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Influence of Target Current on the Structure of Ti-Doped DLC Films

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Abstract. Ti-doped DLC films were synthesized from C_2H_2 and Ti by ion beam deposition and magnetron sputtering, and the influence of Ti target current on the structures and the friction coefficients of Ti-doped DLC films were studied by SEM, AES, XPS, a ball-on-disk friction tester. The Ti content in Ti-doped DLC films is below 1at% when Ti target currents rise up to 3.5 A; but when Ti target current exceeds 3.5 A, Ti content in the films is obviously increased with increasing Ti target current. Ti-doped DLC films have a smooth dense surface with several particles dispersed in it and the surface morphology deposited with different Ti target currents is similar. With the augment of Ti target current, the ratio of sp^3-C to sp^2-C is almost a constant first, and then an obvious rise followed by an abrupt decrease is found; but only when Ti target current is above 5A, the ratio of TiC-C to sp^2-C is obviously increased with Ti target current. The lowest friction coefficients are obtained for the Ti-doped DLC films with the highest ratio of sp^3-C to sp^2-C and a low ratio of TiC-C to sp^2-C.

Introduction

Diamond-like Carbon (DLC) films have been widely used on machinery parts, cutting blades, and moulds for their high hardness and good wear resistance [1-2]. But high stress in the films, poor film-substrate adhesion, high friction coefficient prevents them from applying under severe service conditions. Amongst the methods for the improvement of the mechanical properties of DLC films, the introduction of metal elements into pure DLC films is intensively studied for metal-doped DLC films have an immediate properties between pure DLC and carbide, which can solve the conundrum of the application of DLC films under severe service conditions [3-4]. Ti-doped DLC films are the attractive solid lubricate coating materials for their low friction coefficient, high wear resistance [5], and much emphasis has been attached upon the new fabrication methods of Ti-doped DLC films [5-6]. The films deposition by a linear anode layer ion source is easy to be translated to mass production for many industrial sputtering devices are equipped with linear anode layer ion sources as the ionization and activation sources of reactive gases [7]. The deposition of DLC films using a linear anode layer ion source was developed and the properties of DLC films have be improved by introducing Ti into DLC by magnetron sputtering [8], and the optimization of the deposition process are in progress. The influence of Ti target current on the structures and friction coefficients of Ti-doped DLC films fabricated by ion beam deposition and magnetron sputtering is studied in the paper.

Experimental

DLC films were deposited through the decomposition of ethyne by the linear anode layer ion source, and the doping of Ti was realized by the sputtering of Ti target. Argon with a purity of 99.99% and ethyne with a purity of 99.5% were feed into the vacuum chamber through the linear anode layer ion source and the operation parameters of ion source were determined on the basis of the deposition experience of pure DLC films. The Ti content in DLC films was controlled by adjusting Ti target current. The substrates of 316 stainless steel were first cleaned by argon ion bombardment and a gradual transition layer was deposition before synthesis of Ti-doped DLC films in order to further improve the adhesion between the films and their substrates. The total thickness of Ti-doped DLC films was controlled at about 1.7 mm.

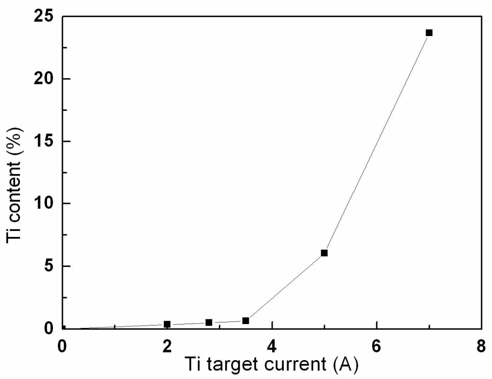

Fig. 1 variety of Ti content in Ti-doped DLC films with Ti target current

The composition of the films was measured with a PHI700 Scanning Auger Nano-probe, and the top surface with a thickness of 10nm was sputtered by argon ion before analysis in order to remove the contaminant. The surface morphology of the films was observed using a SIRON-200 scanning electron microscope (SEM) with an acceleration voltage of 10kV. The bonding status of carbon atoms in the films was analyzed using a PHI Quantera SXM X-ray photoelectron spectrograph, and the top surface with a thickness of 5nm was sputtered before the analysis. The friction coefficients of Ti-doped DLC films in dry air were studied with a MS-T3000 ball-on-disk friction tester; the counterpart was Si_3N_4 balls of 6mm in diameter; the coated 316 stainless steel samples were fixed on a rotary sample stage with a rotation rate of 400rpm and the test duration was set as 30min.

Results and Discussion

The Ti contents in Ti-doped DLC films as a function of Ti target current are shown in fig. 1. It can be found that when Ti target current rises up to 3.5A, Ti content in the films is increased slowly, and a low Ti doping level of 0.65% is obtained when Ti target current is 3.5A; but Ti content is augmented rapidly with increasing Ti target current from 3.5A. The reason why the different rise rates of Ti content with Ti target current appear is for that the sputtered surface on Ti target is distinguishing. During the sputtering of Ti target, there simultaneously exist two inverse processes on the target surface, i.e., sputtering and deposition; when Ti target is sputtered with a low target current, and the effect of the reaction product deposition on target surface surpass the effect of sputtering, which makes the sputtered atoms from Ti target surface quite low and the sputtered atoms include carbon atoms in addition to Ti atoms, so fewer Ti atoms are introduced into DLC films; with the rise of Ti target current, the sputtering of Ti target gradually becomes dominant, and when the fresh Ti atoms substitutes the compound deposit on target surface, an abrupt change of increase rate of Ti content in the films happens, which makes controlling Ti content by Ti target current close to the critical target current difficult. If the ideal Ti contents were close to the Ti content corresponding to the critical Ti target current, other measures should be taken.

The surface morphology of Ti-doped DLC films deposited with different Ti target current is shown in fig. 2. It can be found that Ti-doped DLC films have a smooth dense surface with several particles with a dimension of about 4micron dispersed in the films. The difference of the size and the amount of the particles in DLC films deposited with different Ti target currents is unobvious. If the particles are formed during the deposition of the top layer of Ti-doped DLC films due to the sputtering target arcing induced by target poisoning, the size and the amount of the particles should be obviously affected by Ti target current since Ti target current has an obvious influence of the target poisoning state; so it is concluded that the particles should be formed prior to the deposition of the top DLC

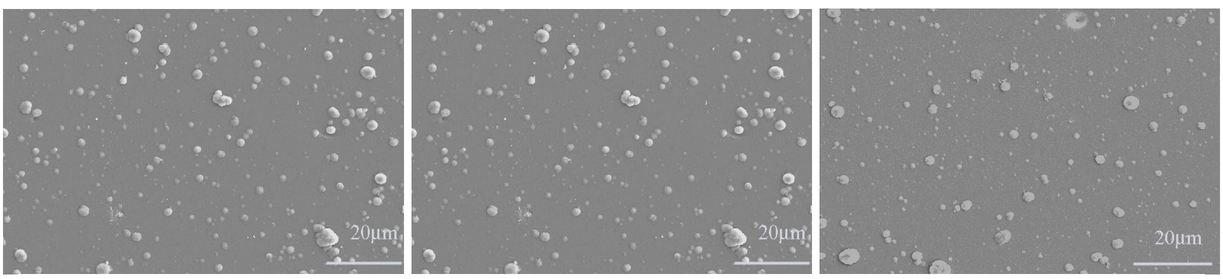

Fig. 2 surface morphology of Ti-doped DLC films deposited with a Ti target current of (a, left) 0A, (b, middle) 2A and (c, right) 5A

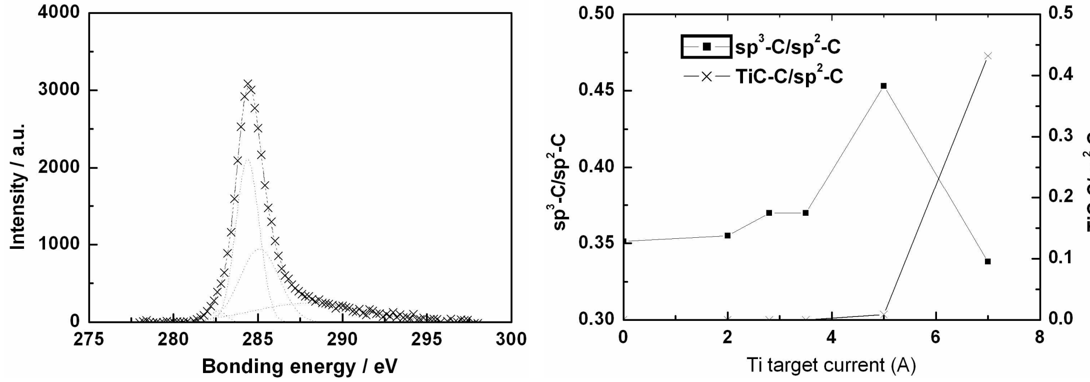

Fig. 3 XPS analysis of Ti-doped DLC films: (a) C_{1s} XPS spectrum of Ti-doped DLC films deposited with a target current of 5A, (b) ratio of sp^3-C and TiC-C to sp^2-C as a function of Ti target current

films, and then the particles is covered with a layer of Ti-doped DLC films whose composition is approximately same as the other zones. The harm of this kind of particles for tribological properties is not so obvious as that produced during the deposition of the top layer of Ti-doped DLC films.

C_{1s} XPS spectrum of Ti-doped DLC films deposited with a target current of 5A is shown in fig. 3a. And the spectrum can be deconvoluted into four components TiC, C1, C2, and C3. TiC component at 282.6 eV reveals the presence of titanium carbide components in Ti-doped DLC films. The C1, C2 components centered at 284.4eV and 285.1eV with FWHM of 1.4eV and 2.3eV correspond to sp^2 bonded carbon(sp^2-C) and sp^3 bonded carbon(sp^3-C) respectively. The C3 peak at 288.0 eV is attributed to C–O bonding. The percentage of different carbon bond status is calculated with the ratio of the area of carbon atoms with a specific bond status to the total area of carbon, and the results are shown in fig. 3b. With the augment of Ti target current, the ratio of sp^3-C and TiC bonded carbon to sp^2 bonded carbon is increased very slowly first, an obvious rise of the ratio of sp^3-C to sp^2-C followed by a rapid reduction is found when the Ti target rises from 3.5A to 7A, and TiC-C to sp^2-C starts to go up markedly when Ti target current is increased from 5A. The DLC films deposited with a Ti target current of 5A exhibit the highest ratio of sp^3-C to sp^2-C and a low ratio of TiC-C and sp^2-C.

The friction coefficients of Ti-doped DLC films are shown in fig. 4. From fig. 4, we can find that the friction coefficients of the films keep stable first, then are reduced remarkably, and the lowest friction coefficients are obtained for the Ti-doped DLC films deposited with a Ti target current of 5A; when Ti target current is further increased, the friction coefficients of Ti-doped DLC films go up again. The Ti-doped DLC films with the lowest friction coefficients correspond the highest ratio of sp^3-C to sp^2-C and a low ratio of TiC-C and sp^2-C.

Conclusion

Ti content in Ti-doped DLC films is below 1 at% when Ti target currents is below 3.5 A, and when Ti target currents exceed 3.5 A, Ti content are obviously increased with increasing Ti target current. Ti-doped DLC films have a smooth dense surface with several particles dispersed in it and the influence of Ti target current on the surface morphology is unobvious. With the augment of Ti target current, the ratio of sp^3-C to sp^2-C is almost constant first, and then an obvious rise followed by an abrupt decrease is found; only when Ti target current is above 5A, the change of the ratio of TiC-C to sp^2-C is obvious. The lowest friction coefficients are obtained for the Ti-doped DLC films deposited with a Ti target current of 5A.

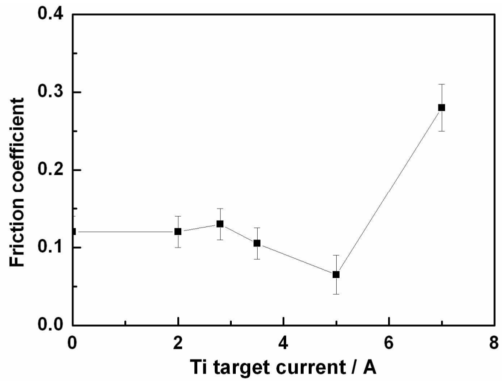

Fig. 4 friction coefficients of Ti-doped DLC films

Acknowledgements

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References

[1] A. H. Lettington. Carbon Vol.36 (1998), p.555
[2] Z.H. Yuan, Z. Q. Fu, Q. S. Deng, et al.: J.Guangdong Non-ferrous Metals Vol. 11 (2001), no. 1, p.55
[3] B. K. Tay, Y. H. Cheng, X. Z. Ding, et al. Diamond Relat. Mater. Vol. 10(2001), p.1082
[4] M. Kalin, J. Vizintin. Thin Solid Films Vol. 515 (2006), p. 2734
[5] J. H. Ouyang, S. Sasaki. Surf. Coat. Technol. Vol. 195 (2005), p. 234
[6] A. M. Peters, M. Nastasi. Surf. Coat. Technol. Vol. 167 (2003), p.11
[7] M. K. Yin, M. Li, C. W. Zou, et al. Journal of Physics D: Applied Physics Vol. 41(2008), p. 22
[8] S. S. Sheng, M. J. Dai, H. J. Hou, et al. Chin. J. Vacuum Sci. Technol. Vol. 27, (2007), p. 418