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Analyses of the Work Required to Delaminate a Coating Film from a Substrate under Oscillating Load Conditions

於振盪負載條件下分析鍍層從底材剝離所需之功

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Abstract

Nanoindentation tests in which oscillating loads were applied to specimens were carried out to study the work (W_f) required for a coating film to delaminate from its substrate. A sharp increase in the indentation depth occurred during coating film delamination. A theoretical model was developed for the present study to evaluate the difference between the work in the load-depth profiles obtained with and without delamination. Arranging that the maximum load was reached at the end of the loading process allowed us to obtain an approximately constant value for the indentation depth propagation rate during in one cycle. This allowed a determination of the number of oscillating cycles, and thus the work required, by assuming there was no delamination during this delamination period. The depth propagation rates and the work required for coating film delamination were investigated by varying P_{max}, P_{mean}, P_0, and frequency (f). The oscillating load-depth cycles appearing after delamination allowed us to establish the film-substrate contact behavior occurring during the loading/unloading process of one oscillating cycle.

Key Words: nanoindentation work for delamination, oscillating load

摘要

本研究之奈米壓痕測試利用不同振盪負載以探討鍍膜從底材剝離所需之功(W_f)。當鍍膜從底材剝離時壓痕深度會大幅增加。本研究發展一套理論模式以評量發生剝離與未剝離時負載-壓深曲線之差異性。最大負載為單一循環中施加負載過程終了壓深傳遞率趨於穩定之值。如此可決定剝離過程中振盪負載循環之次數與所需之功

。施加不同 P_{max}, P_{mean}, P_0, 與頻率(f)之值以探討壓深傳遞率與鍍膜剝離所需之功。負載-壓深之振盪循環顯示剝離後可瞭解單一循環負載/卸載過程薄膜-底材間之接觸行為。

關鍵字: 奈米壓痕之剝離功, 振盪負載

1. Introduction

With the development of the microelectronic and microelectro-mechanical system (MEMS) industries, film coating technology has played an increasingly important role. For most applications, delamination usually causes damage or failure in the system. Delamination may occur due to a residual stress or an applied load. Many testing methods have been proposed to evaluate film adhesion [1], including indentation tests. The depth-sensing indentation test is a powerful tool for determining of the mechanical properties of bulk specimens [2-6] or thin film/substrate systems [7-9]. Hardness, modulus, fracture toughness, and adhesion of the film/substrate interface have been evaluated using nanoindentation experiments [10]. During the indentation tests, an initial interfacial crack is created when the applied load reaches a critical value; the crack then slowly grows as the indentation load is increased. For a stable growth of indentation-induced delamination, the interfacial adhesion energy can be evaluated [11,12].

Dynamic nanoindentation tests, in which an oscillating load is applied to the specimens, were proposed as a convenient way to determine the mechanical properties under different deformations (depths) using one indenter [13]. They are widely used to precisely measure the composite properties of film/substrate specimens. Dynamic indentation tests can also induce fatigue failure, with behavior quite similar to that exhibited in traditional fatigue tests [14-17]. Interfacial fractures and damage due to fatigue failure have been observed during dynamic indentation tests [18-19]. Buckling may occur in the delaminated films, especially when significant residual stress is exhibited in the film. A delaminated film that experienced buckling can be used to determine the interfacial crack length (radius) by measuring the residual profile after tests. Compared to stable growth, the behavior of delamination due to fatigue failure is more complicated and thus evaluating the interfacial adhesion energy is more difficult.

In the present study, the energy in terms of the work (W_f) required for a coating film (SiO_2 film) to delaminate from a silicon substrate is applied to investigate the effects of several factors associated with the oscillating load condition on the work quantity. A new arrangement for the load condition is presented to replace the convential load arrangement widely used in fatigue tests [17-19]. This new load arrangement was designed to have the maximum indentation load in the first dwelling process be greater than the sum of the mean load (P_{mean}) set in front of the oscillating load cycles and the oscillating load amplitude (P_0). This ensures that the indentation depth propagation rate (dh/dN) is an approximately constant value. A constant dh/dN facilitates the calculation of the oscillating load-depth cycles that disappear from the time period delamination occurs. A theoretical model is developed in the present study to evaluate the W_f value from the difference in area between the load-depth profiles obtained from a test with delamination and a test assuming no delamination. The influential factors, including the maximum load (P_{max}), the mean load (P_{mean}), the oscillating load amplitude (P_0), and frequency (f), are varied to investigate their effects on the indentation depth propagation rate and the work required for delamination. Using the oscillating load-depth profiles that appear in the processes after coating film delamination, the contact behavior between the coating film and the substrate can be inferred according to the profiles for loading and unloading processes of the oscillating cycles.

2. A Theoretical Model to Evaluate the Work Needed for a Coating Film to Delaminate from the Substrate in Fatigue Tests

Film fractures in terms of the delamination and spalling of a coating film from its substrate material have been studied by Li and Bushan [10] using single-time nanoindentation tests. In the load-depth profile, there is a sharp increase in the indentation depth (depth shift), even with a very small change in the indentation load, if coating film delamination and spalling occur during the loading process. Under the same load-time condition, the work needed for coating film delamination in an indentation test is denoted by the difference in the area between the load-profile assuming delamination and the profile without delamination. The concept of determining the delamination resistance of a coating film in terms of work is applied to determine the work required for a coating film to delaminate from its substrate in an indentation test in which there is an oscillating load situation.

In order to investigate the delamination behaviors that occur during fatigue tests, an oscillating load with a load amplitude, P_0, and a load frequency, f, as shown in the bottom-right corner of Fig. 1, have been applied in several studies [17-19]. Here, the load set in the first dwelling process (process B) is equal to P_{mean}. The value of P_{mean} is smaller than the maximum load (P_{max}) shown in the oscillating load region (process C). This arrangement has the disadvantage of the envelopes of the oscillating indentation depths shown in the oscillating load region being a nonlinear function of time. As the experimental results show, the indentation depth propagation rate (dh/dN h: indentation depth, N: the number of oscillation cycles) formed in one cycle is no longer a constant value. A variable dh/dN value makes it difficult to determine the number of oscillate cycles during fatigue delamination.

2.1 Determination of the Oscillation Cycles during Delamination

The oscillating depth-time profile linear envelopes in the oscillating load region can be determined by modifying the load conditions before the oscillating load region. The load condition shown in Fig. 2 is arranged in order to obtain envelopes that are approximately linear. This load condition has the maximum load, P_{max}, set in process B, which is higher than the sum of the oscillating load amplitude, P_0, and the mean load, P_{max}, set in process D. The linear envelopes allow dh/dN being a constant value.

Fig. 3(a) shows a schematic diagram of the load-depth profile in response to the oscillating load-time condition shown in Fig. 2. This plot shows a sharp increase in the indentation depth in the $\overline{CD}$ process even after a small change in the indentation load. In this process, the coating film delaminated from the substrate. Beneath the $\overline{CD}$ section, there is a blank area without any load-depth cycles. This blank area should be filled with a number of load-depth cycles if the specimen is assumed to be operating without coating film delamination. A schematic diagram corresponding to the assumption of no delamination is shown in Fig. 3(b). It should be mentioned that the $\overline{CD}$ line in Fig. 3(a) is equal to the $\overline{HI}$ line shown in Fig. 3(b). The number of load-depth cycles beneath the $\overline{HI}$ line, N_i, can be obtained only if the dh/dN value before delamination occurs is available.

In order to determine N_i beneath the $\overline{HI}$ line, the oscillating depth-time cycles before the coating film delaminates are evaluated first from experiments. The dh/dN value can be determined if the oscillating cycles are determined using the depth expression, $h(t)= h_{mean} + h_0 \sin[f(t-t_0) + \theta] + (dh/dN)(t-t_0)$, where h_{mean} denotes the indentation depth corresponding to the P_{mean} load; h_0 denotes the indentation depth amplitude in the oscillating load region; f denotes the oscillation frequency; t_0 is the start time of the oscillating process; and θ represents the lagging phase angle due to the viscous effect intrinsic in the coating film. This expression is valid for oscillating depth-time profile envelopes before delamination that are a linear function of time (or dh/dN= (dh/dt)/f is a constant value).

If dh/dN is available, N_i is determined as:

$$N_i = \frac{h_I - h_H}{(dh/dN)} \quad , \qquad (1)$$

where h_I and h_H denote the indentation depths created at point I and point H, respectively.

2.2 Work Required for Fatigue Delamination of a Coating Film

The work required for a coating film to delaminate from a substrate material in a fatigue test is equal to the difference between the areas beneath the O-A-B-H-I curve in Fig. 3(b) and the O-A-B-C-D curve in Fig. 3(a). Since the profiles are obtained under the same load-time conditions, the difference between the areas shown in these two figures is equal to the difference between the area framed by the cycles in the $\overline{HI}$ section (see Fig. 3(b)) and the blank area □CDHG (see Fig. 3(a)). The blank area is obtained as:

$$(W)_{delamination} = 2P_0 \times (h_{D'} - h_{C'}) - Area(C'-D'-D-C), \qquad (2)$$

where $(W)_{delamination}$ denotes the work required for the coating film to delaminate from the substrate material, and it is evaluated in this blank area only. The work required to produce the oscillating cycles in the $\overline{HI}$ curve can be determined only when the work in one cycle is available. Fig. 4 shows a schematic diagram of the load-depth profile created in one cycle; point 1 denotes the starting point of a cycle, which has a load of P_{max}; point 2 shows where the load is equal to (P_{mean}+P_0); point 3 has a load equal to($P_{mean}-P_0$), and point 4 is in the same position as point 1. The shaded area in Fig. 4 can be determined from the experimental data for one load-depth cycle in the oscillating subregion before coating film delamination occurs. This shaded area is then obtained as:

$$W_N = \int_{h_1}^{h_2} P(t)dh - \int_{h_3}^{h_2} P(t)dh + \int_{h_3}^{h_4} P(t)dh \quad (3)$$

In the present study, the oscillating load in the sinusoidal form is given as the input condition:

$$P(t)=P_{mean} + P_0 \sin(f \cdot t) \quad (4a)$$

where f denotes the oscillating load frequency. The output of the indentation depth as a function of time is expressed as:

$$h(t)=h_{mean} + h_0 \sin(f \cdot t - \theta) \quad (4b)$$

The differential form of Eq. (4b) can be written as :

$$dh(t)=h_0 \cos(f \cdot t - \theta) f dt \quad (4c)$$

The lagging phase angle θ can be determined from the P(t) and h(t) profiles shown as a function of time. If Eq. (4c) is substituted into Eq. (3), then W_N can be rewritten as:

$$W_N = \int_{t_1}^{t_2} P(t) h_0 \cos(f \cdot t - \theta) f dt - \int_{t_3}^{t_2} P(t) h_0 \cos(f \cdot t - \theta) f dt + \int_{t_3}^{t_4} P(t) h_0 \cos(f \cdot t - \theta) f dt \quad (5)$$

The value of W_N can be obtained by substituting the oscillating load condition into the above equation. Then, the work required for the oscillating cycles in the $\overline{HI}$ section (see Fig. 3(b)) is obtained as:

$$W_{no\ delamination} = N_i \times W_N \quad . \quad (6)$$

The work required for fatigue delamination of a coating film from the substrate material (W_f) can be expressed as:

$$W_f = W_{no\ delamination} - W_{delamination}. \quad (7)$$

3. Experimental Details

The effects of the influencing factors shown in the load condition on the fatigue delamination of a coating film are the main subjects of the present study. Therefore, only one kind of specimen was prepared. These specimens were prepared by coating a 545 nm thick SiO_2 film on a silicon wafer substrate using the sputtering method.

The fatigue delamination tests were carried out on a nanoindentation tester (Hysitron, USA). The load condition arranged for the tests is shown in Fig. 2. In general, six parameters for the load condition can influence the fatigue behavior of the coating film: (1) the loading/unloading rate shown in the loading/unloading process (A/C process), (2) the maximum load, P_{max}, set in the first dwelling process (the B process), (3) the mean load, P_{max}, set in the second dwelling process (the D process), (4) the oscillating load amplitude, P_0, (5) the oscillating load frequency, f, set in the oscillating load region (the E process), and (6) the time period, set in the dwelling processes (the B and D processes). In the present study, the five most important influencing factors were chosen as the variables. The time period set in all of the dwelling processes was 0.2 seconds. This time period was chosen for the present study to ensure that the dwelling depth shown in the second dwelling process (the D process) was at almost a constant value. This means that the placement of the first and second dwelling processes (the B and D processes) in front of the oscillating load region could isolate the fatigue delamination in the oscillating load region from the lagging behavior after the loading/unloading process. Table 1 shows the arrangements of the operating conditions for the following four variable parameters: P_{max}, P_{mean}, P_0, and f. Due to the restriction on the oscillation number in the nanoindentation tester; the maximum number of oscillation cycles was 100. The delamination of a coating film could be achieved within this maximum number of cycles if the four influencing factors were properly set.

The semi-angle of the cube corner indenter (α=35.26°) is obviously sharper than that of the Berkovich indenter (α=65.3°). This sharpness has the advantage of having a significant increase in the depth propagation rate (dh/dN) per cycle. That is, the use of the cube corner indenter helps to produce fatigue delamination of the coating film within 100 oscillation cycles.

4. Results and Discussion

Various combinations of the four influential parameters shown in the oscillating load condition have been adopted as the operating conditions of fatigue tests. However, only some of these operating conditions were used to produce the fatigue delamination of the coating film. The operating conditions under which delamination will occur within 100 oscillation cycles required that the maximum indentation depth developing in the oscillating load region must be greater than at least 60% of the coating film thickness. The ratio of the maximum indentation depth to the coating film thickness is defined as a percentage of the film thickness (%).

Increasing the mean load, P_{mean}, but fixing the other three parameters, increased the percentage of the film thickness. Increasing the load amplitude P_0, but fixing the other three parameters, did not increase the percentage of the coating film thickness; conversely, it led to a slight decrease in the percentage. Nevertheless, the influence of the load amplitude on the percentage of the film thickness was limited. Variations of the oscillating load frequency also showed a rather small effect on the percentage of the film thickness. The maximum load, P_{max}, set in the first dwelling process (B), was the most influential factor on the percentage of the coating film thickness; increasing the P_{max} value significantly increased the percentage of the film thickness.

Fig. 5(a) shows the depth-time profile with fatigue delamination of the coating film. In this figure, there are a few oscillating depth-time cycles with relatively small depth amplitude before fatigue delamination occurs. The sharp increase in the indentation depth due to coating film delamination lasted a very short time. After coating film delamination, the oscillating cycles were restored, but they had relatively larger oscillating depth amplitude. Fig. 5(b) shows the load-depth profile obtained under the same operating conditions as those in Fig. 5(a). The arrows show the sequence of the entire process, including the processes before and after coating film delamination.

Several oscillating cycles had a small depth before coating film delamination occurred. They were followed by a sharp increase in the indentation depth even though only a very small change in the load occurred in the fatigue delamination process. After this process, different types of fatigue fractures may form.

The fracture model can be identified based on the forms of the oscillating load-depth profiles shown in the oscillating cycles after the delamination process. The load-depth profile shown in Fig. 5(b) is a typical model of several possible fractures developing in the coating film. The fracture mechanisms, in terms of the coating film delaminating from the substrate, are identified in a later section.

The work required for the coating film from the substrate (W_f) can be determined using Eq.(7) and the load-depth profile. In the present study, every measurement corresponding to the given operating conditions was obtained as the mean value of at least five readings. An error bar is also marked for each of these mean values.

According to the expression shown in Eq.(7), the W_f value is determined as a function of dh/dN. Fig. 6 shows the experimental data obtained by changing the P_{max}, P_{mean}, P_0, and f conditions separately, for cases in which coating film delamination occurred. The W_f curve shows significant declines as (dh/dN) increases. It becomes asymptotic to a constant value as dh/dN increases to a value higher than about 3 nm/cycle. Therefore, for a coating film with a fixed thickness, the work required for the coating film to delaminate from the substrate material is independent of the (dh/dN) value only when the (dh/dN) values are sufficiently high. Fig. 7(a) to Fig. 7(d) show the indentation depth propagation rates per oscillating cycle; they are obtained by varying the maximum indentation load, P_{max}, the mean load, P_{mean}, the oscillating load amplitude, P_0, and the oscillating load frequency, f, respectively. An increase in the maximum load (P_{max}) increases the depth propagation rate slightly (see Fig. 7(a)). The influence of increasing the mean load P_{mean} on the depth propagation rate is not monotonic (see Fig. 7(b)). The dh/dN values corresponding to the four P_{mean} values vary randomly in a small region. Fig. 7(c) shows the effect of changing the oscillating load amplitude (P_0) on the depth propagation rate. The dh/dN values created by changing the oscillating load amplitude also vary within a narrow region. That is, the oscillating load amplitude is not a significant factor affecting the depth propagation rate. The results shown in Fig. 7(a) to Fig. 7(c) imply that the depth propagation rate depends on which of P_{max} and (P_{mean}+P_0) is the larger value.

Fig. 7(d) shows the dh/dN values obtained by varying the oscillating load frequency. The results in this figure show that the (dh/dN) value is generally lower when the oscillating load frequency increases. Since dh/dN can be rewritten as (dh/dN)/f, a significant increase in the oscillating load frequency does not significantly increase dh/dt. This explains why dh/dN decreases when the load frequency increases.

Fig. 8(a) shows the W_f values obtained by varying the maximum load, P_{max}, while fixing the other factors. As shown in the figure, the work required to have the coating film delaminating from the substrate is lessened by increasing the maximum load. Fig. 8(a) shows the W_f values evaluated by fixing all operating conditions except for the mean load, P_{mean}. The P_{mean} values in the present study ranged from 8500 μN to 10000μN. It should be mentioned that only the cases in which delamination occurred are exhibited. The P_{mean} values vary within a narrow range because of the restriction imposed on P_{max} that it must be greater than the sum of P_{mean} and P_0. Since the readings for every set of operating conditions are scattered over a broad range, the error bar with respect to the mean value is quite long. The W_f values for these four P_{mean} values did not show significant deviations from the average of these four W_f values. That is, the W_f value was little affected by the changes in the P_{mean} value if P_{max} = 18000 μN and P_0 = 7900 μN were used. Fig. 8(c) shows the W_f values obtained by fixing all operating conditions except for the oscillating load amplitude, P_0. The mean values show that the W_f value is usually increased by increasing the oscillating load amplitude (P_0). The experimental results of W_f obtained by fixing all operating conditions except for the oscillating load frequency (f) are shown in Fig. 8(d). The W_f curve shows an exponential rise as the load frequency is increased. This is because an increase in the load frequency decreases the dh/dN(=(dh/fdt)) value substantially.

The load-depth profile, shown in Fig. 5(b), is provided to track the deformation behavior occurring at the time periods during and after the delamination of the coating film. Process A in this figure shows a sharp increase in the indentation depth during the delamination process. In this process, the strong bonds between the coating film and the substrate were broken off after several oscillation cycles. Subsequently, the coating film and the substrate demonstrated different contact behaviors due to the differences in the mechanical properties of these two materials and the indentation load.

In Fig. 5(b), the coating film is believed to be unbroken because the oscillation cycles after the delamination process show significant separation in the load-depth profiles between the loading process (denoted by D-C’-B curve) and the unloading process (denoted by B-C-D curve). If a crevice had formed between the coating film and the substrate in the bottom area, the coating film would have lost a solid base of support from the substrate material. The coating film showed pop-in behavior when the indentation load was elevated further. The coating film is believed to have slipped downward when the indentation load was sufficiently large. At the end of process A, the oscillation cycles occurred after the delamination process (process A) and thus completed a loading process of an oscillating cycle with a high stiffness value (the stiffness is defined to be the load required to create one unit of indentation depth change). This high stiffness has a tight contact at the interface of the coating film and the substrate material. After finishing this residual loading process, the test went into the unloading process denoted by the bottom curve, B-C-D, with two different stiffnesses along it. As shown in the figure, the unloading process of an oscillating cycle consists of the BC curve with high stiffness and the CD curve with low stiffness. Point C can be roughly regarded as the border of two subregions with high stiffness and low stiffness, respectively. At the BC curve, the profile shows a high slope value, which has a high stiffness value during the deformation process. This high stiffness condition can be created only when the coating film has tight contact with the substrate. At the CD curve, a small reduction in the indentation load resulted in a great reduction in the indentation depth. This behavior was demonstrated only when the coating film was separated from the substrate as a result of a crevice which appeared in the bottom area. From the above discussion, point C in the unloading process can be identified as the starting point of a crevice closing during elastic recovery. The D-C’-B curve shown at the upper portion of one oscillating cycle denotes the loading process. Similarly, the loading profile consists of two sections with a small slope in the DC’ curve and a large slope in the C’B curve. A low stiffness in the DC’ curve was also caused by a crevice in the bottom area of the interface formed between the coating film and the substrate material. This crevice disappeared when the load was increased up to point C’. A further increase in the indentation load, as shown in curve C’B, brought the coating film and the substrate material into tight contact again, as evidenced by the high stiffness shown in this section. Therefore, the coating film was held at its two shoulders and suspended in the air when indentation occurred in the CD curve in the unloading process and in the DC’ curve in the loading process. The coating film moved up and down repeatedly during these two portions of the oscillation cycle. It is interesting that point C’ in the upper curve (D-C’-B curve) and point C in the bottom curve (B-C-D curve) occur at the same depth. However, the indentation depths corresponding to these two points are quite different. This proves that the coating film after delamination did not break off even under an oscillating indentation load.

The schematic diagrams shown in Fig. 9 describe the contact mechanisms corresponding to the points marked on the load-depth curve. The solid arrows (B→C→D) indicate the evolutional procedures of the contact behavior demonstrated in the unloading process (the B-C-D curve); whereas the dashed arrows (D→C’→B) indicate the procedures of the contact behavior exhibited in the loading process (the D-C’-B curve). The sequences shown by the solid arrows and the dashed arrows constitute the increase in the deformation mechanisms of the coating film and the substrate material operating in one oscillation cycle after coating film delamination.

Fig. 10 shows the load-depth profile arising at a fracture mechanism after fatigue delamination, which is distinct from that shown in Fig. 5(b). The oscillating load-depth cycles occurring after the fatigue delamination process show two features. The unloading curve is very similar to the loading curve in one oscillating load-depth cycle, and the stiffnesses for the loading and unloading processes of one oscillation cycle are rather high. These two features occurred only when the coating film had broken off at its two shoulders and when the coating film and the substrate material were in tight contact during the loading and unloading processes of the oscillation cycle.

5. Conclusions

The indentation depth propagation rate is approximately a constant value when the maximum load in the present load condition is higher than the sum of the mean load (P_{mean}) and the oscillating load amplitude (P_0). A constant value of the depth propagation rate makes it easy to calculate the number of oscillating load-depth cycles arising at a section with coating film delamination if no delamination is assumed in the entire indentation process.

The indentation depth propagation rate is significantly affected by the oscillating load frequency; it is exponentially lowered by increasing the oscillating load frequency. Conversely, the depth propagation rate is increased by increasing the maximum load (P_{max}). The oscillating load amplitude (P_0) and the mean load (P_{mean}) have little effect if the sum of P_0 and P_{mean} is smaller than P_{max}.

The work required for the coating film (SiO_2) to delaminate from the silicon substate (W_f) is exponentially lowered when the depth propagation rate is increased. The work quantity is asymptotic to a small constant value when the depth propagation rate is sufficiently large. The W_f value is significantly increased when the oscillating load frequency is increased, but is lowered slightly when the maximum load (P_{max}) is increased. The influence of the oscillating load amplitude and the mean load (P_{mean}) on the W_f value is not as significant as those of the oscillating load frequency and the maximum load.

The profiles for the oscillating load-depth cycles after the coating-film delamination process can establish the film-substrate contact behavior occurring in the loading/unloading processes of the oscillation cycles. If a significant difference exists between the curves for the loading and unloading processes, the coating film was unbroken as it moved up and down during the oscillating cycles. If the difference is small, the coating film has broken off.

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Table 1 Arrangements of oscillating load conditions for the fatigue tests regarding the SiO_2 film delamination from the silicon substrate. Only some of tests resulted in film delamination.

P_{max} (μN)	6000, 9000, 10000, 18000, 18500, 19000, 20000, 25000, 28000, 30000
P_{mean} (μN)	3000, 5000, 8000, 8500, 9000, 10000, 12500, 14000, 18000, 20000
P_0 (μN)	1500, 3000, 6000, 8000, 8500, 9000, 9500, 10000, 13500
f (Hz)	1, 5, 10, 20, 30, 40, 50
N (cycles)	100
Time period in all dwelling processes (s)	0.2
Time required in the loading / unloading processes (s)	0.5

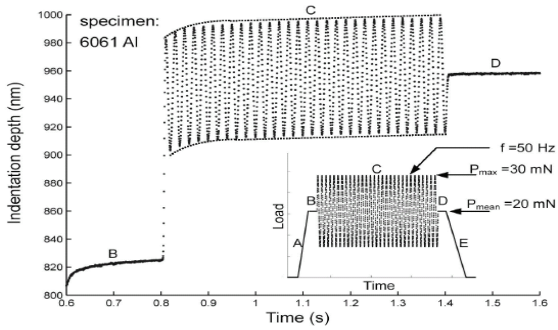

Fig.1 Variations of the indentation depth with time corresponding to the load conditions shown in the bottom-right corner.

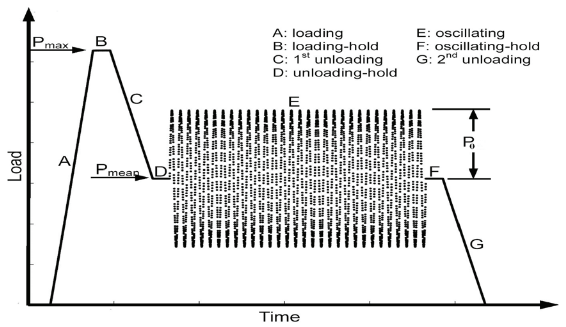

Fig. 2 The new arrangement in the load-time conditions for the present study.

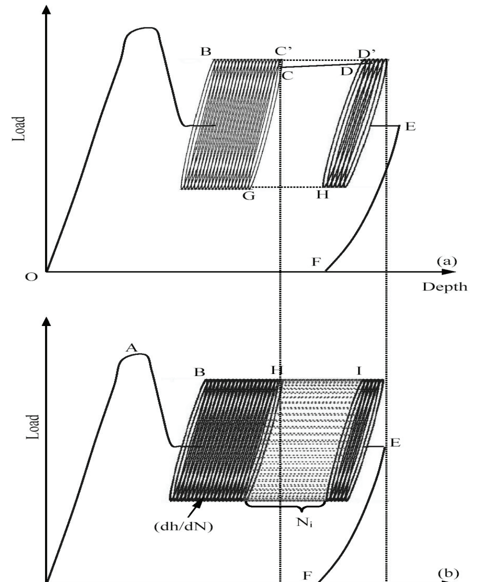

Fig.3 The load-depth profiles in response to the load-time condition shown in Fig. 2. They are obtained for (a) the coating film delaminating from the substrate; (b) the assumption of no coating film delamination.

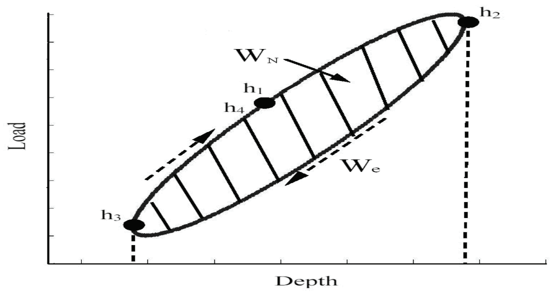

Fig.4 The work required for one oscillating load-depth cycle occurring in the delamination process is denoted by the shaded area surrounded by a closed contour.

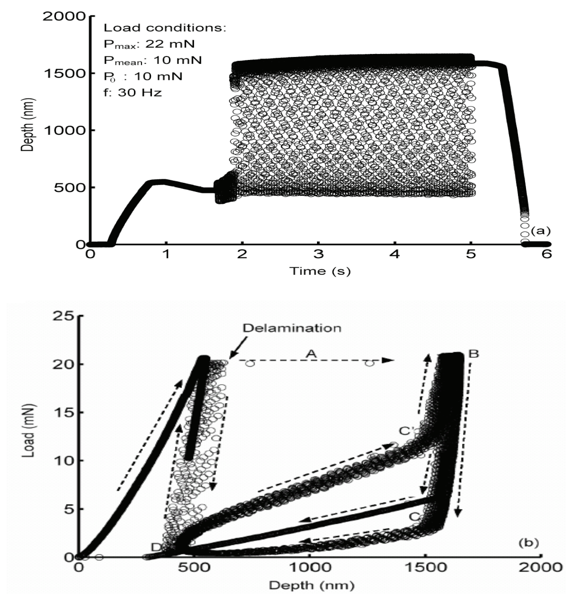

Fig.5 (a) A typical depth-time profile for a coating film delaminating from the substrate material; (b) the load-depth profile including the coating film delamination process and the behavior demonstrated in the oscillating cycles after this delamination.

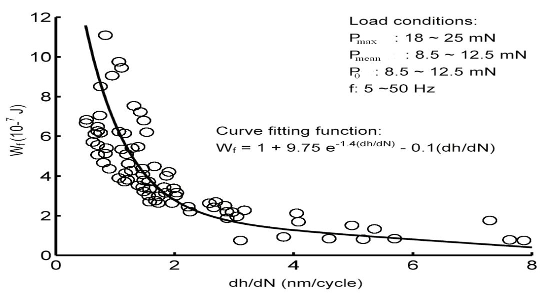

Fig.6 Variations of the work W_f with the

indentation depth propagation rate for the load conditions applied in the present study.

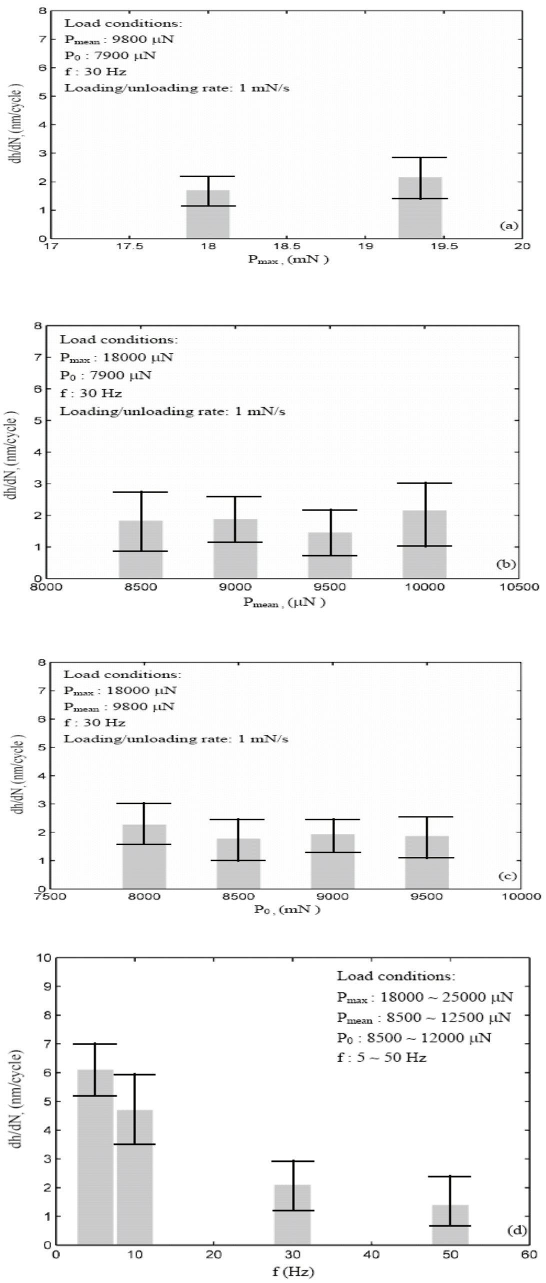

Fig.7 The experimental results of the indentation depth propagation rates obtained by varying (a) the maximum load, P_{max}; (b) the mean load, P_{mean}; (c) the oscillating load amplitude, P_0; and (d) the load frequency, f .

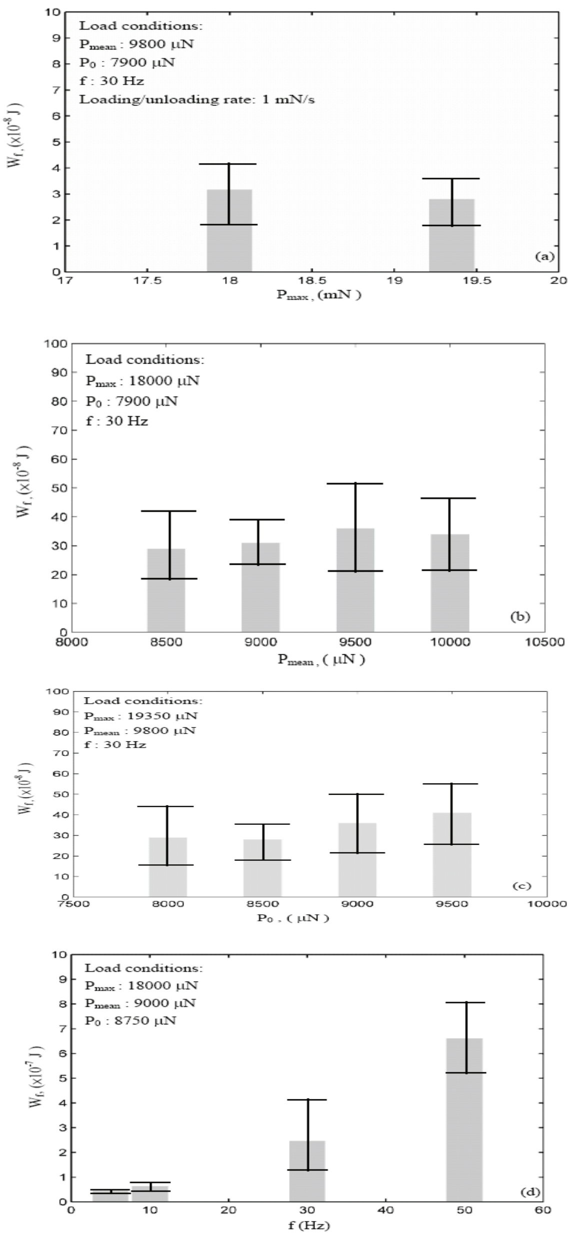

Fig.8 The experimental results of W_f obtained by varying (a) the maximum load, P_{max}; (b) the mean load, P_{mean}; (c) the oscillating load amplitude, P_0; and (d) the load frequency, f.

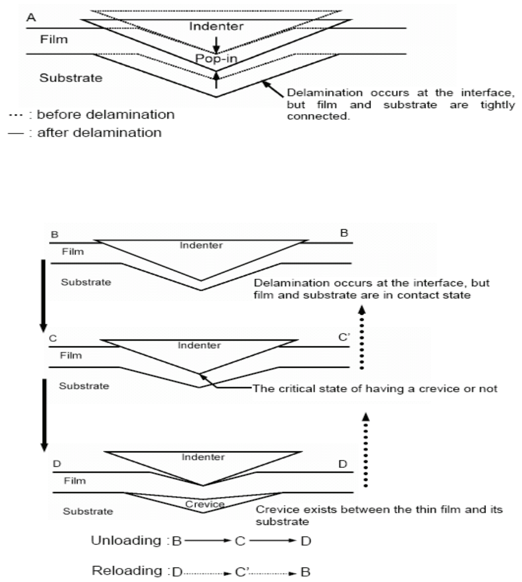

Fig.9 The contact mechanisms of the coating film and its substrate developing during the delamination process and during the loading/unloading processes of the oscillating cycles after coating film delamination.

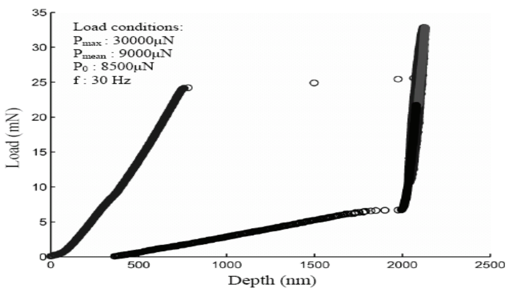

Fig.10 The load-depth profile obtained from a fatigue test in which the coating film broke off after delamination.