

CVD Diamond Coating for Erosion Protection at Elevated Temperatures

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Small solid particles entrained in a gas-fluid turbomachinery flow can cause degradation of the component surfaces containing the flow by erosion and corrosion processes. As diamond is the hardest known material, much work has been done to use polycrystalline diamond (PCD) as a protective coating on parts operating in a hostile and abrasive environment. Little attention is given in the literature to the high-temperature erosion behavior of chemical vapor deposition (CVD) diamond on different substrates. The objectives of this research were to develop CVD diamond erosion barriers for surface protection of cemented tungsten carbide at high temperatures and to study the erosion behavior of the coatings. Microwave plasma chemical vapor deposition (MPCVD) was used to apply diamond films on WC-6%Co. The erosion behavior of the coated specimens was investigated experimentally by exposing them to abrasive particle-laden flow in a high-temperature wind tunnel. The obtained results show the effects of impingement angle, temperature, and particle dose on the erosion rate. The data demonstrate that uncoated substrates suffer 6-7 times higher wear compared to diamond-coated samples at elevated temperatures up to 538 °C when exposed to alumina particle flow. This study indicates that polycrystalline diamond is emerging as a promising erosion protective coating for high-temperature applications.

Keywords cemented carbides, chemical vapor deposition, diamond, tribology, WC-6%Co

1. Introduction

Compressors, engines, and turbines are normally exposed to erosive environments. Ceramic coatings are considered powerful barriers against performance deterioration of machine parts exposed to particulate flow at high temperatures.[1] The erosion resistance of these coatings is strongly dependent on the coating process and on the substrate materials along with the temperature of operation.[2,3] Our previous work demonstrated the excellent protection that chemical vapor deposition (CVD) coatings provided for cemented tungsten carbide and for ceramic substrates in a particulate flow environment.[4,5]

CVD diamond films on cemented tungsten carbide tools have gained a lot of interest for machining of abrasive nonferrous materials such as Al-Si alloys. The wear properties of these coatings on cutting inserts are well studied and documented.[6] As diamond is the hardest known material, a lot of research has been published demonstrating the advantages of using PCD as protective coatings on parts operating in a hostile, abrasive environment.[7-13] Grogler and coworkers[7,8] have already demonstrated superior erosion protection due to CVD diamond coatings on Ti-based alloys. The erosion behavior of diamond films, both as free-standing or on silicon and silicon nitride substrates, has also been reported.[9-12] A comprehensive study on erosive wear of thick CVD diamond coatings on tungsten and on cemented tungsten carbide is presented in Ref. 13. Promising results, showing much better erosion resistance of MPCVD diamond coating compared to uncoated cemented tungsten carbide, are described in Ref. 14.

The erosion data available in the literature are obtained at ambient temperature, using sand-blasting-type facilities. Little attention is given to the high-temperature erosion behavior of CVD diamond on different substrates, and particularly on cemented tungsten carbide, probably because of test rig limitations to operate at elevated temperatures. The demand for high-temperature erosion data is obvious, especially when PCD is considered as a wear barrier in turbomachinery. In this study, we have investigated the erosion behavior of CVD diamond coatings on cemented tungsten carbide (WC-Co) exposed to high-temperature particulate flow.

2. Experimental Details

2.1 Experimental Set-Up

A microwave plasma CVD (MPCVD) system has been created and used for advanced thin-film deposition including polycrystalline diamond. The facility is based on an ASTeX magnetized microwave plasma source,[15] as shown in Fig. 1. Details about this system have been published.[16] It consists of a power generator (1.5 kW), microwave components, symmetric plasma coupler with quartz window, ECR chamber accommodating window and exit electromagnets, downstream chamber, and movable heated platform. In addition, a turbomolecular pump, gas flow control unit, cold traps, and a water-cooling unit support the facility. This system can produce high-quality polycrystalline diamond films in the upper deposition chamber at high pressures such as at 40 torr. Our

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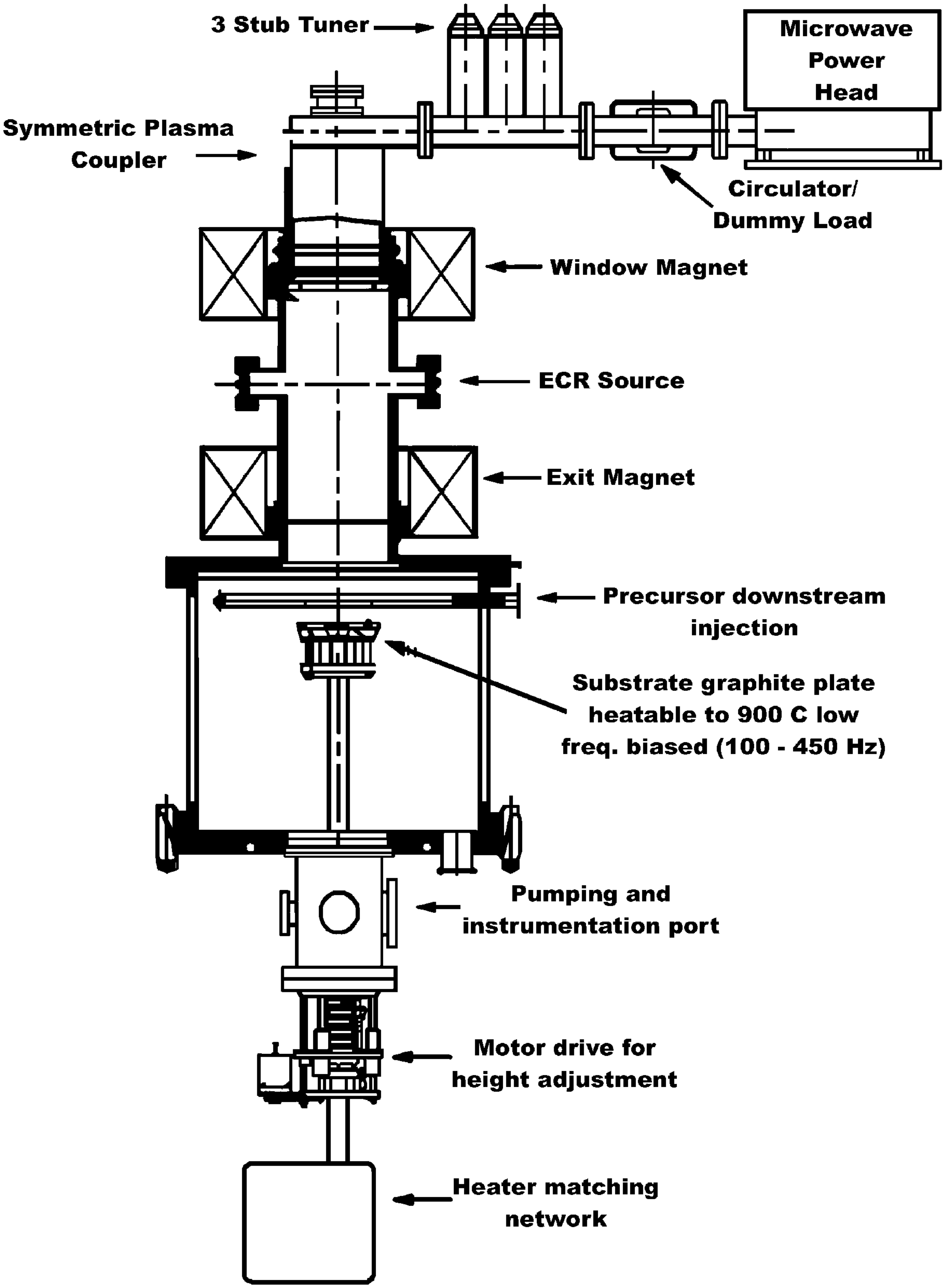

Fig. 1 Schematic of ECR-microwave plasma CVD system (adapted from ASTeX user manual 1.6)

ECR source is coupled to a separate downstream deposition chamber, which allows us to generate plasma at even lower pressures, down to the 2×10^{-5} torr level.

The high-temperature erosion test rig was designed to provide erosion data in the range of operating temperatures experienced in compressors and turbines. In addition to the high temperatures, the facility properly simulates all the erosion parameters that were determined to be important from an aerodynamic point of view. These parameters include particle velocity, angle of impact, particle size, particle concentration, and sample size. A detailed description of the wind tunnel and the particle feeder is given in Ref. 17.

A scanning electron microscope (Cambridge Stereoscan 90) was used to examine the surface morphology of the eroded samples. Micro-Raman analysis (Instrument SA, T 64000, Jobin Yvon triple monochromator system, equipped with an optical multichannel detector-CCD array) was employed for identification of the CVD diamond films. The output power of the Ar^+ laser (514.5 nm wavelength) was 10 mW, and it was focused up to 2 μm.

2.2 Materials and Test Conditions

The particle velocity, particle impingement angle, particle characteristics, and material sample temperature strongly influence the erosion rate. These parameters were varied in the current study for the coated and uncoated specimens. The impact velocity changed from 152 to 305 m/s at temperatures from ambient to 538 °C. Test data were accumulated by setting the particle impingement angle within the range from 15 to 90 degrees. The erosion rate is defined as the ratio between the change of the sample mass and the mass of the impacting particles. The erosion rate tests were carried out in one cycle using a certain amount of particles impacting the sample surface. The wear did not exceed 75% of the total coating mass. After each particle mass increment had impacted the sample, its surface was cleaned from any remaining particles by air jet, the specimen was weighed, and the change in the specimen mass was recorded. In general, the uncertainty of the erosion data obtained by the wind tunnel used in this program is about ±3%. In our experiments, each test was repeated two times under the same conditions, and the scattering of the erosion data was less than 1.5%.

Standard WC-6%Co cemented carbide cutting tool inserts with a square (SPUN) geometry have been employed as substrates for deposition of PCD. When cobalt is used as a binder in cemented tungsten carbides, many difficulties have to be overcome in diamond deposition, because of the solubility of carbon in the metal and the formation of a graphitic layer between the substrate and the coating. Depending on synthesis conditions, cobalt-enriched particles segregate on the substrate surface, leading to poor adhesion. Degradation of the diamond crystals in the early stage of film formation takes place due to their chemical reaction with Co-rich particles.[6,18] Pretreatment was performed on the inserts to enhance the adhesion of the diamond coating. It included leaching of the Co-binder from the surface of the substrate by using *aqua regia* (HNO_3: 3HCl). In some experiments, this step was followed by surface nucleation for 15 min in an ultrasonic bath of ethanol and diamond powder (1–2 μm). The latest procedure was used to plant diamond seeds onto the cemented tungsten carbide surface for increasing of the diamond nucleation density and enhancement of the PCD growth.

Tool-quality diamond coating was deposited by MPCVD using conventional growing conditions,[19] adjusted to the nature and the geometry of the substrates. The typical set of growth parameters included: substrate temperature, 850 °C; pressure, 8 kPa (60 torr); hydrogen gas flow, 500 sccm; methane gas flow, 5 sccm; microwave power, 900 W; and deposition time, 7 h. The growth rate was about 1 μm/h and a 7 μm PCD film was obtained with these deposition parameters.

Fig. 2 Scanning electron micrograph of alumina particles

Because of its great hardness, the PCD coating showed insignificant erosion in the preliminary tests with chromite particles, which frequently cause deterioration of steam and gas turbines.[20] Therefore, electrofused alumina was used as an erodent material. The main constituents of the particles were aluminum oxide (96.53%) and titanium oxide (2.2%). The alumina particles have a grit size of 90 mesh (about 175 μm) and angular shape as shown in Fig. 2.

3. Results and Discussion

The nature of the CVD diamond films was identified by Raman spectroscopy,[21] which is the best-known tool. Figure 3 shows the micro-Raman spectrum of PCD diamond on cemented tungsten carbide. The sharp peak at 1332 cm^{-1} is the diamond (sp^3 bonding) component of the film. The broad peak centered around 1530 cm^{-1} is attributed to the sp^2 bonding of nondiamond forms of carbon (graphite and other, including amorphous carbon). The sensitivity of the Raman technique to sp^2 (nondiamond) bonded phases of carbon is 50 times greater than for the sp^3 bonded phase.[22] Thus the peak around 1530 cm^{-1} represents a much smaller amount of nondiamond in our films. The diamond film surface morphology can be controlled by the substrate pretreatment and by the deposition parameters. Figure 4 shows the SEM micrograph of diamond films grown at deposition parameters listed above. Ball-like morphology can be easily recognized. This spherulitical structure might be contributed by an increased amount of graphite and amorphous carbon in the coating.[19] Columnar growth of PCD can also enhance this structure. Scratching pretreatment with diamond powder reduces the formation of ball-like clusters and gives rise to smoother film.[14]

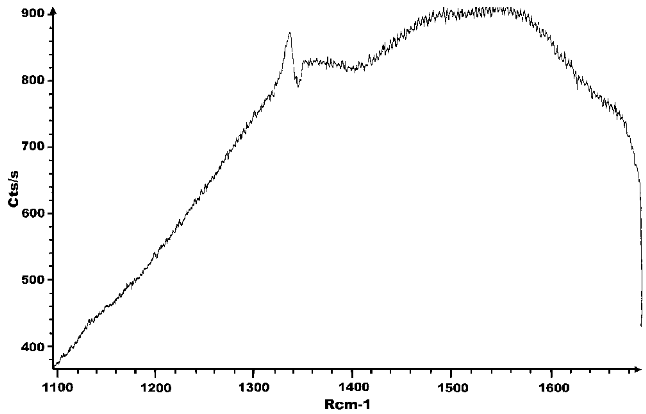

Fig. 3 Micro-Raman spectrum of polycrystalline diamond on cemented tungsten carbide (substrate temperature, 850 °C; pressure, 8 kPa (60 torr); hydrogen gas flow, 500 sccm; methane gas flow, 5 sccm; microwave power, 900 W)

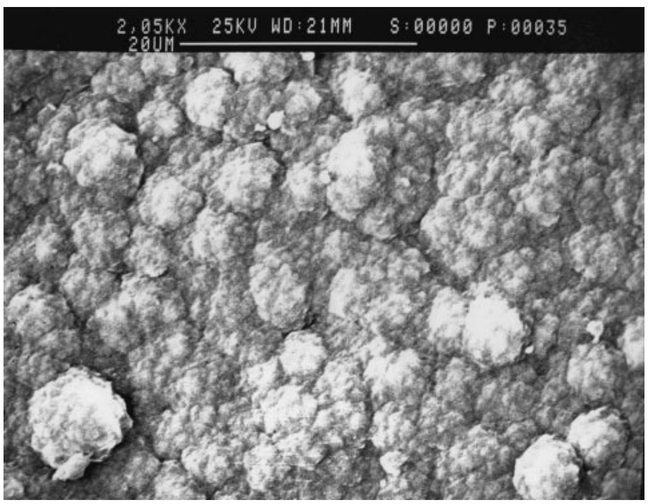

Fig. 4 Scanning electron micrograph of PCD coating on cemented tungsten carbide before erosion

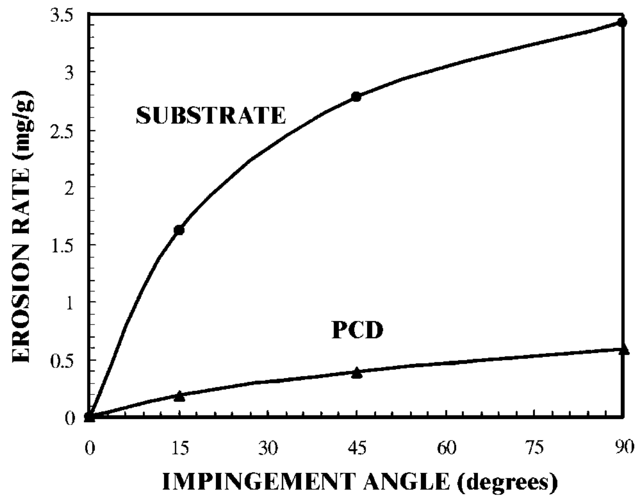

Fig. 5 Effect of the impingement angle on the erosion rate of the PCD coating ($T = 538$ °C, $V_p = 305$ m/s; alumina particles)

3.1 The Effect of Impacting Angle

Predominantly, erosion in brittle materials is caused by propagation and intersection of cracks. The cracks are usually formed by particles impacting on these materials. There are several parameters of interest when considering the erosion rate such as impingement angle, particle velocity, sample temperature, and particle dose.

The impingement angle effect on the erosion rate of uncoated WC-Co and PCD-coated substrates at 538 °C is shown in Fig. 5. Both materials reveal maximum erosion rate at a 90 degree impacting angle, which indicates their brittle nature. The PCD coating is superior to the uncoated cemented tungsten carbide by a factor of 6 to 7 at all angles of attack. The data correspond well with the results obtained by Amirhaghi and colleagues[14] concerning erosion resistance of CVD diamond on WC-Co at ambient temperature. During bombardment by the abrasive particles, mass removal of the coating is predominantly due to microchipping. Figure 6 shows a scanning electron micrograph of the impacted PCD coating tested at 538 °C and 90 degree impingement angle. As seen in this figure, the particulate flow has changed the surface morphology, showing smearing of the alumina particles on the diamond surface similar to that observed by Wheeler and Wood for silica sand erodent.[13] The wear effect is less pronounced at 15 degree impingement angle and 538 °C where the granular structure can still be recognized along with the lateral surface scars (Fig. 7). At lower than 90 degree impact angles, the particles striking the specimen transfer less energy to its surface and cause reduced damage. Inspection of the eroded surface of the uncoated substrates by SEM reveals that the surface morphologies are dependent on the impact angle. Details of the erosion behavior of cemented tungsten carbide are published in our previous work.[4]

From these obtained results, it is obvious that the CVD diamond coating provides excellent protection for cemented tungsten carbide substrates in a particulate flow environment at elevated temperatures.

3.2 Variation of the Erosion Rate with Temperature

To investigate the temperature effect on the erosion rate, experiments were performed at a 90 degree impingement angle and a particle velocity of 152 m/s. The test results are presented in Fig. 8. Similar to our previous finding, the wear of cemented tungsten carbide substrate is slightly affected by the temperature.[4] The PCD coating erosion rate decreases significantly with increasing temperature from ambient to 538 °C. The same behavior has been observed for a CVD TiC coating on a mullite-based ceramic.[5] It is obvious that the coated substrate is better protected from erosion at elevated temperatures. This fact needs additional experimental investigation for correct interpretation. At elevated temperatures, alteration at the interface substrate-coating is not excluded, which might enhance the adhesion of the film. The rise in temperature causes an increase of the energy requirement for erosion of the coated samples. Factors related to the temperature dependence of the

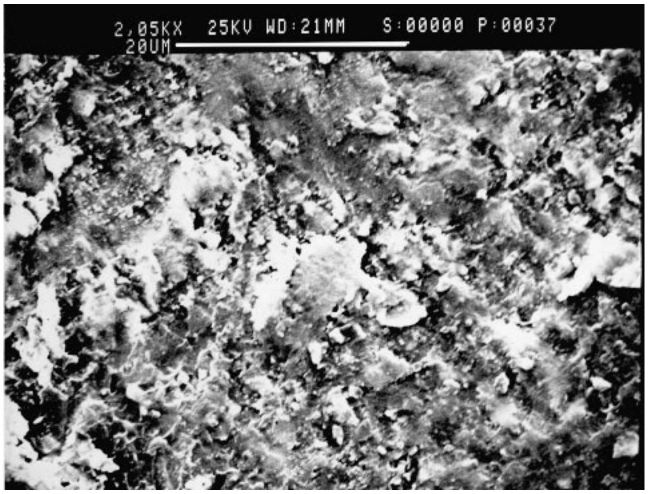

Fig. 6 Scanning electron micrograph of PCD coating on cemented tungsten carbide after erosion at 90 degree impingement angle (T = 538 °C, V_p = 305 m/s; alumina particles)

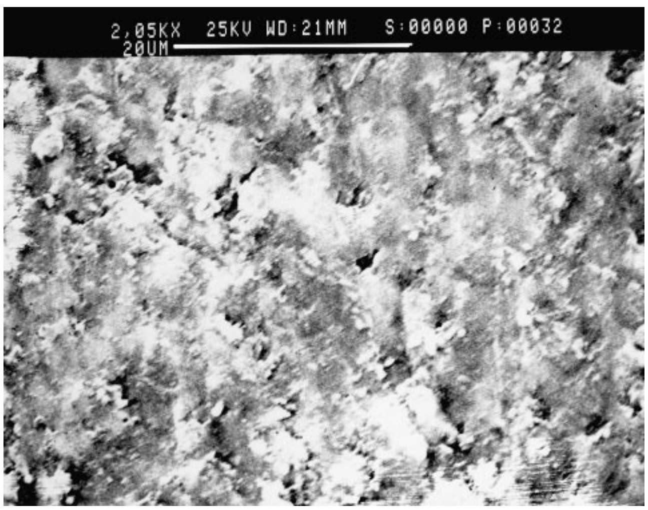

Fig. 7 Scanning electron micrograph of PCD coating on cemented tungsten carbide after erosion at 15 degree impingement angle (T = 538 °C, V_p = 305 m/s; alumina particles)

material's physical properties, including strength and hardness of the coating and also interfacial stress issues, are of significant importance for interpretation of these results.[23-25] For better understanding of this behavior, further coating investigations such as microstructure, oxidation, and fracture mechanics study are required. The results demonstrate that the PCD coating is a promising high-temperature material for erosion protection of cemented tungsten carbide.

3.3 Variation of the Erosion Rate with the Particle Mass

The particle mass effect on the erosion rate of both coating and substrate was studied at the most severe conditions used in this investigation: impingement angle at 90 degrees, T = 538 °C, and V_p = 305 m/s. The specimens were eroded in sequential experiments. The erosion rate variation with the total mass of the impacting particles is shown in Fig. 9. It can be observed that there is a continuous change in the erosion rate of the PCD film. The coating erosion rate initially increases, then reaches a relatively steady state, but remains below the substrate erosion rate. The CVD coating was relatively thin and its penetration was expected at elevated particle mass, which has been confirmed above 35 g of alumina particles. The nonductile nature of both coating and substrate contributes to cracking and delamination of the diamond film in a catastrophic way along the interface. This was observed for a repeatedly eroded sample. A similar failure mechanism is discussed in detail and interpreted in Refs. 13 and 14.

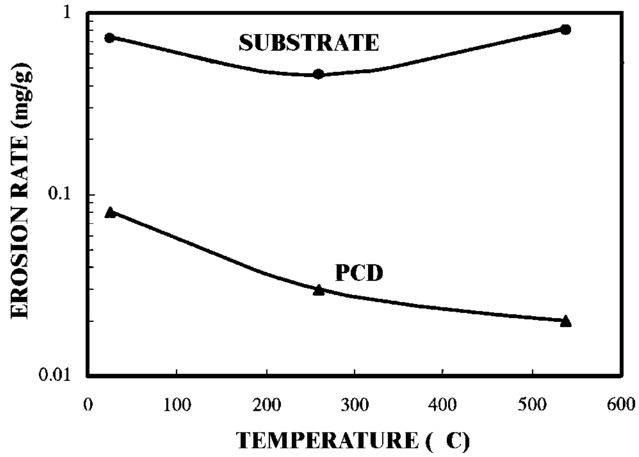

Fig. 8 Variation of the PCD coating erosion rate with temperature at 90 degree impingement angle (V_p = 152 m/s; alumina particles)

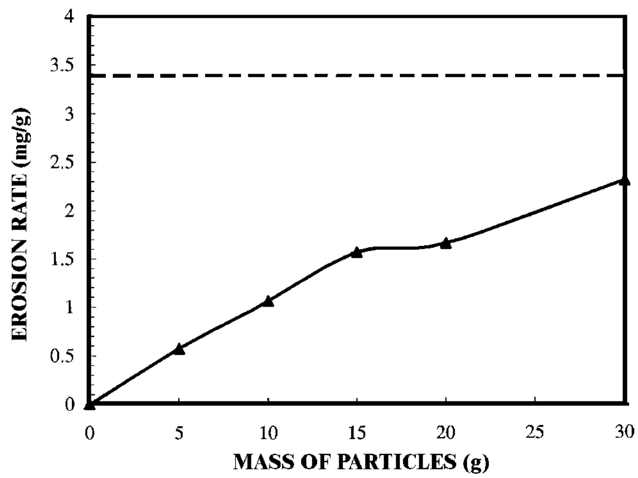

Fig. 9 Variation of the PCD coating erosion rate with particle mass at 90 degree impingement angle (T = 538 °C; V_p = 305 m/s; alumina particles)

4. Summary and Conclusions

Cemented tungsten carbide specimens coated with CVD polycrystalline diamond were tested in an erosion wind tunnel, which simulates steam and gas turbine. The results characterized the tested materials exposed to high-temperature erosion by alumina particles. The effects of impact angle, temperature, and particle dose on the erosion rates of both coatings and substrates have been experimentally investigated.

It was established that the PCD coating exhibits a brittle erosion pattern with erosion resistance superior to the WC-Co substrate. Sample temperature has a strong effect on the erosion rate of diamond films, which reaches a minimum at 538 °C. These erosion tests revealed that the CVD diamond coating efficiently protects cemented tungsten carbide in a harsh, abrasive environment and at elevated temperatures.

Acknowledgments

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