

The Evaluation of The Elastic Property in Nano-scaled Thin Compressive Film on Patterned Substrates.

M.-W. Moon^{1,a}, T.-Y. KIM^{2,b}, K.-R. LEE^{2,c}, K. H. OH^{1,d}

¹ School of Materials Science and Engineering, Seoul National University
Seoul 151-742, Korea

² Future Technology Research Center, Korea Institute of Science and Technology
PO Box 131, Cheongryang, Seoul, Korea

^amwmooon@snu.ac.kr, ^bassun@kist.re.kr, ^ckrlee@kist.re.kr, and ^dkyuhwan@snu.ac.kr

Keywords: Patterned substrate, Euler buckle, Elastic modulus

Abstract The evaluation of elastic property for thin diamond-like carbon (DLC) films has been presented with buckle analysis of compressive stressed film on patterned substrate. When substrate has been patterned with adhesion release layer, the morphologies of buckle configuration on patterned layer has been controlled from straight sided (Euler) buckle to nonlinear telephone cord type buckle with respect to the pattern width. By using the simple equation for Euler buckle, the elastic modulus has been easily calculated, shown well consistent with the results by nano-indentation test.

Introduction

The compressive stresses in thin film would cause interface delamination or buckle over the substrate [1-6]. These failures mainly depend on the mechanical properties of films and interface toughness or adhesion [7-10]. Under high compressive stress condition, the buckle mode has been classified with three types; straight sided (Euler mode), circular and non-linear type of telephone cord buckle. The non-linear instability mode known as telephone cord buckle has been well reported in experiment and theoretical works[2, 6, 10], while it has not been solved with exact solution due to its difficult geometries as shown in Fig. 1 [6, 9]. Straight sided buckle mode would rarely develop in compressive stressed film [1], which, however, has been well analyzed in the form of exact solution. Based on the theoretical condition for the stable straight sided buckle, the confinement of buckle width has been suggested with Diamond-like carbon (DLC) film on patterned substrate in previous work (Fig. 1) [9]. On the region of small width region, the straight sided buckle could be observed, which has been applied to the simple Euler buckle solution.

The associated mechanics for the evaluation of elastic properties by the Euler buckle has been introduced in next section, followed by the experimental procedure for the simple patterning with the release layer and for the deposition of DLC film is briefly addressed. At last section the results by the buckle patterning have been provided and discussed.

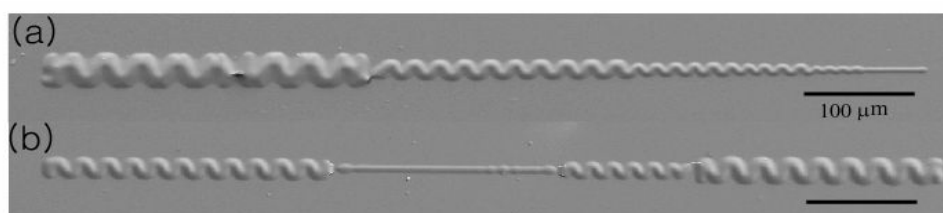


Fig. 1 buckle configurations on the patterned substrate. Straight sided buckle on the smaller width and non-linear telephone cord buckle on the larger width of patterned layer.

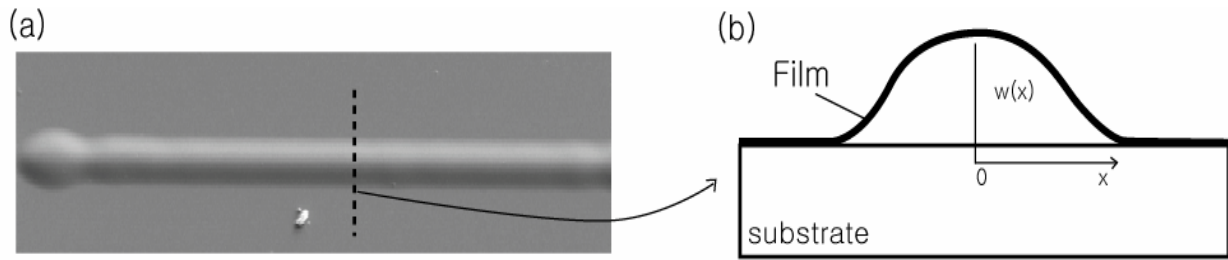


Fig. 2 Straight sided buckle on patterned substrate (a) and schematic of cross section profile

The associated mechanics on Euler buckle.

When the straight sided buckle is formed on small width region in the patterned layers, one can consider the symmetric profiles as shown in Fig. 2. Since the DLC film has the amorphous structure, this system has been taken to be elastic and isotropic materials, and stress state is equi-biaxial. The straight sided buckle on patterned substrate could be analyzed in exact solution with the amplitude and the width of buckled film [5, 9]. The transverse displacement on cross section area is defined as

$$w(x) = y = A(1 + \cos(\frac{\pi}{b}x)) \quad \text{at } |x| \leq b \quad (1)$$

where A and b are the amplitude and width of straight sided buckle under compressive stress in the plane strain condition. The classical membrane stress of a clamped-clamped wide plate is easily defined by [5]

$$\sigma_c = \frac{\pi^2 D}{b^2 h} = \frac{\pi^2 E}{12(1-\nu^2)} \left(\frac{h}{b}\right)^2 \quad (2)$$

Where D and h are bending stiffness and thickness of film, respectively. For evaluating $E/(1-\nu^2)$, membrane stress, σ_c , in eq. (2) should be replaced by the following relationship with residual stress, σ_0 ,

$$\frac{\sigma_0}{\sigma_c} = \frac{3}{4} \left(\frac{w_{\max}}{h}\right)^2 + 1 \quad (3)$$

where the stress ratio would be determined by the measuring the amplitude of buckle and thickness of DLC film [5]. When one inserts the classical membrane stress (eq. (2)) into the eq. (3), the plane strain modulus is easily induced in terms of the buckle width and amplitude and the film thickness as follows;

$$\frac{E}{1-\nu^2} = \frac{48\sigma_0}{\pi^2} \cdot \frac{(b/h)^2}{(3(w_{\max}/h)^2 + 4)}, \quad (4)$$

where the buckle width, b, and amplitude, w_{\max} , can be taken by measuring the straight sided buckle morphologies on patterned substrate. The residual compressive stress, σ_0 , could be directly measured with the curvature method as detailed [7]. As a result, the eq. (4) provides the fundamental value of elastic property of compressive stressed film without other external forces.

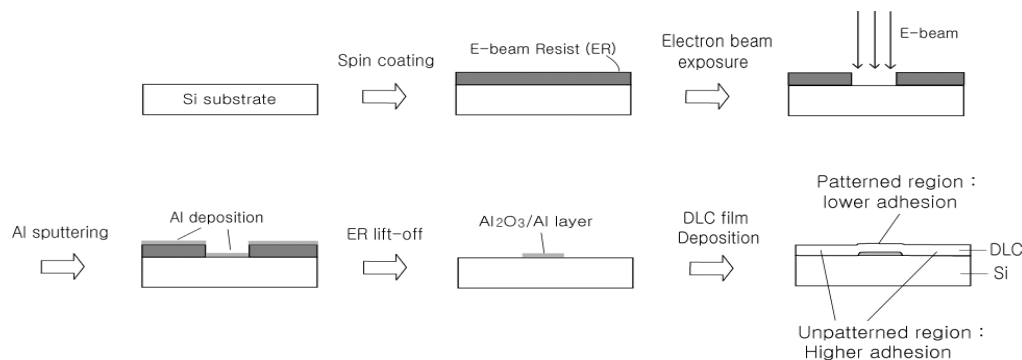


Fig. 3 Procedure for buckle patterning

Experiments : Patterning buckle delamination

In this chapter, a development procedure of adhesion release layer has been expressed and the deposition of the compressively stressed films has been also provided with detailed conditions. The first step for the photolithography is to draw the several patterned layers (Fig. 3) [9, 11]. For this purpose, E-beam lithography technique was adopted in the clean room environment. The positive ER layer was spin-coated on N-type Si (100), where film would be strongly bonded after ER removed, followed by UV exposure. The patterned layers were exposed down to the substrate level, followed by deposition of the release layer. For patterning the layer, very thin Al layer has been deposited on patterned region with the thickness of less than 3 nm with the sputtering method, and oxidized in air. The liftoff for the unpatterned region has been accomplished with PR stripper of acetone solution. The Al_2O_3 thin layer has been deposited on Si layer by sputtering, where the DLC layer has been then deposited. Since interface adhesion of DLC film- Al_2O_3 release layer is smaller than the interface toughness in DLC film-Si substrate, the delamination of film would be expected to propagate only on the Al_2O_3 thin layer [4, 5, 9].

Diamond-like carbon films have been deposited on the patterned substrates by using a *capacitively coupled r.f.* glow discharge, The chamber was evacuated to 10^{-5} Torr by a turbomolecular pump. For the DLC film deposition, a glow discharge of CH_4 at a pressure of 1.33Pa was supplied with negative self-bias voltage controlled between -500Vb by adjusting the r.f. power [7]. With respect to the deposition time, the resulting film thickness ranged from 120nm to 350nm measured by AFM (Atomic Force Microscopy) in contact mode, inducing the high compressive stress which caused buckles only on the release layers. As a reference, nano-indentation technique has been applied for each sample used for the measurement of elastic properties by buckle analysis. The detailed results for elastic property has been presented in Fig. 4.

Results and discussion

As shown in Fig. 1, the precise control of buckle configuration has been provided on the patterned substrate. Non-symmetric telephone cord buckle mode is available on release layer with large width, while straight sided buckle mode is available on the relative smaller width region. With measurement of the straight sided buckle geometries in patterned buckle and with eq. (4), the plane strain modulus have been evaluated for three different film thickness of DLC film in Fig. 4. At the relative thicker film (300nm), the results by nano-indentation and by buckle analysis is quite consistent, but for rather thin film (120nm), the difference between two methods is clear. Since the films in this work have been deposited at the same condition of negative bias condition affecting the mechanical properties of film [4], the plane strain modulus would be similar regardless of film thickness. Moreover

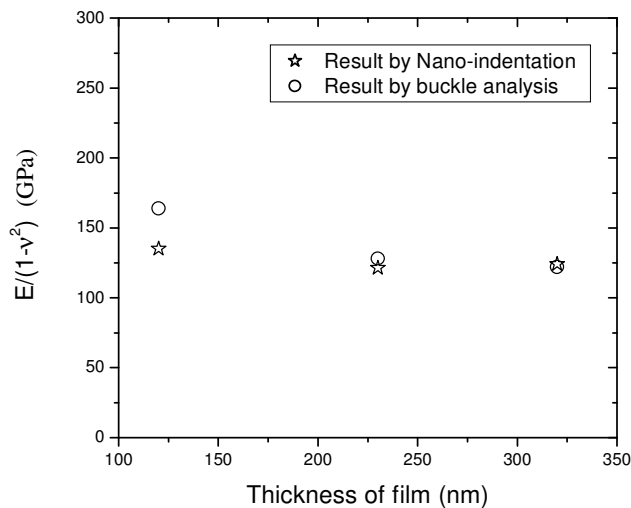


Fig. 4 Plane strain modulus estimated by Nano-indentation and by buckle analysis.

nano-indenting have considered the substrate effect, of which method may not be accurate at thin layer of film on substrate [10]. Therefore, the method by buckle analysis is rather accurate for measurement of mechanical properties of film.

Summary

Patterned buckle configuration has been used for the evaluation of elastic property for thin diamond-like carbon films. With confining of buckle width, the Euler buckle mode has been developed. By using the simple equation for Euler buckle, the plane strain modulus of DLC film has been easily calculated, shown well consistent with the results by nano-indentation test.

References

- [1] N. Matuda, S. Baba, and A. Kinbara, *Thin Solid Films*, **81** (1981) 301.
- [2] J. W. Hutchinson, M. D. Thouless, E. G. Liniger: *Acta metall. mater.*, **40** (1992) 295.
- [3] M. D. Thouless: *J. Am. Ceram. Soc.* **76**(11) (1993) 2936.
- [4] K.-R. Lee, Y.-J. Baik, and K.-Y. Eun: *Diamond and Related Mater.*, **2**(1993) 218.
- [5] J. W. Hutchinson, and Z. Suo: *Adv. Appl. Mech.*, **29** (1991) 63.
- [6] M.-W. Moon, H. M. Jensen, K. H. Oh, J. W. Hutchinson and A. G. Evans: *J. Mech. Phys. Solids*; **50**(11) (2002) 2355.
- [7] S.-J. Cho, K.-R. Lee, K.Y. Eun, J.H. Hahn, D.-H. Ko: *Thin Solid Films* **341** (1999) 207.
- [8] X. Jiang, K. Reichelt, and B. Stritzker: *J. Appl. Phys.* **68**(1990) 1018.
- [9] M-W Moon, K-R Lee, K. H. Oh, J.W. Hutchinson: *Acta mater.*, **52**(10) (2004) 3151
- [10] A. Lee, B. M. Clemens, W. D. Nix: *Acta mater.*, **52** (2004) 2081.
- [11] W. C. Johnson, S. M. Wise, J. Y. Huh, and J. Favergeon: *Mat. Mater. Int.* **9** (2003) 1