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The Inclined Impact Test, an Efficient Method to Characterize Coatings Cohesion and Adhesion Properties

The impact test, supported by its finite elements method (FEM) simulation, has been successfully used to characterize the fatigue performance of coatings. In this test the load is exercised perpendicularly to the coated surface by a cemented carbide ball. In the inclined impact test the successive impacts are applied on an inclined surface. In this way, the coated surfaces are loaded vertically and tangentially simultaneously. The coating fatigue failure modes were classified by means of scanning electron microscopy observations and energy dispersive X-ray spectroscopy microanalyses. The experimental method is supported by a developed FEM simulation, which considers the mechanical elastic-plastic properties of the coating and of the substrate, as well as of the ball indenter during the impact test, thus enabling the elucidation of the coating failure modes. In this way, critical equivalent stresses were determined and the coating cohesive and adhesive impact performance was systematically investigated. The inclined impact test implies a new reference to the prediction of the coatings' cohesive and adhesive failure, managing to approach loading directions for a variety of coated surfaces in different applications. Examples for an efficient use of this test are presented and a characteristic magnitude, the Coating Impact Adhesion (CIA), is introduced.

Keywords: *Inclined impact test, coating, cohesion, adhesion, FEM simulation*

1. INTRODUCTION

The impact tester is applied as a convenient method for the characterization of coatings fatigue properties [1-4]. Furthermore, coating cohesive and adhesive failure modes can be elucidated through this test [6]. Valuable results arise also from the use of the impact test to predict the cutting performance of coated cutting tools [6]. Concerning thick coatings, creep behavior determination builds another interesting application area [7].

In previous investigations [6], the impact tester was applied as a coating cohesion and adhesion test, where the adhesion strength in the coating-substrate interface was described by means of FEM supported calculations using appropriate contact

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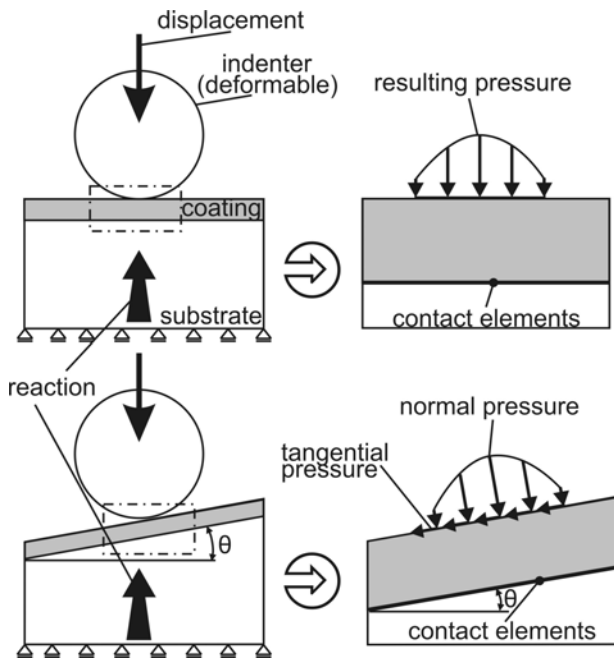*3. CemeCon AG, Würselen, Germany*

elements. On the one hand, in the case of a cemented carbides polished substrate, with elevated superficial Co-content and a poor coating adhesion, the coating failure appeared in distinct regions inside the imprint circumference. On the other hand, in the case of a diminished Co-content on the substrate surface, achieved through micro-blasting, an enhanced adhesion occurred and the first coating failure appeared near the impact crater vicinity.

These coating damage alterations, induced by the film adhesion characteristics, were possible to be theoretically elucidated by means of the mentioned FEM-supported calculations. According to these calculations, the position of the maximum developed stresses in the investigated coating - substrate combination was displaced in the case of a poor adhesion from the crater vicinity towards the crater center [6].

These results gave the idea to further affect the occurrence during the impact test stress distribution in the coating, applying simultaneously normal and tangential loads during this test. In this way, the effect of a poor adhesion on the film impact damage modes could be more distinct. Thus, the inclined impact test was originated in order to enable an effective evaluation of the cohesive and at the same time of the adhesive coating strength.

Figure 1: A FEM simulation of the inclined impact test with cylindrical indenter.

2. STRESS FIELD IN COATINGS WITH VARIOUS ADHESION DURING THE INCLINED IMPACT TEST

Aiming to have a precise overview concerning the stress field during the impact test in coatings with various adhesion strength properties, versus the inclination angle, a plane-strain FEM model was used. In this way, a comparison of the loads in film cases with good or poor adhesion properties can be achieved. The applied FEM model is presented in Figure 1, simulating the contact between a cylinder and an inclined coated plane [8]. In a first calculation stage, the resulting contact pressure between the deformable cylinder and the coating is determined (see left figure part) and in a second calculation stage this pressure is applied to a model with contact elements, being added in the coating - substrate interface (see right figure part). In this way, the quantification of the coating adhesion strength is dependent on the contact stiffness values, which are changeable parameters. The constitutive laws of the cylinder, the coating and the substrate, as well, are taken into account, being determined by means of nanoindentations and the "SSCUBONI" algorithm [9-11].

FEM simulation results elaborated by means of the previously described model, in well- and poor-adherent coating cases, are presented in Figure 2. The developed von Mises equivalent stress distributions are shown in the cases of various inclination angles. As it can be observed, there is a

steep increase in the maximum occurring stress during the inclined impact test with poor-adherent coatings, i.e. low contact stiffness values in the tangential surface direction, in comparison to the corresponding ones in well-adherent coating cases. This tendency is more intense in the cases of 10 and 15 degrees inclination angles, rather than in the case of the perpendicular impact direction, where the occurring maximum stress is almost the same in the cases of good and poor adhesion, however different distributed. The herewith-presented FEM simulation results demonstrate that in the inclined impact test, the coating adhesion strength properties affect significantly the occurrence in the coating stress field, which in turn could facilitate the characterization of the film adhesion in a more distinct way.
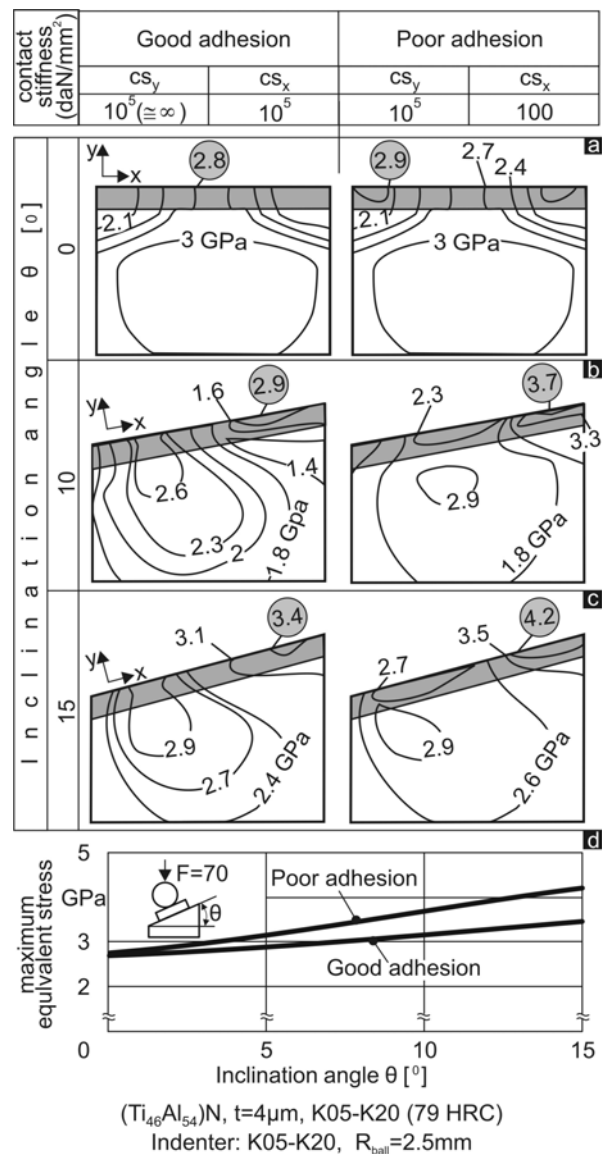

Figure 2: Occurring stress fields in the coatings, determined by means of the FEM simulation of the inclined impact test with cylindrical indenter.

3. THE IMPACT TEST PERPENDICULAR OR OBLIQUELY ON COATED SURFACES

The used impact tester device is presented in Figure 3a. During the impact test a cemented carbides ball indenter penetrates periodically into the coating under a desired maximum load [1,2]. Due to the plastic deformation that develops during the loading stage, the contact area does not fully recover to its initial plane shape, forming herewith a permanent concave imprint. The power supply module supports the whole experimental arrangement.
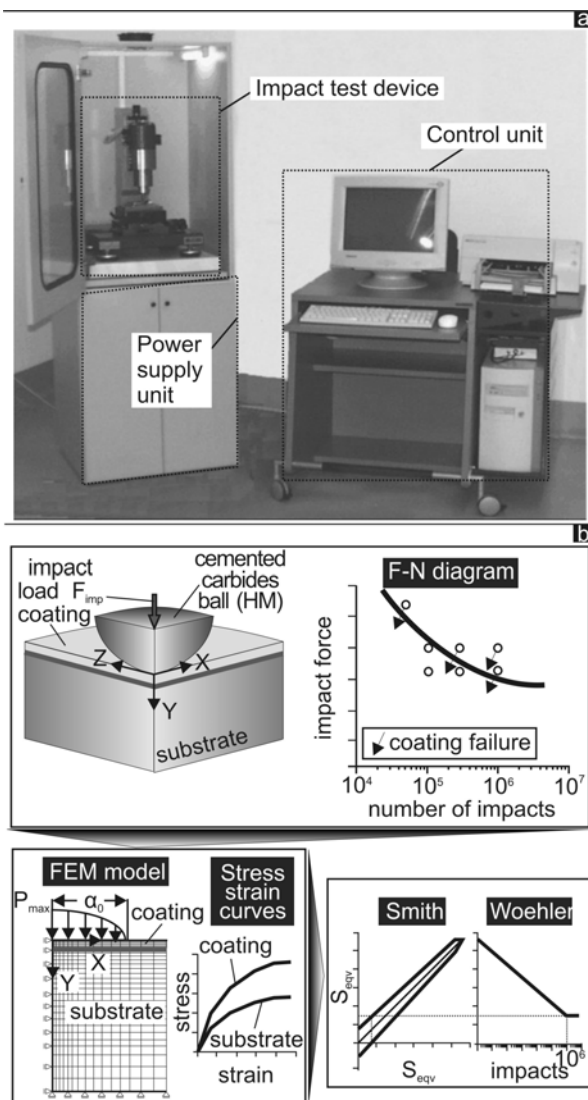

Figure 3: The impact tester working principle and its FEM simulation.

The output voltage of this module is fully controllable, with the aid of a variable transformer, in order to achieve the different impact forces on the impact tester module. In addition, the control module supervises the whole experimental procedure. This module consists of a personal computer (PC) equipped with a PID (Proportional, Integral, Differential) controller. With the aid of this controller it is possible to turn on/off the whole test arrangement, to adjust the output voltage on the variable transformer through a DC (Direct Current) motor, to conduct measurements of current, forces and temperature inside the impact tester. The impact tester is supplied by a fully automated software named "ITEC", which enables the determination of coatings fatigue properties in form of Smith and Woehler diagrams (see Figure 3b) [1], based on the FEM simulation of the impact tester.
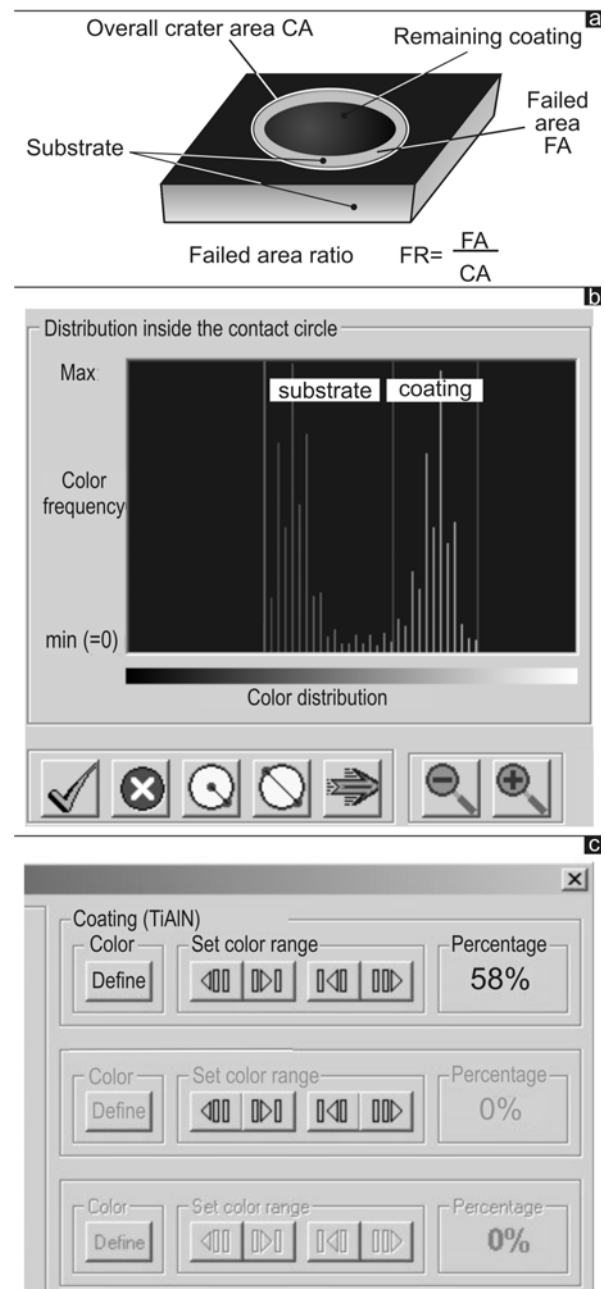

Figure 4: Definition of the coating failed area within the imprint, by means of the algorithm "WEPROC"

The failure extent in the impact test imprint is described through the failed area ratio FR, which is defined as the ratio of the region in which the substrate is revealed versus the overall contact area (see Figure 4a). This ratio is determined automatically with the aid of the developed algorithm WEPROC (WEar PROpagation Code) [12]. The working principle of the WEPROC software is based on the fact that the colors of Scanning Electron Microscopy (SEM) micrographs, especially of back-scattered ones, depend on the materials and in turn on the individual layers deposited on the surface. The analysis of the color density inside the crater area was used in order to determine the percentage of each coating layer revealed in the crater area and thus to calculate the failed area ratio FR [2,12]. Furthermore, the algorithm performs the color density analysis inside the contact circle and the results are illustrated in the color frequency chart and listed as well (see Figure 4b and 4c respectively). Herein, the range of colors that correspond to each layer has been specified, by means of EDX-investigations on ball-cratering test imprints. In order to enable inclined impact test investigations, an appropriate test rig for the coated workpieces was constructed. This test rig is illustrated in Figure 5a.
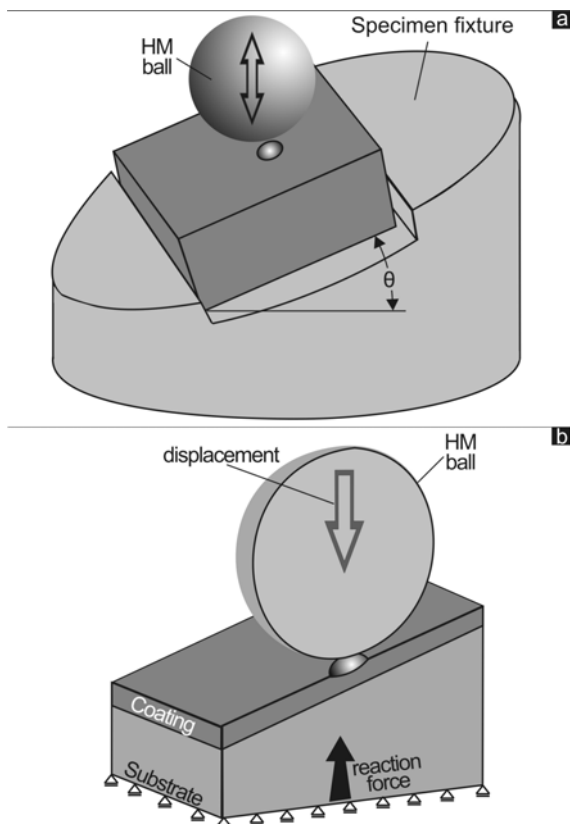

Figure 5: The applied test rig and its 3D FEM simulation.

In order to simulate the contact of a ball penetrating into the specimen, a three-dimensional FEM model was created. This model is demonstrated in Figure 5b, allowing the application of a displacement on the ball indenter and among others enabling the calculation of the resulting reaction force, corresponding to the impact force.
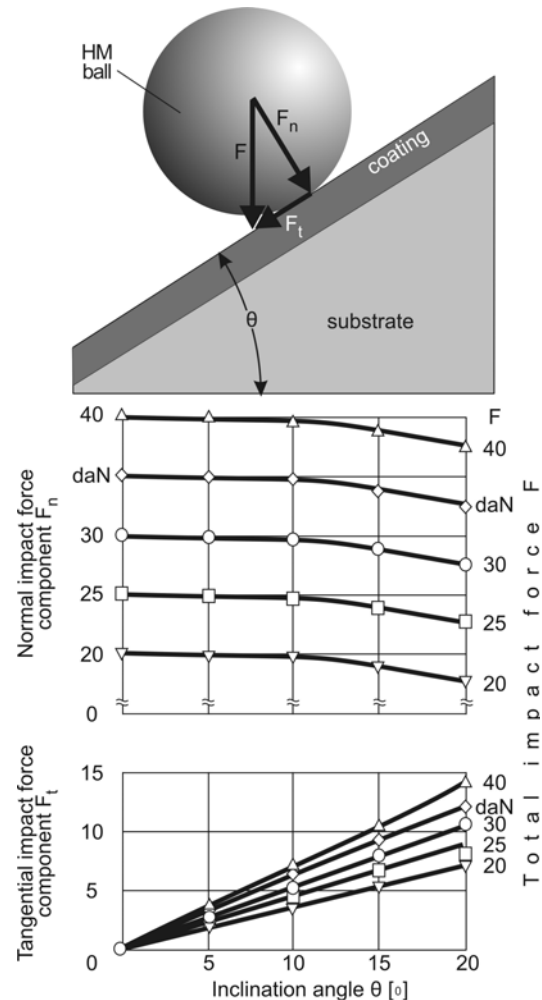

Figure 6: Normal and tangential force components over the applied impact load versus the inclination angle θ.

The impact force, during the inclined impact test, is analyzed into the normal component Fn and the tangential one Ft, with their absolute values depending on the inclination angle θ (see Figure 6). These force components for various impact loads versus the inclination angle θ are presented in the diagrams of this figure. As it can be observed, the tangential impact force component Ft is relatively low up to an inclination angle of 15 degrees, however produces intense adhesion problems, which can be detected through the resulting during the inclined impact test earlier coating failure in the case of poor adherent coatings, as it will be presented in the following sections.

4. CONTACT STRESS FIELD, COATING FRACTURE INITIATION AND FILM REMOVAL PROPAGATION IN THE INCLINED IMPACT TEST

By means of the developed 3D FEM model, simulating the inclined impact test, the stress field occurring in the contract region between the ball indenter and the coated specimen was calculated in various inclination angle cases (see Figure 7).
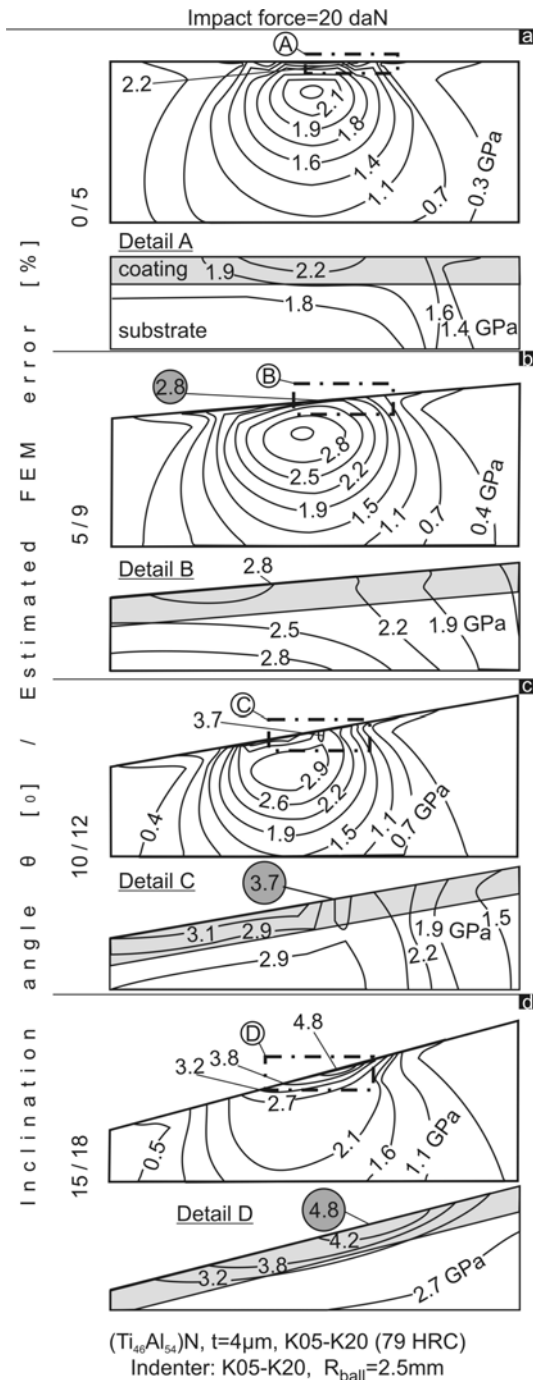

Figure 7: The occurring stress fields during the inclined impact test, deriving from the developed 3D FEM model.

Following an ascending inclination angle order from 0 up to 15 degrees, the corresponding FEM results are demonstrated from the top to the bottom of the figure respectively. The maximum occurring von Mises equivalent stress of a constant impact load has a growing tendency over the inclination angle and consequently it will lead to an earlier coating fatigue fracture initiation and to a consequent more intense film removal propagation.

The coating removal propagation, after the fracture initiation due to fatigue [1,2], can be attributed to its local overstressing owing to excessive relevant motion of the ball indenter to the coating surface. This motion is monitored through the developed 3D FEM simulation of the inclined impact test and is illustrated in Figure 8a.
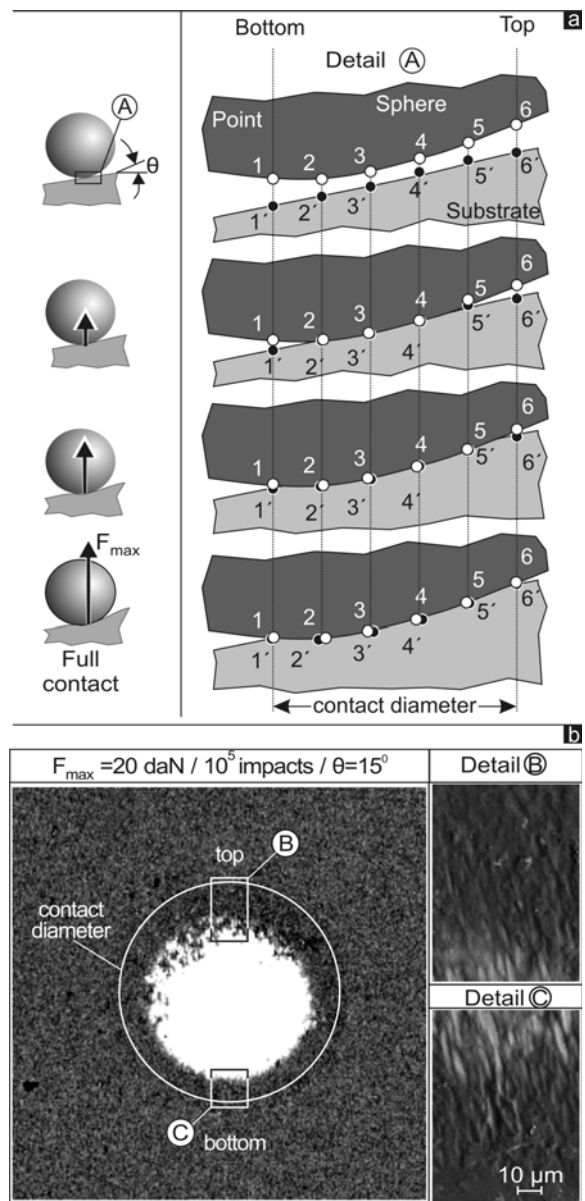

Figure 8: FEM determined relative motions of the ball indenter to the coating surface during the inclined impact test and related SEM micrographs

According to these results, an enhanced film removal is expected in the bottom contact circle area, due to the existence of more intense relative motions between the ball indenter and the coating in this region. SEM photographs taken at the contact region in the top and the bottom imprint region after 10^5 impacts at an inclination angle of 15° verify the previous expectation (see Figure 8b). The scratches on the coating surface, associated to the relative motions of the ball to the coating surface, are more intense in the bottom imprint region, where the film is almost completely removed in comparison to the top crater area, where a smoother coating removal can be observed.

5. COATING COHESIVE FRACTURE INITIATION DURING THE INCLINED IMPACT TEST

In the case of a well-adherent coated specimen, the film fatigue endurance was determined by means of the perpendicular impact test [1,2,12]. The corresponding coating Woehler diagram is presented in Figure 9a.

(Ti$_{46}$Al$_{54}$)N, t=4 µm, K05-K20 (79 HRC), R$_a$~0.04 µm
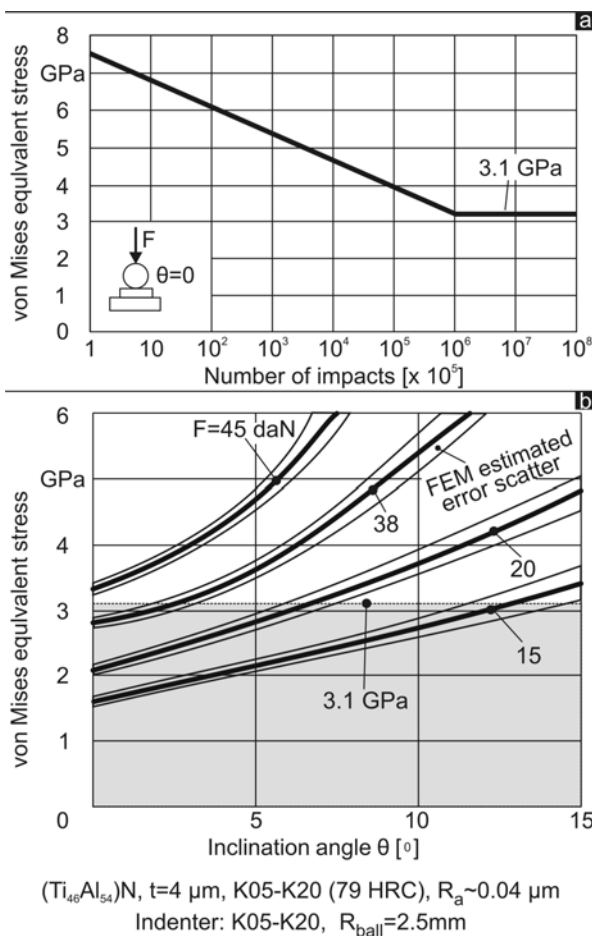Indenter: K05-K20, R$_{ball}$=2.5mm

Figure 9: Coating fatigue endurance limit determination and the occurring stresses during the inclined impact test.

According to this diagram, the coating fatigue stress limit amounts approximately to 3.1 GPa. The experimental procedure revealed that the coating can operate without damage after 1 million impacts below this fatigue endurance limit, corresponding to impact forces lower than 42 daN.

The occurring von Mises equivalent stresses during the impact test, at various impact forces versus the inclination angle, is presented in Figure 9b. These stresses were determined by means of the developed 3D FEM model simulating the inclined impact test. The steady increase of the occurring stress versus the inclination angle is more intense at higher impact loads. Hence, a cohesive coating failure in the inclined impact is expected to appear at lower impact loads is obvious, as already mentioned.

(Ti$_{46}$Al$_{54}$)N, t=4 µm, K05-K20 (79 HRC), R$_a$~0.04 µm
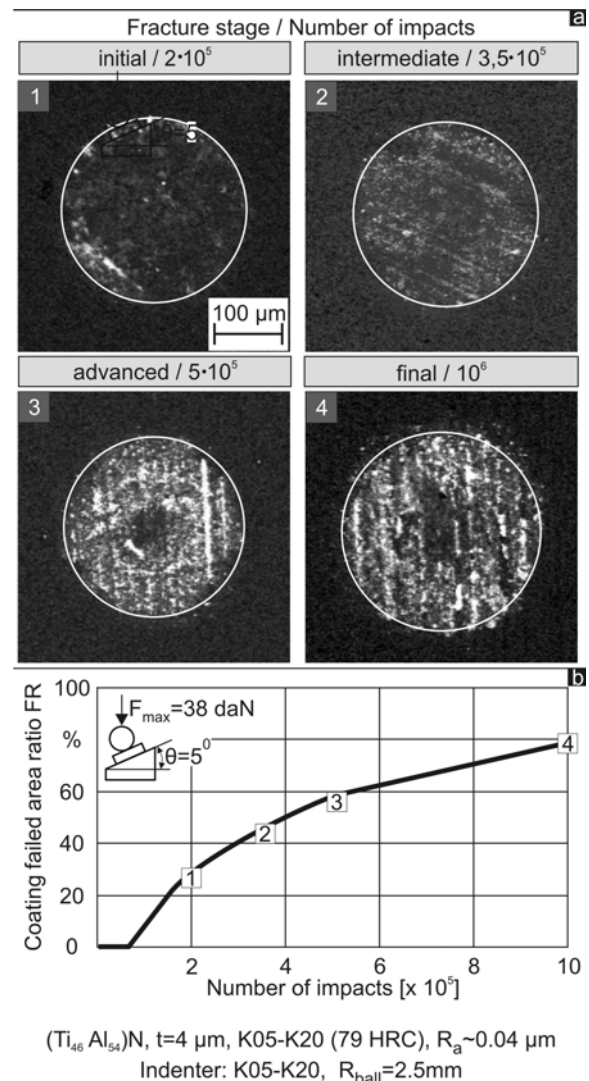Indenter: K05-K20, R$_{ball}$=2.5mm

Figure 10: Elaboration of a coating removal propagation diagram.

This speculation is verified by means of inclined impact tests at an inclination angle of 5 degrees. In the case of a well-adherent coating [6], the SEM

micrographs inserted in <u>Figure 10a</u> illustrate that, as expected, a coating fatigue fracture occurs after approximately 2×10^5 impacts and a film removal takes place after the coating fracture initiation, versus the number of impacts, already at an impact load of 38 daN. This load could not lead to a coating fracture after 106 impacts in the perpendicular impact test case, as already described. The film removal propagation after the first 2×105 impacts can be observed in the further micrographs of Figure 10a. By means of the "WEPROC" algorithm [12], the related curve shown in Figure 10b was elaborated, demonstrating the coating removal propagation within the imprint versus the number of impacts. Hereupon a 90% coating removal was reached after 1 million impacts.

Further similar curves were experimentally determined at higher impact loads and are illustrated in the diagram of <u>Figure 11</u>. These results revealed that the film removal propagation is more intense at higher impact forces, due to the higher occurring stresses according to the results shown in Figure 9b.
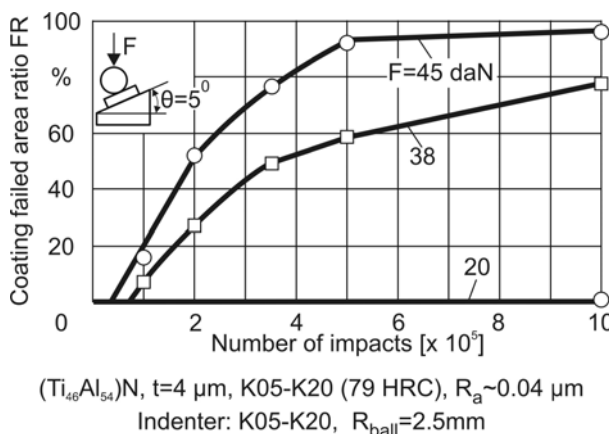

(Ti$_{46}$Al$_{54}$)N, t=4 μm, K05-K20 (79 HRC), R$_a$~0.04 μm
Indenter: K05-K20, R$_{ball}$=2.5mm

Figure 11: Comparison of the coating removal propagation for various impact loads, at an inclination angle of 5 degrees.

6. COATING ADHESION CHARACTERIZATION DURING THE INCLINED IMPACT TEST

As previously described, at higher inclination angles, the impact loads are increased and affect the coating fracture initiation and removal. <u>Figure 12</u> demonstrates the coating removal propagation during the impact test at an inclination angle of 10 degrees at a relatively low impact load of 20 daN, in the cases of a well- and a poor-adherent coating [6]. The well-adherent coating withstands much better the applied loads and has a lower removal propagation rate, compared to the poor-adherent

coating. This can be also observed in the SEM micrographs presented in the bottom figure part. In this way, a poor coating adhesion can be identified through a comparison of the removal rate in the inclined impact test to the corresponding one in the case of a well-adherent film.
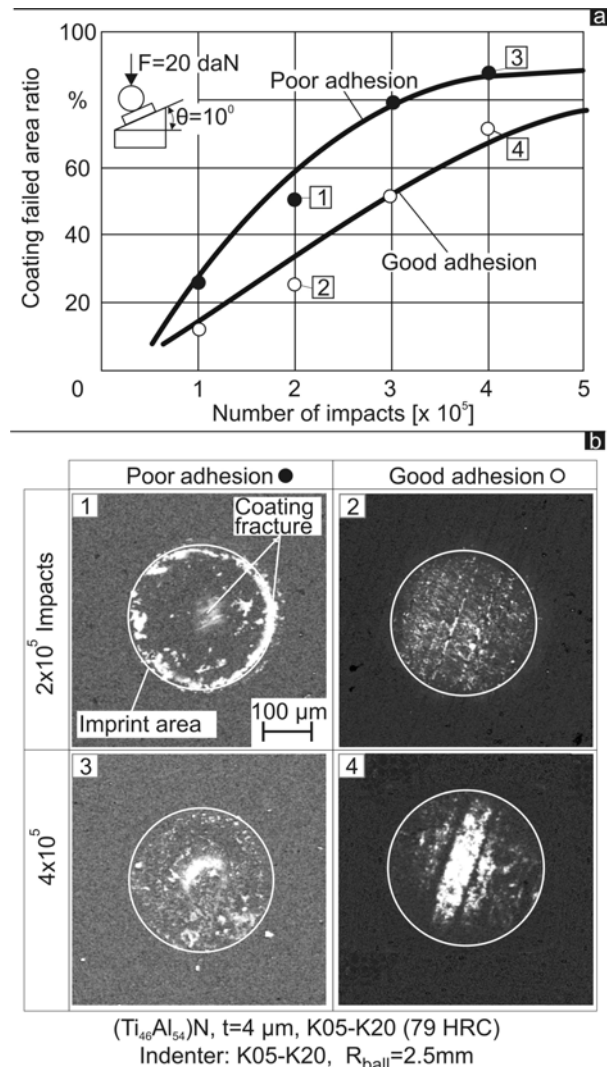

(Ti$_{46}$Al$_{54}$)N, t=4 μm, K05-K20 (79 HRC)
Indenter: K05-K20, R$_{ball}$=2.5mm

Figure 12: Comparison of the coating removal propagation for various impact loads, at an inclination angle of 5 degrees.

Such a comparative presentation of the coatings removal propagations, versus the number of impacts, at various inclination angles and impact loads, is conducted in the cases of a well- and a poor-adherent coating (see <u>Figure 13</u>). In the left figure part, the occurring coating removal propagation rates at an impact force of 20 daN in both coating adhesion cases, are presented. It is obvious that in all the examined inclination angles, there is a significant deviation in the courses of the coating removal propagations, with the poor-adherent coating having the worst behavior. In the right figure part, the corresponding coating wear propagations are presented at a higher impact force.

Due to the fact that this impact load leads to a maximum stress, higher than the coating endurance stress limit in both coating cases, a fast film fracture occurs already at an inclination angle of 5°, in accordance with the results demonstrated in Figure 10. However, in the poor adhesion coating case, the removal propagation is more intense. Herein, the Coating Impact Adhesion (CIA) can be used to quantify the extent of a poor-adherent coating removal, at a given inclination angle, impact force and number of impacts, compared to the corresponding coating removal of a well-adherent film. For example, in the case of an inclination angle of 15°, 20 daN impact force and 10^5 number of impacts, the CIA amounts to 40%, whereas in the case of the same inclination angle, however at higher impact force (38 daN) and lower number of impacts ($0.5x10^5$) to 50%.
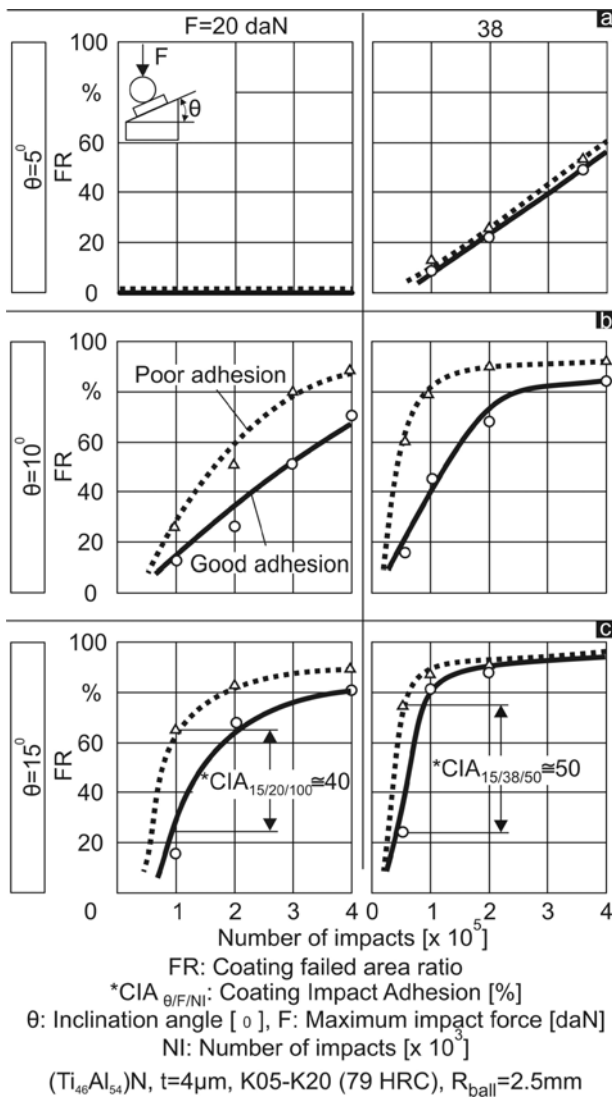

FR: Coating failed area ratio
*CIA $_{\theta/F/NI}$: Coating Impact Adhesion [%]
θ: Inclination angle [^{o}], F: Maximum impact force [daN]
NI: Number of impacts [x 10^3]

$(Ti_{46}Al_{54})N$, t=4µm, K05-K20 (79 HRC), R_{ball}=2.5mm

Figure 13: Comparison of the inclined impact performance of well-and poor-adherent coatings, at various impact loads and inclination angles.

7. CONCLUSIONS

The fatigue and especially the adhesion performance of well- and poor-adherent coatings were examined by means of the inclined impact test. Based on results deriving from the FEM simulation of the inclined impact test, the first coating failure and the subsequent film removal propagation in well- and poor-adherent coating cases were explained.

During the inclined impact test, coatings encounter a cohesive failure at lower impact loads compared to the perpendicular impact test, due to the occurring more intense stress field. The coating removal propagation within the impact imprint is faster, when testing poor-adherent coatings at high inclination angles. Through the introduced Coating Impact Adhesion (CIA) magnitude, the coating adhesion properties can be quantitatively characterized. In this way, the inclined impact test becomes an efficient method to evaluate coatings' cohesion and adhesion properties simultaneously.

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