DETERMINATION OF FRACTURE TOUGHNESS OF AMORPHOUS CARBON COATINGS USING INDENTATION METHOD

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1 Introduction

Fracture toughness is the ability of a material to resist the growth of a preexisting crack. Toughness encompasses the energy required both to create the crack and to enable the crack to propagate until fracture, whereas fracture toughness takes only account of the energy required to facilitate the crack propagation to fracture. For bulk materials and some thick films, fracture toughness is easily measured according to ASTM standards [1]. However, for thin films, fracture toughness measurement remains difficult because of the thickness limitation [2].

Thin coatings have become a key technology in a wide range of industries for a vast range of engineering purposes. The successful performance and reliability of thin coatings is often limited by their mechanical properties. Generally, harder coatings are more brittle and easily damaged by shock loads in practical applications. A necessary criterion for evaluating brittleness of thin coatings is to measure fracture toughness of the coatings. Unlike the bulk materials, however, until now, there is neither standard procedure nor commonly accepted methodology to follow.

Amorphous carbon coatings, often called diamond-like carbon (DLC) coatings, have lots of interesting properties such as very high hardness and elastic modulus, high electric resistivity, high optical transparency and chemical inertness, which are close to those of diamond [3, 4]. These coatings have a wide range of uses including optical, electronic, thermal management (heat sinks), biomedical and tribological applications. In certain applications, there is a need for thin coatings to improve friction and wear performance. Intensive research has been done on the measurement of hardness and elastic modulus of such thin DLC coatings deposited by different deposition techniques [5-8]. However, very little is understood on their fracture toughness. The objective of this study was to deposit a thin coating of DLC on a ceramic substrate by plasma-enhanced chemical vapor deposition (PECVD) via evaluating their fracture toughness using micro Vickers's indenter based on the energy release.

2 Experimental Details

The amorphous carbon was deposited on ZrO_2 substrate by plasma-enhanced chemical vapor deposition (PECVD). PECVD is that the process can be operated at low temperature while the deposition rate is comparable to other CVD process. To investigate the effect of coating thickness on fracture toughness three types of coating thickness (6.4, 40.4, and 53.2 μm) has been considered. Thickness was examined by SEM and took an average value. The microstructures of DLC coated ZrO_2 material was also observed by scanning electron microscope (SEM), Model JSM-5610 (JEOL, JAPAN). Prior to the test, the samples were coated with a thin layer of Platinum to avoid sample charging under the electron beam. The observation was performed in high vacuum mode with secondary electron detector and accelerating voltage between 5 and 10 kV. Energy dispersive X-ray spectroscopy (EDS) was carried out on our experimental substrate to clarify the compositions of materials.

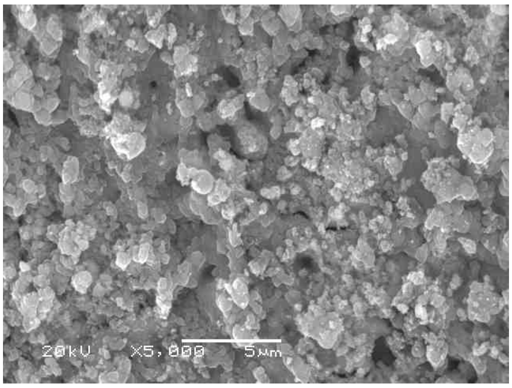
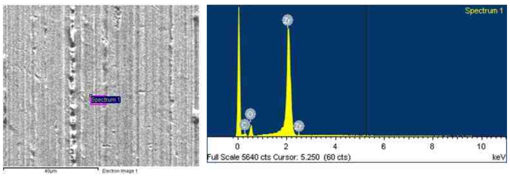
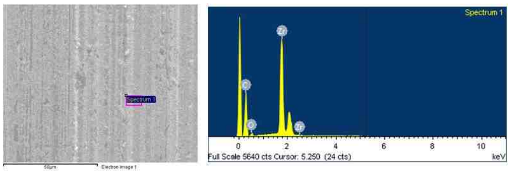


Fig. 1 SEM image of a DLC coated surface



(a)



(b)

Fig. 2 Surface characterization of (a) ZrO_2 and (b) DLC coated ZrO_2 investigated by SEM/EDS

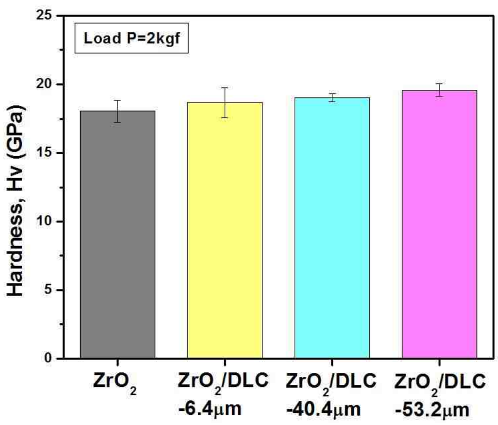


Fig. 3 Vickers hardness for ZrO2 and DLC coated ZrO2 of various thicknesses under applied load 2 kgf

Vickers hardness test was conducted with a normal-load hardness tester at 2 kgf load and at a constant indenter dwell time of 15 s. After indentation, the length of each of the two diagonals of the square-shaped creates surface projected diagonals on surface. Vickers indentation was immediately measured by optical microscopy. At least three indentations test were performed for each specimen. The Vickers diamond pyramid hardness number, H_V, is defined as the ratio of the applied load, P, to the pyramidal contact area, A, of the indentation

$$H_v = P/A = \propto P/d^2 \tag{1}$$

Where, d is the length of the diagonal of the resultant impression, and α = 1.8544 for Vickers indenter.

The Vickers indenter is applied onto the surface and cracks can be generated at the extremities of the indent. The average crack lengths are used for measuring the fracture toughness. Since it is impossible to compare VIF toughness to K_{IC}, it is simply suggested to calculate a mean value for K_C by using the average equation. The crack equations for radial-median and Palmqvist are the following

$$K_{C(R-M)} = 0.016\left(\frac{E}{H}\right)^{1/2}\frac{P}{C^{3/2}} \tag{2}$$

$$K_{C(P)} = 0.0089\left(\frac{E}{H}\right)^{2/5}\frac{P}{al^{1/2}} \tag{3}$$

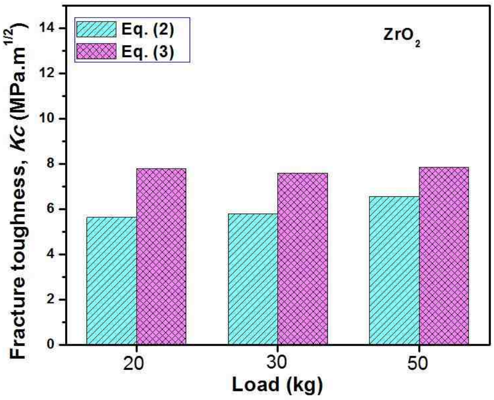
Where, E is the Young's modulus, H is Vickers hardness, P is applied load, c is total crack length measure from half diagonal of indenter to crack tip, a is half diagonal length and l is radial crack length.

3 Results and discussion

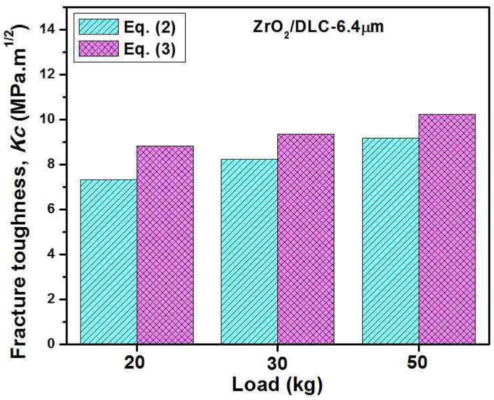
Figure 1 shows the surface morphology of DLC coating surface. From this figure it is observed that carbon grains are well spread throughout the surface making sp3 bond which gives high hardness for coating surface.

Figure 2 shows the area SEM/EDS images for ZrO_2 and the presences of amorphous carbon on this substrate. From these figures it can be easily identify the compositions of substrate materials. The weight percentages of these compositions are duplicated in Table 1.

Vickers hardness for ZrO_2 and DLC coated ZrO_2 of various thicknesses under normal applied load 2 kgf is shown in Fig. 3. From this figure it can be seen, hardness values improved significantly while increasing thickness. Compare to base



(a)



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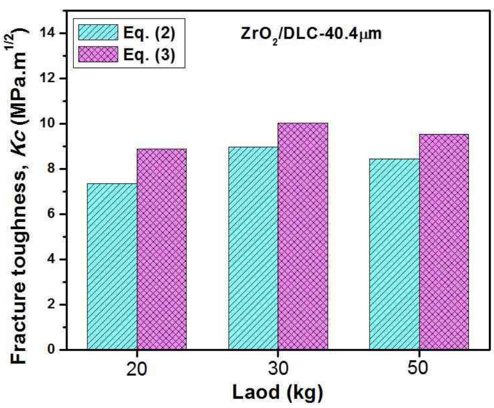
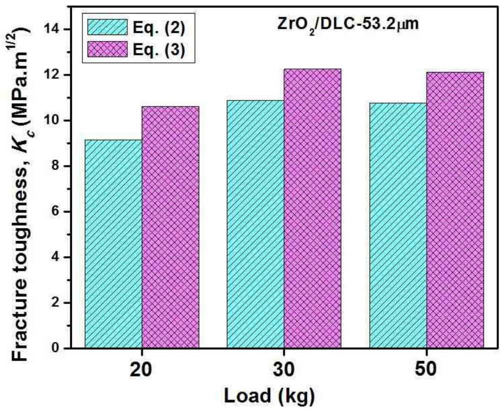


(c)



(d)

Fig. 4 Fracture toughness as a function of load using Eqs. 2 and 3 on (a) ZrO_2, (b) ZrO_2/DLC-6.4 μm, (c) ZrO_2/DLC-40.4 μm, and (d) ZrO_2/DLC-53.2 μm coatings surface.

materials, 3.37%, 5.54%, and 8.48% hardness improvements have been observed in 6.4, 40.4, and 53.2 μm DLC coated ZrO_2 materials, respectively.

Table 1: Chemical Compositions by EDS

Material	Element	Weight (%)
Zirconia (ZrO_2)	Zr	41.92
	O	32.9
	C	25.18
Zirconia (ZrO_2)/DLC	Zr	15.57
	O	9.43
	C	75

The Fracture toughness values obtained using equations 2 and 3 are presented in Fig. 4. For all tested samples, the indentation loads were 20, 30, and 50 kgf and Young's modulus was 205 GPa. The average crack lengths produced from the Vickers indentation tests on ZrO_2 surface were 191.6, 124.5, and 76.9 μm under applied load 50, 30, and 20 kgf, respectively. If we go through Fig. 4(a), we can see that the fracture toughness values ranges from 5.6 to 6.5 $MPa.m^{1/2}$ and 7.7 to 7.8 $MPa.m^{1/2}$ using equations 2 and 3, respectively. DLC coatings have significantly improved the fracture toughness values. Fig. 4(b) to Fig. 4(d) revealed this truth. The fracture toughness values go to 8.4 from 7.36 $MPa.m^{1/2}$ and 9.5 from 8.8 $MPa.m^{1/2}$ using equations 3.3 and 3.4, respectively if ZrO_2 is coated 6.4 μm with DLC material. Highest fracture toughness values were found on 53.2 μm coated DLC materials ranging from 9.16 to 10.76 $MPa.m^{1/2}$ and 10.6 to 12.13 $MPa.m^{1/2}$ by using equations 2 and 3, respectively

4 Conclusions

In this study, fracture toughness of thin film coating has been studied. Micro-meter of DLC coatings were deposited on ZrO_2, faces by PECVD method at 200^0C. Three types of coating thickness (6.4, 40.4, and 53.2 μm) have been examined. Hardness values improved significantly while increasing thickness of coating materials. Compare to base materials, 3.37%, 5.54%, and 8.48% hardness improvements have been observed in 6.4, 40.4, and 53.2 μm DLC coated ZrO_2 materials, respectively. The fracture toughness values are raised 37.4 and 71 percent on 6.4 and 53.2 μm DLC coated surface over base ZrO_2 material by using equation 2 while applying equation 3 it was 22.48 and 50.77 percent respectively.

Acknowledgement

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