

Diamond-Like Nanocomposite (DLN) Films for Microelectro-Mechanical System (MEMS)

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ABSTRACT

Diamond-like nanocomposite (DLN) thin films were deposited on pyrex glass or silicon substrate by plasma enhanced chemical vapor deposition (PECVD) method. These types of films have their unique number of structural, mechanical and tribological properties, which are quite similar with MEMS material properties. DLN films provide a number of unique and attractive characterization properties that are unattainable from diamond-like carbon (DLC) films, silicon or other materials. These properties include high hardness, high modulus of elasticity, very low surface roughness, low friction coefficient, high tensile strength, low thermal expansion coefficient, good wear resistance property and biocompatibility. Due to these properties, DLN films can highly applicable in MEMS/NEMS devices. There are two different ways of applications of DLN films in MEMS/NEMS: either a surface coating material or a structural material. In this paper, we suggest the use of DLN films as a coating material mainly to improve the wear and friction of micro components and reduce stiction between microstructure and their substrate. The high mechanical properties of this type of DLN films exploited the design of high frequency resonator and comb driver for sensing and actuating applications. As a biocompatible material, we can use DLN films for detection of bio-molecules in biological research and disease diagnosis.

Keywords

DLN films, Raman spectroscopy, HRTEM, AFM, MEMS, NEMS.

1. INTRODUCTION

Recently, science and technology pay much attention to use diamond-like nanocomposite (DLN) thin films in MEMS/NEMS devices due to its high hardness, high elastic modulus, high tensile strength, very low surface roughness, good wear properties, low friction coefficient, high thermal conductivity and biocompatibility [1-3]. Actually, MEMS technology is dominated by silicon based materials due to advantages for their suitable fabrication. But the silicon materials have some disadvantages like it has relatively lower values of Young's modulus and fracture toughness. Also, the silicon materials have large coefficient of friction (COF), high wear rate, high surface energy and small band gap energy compared to DLC/DLN films. The fact is that this lack of silicon properties can create problem during MEMS device fabrication and operation. Like, if we fabricate the rotor or linear MEMS related motor by silicon material, which contain sliding interfaces typically suffer from rapid failure caused either by high frictional wear or by stiction. It

produces the unacceptable high static friction which can be exacerbated by capillary adhesion in humid environments [4-5] or by low operation temperature and heat dissipation due to small energy band gap. Hence, silicon based materials are not ideal for MEMS device-fabrication. Presently, people have given much attention in continuous study on advanced materials like SiC, SU8, PMMA, or PDMS for their suitability in MEMS/NEMS. Except these materials, diamond, DLC and DLN thin films are believed to be strongest materials for MEMS application. They possess a number of excellent properties over silicon and other advanced materials stated above [6-7]. Also, among the diamond materials, we suggest the DLN films which are more promising materials for MEMS.

2. PREPARATION OF THE FILMS

DLN thin films are deposited on glass (pyrex) /silicon substrate by PECVD technique using siloxane or silazane based gas precursor with argon as a source gas. First, the substrate was cleaned by ultrasonic bath using acetone as well as warm trichloroethylene (TCE) for five minutes and was dried by using nitrogen gas. Afterwards, the substrate was cleaned by methanol in ultrasonic bath for ten minutes and dried by nitrogen gas. Then, the substrate was rinsed in de-ionized water for two to three minutes and was again dried by nitrogen gas. In this deposition, we have used DC as well as RF bias for deposition of the films. Due to hot filament, the gas precursors or their combinations are ionized; as a result, the DC discharge plasma was produced. Substrate temperature was kept around 250 °C by flowing cold water through the substrate holder assembly. To ensure the uniform thickness of the films, the arrangement of the vaporization chamber was maintained to provide a planetary motion to the substrate holder plate. The growth rate was maintained at 1 µm/hour.

3. CHARACTERIZATION OF THE FILMS

The structural properties of DLN films were analyzed by high resolution transmission electron microscopy (HRTEM), Raman spectroscopy and the tribological property were analyzed by atomic force microscopy (AFM), nanoindentation test and scratch test. These properties are most important parameters for application of any material in MEMS device fabrication. All of the characterizations we have done using pyrex glass / silicon substrate. Fig.1 shows the HRTEM image of diamond-like nanocomposite (DLN) thin films. From this HRTEM image,

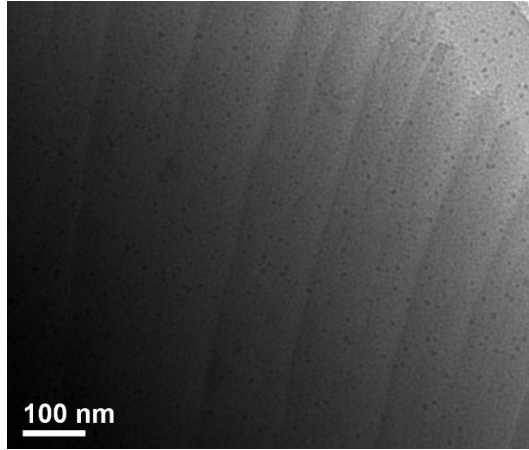


Fig.1: HRTEM image for siloxane precursor based DLN films.

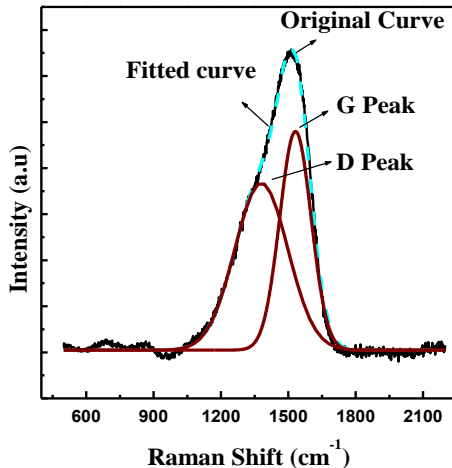


Fig. 2: Gaussian deconvoluted Raman spectrum of the D and G peaks for DLN films based on siloxane based gas precursor.

(for siloxane precursor based DLN) we have confirmed the nucleation and growth of SiO_x nanoparticles of sizes 2-15 nm in the amorphous matrix. Actually, DLN films are mostly consisting of two interpenetrating networks of a-Si:O and a-C:H.

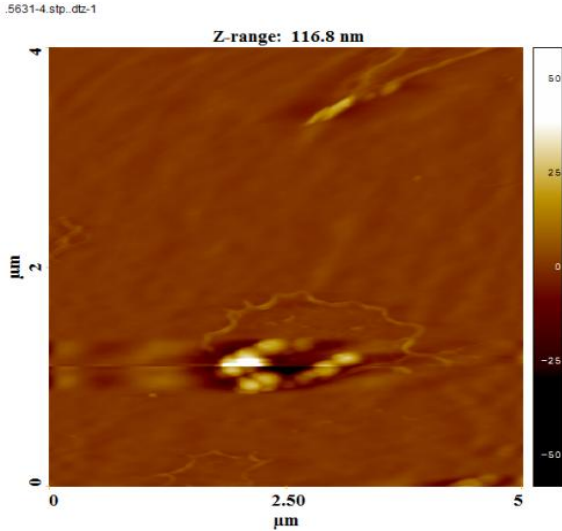
The first order Raman spectra of DLN films is shown in Figure 2. Which is usually deconvoluted into Gaussian D and G bands (here D stands for disorder graphite band and G stands for single crystal graphite band). Two broad peaks (D and G) are assigned in the Raman spectra of the DLN films, which are appeared in the wavenumber region $1000\text{-}1800\text{ cm}^{-1}$, are the typical characteristics of amorphous carbon films. The shape of the Raman spectra varies with substrate material composition. The positions and widths of D and G peaks can be correlated to the films properties (e.g. hardness, wear and electrical properties) like conventional DLC films [7]. The position of the D and G peaks are shifted due to variation of structure of DLN films using

different precursors. In diamond-like films, the intensity and position of D and G peaks are generally related with the ratio of $\text{sp}^3:\text{sp}^2$ which determines the mechanical and electronic properties of the films. The measured intensity ratios (I_D/I_G) for the DLN samples with silazane and siloxane based precursors are 1.84 and 1.66, respectively. As I_D/I_G ratio decreases, the sp^3 content into the films increases and hence, the hardness of the films increases.

The surface morphologies of DLN films are analyzed by using atomic force microscope (AFM). Figure 3 shows the AFM image of DLN films (for silazane based precursor) in two dimensional (2D) and three dimensional (3D) views. From AFM analysis, we have estimated the mean surface roughness (R_a) and maximum peak-to-valley height (R_{max}) of the DLN films, which are 3-8 nm and 6-20 nm, respectively. From this analysis, we can assume that all of the DLN films have very less surface defects (such as macroparticles and pinholes). Again, very less surface roughness influences the mechanical and tribological performances of the films for micro and nano scale devices. Hence, DLN films will provide the better performance for the applications in MEMS or NEMS devices compared to DLC films [8].

The method of measuring hardness and elastic modulus by nanoindentation technique is produced by Oliver and Pharr [9]. This method has been widely adopted to characterize the mechanical behavior of materials in small scales, while numerous change and refinements have been made to further improvement of its accuracy. The hardness and elastic modulus of DLN thin films was measured by Nanoindentation technique. Figure 4 shows the loading and unloading curve of DLN sample which was given by load vs. displacement into the surface of DLN films. This curve shows the good reproducibility by nanoindentation test. The average hardness of the DLN films was measured using three indents with 20 mN load which is around 8.2-17 GPa. The average reduced elastic modulus of the DLN films is measured under the 20 mN loading force with 300 nm displacement into the surface and which is around 90-160 GPa. Whereas for SiO_2 thin films, the elastic modulus is around 69-74 GPa [10-11], for bulk silicon, the hardness and elastic modulus is 11.9 GPa and 62 GPa, respectively [12]. The hardness of bulk silicon dioxide is 6.9 GPa [13]. Hence, for DLN films, the hardness and elastic modulus is better compared to SiO_2 thin films and bulk silicon films.

The coefficient of friction (COF) of DLN films are very important parameter for MEMS application. The low COF can reduce the stiction between two MEMS microstructure. To measure the COF of the DLN films by scratch test method, the loading rate was 16 N for both normal force and tractional force and the scratch length was about 3 mm. We have estimated the COF of the samples by taking the ratio of normal force and lateral force. The measured average COF for DLN films is around 0.02-0.05. For bulk silicon and silicon nitride (Si_3N_4) films, the COFs are 0.1-0.6 and 0.32, respectively [14]. Figure 5



d:\all\dlm\data\m1\afm\0.5631\5631-1.stp.dtz-1

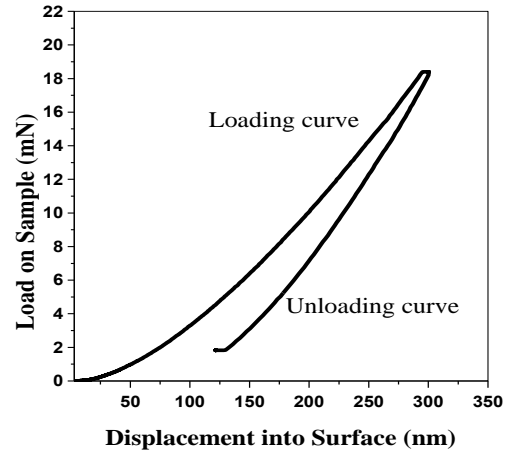


Fig. 4: Loading and unloading curves by nanoindentation test for DLN films using siloxane based precursor.

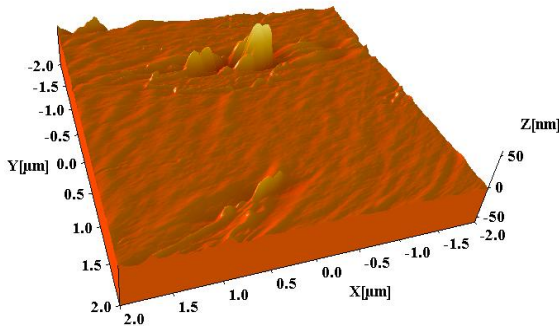


Fig. 3: Surface morphology of DLN films (for siloxane based precursor) deposited on glass substrate: 2D view (top) and 3D view (bottom).

shows the scratch behavior of DLN samples with normal and tractional forces, while Fig. 6 shows the variation of coefficient of friction (COF) with normal load using scratch test results.

4. DISCUSSION

For a typical MEMS device, the overall dimensions are ranging from tens of micrometer to a few millimeters and the separation distance of individual elements of this device (determine by the thickness of the sacrificial layer) is in the range of sub-micrometer to less than few micrometers. In this case, surface adhesion and coefficient of friction (COF) potentially dominate the performance of MEMS devices [15-16]. For microgear, microactuator, or any moving parts of MEMS devices generate lot of friction during the experience of surface contact operation. Silicon has very high COF (nearly 0.6), can hamper the MEMS devices during operation [15]. In the literature, it is reported that microengines and micromotors could only be operated only for few minutes before their

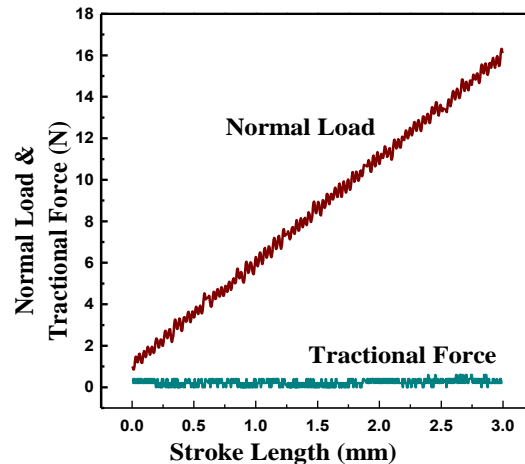


Fig. 5: Scratch behavior for DLN coating sample. Variation of normal load and tractional force with stroke length.

tribological collapse [17]. DLN films have very low COF and wear rate is also exceptionally small, which can act as potentially solid lubricant for MEMS devices.

5. CONCLUSIONS

Diamond-like nanocomposite (DLN) films possess a unique number of material properties which particularly make it attractive for application in MEMS devices. The structural properties of this films shows that films nucleation and growth of SiO_x based nanoparticle with particle sizes around 2-15 nm. The Raman spectroscopy shows the higher sp^3 content into the DLN films compared to conventional DLC films, which improve the mechanical properties of the DLN films. This films have very low surface roughness (3-8 nm), very high hardness (8.2-17 GPa), very high Young's modulus (90-160 GPa) and very low COF (0.02-0.05), compared to other diamond like materials which we have discussed earlier. Hence, these properties improve the friction and wear resistance of the operating

MEMS devices. The biocompatibility of DLN material exploited it as biosensors, micro-fluidic devices for lab-on-chip applications and implantable medical devices. The tribological properties of the DLN films strongly suggest that, we can use this DLN films as coating materials for MEMS devices.

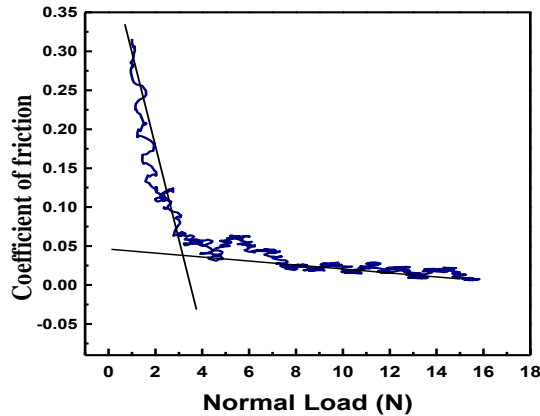


Fig. 6: Variation of coefficient of friction (COF) with the normal load.

6. ACKNOWLEDGMENTS

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7. REFERENCES

- [1] Yang, W. J., Choa, Y. H., Sekino, T., Shim, K. B., Nihara, K. and Auh, K. H. 2003. "Structural characteristics of diamond-like nanocomposite thin films grown by PECVD", *Mater. Lett.* 57, 3305–3310.
- [2] Neerincck, D., Persoone, P., Sercu, M., Goel, A., Venkatraman, C., Kester, D., Halter, C., Swab, P. and Bray, D. 1998. "Diamond-like nanocomposite coatings for low-wear and low-friction application in humid environments", *Thin Solid Films* 317, 402-404.
- [3] Das, T., Ghosh, D., Bhattacharyya, T. K. and Maiti, T. K. 2007. "Biocompatibility of diamond-like nanocomposite thin films", *J. Mater. Sci.: Mater. Med.* 18, 493-500.
- [4] Spengen, W. M., Puers, R. and Wolf, I. D. 2003. MEMS reliability from failure mechanism perspective 17, p. 563.
- [5] Grill, A. 1997. "Tribology of Diamond-like carbon and related materials: an updated review", *Surf. Coat. Technol.* 94-95, 507-513.
- [6] Marchon, B., Heiman, N., Khan, M. R., Lautie, A., Ager, W. J. and Veris, D. K. 1991. "Raman and resistivity investigations of carbon overcoat of thin film media: correlation and tribological properties" *J. Appl. Phys.* 69, 5748-5750.
- [7] Luo, K., Fu, Y. Q., Le, H. R., Williams, J. A., Spearing, S. M. and Milne, W. I. 2007. "Diamond-like carbon for MEMS" *J. Micro. Macro. Eng.* 17, S83-S90.
- [8] Santra, T. S., Liu, C. H., Bhattacharyya, T. K., Patel, P. and Barik, T. K. 2010. "Characterization of diamond-like nanocomposite (DLN) thin films grown by PECVD", *J. Appl. Phys.* 107, 124324(1-9).
- [9] IEEE Transactions on electron devices 1978. vol. ED25, no.10, p.1249.
- [10] IEEE MEMS Workshop, 1993. Florida, P. 25.
- [11] Bhusan, B. and Li, X. 1997. Micromechanical and tribological characterization of doped single-crystal silicon and polysilicon films for microelectromechanical systems devices, vol. 12, pp. 54-63.
- [12] CRC Materials Science and Engineering Hand-Book, 2002, p. 474.
- [13] IEEE MEMS Workshop 1991, Nara, Japan, p. 151.
- [14] Tanner, D. M., Miller, W. M., Peterson, K. A., Dugger, M. T., Eaton, W. P., Irwin, L. W., Senft, D. C., Smith, N. F., Tangyunyon, F. P. and Miller, S. L. 1999. "Frequency dependence of the lifetime of a surface micromachined microengine driving a load", *J. Microelectron. Reliab.* 39, 401-414
- [15] Kim, D., Cao, D., Bryant, M. D., Meng, W. J. and Ling, F. F. 2005. "Tribological study of microbearing for MEMS applications", *J. Tribol.* 127, 537-547.