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W-doped DLC Films by IBD and MS

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Abstract. W-doped DLC films were synthesized from ethyne and tungsten by ion beam deposition and magnetron sputtering, and the influence of W target current on the structures and the properties of W-doped DLC films were studied. There exist some defects smaller than 3micron in W-doped DLC films and the influence of W target current on the defects is unobvious. The W content in the films is tardily increased with W target current below 3.5A, and then acutely rises with W target current. When target current is below 3.5A, the ratio of sp^3-C to sp^2-C is first decreased and then increased with the rise of target current, and the ratio of WC-C to sp^2-C is close to 0; but when the target is above 3.5A, the ratio of sp^3-C to sp^2-C is decreased and the ratio of WC-C to sp^2-C is augmented with further increasing target current. The hardness and the modulus is first decreased with target current and the minimum value is reached for the W-doped DLC films deposited with a target current of 2.6A. The W-doped DLC films deposited with a target current of 2.6A exhibit the best film-substrate adhesion. The W-doped DLC films deposited with a low target current exhibit a friction coefficient while the wear resistance of the W-doped DLC films deposited with a medium target current of 2.6A is best.

Introduction

The applications of diamond-like Carbon (DLC) films, which is the ideal wear-resistant coatings for precise machinery parts, cutting tools, and et al for their high hardness, low friction coefficient and good wear resistance [1], has been restricted by their poor film-substrate adhesion, high residual stress, low toughness[2]. The introduction of W into pure DLC films can greatly improve the synthetic properties of DLC films through forming a WC/a-C composite structure, reducing the structural differences at film/metal interface and cutting down the residual stress in the films [3], which is one of the fascinating methods to overcome the key technological difficulties for the application of DLC films. And verifying the correlation between the fabrication process and the performance of W-doped DLC films is a hotspot of the fangle DLC films technology [4-5]. A hybrid deposition method composed of ion beam deposition (IBD) and magnetron sputtering (MS) is a hopeful industrial deposition method for its large deposition area and good properties [6]. In the study, W-doped DLC films were synthesized by ion beam deposition and magnetron sputtering, and the correlation between W target current and the performance of W-doped DLC films were studied.

Experimental

W-doped DLC films were synthesized through ion beam deposition using an anode layer ion source and W doping by magnetron sputtering. Argon with a purity of 99.99% and ethyne with a purity of 99.5% were feed into the vacuum chamber through an 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 W content in the films was controlled by W target current. The substrates of on 316 stainless steel were carefully cleaned and a gradual transition layer was fabricated before the synthesis of W-doped DLC films in order to further improve the film-substrate adhesion. The total thickness of W-doped DLC films was controlled at about 2.6 micron.

The surface morphology of the films was observed using a SIRON-200 scanning electron microscopy (SEM) using an acceleration voltage of 10kV. The composition and the carbon bonding status of the films were analyzed by PHI Quantera SXM X-ray photoelectron spectroscopy (XPS). The hardness and the modulus of the films were measured with a MTS XP nano-indentation tester with an indentation depth of 1000nm. The adhesion between W-doped DLC films and 316 stainless steel was evaluated by a MFT-4000 multi-functional material property tester under the conditions with a loading rate of 40N/min, an end load of 100N and a scratch length of 5mm. The tribological behaviors of coated samples in dry air were studied with a MS-T3000 ball-on-disk friction tester; the counterpart is a Si_3N_4 ball of 6mm in diameter; the coated 316 stainless steel plates were fixed on a rotary sample stage with a rotation rate of 400rpm and the test duration was set as 30min; the friction coefficients were recorded continuously during the test and the wear volume was established by the perimeter and the cross-section area of the worn trace.

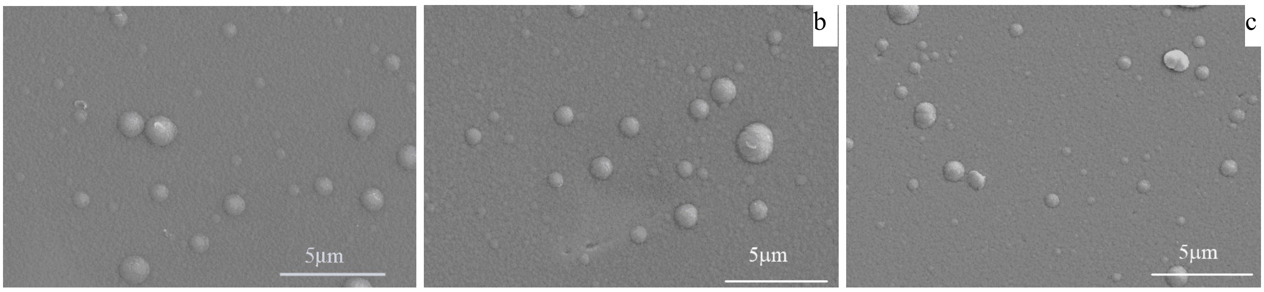

Fig. 1 surface morphology of W-doped DLC films deposited with W target current of: (a) 0A, (b) 3.5A, (c) 5A

Results and Discussion

Fig. 1 shows the surface morphology of W-doped DLC films deposited with different W target currents. There exist several defects including particles and pit on the film surface, whose size is less than 3 micron; and the difference amongst the surface morphology of W-doped DLC film deposited with different W target current is unobvious. The composition analysis of different zone by AES shows that the composition in the defect zones is similar to that in the defect-less zones even after the films are sputtered for 200nm, which means the defects should be formed before the deposition of the top W-doped DLC layer and a layer of W-doped DLC is deposited on the defect surface.

W contents and the ratio of carbon with different bonded status to sp^2 bonded carbon (sp^2-C) in W-doped DLC films as a function of W target current are shown in fig. 2. It can be found that W contents in W-doped DLC films rise slowly with W target current first, and then are increased markedly with W target current after W target current is above 3.5A. When W-doped DLC films are deposited with a low target current, a layer of compound deposit formed on the target surface obviously reduces the sputtering efficiency, and the sputtered W amount is small for it depends on both the sputtering target current and the sputtering efficiency. But when the target current is increased to a critical

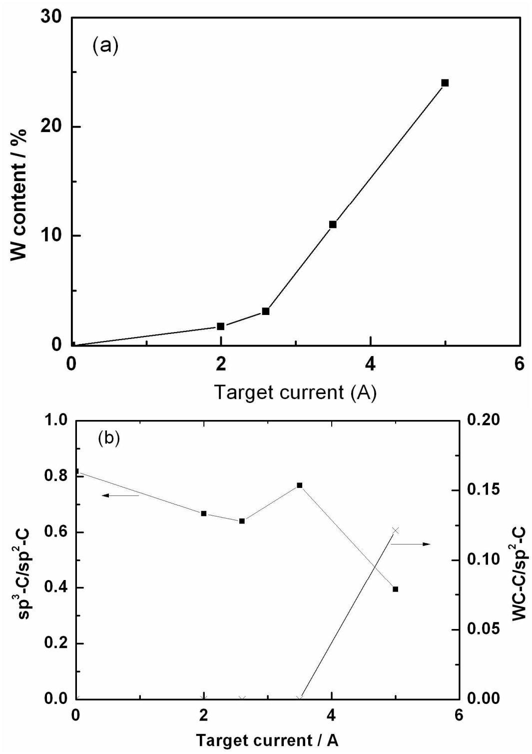

Fig. 2 tungsten content and carbon atom bonding status of W-doped DLC films: (a) tungsten content, (b) ratio of sp^3-C to sp^2-C and WC -C to sp^2-C value or above, the sputtering effect on the target surface surpasses the deposition effect, and a layer of compound deposit on the target surface is substituted by the fresh tungsten atoms, which makes an abrupt increase of the sputtering efficiency, and W contents start to go up rapidly with W target current.

When W target current is below 3.5A, the variety curve for the ratio of sp^3 bonded carbon (sp^3-C) to sp^2-C as a function of W target current exhibits a minimum, and the ratio of WC bonded carbon (WC-C) to sp^2 bonded carbon (sp^2-C) is almost equal to 0; but an obvious decrease in the ratio of sp^3-C to sp^2-C and a abrupt increase in WC-C to sp^2-C with the rise of target current are found when the target current is augmented from 3.5A to 5A, which means WC crystalline starts to be precipitated in the films.

The hardness, the modulus and the film-substrate adhesion of W-doped DLC films are shown in fig. 3. The lowest hardness and the lowest modulus are found for the W-doped DLC films deposited at a target current of 2.8A, which corresponds to the structure of the minimum ratio of sp^3-C to sp^2-C and a low ratio of WC-C to sp^2-C as mentioned above. The scratch test shows a best film-substrate adhesion (L_c=100N) for the W-doped DLC films deposited with a target current of 2.8A, which is attributed to the stress relax in films through the opportune introduction of W atoms into DLC matrix; but when the brittle WC crystalline starts to be precipitated in the films, the film-substrate adhesion becomes worse.

The tribological properties and worn surface morphology of W-doped DLC films are shown in fig. 4. The W-doped DLC films deposited with a low W target current exhibit a low friction coefficient, but the friction coefficient of W-doped DLC films is abruptly increased to above 0.2 when the target current is above 2.6A. The W-doped DLC films deposited with a target current of 2.6A, which have the highest film-substrate adhesion, the minimum hardness and the lowest modulus, exhibit a minimum wear rate. From the worn surface morphology, we can find that there exist some fractured zones and debonded zones on the worn surface, and the mass loss is mainly caused by the fracture and debonding of the films during the friction process, so the toughness and the film-substrate adhesion are the key controlling factors of wear rate instead of the hardness. Therefore a high film-substrate adhesion is more important than a high hardness for the improvement of the wear resistance of DLC films.

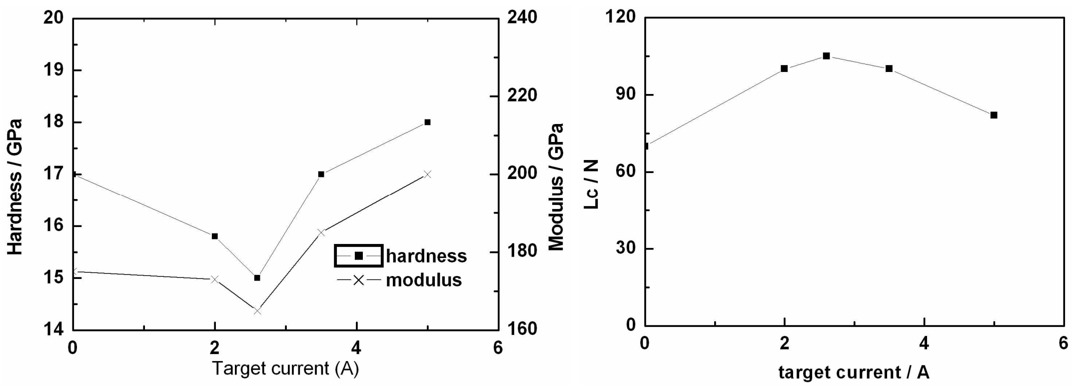

Fig. 3 mechanical properties of W-doped DLC films: (a) hardness and modulus, (b) critical load

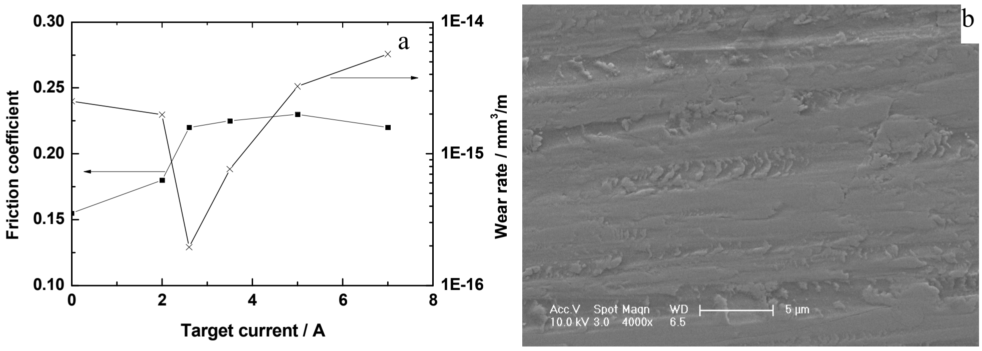

Fig. 4 tribological properties and worn surface morphology of W-doped DLC films

Conclusion

The influence of W target current on the size and the amount of the defects in W-doped DLC films is unobvious. W contents in the films first gradually rise with W target current in the range of low W target current and then the rise rate becomes rapid at higher W target current. When W target current is below 3.5A, the DLC films deposited with a target current of 2.6A exhibit a minimum ratio of sp^3-C to sp^2-C and the ratio of WC-C to sp^2-C is close to 0; but when W target current is above 3.5A, the ratio of sp^3-C to sp^2-C is decreased and the ratio of WC-C to sp^2-C is augmented with increasing target current. The hardness and the modulus is first decreased with W target current and the minimum values are reached for the W-doped DLC films deposited with a target current of 2.6A; and the W-doped DLC films deposited with a target current of 2.6A exhibit the best film-substrate adhesion. The friction coefficients are found for W-doped DLC films deposited with a low target current, and the wear resistance of the W-doped DLC films deposited with a medium target current of 2.6A is best.

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References

[1] Z.H. Yuan, Z.Q. Fu, Q.S. Deng, et al.: J. Guangdong Non-ferrous Metals Vol. 11 (2001), no. 1, p.55
[2] A. H. Lettington. Carbon Vol.36 (1998), p.555
[3] Y. T. Pei, X. L. Bui, X. B. Zhou, et al. Surf. Coat. Technol. Vol. 202 (2008), p.1869
[4] K. Bewilogua, C.V. Cooper, C. Specht, et al. Surf. Coat. Technol. Vol. 127(2000), p.224
[5] K. Baba, R. Hatada, Y. Tanaka. Surf. Coat. Technol. Vol. 201 (2007), p. 8362–8365
[6] M. K. Yin, M. Li, C. W. Zou, et al. Journal of Physics D: Applied Physics Vol. 41(2008), p. 22