Synthesis, Reliability and Applications of Nanocrystalline CVD-grown Diamond and Micro Device Fabrication

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ABSTRACT

Nanocrystalline diamond layers have been deposited using hot-filament chemical vapor deposition. By variation of the deposition conditions we are able to adjust the grain size over a wide range from 300 nm up to values lower than 10 nm resulting in very smooth surfaces. Investigations of the mechanical properties show a decrease of the Young's modulus with decreasing grain size. Finally, potential industrial applications of nanocrystalline layers like diamond coatings of cutting tools and diamond tooth wheels are presented.

Keywords: nanocrystalline diamond, hot filament chemical vapor deposition, micro mechanical parts, cutting tools

1 INTRODUCTION

The correlation between the micro- and nanostructure of a material and its physical and chemical properties is the key issue in materials development. Considerable progress has been achieved recently by the development of new processing technologies (hot filament chemical vapor deposition – HFCVD [1-3]) and new materials in a nanocrystalline state with superior mechanical strength and tribological properties [4-6].

Further processes based on lithographic techniques known from silicon technology allow further microstructuring of CVD-diamond. So far, the microstructuring of highly oriented columnar diamond [7] has been hampered by the fact that the internal microstructure is being reproduced by plasma etching yielding rather rough surfaces. This problem now can be overcome by the production of nanocrystalline diamond, i.e. when the grain size is smaller than the acceptable surface roughness. It can be expected that microparts (microtoothed wheels, atomically sharp cutting edges, functionalized diamond surfaces etc.) can be produced on a reliable basis in the near future.

2 EXPERIMENTAL

Nanocrystalline diamond (NCD) films were grown in a CemeCon CC800/Dia hot-filament CVD reactor [8]. Both silicon (100) wafers with a thickness of 350 µm and a diameter of 75 mm and WC-Co hard metal components were used as substrates. In order to achieve a nanocrystalline structure, a new process has been developed to achieve high nucleation densities and controlled growth conditions using a mixture of H_2, CH_4, N_2, O_2 and Ar as a feedstock gas. Only this allows to obtain extremely smooth surfaces which are not available using other techniques, for example by microwave plasma enhanced CVD. The substrates were heated only by thermal radiation of the filaments to a temperature of ~ 750 °C. The gas pressure was fixed to a constant value during deposition in the range between 3 and 25 bar.

Scanning electron microscopy (SEM) was performed using a Zeiss Leo 1540. The roughness of the diamond surface was measured by an atomic force microscopy (AFM) (Dimension 3100, Digital Instruments). A Laser surface acoustic wave system (LSAW) by Fraunhofer IWS Dresden carried out the determination of the Young's modulus. A Philips X'Pert instrument was used for X-Ray diffraction (XRD) measurement. For the analysis presented here, we used a standard Bragg-Brentano configuration. An estimation of the average grain size in the films was obtained by application of the well-known Scherrer formula [9]

$$L = \frac{K\lambda}{\beta_{FWHM}\cos\theta} \tag{1}$$

where θ and β_{FWHM} are the angle and the full width at half maximum of the considered XRD peak, respectively, λ the wavelength of the used $Cu_{K\alpha}$ X-Rays and K a form factor, which is assumed to be equal to one for our estimation.

3 SYNTHESIS

For comparing the morphology of nanocrystalline diamond it is of great importance that all films are of the same thickness, as both the grain size and the roughness of a thin film usually increases with increasing film thickness. Thus, all films shown in this section are ~ 1.5 µm thick. Figure 1a, 1c and 1e show three representative SEM micrographs of diamond thin films deposited on Si wafers. The morphology changes from polycrystalline structure (Fig. 1a) with a grain size of 300 nm over a nanocrystalline (Fig. 1c) with a grain size of 60 nm to a fine-grained nanocrystalline morphology (Fig. 1c) with a grain size of 8 nm. By slight changes of the deposition conditions all grain sizes within this range can be deposited.

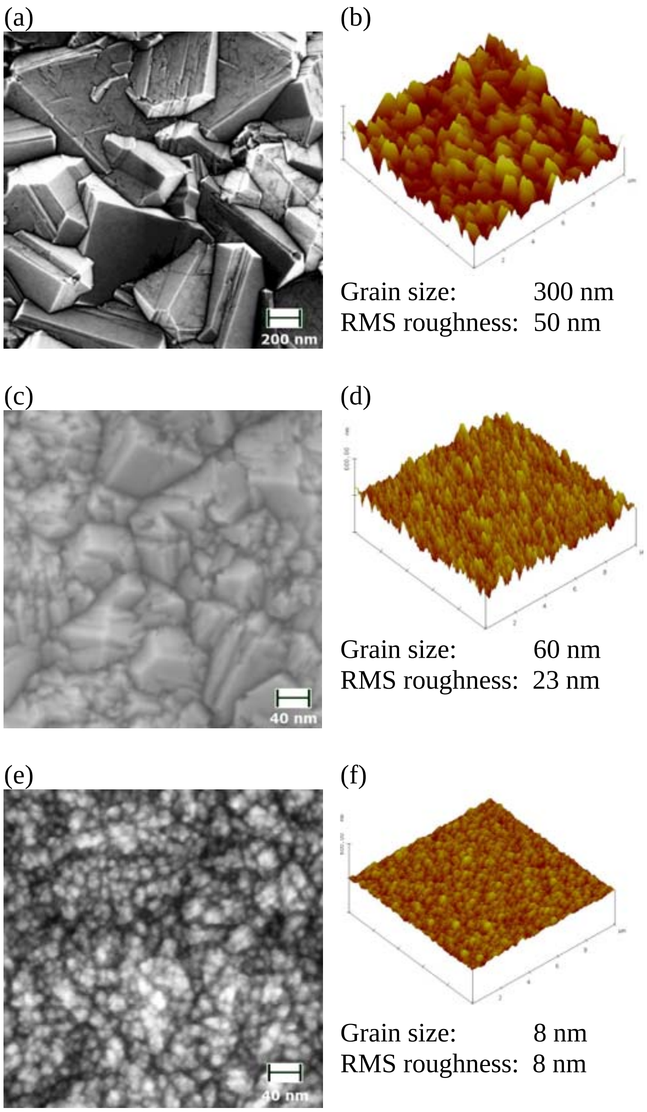

Figure 1: SEM (a, c, e) and AFM (b, d, f) micrographs of diamond samples with grain sizes of 300 nm (a, b), 60 nm (c, d) and 8 nm (e, f).

The AFM measurements (Fig. 1b, 1d, 1e) reveal that the RMS roughness of the diamond surface decreases with decreasing grain size leading to a very smooth surface with an RMS roughness of ~ 8 nm for the fine-grained sample. Samples with such a low roughness will show low friction which is important for applications shown in the next section.

The Young's modulus is an important material property which affects the stress – strain behavior, bending, propagation of cracks and delamination from a substrate. Figure 2 illustrates the dependence of the Young's modulus on the grain size. A decreasing grain size leads to a decrease of the Young's modulus. This is caused by a higher ratio of the grain boundary regions at the fine-grained samples leading to a higher fraction of non-sp^3-bonds which reduces the Young's modulus.

The nanocrystalline samples almost reach the calculated value for randomly oriented polycrystalline diamond of 1143 GPa [10]. However, a Young's modulus of ~ 750 GPa for the fine-grained samples with a grain size lower than 10 nm is still a remarkable result compared with other materials like tungsten carbide which exhibits a Young's modulus of ~ 500 GPa [11].

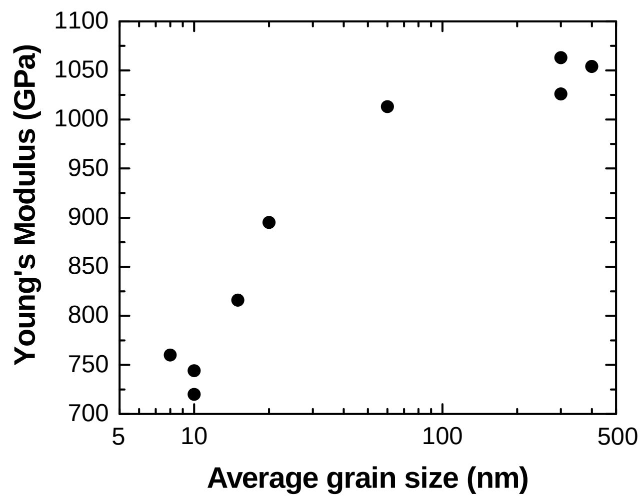

Figure 2: Young's modulus depending on the grain size.

4 APPLICATIONS

Because of the fact that the NCD show a low residual stress, a high hardness and Young's modulus this material is suitable for industrial applications. In this chapter we present the use of diamond for toothed wheels as an example for micro mechanical parts and the diamond coating of cuttings tools.

4.1 Diamond micro mechanical parts

The main advantage can here be taken from the excellent mechanical properties and the low coefficient of friction. Thus, a lubricant-free operation of diamond micro gears at high revolution speed, low wear, low moment of inertia, high efficiency and high reliability is achievable. To evaluate the potential of this application, diamond micro gears have been designed, fabricated and characterized. Figure 3 shows a Scanning Electron Micrograph (SEM) of a diamond micro gear.

Regarding the small size of the diamond toothed wheels, mounting and adjusting them one to another, leads to problems not known in standard gear production. To enable a sufficient vertical overlap for precise movement, their thickness was chosen to be 150 µm. Even with very high aspect ratio etching processes, a lateral under etch of the mask cannot be entirely suppressed. However, the optimization of the plasma processes allowed the realization of micro structured diamond toothed wheels having sidewall angles of 90° ± 2° at a thickness of 150 µm.

From the toothed wheels, a micro gear was assembled using an aluminum base plate with bores of 0.2 mm diameter for the center axis of each wheel. A nickel-wire of 200 µm diameter was used as axis material. The vertical adjustment of the wheels was realized gluing copings on top of the axis. For actuation, a Faulhaber micro motor series 0206H was used, which was specified to max. 100,000 rpm with a maximum torque of 7.5 µNm. The original output-wheel was replaced by a diamond wheel.

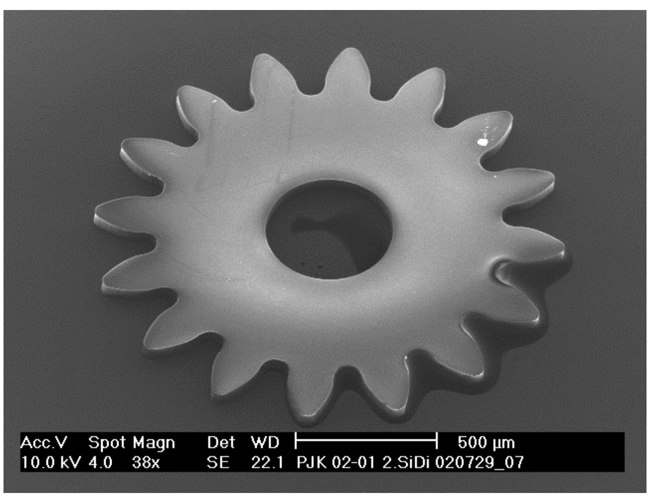

Figure 3: Scanning electron micrograph of a micro mechanical diamond toothed wheel.

The diamond gear could be successfully driven in this test setup with a maximum revolution speed of 35,000 rpm. After more than 200 min at 20,000 rpm, the gear showed a parasitic destructive breakdown due to a failure of the motor, which was caused by metal particles from the base plate and the axes. A strong wear can be observed after the operation, which is caused by the still too rough diamond sidewall surfaces. The blocking of the gear caused by particles, resulted not in a breakdown of the wheels, but of the motor itself. Wear of the diamond wheels could not be observed.

4.2 Diamond cutting tools

After a chemical pre-treating in order to decrease the cobalt concentration of our tungsten carbide (WC) substrates, we deposited a 10 to 15 µm-thick NCD film onto the substrate. The radius of curvature of the cutting edge, however, increases from an initial value of ~ 1 µm to about 15 µm after deposition. Although the hardness of the tool surface has been significantly increased by the diamond film, the tool is no longer usable since the radius of curvature is too large and the tool is not cutting any more. This is the reason why we introduced a plasma sharpening procedure in order to reduce the procedure. Figure 4 shows a schematic view of this process. By etching the diamond layer using Reactive Ion Etching (RIE) from both the top side and the bottom side the radius of curvature of the cutting edge is decreased to values of smaller than 0.2 µm, which is noticeable smaller than that of the original work piece.

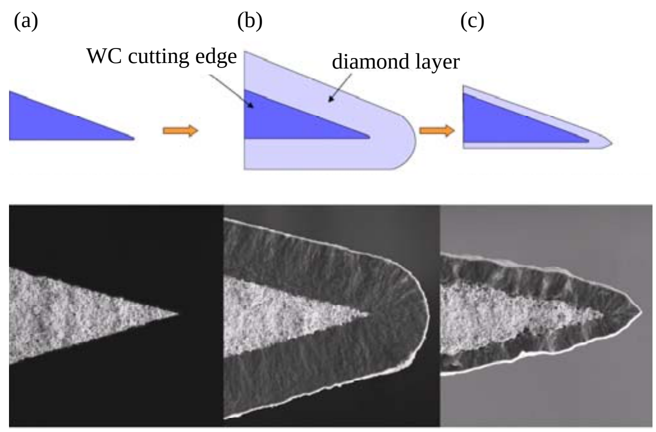

Figure 4: Principle of the plasma sharpening. After depositing the uncoated tungsten carbide (WC) cutting tool (a) with a NCD layer of the thickness ~ 15 µm the radius of curvature is increased. After etching the diamond layer by RIE from the top side and the bottom side (c) the radius of curvature of the cutting edge is decreased to values even smaller than the original work piece.

Several blades were tested in order to compare the life span of the diamond coated, sharpened, cutting edges with those of uncoated carbide or ceramic blades. The experiment was performed using cutting edges having a cutting angle of 15° by cutting a plastic foil with Titanium oxide filling material. The thickness of the foil was approx.

0.2 mm. The number of tested blades was 150. The carbide and the ceramic blades had comparable life spans of 1.5 and 2.0 days respectively. The sharpened, diamond coated, blades showed life spans of approx. 36 days. The life span increase of the sharpened, diamond coated, blade compared to the carbideblades was therefore 24 times (see also Fig. 5).

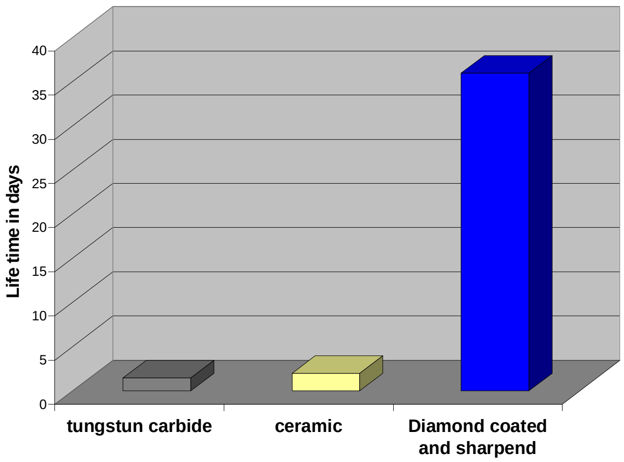

Fig. 5: Comparison of the life span of tungsten carbide, ceramic and sharpened, diamond coated, cutting blades.

5 CONCLUSION

Nanocrystalline diamond layers on silicon and on tungsten carbide were prepared using hot-filament vapor deposition. By slight changes of the source gases we are able to adjust the crystalline size over a wide range from 300 nm to less than 10 nm. The latter sample exhibit a very smooth surface with an RMS roughness of 8 nm.

We presented the use of diamond for toothed wheels as an example for micro mechanical parts and the diamond coating of cuttings tools. For the latter we showed a new technique to reduce the radius of curvature of the cutting edge improving the cutting properties and extending the lifetime of the tool by a factor of 10.

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REFERENCES

[1] S. Matsumoto, Y. Sato, M Tsutsami and N Setaka, J. Mater. Sci. **17** (1982) 3106.

[2] A. Afzal, C. A. Rego, W. Ahmed and R. I. Cherry, Diamond Relat. Mater. **7** (1998) 1033.

[3] S. Pecoraro, J. C. Arnault and J. Werckmann, Diamond Relat. Mater. **13** (2004) 342.

[4] M. Roy, V. C. George, A. K. Dua, P. Raj, S. Schulze, D. A. Tenne, G. Salvan and D. R. T. Zahn, Diamond Relat. Mater. **11** (2002) 1858.

[5] C. Z. Gu and X. Jiang, J. Appl. Phys. **88** (2000) 1788.

[6] S. O. Kucheyev, J. Biener, J. W. Tringe, Y. M. Wang, P. B. Mirkarimi, T. van Buuren, S. L. Baker, A. V. Hamza, K. Brühne and H.-J. Fecht, Appl. Phys. Lett. **86** (2005) 221914.

[7] A. Flöter, H. Güttler, G. Schulz, D. Steinbach, C. Lutz-Elsner, R. Zachai, A. Bergmaier and G. Dollinger, Diamond Relat. Mater. **7** (1998) 283.

[8] S. Schwarz, S.M. Rosiwal, M. Frank, D. Breidt and R.F. Singer, Diamond Relat. Mater. **11** (2002) 589.

[9] P. Scherrer, Gött. Nachr. **2** (1918) 98.

[10] F. Szuecs, M. Werner, R.S. Sussmann, C.J. Pickles, H.J. Fecht, J. Appl. Phys. **86** (1999) 6010.

[11] ASM Engineered Materials Reference Book, ASM International (1989), pp.182.