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Thickness Dependent Properties of Diamond like Carbon Coatings Prepared by Filtered Cathodic Vacuum Arc

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Abstract: The diamond like carbon (DLC) coatings of thickness 300 nm, 500 nm and 650 nm were deposited on stainless steel substrates by Filtered Cathodic Vacuum Arc system combined with magnetron sputtering The chromium interlayer of 100 nm thickness was deposited prior to the deposition of diamond like carbon coatings. The research work focuses on effect of diamond like carbon coating thickness on microstructural, adhesion strength and tribological properties. The films were characterized by field emission scanning electron microscopy, Raman spectroscopy, scanning Electron microscopy, adhesion and wear tests. The Raman studies indicated that the I_D decreased with increasing coating thickness. The maximum value of I_D/I_G was 0.84 for 300 nm diamond like carbon coated stainless steel. The low friction coefficient of 0.12 was obtained for 500 nm coating thickness. Moreover, friction coefficient decreased with increasing coating thickness in initial stages and increased directly proportional to thickness after certain time. For adhesion strength, the higher critical load was found good for 500 nm film compared to other specimens.

Keywords: Diamond like Carbon, Coating thickness, plasma etching, RF sputtering, Cathodic Vacuum Arc, Cutting tools

1. Introduction

Diamond like carbon (DLC) coatings are used as protective coatings for metal, ceramics and polymer substrates. Diamonds like Carbon (DLC) coatings received industrial interest due to its excellent mechanical, optical, electrical, thermal, chemical and biomedical properties. For mechanical properties, DLC possess high wear resistance, high hardness, low friction coefficient, good protection from erosion corrosion with good surface finish. Due to good insulating properties, it finds applications in electronic industry. The thermal stability makes it suitable for high temperature applications. It is a good protective and antireflective layer for solar panels as well as good erosion corrosion resistant for bio implants. The applications of DLC includes gears [1], bearings [2], pins and piston rings [3-4], direct injection fuel systems, forming and cutting tools [5-6], orthopedic applications includes hip joints [7] and hard disk interface in computer industry [8-11]. The properties of DLC coating depends on many parameters such as, method of deposition, deposition variables, i.e bias voltage, sputter power, gas flow, chamber pressure etc. The specific application needs desired properties and can be produced by controlling the parameters.

The effect of initial etching of the substrate and its consequences on bulk structure of the films was reported in the literature [12]. The optimum etching increases the adhesion strength, enhances the sp^3/sp^2 ratio and hence reduce the residual stresses without considerably affecting the film hardness. The young's modulus range 150-750 GPa is obtained depending on the sp^3/sp^2 content and deposition method. The highest sp^3 content of 88% was reported by Ferrari et al [13]. The young's modulus of 330 GPa for 40% sp^3 structure and 630 GPa for 80% sp^3 structure with hardness range of 33-63 GPa was obtained [14]. DLC coatings have good wear resistance, low friction coefficient and provide protection to coated substrate and uncoated counter face [15-21]. Excellent adhesive strength of coating is the basic requirement in practical atmosphere. Different parameters influence the adhesion strength including residual stress, contamination, chemical bonding and surface roughness [22]. If there is a poor adherence, premature of the film will arise by peeling off coating from substrate interface due to high stress. In order to reduce the residual stress, many researchers adopted various techniques as reported in literature [23,24].

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The introduction of soft metal buffer layer or mixing of additives is common techniques. The amount of residual stress also depends on the method of deposition. DLC films have been deposited by different techniques, which includes physical vapor deposition (PVD) and plasma enhanced chemical vapor deposition (PECVD) methods. The properties of DLC films depend on application and preparation method. In the present study, DLC coatings were deposited by Filter Cathodic Vacuum Arc (FAVC) System. The FCVA technique produced high quality DLC coatings [25-26] and argon plasma generated by filtered vacuum arc was used for substrate etching [27]. The effect of coating thickness on microstructural, adhesion strength, tribological properties was evaluated.

2. Experimental design

The Stainless Steel (SuS) and silicon were used as substrates for the DLC deposition. The Stainless Steel substrates were polished with SiC papers and then diamond paste. The deposition includes sputtering of chromium interlayer of 100 nm thickness and diamond like coatings by Filtered Cathodic Vacuum Arc System. Before the deposition, plasma etching rate and DLC deposition rate were found separately as discussed below.

2.1 *Plasma etching*

The specimens were highly polished using diamond paste. The plasma etching process was adopted to achieve the good surface finish. The plasma etching was conducted using Argon flow rate of 50 sccm and 70 sccm. To find the thickness, six points starting from center of specimen to outward in radial direction with increment of 15 mm were marked. The etching time of 10 minutes was kept constant for both types of argon flow rates. The etching process was carried out with 600 V bias voltage and 0.3 A current. The thickness was measured using profilometer. It was found that the etching increased with increasing Argon flow rate. The etching depth of 21.4 nm at 50 sccm Ar flow rate and 2.5 nm at 70 sccm was observed. The details of etching depth on different points for each test are given in Table 1.

Table 1 Effect of Ar flow rate on sbstrate etching

*Pt No.	Thickness (nm)	Ar flow rate
	50 sccm	70 sccm
1	20.0	2~2.5
2	21.2	2~2.5
3	22.0	2~2.5
4	24.5	2~2.5
5	22.4	2~2.5
6	18.5	2~2.5
Avg	21.4	2~2.5

*From center of specimen to outward direction with increment of 15 mm

2.2 *Deposition rate*

The deposition rate was evaluated by varying the bias voltage. Figure 1 presented the effect of bias voltage on deposition rate. It was observed that the deposition rate decreased gradually with increasing bias voltage.

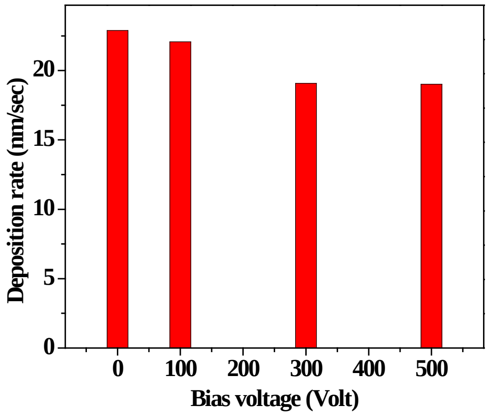

Fig. 1 Effect of bias voltage on deposition rate.

2.3 *Deposition process*

After finding the etching conditions and deposition rate, deposition was designed for stainless steel as described below,

a) Etching using 70 sccm Ar flow rate and chamber pressure of 9.4×10^{-3} Torr with 600 V bias voltage for 10 min.

b) Cr interlayer of 100 nm thickness was deposited using RF sputtering. The Ar flow rate of 40 sccm, 200 W, 600 V bias voltage and 8.4×10^{-4} Torr chamber pressure were used as deposition parameters.

c) DLC coatings of thicknesses 300 nm, 500 nm and 650 nm were deposited over chromium interlayer. The deposition parameters include bias voltage of 100 V, 4.9×10^{-5} Torr of chamber pressure without argon.

Before the deposition, effect of bias voltage was evaluated. Specimens were prepared with 0 V, 100 V, 300 V and 500 V bias voltages. The FESEM, Raman spectroscopy, wear and scratch tests were performed. DLC coatings have maximum hardness and residual stress in range of 70 V -140 V [28] bias voltage.

3. Results and Discussion

3.1 *Field Emission Scanning Electron Microscopy (FE-SEM)*

The surface morphology was examined using Field emission scanning electron microscopy TESCAN-CZ/MIRA-I-LMH with 5000X. The FESEM images of DLC coated SuS are shown in Fig 2. It was observed that the 300 nm DLC coating is highly smooth as compared to 500 nm and 650 nm films.

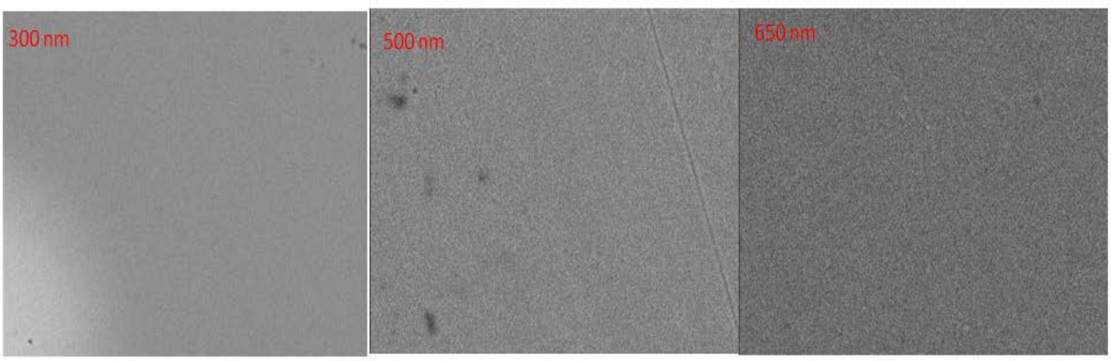

Fig. 2 FESEM images show the surface morphology of DLC coatings on SuS

3.2 *Raman Spectroscopy*

Raman spectroscopy of DLC coated stainless steel specimens were performed using JASCO, NRS-3300. The studies were performed in the range 100-4000 cm^{-1} with interval of 0.01 cm^{-1}. The I_G

peaks of DLC coated SuS were in range 1570 cm^{-1}-1580 cm^{-1}. It was observed that the graphite structure increased with increase of thickness. The specimen with 300 nm coating showed I_D peak ~ 1320 cm^{-1}, while the specimens deposited with 500 and 650 nm coating thickness did not show I_D peaks. The maximum I_D/I_G ratio of 0.84 was achieved for 300 nm DLC thickness. These results are consistent with the reported results. The Raman spectra of DLC coatings are shown in fig. 3.

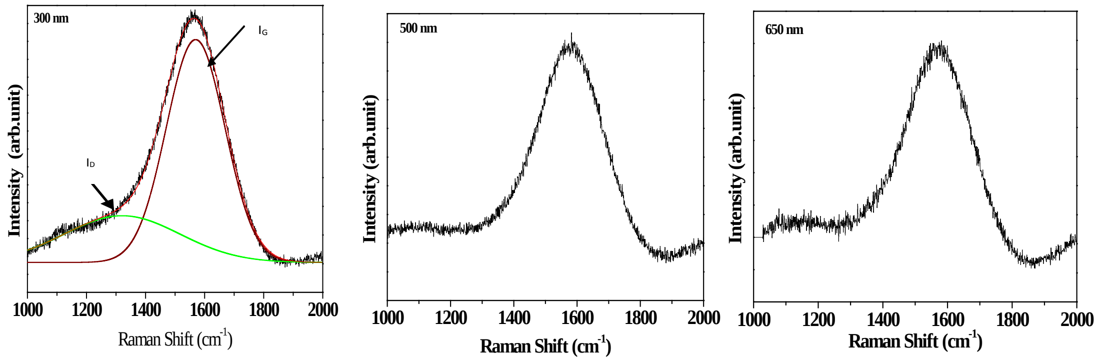

Fig. 3 Full and Decomposed I_G and I_D peaks for DLC coated SuS materials

3.3 *Wear test*

For mechanical applications, low friction suggests a lower energy loss, high reliability and an improved wear resistance. The tribological behavior was evaluated using ball-on-disk sliding (CSM wear tester) in dry air. The tribology test conditions were, 100Cr6 ball of 3.0 mm diameter, track radius of 5 mm, normal load of 5 N and stop condition of 100 m were used. The test was acquitted at 10 Hz with a linear speed of 0.06328m/s at room temperature (20°C). The mean friction coefficient of 0.149, 0.115 and 0.155 were obtained for the DLC thickness of 300 nm, 50 nm and 650 nm respectively. The minimum friction coefficient of 0.143, 0.093, 0.092 and maximum of 0.187, 0.183 and 0.239 was recorded for 300 nm, 500 nm and 650 nm DLC coated on stainless steel respectively. Table 2 provides the summary of friction coefficient for DLC coated on SuS. Fig. 4 shows the variation of average friction coefficient with coating thickness

Table 2 Effect of DLC Thickness on Wear of SuS

Specimen	Min.	Max.	Mean
300 nm DLC/SuS	0.143	0.187	0.149
500 nm DLC/SuS	0.093	0.183	0.115
650 nm DLC/SuS	0.092	0.239	0.155

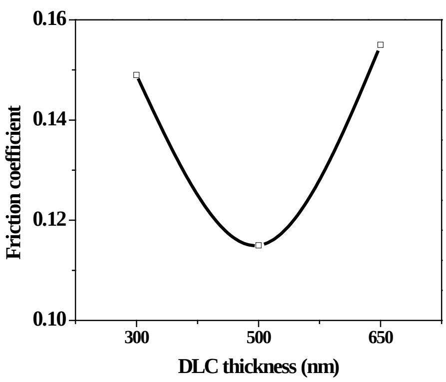

Fig. 4 Effect of DLC thickness on wear

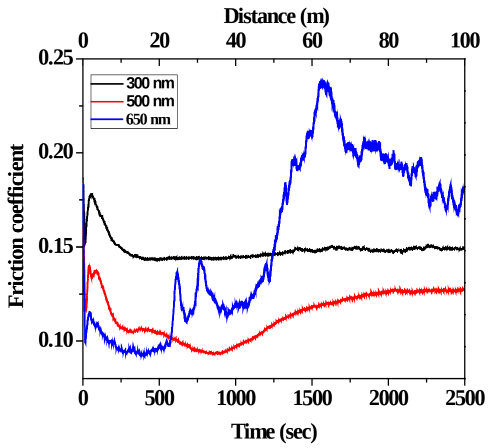

Fig. 5 Tribology of DLC coated SuS

Figure 5 shows the friction behaviour with time and distance for different coating thickness. The figure shows that the specimen having 300 nm thickness possess average friction coefficient of 0.115 and smooth wear rate up to 100 m. The specimen having 500 nm thickness have small friction coefficient up to 40 m and the wear rate increased due to the damage of coating. Similarly, the wear rate for 650 nm coating, increased abruptly after 40 m due to severe damage of coating. Hardness is directly proportional to thickness [29] hence at high stresses wear rate increased rapidly after certain time and distance. However, it was observed that the thicker coatings have low friction coefficient in initial stage of wear tests.

3.4 *Scanning Electron Microscopy*

Wear track images were observed using scanning electron microscope JP / JSM 5200. Figure 6shows the SEM images of the wear track of 300 nm, 500 nm and 650 nm respectively. It can be observed that, the 300 nm and 500 nm thick coating have friction coefficient of 0.149 and 0.115 respectively and have smooth curves throughout test of 100 m. In the case of 650 nm coating, it was observed that the coating damage was occurred on wear track. The damage of coating is also observed from friction coefficient curve.

3.5 *Adhesion Strength*

Etching plays an important role on adhesion strength of coating with substrate. Adhesion strength of plasma etched DLC coated on SuS was observed using CSM scratch tester. The scratch length was 10 mm with a load of 1-20 N. Figure 7 shows the adhesion strength of DLC coatings of different thickness. It can be seen that the 650 nm film showed the lower critical load of 4.3 N and 500 nm film showed the higher critical load of (LC2) 7.9 N. The summary of adhesion test results is given in Table 3.

Table 3 Effect of Coating thickness on Adhesion Strength

S. No	Specimen Name	LC-1 (N)	LC-2 (N)
1	300 nm DLC/SuS	4.1	6.7
2	500 nm DLC/SuS	4.0	7.9
3	650 nm DLC/SuS	4.3	7.1

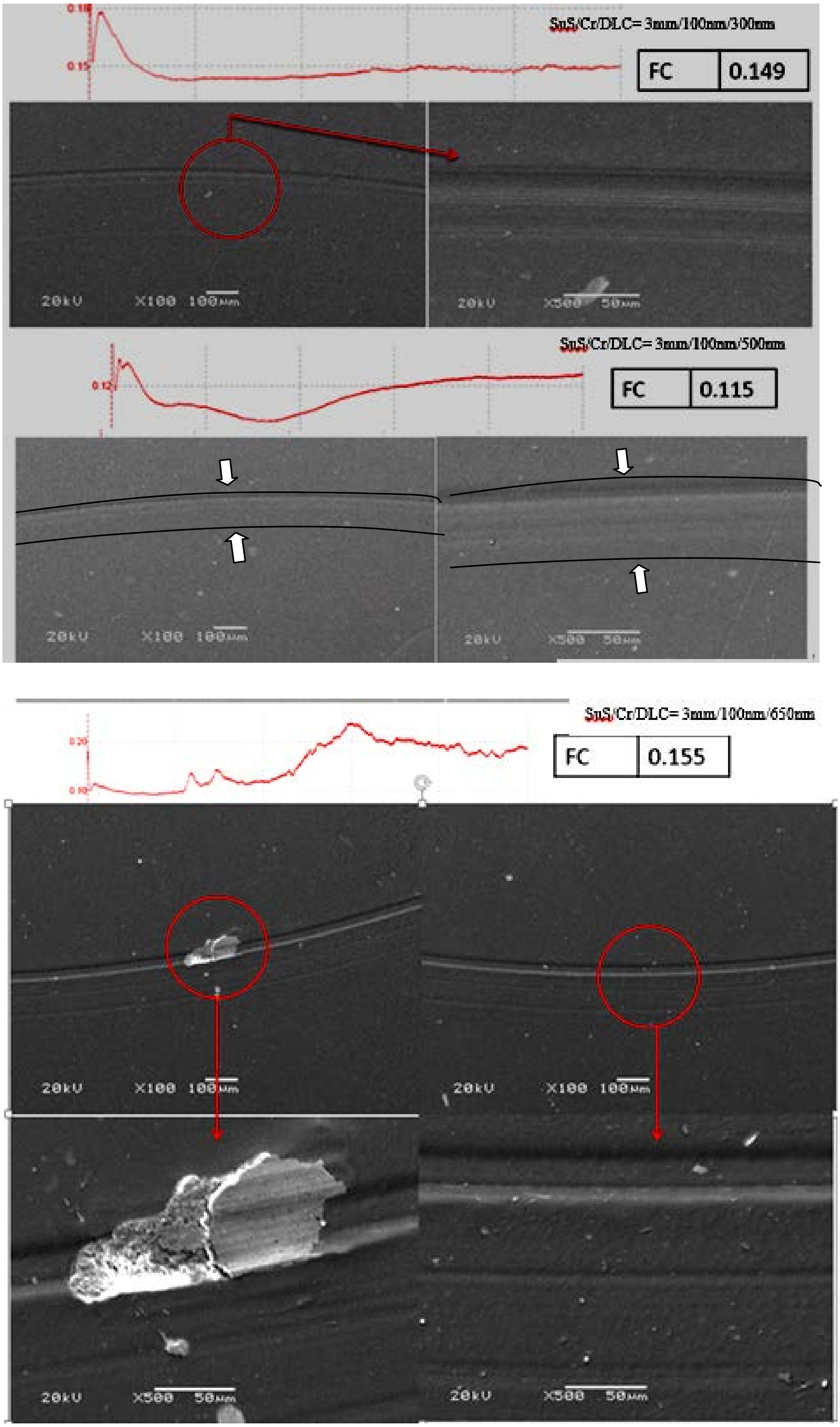

Fig. 6 Scanning Electron Miroscopy of Wear Tracks

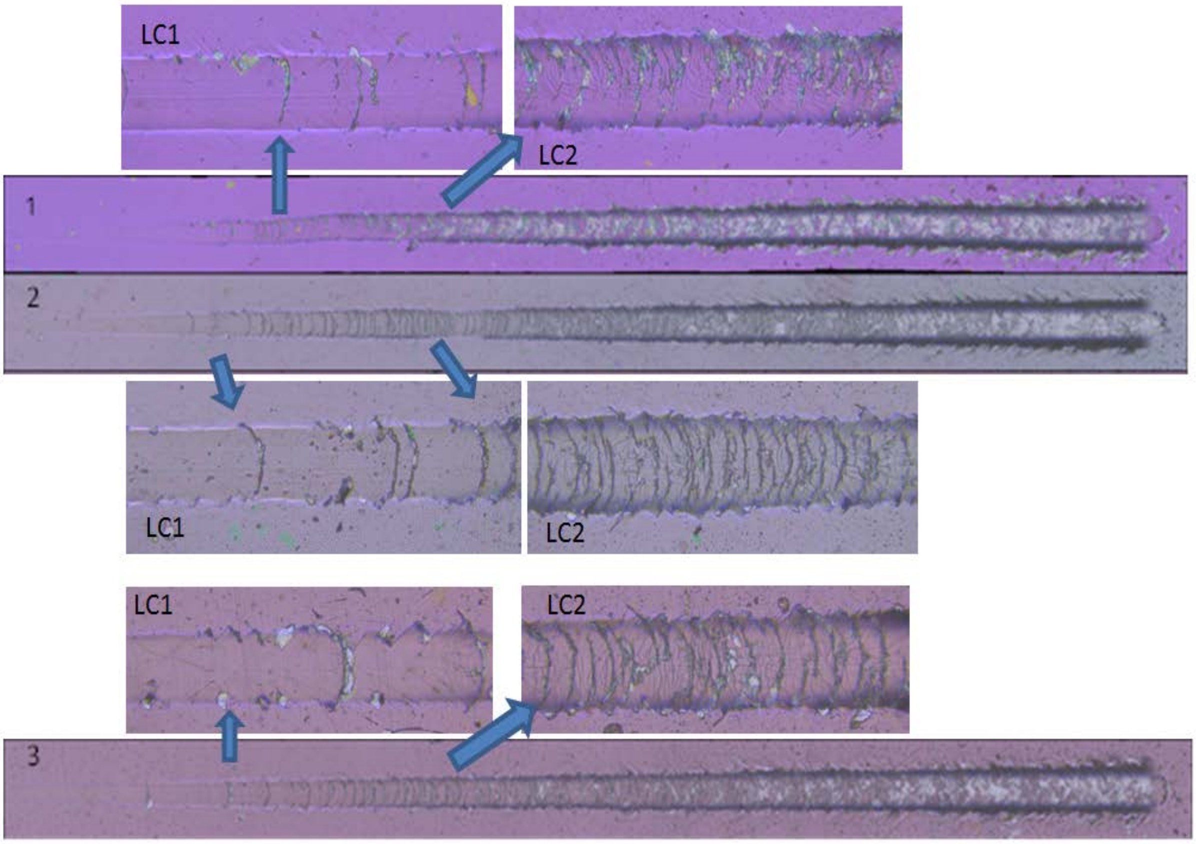

Fig. 7 Panorama picture of scratch test of DLC coated SuS (1) 300 nm (2) 500 nm and (3) 650 nm

4. Conclusions

DLC coatings of different thicknesses were deposited on stainless steel substrates after plasma etching. The effect of coating thickness on microstructure and tribological properties were evaluated. The DLC coated SuS showed the I_G peaks ~1570 cm^{-1}-1580 cm^{-1} range, and I_D peaks were in range ~1170 cm^{-1}-1320 cm^{-1} with maximum I_D/I_G of 0.84 for 300 nm DLC. The wear resistance increased with increase of coating thickness just for initial stages but high wear occurs after a small sliding distance. The adhesion strength was good for 500 nm DLC coated samples. Hence the DLC coating of 500 nm thickness can be suggested for cutting tool applications.

Acknowledgment

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