ELSEVIER

Diamond and Related Materials 12 (2003) 1041–1044

DIAMOND AND RELATED MATERIALS

www.elsevier.com/locate/diamond

Micro-machine fabrication using diamond-like carbon films

A.P. Mousinho[a,*], R.D. Mansano[b], M. Massi[c], J.M. Jaramillo[c]

[a]*CEETEPS – FATEC-SP, São Paulo, SP, Brazil*
[b]*LSI PSI EPUSP, Av. Prof. Luciano Gualberto, 158 – trav. 3, CEP 05508-900, São Paulo, SP, Brazil*
[c]*LPP, Departamento de Física, ITA, CTA, CEP 12228-900, São José dos Campos, SP, Brazil*

Received 19 July 2002; accepted 21 August 2002

Abstract

In this work we use diamond-like carbon (DLC) films deposited by a RF magnetron sputtering system in micro-electro mechanical systems (MEMS) development. The principal applications of DLC films in MEMS application are micro-channels for microfluidric devices, mechanical micro-machines, micro-optical devices, and mechanical actuators. These films were produced by a reactive RF magnetron sputtering system from a target of carbon in a stable graphite allotropic form with purity of 99.9999% and methane plasma. The DLC films were deposited on silicon substrates. The deposition rate was 2.5 nm/min and the films showed low mechanical stress. The films were patterned by a lithography step and the structures were obtained by a reactive ion etching using oxygen plasma. The etching rate in this process was 1 μm/min. The film thickness was measured with a height step meter and a ellipsometer. Fourier transform infrared (FTIR) and Raman spectroscopy were used to identify the sp^2 and sp^3 hybridization of C, –CH bonds and other possible bonds that can appear; atomic force microscopy (AFM), was used to measure film roughness.

Keywords: Plasma etching; Sputtering; Diamond-like carbon films; Micro-machining; Amorphous hydrogenated carbon

1. Introduction

Diamond-like carbon (DLC) films are promising materials for micromechanical devices [1], because they are hard, hydrophobic, exhibit relatively low surface energies, and can also be doped to enhance their electrical conductivity. They have other properties, such as thermal stability, chemical inertness [2] and low stress, that are very interesting for other applications. Endurance of the carbon films is also important, especially in applications where wear due to repetitive contacts with the film (gears) or contacts with other materials (microchannels) may occur [3].

These films can be deposited by several techniques, such as chemical vapor deposition (CVD), mass selected ion beam systems, laser ablation and sputtering [4]. The film characteristics depend on several different deposition process parameters, such as the gas composition and pressure, the substrate temperature, the energy of the impinging ions, etc. [5]. The presence of hydrogen in the gas phase promotes the formation of sp^3 C structures [6]. For micromachine purposes, these films are usually obtained by PECVD techniques using different hydrogen sources, such as H_2 and CH_4 [6–8], and by reactive sputtering of carbon, adding one of these gases to argon [7,9].

Raman spectroscopy and Fourier transform infrared spectroscopy (FTIR) are the techniques more frequently used to characterize these materials [9–13].

2. Experimental

The films were produced in a reactive RF magnetron sputtering system. The target is a 99.9999% pure, 6-inch diameter, graphite plate, located at 100 mm from the substrate. A pump system composed of a rotary vane and a turbo-molecular pump, was used to attain a

*Corresponding author. LSI/PSI/EPUSP – Universidade de São Paulo, Av. Prof. Luciano Gualberto, 158 –trav 3, CEP: 05508-900 São Paulo, SP, Brazil. Tel.: +55-11-3091-5660; fax: +55-11-3091-5665.

E-mail address: mousinho@lsi.usp.br (A.P. Mousinho).

0925-9635/03/$ © 2002 Elsevier Science B.V. All rights reserved.
PII: S0925-9635(02)00219-4

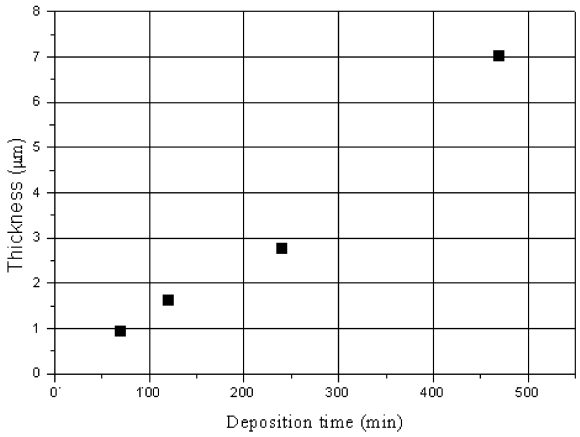

Fig. 1. Thickness of the layer of the diamond-like carbon as a function of deposition time.

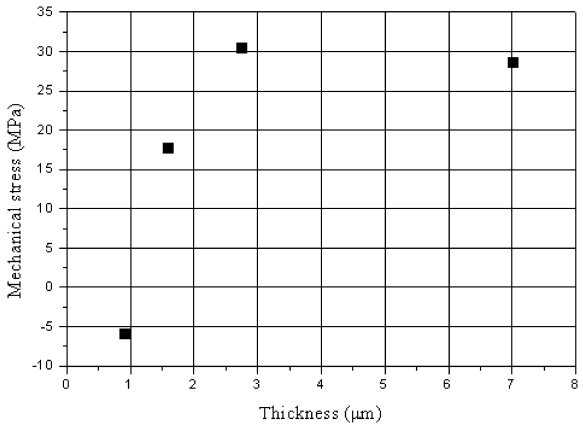

Fig. 2. The internal stress of diamond-like carbon films as a function of thickness.

residual pressure of 4×10^{-6} torr. The process pressure was kept constant at 5×10^{-3} torr. The total flow was also kept constant at 70 sccm of the CH_4. The RF power was applied at 13.56 MHz and kept constant at 150 W. The substrate temperature was not controlled, but measured by a type K thermocouple, which indicated that the temperature never exceeded 90 °C.

Two types of wafers were used: traditional three-inch diameter, 280 μm thick, p-type, (100), test quality silicon wafers with resistivities in the range of 10–20 Ω cm, and three-inch diameter, 400 μm thick, double side polished, p-type, prime quality wafers with a resistivity of 5 Ω cm. The high quality wafers were used for FTIR measurements, the lower quality wafers for the other tests. All were submitted to a Piranha clean, followed by a diluted HF dip, before the film deposition.

A thin silicon layer (100 nm), was deposited on the DLC films to be used as a mask in the plasma etching process of the structures. This film was also deposited by a magnetron sputtering using a pure silicon target (99.999%) and argon plasma. In order to obtain devices, after the silicon films deposition, structures were defined using conventional and e-beam lithographs. The silicon film was etched by a SF_6 plasma (50 mtorr pressure and 100 W RF power at 13.56 MHz) [14]. After this, an oxygen plasma was produced in the reactive ion etching (RIE) reactor[15] in order to etch the DLC films and produce the devices.

The thickness of the films was measured with a Dektak 3030 step height meter, and also with a Rudolph AUTOEl-NIR3 ellipsometer. The surface roughness of the as deposited and of the etched films was measured using an atomic force microscope (AFM) Nanoscope III from Digital, by scanning areas of 5 μm×5 μm, on the film surface.

The mechanical stress was analyzed by measuring the curvature of the wafers before and after the film depositions. In the case of the a-C:H films an analysis of the internal stress was performed for the case of film deposition just on the polished surface of the substrates. The Raman spectra of the films, in the range of 1060–1930 cm^{-1}, were obtained by the Renishaw micro Raman imaging microscope system.

The chemical composition was measured by a Bio-RAD FTS 40 FTIR spectrometer in the range of 400–4000 cm^{-1} and the structures were observed by scanning electronic microscopy.

3. Results and discussion

Fig. 1 shows the thickness of the layer of the DLC as a function of deposition time. The process time was varied from 1 to 7 h and the deposition rate of the investigated processes remained constant.

The FTIR analyses show the different C–H_x sp^2 and sp^3 peaks for the a-C:H film. In the Bessel deconvolution fit for the a-C:H films are shown the following peaks: sp^3 CH_3 at 2875 cm^{-1}, sp^3 CH_2 at 2920 cm^{-1}, sp^3 CH_3 at 2960 cm^{-1} and sp^2 CH at 3000 cm^{-1} [2].

In order to quantify the results of the FTIR analyses, Bessel deconvolutions were performed on the FTIR spectra obtained and the area of the sp^2 CH peak was compared with the sum of the areas of the three sp^3 peaks, the C–H sp^3/sp^2 ratio is 228.

The Raman imaging microscope system (Renishaw) using 632.8 nm laser light obtained the Raman spectra of the films, in the range of 200–2000 cm^{-1}. The spectra were fitted into two Gaussian lines to analyse the shape, frequency position and width of the D and G bands. The Raman spectra of these films result in 1359 and 1585 cm^{-1} D and G band position, with 225 and 117 band width, respectively. The intensity ratio of D and G bands (I_D/I_G) is 2.18.

The DLC films present low values of internal stress,

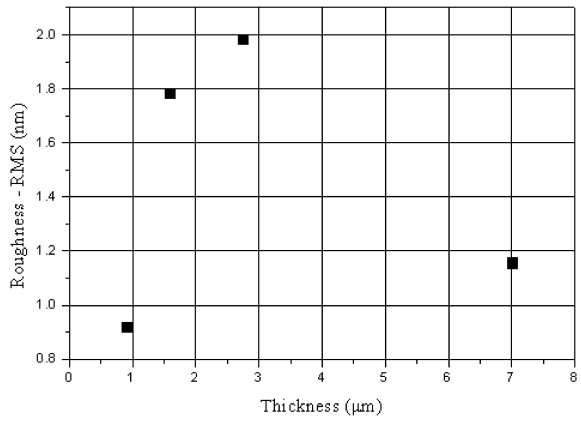

Fig. 3. Surface roughness as a function of the thickness of the diamond-like carbon films.

especially at a high thickness when compared with SiO_2 and Si_3N_4 films. The internal stress of DLC films as a function of thickness is shown in Fig. 2. The decrease of the stress with the thickness is a contrary behavior when compared with SiO_2 and Si_3N_4 films where the total stress increases with the thickness.

The variation of the deposited film surface roughness as a function of the thickness of the DLC films is shown in Fig. 3. These films were deposited on Si wafers with RMS roughness of 0.16 nm; the roughness level increases to 2 nm for a 2-μm-thick film and decreases to 1.2 nm for a 7-μm-thick film. These results were independent of the size of the scanned area.

The DLC films were etched in oxygen plasmas (Fig. 4). The etch rate increased with process pressure and RF power. At higher power levels, more oxygen molecules are dissociated into oxygen atoms, thereby increasing the chemical etching of the carbon atoms of the DLC films. The scattering of the incoming ions for gas phase molecules causes the lower anisotropy in the higher pressure. In this case the ideal process is a low

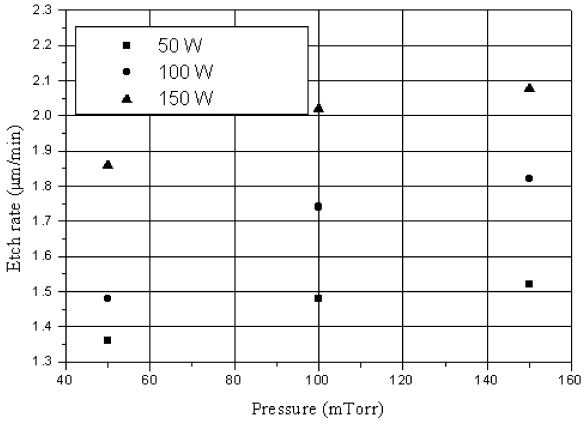

Fig. 4. Plasma etch rate of DLC films, etched in oxygen plasmas.

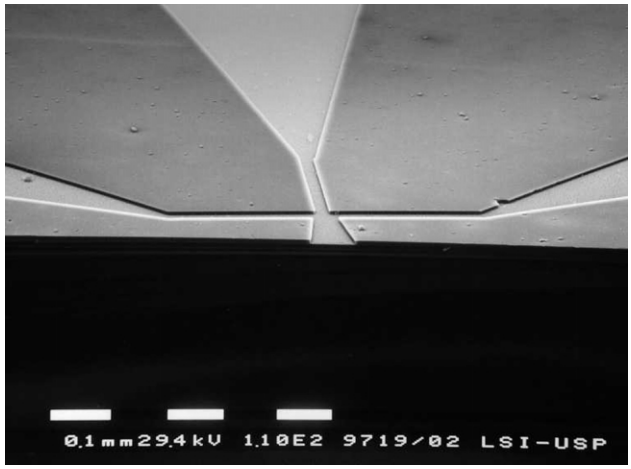

Fig. 5. Micro-channel made in a DLC film with a height of 8 μm and width of 25 μm.

pressure (100 mtorr) and high RF power (150 W) (Figs. 5 and 6).

Fig. 5 shows a micro-channel made in the DLC film of 8 μm height and 25 μm width and Fig. 6 shows a mold for electrochemical deposition with 8 μm height and 80 μm diameter.

4. Conclusions

DLC films were sputter-deposited using a graphite target with CH_4 plasmas, at low temperatures. FTIR and Raman characterization showed that the films deposited with CH_4 containing plasmas are richer in sp^3 type carbon. The deposited films present low levels of stress (30 Mpa) while the RMS surface roughness of the deposited films was always lower than 2 nm, with possible applications to micromachines (gears) microstructures (micro-channels) using lithography and the

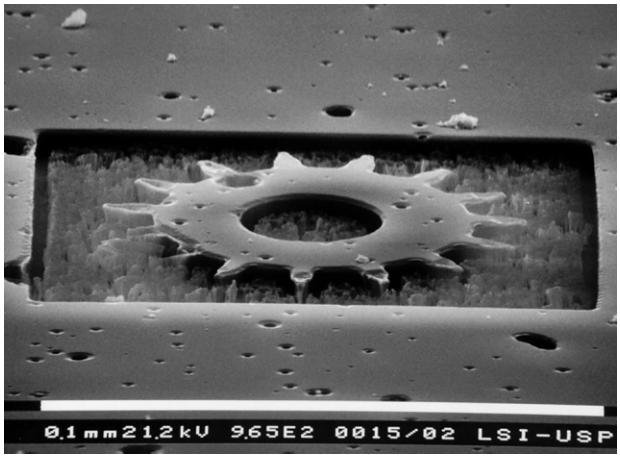

Fig. 6. Gear made in DLC film with a height of 8 μm and diameter of 80 μm.

plasma etching process. The characteristics of DLC films such as low mechanical degradation, high chemical resistance, low temperature deposition and easy patterning and etching observed in this work show that these films are promising materials to be used as structural layers in MEMS development.

Acknowledgments

The authors would like to thank Dr Antonio C. Seabra for the support in e-beam lithography, technician Gilberto K. Nishioka for the technical support in AFM analyses, technician Alexandre Marques Camponucci for the technical support in SEM analyses. They also thank Lucia Vieira Santos for the Raman analyses. The financial support of Fapesp, CNPq and CAPES is also appreciated.

References

[1] R. Maboudian, Surface Processes in MEMS Technology Surface Science Reports 30, Elsevier, 1998.
[2] J.M. Jaramillo, R.D. Mansano, L.S. Zambom, M. Massi, H.S. Maciel, Diamond Relat. Mater. 10 (2001) 976.
[3] M. Madou, Fundamentals of Microfabrication, CRC Press LLC, Florida, 1997.
[4] Y. Lifshitz, Diamond Relat. Mater. 5 (1996) 388.
[5] A. Grill, Diamond Relat. Mater. 8 (1999) 428.
[6] J. Robertson, Adv. Phys. 35 (1986) 317.
[7] H-C Tsai, D.B. Bogy, J. Vac. Sci. Technol. A 5 (1987) 3287.
[8] A. Grill, B.S. Meyerson, Synthetic Diamond: Emerging CVD Science and Technology, Wiley, New York, 1994.
[9] M. Massi, R.D. Mansano, H.S. Maciel, C. Otani, P. Verdonck, I.N.B.N. Nishioka, Thin Solid Films 343/344 (1999) 378.
[10] G.A. Clarke, Y. Xie, J.E. Eldridge, R.R. Parsons, Thin Solid Films 280 (1996) 130.
[11] X.L. Peng, T.W. Clyne, Thin Solid Films 312 (1998) 207.
[12] M.J Paterson, Diamond Relat. Mater. 7 (1998) 908.
[13] A. Khan, J.A. Woollam, Y. Chung, Solid State Electronics 27 (1984) 385.
[14] R.D. Mansano, P. Verdonck, H.S. Maciel, Appl. Surf. Sci. 110/111 (1996) 583.
[15] M. Massi, R.D. Mansano, H.S. Maciel, C. Otani, P. Verdonck, I.N.B.N. Nishioka, Thin Solid Films 343/344 (1999) 378.