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Deposition of Diamond like Carbon Thin Film by Pulse Laser Deposition for Surgical Instruments

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Abstract: Thin film of amorphous carbon (DLC) was deposited on 316 steel using Nd:YAG laser having energy 300mJ. Pure graphite was used as a target. The vacuum in the deposition chamber was generated in the range of 10^{-6} mbar by turbo molecular pump. Ratio of sp3 to sp2 content shows amorphous nature of the film. This was confirmed by Raman spectra having two peaks around 1300 cm-1 i.e. D-band to 1700 cm-1 i.e. G-band. If sp3 bonding ratio is high, the films behave like diamond-like whereas, with high sp2, films are graphite-like. The ratio of sp3 and sp2 contents in the film depends upon the deposition method, hydrogen contents and system parameters. The structural study of the film was carried out by XRD. The hardness of the films as measured by Vickers hardness tester and was found to be 28 GPa. The EDX result shows the presence of carbon contents on the surface in high rate and optical microscopy result shows the smoothness of the film on substrate. The film possesses good adhesion and can be used to coat surgical instruments.

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Key words: DLC · Thin Film · Raman Spectroscopy · XRD · EDX

INTRODUCTION

Thin films are the materials having low-dimension fabricated by depositing one-by-one, atomic/ ionic/ molecular species on the surface of substrate. Generally, thin film is prepared by deposition process of individual atoms of materials (Such as TiO2, SiO2, MgF2, Au and Al etc) which can be deposited to the surface of a substrate like glass, metals, crystals or ceramics to modify their physical, chemical properties and morphology of a surface or substrate [1]. The quality of thin films strongly depends upon the deposition method, gas ambient, source energy, base pressure and topography of the substrate. The main applications benefiting from thin film construction in many manufacturing disciplines are optical coatings, hard surface device coatings, decorative parts and in making electronic as well as semiconductor devices [2].

Diamond like carbon (DLC) film is the mixture of sp3 sites having four-fold coordination and sp2 sites having three-fold coordination with a few of the terminated hydrogen bonds. The range of this hybridization is very short so they are amorphous in nature. DLC thin films are very smooth and can make at low temperature using different carbon sources and on different substrates materials [3]. As from its name indicates that DLC have identical properties to that of diamond such as chemical inertness, high wear resistant, high hardness, biocompatibility and smooth surface behavior but they do not obey crystalline structure as they are amorphous in nature and may contains very small crystalline domains. DLC plays very wide role in the field of coating and used as protective coating such as corrosion resistant coating for industrial tools, wear resistant coating, antireflective coating, etc. [4]. The most important property of DLC is its hardness. DLC films possess very good tribological properties and various researchers have studied this behavior extensively. There are two factors which strongly affect the wear and friction of DLC films, (1) characteristics of films, which are controlled by the deposition process (2) tribological testing conditions such as mechanical parameters, kinematic parameters, physical and chemical parameters. So in short, the carbon-based materials have the desired set of tribological

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properties providing not only low friction but also high wear resistance. They are usually harder than most of the metals and alloys, thus showing a very high wear resistance and impressive friction coefficients [6-7]. Presently, DLC films of high quality are easily accessible from commercial sources. Several of these DLC films are very tough and hard and are able to face all kind of environments, while some are relatively soft which also offer low friction property and good wear coefficients [8]. Owing to their hardness, biocompatibility and tribological properties these DLC coatings can be used to coat on surgical instruments to increase the life and uses of the instruments.

Experimental Procedure for Deposition of DLC Coatings: To synthesize, DLC film on stainless steel, 316L pulsed laser depositions (PLD) technique was used in which the laser was Nd: YAG. The term Nd: YAG stands for Neodymium Doped Yttrium Aluminum Garnet (Y3 Al5 O12). In this technique the laser beam was focus over the target material using optical component (convex lens). To vaporize materials from the target surface Nd: YAG (YAG: 981/c Quantel France) laser was used as an external high energy source. The high-energy beams of each pulse penetrate and broke their chemical bonding in the target material. The few ablated layers produced plasma plume which had atoms/ions/molecules species or clusters, this plume moved in forward direction with different energies towards the substrate holder and struck with substrate material. The species having high energy stuck with surface of substrate forming a continuous dense film. As a result, each pulse increased the thickness of the film in the range from fractions of a nanometer to several micrometers. To obtain a good adhesion of film the substrate holder and target holder were placed parallel to each other inside the chamber so that the ablated plasma plum move in forwarded direction towards substrate. The substrate holder was attached with heater and the target holder was attached with rotator machine that rotate the target in order to obtain a uniform evaporation of target materials.

The whole system consists of deposition chamber, Nd: YAG laser, power supply, voltage supply to substrate heater, gas cylinders, multi meter, optical (convex) lenses, vacuum gauges, heat sensor, chiller, rotary and turbo pumps.

To grow DLC thin film of high quality different parameters has been set off. The laser energy at 300 mJ, frequency of laser pulse 10 Hz, wavelength of laser 532 nm, pulse duration 6 ns, 5000 pulse shots given, target to substrate distance 5 cm and base vacuum in the chamber was 4×10^{-6} m-bar were used in the experiment at room temperature This arrangement helps us to reduce the impurities on the surface of the thin film The pure graphite (99.99%) inside the chamber with 25 mm of square size is rotated at the rate of 7 rpm as a target material. To focus the laser beam at an angle of 450 on the target surface, optical Plano convex lens was used, placed at a distance of 40 cm apart to the chamber.

RESULTS AND DISCUSSION

This section explains characterization techniques, details of various properties of DLC film including their amorphous nature, elemental composition and micro hardness etc briefly.

Raman Spectroscopy: Raman spectra of DLC film on steel is depicted in Figure 1, showing two broad peaks around 1330-1350 cm^{-1} and 1500-1630 cm^{-1} known as D-band and G-band correspondingly as shown in Figure 1. The ratio ID/IG of peak intensity has related to the ratio of sp3/sp2. If the ratio of sp3 bonding is high, the films behave as diamond-like whereas, with high sp2, films are graphite-like. The ratio of ID/IG was calculated and found to be 3.0. The structure is amorphous in nature as peak occurs at 1550 cm^{-1}, that is totally depends upon the deposition method, hydrogen contents and amount of doping materials.

The two clearly broad peaks of our observed results in the Raman spectra are centered around 1342.57 cm^{-1} and 1539.62 cm^{-1} designated as D-band G-band correspondingly that is the signature of diamond-like carbon films corresponding to stainless steel as shown by P1 and P2 in Figure 1. The result of the spectra shows that the deposited film is composed of amorphous carbon. It is also DLC in nature which is appeared by the strong covalent bonds between the atoms.

X-Ray Diffraction (XRD): Figure 2 shows the XRD pattern of DLC film on stainless steel 316L in this pattern no main diffraction peak appears and it shows that DLC film is amorphous in nature which is property of DLC coatings and observed by many scientists [9]. When annealed at 400°C DLC shows a slight shift toward crystallinity and diffractions peaks start to emerge as shown in Figure. 3.

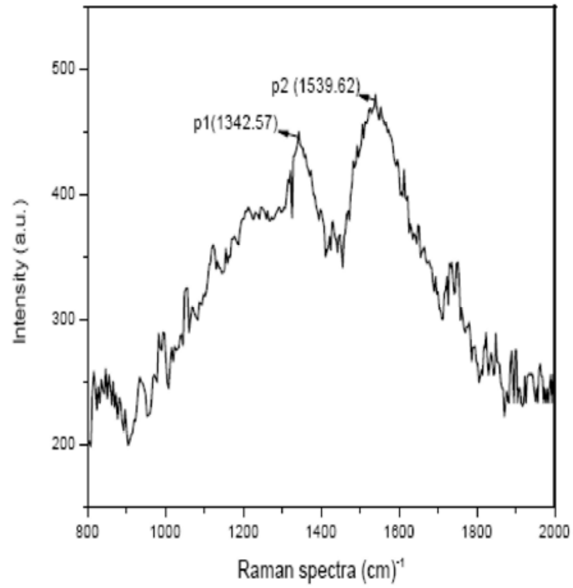

Fig. 1: Raman spectra of the deposited carbon on stainless steel.

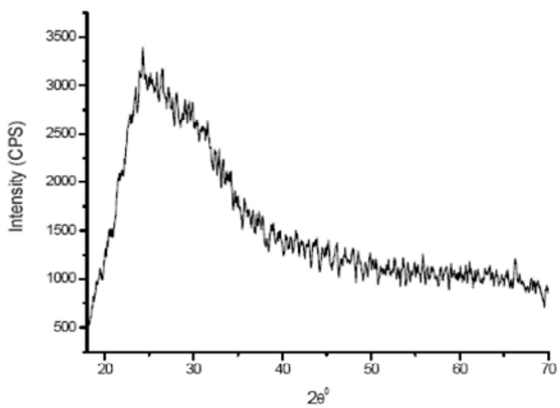

Fig. 2: Typical plot of XRD spectrum.

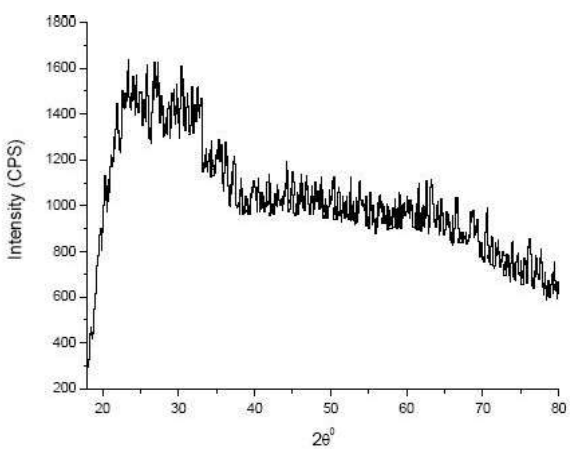

Fig. 3: Typical plot of XRD spectrum after annealing

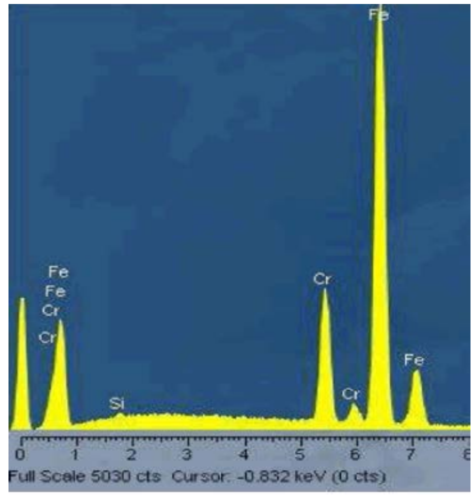

Fig. 4: EDX spectra of untreated stainless steel

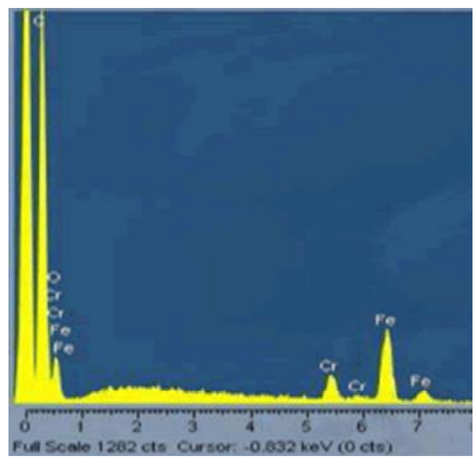

Fig. 5: EDX spectra of stainless steel treated with DLC

Energy Dispersive X-Ray (EDX) Analysis: EDX spectrums of DLC thin film and untreated specimen as shown in the following figures. The EDX spectrum shows two main peaks of the corresponding element. The higher peak shows greater concentration of the corresponding elements in the specimen. The untreated stainless steel spectrum shows peaks of iron (Fe), chromium (Cr) and small amount of silicon (Si) as shown in Figure 4.

EDX Spectra of Stainless Steel Treated with DLC: Comparing the graphical results of EDX of treated with untreated specimen gives the following Information, the silicon layer, which was present in the untreated sample, vanishes after coating. Now the coated sample contains carbon species in large amount on stainless steel substrate which confirms that DLC thin film has been formed.

Untreated stainless steel   Treated stainless steel

Fig. 6: Optical images of stainless steel and treated DLC thin films.

Micro hardness Measurement: Hardness of DLC (diamond-like carbon) coating has conducted by using Wilson Wolpert 401 MVA Vickers hardness tester. The sample was located at a distance of 6 cm to 10 cm for different focus shots of 10 to 30. To measure the hardness of a film load of 10 gf to 25 gf are smoothly applied onto the film under specific conditions for a fixed time of 10 seconds. Furthermore, with an increasing test load the indenter is continuously pressed into the specimen. Finally hardness are measured which is in the range of 28-35 GPa. The hardness of DLC coating is found many times harder than that of stainless steel. So these coatings can be used as hard coating on different instruments specially on surgical instruments.

Optical Microscopy: For magnification of the images of diamond-like carbon coating, optical microscope is used. To obtain wider or narrower field of view of different magnification the optical microscope is adjusted at different angles and distances. Optical microscope is also capable to resolve the image in details. The optical microscope of untreated and treated specimens with different magnification of DLC films is depicted in Figure 6. The micrographs were taken on it at various angular positions placed at a distance of 6 cm. The images of untreated sample is very rough, non-uniform and having number of cracks while due to deposition of carbon coating the treated samples are very clear, uniform and smooth having less cracks. The closer substrates have denser film coating with some pits on the surface that are induced by the etching and sputtering of energetic ion flux. At relatively far position, the films are smoother showing no crack at the surface. The films contain no grain and have amorphous behavior at room temperature. This result again proves that the DLC coating is promising candidate for coating as it is hard, smooth, biocompatible and amorphous.

CONCLUSION

Thin films of DLC were sucessfully deposited on stainless steel 316L substrate using Nd:YAG laser of energy 300 mJ at room temperature. The vacuum in the chamber was kept at 6×10^{-6} mbar. The graphite target having 99.99% purity with dimension of 25 mm and rotate at 7 rpm was used. Films were analyzed with Raman spectroscopy, Energy dispersive X-Rays (EDX) technique, XRD, Optical microscopy and Hardness tester. Raman spectra revealed two distinguishable G and D peaks centered at 1539.62 cm^{-1} and 1342 cm^{-1} respectively. The ID/IG ratio is found to be 2.8. Film hardness has been calculated which is equal to 28 GPa.. XRD patterns show that this thin film is amorphous in nature at room temprature but when temperature is increased upto 400°C then crystallinity starts to show up. Optical microscopy reveals that when substrate is coated with DLC, the substare suface became smooth and clear having no cracks which indicates that uniform thin film has been made. These films can be used to coat surgical instruments because of their hardness and biocompatablity.

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