Microwave Plasma-Assisted Etching for

Smoothing Polycrystalline Diamond Films

Dzung T. Tran¹, T. A. Grotjohn^{1,2}, D.K. Reinhard^{1,2} and J. Asmussen^{1,2}

¹Michigan State University, Electrical & Computer Engineering Department, East Lansing, MI 48824

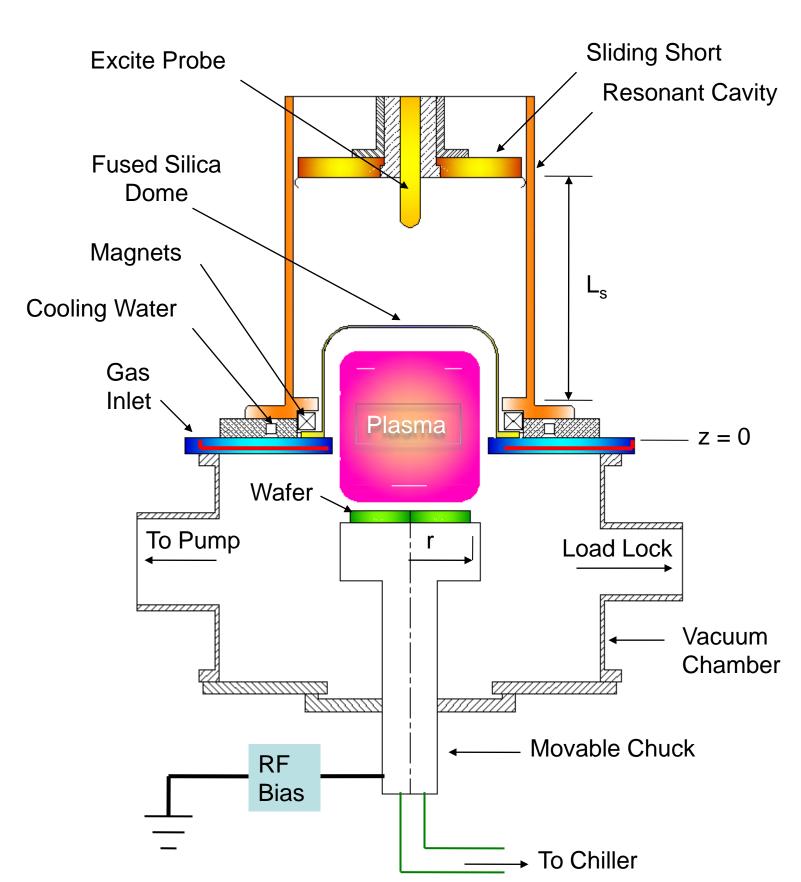
²Fraunhofer USA, Center for Coatings and Laser Applications, East Lansing, Michigan 48826-1226

Abstract

Polycrystalline diamond (PCD) films are promising for several applications for which a smooth surface is important, such as optical windows, X-ray masks, SAW filters and other electronic devices. Mechanical lapping and polishing techniques are fairly well established, however the diamond lapping removal rate is very low. Consequently there is interest in the development of more efficient smoothing methods. Examples include thermal-chemical polishing [1], laser polishing [2] and planarizing layers combined with oxygen ion-beam etching [3]. This paper describes methods to smooth polycrystalline diamond films using microwave plasma assisted etching combined with mechanical polishing and combined with sacrificial planarizing layers.

A 2.45 GHz, microwave plasma-assisted etching reactor is utilized for high etch rate processes on diamond substrates as previously described [4]. One surface smoothing method combines plasma etching with mechanical lapping/polishing. With this method the surface roughness R_a was improved from 3802 nm to 53 nm. Two other methods combine plasma etching and planarizing layers such that the etch is designed (1) to remove the planarizing layer and diamond at comparable rates or (2) to preferentially remove the exposed diamond and not the protecting planarizing layer. Planarizing layers tested include photoresist, Si₃N₄, and SiO₂. Sequential applications of the procedure produce optically smooth surfaces.

Experimental Equipment:



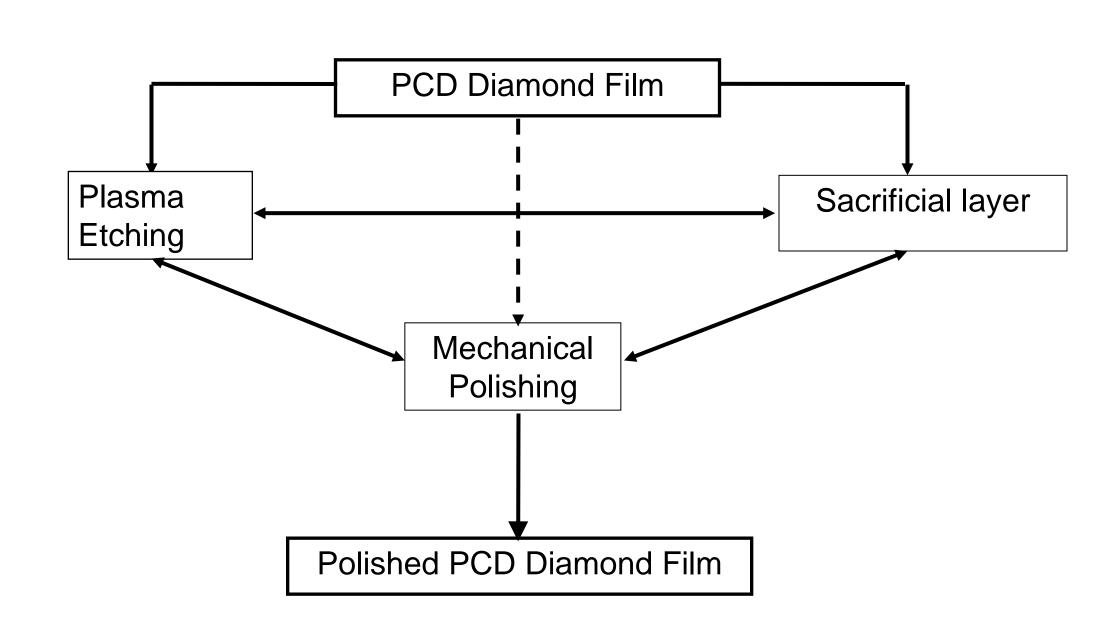


The ECR Microwave Plasma Diamond Etching Reactor Microwave Plasma Assisted Diamond Etching

-The etching system used in this study is a Lambda Technologies etcher that consists of a 25 cm diameter discharge located inside a 30.5 cm cavity applicator. It operates in two distinct excitation modes: (a) an ECR (electron cyclotron resonance) plasma source operating at pressures of 1-50 mTorr (used in this study) and (b) a non-ECR, non-magnetized mode operating at higher pressures of 50 mTorr-100 Torr.

-The surface roughness (both Ra and Rz) are measured using a Dektak surface profilometer.

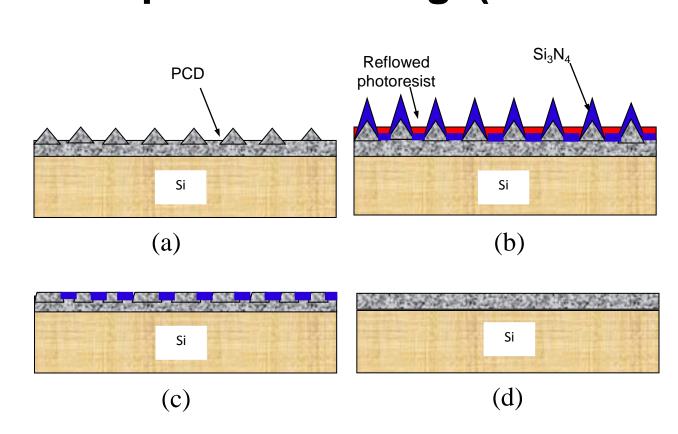
Diamond Smoothing



PCD Diamond Smoothing Techniques using Microwave Plasma Assisted Etching.

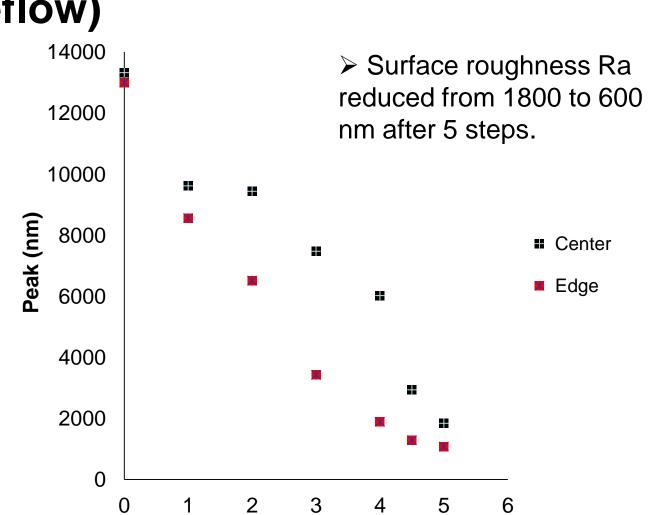
Polycrystalline diamond smoothing methods and results

 Method 1: Formation of hard mask in PCD surface valleys combined with plasma etching. (Photoresist reflow)



Photoresist Reflow Method

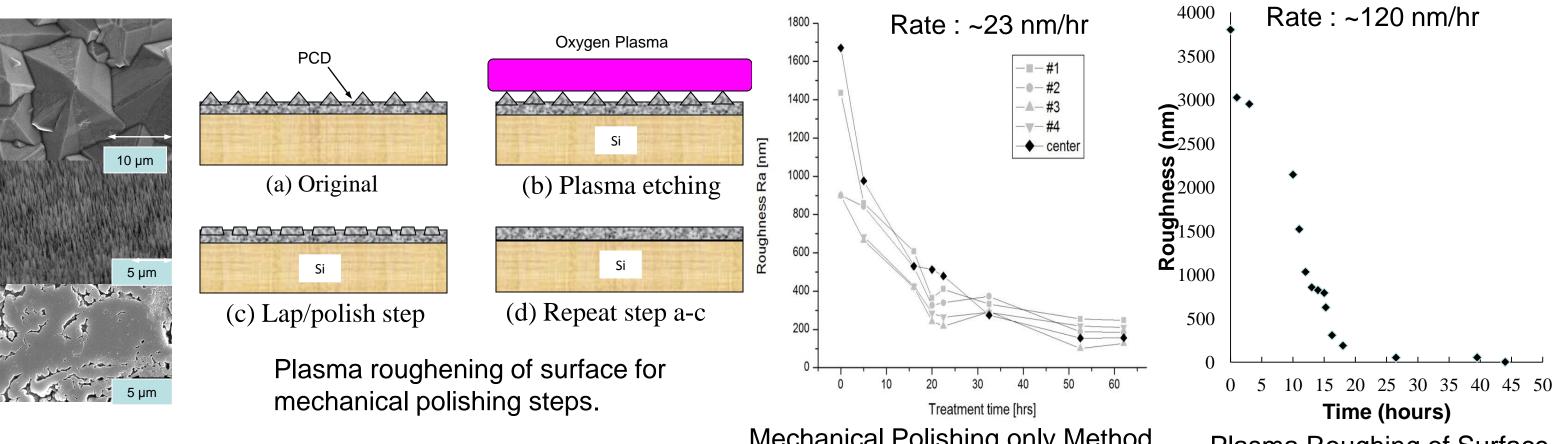
- (a) The original PCD diamond surface.
- (b) Surface formed by depositing Si₃N₄ (1μm thick) followed by photo resist (PR) 1813 (1.6 μm thick) on top of the Si₃N₄ layer. Then the PR is reflowed at 150 C followed by a Si₃N₄ plasma etch.
- (c) Etch the diamond for 30 min to get rid of the peaks of diamond surface. Use etch conditions that give Si₃N₄ a high selectivity.
- (d) Repeat steps a through c until the PCD surface is smooth.



PCD surface roughness (R_z) versus time <u>Etching condition:</u> Microwave power of 700 W, pressure of 4 mTorr, -125 V substrate bias, $Ar:O_2:SF_6 = 6:20:2$ sccm

➤ The purpose of using the sacrificial layers (photoresist and Si₃N₄) is to remove the protruded portion of diamond film using plasma etching. The process is repeated multi-cycle until the surface roughness is smoother.

Method 2: The microwave plasma etching to roughen the surface combined with mechanical polishing [5].



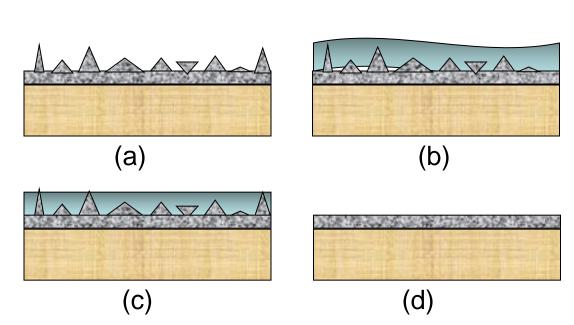
The whiskers that appear on the PCD diamond surface after oxygen plasma etching are easily removed by the mechanical lapping/polishing.

Mechanical Polishing only Method
(This work was done by K. Loewe
using a Logitech LP 50 machine).
#1-#4 are locations on substrate

Time (hours)

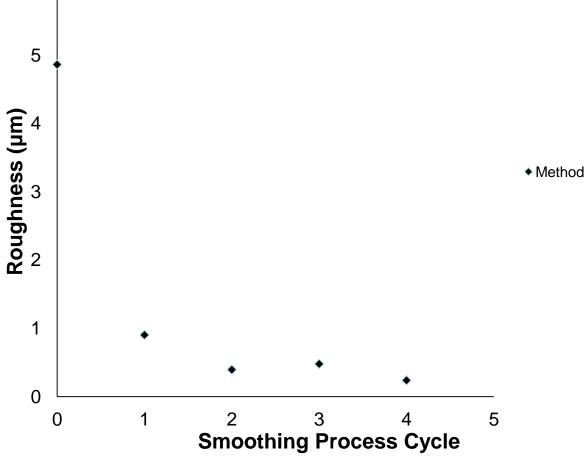
Plasma Roughing of Surface for Mechanical Polishing method up to 23 hours. At 27 hours roughness is Ra=53 nm. Final Ra=2 nm.

Method 3: The microwave plasma etching of diamond/sacrificial layer with selectivity of one combined with mechanical lapping/polishing [6].



Schematic of smoothing process using method 3:

- a) The original PCD diamond surface.
- b) PCD surface filled with SiO₂ film using PECVD system.
- c) Polishing surface using Logitech LP50 system.
- d) Plasma etching with selectivity of 1 to get rid of the protrude portion on PCD surface.
- e) Repeat steps b, c, d until the surface getting smooth.



PCD surface roughness (Ra) versus process cycles.

Summary

- Three methods to smooth thick PCD diamond substrates were investigated using microwave plasma etching combined with mechanical lapping/polishing and sacrificial layers.
- The method of forming a hard mask in the valleys of the PCD surface using a photoresist reflow technique worked well for removing the larger protrusions (>500nm) from the surface.
- The method of plasma etching with selectivity of 1 combined with mechanical lapping/polishing of a sacrificial SiO₂ layer quickly reduced the roughness to 300 nm. This method provides a flat surface (planar).
- The method of plasma roughening combined with mechanical polishing gave the smoothest surface at a rate faster than mechanical polishing alone. Below a surface roughness of about 50 nm, plasma-assisted methods appear to have no strong advantage over mechanical polishing alone.

References

- [1] M. Yoshikawa, Diamond Optics III, SPIE, 1325, 210 (1990).
- [2] A.M. Ozkan, A.P. Malshe, W.D. Brown, Diamond and Relat. Mater., 6, 1789 (1997).
- [3] D. F. Grogan, T. Zhao, B.G. Bovard, and H. A. Macleod, Applied Optics, 31, 1483 (1992).
 [4] D. T. Tran, T. A. Grotjohn, D. K. Reinhard, J. Asmussen, Diamond and Relat. Mater., 17, 717 (2008).
- [4] D. T. Tran, T. A. Grotjohn, D. K. Reinhard, J. Asmussen, Diamond and Relat. Mater., 17, 717 (2008). [5] Z. Xianfeng, M.A. Zhbin, W.U. Zhenhui et al, Plasma Science and Technology, 10, 3, 336-339 (2008).
- [5] Z. Xianfeng, M.A. Zhbin, W.U. Zhenhui et al, Plasma Science and Technology, 10, 3, 336-339 (2008).[6] Vivensang, L. Ferlazzo-Manin, M.F. Ravet, G. Turban, F. Rousseaux and A. Gicquel, Diamon and Relat. Mater, 5, 840 (1995).



