Surface & Coatings Technology 206 (2011) 1873–1878

Contents lists available at SciVerse ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

A modified scratch test for the mechanical characterization of scratch resistance and adhesion of thin hard coatings on soft substrates

T. Sander *, S. Tremmel [1], S. Wartzack [2]

Friedrich-Alexander-University Erlangen-Nuremberg, Martensstrasse 9, 91058 Erlangen, Germany

ARTICLE INFO

Available online 31 August 2011

Keywords:
Scratch test
Adhesion
Scratch resistance
Critical load
Characterization
Soft substrates

ABSTRACT

The scratch resistance of coatings and the adhesion between coating and substrate are usually determined in model experiments performed with sharp diamond indenters. These common methods as described for example in EN 1071-3 often fail for the combination of hard coatings on soft substrates due to very small critical loads or no detectable failure modes at all. Hence in the industry a lot of highly subjective and provisional test methods are used. On the one hand, quantitative comparability is difficult with common methods since defects already occur at very small loads for ductile and relative soft substrate materials like plastics. On the other hand, wear of the indenter in contact with hard coatings like pure diamond coatings requires its cost-intensive replacement.

In this article a macroscopic tribological–mechanical test method is suggested which uses balls of hardened steel as indenters. A wide scope can be applied for both soft and hard coatings and different substrate materials. By variation of the ball-diameter, the normal contact force and the sliding speed different levels of stress and wear can be induced to analyze the tribological and mechanical behavior between body and counterpart as well as the interface of coating and substrate. To determine scratch resistance close to reality as usual scratch conditions on consumer products are better represented by a small ball than a sharp diamond indenter. Another benefit of the presented test method is the cost saving acquisition of the balls for indentation in very high quality as they are standard parts in the ball bearing industry. For every test on very hard coatings a new ball can be used with the possibility to detect the wear both on the base object and the counterpart. The occurring failure modes of coating and substrate also can be compared with comparatively easy numerical models to verify the results. Additionally to the test concept, first results of different coatings will be presented in this paper and compared with the results of common scratch tests.

1. Introduction

The determination of adhesion and the scratch resistance is essential for the mechanical characterization of thin and hard films both for research and industry. To evaluate the performance of coatings the scratch test with a Rockwell-C diamond stylus as indenter is a commonly used test for the characterization of hard coatings on predominant metallic substrates due to its comparatively robust experimental setup, the easy use and the representative results. However, the versatility as compared to other mechanical test methods [1,2] is limited for several applications. While testing hard coatings on soft substrates, the failure modes occur at very small critical loads, which are difficult to monitor. In other cases, there cannot even be detected any failure modes at all. Further problems arise when ultra hard diamond layers are tested. The tip of the indenter abrades quickly and, as a consequence, the ideal geometry is destroyed.

The scratch test was modified by using bearing balls instead of the Rockwell-shaped indenter. This allows to easily change the contact radius and the material of the indenter and therefore to influence the stress field forming in the composite of coating and substrate. The conditions of measuring can be better adapted to the parameters of the subsequent application. In this paper the modified scratch test is introduced and applied on hard layers of CrN and TiN deposited on soft polymeric ASA substrates as well as on ultra hard diamond layers on steel substrates. Exemplary results are presented and compared with the common scratch test with a Rockwell-C indenter.

2. Experimental methods

2.1. Description of common scratch test and failure modes

The scratch test which is standardized in EN 1071-3 and compiled by CEN/TC 184 uses a sharp diamond indenter. The indenter is

* Corresponding author. Tel.: +49 9131/85 23224.
E-mail addresses: sander@mfk.uni-erlangen.de (T. Sander), tremmel@mfk.uni-erlangen.de (S. Tremmel), wartzack@mfk.uni-erlangen.de (S. Wartzack).
[1] Tel.: +49 9131/85 23222.
[2] Tel.: +49 9131/85 27987.

0257-8972/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.surfcoat.2011.08.035

dragged over a coated surface with a defined normal force. This load can be constant or linearly increasing during the test. The dimensions of the diamond indenter are similar to the Rockwell-C hardness test and are thereby well-defined in EN ISO 6508-2 as depicted in Fig. 1. In practice, however, there are often small differences in the geometry between the standard and the deployed indenter, mainly in the tip radius and the taper angle.

The load at which at least one of certain failure modes occurs at the coating and/or substrate is referred as the critical load L_C. For the different failure modes, the accordant critical loads describe the capability characteristics like adhesion, scratch resistance or utility value. The failure of coating or substrate can be determined by microscopic examination, measurement of friction force or acoustic emission. Bull [3] splits the normally occurring failure modes of hard coatings in a scratch test into three categories:

1. Through-thickness cracking: tensile cracking behind the indenter, conformal cracking and Hertzian cracking.
2. Spallation: compressive spallation ahead of the indenter, buckling spallation ahead of the indenter or elastic recovery induced spallation behind the indenter.
3. Chipping in the coating

The several failure modes are further described in Refs. [4–6] and can be compared and classified in failure mode maps with the microscopic images. The common scratch test with the Rockwell-shaped stylus delivers good and comparably reliable results for the determination of the adhesion,as long as the material properties like Young's modulus and Poisson's ratio of coating and the substrate material are close [7].

2.2. Sample preparation

Extruded sheets of ASA polymer (acrylonitrile–styrene–acrylester) of 3 mm thickness were used as substrates. CrN and TiN coatings were deposited by reactive cathodic arc evaporation on a downscaled hybrid PVD/PACVD industrial coating plant. The substrates were plasma pretreated in the same coating plant before the coating process. In order to be able to review the test method on a sample with reduced adhesion of the coating the plasma pre-treatment was intentionally omitted for one sample. The thickness of the coating was investigated via SEM analysis and optical microscopy of cross sections. The averaged thickness was 0.52 μm for the CrN and 1.51 μm for the TiN layer. The surface roughness $Ra = 0.38$ μm of the uncoated substrate was reduced to $Ra = 0.20$ μm (CrN) and $Ra = 0.29$ μm (TiN).

2.3. Limits of the scratch test with Rockwell-C indenter

Significant failure modes often cannot be detected at reasonable loads with the common Rockwell indenter. This applies in particular to layer composites with thin hard films on soft substrates. Fig. 2

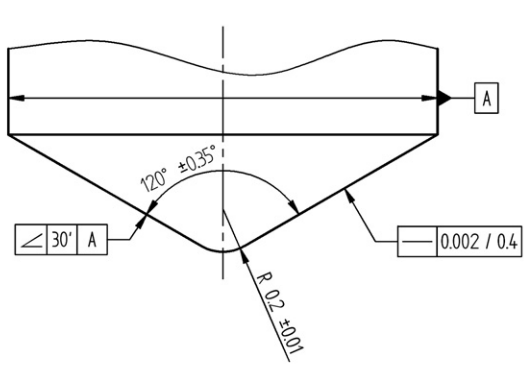

Fig. 1. Dimensions and tolerances of a commonly used Rockwell-C indenter according to EN ISO 6508-2.

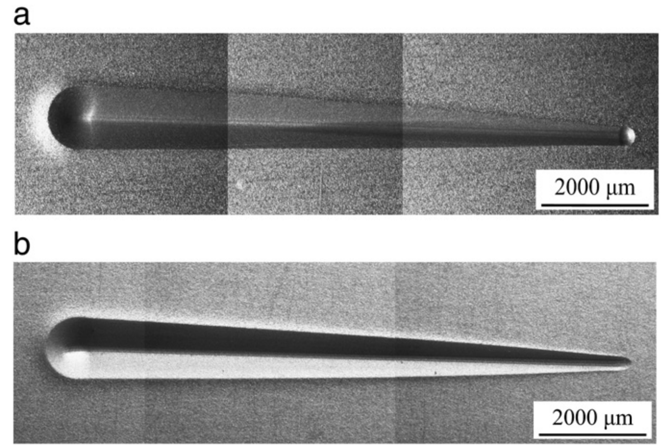

Fig. 2. Scratch test with common Rockwell-C indenter, linearly increasing load 0–100 N on soft ASA-substrate and thin hard layers (a) CrN and (b) TiN.

shows a CrN coated (a) and a TiN coated (b) polymeric ASA substrate, tested with a usual scratch test with a Rockwell-C diamond indenter and a linearly increasing normal force from 0 to 100 N. Apart from the large plastic deformation, neither buckling, spallation, chipping or cracking occurs. The layers are still completely undamaged. Bull [3] suggests that the trough-thickness cracking should be detected, as the polymeric substrate is very soft and the hardness of the layers are in the range of typical wear protective coatings, as shown in the map of the main scratch test failure modes in terms of substrate and coating hardness (Fig. 3).

With the measured critical loads of $L_C > 100$ N, the tested coatings seem to have a good adhesion to the substrate and to be very scratch resistant. However, in reality, the contact with everyday objects like pens or screwdrivers even at relative small loads can damage the composite of substrate and coating. This raises the question why this discrepancy in the tribological properties between the common scratch test and the utility value occurs and how it can be quantitatively registered. The friction between the indenter and the tested surface might have a considerable influence. The earlier analytical models for the scratch test [8–10] derive the stress condition mainly from the geometry of the indentation and the plastic zone. Nevertheless, besides the plowing component, a tangential force which deforms the material, Xie and Hawthorne [7] also describe the traction component, an additional tangential force caused by adhesion between surface and indenter. Hereby the friction and the indenter

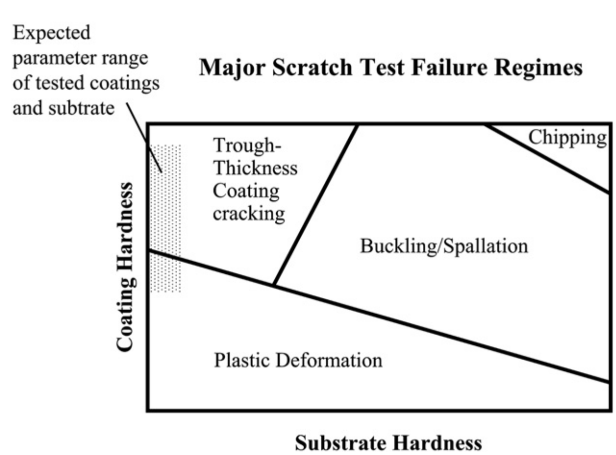

Fig. 3. Expected parameter range of tested coatings (CrN and TiN) and substrate (ASA) in the map of the main scratch test failure modes in terms of substrate and coating hardness by Bull [3].