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A method for predicting critical load evaluating adhesion of coatings in scratch testing*

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Abstract: In this paper based on the experiment principle of evaluating adhesion property by scratch testing, the peeling mechanism of thin films is discussed by applying contact theory and surface physics theory. A mathematical model predicting the critical load is proposed for calculating critical load as determined by scratch testing. The factors for correctly evaluating adhesion of coatings according to the experimental data are discussed.

Key words: Coating, Adhesion, Scratch testing, Critical load prediction
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INTRODUCTION

Hard ceramic films deposited by chemical vapor deposition (CVD) and physical vapor deposition (PVD) techniques had been widely applied in industry. Coating adhesion is a very important property for evaluating the coating quality. The method evaluating the adhesion strength of the coating by the scratch test has been widely accepted in industry (Burnett and Rickerby, 1987; Jalli *et al.*, 1987; Shiozawa *et al.*, 1994).

Scratch adhesion testing is simple to perform. A loaded diamond stylus (usually a Rockwell C profile) is drawn across the coated surface at ever increasing loads until the coating is stripped from the substrate at some critical load L_c. The coating adhesion is thus evaluated by the critical load L_c.

Essentially, the scratch test involves deforming the coating-substrate interface by straining the substrate. The mechanical resistance of the interface or the coating is characterized by a critical load which is the minimum load at which damage can be observed by lack of adhesion.

The critical load L_c depends not only on the coating's adhesion but also on several other parameters; some of which are directly related to the test itself (the intrinsic parameters). For example, loading rate, scratching speed, tip radius and wear rate. Others are related to the coating-substrate combination, i.e., the extrinsic parameters such as substrate hardness, coating thickness, substrate and coating roughness, friction coefficient between tip and coating and the friction force in the scratching direction (Steinmnann *et al.*, 1987).

Based on experimental study (Hirose *et al.*, 1996), this paper reports some theoretical analyses based on contact theory about the critical load L_c. A mathematical model and associated simulation program to predict the critical load L_c is also proposed.

MATHEMATICAL MODEL

Contact problem

Hertz contact theory says that the distributed pressure around the indentation of the plane sample could be given by the following equation (Sakurai and Hironaka, 1984), when a spherical indenter contacts a plane sample:

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* This work was carried out in Kanazawa University, Japan

$$P = \frac{3L}{2\pi a^2}\left[1 - \frac{x^2 + z^2}{a^2}\right]^{\frac{1}{2}} \qquad (1)$$

Where P is the distributed pressure, L is the normal load, a is the indentation radius, x and z are X-axis and Z-axis on the co-ordinate values (see Fig. 1). The indentation radius a is given by the following equation

$$a = \left[\frac{3Lr}{4E}\right]^{\frac{1}{3}} \qquad (2)$$

Where r is the diamond indenter radius, E is the effective elastic modulus.

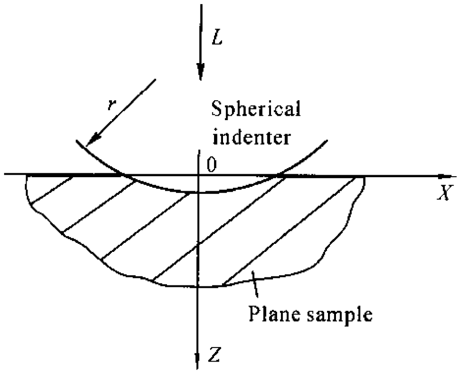

Fig. 1 Schematic of contact between spherical indenter and plane sample

The effective elastic modulus E may be given by following equation

$$\frac{1}{E} = \frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \qquad (3)$$

Where E is the elastic modulus and ν is Poisson's ratio for the indenter (indicated by subscript 1) and the plane sample (indicated by subscript 2).

As the indention pressure on the plane sample is increased to a critical value P_c, the complete plastic deformation zone is formed in the indentation of the plane sample. The critical pressure P_c could be given by following equation

$$P_c = 3Y \qquad (4)$$

Where Y is the yield strength (Bowden and Tabor, 1954).

Surface physics theory, reveals that contact essentially involves the effect between the molecules or atoms of the contact surfaces. The effect is related not only to the crystal structure and micro or macro defects of the contact surfaces but also other surface state factors.

Friction contact problem

In the scratch test, the indenter is slid in the direction tangent to the coating surface, as the normal load is applied between the indenter and the coatings surface. Parameters, such as the coating-substrate hardness, films thickness and surface state, that affect the tribology contact process must be considered. To correctly use the above-mentioned equations of Hertz's contact theory for the scratch test, they have to be modified according to the conditions of the scratch test.

In the scratch test of the coating-substrate the effective elastic modulus E is corrected from Eq. (3) to following equation

$$\frac{1}{E} = \frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_f^2}{E_f} \qquad (5)$$

where E is the modulus of elasticity and ν is Poisson's ratio for the substrate (indicated by subscript s) and the film coated (indicated by subscript f). In regard to the effect of the tangent friction force and the film coated, the friction coefficient μ and film thickness h are used for correction of Eq. (1) and Eq. (3). The equations are corrected as follows

$$a = \left[\frac{3L(1 + \mu^2)^{1/2} r}{4E}\right]^{\frac{1}{3}} \qquad (6)$$

$$P = \frac{3L(1 + \mu^2)^{1/2}}{2\pi(a^2 + h^2)}\left[1 - \frac{x^2 + z^2}{a^2}\right]^{\frac{1}{2}} \qquad (7)$$

Regarding the influence of the coating-substrate hardness and relation between yield strength and hardness, yield strength Y in Eq. (4) could be substituted by the effective hardness of the substrate hardness HV_s and film coat hardness HV_f. As a result, finally, the critical pressure P_c is given by the following equation

$$P_c = 0.63\left[HV_s^2 + \left(HV_f \frac{h}{a}\right)^2\right]^{\frac{1}{2}} \qquad (8)$$

Method and program for predicting critical load L_c

In the scratch test, pressure P could be given using Eq. (5), Eq. (6) and Eq. (7) for the known coating-substrate sample on which the load L_1 is applied L. If the critical pressure P_c

is reached by a load L; this load L is considered as the critical load L_c. In the critical load L_c predicting program, the initial conditions E_1, ν_1, E_s, ν_s, E_f, ν_f, HV_s, HV_f, h are used as inputs. The load L is calculated and recorded from 0.5 N to 200 N at steps of 0.5 N. Both x and z are 0.5 a for the position of maximum shearing stress at half indentation radius from the indentation center. Then the initial conditions are substituted into Eq.(5), Eq.(6) and Eq.(7). Pressure P is extracted for every step load L. Critical pressure could be obtained from Eq.(8). The pressure P is calculated at every load L from 0.5 N to 200 N and compared with P_c until $P = P_c$. The load output is critical load L_c. The flowchart of the program predicting critical load L_c is described in Fig.2.

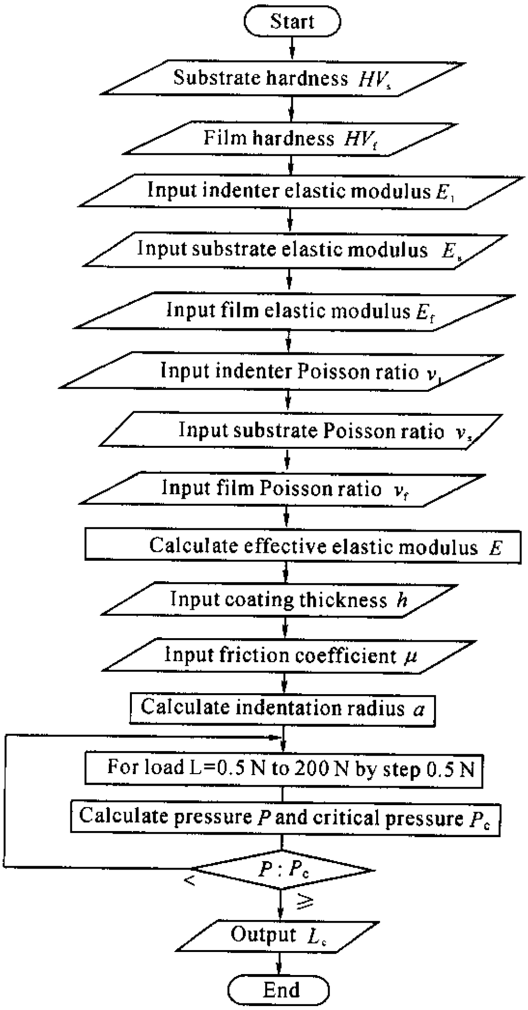

Fig.2 The flowchart of the program predicting critical load L_c

RESULTS OF NUMERICAL SIMULATION

The method and the program predicting the critical load L_c described above are used for numerical simulation predicting the critical load L_c. Fig.3 and Fig.4 are the simulation results obtained for TiN films and TiC films coated on different substrates, respectively.

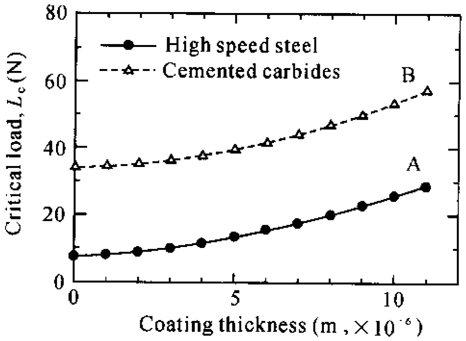

Fig.3 The dependence of critical load L_c for TiN coating on coating thickness and substrate type

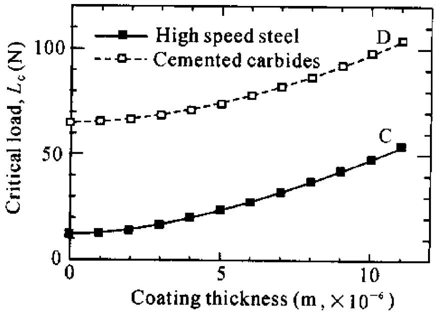

Fig.4 The dependence of critical load L_c for TiC coating on coating thickness and substrate type

The initial parameters predicting the critical load L_c are indicated as follows:

The diamond indenter elastic modulus is $E_1 = 1250$ GPa. The diamond indenter Poisson's ratio is $\nu_1 = 0.069$. The diamond indenter radius is $r = 200$ μm. The coefficient of friction between the indenter and surface of the samples is $\mu = 0.08$.

The initial parameters for the TiN film coat are $HV_f = 19.5$ GPa, $E_f = 450$ GPa, $\nu_f = 0.19$. The initial parameters for the TiC film coat are $HV_f = 32$ GPa, $E_f = 250$ GPa, $\nu_f = 0.2$.

The initial parameters for the substrate of high speed steel are HV_s = 7 GPa, E_s = 210 GPa, $\nu_s = 0.297$. The initial parameters for the substrate of cemented carbides are HV_s = 16 GPa, E_s = 620 GPa, $\nu_s = 0.21$.

Fig.3 and Fig.4 show the variation of critical load of TiN and TiC films, respectively. Curve A in Fig.3 and curve C in Fig.4 are for samples on high speed steel substrates while curve B in Fig.3 and curve D in Fig.4 are for films on cemented carbides substrates. The thickness of each film varied from 1 μm to 10 μm, In such a case, the critical load L_c varied from 34.5 N to 53.5 N in curve A, from 34.5 N to 53.5 N in curve B; from 12.5 N to 48 N in curve C, and from 64.5 N to 98 N in curve D respectively.

Moreover, these results show that the critical load L_c increased with the film coat thickness. For the same kind of film with the same thickness, the critical load L_c increases with the substrate hardness. For the same substrate material, TiC coatings showed higher L_c than TiN coatings of comparable thickness.

The comparison between the predicted values and experimental ones are presented in Fig. 3 and Fig.4. For the TiC film coated on cemented carbides (thickness h = 3 μm), the predicted and experimental L_c were 68 N and 68 N (Burnett and Rickerby, 1988), respectively. And for the TiN film coated on cemented carbides (thickness h = 2 μm), we found L_c was 35 N while the experimental value was 38 N (Kawata, 1989). We found that they both agreed well.

However, experimental values could sometimes be very different for the same kind of film coated on the same substrate. The reason is as follows. On the basis of surface physics theory, the adhesion of the film coat involves the effective force between the molecules or atoms of the coatings-substrate interface. But the adhesion is evaluated through the critical load L_c in the scratch test. The critical load L_c relates not only to the adhesion, but also to the scratch test parameters such as loading rate $\mathrm{d}L/\mathrm{d}t$, scratching speed of the indenter $\mathrm{d}x/\mathrm{d}t$, indenter radius r, mechanical properties of the indenter material, indenter surface state. It also relates to the contact tribology factors, such as elastic and plastic mechanical properties, surface state, friction state, defects or internal stress, that may be involved in the coating process (Steinmnann *et al*., 1987).

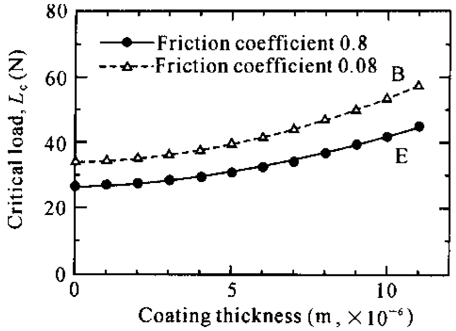

Fig.5 The dependence of critical load L_c for TiN coating on friction coefficient value

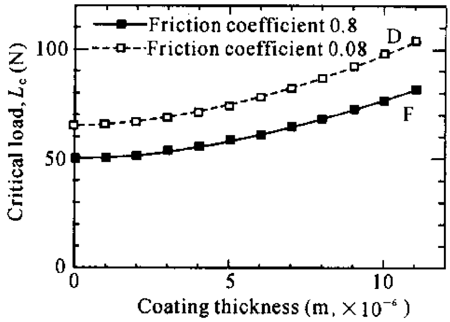

Fig.6 The dependence of critical load L_c for TiC coating on friction coefficient

A rule-of-thumb is that, in order to sample the coating only, the indentation depth is required to be less than one-tenth of the coating thickness. Thereafter, in the scratch test, where critical loads L_c for coating failure are of the order of kilograms, deformation of both coating and substrate will always occur (Burnett and Rickerby, 1987).

The extent of the coating-substrate hardness assembly deformation caused by the scratching point is mainly dictated by the substrate deformation. When the substrate hardness increases, a higher load is required to obtain the same plastic deformation. If it is assumed that the adhesion is the same and that the critical load is determined by the degree of deformation, then the critical load should increase with the substrate hardness.

For the same reason, it can be stated that a coating thickness increase requires an increased load to obtain the same degree of deformation and therefore the critical load increases with the coating thickness (Steinmnann *et al*., 1987). However, in some cases L_c appeared to decrease with increasing coating thickness and in extreme cases the coating can spall spontaneously when a critical thickness is exceeded without additional mechanical energy input. This tends to occur when the mechanical adhesion is poor and/or the internal stress is high so that the coating failure is dominated by the stress levels already present within the film; the additional stress-energy provided by the scratch test is small (Burnett and Rickerby, 1987).

If a specific coating-substrate system is considered, it can be said that the critical load depends on the frictional coefficient; higher values for the coefficient of friction correspond to lower critical load values (Steinmnann *et al*., 1987). L_c calculation was performed for TiN and TiC coating on cemented carbides changing the friction coefficient from 0.8 to 0.08. Our results showed that, in all the cases, the critical load L_c was decreased when the friction coefficient increased as presented in Fig.5 for the TiN coating on cemented carbides.

CONCLUSIONS

Factors effected on the critical load L_c in scratch test were discussed. Mathematical model and program predicting the critical load L_c in scratch test were proposed. Numerical simulation was carried out for TiN film coated and TiC film coated on two different substrates. It was observed that the critical load L_c should increase with the film coated thickness. For the same films with the same thickness the critical load L_c should increase with the substrate hardness. For the same kind of substrate material TiC coatings exhibit higher L_c than TiN coatings of comparable thickness. But higher values for the coefficient of friction correspond to lower critical load values.

This study theoretically investigated the scratch testing which is widely used for evaluating the adhesion of film. The mathematical model and program are applicable for predicting the critical load L_c in scratch test of hard film. The scratch test provides a rapid means by which the adhesion of coatings can be qualitatively assessed. The results obtained from the scratch test reflect not only the adhesive strength of the coating-substrate interface but also the action of all the stresses across the interface. In the testing process, many phenomena take place at once. Therefore, it is extremely difficult to make a model for analysis. In this paper, the focus is put on mainly the deformation analyzed by the simple model. The mathematical model predicting the critical load is proposed based on application of contact theory and surface physics theory. In order to improve the evaluation of coatings adhesion of the scratch test possibly, related factors influenced on the critical load L_c in scratch test must be investigated on the basis of conditions of the scratch testing and coating processes.

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