Vol.25 No.5 TRANSACTIONS OF MATERIALS AND HEAT TREATMENT PROCEEDINGS OF THE 14TH IFHTSE CONGRESS October 2004

Evaluation of the Interfacial Adhesion between Brittle Coating and Ductile Substrate by Cross-Sectional Indention

SU Jian-yu[1,2], ZHANG Kun[1], CHEN Guang-nan[1]

1. Institute of Mechanics, Chinese Academy of Sciences, Beijing, China

2. Graduate School of the Chinese Academy of Sciences, Beijing, China

Abstract: The cross-sectional indentation method is extended to evaluate the interfacial adhesion between brittle coating and ductile substrate. The experimental results on electroplated chromium coating/steel substrate show that the interfacial separation occurs due to the edge chipping of brittle coating. The corresponding models are established to elucidate interfacial separation processes. This work further highlights the advantages and potential of this novel indentation method.

Key words: coating, interface adhesion, indentation

THE EVALUATION of the interfacia adhesion is the predominant factor in determining the performance and reliability of coating substrate system [1]. There are more than 200 different methods for measuring adhesion, but most of which are problematic at high adhesive strength levels [2].

Cross-sectional indentation has been designed to examine the interfacial fracture properties of thin film on brittle substrate, whatever the film is ceramics [3] or metals [4]. The novelty of this indentation technique is the usage of cross-sectional sample and edge chipping effect.

The aim of this paper is to extend the cross-sectional indentation technique to the study of interfacial fracture in brittle coating/ductile substrate system, and to describe the corresponding analysis to account for interfacial separation at high adhesive strength levels.

1. Experimental Procedure and Principle Analysis

The material considered in this work consists of an electroplated chromium coating with the thickness of 170 micrometer on a steel substrate. Cross-sections are prepared by refined rubbed without further polishing. Appropriate etching process is adopted to reveal the interface.

During the indentation, the Rockwell indenter is jostled into the steel substrate smoothly near the interface, as shown in Figure 1. The indentation process can be observed by the control of the loading force. Due to the brittle coating/ductile substrate system, cracks are not initialized in the substrate but in the coating. There are three cracks *l*, *m*, *n* initialized and propagated in the coating successively. Among them, the initialized and propagated of crack *l* at point *b* is extremely abrupt. Cracks *m* and *n* grow steady from points *d* and *e* to points *a* and *c*. When they grow to some degree, they run out to the surface quickly. After that, with the indentation going on, owing to the edge chipping effect, because inner coatings between *m* and *n* have nothing to crutch in *x* direction, they move outwards as a whole. The interfacial cracks initialize at the points *d* and *f* then grow towards inside. Finally, when interfacial cracks *m* and *n* reach to point *e*, the cracks line up and the whole coating is broken off.

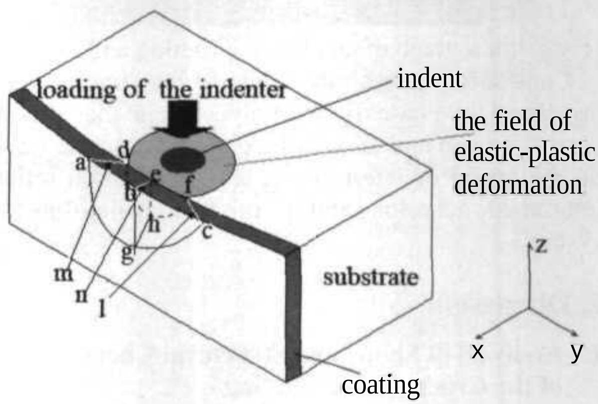

Fig. 1 Schematic of the cross-sectional indentation test

According to the analyses above, the process can be divided into three following stages:

Stage I: The stage as an integer: from the beginning of indenter's reach to the cross-section to the rapid propagation of crack *l*.

The experiment has showed that the process of initialization and propagation of crack *l* are very quickly. We can figure that once the stress on point *b* reach to the limit strength of fracture, crack *l* initializes immediately and run out rapidly. Before that, the system of coating/substrate can be considered as a whole object.

Stage II: The stage of the crack propagation in the coating, as shown in Figure 2: from the appearance of crack / to the running out of cracks *m* and *n*.

After the appearance of crack *l*, there are two strong acoustic emission signals in a short time. These mark the running out of cracks m and *n* to points *a* and *c* at the surface of coating. Before the signals there are not apparent cracks in the interface thought the observation from the cross-section. So it can be considered that there are no interfacial cracks in this stage.

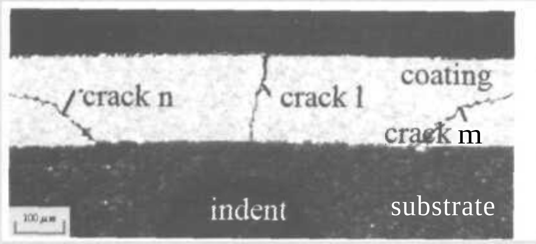

Fig.2 Micrograph of crack in the coating

Stage III: The stage of interfacial crack propagation, as shown in Figure 3: from the cracks *m* and *n* running out to the coating chipped from the system.

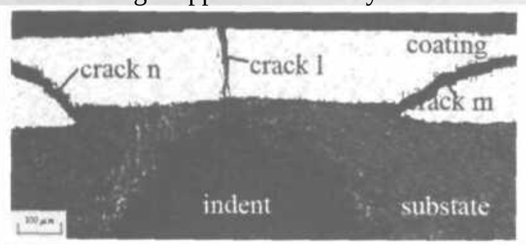

Fig.3 Micrograph of crack in the coating and interface

Considering that the interfacial cracks have initialized in Phase II, we only explain the interface cracks' growth and the coating chipped from the system. So the speed of interface crack's propagation reflects interfacial adhesion ability of the coating/substrate system.

2. Discussion

2.1 Analyses of Shape and Structural Characteristic of the Cracks in the Coating

The first application of the edge chipping effect during the indentation is not used in the system of coating/substrate. At the end of 1980s, it had been used to evaluate the edge toughness of hard materials [5].

In the experiment of edge chipping of hard materials, the three cracks in hard materials are similar to those in the brittle coating of our experiment [6]. Because the cracks' initialization and propagation relates to edge chipping effect during the indentation, the position and condition of cracks initiation in the experiment of hard materials can be easily calculated [5]. Although there are a ductile substrate layer between indenter and brittle coating in our experiment, and the force and the displacement of indenter are transferred to brittle coating by the elastic-plastic deformation of ductile substrate, the pressure on the coating is slimily to that direct loaded by indenter. So in our experiment the cracks in coating display a similar shape to those in the edge chipping experiment.

In addition, McCormick' analysis showed the following feature [5]. In the hard materials' experiment, under an increasing indentation force, cracks normally develop and grow stably and steadily into a declining stress field, before the cracks run to the critical position. However the location of this position remains unclear, but is presumably between 50% and 70% of the final crack length. We can conclude that the assumption the cracks *m* and *n* grow stably before the acoustic emission signals in the stage II of our experiment is reasonable. And the critical chipping force can be determined when by the acoustic emission signals.

2.2 Characteristics of Substrate and Interface in the Cross-Sectional Indentation Process on Ductile Substrate

Although the location of indenter on ductile substrate and brittle substrate are similar, there is great difference between them [3, 4]. For the brittle substrate, the substrate will be fractured directly when the indenter jostles into it. The chipped substrate will push the film outwards when the loading goes on. So the interface can be broken without much pressure and displacement. It can be done even by a nanoindenter. For the ductile substrate, however, because the pressure and displacement loaded on coating must be transferred indirectly by elastic-plastic deformation of substrate, and the value of them is affected by both the rigid movement of the indenter jostling into the substrate and elastic-plastic deformation of substrate, the indent is much bigger than that on the brittle substrate. Furthermore, due to the deformation of substrate, the appearances of the experiment of both methods are different too. The interfacial cracks on brittle coating expand to outside but the interfacial cracks on ductile coating expand to inside.

There is still similarity between the two methods. Firstly, the interfacial cracks develop and grow stably [3] no matter the interfacial cracks expanding to outside or inside. Secondly, because the crack in coating interface is always semi-circular on the surface whatever the coating is ductile [4] or brittle [3], the coating can be simplified as a tapered beam such as the assumption of brittle substrate by ignoring the influence of hoop stress in the coating as the scheme of Figure 4, then the deformation of coating can be analyzed as the bending of tapered beam. At last, the method of energy analysis is still to be used to calculate interfacial adhesion ability in brittle substrate as ductile substrate.

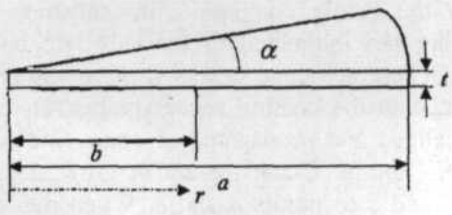

Fig. 4 Plate modeled an assembly of tapered beams

2.3 Transfer of the Force and Replacement during the Loading and the Corresponding Model

The translation of force and replacement is mainly based on the physical description of different stages of the analysis of indention process above.

2.3.1 Stage I: Stage as an Integer

When the indenter jostles into the substrate, because of the weaker crutch on the coating side, the indenter tends to move towards the side of coating, there is a side force added in the substrate. At the same time, because of the shape of the Rockwell indenter, the force on point *e* is bigger than points *d* and *f*, then there is a bending on the coating and a maximum tensile stress on point *b*. Because the inner coating of the points *d* and *f* is hustled out much more than the outer coating, there is another two maximum tensile stress on these points. But the restriction of substrate makes the stress on these two points much smaller than it on point *b*. When the loading of indenter is increasing to some value, the tensile stress on point *b* reaches to the limit stress of fracture and the coating is broken. Because there is not any restriction, we consider that the crack propagates rapidly to the end and does not grow anymore after this fracture process.

2.3.2 Stage II: Stage of the Crack Propagation in the Coating

When the indenter jostles into the substrate continually, the substrate outside is hustled by the indenter. When tensile stress on the danger points *d* and *f* reach the limit, crack *m* and crack *n* initialized. The displacement on point *e* is the greatest one according to the shape of indenter. Block *agb-dhe* and block *bgc-dhf* can be simplified to taper beams based on the analysis above. The plate model is shown in Figure 5. In the figure, *M*, F_0 is the resultant force loading on the coating by substrate, and u_0 is the resultant displacement loading. We can analyze the translation of force and replacement in loading on the model before the cracks run to the critical position. After that, surface *adhfcga* is the section of coating, and blocks *agb-dhe* and *bgc-dhf* are chipped from the whole coating, only connected with substrate by interface *dhfed*.

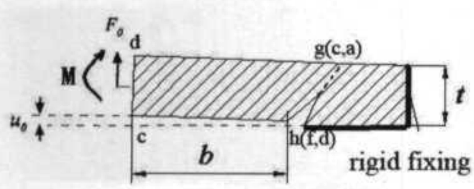

Fig.5 The model of Stage II

2.3.3 Stage III: Stage of Interfacial Crack Propagation

If the loading on the indenter keeps growing after coating cracks run out to the surface, without the whole coating's crutch, the inner coating tends to move outwards as a whole by the hustle of the substrate under the side force of indenter, but the substrate on the outsides of interface *dhfed* is restricted by the whole coating. As a result, the crack in the interface on the arc *dhf* propagates because of the discontinuousness of the displacement between the outer substrate and the inner coating around arc *dhf*. So the model as a simplified taper beam has been established as the Figure 6. The chipped coating taken as rigid member, the substrate outline along to the crack path must be arc-shaped because of the indenter's shape. So there is a tearing process in the interfacial fracture of brittle coating and ductile substrate.

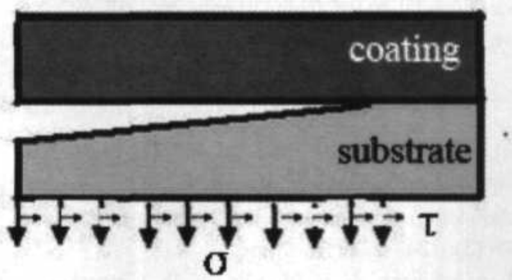

Fig. 6 the model of Stage III

2.4. Compared with Previous Measuring Method

The evaluation of the interfacial adhesion at high strength levels has always been a rough task because the interface separation is difficult to realize directly by tension, shearing or bending. One may make a pre-crack on the interface before the test and evaluate the adhesion by the crack propagation such as four-point bending experiment or interface indentation experiment. For the brittle coating, however, a slight disturbance may cause the crack propagating and the coating chipping quickly. So, in practice, the evaluation of strong adhesion interface is normally based on the engineering method, such as whetting/sawing methods.

The whetting/sawing methods are to scrape the cross-section by grinding wheel or saw from the substrate to the coating, and make the coating separate from the substrate. In fact, there is the same effect of edge chipping between cross-sectional indentation methods and the whetting/sawing methods. The loading on the ductile substrate instead of the brittle coating avoids the centralization of stress and the chipping in a short time. The cross-sectional indentation process on ductile substrate could be considered as quasi-static, and the pre-crack has been initialized indirectly in the stage II. As a result, one may easily control the process of the stage III i.e. that of the interfacial cracks propagation. The cross-sectional indentation is believed to widely accepted, provided that one may reveal the relation between the interface fracture and the parameters including the loads and the distance from the indenter to the interface.

3. Conclusions

(1). Cross-sectional indentation may be extended to evaluate the interfacial adhesion in brittle coating/ductile substrate system, especially at high adhesion strength levels.

(2). Like the engineering method of whetting/sawing, this novel method with the same effect of edge chipping will be practically applied.

Acknowledgment

The financial support provided by the National Natural Science Foundation of China (Grant No.59836220) is greatly acknowledged.

References

1 HUANG Li-ye, XU Ke-wei, LIU Jian. Nano-scratch Behaviors of Diamond Like Carbon Films during Loading-Unloading Processes. Acta Metallurgical Sinica, 2001, 37(12): 1247-1250 (in Chinese).

2 A A Volinsky, N R Moody, W W Gerberich. Interfacial Toughness Measurements for Thin Films on Substrates. Acta Mater, 2002, 50(3): 441-466.

3 J M Sanchez, S El-Mansy, B Sun, T Scherban, N Fang, D Pantuso, W Ford, M R Elizalde, J M Martínez-Esnaola, A Martín-Meizoso, J Gil-Sevillano, M Fuentes and J Maiz. Cross-sectional Nanoindentation: A New Technique for Thin Film Interfacial Adhesion Characterization. Acta Mater, 1999, 47(17), 4405-4413.

4 M R Elizalde, J M Sánchez, J M Martfnez-Esnaola, D Pantuso, T Scherban B Sun, G Xu. Interfacial Fracture Induced by Cross-Sectional Nanoindentation in Metal-Ceramic Thin Film Structures. Acta Mater, 2003, 51(14), 4295-4305.

5 N J McCormick, E A Almond. Edge Flaking of Brittle Materials. J. Hard Mater, 1990, 1(1), 25-51

6 R Morrel, A J Gant. Edge Chipping of Hard Materials. Int. J. Refract. Met. Hard Mater, 2001, 19(4-6), 293-301.

Corresponding author. Dr. Zhang Kun,
E-mail: kzhang@imech.ac.cn
Mail address: Institute of Mechanics, Chinese Academy of Sciences, 15 Beisihuanxi Road, Beijing 100080 China,
Tel: 86-10-62547527-3, Fax: 86-10-62561284