P. A. Dennig and D. A. Stevenson, to be published, "Applications of Diamond Films and Related Materials", Proceedings of the First International Conference on the Applications of Diamond Films and Related Materials, August 20-22,1991, Auburn, Alabama, USA, Edited by Y. Tzeng et al

INFLUENCE OF SUBSTRATE TOPOGRAPHY ON THE NUCLEATION OF DIAMOND THIN FILMS

Paul A. Dennig and David A. Stevenson

CONF-910854--3

DE92 010777

Dept. of Materials Science and Engineering, Bldg. 550,
Stanford University, Stanford, CA, U.S.A. 94305-2205

FG05-88ER45345

Abstract

Polycrystalline diamond films are of interest because of their unique materials properties. However, depositing films with the desired morphology is hindered by the difficulty in controlling the nucleation process. A popular method of enhancing the nucleation rate and density is to abrade the substrate with diamond powder before deposition. Although effective, this procedure is difficult to analyze since it may leave residual powder, may introduce damage, and may modify the surface topography. In order to separate these effects, we have studied the nucleation on chemically etched silicon substrates. Our results show that the majority of nucleation events occur on protruding surface features.

1. INTRODUCTION

The growth of a polycrystalline diamond thin film begins, after some incubation time, with the nucleation of individual particles on a substrate. A film is formed when the particles merge together to completely cover the substrate. The nucleation rate and density for a particular substrate may be reduced or enhanced by various means of substrate modification prior to deposition. A common method used to enhance nucleation is to scratch the surface with abrasive powder. Several explanations have been proposed for this effect, but none have gained universal acceptance. In an effort to separate the factors that are introduced with the scratching treatment, we have studied the respective behavior of scratched, fractured and chemically patterned silicon surfaces [1,2]. We have shown preferential nucleation behavior on sharp convex features. We have proposed nine possible explanations for this observation and have stated that carefully designed experiments are necessary to establish the relative importance each of the proposals [2]. In the present study, we extend our investigation of the influence of substrate topography on the nucleation of diamond particles grown on silicon substrates using new patterned surfaces.

2. EXPERIMENTAL

In the present study, we employ a unique topography originally designed to minimize reflections from the surfaces of solar cells. This substrate was chosen to provide additional information about nucleation on convex surfaces. The surface treatment consisted of masking a high resistivity <100> silicon substrate with a pseudo-random pattern of $2\mu m$ squares using standard integrated circuit photolithography techniques. The wafer is then etched in a KOH solution to form pyramids composed of (111) planes [cf. reference 3, for example]. In this surface treatment, there was no abrasion step and, hence, no possible residue of abrasive particles.

The following deposition conditions were used: a deposition time of 6 hours (10 hours for specimen of Fig. 5); gas activation with a tantalum hot filament (~2000° C) with no applied substrate bias; a gas composition of 1% methane in a balance of hydrogen; an ambient pressure of 30 Torr; and a substrate temperature of 900-1000° C. The quality of the deposited diamond was evaluated by producing a film under identical deposition conditions. The resulting film was evaluated by SEM, Raman spectroscopy, and ESCA and was shown to be of good quality for the same deposition conditions. For the present study, the initial stages of diamond particle growth were characterized by SEM. The results are shown in Figs. 1-4. We did not attempt to compare nucleation densities quantitatively for these substrates. However, in Fig. 5, the results of deposition on a linear v-groove pattern is provided and a comparison is made between the linear nucleation density for edges of roughly 54° versus 117°.

3. RESULTS

Figure 1 shows nucleation taking place in a non uniform pattern. Such non uniformity is often observed in diamond particle nucleation, indicating that subtle surface features are important. Figure 2 is an enlargement of the region shown in Fig. 1. Nucleation takes place predominantly on the upper portions of the pyramids rather than in the crevices. Figure 3 shows nucleation occurring along a path from the lower left to upper right of the view, with Fig. 4 portraying an enlargement of the region shown in Fig. 3. Nucleation takes place consistently on the tips of the pyramids in this location. In this experiment, we concur with the observations of Jansen, et al. [4], who report that nucleation was not observed in sharp concave features that were produced by chemical etching. In addition, we observe that nucleation takes place predominantly on convex surface features.

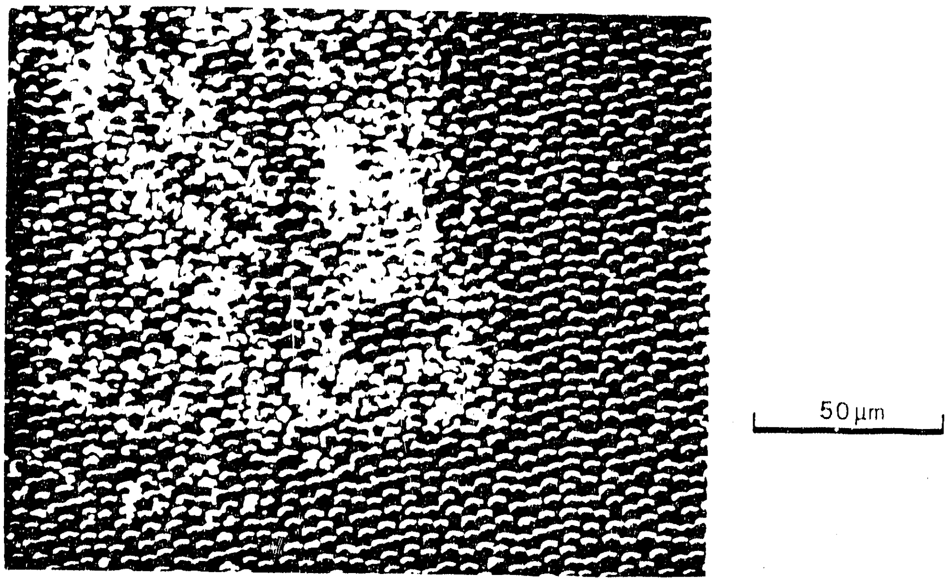

Figure 1. Enhanced nucleation on pyramids etched into <100> high resistivity silicon, 45° tilt.

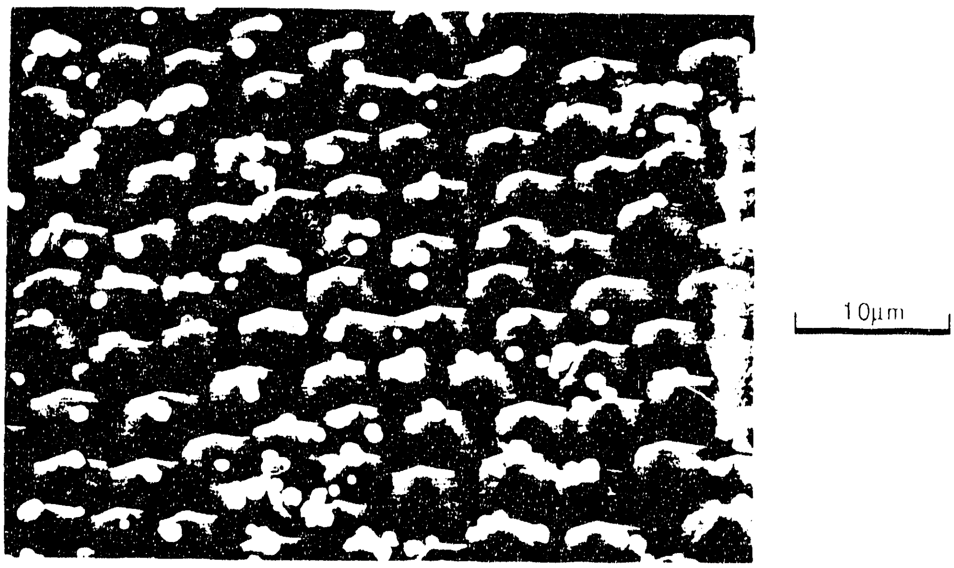

Figure 2. Enlargement of region shown in Fig. 1, 45° tilt. Nucleation occurs predominantly on the upper portions of the pyramids in this location, rather than in the crevices.

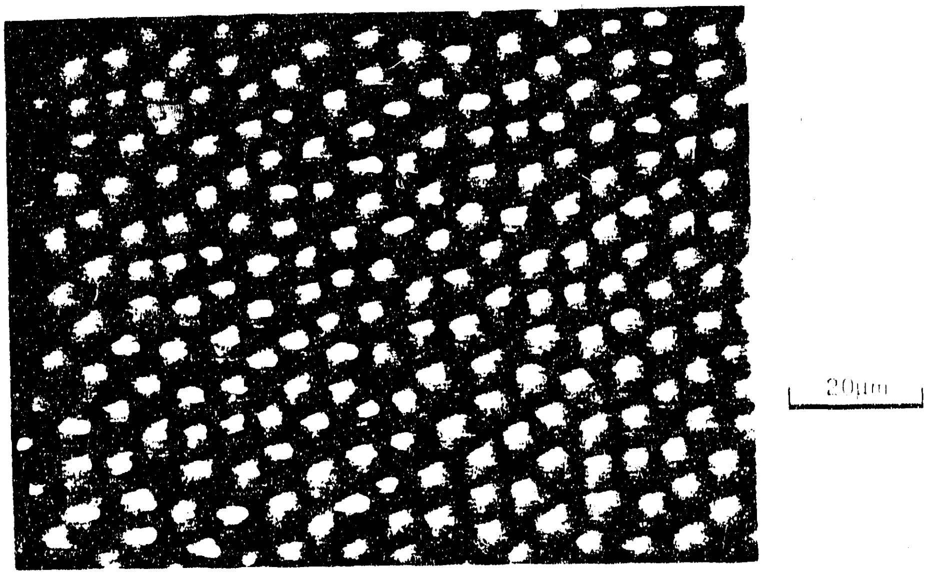

Figure 3. Nucleation on pyramids etched into <100> high resistivity silicon, 15° tilt. Nucleation occurs along a path from lower left to upper right.

Figure 4. *Enlargement of region* shown in Fig. 3, 45° tilt showing nucleation on the tips of the pyramids. Note the unusual diffuse structural features on the pyramid tips in the path of nucleating particles.

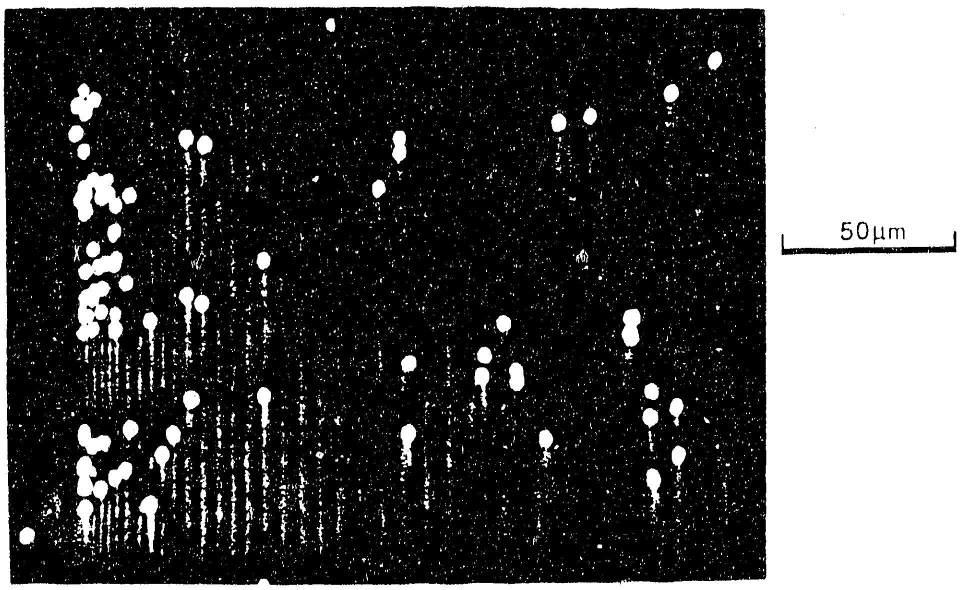

Figure 5. Enhanced nucleation on edges, 15° tilt, showing a preference for nucleation on the more acutely angled edges.

In Fig. 5, V-grooves of increasing width (but constant depth) are etched into a silicon <100> surface, without using an abrasion treatment. Nucleation occurs preferentially on the edges of silicon bounding the grooves. There is roughly 8 times the nucleation density (particles/unit length) on the more acutely angled edges on the left, as compared to those on the right.

4. CONCLUSIONS

There is extensive use of surface abrasion procedures to enhance diamond nucleation, usually using diamond powder as the abrasive. The enhancement might arise from residual diamond powder, mechanical damage, and changes in the surface topography. We propose that at least part of this nucleation enhancement is due to a change in the surface topography in the form of prominent surface features developed on the sides of the scratches. We have endeavored to demonstrate this influence by patterning surfaces so that mechanical damage and any residual powder used in the abrasion process is absent. In an earlier study, we patterned silicon surfaces to form ridges, pits, and mesas by preferentially etching silicon <100> substrates using photolithographic techniques. We observed that nucleation was favored on prominent features of the substrate surface, i.e., features that protrude with sharp edges or apexes as opposed to valleys or pits [1,2]. (We have made similar observations with molybdenum and silicon substrates.) Our present work with pyramid textured surfaces lends additional support for these observations. We have made a list of nine possible explanations for the preferential nucleation of diamond on sharp convex edges [2]. The most likely of these are: minimizing the interfacial energy for diamond nuclei forming on sharp convex surfaces; the presence of more dangling bonds at these sites; and a larger reactant flux at these sites.

6. ACKNOWLEDGEMENTS

The authors acknowledge Professor Richard Swanson and his research group and David Kane of Stanford University for providing the textured substrates, and the generous support provided by the U.S. Department of Energy, contract No. DE-FG05-88ER45345.

6. REFERENCES

1 P.A. Dennig and D.A. Stevenson, New Diamond Science and Technology (Proceedings of the 2nd International Conference on New Diamond Science and Technology), edited by R. Messier and J.T. Glass, MRS International Conference Proceedings Series (Materials Research Society, Pittsburgh, PA, 1991).
2 P.A. Dennig and D.A. Stevenson, submitted to Appl. Phys. Lett.
3 S. Wolf and R.N. Tauber, Silicon Processing for the VLSI Era, Lattice Press, 1986, 531-532.
4 F. Jansen, M.A. Machonkin, and D.E. Kuhman, J. Vac. Sci. Technol., A 8 (1990) 3785 .

END

DATE
FILMED
5/13/92