Solid State Science and Technology, Vol. 15, No 2 (2007) 22-27
ISSN 0128-7389

SYNTHESIS OF DIAMOND COATING FROM METHANE GAS IN MICROWAVE PLASMA ENVIRONMENT

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ABSTRACT

Thin diamond film can be employed as a protective, hard and inert coating on metallic materials. But, the application of diamond on metal, such as steel material, is restricted, mainly because of poor adhesion between diamond films and the metal substrate. Diamond film cannot be formed directly on the steel surface because the precursor gas, methane, simply diffuses into the substrate, thus hindering the diamond nucleation. In this work, a diffusion barrier of carbonitriding layer was employed as a pretreatment process before diamond deposition. It was noticed that an interlayer by plasma carbonitriding was an effective method in improving the nucleation of diamond coating on a steel substrate.

INTRODUCTION

Diamond has unique properties such as extremely good mechanical, thermal, chemical and electrical properties, which makes it a good candidate for a great number of novel applications. The nucleation and growth of diamond coatings on steel substrate can be improved with three types of surface treatment: (i) mechanical treatments such as polishing and sand blasting [1]; (ii) bias enhancement [2]; and (iii) deposition of an interlayer on the substrate. The function of an interlayer is to provide a carbon diffusion barrier layer to enhance sufficient carbon concentration for diamond nucleation and to enhance the diamond nucleation rate [3]. In this study, the effect of power on the diamond nucleation using tool steel as substrate with carbonitrided interlayer will be discussed.

EXPRIMENTAL DETAILS

In this work, an interlayer of carbonitriding layer was used in order to overcome the problem of carbon atom diffused into the steel substrate during the diamond deposition process. Commercial M2 tool steel disks with diameter 30 mm and thickness of 6 mm (1.83 wt. % C, 0.35 wt. % Co, 0.7 wt. % Mn, 7.64 wt. % Cr, 0.51 wt. % Mo, 0.88 wt. % V, 0.27 wt. % Si, 0.7 wt. % Fe were used as a substrate in this study. In the carbonating process, the samples were first polished to 1200 grid using SiC emery papers and subsequently polished to mirror finish of 6 μm using emery cloth and diamond paste. The samples were then ultrasonic cleaned with acetone for 30 minutes before commencing the carbonitriding process. For carbonitriding process, various CH_4:N_2:H_2

flow ratios were initially produced and then subjected to hardness test. The parameter set that produced the highest hardness was selected for subsequent diamond coating production. Plasma carbonitriding was performed under a microwave power of 1.5 kW and a duration of 4 hours using CH_4:N_2:H_2 flow ratios of 3:87:10 with the thickness of the carbonitrided layer was approximately 19 μm.

Prior to diamond coating process, diamond seeding was performed using ultrasonic machine with diamond suspension particle size of 15 μm. The samples were then subjected to two different microwave powers, whereas other coating parameters such as substrate temperature, deposition duration, methane hydrogen ratio and chamber pressure were kept constant. Diamond coating parameters are shown in Table 1. Each sample was subjected to microstructural examination using Filed Emission Scanning Electron Microscopy (FESEM).

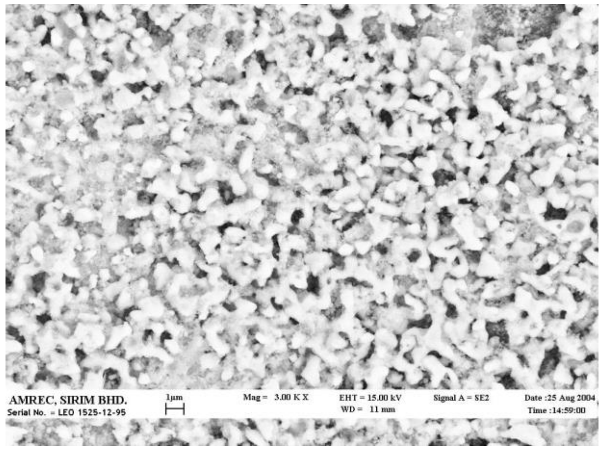
Table 1: Diamond coating parameters

Step	Sample A	Sample B	Time (hours)	Gas flow (sccm)	Pressure (Tor)
	Power (kW)				
1	2.0	3.0	4	$200H_2 : 4CH_4$	35
2	2.5	3.5	8	$200H_2 : 3CH_4$	50

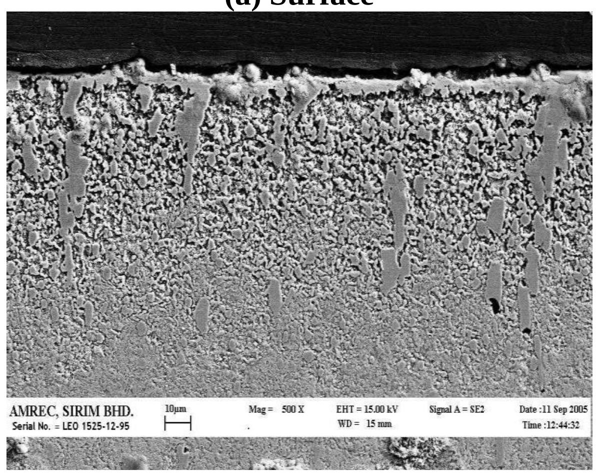
RESULTS AND DISCUSSION

SEM morphology and cross-section micrograph shows that carbonitrided layers were formed on the tool steel substrate (Figure 1). EDX analysis also shows that the carbon and nitrogen atom diffused into the subsurface of the substrate (Figure 1c). Plasma carbonitriding can generate a carbide layer, which serves as the precursor for the nucleation of diamond crystals. During plasma carbonitriding process, the surface bombardment effect can also provide nucleation sites for diamond nucleation. The formation of carbide layer such as Cr_3C_2 during thing this stage is believed to provide an effective solution to prevent the diffusion of carbon atoms into the iron matrix and iron atoms to the surface. These phenomena enhance the diamond nucleation due to saturation of carbon and nitrogen at the substrate surface.

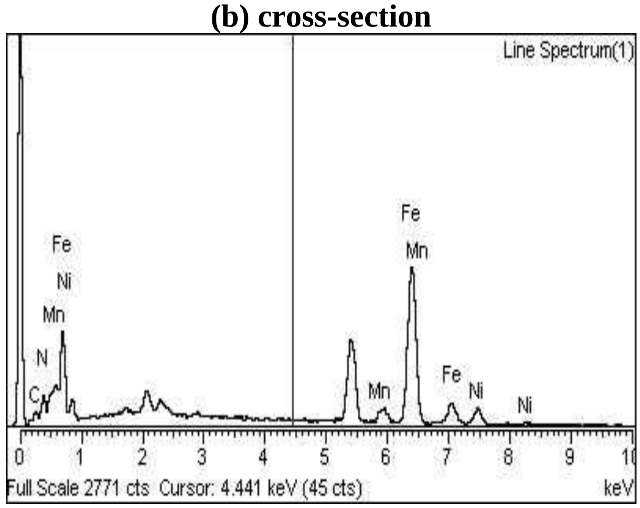
Figure 2 shows the surface morphology of the substrate under different power. In the early stage of diamond coating process, it was observed a few clusters of ball shaped features were formed on the substrate surface under the power of 2.5 kW. Subsequently as the power increased to 3.5 kW, continuous coating was formed. The balls shaped features are generally referred as graphite or microcrystalline graphite [4].



(a) Surface



(b) cross-section



(c) EDX analysis

Figure 1: SEM of carbonitrided layer and EDX analysis on carbontrided layer

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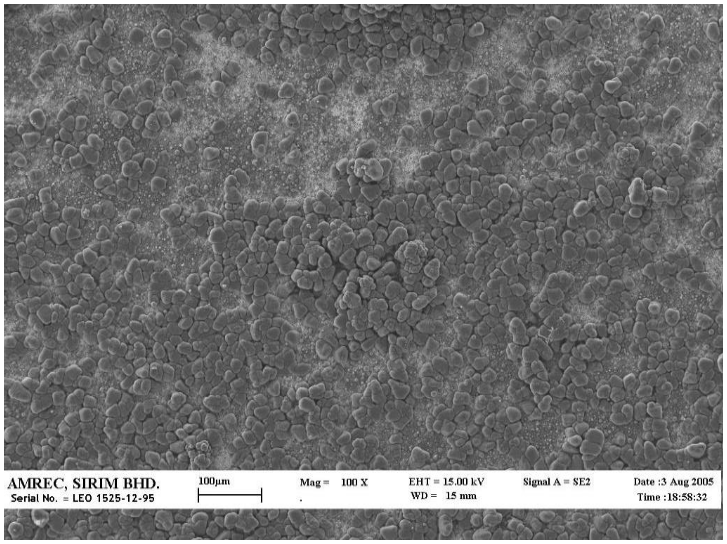
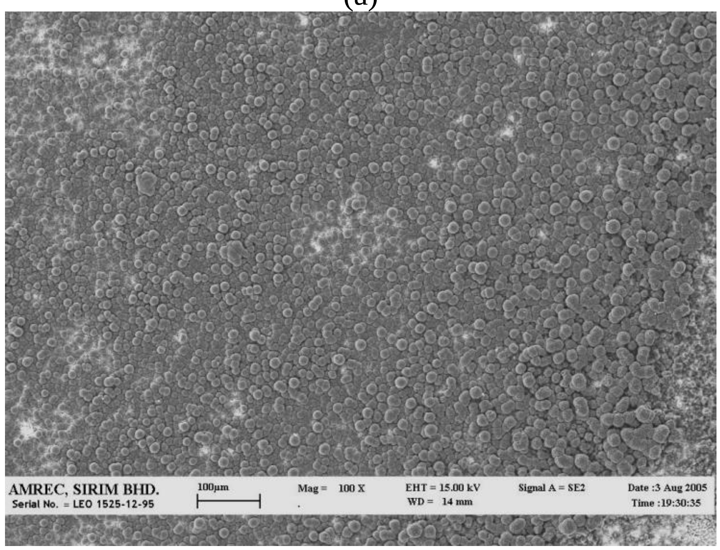


(a)



(b)

Figure 2: SEM micrograph of the substrate surface under different power; (a) 2.5 kW, (b) 3.5 kW

Figure 3 shows the surface morphology of diamond coating. Figure 3a, 3b and 3c show the surface morphology of diamond coating under power of 2.5 kW. In early stage of diamond nucleation, some ball-like with different sizes and morphology as well separated particles form rather than a continuous film were observed as shown in Fig. 3a. Subsequently, the cauliflower morphology with the initial stage of diamond nucleation can be observed and the surface is still rough as shown in Figure 3b. Finally full diamond coating was observed on the cauliflower as the diamond coating time increases. Whereas under the power of 3.5 kW, continuous diamond coating was observed on the substrate surface as shown in Figure 3d.

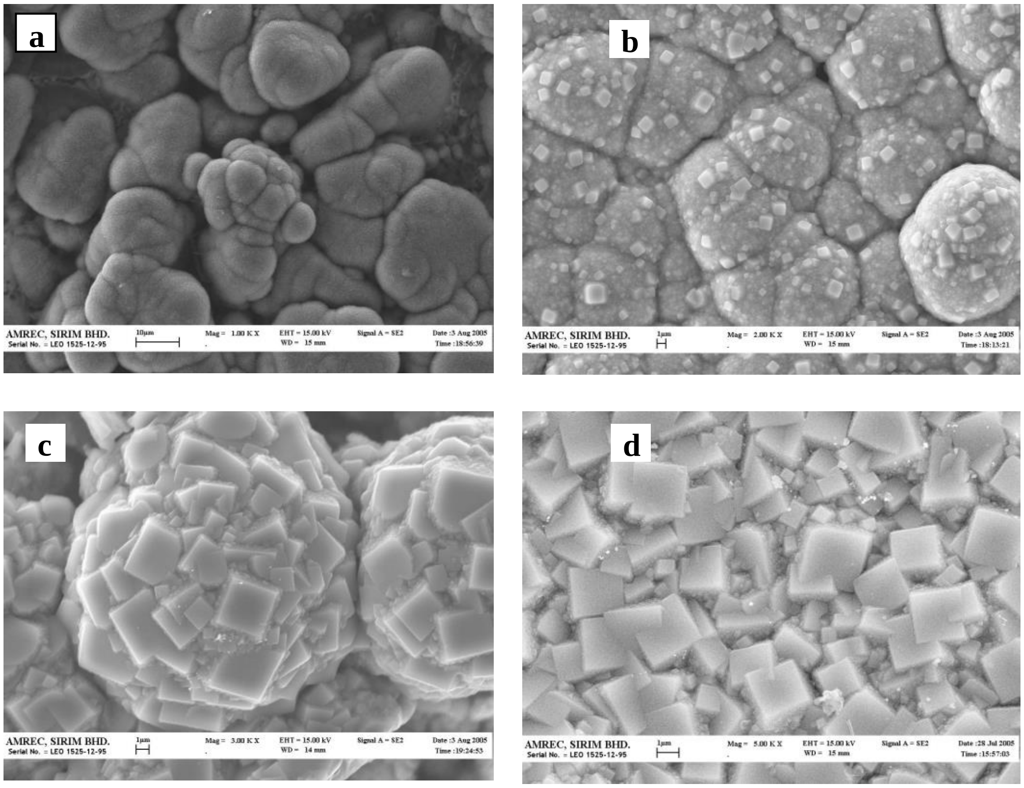


Figure 3: Surface morphology of the diamond coating: (a) early stage of diamond nucleation, power 2.5 kW, (b) generation cauliflower morphology with initial stage of diamond nucleation, power 2.5 kW, (c) diamond coating on the cauliflower, power of 2.5 kW, and (d) continuous diamond coating, power 3.5 kW.

CONCLUSION

This study shows that plasma carbonitriding was effective method to prevent the rapid diffusion of carbon into the substrate, thus improving the nucleation rate of diamond coating. It was also observed that the formation diamond coating was improved with increasing microwave power. The diamond coating changes from cauliflower morphology to continuous diamond coating on the too steel substrate.

ACKNOWLEDGEMENT

The authors are grateful to the Government of Malaysia for funding this research project through IRPA grant 05-09-0101-0032.

REFERENCES

[1] B. Zhang, L.Y. Zhou, (1997), *Thin Solid Films* 307, 21- 28.
[2] S. Yugo, T. Kanai, T. Kimura, T. Muto, (1991), *Appl. Phys. Lett.* 58, 1036.
[3] I.Y. Konyashin, M.B. Guseva, V.G. Babaev, V.V. Khvostov, G.M. Lopez, A.E. Alexenko, (1997), *Thin Solid Films 300,* 18 - 24.
[4] M. Gowri, H. Li, J.J. Schermer, W.J.P. van Enckevort, J.J. Ter Meulen., (2006), *Diamond & Related Materials* 15, 498 – 501