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ELASTIC MODULUS OF COATINGS BY PENDULUM IMPACT TEST

Stela CONSTANTINESCU
“Dunarea de Jos” University of Galati, Romania
e-mail: constantinescu_stela@yahoo.com

ABSTRACT

The pendulum impact scratch device with high precision system in measuring normal and tangential forces during scratching has been used to study elastic modulus of coatings. The criterion for evaluation of elastic modulus of coating and its experimental principle have been described and a series SiC and SiN thin films coated by PNCVD have been tested. The test results indicate that elastic modulus of SiC coatings has a good line relationship with Si content and there is a peak value in middle Si content. The experimental results also show that the newly developed device is useful in the evaluation of elastic modulus of coating especially under dynamic state and the method is characterised by reliability, validity and simplicity.

KEYWORDS: coatings, elastic modulus, pendulum impact test

1. Introduction

Being an active research topic in the field of materials sciences and technology surface coatings have been applied in many industries.

Their performances are related to mechanical properties such as cohesion strength and elastic modulus, etc. The stress in coatings is a main reason to make coating invalid, which may occur during the process of manufacture and machining or appears due to temperature change, phase transformations and magnetic field [1-2].

Elastic modulus (E) is an important factor for the determination of coating stress and it is usually measured by microbending and microtensile test. In bending tests the errors are due to inaccuracies in measuring geometry, deformation of the beam as well as simple model. There are many practical difficulties associated with achieving a straight portion at the beginning of the stress–strain curve in tensile test. Therefore it is urgent to develop effective and practical techniques for evaluating dynamic elastic modulus of coatings and for promoting the widespread use of coating technology.

In this paper, the single pendulum impact scratching device with high precise measuring system has been developed [3].

Using the device a novel tribological method for evaluating elastic modulus of coating has been established and the experimental results have been discussed.

2. Experimental procedure

2.1. Instrument and parameters measurement

The single pendulum impact scratch device is based on the combination of an impact tester and a scratch tester. This new developed instrument has a swinging pendulum (length L) with a sintered hard metal (YT15) stylus at the top of hammer (weight M). In order to control the incursion depth of grooves on the surface of specimen there is an adjustable specimen holder with a precision regulator for lifting or dropping samples, as in Fig. 1.

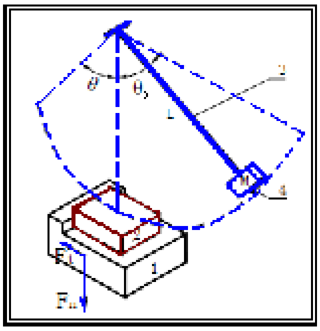

Fig. 1. *Diagram of single pendulum impact scratch device (1 - Adjustable specimen holder; 2 - Specimen; 3 - Pendulum; 4 - Stylus; Fn - Normal force; Ft - Tangential force; θ_0 - Initial angle; θ - Final angle)*

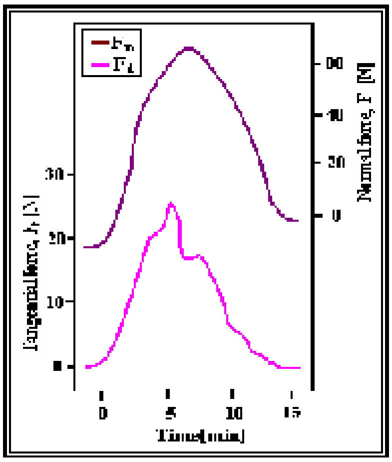

***Fig. 2.** Typical curves of Ft and Fn vs time during a scratching process*

The specimen holder is constructed by a rigid frame and strain gauges are mounted in the frame for measuring normal force (*n F*) and tangential forces (*t F*) simultaneously in scratching and the forces curves can be recorded continuously by a digital storage oscilloscope [4].

Typical force curves of a single-pass impact scratch are shown in Figure 2.

Table 1 shows the parameters and precision of the single pendulum impact scratch device.

During impact scratching test a series of grooves with different sizes were produced for each specimen by changing the height of holder. After the test the geometry of grooves such as length (*l*), depth (*h*), width (*b*) were determined by measurement microscope and a profilometer in order to calculate the maximal normal projective area of stylus (A_N) and the maximal cross-sectional area of groove (A_T).

***Table 1.** The parameters and precision of the single pendulum impact scratch device*

Parameters	Simbol	Unit	Variable range	Precision
Pendulum length	L	mm	362	0.5
Tangential force	F_t	N	2 ~ 100	0.8
Normal force	F_n	N	5 ~ 200	1.25
Speed of stylus	v	m/s	0.64 ~ 4.42	0.005
Specimen size	-	mm	30 x 50 x 1 ~ 4	-
Lifting (dropping) of Specimen holde	-	mm	0 ~ 5.00	0.002

2.2. Elastic modulus criterion

In typical and traditional situation, due to the un symmetry of depth of microindentation in loading and unloading processes, the elastic deformation of materials could be obtained by continuously recording the indenter displacement as a function of the applied load.

A similar treatment as those used in microindentation techniques is employed in the present study [5-6].

There is almost the same process of loading in incursion and unloading in sweepback during single pendulum impact scratching test (SPIST). As shown in Fig. 3, from the maximal normal force *F N* and the recovery behavior of groove in sweepback process at the deepest place, the mean slope of unloading curve K_e could be expressed as follows:

$$K_e = 2 / \pi^{1/2} \beta E_r$$

where: β is geometric shape factor, E_r is the synthetic elastic modulus of indenter and tested material, S_c is the contact area of indenter, *h* is the indentation depth [7].

Ke is a constant for certain material and it is obtained by fitting a series of F_N *vs*. (y - h) $S_c^{½}$ from different grooves.

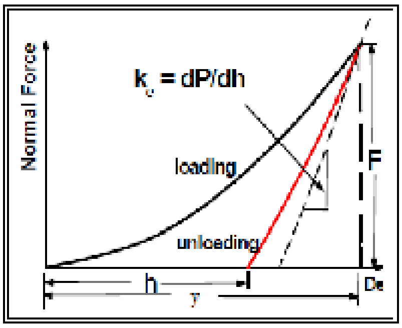

***Fig. 3.** Normal force vs depth of groove from loading and unloading in pendulum testing*

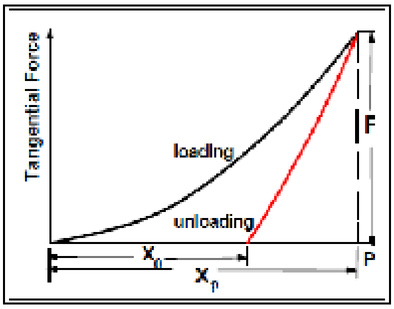

***Fig. 4.** Tangential force vs position of stylus from loading and unloading in pendulum testing*

According to the same principle, as shown in Fig. 4, meantime notice groove length $L = 2x_p - x_0$, i.e. $x_p - x_0 = L - x_0$, the shear process could also be described by the equation:

$$F_T = k_g (L - x_p) S_t^{1/2}$$

where: x_p is the groove length part under loading and x_0 is part under unloading, S_t is the contact area of stylus in tangential direction, k_g is also a constant for certain material and obtained the same as k_e by a series of grooves from the test. Fig. 5 and Fig. 6 show relations both in normal and tangential force vs normal and tangential displacement and square root of the corresponding area of cobalt samples. The slope k_e of F_N vs. $(y - h) S_c^{1/2}$ and the slope k_g of F_T vs. $(L - x_p) S_t^{1/2}$, can be obtained by fitting curves [8].

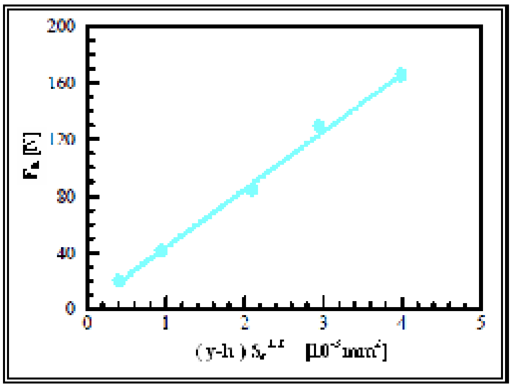

Fig. 5. *Normal force vs displacement by square root of contact area of a series of grooves on cobalt specimen*

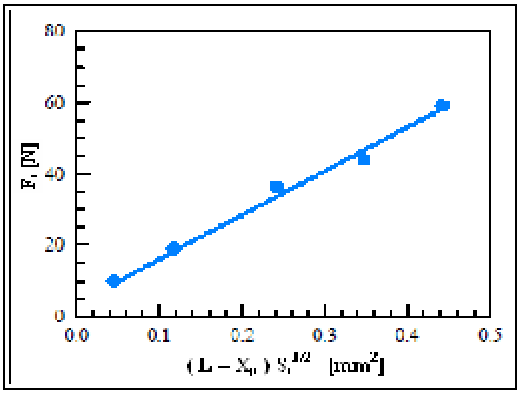

Fig. 6 *.Tangential force vs displacement by square root of contact area of a series of grooves on cobalt specimen*

The relation of *E* and *G* is:

$$E = 2 (1 + \upsilon) G$$

The *E* of any material or coating could be determined by the following expression:

$$E = 4G - 4G^2 [1/E_r - (1 - \upsilon_i^2)/E_i]$$

To verify growth mechanisms of SiN_x thin films, the bulge test specimens were cross sectioned and observed using scanning electron microscopy.

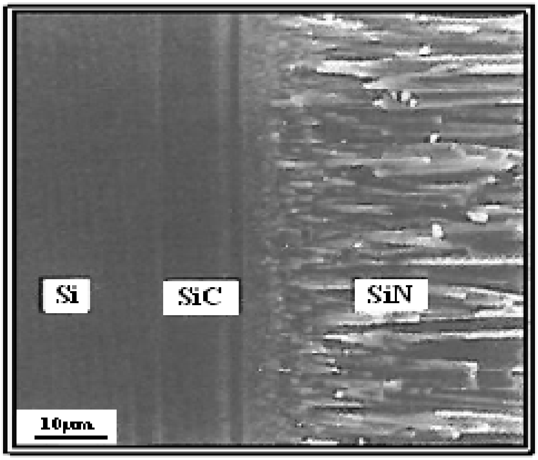

Fig. 7. *SEM micrographs showing the cross section of thin deposited on silicon wafer Si (100)*

Both coatings exhibit columnar morphology that arises from transverse fracture along the weak, low density intercolumnar regions. These are usually two distinct growth morphologies within the film thickness. The columnar structure is almost perpendicular to coating surface [9-10].

The SEM cross sectional views (Fig. 7) showed the microstructure throughout the film thickness is not uniform. This means that the biaxial measurements should be considered as mean values corresponding to modulus average of different layers.

3. Result and discussions

A series of SiN coatings with different content was obtained by chemical vapour deposit at normal pressure (PNCVD). Their elastic modulus was measured by SPIST and the test results were shown in Fig. 8. From Fig. 8, the elastic modulus of pure silicon coating was 140 GPa, which is lower than that of bulk (237 GPa) and E of SiN coating was 180 GPa. The scattered E of similar coatings was due to different composition, CVD process and method [11]. Comparing to the scattered result, coating’s E from SPIST is on the high side, which is due to the fact that in this test E is calculated by the maximum normal force and the maximum normal strain and this method decreases the effects of porosity and layer structure. However coating’s E from this test is more efficient than other test on the aspect of expressing dynamic behavior of coatings.

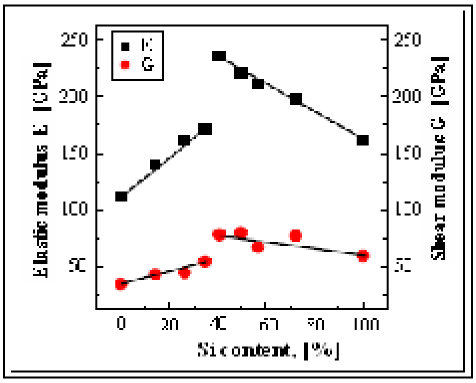

Fig. 8. *The relation between Si content, E and G*

Fig. 8 shows the relation between E or G and silicon content. For SiC and SiN coating, the curves are divided into two segments and the relationship can be described by a line’s regularity. In SiC coating E and G of coating increase with silicon content and in SiN coating E and G decrease with Si content, therefore E and G of coating appear to reach a peak value when SiC content is similar to SiN content. The relationship between E and content is important to design a stepped coating [12]. To realize the continue transition of mechanical property, the peak value of E and G should be decreased as low as possible.The elastic modulus of three kinds of Si(C,N) coatings on silicon substrate obtained by Chemical Vapour Deosition was also measured by SPIST. SiC coating and thickness of Si(C,N) coatings were shown in Table 2.

Table 2*. Si content and thickness of Si (C,N) coatings*

Coating	Low Si	Middle Si	High Si
Si content (wt %)	2.5	4	6
Thickness (µm)	10	10	10

Some of Si(C,N) coatings were heated for 4 hours at different temperature.

Fig. 9 showed the relationship between E of coatings and Si content, temperature of heat treatment.

Form Fig. 9 it can be seen that with SiN coating increasing E decreases, mainly due to the fact that the more Si content the more brittle the coating is. It can also be seen in Fig. 9 that there is a maximum point of E at heating temperature of 1090 °C for three coatings. As far as SiN coating is concerned, LSi coating has the largest E (240 GPa) after heat treatment at 1090 ℃ for 4 hours and HSi coating has the least E (137 GPa) without heat treatment, which corresponds to the result (140 GPa ～ 237 GPa). To obtain good mechanical property of coating, the appropriate content and mode of heat treatment must be selected. The advantage of this method comparing to traditional ways is that it can simulate service environment, especially under shearing action induced by dynamic loading [13].

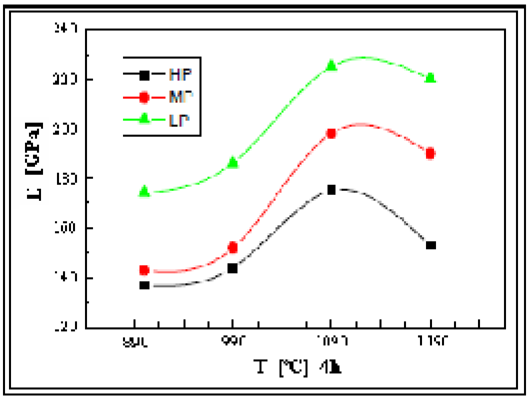

Fig. 9*. The relationship between E and the procedure of heat treatment*

4. Conclusions

The single pendulum impact scratch device with precise measurement of normal and tangential forces has been used to study the elastic modulus of coatings. The criterion for evaluating elastic modulus of coating is defined and its efficiency is identified by a series of SiC and SiN thin films coated by PNCVD. The measurement technique not only emphasizes the evaluation of elastic modulus of coating under dynamic state but it also is characterized by reliability, validity and simplicity.

The relationship between E of SiC coatings and their content can be divided into two segments, which present different line’s regularity respectively and a peak value appears in the middle of the content. The content of Si and the temperature of heat treatment have an important influence on E of SiN coating and LSi coating possesses the largest E after heat treatment at 1090^{o}C for 4 hours.

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