Low-stress diamond films deposited on stainless steel by a two-step dropped power process in chemical vapor deposition

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Abstract:

A continuous low-stress diamond film has been prepared on stainless steel by a novel two-step dropped power process using a Cr/CrN/CrTiAlN film as an interlayer. After the interlayer was deposited on the stainless steel, isolated diamond particles were firstly prepared under high power of 1800 W for 10 min in CVD, which has low stress, releasing most of thermal stress. Then, the low power of 1500 W was adopted to form continuous diamond films. The stress is obviously lower than that of the continuous diamond film deposited only in the first CVD process, indicating that the two-step dropped power process surely decreases the internal stress of the film. The blocky microstructure on sandblasted stainless steel and the existence of very small amount of pores in the diamond film are also beneficial to releases the stress.

Keywords: Diamond film; Interlayer; Stainless steel; Low stress

1. Introduction

Diamond is a kind of carbon material which has lots of outstanding chemical and physical properties, such as excellent corrosion resistance, extremely high thermal conductivity, biocompatibility, etc [1-3]. Stainless steel is a kind of important metal

materials, which is widely used in food industry, medical apparatus and so on. Deposition of diamond coating on stainless steel can enhance the surface properties of steel and prolong its life. However, there exist two problems to successfully deposit diamond films on it. First, the graphitic soot is easily formed at the interface due to Fe atoms' catalytic effect, which leads to poor adhesion [4,5]. Second, the difference of thermal expansion coefficient (CTE) between diamond and stainless steel is very large, which results in the large internal stress and makes the film delaminate from the substrate easily [6,7]. To solve the problems, the introduction of an interlayer is an available method.

Up to now, many kinds of interlayers have been studied, such as Cr [8], Ti [8], Al [9], Al/W [10], Al/AlN [11], CrN [12-13], etc. These interlayers can serve as diffusion barriers, blocking C inward diffusion and Fe outward diffusion and continuous diamond film can be deposited on stainless steels. However, the prepared diamond film using the interlayer has large internal stress, which results in instability in the application and the film easily detaches from the substrate. Applying Fe_2B phase as an interlayer, the low-stress film was prepared, but its quality is poor [14]. Using a SiC layer by laser cladding as an interlayer, the stress is partly relaxed, but it is difficult to obtain a low stress diamond film [15]. In all, up to now, preparing low-stress diamond film on stainless steels is still very challenging. Therefore, it is significant to find a new interlayer and process to solve it.

It was shown that materials containing aluminum element could enhance diamond nucleation rate [9] and was beneficial to the deposition of diamond film. A

Cr/CrN/CrTiAlN film is a kind of hard film which includes Al element. It has high toughness and high hardness [16], which can suppress the formation of crack and its propagation. It is advantageous to enhance the adhesion. So, in this work, the Cr/CrN/CrTiAlN film was selected as an interlayer.

Our previous study showed that the sandblasting pretreatment preferred to decrease the stress and improve the adhesion, but the stress is still high [17]. So, the sandblasting was used in the work and it still needs to look for other method to further decrease the stress. Generally, thermal stress is an important component in the stress of diamond films [13]. It is proportional to the temperature difference from deposition temperature to room temperature. In the experiment, high (low) deposition power means high (low) deposition temperature, which can produce large (small) stress. Therefore, it is anticipated that low stress diamond films can be obtained at low power. However, if the deposition power is very low, the deposition temperature will be too low to form diamond film. Our recent experiments show that the stress in the films is mainly related to the temperature when a continuous film forms, but not the temperature when diamond crystal nucleus form. So, we firstly used high power in CVD deposition to prepare isolated diamond particles to release the stress, and then we decreased the power to form continuous films. We call this process as two-step dropped power process.

2. Experiment

3Cr13 stainless steels with dimensions of $20 \times 20 \times 2 \text{ mm}^3$ were used as substrates in the experiment. In order to improve the adhesion, the substrates were firstly

sandblasted, and the process parameters can be found in our previous publication [17]. Then the Cr/CrN/ CrTiAlN interlayer was deposited on the substrates using a close field unbalance magnetron sputtering ion plating udp650 (Teer coatings Ltd). In the deposition, base pressure and working pressure are 1.3×10^{-3} and 0.13 Pa, respectively. Rotation speed of the substrate is 4 rev/min and bias voltage is 60 V. The thicknesses of Cr film and CrN/CrTiAlN layer are ~ 0.2 and ~ 1.5 μm , respectively. In the deposition of CrN/CrTiAlN layer, N_2 flow rate was gradually increased to the appropriate value. The currents for Cr, Ti and Al target are 4, 6 and 6 A, respectively. After that, the diamond film was deposited on the sample with the interlayer by HFCVD apparatus. In CVD process, the samples were firstly agitated for 30 min in slurry with a mixture of 20 ml acetone, 0.2 g diamond particles (average diameter 0.1 μm) and 0.2 g alumina powders (average diameter 63 μm). The working pressure is 1.6 kPa in the deposition process. The bias current is 4 A. In the first step, the power is set as 1800 W for 10 min; in the second step, the power is decreased to 1500 W. The deposition time is 20~120 min. The carbon source is acetone, which is added into the reactor through the carrier gas H_2 . The flow ratio of the carrier gas to H_2 is 80:200. The substrate temperature is about 650 and 600 $^\circ\text{C}$ at 1800 W and 1500 W, respectively.

The surface morphology and element's distribution was characterized using a Tescan Vega 3(Czech Republic) scanning electron microscopy with EDS. The cross sectional sample was prepared by the conventional metallographic method. The details can be found in our previous publication [17]. The stress of diamond film and diamond phase

composition was characterized by a Renishaw inVia Reflex Raman spectroscopy using a 532 nm laser. The spot size is $\sim 2 \mu\text{m}$. Raman image ($6 \times 6 \mu\text{m}$) was carried out by scanning the sample in a raster pattern at an image resolution of 12×12 pixels (144 spectra). Phase identification of the film was characterized by grazing incidence X-ray diffraction (GIXRD) with incidence angle 2° using a Bruker D8 Discover. Adhesion of film was assessed by Rockwell C indentation method using a load of 1470N.

3. Results and discussion

We firstly investigate the effects of growth time in the first step on the microstructure of diamond films. Fig. 1 shows SEM images of the diamond films deposited at 1800 W after different times. It is observed that there are comparatively sparse diamond particles with average size $\sim 100 \text{ nm}$ on the surface after 10 min deposition. When the time increases to 19 min, diamond particles become large and basically contact each other. But there are no particles observed on some concave places, as shown in the arrow of Fig. 1(b), which is attributed to high thermodynamic barriers on the concave sites [18]. After 25 min, some local diamond film peels off, indicating a continuous diamond film cannot be obtained on the steel at 1800 W.

In order to understand the reason, Raman mapping of the films were measured, as shown in Fig. 2. Each spectrum in the Raman mapping was deconvoluted to six peaks using Gaussian function, which are ~ 1130 , ~ 1220 , ~ 1332 , ~ 1350 , ~ 1460 and $\sim 1580 \text{ cm}^{-1}$, respectively. The peaks at ~ 1130 and $\sim 1460 \text{ cm}^{-1}$ are ascribed to transpoly-acetylene (TPA) existed in grain boundaries [19]. The peaks at ~ 1350 and

Fig.1 SEM images of diamond film deposited at 1800 W and after different time (a) 10 min, (b) 19 min, (c) 25 min

$\sim 1580\text{cm}^{-1}$ correspond to D-band and G-band of sp^2 -bonded carbon, respectively. The peak at $\sim 1220\text{ cm}^{-1}$ is attributed to extremely small diamond grains or amorphous carbon [19]. The diamond peak position distribution and average Raman spectrum were displayed in the figure. It is observed that the diamond peak position of the diamond film after 10 min deposition is obviously smaller than that after 19 min deposition. The shift of diamond position from 1332 cm^{-1} is proportional to the stress. When the peak position is smaller than 1332 cm^{-1} , the film shows the tensile stress. Otherwise, the film presents the compressive stress [20]. Therefore, the stress after 10 min deposition is compressive and it is obviously smaller than that after 19 min deposition. As there exists gaps between diamond particles after 10 min deposition, the diamond particle contracts easily during the cooling and the film shows low stress value. After 19 min deposition, the diamond particles join together and form the continuous film. As the diamond particle is difficult to contract during the cooling, the film shows large stress value. After 25 min deposition, due to the delamination of some local diamond films, the stress of the film releases mostly and shows low stress value. In addition, diamond content was calculated considering different Raman cross

sections of diamond and non-diamond carbon [21]. The results are 71.1%, 58.8% and 64.6%, respectively. Therefore, above results show that the growth time of about 10

Fig.2 Raman mapping, diamond peak position distribution and average Raman spectrum of diamond film deposited at 1800 W for 10 min, 19 min and 25 min, respectively.

min at high power of 1800 W prefers to prepare the isolated diamond particles with comparatively high diamond content and to decrease the internal stress.

In order to determine the appropriate power of second step, four samples at 1400-1700 W were deposited and the growth time is chosen as 10 min. Fig. 3 shows their Raman spectra. Obvious diamond peaks are observed at 1500-1700 W,

indicating that the diamond phase has been formed in this power range. However, no diamond peak but graphite peaks of 1350 and 1580 cm^{-1} is found at 1400 W, which indicates that the diamond phase cannot be obtained at 1400 W. So, the second step's power was chosen as 1500 W in order to obtain diamond phase.

Fig. 3 Raman spectra of diamond films deposited at 1400-1700 W

In order to obtain a continuous diamond film with low stress, effects of different times in second step on the stress, phase composition and microstructure were studied. Fig. 4 shows the Raman mapping of the diamond films after different times in second step. It is observed that diamond peak positions for the samples growing for 20, 30, 60 and 90 min are all obviously smaller than that at first step for 19 min. According to the relation between diamond peak shift and the stress as above mentioned, it is concluded that the magnitude of the stresses are obviously lower than that of the diamond film growing for 19 min in the first CVD process, indicating that the two-step dropped power process surely decreases the internal stress of the film. From Fig. 4, it is also observed that after 20 min and 30 min deposition, the diamond peak

Fig. 4 Raman mapping, diamond peak distribution and average Raman spectrum of the diamond

films at different times in second step

position of diamond films is in the range of 1331.3-1338.0 cm^{-1} . After 60 min deposition, the peak position obviously become small, it is in the range of 1331.6-1334.0 cm^{-1} . After 90 min deposition, the peak position becomes large within the range of 1335.1-1337.2 cm^{-1} . After 120 min, the peak position becomes small again, within the range of 1332.8-1335.5 cm^{-1} . This suggests that the stresses of the film deposited after 20 min and 30 min deposition are large and it becomes small after 60 min deposition. After 90 min deposition, the stress becomes large, while it was small again after 120 min. Diamond content was calculated and they are 46.3%, 61.0%, 70.8%, 56.3% and 60.2% after second step deposition with the time of 20, 30, 60, 90 and 120 min, respectively. Low diamond content after 20 min deposition is related to low power deposition. When the power is low, the filament temperature is low, the concentration of dissociated atomic hydrogen is low [22]. And the atomic hydrogen etches sp^2 carbon faster than sp^3 carbon. So, low concentration of atomic hydrogen leads to low content of the diamond. Diamond formation in CVD process involves a set of complex physical and chemical processes [23]. It is related to adsorption and diffusion of atoms, which is related to substrate materials. After the deposition time large than 30 min, it is considered that new diamond phase forms on the diamond film due to homoepitaxial growth and the film presents comparatively high diamond content. Stress in diamond film generally includes thermal stress and intrinsic stress. Thermal stress is in relation to CTE mismatch between the stainless steel and the diamond film is compressive in general. Intrinsic stress is mainly related to the existence of nondiamond phase, defects, etc. Here, change of the stress is

considered to be associated with change of the content of nondiamond phase, as the nondiamond phase is source of compressive intrinsic stress. When the content is higher, the value of intrinsic compressive stress is larger [24]. As mentioned above, when the deposition time increases from 20 to 60 min, diamond content increases, i.e. nondiamond content decreases. So, the contribution of the compressive stress becomes smaller and the total stress becomes smaller with deposition time increasing from 20 min to 60 min. After 90 min, the contribution of compressive stress increases due to the increase of non-diamond content on one side. Also, thermal compressive stress becomes larger as the film becomes denser and thicker. As a result, the stress becomes large after 90 min. The comparatively low stress after 120 min is attributed to the local spalling of the diamond film, as the delamination accommodates the stress.

Fig. 5 shows the SEM images of the diamond films after different second step's times. It is observed that a diamond film already forms on the surface after 20 min deposition, but some apertures between blocky structures (see the arrow indicating in Fig. 5(a)) can still be seen on the surface. Further, from the magnified image (i.e. Fig. 5(b)), it is observed that no diamond particles existed in the aperture and some diamond particles do not contact each other on the surface. This indicates that the continuous diamond film is not obtained after 20 min growth. After 30 min deposition, the apertures almost cannot be observed on the surface of the film (as seen in Fig. 5(c)), diamond particles contact each other closely and a fully continuous diamond film forms on the surface (see the inset in Fig. 5 (c)). This suggests that the

continuous diamond film forms at between 20 and 30 min. After 60 and 90 min deposition, the film is still continuous and no delaminated area can be found on the

Fig. 5 SEM images of diamond films deposited after second step time (a) 20 min (low magnification), (b) 20 min (high magnification), (c) 30 min, (d) 60 min, (e) 90 min, and (f) 120 min. The inset of (c) shows the corresponding magnified image.

surface, as shown in Fig.5 (d) and (e). But some tiny amounts of pores (e.g. circle A in Fig. 5(e)) are observed on the surface. After 120 min, some local diamond film flakes off, indicating that the continuous diamond film only can be obtained at between 90 min and 120 min.

Fig. 6 (a) shows GIXRD of the sample after second step time 60 min. It is observed that the film is composed of a large amount of $(\text{Cr,Ti,Al})\text{N}$ and $(\text{Cr,Ti,Al})_2\text{N}$ phase and a small quantity of Cr_2C_3 phase. The appearance of the carbide phase suggests that the interlayer is a little carburized, chemical bonding at the interface between diamond film and the interlayer is formed. Fig. 6 (b) shows EDS line scan of the

sample. It is observed that the thicknesses of the diamond film and the interlayer are ~ 0.4 and $1.7 \mu\text{m}$, respectively. And nitrogen content is still high in the interlayer, proving the low extent of carburization of the interlayer again. From the line scan, it is

Fig. 6 GIXRD (a) and EDS line scan (b) of the sample after second step time 60 min

also observed that iron element does not diffuse out the interlayer and carbon element does not diffuse into the steel, indicating that the interlayer effectively block C inward diffusion and Fe outward diffusion. Compared to carburization extent of the Cr/CrN interlayer in our previous work [17], the extent is obviously small. In general, when the deposition temperature is low, the extent of carbon reaction is low. Also, the existence of aluminum element in the interlayer enhances the nucleation rate [9], alleviates carbon reaction's extent. Therefore, it is believed that the low carburization extent is related to low power deposition in the two-step process and the existence of aluminum element in the interlayer.

Fig. 7 shows Rockwell indentation morphology of the films after different second step times. It is observed that there is basically no chipping or cracking on the border of the indentation on the film after 20 min deposition, meaning that the film has good adhesion. There are some chippings on the border of the indentation on the film after 30 min deposition. There are more chippings on the border and some local flaking

areas are also observed on the periphery of the film after 60 min deposition. And more flaking areas are observed on the film after 90 min deposition. This suggests that the adhesion become poor as second step time increases. The composition of the

Fig. 7 Rockwell indentation morphology of the films after different second step times (a) 20 min (b) 30 min (c) 60 min (d) 90 min

delaminated area (for example, the arrow indicates in Fig. 7(c)) was tested using the EDS. The results show that the residual film includes 15.9 wt.% C, 25.1 wt.% N, 3.1 wt.% Al, 5.2 wt.% Ti, 49.9 wt.%Cr, 0.6 wt.%Fe, indicating that the interlayer doesn't peel off from the substrate. This means that the adhesion between the interlayer and the stainless steel is good. The weakest place in the film is interlayer/diamond interface. The film after 20 min deposition does not chip, as the isolated diamond particles deform freely when the indenter is pressed into the sample. The diamond film after 30 min deposition becomes continuous, and the film's deformation is

restrained in the indenting process. As a result, some areas of the film on the border peel off. The film after 60 min deposition is vulnerable to fracture and peels off in the indenting process due to the increase of diamond content and thickness, so that more delamination areas are found. More peeled areas are observed on the film after 90 min deposition, as the stress and thickness all become large.

Above results show that low-stress diamond films can be prepared on 3Cr13 steel using the two-step dropped power process. Fig. 8 shows the stress relief mechanism of diamond film deposited in the two-dropped power process. As seen, diamond particles are firstly randomly formed on the interlayer's surface at first step (i.e. at high power). Then the power is decreased quickly to the low power. Deposition temperature is high (low) at high (low) power. Thermal stress arising from temperature change from high power to low power can mostly be released, as the discrete diamond particles contract freely. As above mentioned, the substrate temperature is 650 and 600 °C at 1800 and 1500 W, respectively. CTEs of stainless steel and diamond are 17.8×10^{-6} and 1×10^{-6} , respectively. Assuming that thermal stress arising from the temperature decrease is released, it is estimated that the stress decreases by 7.7% according to the thermal stress equation [17]. Then the continuous diamond film with blocky structure was formed on the rough interlayer's surface at low power. Singh R K's study showed that rough surface decreased the stress [25]. So it is considered that the blocky structure is beneficial to the release of the stress. In addition, it is considered that some amount of pores existed in the film also contribute to the accommodation of stress, as it can offer more spaces to shrink. As a result, the

stress in the film is small.

Fig. 8 stress relief mechanism of a two-step dropped power process

4. Conclusions

A continuous diamond films has successfully been deposited on stainless steel with a Cr/CrN/CrTiAlN interlayer using a new two-step dropped power process. In the first step, isolated diamond particles are deposited on the interlayer at high power 1800 W for 10 min. As the discrete particles contract freely, thermal stress arising from high power to low power is mostly released. Subsequently, the continuous diamond film is deposited at low power 1500 W, which further decreases the thermal stress. In addition, the formed blocky microstructure and a small quantity of pores in the two-step process also contribute to stress accommodation. As a result, the film shows lower stress compared to the continuous film deposited only in the first step. This work provides a new way to prepare low-stress diamond films on stainless steel.

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