HFCVD DIAMOND PARAMETERS GROWTED ON TOOL STEEL APLAING VANADIUM CARBIDE INTERMEDIATE LAYER

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1. Introduction

The use of an intermediate material to growth diamond on steel substrate had been studied by many researchers [1]. The interlayer have to full fill three requirements: reduce thermal residual compressive stress after cooling; block the iron diffusion from substrate to material surface; and block the carbon diffusion from gas phase to substrate during HFCVD diamond growth. The vanadium carbide (VC) interlayer was proposed by Barquete et al [2]. The mismatch between diamond coefficient thermal expansion (CTE) ($0.8x10^{-6}K^{-1}$) and steel ($11.6x10^{-6}K^{-1}$) after cooling causes a high compressive stress in the diamond film. The VC CET around $6.06x10^{-6}K^{-1}$ [3], intermediate, can mitigate the residual stress. Also, the VC layer has elevate mechanicals properties (2600-3200HV) [4] and can act as an efficient diffusional barrier[5]. Iron in the diamond growth reactive region acts as catalyze for graphite formation (sp^2 bond) and the presence of graphite at the material surface reduces adhesion and the quality of the diamond film [6].

Adherent and continuous diamond coatings on steel substrates with VC interlayers is determined by several parameters such as: surface morphology; interlayer thickness; substrate temperature; chamber pressure; total gas flow; work distance; CH_4 concentration and diamond deposition time. In this work a detailed study is carried out to probe the effect of some these parameters on diamond nucleation and growth on VC interlayer. The VC layer deposited on AISI D2 tool steel substrates and the HFCVD diamond film were characterized by scanning electron microscopy (SEM-EDS), X-ray diffraction, Raman spectoscropy and Rockwell C indentation test. The results showed that VC layer has a strong bond with both materials: diamond and principally steel substrate. A well adherent diamond film with high quality and purity could be grouted on the VC interlayer.

2. Experimental Procedures

Thermo reactive diffusion is a deposition process which carbide coating using a borax bath grows onto carbon-containing substrates, through the combination of the carbide-forming element atoms in the bath with carbon atoms supplied from the substrates[7]–[9]. In this experiment the salt bath composition was 68% of borax ($Na_2B_4O_7.10H_2O$), 24 % of vanadium pentoxide (V_2O_5) and 8% of boron carbide (B_4C) mixture in a melting pot, which is heated at 1050°C for 3h of TD-VC deposition time. After vanadium carbide deposition the samples are cleaned in boiling water and in the ultrasonic acetone bath.

The thermodiffused samples are submitted to diamond seeding process to improve diamond nucleation and the growth rate in the HFCVD reactor. Once introduced in the HFCVD reactor, the diamond deposition was conducted with the following parameters: 1-3% of CH_4; total flux of 100sccm; 50 torr; work distance of 5mm; substrate temperature at 650°C - 750°C; and deposition time of 3h.

3. Results and Discussions

A continuous and homogeneous vanadium carbide film was obtained on the AISI D2 tool steel and the carbide covered the entire samples surface with 16-18µm thickness (Fig.2). The film showed the formation of the phase V_8C_7, it can be happening as function of borax concentration. The borax acts reducing the melting point and give freedom to the salt bath elements to combine in the most complex form possible that it in this case the V_8C_7 phase. An EDS semi quantitative characterization on the vanadium carbide surface showed a residual iron concentration below 3%, it probe the efficiency of the VC diffusional barrier. In large proportions the iron catalyzes the CVD diamond reaction forming sp^2 bonds characteristics of graphite. However, on a small scale it acts as an accelerator, increasing the nucleation and growth of the CVD diamond.

The HFCVD diamond deposition was conducted keeping the following parameters constants: pressure, work distance and diamond deposition time. Nine samples were obtained after diamond growth varying methane concentration (1-3%) and substrate temperature (650-750°C). We noted that as the temperature was increased the higher was the rate of the growth. However, under some conditions with elevate temperature, a

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high graphite formation was seeing in some sites. With 1% of methane a high quality of the diamond crystals was reached. The hydrogen was more reactive with this gas mixture controlling better the graphite formation. Increasing the methane concentration up 2% and 3% the quality of the film was reduced. On the other hand, the adhesion of diamond film to the VC surface increases, reducing cracks and the residual thermal stress after cooling. The Raman spectrum obtained with 2% of methane at 700°C is presented in the Fig. 3.

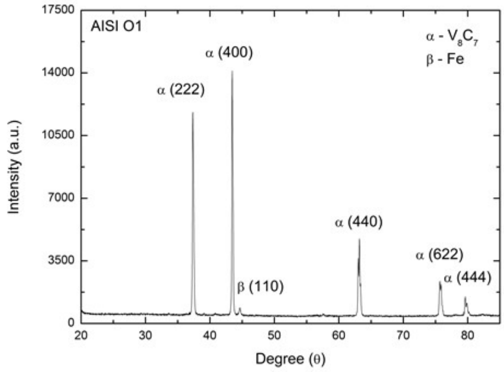

Fig. 1. The vanadium carbide phase.

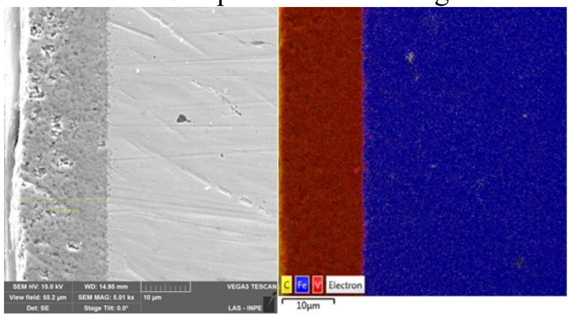

Fig. 2. The vanadium carbide thickness.

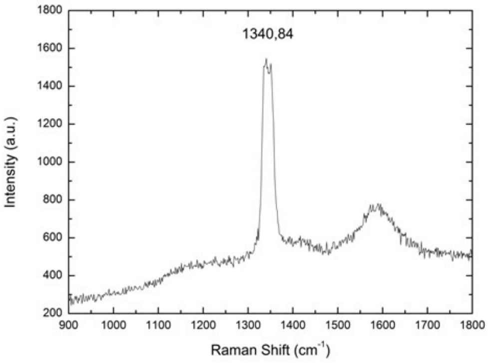

Fig. 3. The HFCVD diamond Raman spectrum.

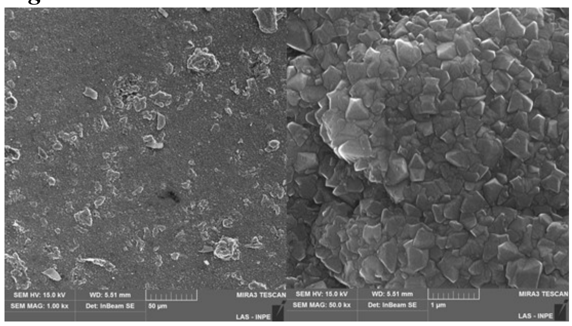

Fig. 4. The HFCVD diamond morphology.

4. References

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