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SCRATCH TESTERS ON THE APPLICATION TO THE ADHESION MEASUREMENT OF THIN COATINGS

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ABSTRACT

Two models of scratch testers developed recently are applied to the adhesion measurement of thin coatings. In both models the critical load is determined from the frictional irregularity when coated surface is broken in the scratching process. One model is effective for coatings thicker than 1m and the other for thin coatings down to 0.1μm.

The mechanism and the measurement principle of these testers are described. High sensitivity and good reproducibility of the measurement are attained primarily by the employment of new detection devices. These testers are even more useful for the quality control and the inspection of commercial products of coatings because only a small area of specimen is required for the test.

INTRODUCTION

For various engineering applications, functional devices and machinery parts of high performance can often be realized by coating the base materials with thin skins of other materials of metals, ceramics and polymers. This coating technique has been developed more and more as the actual evidence of the advantage has been reported even from the economical point of view. In these composite materials, however, the heterogeneous structure sometimes raises a new problem about the interfacial strength i.e. the adhesion of coating to the substrate. A lot of testing methods have been designed to study and evaluate the interfacial strength in order to examine and also to improve the property of the composites. Historically coatings whose thickness was greater than 10μm was first put into a practical use to obtain a good cutting property of machining tools, where testing methods[1] such as the Elicsen test, scratching, indentation, bending, and friction could have been employed successfully.

Recent trends in modern engineering and advanced technology aim at the use of much thinner coatings, and cases to meet a limitation for the adhesion measurement with the conventional testing methods have been increasing from the viewpoint of the sensitivity. In the present report, the application of two scratch testers developed in RHESCA Co., Ltd. to such thin coatings are described.

These testers basically detect the critical stylus load for the detachment of the coatings. It is demonstrated that the reliability, the reproducibility and amongst the sensitivity can be improved greatly with these testing machines.

DETAILS OF OUR SCRATCH TESTERS

Model CSR-01 : for coatings > 1㎛ thick

The scratch tester CSR-01 is designed to measure the adhesion of thick coatings. A schematic illustration of the scratching mechanism is shown in Fig. 1. A coated specimen is scratched with a Rockwell-C type (120° cone with a tip of 0.2㎜ in radius) diamond stylus. The stylus load is applied up to a maximum of 1~20kgf with an accuracy of 1/1000 of the full scale. The maximum load or the resolution can be changed by choosing a spring of proper elasticity which supports the specimen stage. The stage is driven at a constant rate of 0~20㎜/min. The stem of the stylus nose equips a semiconductor 2-axis load sensor to measure the load and friction. Another transducer to detect the acoustic emission can be equipped optionally in the same way as a conventional scratch tester[2] if one wishes to observe it.

Model CSR-01 can be applied to coatings of from soft resin to hard ceramics whose thickness is greater than 1㎛ typically. As the Rockwell diamond stylus is pressed to the specimen surface gradually from 0 to a preset value, the specimen is moved horizontally. The test sequence can be programmed by a micro-computer to exclude an ambiguity due to the operator's technical skill and also to

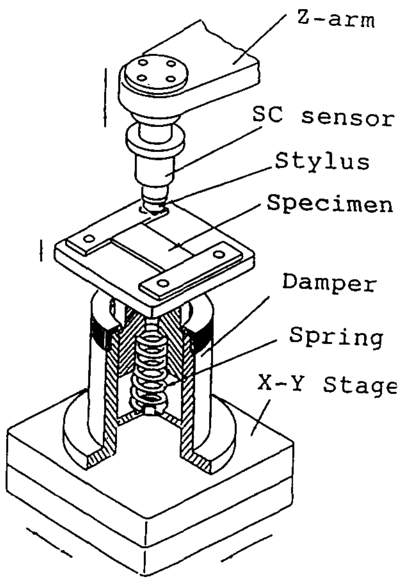

Fig. 1 Schematic view of the model CSR-01

study the effect of the scratching conditions. A damper unit is employed in the specimen stage to reduce the load fluctuation in the loading process.

The vertical load and the friction force are recorded in a chart sheet. The critical load value where the detachment of the coating is observed with an anomaly in the friction force is taken as a measure of the adhesion strength of the coating. To detect the onset of the friction anomaly in a clear manner, time derivative of the friction signal is also recorded.

An example of the adhesion measurement using CSR-01 is given in Fig. 2, where a glasssubstrate coated with Si_3N_4 of 2㎛ thick was examined. A passing irregularity was observed at the load of 2.3kgf, and the critical detachment of Si_3N_4 occurred at 3.2kgf. As shown in the dF/dt signal, the coating fracture could be detected with enough margin above the noise level. A damper unit in the

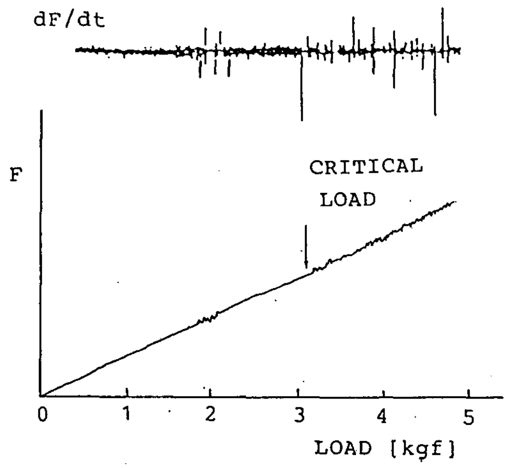

Fig. 2 Scratch test of Si_3N_4 coating of 2μm thick by CSR-01.

specimen stage was found effective to reduce not only the load fluctuation but also the background noise.

Model CSR-02 : for coatings < 1μm thick

The scratch tester CSR-02 is designed to measure the adhesion of thinner coatings compared to CSR-01. As the coating thickness decreases, the signal emitting upon the coating detachment becomes small. To detect such small signal, a new detection mechanism is devised.

The new scratch head consists of a stylus of light mass and an electromagnetic pickup of the stylus vibration. An illustration of the stylus and the sensor unit is shown in Fig.3, whose feature is almost the same as the pick-up cartridge of the phonograph. It is, however, the cartridge body that is forced to vibrate in the new tester while the stylus is forced to trace the music channel on a record disc in the phonograph. The vibrating stylus works like a vibration wear tester. The voltage signal generated in the cartridge Ke

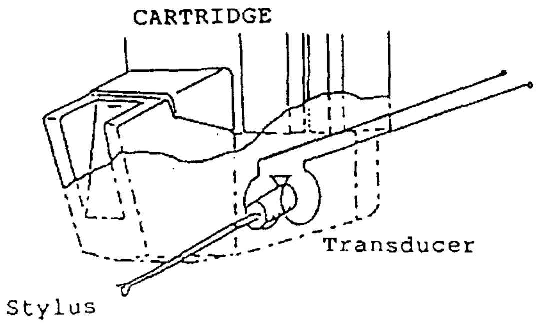

Fig. 3 Schematic View of the Sensor Head of CSR-02.

flects the frictional characteristics of the coated surface. It has been reported that the signal can be analyzed through a simple equation of motion[3]. In a word the signal power is proportional to the friction working on the stylus.

The surface of thin coatings showed a variety of morphology than that of thick coatings does. It depen ds on the degree of surface finish of the substrate and also on the preparation condition of the coatings. Thin coatings cannot be regarded as flat plates any longer, and a sharp stylus is required to investigate the interface between a thin coating and the substrate. All these things make the modeling and the analysis of the scratching process difficult for very thin coatings. Hence, there is not any standard test condition that can be applicable to all of the thin coatings. If one could establish a good test condition for a coating, it means that he could get an important information about the nature of the particular coating. In the model CSR-02, the stylus vibration amplitude of 0~80μm, the weight loading rate of 0~10 mN/s, and the scratching speed of 0~60μm/s can be varied as test conditions. The tip radius of the hemispherical diamond stylus is chosen from 5 to

100m.

The test procedure is essentially the same as that of CSR-01. That is, the vibrating stylus is pressed to the specimen surface gradually, and at the same time the specimen is moved laterally. When the coating is broken, an irregular frictional response is observed.

It is the special point of CSR-02 that the horizontal translation (i.e. scratching motion) of specimen is not necessary to detect the fracture signal. Even if the scratching speed is zero, the critical fracture can be observed. With the low scratching speed, however, the surface is subject to repeating stimulus due to the stylus vibration, and the result will be affected by wear and fatigue.

An example of the adhesion measurement using CSR-02 is shown in Fig. 4, where a glass substrate coated with SiC of 0.04μm and Cr of 0.08μm thick was examined. The radius of the used stylus was 14μm, and the scratching speed was 20μm/s. The critical detachment of SiC occurred at 8.2gf. The photograph of the scratched surface is given in the upper part of Fig. 4, and the critical fracture of the coating can be seen in correspondence to the friction signal.

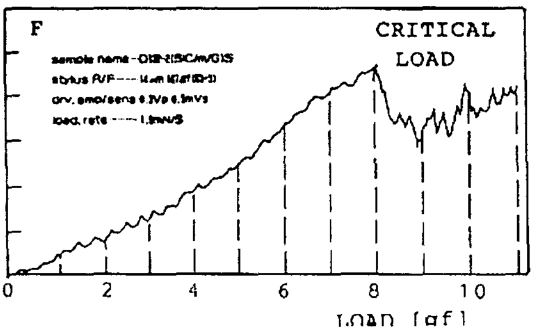

Fig. 4 Scratch test of SiC coating of 0.04μm thick by CSR-02.

DISCUSSION

In the study of the scratching process historically, the correlation of the adhesion and the friction has been considered as one of the main interest[4]. Nevertheless for the application of scratch test to the coating adhesion, the detection of acoustic emission has been a leading tool to determine the critical load for the coating detachment probably because the sensor for the acoustic emission is convenient to install in conventional scratch testers and it has enough sensitivity for thick coatings. Recently the utilization of friction to the adhesion measurement for thin coatings was reported successfully[5]. We also could make use of friction to the new testers in a much sophisticated way with high sensitivity as shown in the present report, and the usefulness of the testers could be demonstrated. Furthermore since these testers require only a limited area of specimen surface, they have a wide applicability such as the life test of actual products.

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