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PROPERTIES OF DLC COATING INSIDE OF MICRO-CHANNEL CAVITIES PREPARED WITH PSII

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Abstract. With the miniaturization of parts, friction has more obvious effect on the microforming process than that in macroforming. For the existing of friction size effect, liquid lubrication is not suitable for the microforming. More attentions have been paid to DLC coating for its superior tribological characteristics. In this investigation, several dies with micro-channel of different width from 0.6 mm to 1.4 mm were developed. Plasma source ion implantation (PSII) was selected to deposit DLC coating at the inner surface of micro channel dies, and analysis of micro-channel dimension effect was carried out by measuring the properties of DLC coating inside of micro-channels. The surface topography of DLC coating shows that the DLC coating exhibits an obvious island growth. With decreasing of micro-channel width and increasing of distance from upper surface, the nanohardness becomes small. This indicates that the mechanical properties become poor. Scratch tests show that the failure mode is brittle fracture for high contact pressure. With the decreasing of channel width, the DLC coating is not easily peeled off even when DLC coating is broken for the large plastic deformation of substrate. The ratio of sp^3 and sp^2 bonds is changed by the channel width and distance from upper surface. The fraction of sp^3 bond in DLC coating becomes smaller. The results shown above indicate the mechanical properties of DLC coating becomes poor with decreasing of channel width, and the adhesion strength is improved.

1. INTRODUCTION

Microforming is very suitable for mass production of microparts which are widely used in micro-electro-mechanical system (MEMS) [1-3]. However, friction exhibits more effect on the microforming processes than that in traditional macroforming. And when a liquid lubrication is applied, friction increases obviously with the miniaturization of part [4-6]. More attentions have been paid to diamond like carbon (DLC) coating for low coefficient of friction (COF) and wear rates, environmentally benign characteristics [7]. Osakada found that DLC was effective to reduce the friction with aluminum in dry metal forming with coated tools [8]. The wire drawing process showed that utilizing the DLC-coated bisected die were effective in realizing semidry and dry for aluminum materials [9]. To protect dies and eliminate washing processes of lubricant, DLC coating was applied in macroforming process. DLC coating were successfully tested by Reisel in a contact temperature range between room temperature and 500 °C to protect the tool against high sliding friction and adhesive wear. And lower roughness of produced bores compared to uncoated punches was achieved [10,11]. For high contact pressure, the lifetime of DLC coating becomes a concerned problem, and failure mechanism should be discussed. Lin's investigation showed that the lifetime of the proof minting dies coated with DLC coatings was extended 8 times and 4 times compared to that of un-coated and Cr-plated dies, respectively. And failure mechanism of DLC coating were plastic deformation of substrates, microcraks and local breakdown due to surface pin hole, high stress abrasion or impact wear [12]. Wang's investigation showed that the generation of horizontal

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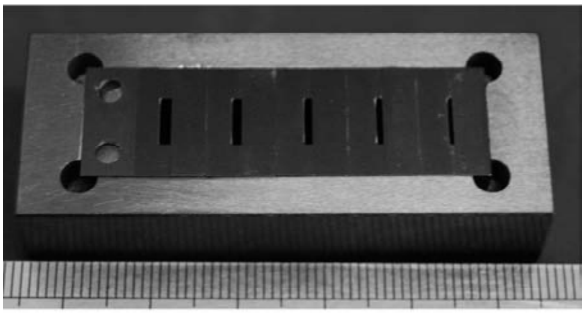
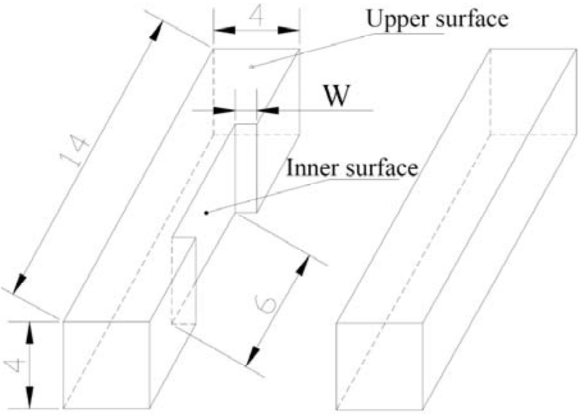
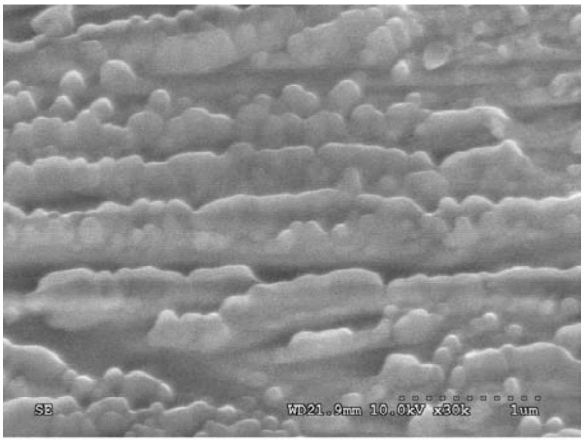


(a) Photograph of micro channel die

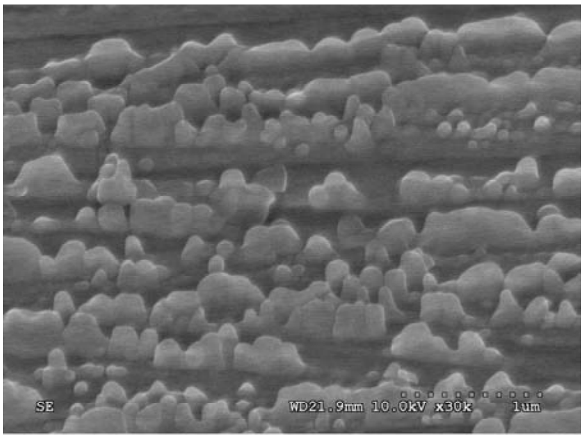


b) Schematic of micro channel die

Fig. 1. Micro channel die.



(a) *D*=0.5 mm



(b) *D*=1.5 mm

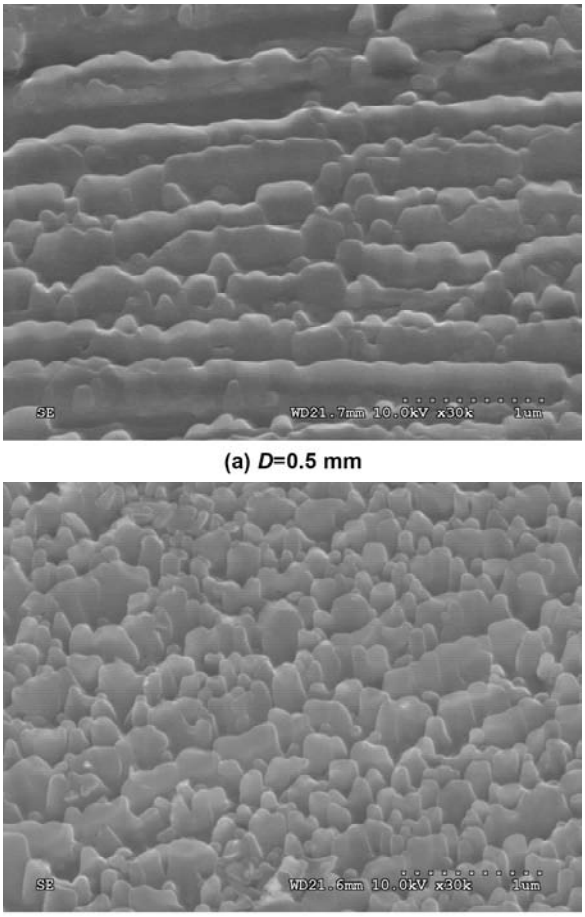
Fig. 2. Inner surface SEM photography various distance from upper surface (*W* is 0.8 mm).

cracks in the DLC-Si coating layer could be hardly influenced by the coating thickness and substrate material [13]. Kitamura evaluated the properties of DLC-Si, and they found that DLC-Si coating had high enough potential to be applied to a die for cold metal forming [14]. In microforming, Yang deposited DLC coating and DLC coating with gradient properties on the surface of die to improve the wear–resistance. The results showed that DLC coating could bear larger strain/stress with very strong adhesion with substrate, and be able of strong wear resistance [15,16]. Takatsuji investigated the aluminum micro extrusion process with various coatings, and the results showed that the maximum extrusion force was reduced from 32 kN to 25 kN when DLC coating was applied [17]. Since it may be difficult for depositing DLC coating on inner surface of micro cavity, Yang's results show that the micro-coating method can be applied for inner wall surface treatment of components with thin holes [18].

In this investigation, the properties of DLC coating inside of micro-channel with different width were studied deposited with plasma source ion implantation (PSII) used for the micro-U deep drawing. Analysis of micro-channel dimension effect was carried out by measuring surface topography, nano-hardness, microstructure and adhesion strength. The results indicate that the width of micro-channel has an obvious effect on the properties of DLC coating.

2. EXPERIMENT PREPARATION

The photograph and schematic illustration of dies is shown in Fig. 1. To easily measure the properties of DLC coating at the inner surface of micro channel, a serial of two-body dies with micro channel of different width were manufactured with SKD 11 steel. The width of micro channel *W* is 0.6 mm, 0.8 mm, 1.0 mm, 1.2 mm, and 1.4 mm, respectively. DLC coating was deposited at room temperature with the plasma source ion implantation (PSII). The base pressure in the chamber was about 0.5 Pa, and the dies are cleaned by the Ar sputtering. Before the deposition of DLC coating, a very thin nitrogen layer as buffer film was deposited at the base pressure of 0.5 Pa and pulse voltage of 20 kV. Then, radio-frequency power was selected as 300 W, and the



(a) *D*=0.5 mm

(b) *D*= 1.5 mm

Fig. 3. Inner surface SEM photography various distance from upper surface (*W* is 0.6 mm).

DLC coating was deposited at the base pressure of 0.5 Pa and pulse voltage of 20 kV for 2 hours.

AFM and SEM (HITACHI, S-4300) were selected to observe the topography of DLC coating. A nanoindenter (MTS Nano Indenter XP, USA) was selected to evaluate the nanoindentation. Scratch tests were performed to analyze the failure mechanism and evaluate the adhesion strength between DLC coating and substrate. The microstructure of DLC coating was analyzed with Raman spectra (JY, HR800). The ratio of sp^3 and sp^2 bond was achieved with excitation wavelength of 532 nm at power of 0.1 mW.

3. RESULTS AND DISCUSSION

3.1. Surface topography

The topography of inner surface DLC coating is observed with SEM, and the results of 0.8 mm and 0.6 mm in micro channel width are shown in Fig. 2 and Fig. 3. The inner surface is not smooth, lathlike and island topography is observed. With the increasing of distance from upper surface *D*, the amount of lathlike structure decreases, and island structure increases. Comparison between width of 0.8 mm and 0.6 mm shows that amount of island structure increases clearly when a small channel is accepted. As shown in Fig. 3b, the DLC coating mainly consists of island structure. The reason may be that the distribution of electric field is changed because that width channel is very small. The energy and moving direction of plasma in micro channel is different from that of deposition on a body.

To analyze the growth mode in details, the topography is measured with AFM as shown in Fig. 4. The photography of upper surface shows that the DLC coating consists of many very small island structures. And the distribution of island is very homogeneous. With the increasing of distance from upper surface, large island structures are appeared at the inner surface, and the density of island becomes small. This result is according to that observed with SEM. The topographies indicate that

Table 1. Nanohardness and elastic modulus of DLC coating at upper surface (GPa).

W (mm)	0.6	0.8	1.0	1.2	1.4
Nanohardness	10.9	12.3	11.88	12.47	13.7
Elastic modulus	122	136.3	131.85	140.15	150

Table 2. Nanohardness of DLC coating at various distance from upper surface (GPa).

D(mm) *W*(mm)	0.5	1.0	1.5	2.0	2.5
1.4	1.68	1.95	0.576	0.46	0.2
1.2	0.165	0.15	0.83	0.54	3.74
1.0	0.48	0.58	0.3	0.14	0.55
0.8	0.11	0.08	0.32	0.15	
0.6	0.103	0.08	0.3	0.14	0.049

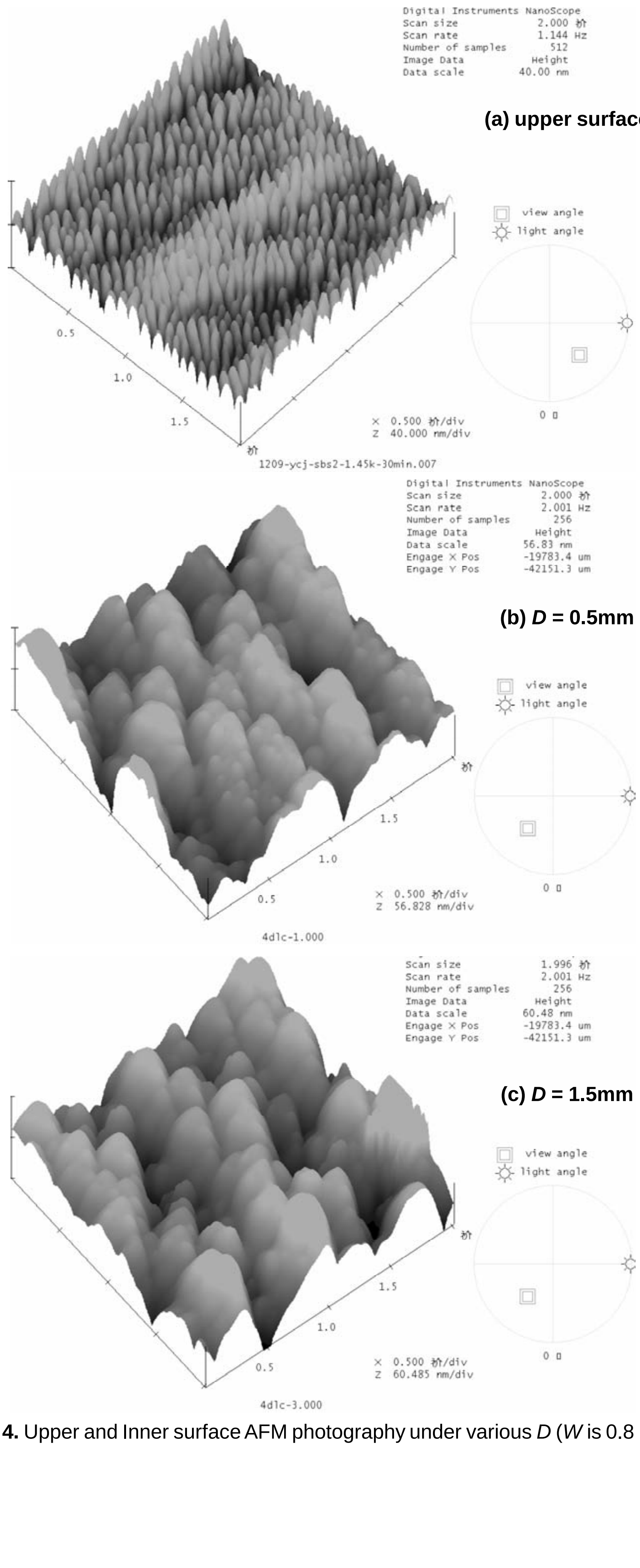
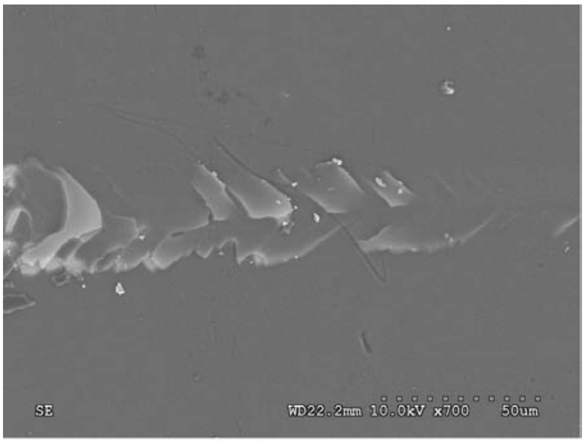
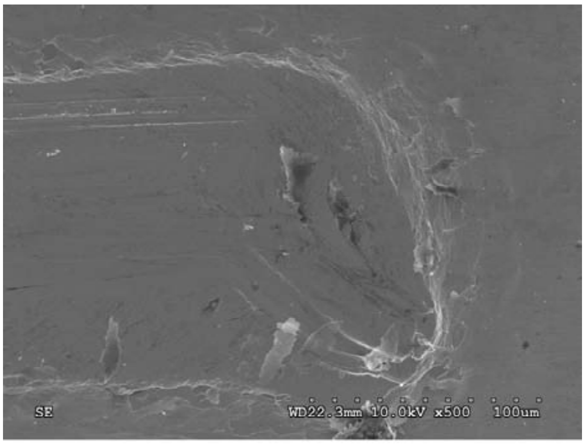


Fig. 4. Upper and Inner surface AFM photography under various *D* (*W* is 0.8 mm).

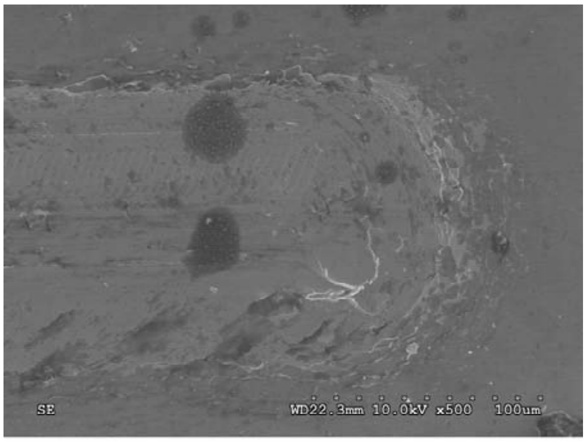


(a) Start point

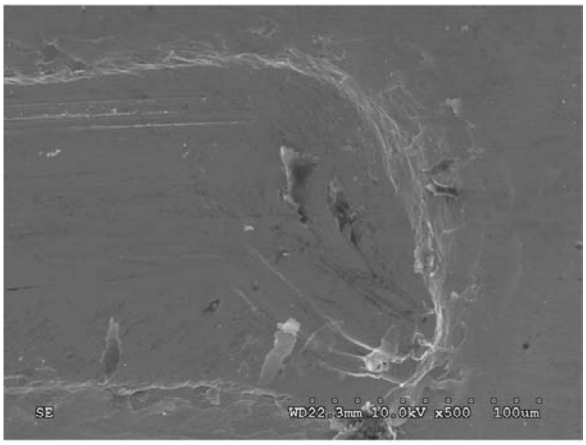


(b) *W*=0.6 mm

Fig. 5. SEM photography of scratch mark at upper surface.



(a) *W*=1.0 mm



(b) *W*=0.6 mm

Fig. 6. SEM photography of scratch mark at inner surface (D = 0.5 mm).

the compactness of DLC coating becomes lower with the decreasing of micro channel width and increasing of distance from upper surface.

3.2. Nanohardness

To evaluate the mechanical properties of DLC coating, nanohardness is measured. Table 1 shows the nanohardness of DLC coating at upper surface. The value of nanohardness is from 10.9 GPa to 13.7 GPa of different micro die, and the difference value is very small. However, the nanohardness of DLC coating at the inner surface is much lower as shown in Table 2. And the width of micro channel has an obvious effect on the nanohardness of DLC coating. With the decreasing of channel width, the hardness becomes lower. When we measure the hardness of points at the inner surface with different distance from upper surface, decreasing of hardness with increasing of D is observed. These results can be analyzed with energy of plasma. Since the width of micro-channel is very small, it has an obvious effect on the distribution of electric field, which leads to the energy of plasma inside of channel is much lower than that around upper surface. Then the deposited DLC coating becomes loose, which can also be proved by the SEM photography of inner surface.

3.3. Adhesion strength

Adhesion strength is a concerned parameter for the microforming process because of high contact pressure. The adhesion strength between DLC coating and substrate at the upper surface and inner surface is measured by scratch tests with load of 100 mN. The value of adhesion strength at upper surface is about 125 mN. But the value of adhesion strength at inner surface is not achieved. The scratch marks are observed with SEM as shown in Fig. 5 and Fig. 6. At the upper surface, only small part of DLC coating is peeled off, which indicates that the adhesion strength is higher. The failure mode is mainly brittle fracture when high contact pressure is applied. Fig. 6 shows the scratch marks at inner surface of different channel width. With decreasing of channel width, the peeled off part of DLC coating

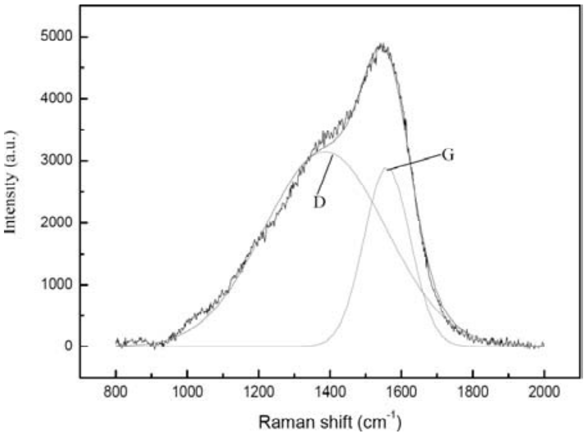


Fig. 7. Raman spectra of DLC coating inner surface (W = 0.6 mm).

becomes very small. Even the DLC coating is broken for the large plastic deformation of substrate, the DLC coating is not peeled off.

3.4. Microstructure

The results shown above indicate that channel width have an obvious effect on the mechanical properties of DLC coating. To investigate the phenomena in details, the microstructure of DLC coating at inner surface of channel is analyzed with Roman spectra, and the ratio of sp^3 and sp^2 bonds is calculated. Fig. 7 shows the results of Roman spectra. Two broad peaks are appeared, and they are D peak (Diamond) at about 1332 cm^{-1} and G peak (Graphite) at about 1580 cm^{-1}. The fraction of sp^3 and sp^2 bonds in DLC coating is correlated to the mechanical properties. Fig. 8 shows the ID/IG of inner surface DLC coating under various channel width. With the decreasing of channel width, the ratio of ID/IG increases significantly, and this indicates that the fraction of sp^3 bonds becomes lower. This is the

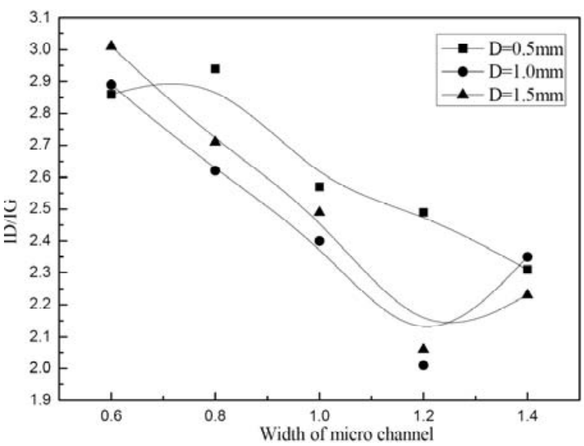


Fig. 8. ID/IG of DLC coating inner surface under various channel width.

reason of decreasing of nanohardness. The DLC coating becomes easily broke when the width of channel decreases. And the adhesion strength between DLC coating and substrate is improved for the increasing of sp^2 bond.

4. CONCLUSIONS

1) The inner surface is not smooth, lathlike and island topography is observed. With the increasing of distance from upper surface *D* and decreasing of channel width, the amount of lathlike structure decreases, and island structure increases. The compactness of DLC coating becomes lower.
2) The nanohardness of DLC coating is much lower at the inner surface than that at upper surface. And with the decreasing of channel width, the hardness becomes small. The reason may be that the energy of plasma inside of channel is much lower than that around upper surface.
3) At the upper surface, only small part of DLC coating is peeled off, which indicates that the adhesion strength is higher. The failure mode is mainly brittle fracture when high contact pressure is applied. With decreasing of channel width, the peeled off part of DLC coating becomes very small.
4) With the decreasing of channel width, the ratio of ID/IG increases significantly, and this indicates that the fraction of sp^3 bonds becomes lower. This is the reason of channel width effect on the properties of DLC coating.

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