Advanced Materials Research *Online: 2012-12-13*
ISSN: 1662-8985, Vol. 619, pp 589-593
doi:10.4028/www.scientific.net/AMR.619.589

Preparation and Film-Substrate Stress Analysis Of CVD Diamond Film Coating Tools

Zheng You-Yi[1,a], Zhang Chun-Lin[1,b], Xu Xing-Xin[1,c]

[1]School of Mechanics & Power Engineer, Henan Polytechnic University,
Jiaozuo 454003, Henan, China
[a]zhengyy@hpu.edu.cn [b]zhangchunlin.1987@163.com [c]xuxx@hpu.edu.cn

Keywords: hot filament CVD; diamond films; finite element analysis; stress

Abstract: The diamond thin films was deposited by hot filament CVD method in the Cemented Carbide (YG6) substrate. The surface morphology and quality of the diamond film coating was detected by scanning electron microscopy and Raman spectrometer. The internal stress of the diamond thin films and Cemented Carbide substrate, exerted by load, was calculated by the method of finite element numerical analysis. The analysis result shows that the internal stress of the film is tensile stress. The maximum appear in the blade and the tip place, the stress distribution more evenly in the middle area of the film. The stress is a zonal distribution and reduced gradually from the matrix surface to bottom in the Cemented Carbide, the results of the finite element analysis can provide the reference for the other methods to determin the diamond film stress and improve the stress between the membrane and substrate.

1 Introduction

With the wide application of the new composite materials and the difficult to process materials, it put forward higher request for cutting tool in the modern manufacturing. The traditional high speed steel and Cemented Carbide cutting tools hasn't met its processing requirements of high speed and high precision. However the CVD diamond film has a lot of excellent performances, such as very close to the hardness of natural diamond, big elastic modulus, high thermal conductivity, low thermal expansion coefficient, low friction coefficient, chemical stability and very good fight acid, alkali, corrosion resistant. It is honoured as the most potential new coating material. So the diamond film coating tools become the ideal tool of processing new materials[1,2].

The failure of the coated tool often is the shedding of the coating. Numerous studies show that: the internal stress is caused by that the defects are generated by the film due to its own internal structure, with different of the thermal expansion coefficient of the substrate and the uneven distribution of the growth temperature will affect the binding force between the film coating and substrate, in severe cases, it will cause the coating to fall off, it is urgently needed to resolve the problem that how to reduce the internal stress of thin films to improve the binding force between the coating and substrate[3].In this paper, diamond film is prepared by hot filament CVD method, the size and distribution of the stress are analyzed by finite element simulation after the load is applied in the certain thickness.

2 Preparation and Characterization of CVD diamond film coated tool

The tool base material was the WC-Co6% Cemented Carbide YG6 blade, the substrate surface needed to pretreat before the diamond film was deposited. In this paper the YG6 matrix blade was pretreated by the acid-base two-steps. As follows: ①Putting the Cemented Carbide YG6 blades into the Murakam solution (mass ratio: $KOH:K_3(Fe(CN)_6):H_2O$ = 1:1:10) to ultrasonic cleaning 20min; ②The blade was putted into the $H_2SO_4:H_2O_2$ = 3:7 (volume ratio) solution to ultrasonic

cleaning 30s;③Using the distilled water to wash away the residual reaction solution on the blade; ④Acetone ultrasonic cleaning 5min. The purpose of preprocessing in order to make the substrate surface to become roughening and purification, thereby change the microstructure of the substrate surface. In the first step, the tungsten particles was broke and removed on the cutting tool surface, making tool surface to coarsen and forming a large number of defects that could increase the contact area between the reaction of gas source and substrate and the can of the substrate surface, improve the nucleation density of diamond on the cemented carbide substrate, and then enhance the adhesion of the diamond film in the substrate surface. The second step pretreatment was to remove the tool surface of cobalt, reducing the reminder graphite role of cobalt in the diamond particle deposition process and thus improving the quality of the diamond film and substrate adhesion. The last two steps pretreatment in order to purify the tool surface and remove the oxides and contaminants of the substrate surface[4,5]. The hot filament chemical vapor deposition (HFCVD) device was used the film-forming device, the reaction gas were the hydrogen and acetone. The hot wire was tantalum wire of the 0.5mm in diameter. Using four tantalum wire that the space was 20mm approximately, parallel and equidistant arrangement each other.

It can be seen from the Table 1, the deposition process of the diamond film can be divided into three stages that are the carbonization stage, the nucleation stage and the growth stage. Carbonization stage in order to reduce impurities and obtain high-quality diamond film. Because the hot wire have the oil and other impurities, if not removed in the deposition process. It would affect the quality of the deposited film. In the nucleation stage, using the lower reaction pressure and high current bias can increase the diamond nucleation density that it was conducive to generate a uniform and dense diamond film. In the growth stage, increasing the reaction gas pressure and reducing the carbon source flow that would result hydrocarbon groups adsorbed to the substrate surface was etched by a large number of Hatoms, this in order to reduce the non-diamond carbon components [6,7]. Depositing 6h and the film's thickness was about 10μm.

Table 1 The process parameters of the hot filament CVD deposit diamond films

Deposition parameters	Carbonization stage	Nucleation stage	Growth stage
Hydrogen / Acetone[sccm]	200/60	200/60	200/50
Hot filament temperature[^{0}C]	450-700	1800-2000	2100-2200
Hot filament power[W]	150-600	1500-1700	1900-2100
Substrate temperature[^{0}C]		650-750	750-850
the distance of Hot filament substrate [mm]	6	6	6
Pressure[Torr]	45	12	40
Current bias[A]		3	3
Time[h]	1	0.5	6

The diamond film coating's surface morphology was tested by scanning electron microscopy. Figure 1 shows the diamond particles is thick, dark gray, metallic luster and an irregular lozenge shape. Grain shape is basically the same, the crystal edges and grain boundaries are very clear. The tool surface and the cutting edge is covered by a layer of uniform and dense diamond film.

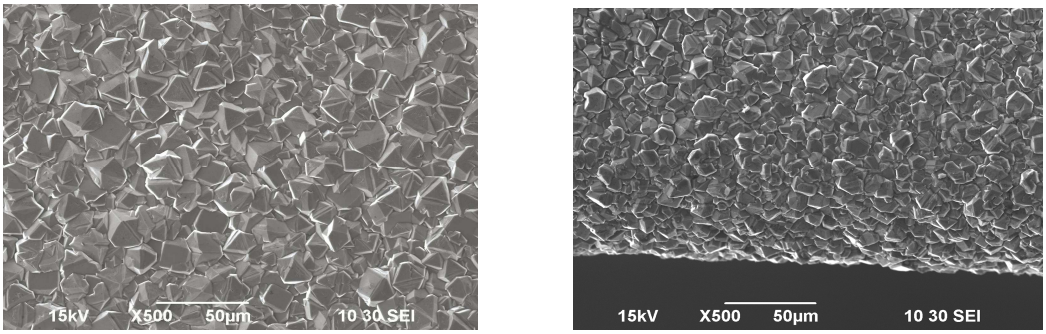

Fig .1 SEM images of the surface of the diamond film coating tool and cutting edge

The diamond films composition and quality were analyzed by using laser Raman spectroscopy. Laser Raman scattering spectroscopy play an important role for analysing the physico-chemical properties of diamond films. Raman spectroscopy is very sensitive to the carbon bond and it can produce different Raman peak to the different types of the carbon structure such as diamond, graphite, amorphous carbon ,hydrocarbons and so on. However diamond Raman peak at 1332cm$^{-1.}$ The test results of the diamond film coating tool Raman spectrum is shown in Figure 2. The section of diamond film at the tool surface and the cutting edge has a very sharp Ranman peak at 1332cm^{-1}. Which in accord with the natural diamond intrinsic peaks, indicating the good quality of diamond films. This indicates that the diamond films has a good quality. But the different is that non-diamond peaks emerge in the Raman spectra of the blade surface. That is to say there have a small amount of graphite or amorphous carbon and other impurities in this region, because of its content is very low, so it will not have an effect on the overall performance of the diamond films[8].

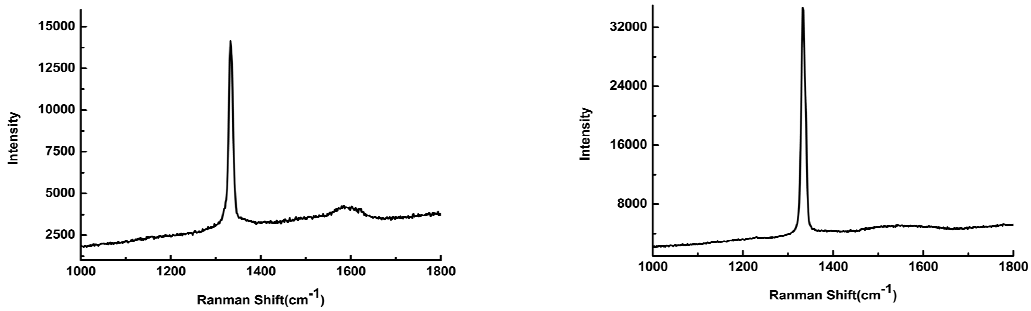

Fig .2 The Raman spectra images of the diamond thin film coating tool surface and the cutting edge

3 Stress analysis of the three-diamond thin film coating tool

The internal stress of diamond film is caused by the defects generated by the film due to its own internal structure, with different of the thermal expansion coefficient of the substrate and the growth temperature of the uneven distribution. The focus of the film stress will affect the coating and substrate's binding strength at the joint surface and reduce the adhesion of the film and substrate, reduce severe, causing the film off, in severe cases, it will cause the coating to fall off. So we need to analyze and calculate the distribution and size of the coating stress. The measure method of Stress have the Ranman spectrometry, X-ray diffraction method, film vibration France, bending method and so on. The above method of measuring stress only to obtain a point or a small area of stress message in the sample, it does not have a comprehensive understanding of the stress of the diamond film coating. Using the finite element analysis of solving continuous problem's numerical methods is an effective means to settle this problem[9,10].

3.1 Establish the finite element analysis model

The substrate material is the WC Cemented Carbide and the size is 15mm×15mm×4.5mm. Coating is the diamond films of thickness 10μm. These two kinds of material properties are shown in Table 2.The diamond film coating blades showing a geometric symmetry structure. So we could choose the longitudinal profile of the blade coating to set a plane finite element model, select the appropriate thermal analysis unit and define the material parameters.

Table 2 The material properties of the substrate and coating[11]

Material Name	Modulus of elasticity [E/GPa]	Poisson's ratio	Thermal conductivity [W/cm. k]	Coefficient of thermal expansion [10^{-6}/K]
Cemented Carbide	100	0.33	0.29	5.7
Diamond Coating	1035	0.07	20	0.81

3.2 Meshing of the model

Meshing the finite element analysis model. As the coating thickness was too thin, and it wasn't easy to be divided. So we using the same size grid on the coating and substrate. The grid of Some part of the regionals was refined on the film, especially in the point and edge.So that in the process of the subsequent calculation in order to obtain more accurate results.

3.3 Loading of the model

(1)The combination of between diamond films and the substrate surface and the connection of the different planes of the film were ideal, when they were affected by external load, the displacement of the substrate surface and film surface was zero.

(2)Coating was cooled by the role of the water from 850^{0}C to room temperature in the vacuum reactor internal, it was a steady-state thermal analysis and the process did not take into account the thermal radiation and phase change.

3.4 Viewing the solution results

Figure 5 is the temperature field contour map of the model after by the thermal load. It can be seen from the figure, the lowest temperature of the substrate is 350^{0}C, the thermal load is applied to the coating tool surface, the temperature decreases gradually after heat conduction and the temperature is zonal distribution in the substrate.

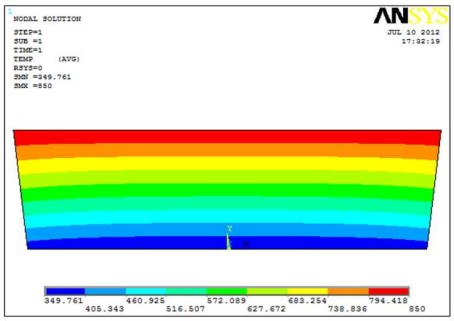

Fig 5 Temperature field contour diagram

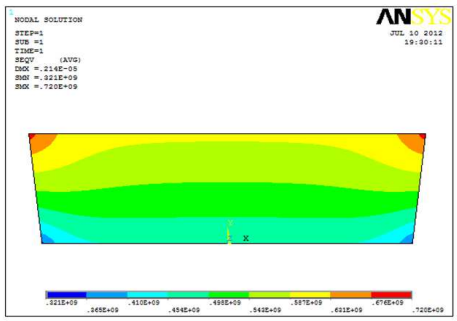

Fig 6 Stress field contour diagram

Figure 6 is the whole stress field contour diagram of the diamond film coating tool. It can be Seen from the figure that the maximum stress focus on the cutting edge and the size of the stress is 0.7GPa, from edge to middle the stress is reduced by degrees. The stress value of the middle areas is 0.59GPa, it show that the stress distribution is uniform in the diamond coating and the stress state is expressed as the tensile stress. Stress is the zonal distribution in the substrate and it is reduced

gradually from the substrate surface to downward, the average stress value is 0.4GPa. In diamond films stress is less than the tensile strength (tensile strength is 16GPa), so the diamond film should not be ruptured on the substrate [12,13].

4 Conclusion

(1)Diamond film is prepared by hot filament CVD method, we must abide The deposition process strictly and control the different of deposition parameters reasonably, especially temperature, so as to obtain good quality diamond films.

(2)The stress of the membrane-based systems is analysed by finite element simulation, as the result shows that the magnitude of the stress is GPa in the diamond film, the largest stress of the film-substrate systems concentrate at the tip of the blade, its value is 0.7 GPa. The stress of the CVD diamond film is uniform distribute in the middle regions, and the stress state is expressed as the tensile stress. Stress is reduced gradually from the surface to down in the cemented carbide substrate.

References

[1] Lv Jilei, Man Weidong, Chen Peng, Zhu Jingfeng, Wu Feifei. The Latest Development of CVD Diamond Coated on Cemented Carbide [J].Cemented Carbide ,2011,(05):321-331.

[2] Zhao Zhiyan, Deng Fuming, Lu Xuejun, Xu Guojun. Progress in the Research and Application Prospect of CVD Diamond Coating Tools [J].Cemented Carbide,2009,(4).

[3] Rash Ahmadi S, Shakeri M,Sadough A. Stress Analysis in hin Coatings and Substrate Subjected to Point Contact Load-ing(Mechanics of Scratchest)[J].Journal of Materials Processing echnology,2008,205(1-3):89-98.

[4] CHEN Sheng-li.Study on Improving Adhesive Force of CVD Diamond Film Coated Tool[J].Surface Technology,2006,(2).

[5] Sarangi S K, Chattopadhyay A, Chattopadhyay A K.Effect of pretreatment seeding and interlayer on nucleation and growth of HFCVD diamond films on cemented carbide tools[J].International Joumal of Refractory Metals &Hard Materials 2008,26(3):220.

[6] Shen Bin, Sun Fanghong, Zhang Zhiming, Shen Hesheng, Guo Songshou, Fabrication and application of CVD diamond coated cutting tools[J]. Diamond&Abrasives Engineering ,2011,(1).

[7] HU Ru-fu1,SUN Fang-hong.CVD Process for Improving Mechanical Properties of HFCVD Diamond-coated Thin Film Tools[J].Machine Design & Research,2007,(1).

[8] YANG Bao-hua, CHEN San, XIONG Yong, WANG Hong-yan, Preparation, characteristics and field emitter property of diamond films Journal of Yunnan University(Natural Sciences Edition),2010,(4).

[9] XU Feng,ZUO Dun-wen,WANG Min,LI Xiang-feng,LU Wen-zhuang,PENG Wen-wu .Study on Mechanical Polishing for CVD Diamond Thick Film and Its Residual Stresses[J].Journal of Synthetic Crystals,2004,(3).

[10] Zhu Jilei,Gou Li,Yan Shuangfeng,Ran Junguo.Progress in CVD Diamond Coated Cemented Carbide Cutting Tools[J].Cemented Carbide,2004,(3).

[11] Xue Hong-guo, Research On Deposition Of high Quality Diamond Film On Cutting Tools With Complex Shape And Their Cutting Performance.2007,2-3.

[12] Lin Zeng,Sun Shaoni,Wang Yong,Ba Dechun,Han Qingkai,Liu Chunming.Finite Element Analysis of Internal Stress of Diamond-Like Carbon Films[J].Chinese Journal of Vacuum Science and Technology.2011,(5).

[13] Zhang Zhaohui,Ansys Thermal Analysis Tutorials And Examples Of Resolution.China Railway Publishing House, 2007.