by oxygen plasma to obtain re-structured carbon coatings. Scratch behaviors of the as-deposited and re-structured carbon coatings on silicon substrates were evaluated by atomic force microscope (AFM) scratch test. It was found that the scratch resistance of the re-structured carbon coating was improved. Then X-ray photoelectron spectroscopy (XPS) analysis showed that after oxygen plasma etching, the C=C (sp^2) content decreased from 55% to 17%, the C–C (sp^3) content increased from 33% to 58% and the C–O content increased from 7% to 18%. It indicated that the oxygen plasma etching induced the increase of the sp^3 bonding carbon, which contributed to the improved scratch resistance. Further transmission electron microscope (TEM) observation was conducted and the rising of the scratch resistance was inferred to be caused by the formation of the interlayer cross-linking after preferentially breaking the sp^2 bond. Based on the above results, the ultrathin re-structured carbon coatings by oxygen plasma etching were prepared on uncoated magnetic disk substrates and showed a better scratch behavior and a comparable surface roughness in comparison with the commercial magnetic disk.

© 2014 Elsevier B.V. All rights reserved.

1. Introduction

The storage density of magnetic media increases exponentially with the decrease in the magnetic spacing [1–6]. Decreasing the thickness of carbon protective coating is an effective way to reduce the magnetic spacing. The reduction of magnetic spacing can also be achieved by decreasing the flying height. However, the flying height reduction will increase the possibility of the accidental contacts of head/disk and slider/asperities, which can generate problems of collision and mechanical scratch [7], and thus the high scratch resistance and smooth surface are required for the carbon protective coating. Therefore, the preparation of ultrathin carbon protective coating with high scratch resistance and low surface roughness for magnetic disk becomes an important issue.

The ultrathin carbon coating for magnetic disk application is usually prepared by physical or chemical vapor deposition. During deposition, to ensure the formation of a continuous coating, the carbon phase must nucleate everywhere on the substrate, and at the same time the carbon species cannot move across the substrate to form islands and leave some parts of the substrate uncovered [8]. These requirements

are satisfied by the use of a deposition flux with high ionization rate and appropriate energy, limiting available deposition methods and preparation parameters. For example, the conventional magnetron sputtering method is unable to deposit continuous coating as thin as 2 nm [2,3]. To solve the problem, plasma treatment by embedment of C^+ ions into surface has been reported to allow sub-surface coating growth and formation of protective coatings thinner than 2 nm [5,9,10]. In addition, plasma treatment by atomic scale etching of as-deposited coating is also an effective way to obtain such a coating. For physical etching, the displacement threshold energy of carbon substance with sp^2 and sp^3 allotropes is about 30–40 eV [11–13]. In order to physically etch carbon atoms, high kinetic ion energy exceeding the threshold energy is needed but higher kinetic ion energy will cause heavier ion impingement on the treated magnetic disk surface. In contrast, chemical etching can remove the surface carbon atoms by chemical interaction at lower kinetic ion energy [14]. Therefore, chemical etching is supposed to be an optimum choice.

Oxygen plasma with energetic oxygen species has been used to modify carbon related materials [15–20]. Paredes et al. [17] suggested that the graphite surface was observed to develop an amorphous character after oxygen plasma treatment. The modification made the treated graphite exhibit superior mechanical properties. Liou et al. [18] stated that oxygen played an important role in diamond film growth by preferentially etching the non-diamond carbon, which was proved by

* Corresponding author. Tel.: +86 755 26902415.
E-mail address: dfdiao@szu.edu.cn (D. Diao).

the decrease of the graphitic component in the film. Jiang et al. [19] reported that oxygen ions preferentially etched the soft graphite-like sp^2 clusters in sp^3 and sp^2 carbon network. Cateledge et al. [20] also suggested that oxygen in DLC helped to reduce non-diamond phase such as graphite-like sp^2 sites and enhance diamond-like sp^3 sites. Therefore, it can be inferred that the oxygen plasma etching has a potential to increase the sp^3 bonding carbon and improve the scratch behavior of carbon coating.

In this paper, the oxygen plasma etching technology in ECR plasma system was presented as a potential method to obtain ultrathin carbon coatings for magnetic disk application. The scratch behaviors of carbon coatings before and after oxygen plasma etching on silicon substrates were compared by AFM scratch test. The chemical structures and nanostructures were examined by XPS and TEM for studying the mechanism of the improved scratch resistance induced by oxygen plasma etching. Finally, the ultrathin re-structured carbon coatings by oxygen plasma etching were prepared on uncoated magnetic disk substrates, and the scratch resistance and surface roughness were measured and compared with those of the commercial magnetic disk.

2. Experimental method

2.1. Preparation of re-structured carbon coating by oxygen plasma etching technology

A divergent ECR plasma processing system was used to prepare the ultrathin re-structured carbon coatings by oxygen plasma etching. Full details of the system have been reported in our previous works [21, 22]. The experimental procedure is shown in Fig. 1. First, the chamber was evacuated to the pressure of 3×10^{-4} Pa, and argon was introduced keeping the working pressure to be 0.04 Pa. ECR plasma was generated when the microwave with power of 200 W was introduced and the magnetic field was applied. With a target discharge voltage of -300 V and a substrate bias voltage of -5 V, the as-deposited carbon coating was prepared (gray strip in Fig. 1(a)). After that, as shown in Fig. 1(b), oxygen and argon gas mixture with oxygen gas concentration of 12% was injected into the chamber to generate the oxygen plasma, and the working pressure was 0.04 Pa. The target voltage was set to zero and a substrate bias voltage of -10 V was applied to etch the as-deposited carbon coating. Then the re-structured carbon coating was obtained. In the ECR plasma, not all of the ions had the same energy, but the energy dispersion of the ions extracted was narrow and the upper end of the energy profile was short [23]. In this study, we calculated the mean ion kinetic energy by considering the effects of plasma floating voltage (about 0 V) and substrate bias voltage. Therefore, the mean ion kinetic energy was 10 eV when substrate bias voltage of -10 V was applied.

For the ease of coating characterizations, the as-deposited and re-structured carbon coatings were firstly prepared on p-type (100) oriented silicon substrates to study the scratch behavior change induced by oxygen plasma etching. In order to exclude the substrate effect on scratch behavior comparison, large thickness coatings were used with the deposition time of 20 min and the plasma etching time of 20 min. For further evaluating the performance of the oxygen plasma etching technology in magnetic disk application, the re-structured carbon coatings with small thickness were then prepared on uncoated magnetic disk substrates. The substrate was obtained by etching away the original carbon coating of the commercial magnetic disk with 50 eV ECR Ar plasma. To ensure the complete removal of the original carbon coating with the underlying magnetic layer intact, the etching time of 10 min was determined when the surface electric resistance just decreased to zero. In the whole preparation process, the substrate temperature was below 100 °C, which was much lower than Curie temperature of the magnetic recording media.

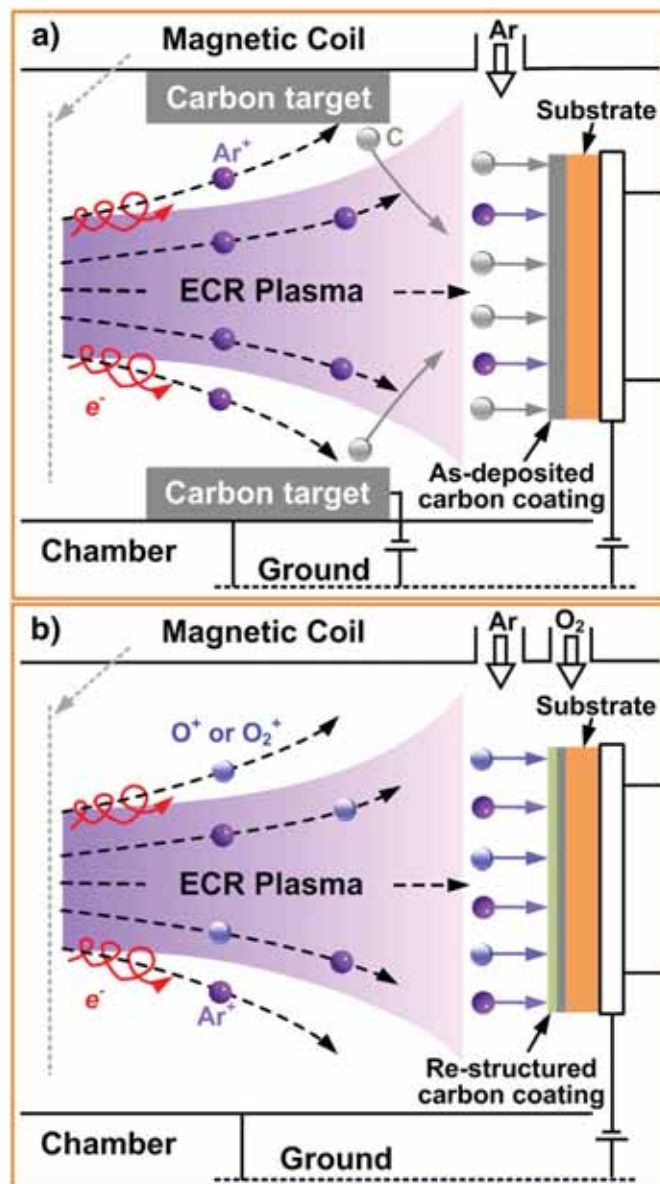


Fig. 1. Preparation procedure of (a) the as-deposited carbon coating by ECR plasma sputtering and (b) the re-structured carbon coating by oxygen plasma etching.

2.2. Scratch test

A retrofitted AFM system (AutoProbe CP-R, Veeco) with a 100-nm-radius Berkovich diamond tip (TI-0039, Hysitron) was developed to evaluate the scratch behavior of carbon coating. A manganese steel cantilever (normal stiffness: 24.43 N/m) with a diamond tip fixed at the free end was mounted on the high-precision guide screw movement stage of the AFM. The normal load was applied to the sample by driving the guide screw to move in z direction and monitored by a strain-gauge force sensor (KFG-1N-120-C1, Kyowa). Then with the normal load held constant, the sample was laterally displaced for 150 μm at a speed of 6 $\mu\text{m/s}$ controlled by the AFM piezoelectric scanner. After that, an AFM (Innova, Bruker) with a 2-nm-radius silicon tip (SNL-10, Bruker) was used to scan the scratched surface in the contact mode. The scan direction was perpendicular to the scratch direction and the scratch depth was measured by the cross-sectional topography across the scratch. By comparing the scratch depths of different carbon coatings under the same normal load, the scratch behaviors can be evaluated.

In order to calibrate the test device, silicon (100) was scratched under the normal load of 500 μN before and after every scratch test. The almost constant scratch depth indicated that the diamond tip wear was negligible and the data of scratch test were reliable.

2.3. Characterizations

In order to measure the deposition thickness and the etching thickness, part of the surface was pre-masked. After deposition and etching procedures, the masks were removed and steps formed on the surface. Then the steps were scanned by AFM in the contact mode to measure the deposition thickness and etching thickness. The root mean square (RMS) roughness was also obtained using AFM with an 8-nm-radius silicon tip (RTESP, Bruker). The tests were operated in the tapping mode with scan size of $1\ \mu\text{m} \times 1\ \mu\text{m}$, scan rate of 0.8 Hz and scan lines of 256. The AFM noise was less than 0.03 nm in the vertical dimension.

The chemical compositions and bonding configurations of the as-deposited and re-structured carbon coatings were analyzed by an AXIS ultra DLD multifunctional XPS with a monochromatic Al K α X-ray source. To compensate for surface charging, the binding energies were referred to the Ar 2p $_{3/2}$ peak which was introduced in the coating preparation process [24]. The O/C atomic ratios were calculated after correcting the relative peak areas by sensitivity factors. The C 1s spectra were fitted by Gaussian–Lorentzian sum components after the inelastic scattering backgrounds were subtracted using Shirley's method. The oxidative state of the magnetic recording layer covered with the re-structured carbon coating was also analyzed by XPS.

The nanostructures of carbon coatings were observed by a JEM-2100 TEM operated with the electron acceleration voltage of 200 kV. TEM specimens for the cross-sectional view were prepared by mechanical polishing and argon ion beam milling.

3. Experimental results

3.1. Oxygen plasma etching rate

In order to obtain the etching rate, AFM scan was conducted at the step formed on the coating after oxygen plasma etching. According to the results, when the etching time was 20 min, the step height was around 9 nm suggesting the etching rate was around 0.45 nm/min. In the oxygen plasma etching process, argon plasma etching as a physical etching was a factor that cannot be ignored. In order to make clear the effect of oxygen and argon, carbon coating was etched by pure argon plasma with the same ion kinetic energy of 10 eV. It was found that the etching rate measured was zero. The reason was that the ion kinetic energy of 10 eV was too low to sputter the carbon atoms physically. Hence it was believed that in the oxygen plasma etching process, the chemical interaction between oxygen and carbon atoms played a major role in removing the surface carbon atoms in the form of CO or CO $_2$ [25,26]. The oxygen plasma etching with ion kinetic energy of 10 eV as a chemical etching could remove the surface carbon atoms and get ultrathin coatings easily.

3.2. Scratch resistance

When the normal load of 800 μN was applied, the three dimensional images of the scratched surfaces of the as-deposited and re-structured carbon coatings on the silicon substrates are presented in Fig. 2(a) and (b). Clear scratch tracks can be seen from the images. Fig. 2(c) compares the scratch depths of the coatings and shows that the scratch depth decreased from 3.5 nm to 2.3 nm after oxygen plasma etching. The scratch depths of carbon coatings under the normal loads of 400 μN , 600 μN and 800 μN are presented in Fig. 3. In both of the carbon coatings, the depth approximately increased linearly as the normal load increased. And under all measured normal loads, the scratch depths of the re-structured carbon coating were lower than those of the as-

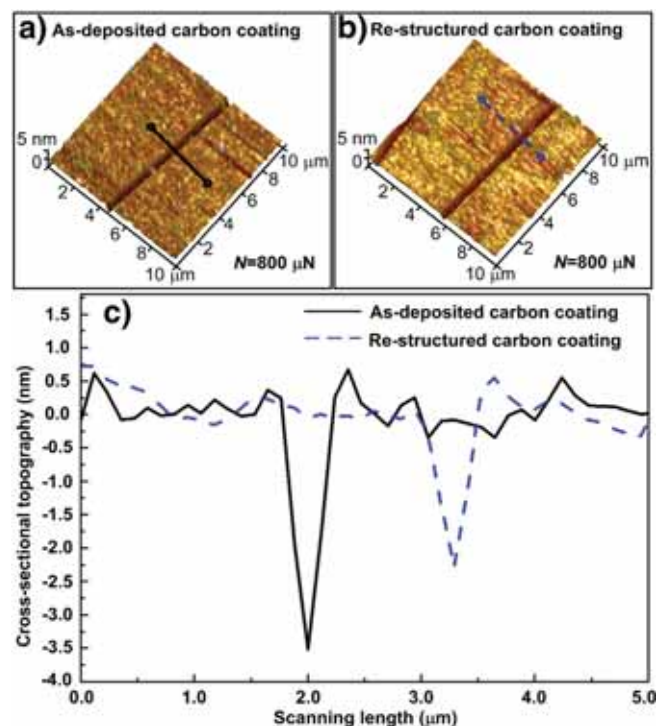


Fig. 2. Three dimensional images of the scratched surface of (a) the as-deposited carbon coating and (b) the re-structured carbon coating under the normal load of 800 μN . (c) Cross-sectional topographies across the scratches of two carbon coatings.

deposited carbon coating, indicating that the scratch resistance of the re-structured carbon coating by oxygen plasma etching was improved.

The thicknesses of the as-deposited and re-structured carbon coatings measured by AFM were about 80 nm and 70 nm, respectively. In the scratch tests, as the maximum scratch depth around 3.5 nm was lower than one tenth of coating thickness, the scratch results were not affected by substrates and could reflect the intrinsic scratch behavior of carbon coatings.

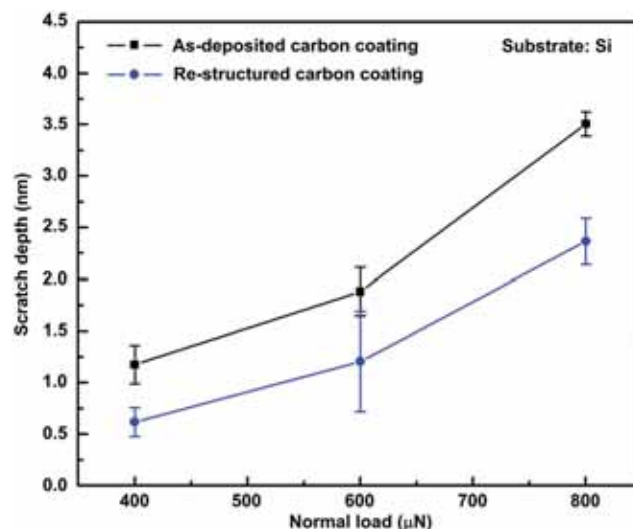


Fig. 3. Scratch depths of the as-deposited and re-structured carbon coatings under different normal loads of 400 μN , 600 μN and 800 μN .

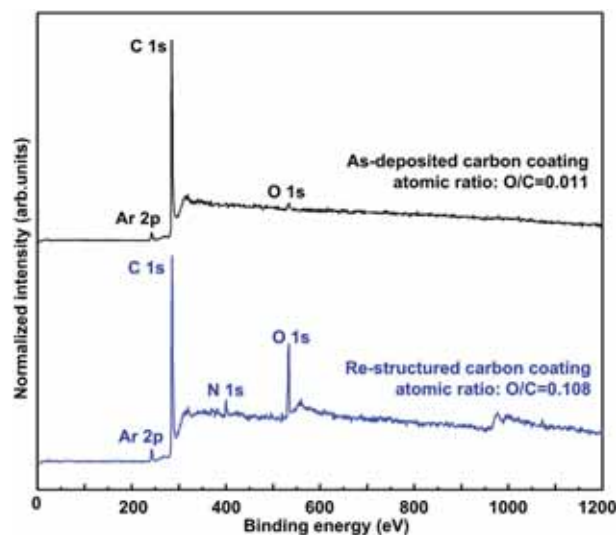


Fig. 4. XPS survey spectra of carbon coatings.

3.3. Chemical structure evolution

In order to study the scratch resistance rising mechanism, XPS tests were performed to specify the chemical structure evolution of carbon coatings induced by oxygen plasma etching.

Fig. 4 shows the XPS survey spectra of the as-deposited and re-structured carbon coatings. The main peaks observed at 285 and 531 eV are due to photoelectrons excited from the C 1s and O 1s levels, respectively. In the as-deposited carbon coating, the O/C atomic ratio was 0.011. After oxygen plasma etching, the ratio increased to 0.108. In other words, the oxygen content of the re-structured carbon coating was larger than that of the as-deposited carbon coating. Hence, in the re-structured carbon coating, there could be more oxygen atoms adsorbed and bonded with the carbon atoms.

To examine the bonding configuration change caused by oxygen incorporation, the C 1s XPS spectra of the as-deposited and re-structured carbon coatings were decomposed into five components. The two main components around 284.6 and 285.4 eV correspond to sp^2 and sp^3 carbon hybridization. The remaining three components around 286.7, 288.1 and 289.2 eV are attributed to C–O, C=O and O–C=O [27–29]. The results are shown in Fig. 5. According to the peak integrated area ratios of different bonds, the contents of the five bond types in carbon coatings are presented in Table 1. These data showed that after oxygen plasma etching, the C=C (sp^2) content decreased from 55% to 17%, while the C–C (sp^3) content in carbon coating increased from 33% to 58%. And the C–O content also increased from 7% to 18% obviously. These results indicated that the oxygen plasma etching induced the increase of the sp^3 bond, which promoted the improvement of the mechanical properties of the coating remarkably. Therefore, the scratch resistance of the re-structured carbon coating etched by oxygen plasma was better than that of the as-deposited carbon coating.

3.4. Application of oxygen plasma etching technology to carbon coating for magnetic disk

Re-structured carbon coatings with different thicknesses ranging from 0 to 7.3 nm were prepared on the uncoated magnetic disk substrates to explore the application of oxygen plasma etching technology to carbon protective coatings for magnetic disk. The scratch depths of the re-structured carbon coatings with different thicknesses under the normal load of 300 μ N are presented by the blue dots in Fig. 6. When the coating thickness was less than 2 nm, the scratch depths were 3–6 nm, which means the magnetic layer has been damaged and the demagnetization of the magnetic disk occurred [7]. When the thickness

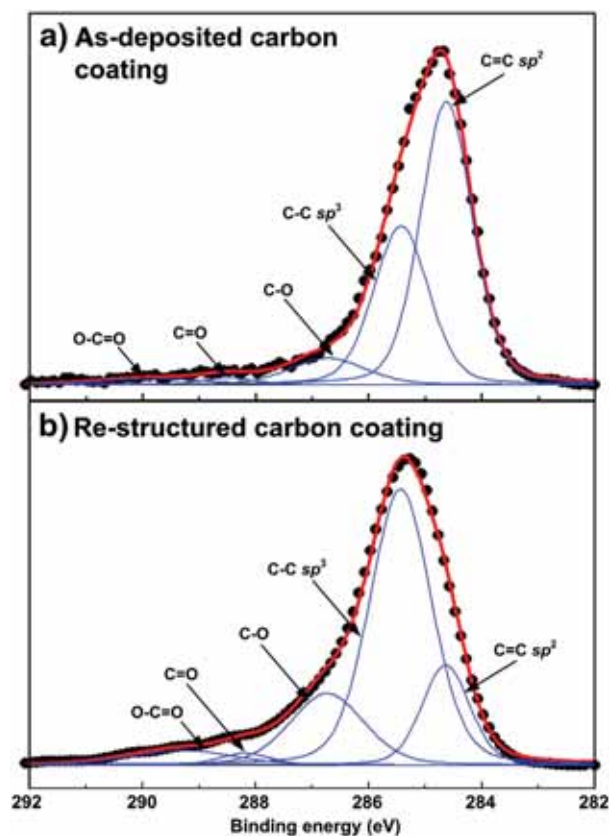


Fig. 5. C 1s XPS spectra of carbon coatings (black dots: original data; red lines: the peak fitting curves; blue lines: five components). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

increased to 2.5–3.5 nm, the scratch depths were kept at about 1.5 nm, which was less than the coating thickness, and thereby the magnetic layers were protected from damage. For comparison, the scratch depth of the commercial magnetic disk under the normal load of 300 μ N was also measured. The commercial magnetic disk contained a 2–3 nm carbon coating [4] and a lubricant layer. The lubricant layer was fully removed by 10 eV ECR Ar plasma etching before test. According to the result, the depth was about 2.5 nm (red square in Fig. 6). Thus, it can be concluded that the re-structured carbon coatings with thickness of 2.5–3.5 nm had a better scratch behavior than the commercial magnetic disk. With a comparable scratch depth to the commercial magnetic disk, the thickness of the re-structured carbon coating could be decreased to 1.5–2 nm, so that the thinner protective coating could be provided for higher storage density magnetic recording media.

Fig. 7(a) and (b) shows the surface topographies of the commercial magnetic disk and the re-structured carbon coating on the uncoated magnetic disk substrate, respectively. The RMS roughness of the re-structured carbon coating was 0.11 nm, which was comparable to that of the commercial magnetic disk surface (0.13 nm). The results indicated that the oxygen plasma etching technology could meet the surface roughness requirement for magnetic recording media.

Table 1
Bond contents of carbon coatings.

Bond type	Content	
	As-deposited carbon coating	Re-structured carbon coating
C=C (sp^2)	0.55	0.17
C–C (sp^3)	0.33	0.58
C–O	0.07	0.18
C=O	0.01	0.02
O–C=O	0.04	0.05

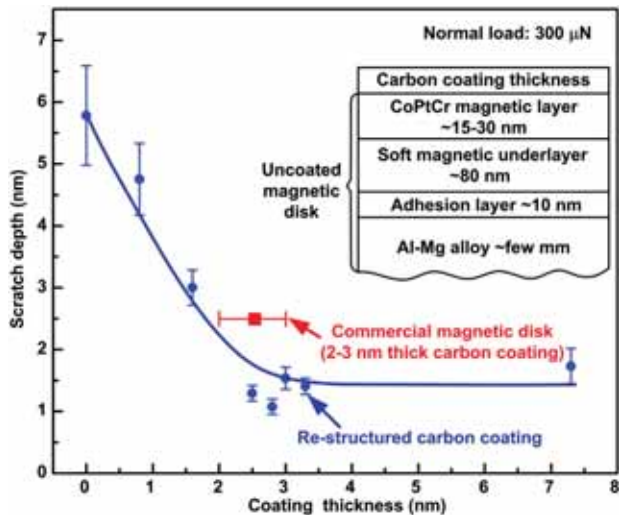


Fig. 6. Scratch depths of the re-structured carbon coatings with different thicknesses (blue dots) and the commercial magnetic disk with 2–3 nm thick carbon coating (red square) under the normal load of 300 μN . (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

In order to further explore whether the oxygen atoms diffused through the carbon coating to the magnetic recording layer and oxidized it, the oxidative state of the magnetic layer covered with re-

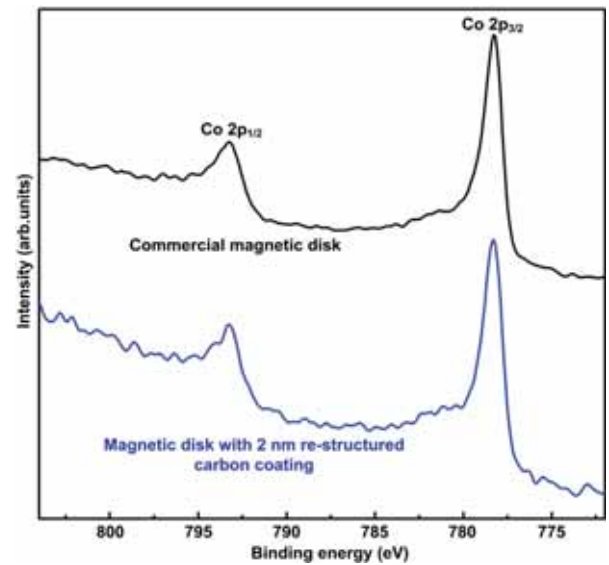


Fig. 8. Co 2p XPS spectra of the commercial magnetic disk and the magnetic disk with 2 nm re-structured carbon coating.

structured carbon coating was studied by XPS. Fig. 8 shows the Co 2p XPS spectra of the commercial magnetic disk and the magnetic disk with 2 nm re-structured carbon coating. It was found that the magnetic recording layer covered with re-structured coating showed the narrow Co 2p_{3/2} peak comparable to the commercial magnetic disk, indicating that the cobalt still existed in the metallic state [5]. Therefore, the oxygen plasma treatment did not change the structure of the magnetic recording layer.

In summary, the oxygen plasma etching technology can be used to prepare ultrathin carbon coatings with high scratch resistance, low surface roughness and good protection for the magnetic recording layer from oxidation for magnetic disk application.

4. Discussion

In the oxygen plasma etching process, carbon atoms in the upper surface were removed in the form of CO and CO₂ layer by layer [30, 31]. And the top surface after oxygen plasma etching was re-structured with the increase of the *sp*³ bonding carbon. In order to clarify the re-structure process of coating surface, the cross-sectional TEM images of the as-deposited and re-structured carbon coatings are shown in Fig. 9(a) and (b), respectively. It can be seen that the as-deposited and re-structured carbon coatings both contained the nanocrystallites with graphene sheets vertically oriented to the coating surface. In the nanocrystallites, graphene sheets were interconnected by cross-linking bonds, which has been studied in detail in the previous works [32,33]. After oxygen plasma etching, the chemical structure evolution would cause the re-structure in coating surface. However, the change was not obviously observed from TEM images. The reason was probably that the effect depth or transformation extent induced by plasma etching was not large enough to be distinguished easily.

The re-structure process in the coating surface was inferred as follows. The oxygen plasma may preferentially break the *sp*² bonding carbon, which is proved by the decrease of the *sp*² content after oxygen plasma etching. Then, the formed dangling bond may connect with a carbon atom or oxygen atom to form new bond. When the new bond connection occurred between different parallel basal planes, the cross-linking increased. In order to visualize the re-structure process in the coating surface, Fig. 10 gives a simplified model illustration when the *sp*² bond in graphene sheets was etched and the new connection occurred between different parallel layers. After the interlayer

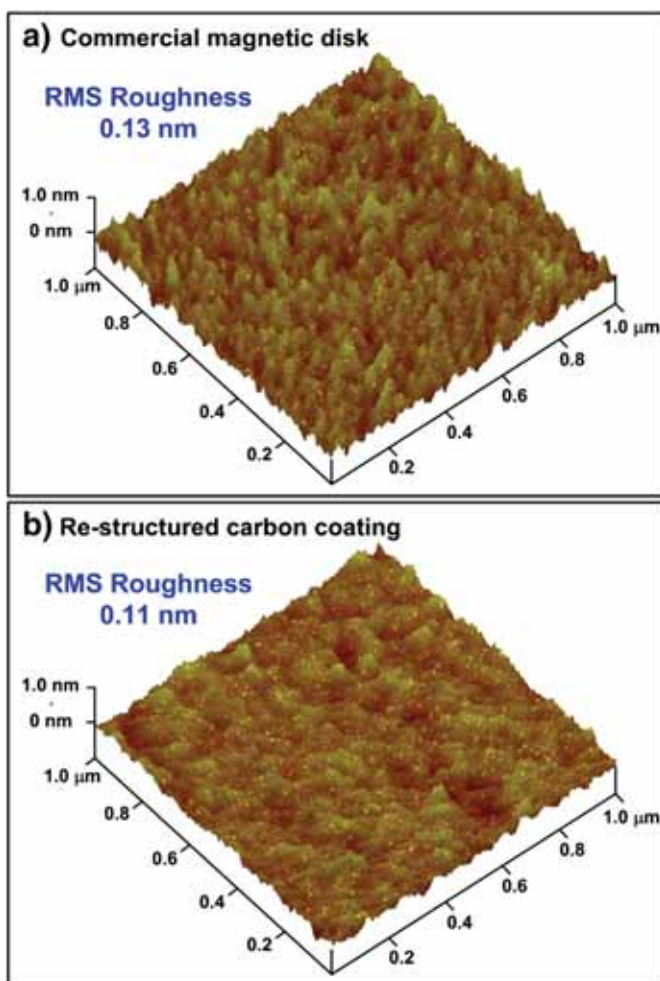


Fig. 7. Surface topographies of (a) the commercial magnetic disk and (b) the re-structured carbon coating on uncoated magnetic disk substrate.

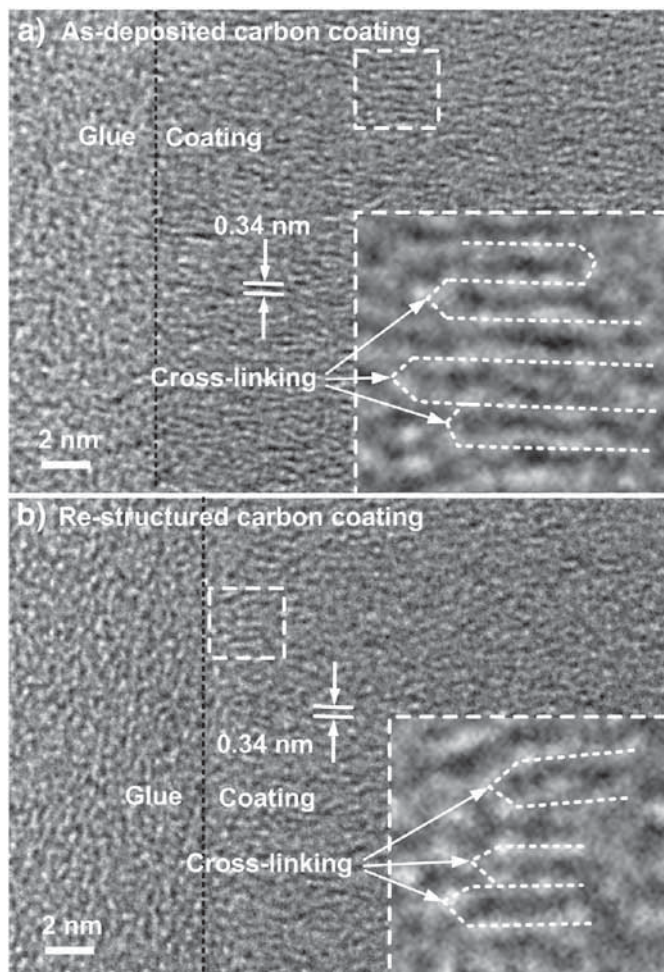


Fig. 9. Cross-sectional TEM images of (a) the as-deposited carbon coating and (b) the re-structured carbon coating.

interconnection, the original weak van der Waals force between different parallel layers changed into covalent bond force, which extended the extraordinary strength of a planar sp^2 -coordinated carbon network into three dimensions and improved the scratch resistance of carbon

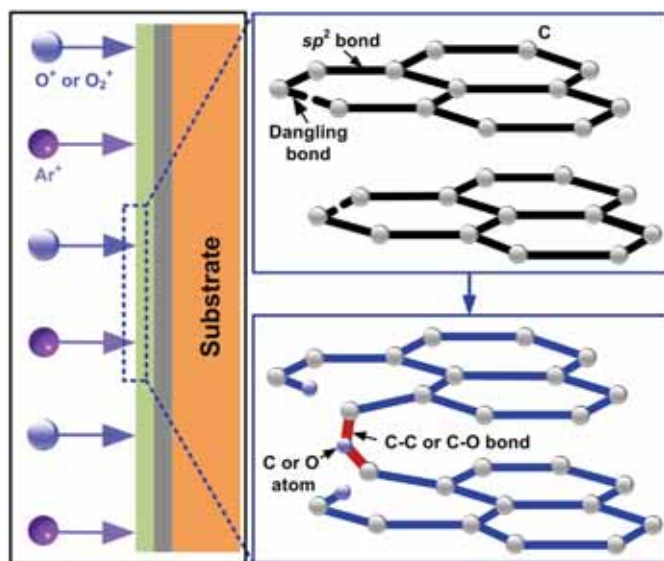


Fig. 10. Simplified model illustration of re-structure process in the coating surface.

coating obviously [34]. Therefore, the scratch depths of re-structured carbon coating effectively decreased. However, the re-structure was not clearly observed by TEM. This is because the re-structure process just occurred near the top surface and the degree of the structural evolution was not large enough to be observed in the TEM images. And further work on identifying the structural difference near the top surface will be carried out with future effort.

5. Conclusions

An oxygen plasma etching technology in ECR plasma sputtering system was developed to prepare ultrathin carbon coatings for magnetic disk application. This technology enables us to obtain ultrathin carbon coatings easily with etching rate of 0.45 nm/min. Compared with the as-deposited carbon coating, the re-structured carbon coating after oxygen plasma etching got an improved scratch resistance. The reason was that after oxygen plasma etching, the $C=C$ (sp^2) content decreased from 55% to 17%, the $C-C$ (sp^3) content increased from 33% to 58% and the $C-O$ content increased from 7% to 18%. Such an increase in sp^3 bonding carbon contributed to the improved scratch resistance. Then the re-structured carbon coatings by oxygen plasma etching were prepared on uncoated magnetic disk substrates and showed a better scratch resistance than the commercial magnetic disk and a low surface roughness of 0.11 nm, indicating that the oxygen plasma etching technology can be used to prepare ultrathin carbon protective coatings with high scratch resistance and low surface roughness for magnetic disk application.

Acknowledgment

The authors would like to thank the National Nature Science Foundation of China with grant numbers of 91323303 and 51305332. The authors also wish to thank Dr. S. Hirono for helpful discussion.

References

- [1] A.C. Ferrari, Surf. Coat. Technol. 180–181 (2004) 190–206.
- [2] J. Robertson, Thin Solid Films 383 (2001) 81–88.
- [3] C. Casiraghi, A.C. Ferrari, R. Ohr, D. Chu, J. Robertson, Diamond Relat. Mater. 13 (2004) 1416–1421.
- [4] M. Zhong, C.H. Zhang, J.B. Luo, X.C. Lu, Appl. Surf. Sci. 256 (2009) 322–328.
- [5] H.S. Zhang, K. Komvopoulos, J. Appl. Phys. 106 (2009) 093504.
- [6] R. Brunner, F.E. Talke, Tribol. Lett. 40 (2010) 23–29.
- [7] L. Yang, D.F. Diao, W.J. Zhan, Tribol. Lett. 46 (2012) 329–335.
- [8] J. Robertson, Tribol. Int. 36 (2003) 405–415.
- [9] E. Rismani, M.A. Samad, S.K. Sinha, R. Yeo, H. Yang, C.S. Bhatia, Appl. Phys. Lett. 101 (2012) 191601.
- [10] M.A. Samad, E. Rismani, H. Yang, S.K. Sinha, C.S. Bhatia, Tribol. Lett. 43 (2011) 247–256.
- [11] J. Robertson, Diamond Relat. Mater. 2 (1993) 984–989.
- [12] Y. Lifshitz, T. Köhler, T. Frauenheim, I. Guzman, A. Hoffman, R.Q. Zhang, X.T. Zhou, S. T. Lee, Science 297 (2002) 1531–1533.
- [13] F. Banhart, Rep. Prog. Phys. 62 (1999) 1181–1221.
- [14] M. Mozetic, Vacuum 71 (2003) 237–240.
- [15] H. Fredriksson, D. Chakarov, B. Kasemo, Carbon 47 (2009) 1335–1342.
- [16] V. Chirila, G. Marginean, W. Brandl, Surf. Coat. Technol. 200 (2005) 548–551.
- [17] J.I. Paredes, A. Martinez-Alonso, J.M.D. Tascon, Langmuir 23 (2007) 8932–8943.
- [18] Y. Liou, A. Inspektor, R. Weimer, D. Knight, R. Messier, J. Mater. Res. 5 (1990) 2305–2312.
- [19] L. Jiang, A.G. Fitzgerald, M.J. Rose, R. Cheung, B. Rong, E.V. Drift, Appl. Surf. Sci. 193 (2002) 144–148.
- [20] S.A. Cateledge, R. Vaid, P. Diggins IV, J.J. Weimer, M. Koopman, Y.K. Vohra, J. Mater. Sci. Mater. Med. 22 (2011) 307–316.
- [21] X. Fan, D.F. Diao, K. Wang, C. Wang, Surf. Coat. Technol. 206 (2011) 1963–1970.
- [22] C. Wang, D.F. Diao, X. Fan, C. Chen, Appl. Phys. Lett. 100 (2012) 231909.
- [23] M. Matsuoka, K. Ono, J. Vac. Sci. Technol. A 6 (1988) 25–29.
- [24] H. Nakazawa, Y. Yamagata, M. Suemitsu, M. Mashita, Thin Solid Films 467 (2004) 98–103.
- [25] J.J.S. Acuña, C.A. Figueroa, D. Biggemann, M.U. Kleinke, F. Alvarez, J. Appl. Phys. 103 (2008) 124907.
- [26] J.J.S. Acuña, M. Escobar, S.N. Goyanes, R.J. Candal, A.R. Zanatta, F. Alvarez, J. Appl. Phys. 109 (2011) 114317.
- [27] S. Stankovich, D.A. Dikin, R.D. Piner, K.A. Kohlhaas, A. Kleinhammes, Y. Jia, Y. Wu, S.T. Nguyen, R.S. Ruoff, Carbon 45 (2007) 1558–1565.
- [28] D.R. Dreyer, S. Park, C.W. Bielawski, R.S. Ruoff, Chem. Soc. Rev. 39 (2010) 228–240.

- [29] S. Park, K.S. Lee, G. Bozoklu, W. Cai, S.T. Nguyen, R.S. Ruoff, *ACS Nano* 2 (2008) 572–578.
- [30] J.I. Paredes, A. Martinez-Alonso, J.M.D. Tascon, *J. Mater. Chem.* 10 (2000) 1585–1591.
- [31] A. Nourbakhsh, M. Cantoro, T. Vosch, G. Pourtois, F. Clemente, M.H. van der Veen, J. Hofkens, M.M. Heyns, S.D. Gendt, B.F. Sels, *Nanotechnology* 21 (2010) 435203.
- [32] S. Hirono, S. Umemura, M. Tomita, R. Kaneko, *Appl. Phys. Lett.* 80 (2002) 425.
- [33] C. Chen, D.F. Diao, *Tribol. Lett.* 50 (2013) 305–311.
- [34] Q. Zhang, D.F. Diao, L. Yang, *Surf. Coat. Technol.* 237 (2013) 230–233