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Impact Test, an Innovative Technique to Characterize Various Coating Properties

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Abstract

The main research scope of the impact tester is the characterization of coatings' cohesive and adhesive properties, in dry or lubricated environment, in room or elevated temperatures. The coating evaluation can also be correlated with other experimental procedures, such as cutting (milling, turning). This experimental method is supported by developed FEM simulations, which consider the mechanical elastic-plastic properties of all involved materials and predicts the coating failure under various test conditions.

Key words: *Impact test, PVD coatings, fatigue, adhesion, micro-abrasion*

Introduction

The developed impact tester is presented in figure 1. The perpendicular impact test has been applied successfully for the characterization of coatings fatigue properties [1-5]. 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 arrangement. The output voltage is fully controllable, with the aid of a variable transformer, in order to achieve the various impact forces. 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, cooling air pressure and coil temperature during the impact test.

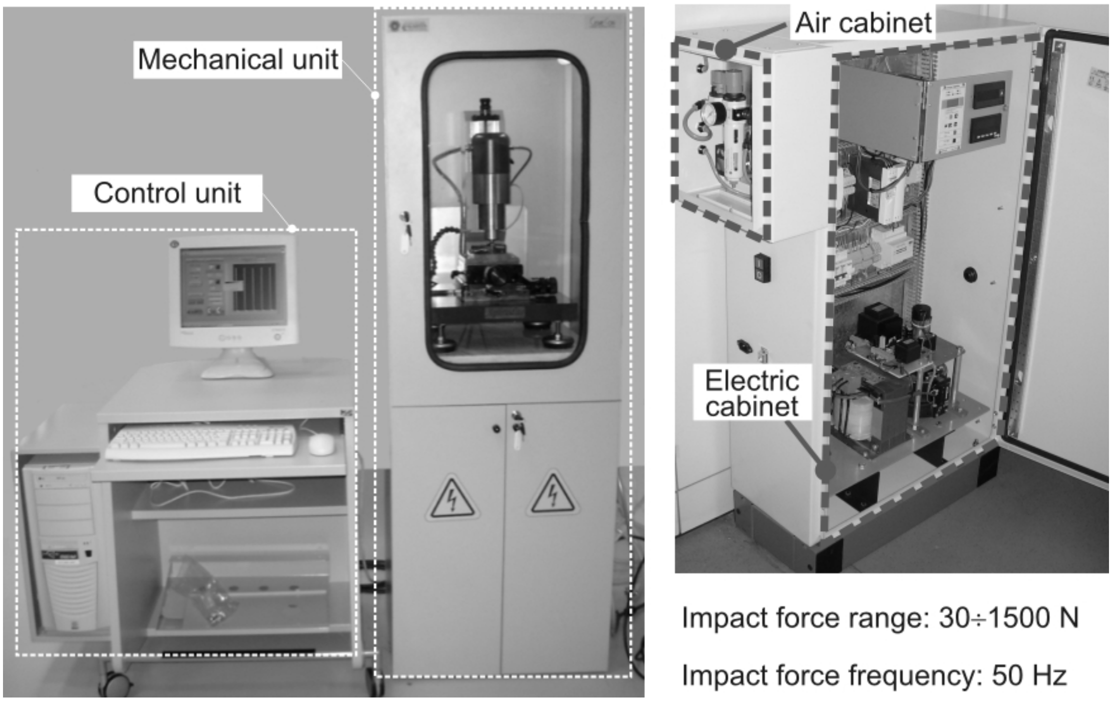


Fig. 1. The developed impact tester

The perpendicular impact test to predict coatings' fatigue behaviour

During the impact test a ball indenter penetrates periodically into the coating under a desired maximum load. Depending on the impact load and on the number of impacts, a coating failure may occur. Through the impact testing of a specimen at various loads and numbers of impacts, a Woehler-like diagram can be developed, as illustrated in figure 2. The horizontal region of the curve is associated with the coating continuous fatigue endurance.

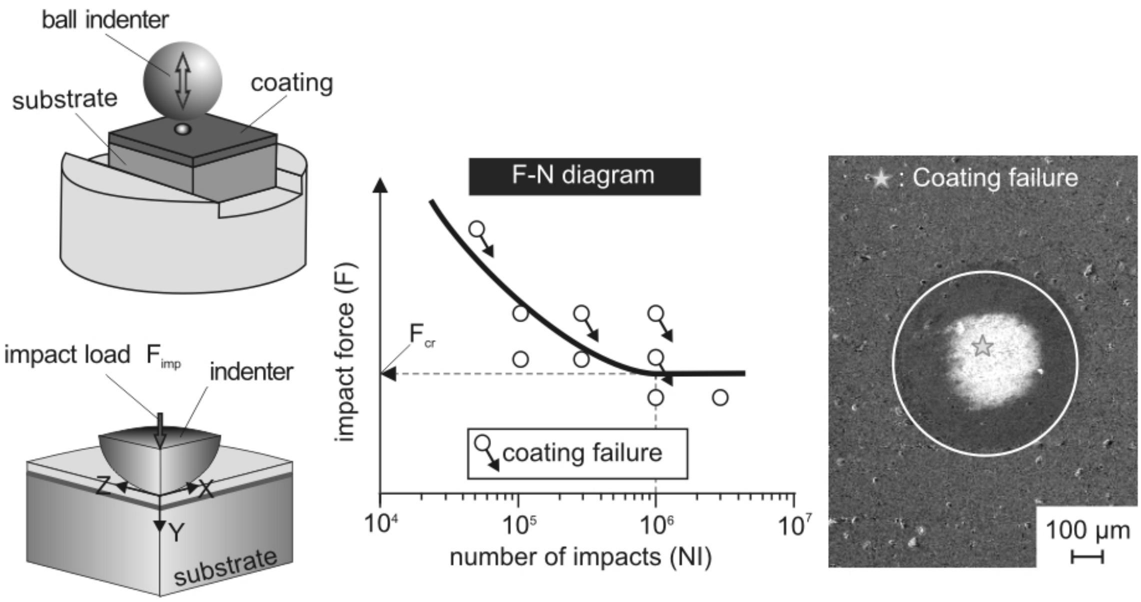


Fig.2. The perpendicular impact test; fatigue critical impact force determination

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 3). The failed area ratio FR of an impact test imprint at a specified impact force and number of impacts is determined through color analysis of the occurring imprint SEM micrograph, by appropriate software. In this way, diagrams of the coating failed area ratio FR development versus the number of impacts can be determined, as shown in figure 3.

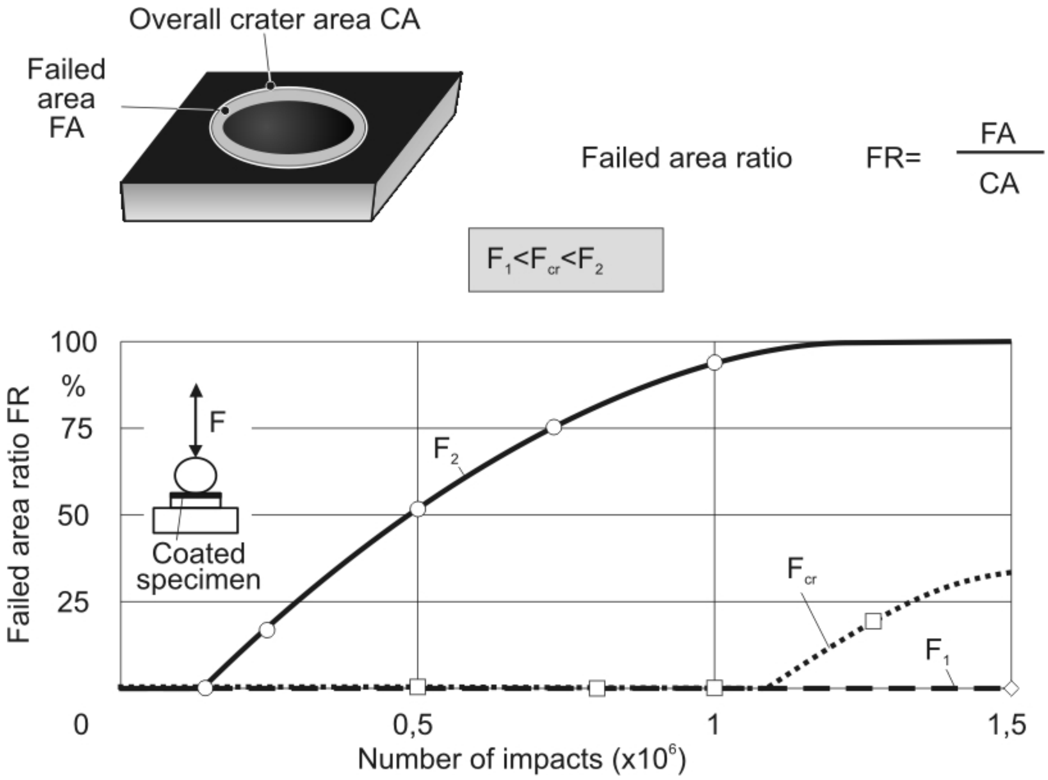


Fig. 3. Advanced impact test results registration through the coating failed area ratio versus the number of impacts

The impact tester is supplied with a fully automated software named “ITEC”, which enables the determination of coatings fatigue properties in form of Smith and Woehler diagrams (see figure 4), based on the FEM simulation of the impact test. In the demonstrated example, a characteristic cutting tools’ $(Ti_{46}Al_{54})N$ coating was applied. The stress-strain data of this PVD film, as well as those of the cemented carbide substrate, are presented in the top part of this figure. Additional data regarding the coating deposition process and the substrate pretreatment are also provided in this figure, together with the perpendicular impact test FEM simulation model. Appropriate contact elements with variable stiffnesses cs_x, cs_y in normal and tangential directions respectively, were applied, to take into account the coating adhesion [6]. The contact stiffness was quantified according to procedures described in this paper [7], and corresponds to stiffness of a well-adherent coating on a ground and micro-blasted substrate. With the aid of calculations based on this FEM model, the developed coating stresses, during the loading and unloading test stages were obtained (see top-right part of the figure).

The film damage, due to fatigue, starts at the intensively stressed area near the vicinity of the formed imprint, as displayed in the diagram in the bottom-right part of the figure. Taking into account these results, the coating Smith and Woehler diagrams, illustrated in the bottom-left part of figure 4, are then developed. The coating fatigue endurance limit of the applied film, amounts approximately to 2.8 GPa, which corresponds to an impact load of 400 N.

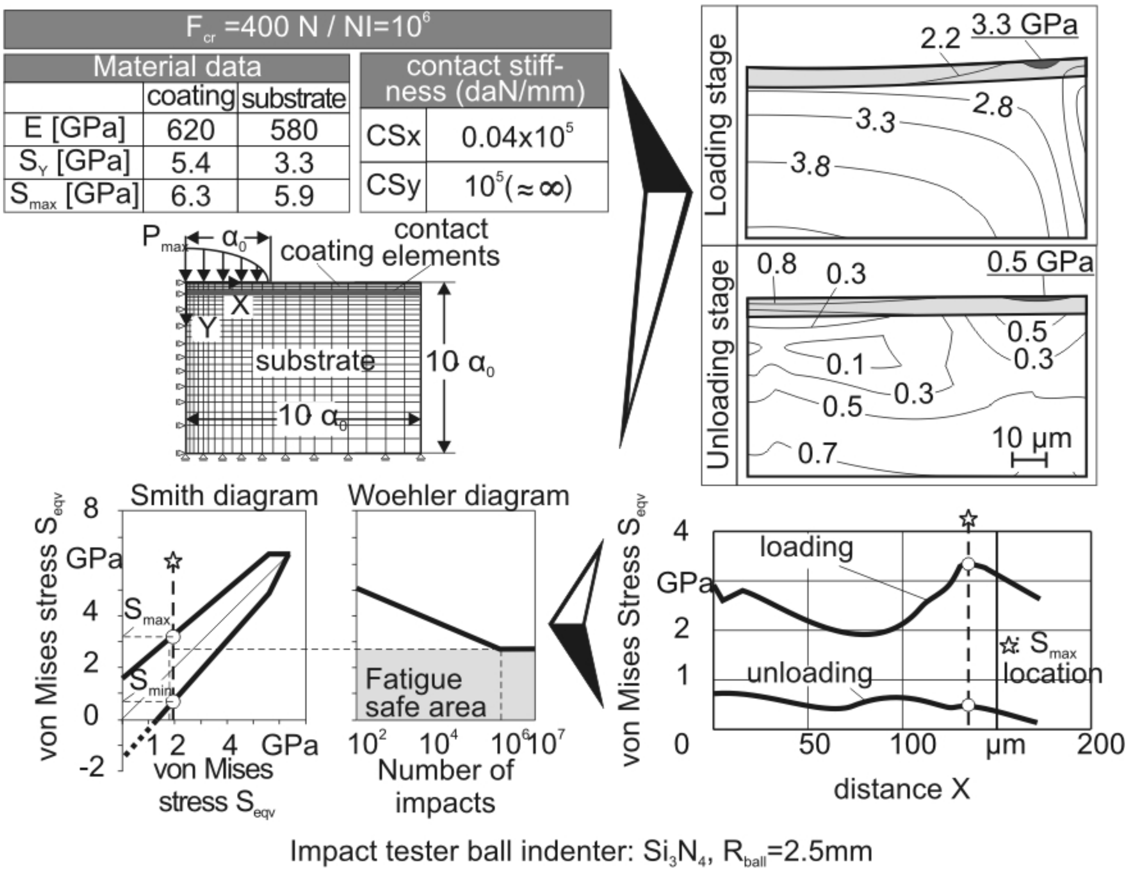


Fig. 4. Determination of film's Smith and Woehler diagrams through a FEM-based simulation of the contact between ball indenter and coated specimen

The SEM-micrographs of the occurring imprints at various impact loads and numbers of impacts are illustrated in figure 5. The coating is almost removed at an impact load of 430 N after one million (10^6) impacts. This load is higher than the corresponding fatigue endurance critical load of approximately 400 N, as shown in the Woehler diagram of the figure. Moreover, restricted coating fractures can be observed at an impact load of 400 N, equal to the fatigue endurance limit, located in the overstressed area near the crater vicinity. The film damages can be detected by the illustrated EDX-microanalysis. In the locations with film fractures, elements of the cemented carbides substrate as W and Co were detected. The coating withstands a theoretically infinite number of impacts, when it is stressed under the fatigue endurance critical stress, amounting to 2.8 GPa. As it can be seen in the corresponding SEM micrograph of figure 5, at a load of 300 N, corresponding to a maximum equivalent stress of ca. 2.2 GPa, no film damage occurs after twenty million impacts (see detail A of figure 5).

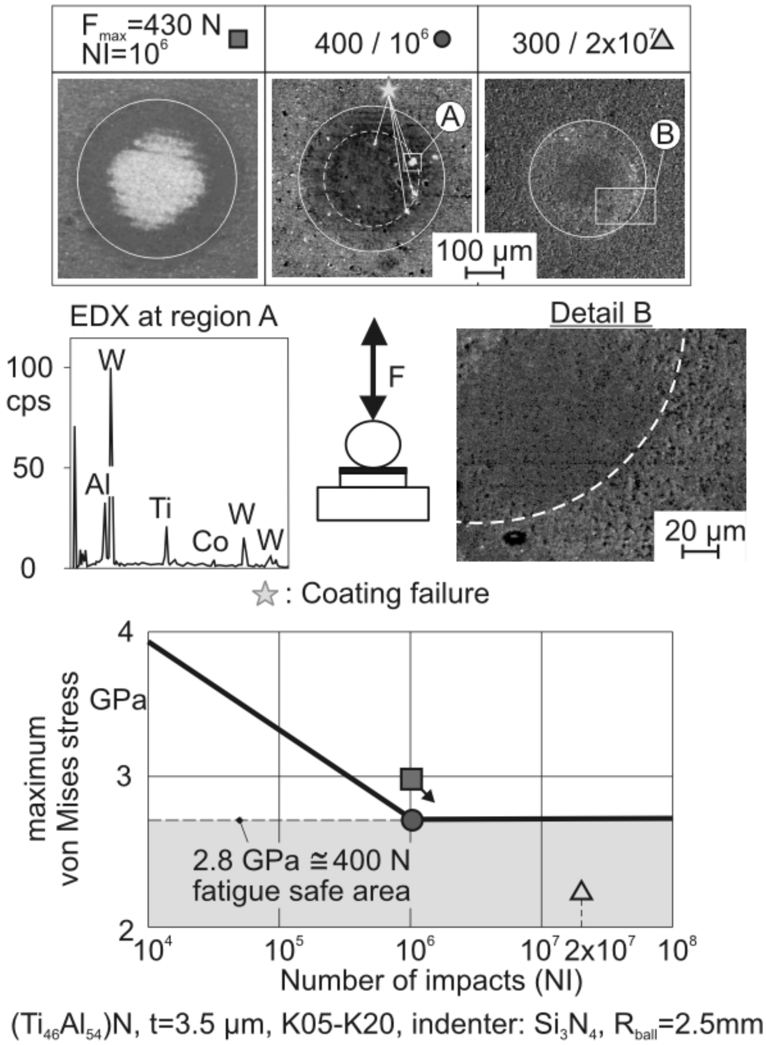


Fig. 5. Verification of a determined film Woehler diagram

The inclined impact test to evaluate coating adhesion

During the inclined impact test, the coated surfaces are loaded vertically and tangentially simultaneously. 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 adhesive coating strength [7-9].

A corresponding example is illustrated in the case of the investigation of the effect of various substrate treatments on the film adhesion. For this reason, ground, polished and micro-blasted substrates were applied, all coated with the same PVD film. All samples were tested by means of the inclined impact test. The obtained results displayed in figure 6 exhibit that the applied polished and subsequently micro-blasted well-adherent coating is overloaded concerning fatigue, at an inclination angle of 15^0 and at impact forces larger than ca. 140 N, associated with the fatigue critical stress of 2.8 GPa. These results were provided by FEM calculations of the developed stress field in the imprint area during the inclined impact test, as described in the following section. As expected, the coating withstands one million impacts, i.e. it possesses fatigue endurance, at loads less than approximately 140 N and it is damaged earlier, i.e. it possesses a time-limited resistance against fatigue at higher impact forces. At impact forces greater than 140 N the maximum equivalent stresses in the contact region between the ball indenter and the coated specimen exceeds the fatigue critical stress of 2.8 GPa and the film damage development is accelerated by the impact load growth. The fracture ratio FR, for instance, at an impact force of 200 N, amounts to approximately 15% after $0{,}2\text{x}10^6$ impacts, whereas after the same number of impacts, at a load of 300 N it amounts to ca. 75%; this is further displayed by the corresponding curves in figure 6. Taking into account these dependencies, the fatigue over-critical force of 200 N was selected for an efficient monitoring of the film damage development and for short test durations, i.e. up to two hundred thousand impacts. In this way, the expected coating failure ratios in the worse substrate adhesion cases is larger than 15% after two hundred thousand impacts and the film damage start and development can be detected in a short time easily and registered accurately, even in the worst polished substrate case (p), where the film fracture rate is accelerated, as it will be demonstrated.

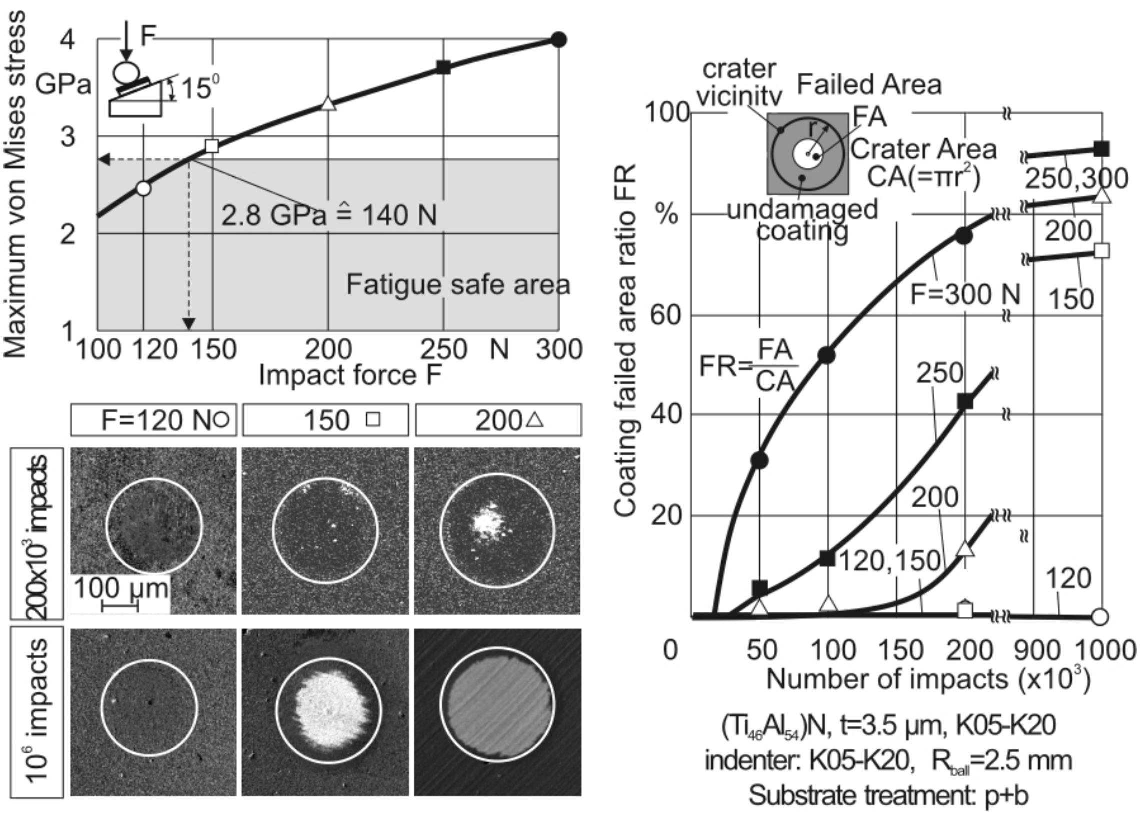
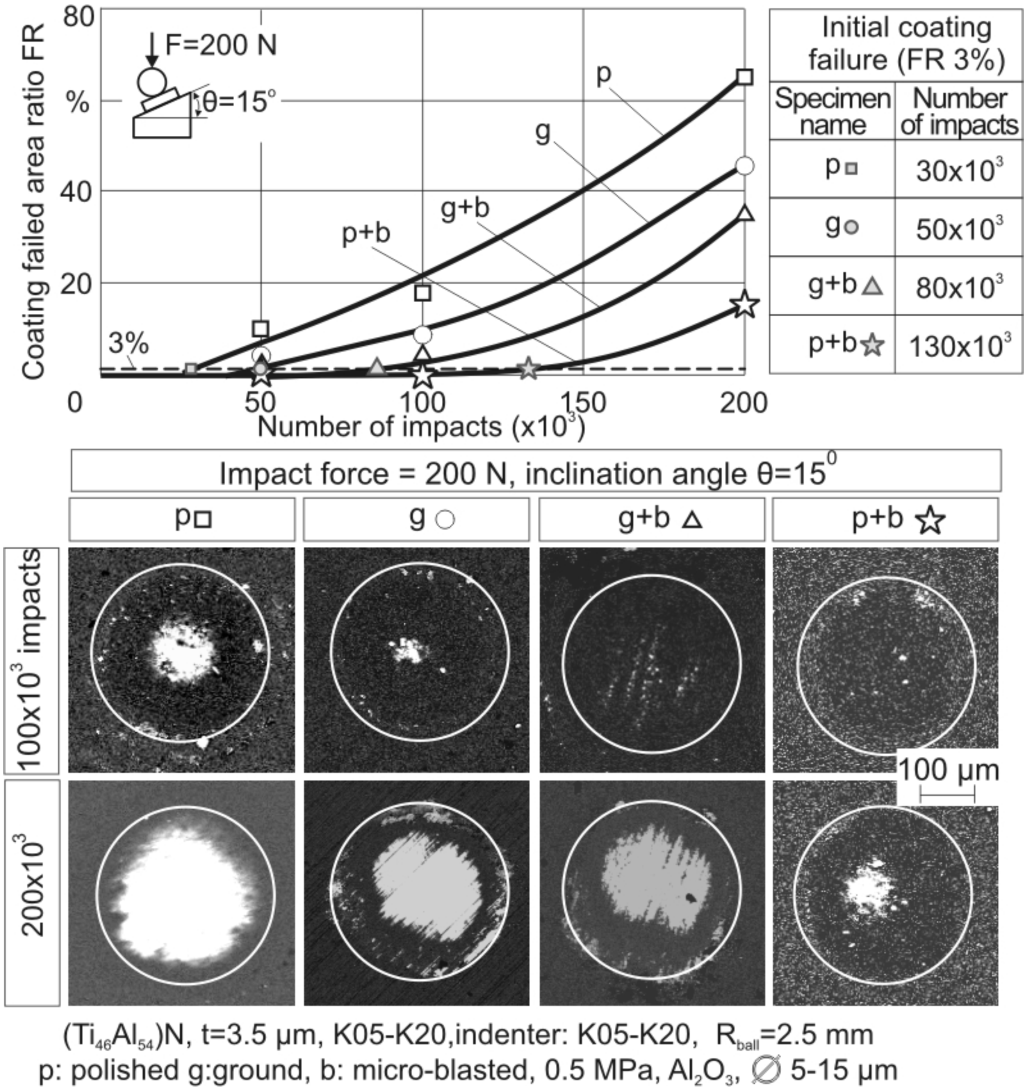


Fig. 6. Maximum von Mises stress occurring during the inclined impact test at various impact loads, corresponding SEM micrographs and coating failed area ratio at various impact forces versus the number of impacts

Quick coating adhesion characterization

The coating failed region versus the number of impacts, during the inclined impact tests, in the various substrate treatment cases, is monitored in figure 7. The coated inserts, subjected to substrate micro-blasting, withstand more effectively the applied loads in comparison to the unblasted inserts and have a slower coating failed area ratio FR increase. Moreover, the failure of a coating on a poor-adherent polished insert possesses the most intense development due to restricted film-substrate mechanical interlocking; however, after two hundred thousand impacts, was not removed totally. In this way, even in this insufficient adhesion case, the film damage start-up and its intense removal rate can be conveniently registered and evaluated. The coating fracture initiation is encountered in all substrate cases after approximately the number of impacts indicated in the table of figure 7, depending on the substrate superficial mechanical treatment. Hereupon, a coating failed area ratio FR larger than 3% was used as the criterion of the film damage start-up. Considering these results, it is obvious that the appropriate selection of the fatigue over-critical load of 200 N facilitated the monitoring of the film failure start and development, with sufficient accuracy, in all investigated cases.



Initial coating failure (FR 3%)	
Specimen name	Number of impacts
p □	$30x10^3$
g ○	$50x10^3$
g+b △	$80x10^3$
p+b ☆	$130x10^3$

Fig. 7. Film impact resistance due to various coating-substrate adhesion, induced by different substrate superficial treatments

The magnitudes of the divergences between these various film-substrate adhesion stages and that one of the well-adherent polished and micro-blasted insert, which is considered as reference, are illustrated in figure 8. Hereupon, the coating impact adhesion CIA was used to characterize the previously mentioned divergence, at given inclination angle, impact force and number of impacts. The CIA amounts to 20% for the ground and micro-blasted substrate, compared to the reference one. The failed area ratio FR increases up to 30%, in the case of a ground substrate, compared to the reference one, and up to 50% in the case of a polished substrate. In this way, the adhesion strength can be qualitatively characterized [8].

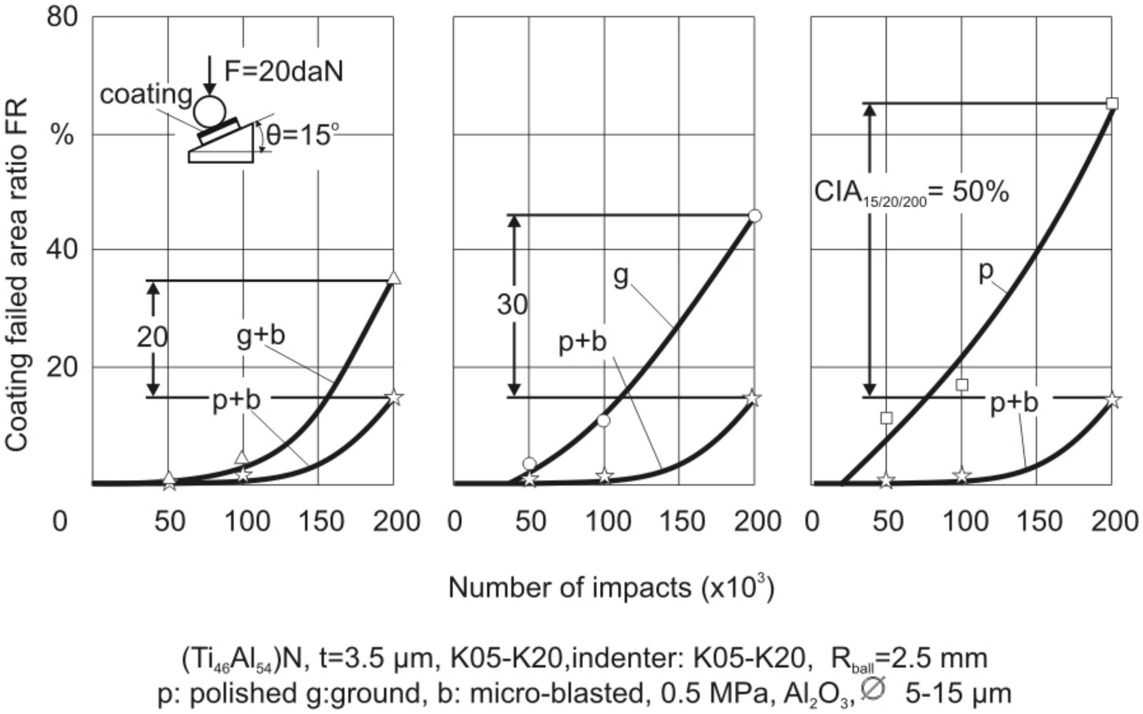


Fig. 8. Fast coating adhesion characterization by means of the CIA metric

Quantification of coating adhesion

The adhesion strength can also be quantitatively characterized through a developed FEM simulation of the inclined impact test, exhibited in figure 9. At first, a three-dimensional model

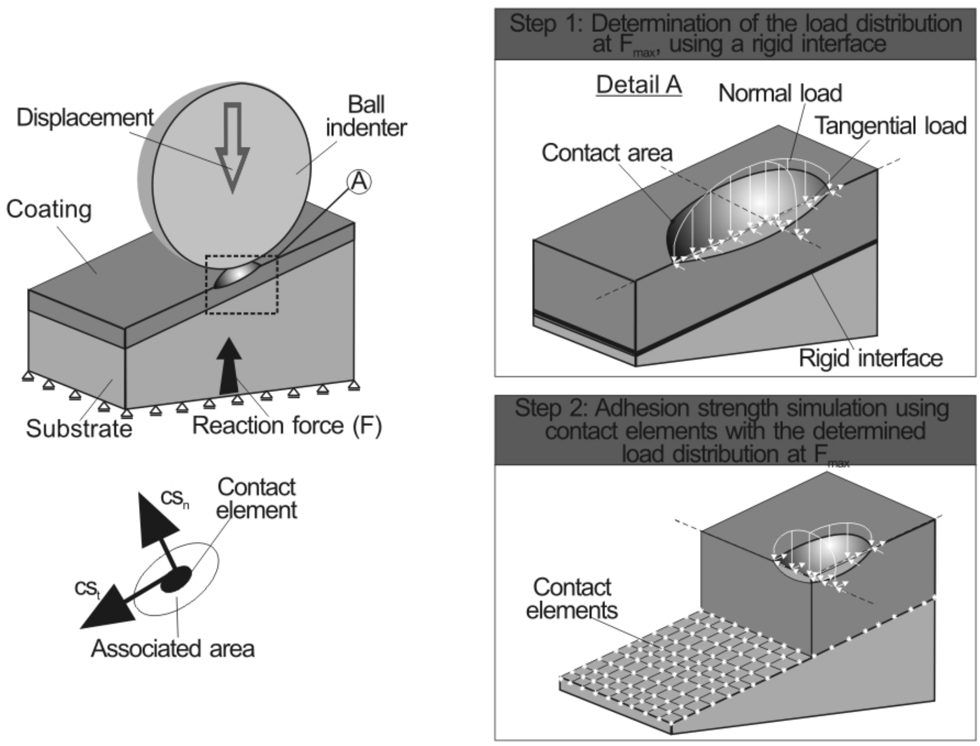


Fig. 9. The inclined impact test FEM simulation of the film-substrate interface

was created, with a rigid film-substrate interface, considering the mechanical elastic-plastic properties of the coating, the substrate, and of the ball indenter. Using this model, the developed contact loads between the ball and the coated surface were determined. Moreover, these data were further applied in a similar FEM model, with suitable contact elements between the coating and substrate surfaces, as shown in figure 9. The applied contact elements are characterized by the normal cs_n and the tangential cs_t contact stiffness. The same PVD coating, deposited on various adherent substrates, was simulated with the aid of the developed FEM models of the inclined impact test, using these contact elements to describe the normal and tangential film-substrate interface stiffness. Hereupon, the ratio CSR of the tangential contact stiffness cs_t, to the normal one cs_n, was applied to characterize the adhesion strength in the coating-substrate region [10].

The maximum von Mises equivalent stresses versus the contact stiffness ratio, developed during the inclined impact test, are shown in figure 10, in the case of an impact force of 200 N, at inclination angles of 0^0 and 15^0. The FEM calculations were conducted for two adhesion quality stages, an ideal one with contact stiffness ratio CSR equal to 1 and an insufficient one, concerning adhesion, with CSR equal to 0.01. As can be seen in figure 10, the contact stiffness ratio has no effect on the developed maximum equivalent stress in the perpendicular impact test. On the other hand, a slight displacement of the maximum equivalent stress appears toward the imprint center, in the case of a poor adhesion (CSR=0.01). Furthermore, during the inclined impact test, the decrease of the contact stiffness ratio, i.e. the film adhesion deterioration, results in a nonlinear growth of the maximum equivalent stress. The stress augmentation is more intensive as the coated surface inclination angle increases. These results indicate that at the same impact force, the film adhesion affects the developed maximum stress during the inclined impact test and therefore the film damage, as already experimentally found.

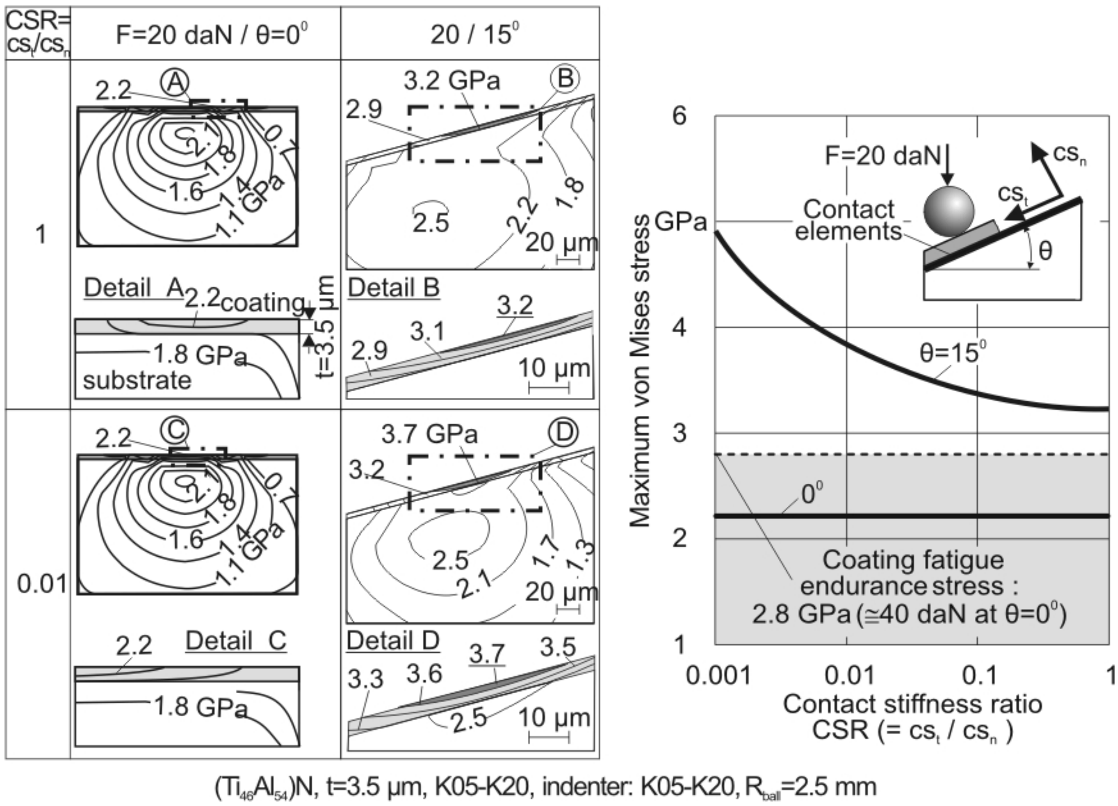
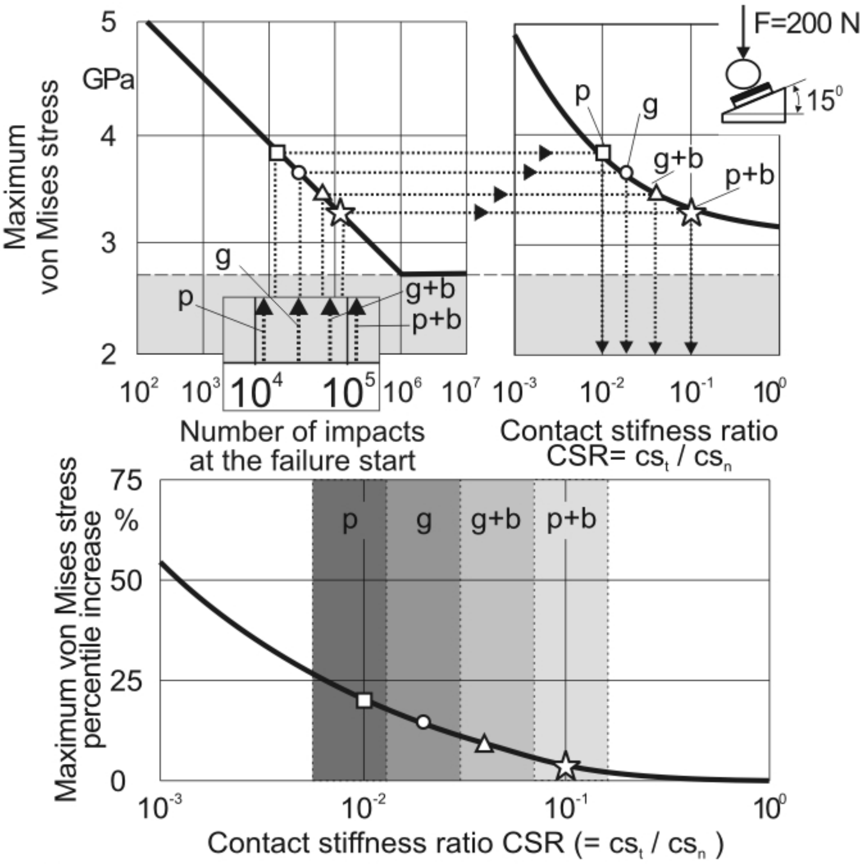


Fig. 10. FEM determined equivalent stress distributions during the perpendicular and the inclined impact test and the effect of he contact stiffness ratio CSR on the maximum equivalent stress in an ideal and a poor film-substrate adhesion cases

With the aid of the contact stiffness ratio CSR, it was possible to quantify the contribution of the substrate treatments to the coating adhesion strength by applying the following method. Herein, the maximum equivalent stress during the inclined impact test, in the case of an inclination angle of 15^0 and at 200 N impact load, was used to quantify the contribution of the substrate treatments to the coating fatigue failure and thus, to the contact stiffness ratio, as exhibited in figure 11. The maximum equivalent stress, leading to the film fatigue fracture start, was determined as demonstrated in the figure. The number of impacts associated with the film failure start was detected experimentally for each substrate treatment case and were correlated with the related film Woehler diagram Further, the equivalent stress versus the contact stiffness ratio diagram was applied to quantify the contact stiffness ratio.



substrate treatment	p □	g ○	g+b △	p+b ☆
CSR	0.01	0.02	0.04	0.1

$(Ti_{46}Al_{54})N$, t=3.5 μm, K05-K20, indenter: K05-K20, R_{ball}=2.5 mm
p: polished g: ground, b: micro-blasted, 0.5 MPa, Al_2O_3, ∅ 5-15 μm

Fig. 11. Adhesion quantification by means of the contact stiffness ratio CSR in various substrate treatment cases

According to these results, coatings on poor-adherent substrates are loaded more intensely, in comparison to the corresponding well-adherent films loading cases, when also tangential loads are exercised to the film-substrate interface. A contact stiffness ratio CSR ca. 0.1, implemented by the polishing and additionally micro-blasting of the applied cemented carbide substrate, close to the one of the ideal-adherent film-substrate interface, causes an overloading only of approximately 3% (see figure 11). On the other hand, in the poor adhesion polished substrate case with CSR less than 0.01, overloadings larger than 20% can occur. For instance, at a CSR of

0.001 a 53% overloading develops. In this way, in FEM simulations of coated components, the contribution of the coating adhesion on the film loadings can be described by means of contact elements, possessing stiffness data, determined with the aid of the introduced procedures. These data can also be used in various applications, for instance, in the FEM simulation of the loaded cutting edge, to capture the adhesion effect on the wear propagation of coated tools in milling.

Correlation among cutting performance, coating impact resistance and adhesion

The coating adhesion improvement leads to a significant cutting performance increase, as displayed in figure 12. The shown results were achieved through milling investigations, using cemented carbide inserts coated with the same PVD film. The coated inserts with ground and micro-blasted substrates reach a tool life of approximately $55x10^3$ cuts, at a flank wear width of 0.2 mm. Moreover, the results exhibit an additional increase in wear resistance, by means of polishing and subsequent micro-blasting of the substrate. On the other hand, inserts with polished or ground substrates managed to cut only ca. $28x10^3$ and $35x10^3$ times respectively, up to the same flank wear width.

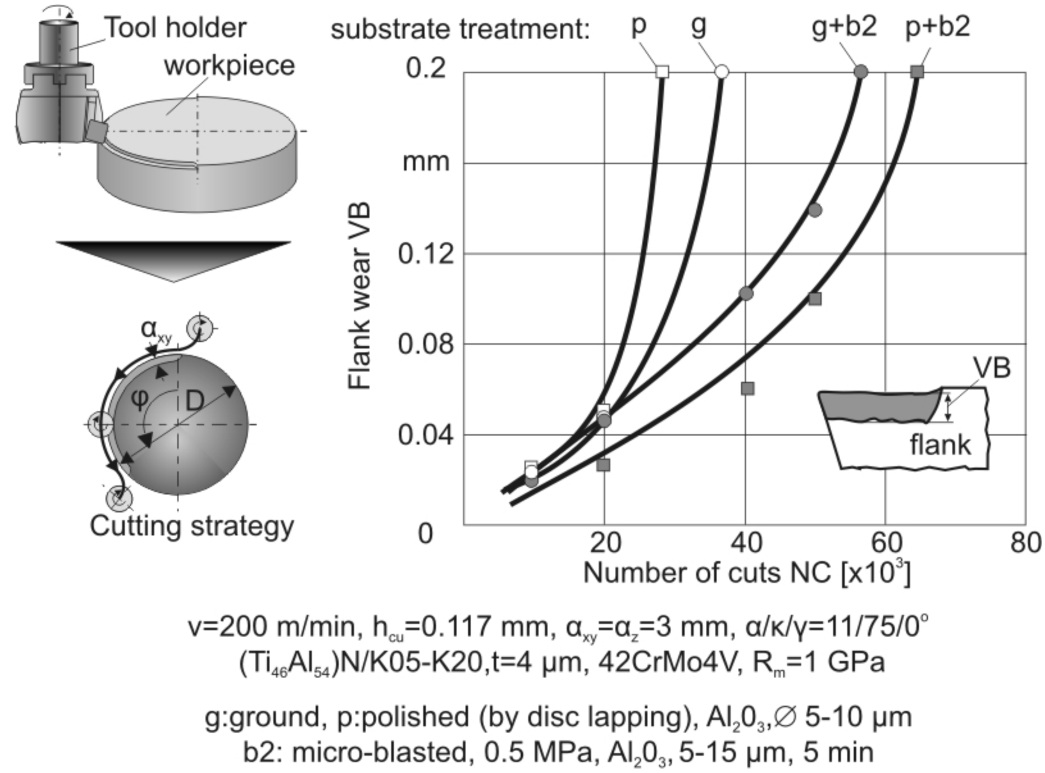


Fig. 12. Flank wear development in various substrate mechanical treatment cases

The potential to quantify the occurring coating adhesion in each substrate treatment case, allows the correlation among the coating inclined impact resistance, the number of cuts up to a flank wear of 0.2 mm and the contact stiffness ratio CSR, as demonstrated in figure 13. It is quite impressive that there is an almost identical behaviour of the coating cutting performance with the inclined impact test resistance, expressed by the remaining area ratio (1-FR) and with the contact stiffness ratio in all the substrate treatment cases [11].

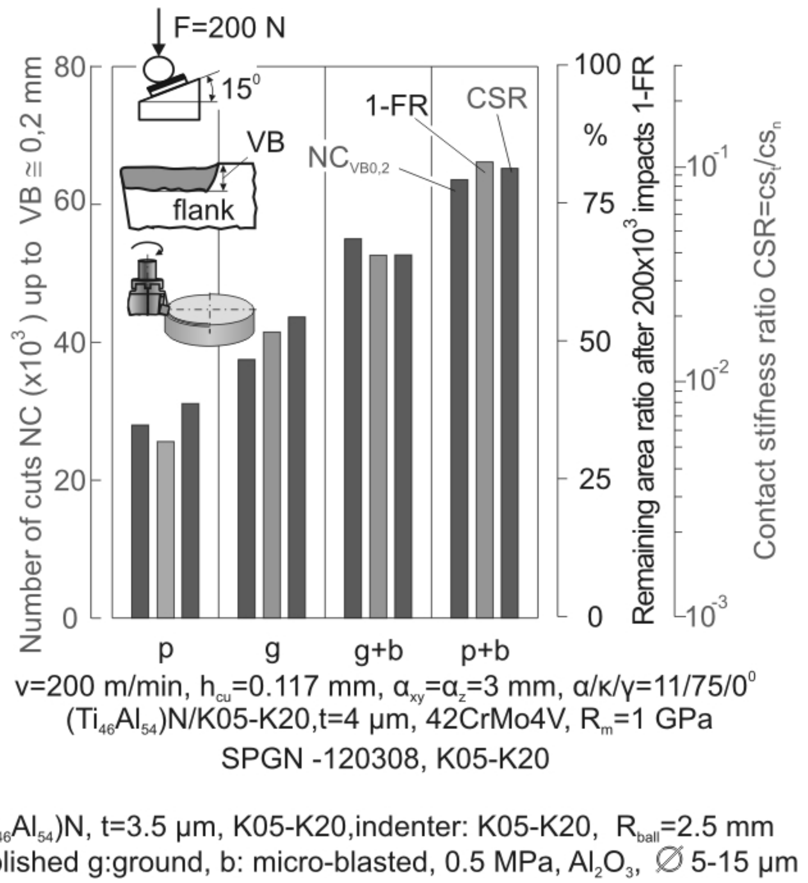


Fig. 13. Correlation among the milling performance, the remaining area ratio (inclined impact resistance) and the contact stiffness ratio CSR in various substrate treatment cases

Coating micro-abrasion evaluation

The inclined impact test is a very efficient method for detecting coating resistance against impact micro abrasion, with or without lubrication and according to the test conditions for the synchronous contribution of fatigue phenomena to the film wear. The abrasive wear of a well-adherent $(Ti_{46}Al_{54})N$ coating was investigated by the inclined impact test, at impact loads higher, equal or lower than the fatigue critical impact load of the applied film. These investigations were conducted for few thousands up to several millions of impacts both under dry and lubricated conditions.

The experimental results exhibited in figure 14 indicate that the coating reaches its fatigue endurance, at an inclination angle of 15^0 and at impact forces less than approximately 140 N, since up to one million impacts at the applied loads of 140 and 80 N no film damage occurs. Furthermore, a film failure appears after only half a million impacts, at the fatigue over-critical impact force of 200 N. On the other hand, the coating is damaged at both the loads of 140 N and 80 N, over one million impacts, due to abrasion mechanisms. The coating fails in its central imprint region, due to excessive relative micro motions, between ball and film surface. In Detail A of figure 14, it can be observed that at the under-critical impact load of 80 N, the film micro abrasion led to a roughness decrease and a restricted film removal in the central imprint area, after two million impacts [12].

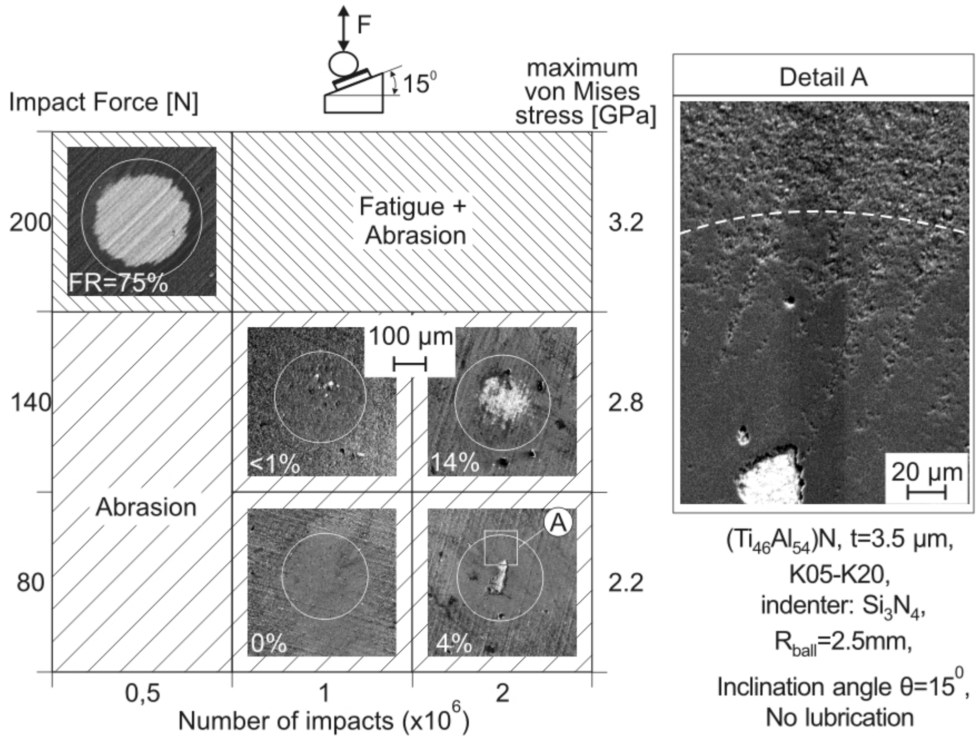


Fig. 14. Coating removal due to fatigue or abrasion at test conditions without lubrication

The coating failure at over- and under-critical loads is further explained through the analysis of figure 15. For an impact load of 80 N, lower than the fatigue critical load, the previously mentioned restricted coating failure develops approximately in the center of the imprint after

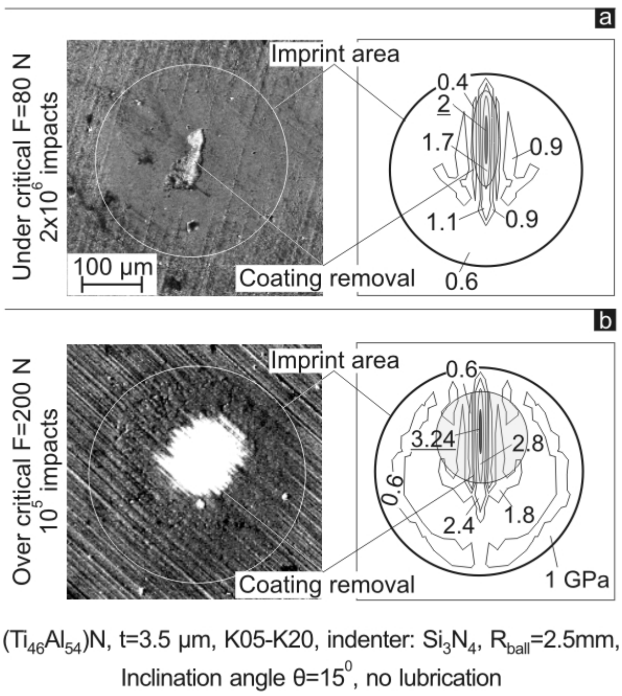


Fig. 15. Developed imprints and superficial stress fields during the inclined impact test at over- and under-critical impact loads

two million impacts (see figure 15a). In this case, the coating is slowly removed due to low-rate developing micro abrasion effects. This coating failure, as expected, appears near the center of the imprint, since at this location the corresponding superficial stresses possess their maximum, as the related FEM calculation clearly demonstrates.

On the other hand, at the over-critical load of 200 N, after one hundred thousand impacts, the coating is removed due to fatigue failure (see figure 15b). The first damage appears in the overstressed central imprint region, where a local stress of 3.24 GPa is developed, exceeding the fatigue critical one of 2.8 GPa, as ascertained by the corresponding FEM calculation of the superficial stresses. Furthermore, due to the restricted number of impacts, the film micro abrasion is negligible and the roughness in the imprint remains unaffected.

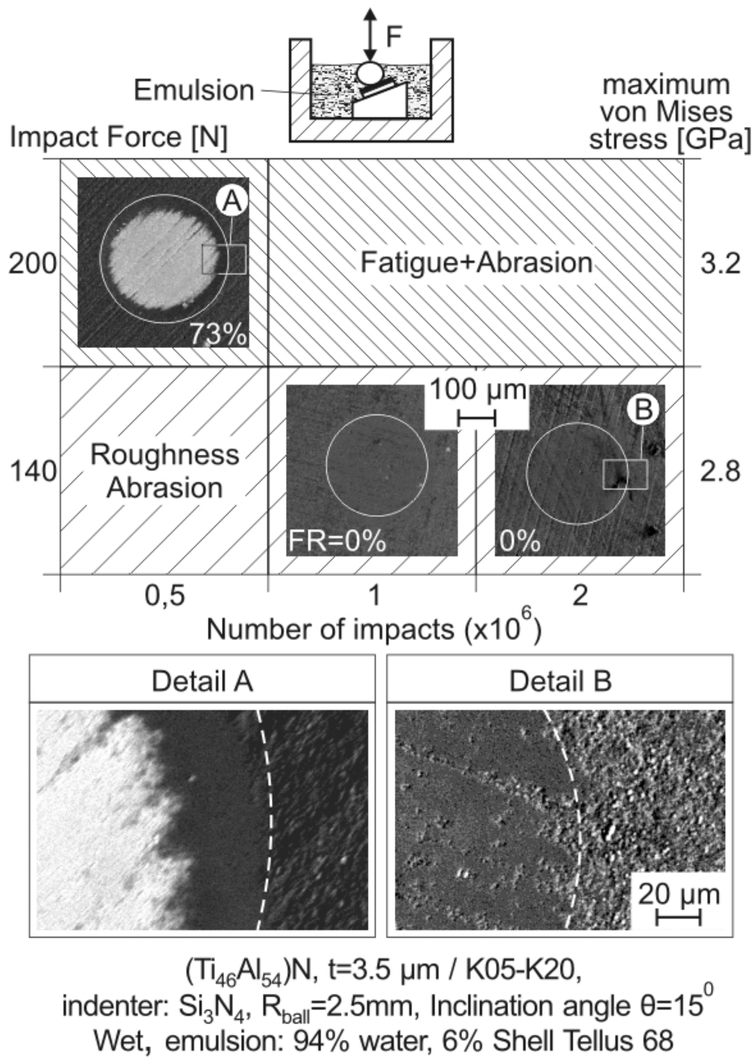


Fig. 16. Coating removal due to fatigue or abrasion a lubricated test conditions

Additional inclined impact tests were conducted under lubricated conditions. The coated specimen was placed in emulsion, used in cutting processes. The corresponding results, as well as the emulsion data are exhibited in the right part of figure 16. As expected, the coating fails due to fatigue at an impact load of 200 N, higher than the fatigue critical load, after half a million impacts (see Detail B). At impact loads equal and lower than the fatigue critical one of 140 N for over one as well as two million impacts, no film failure develops (see Detail C). In the latter cases, only roughness peaks are removed and the roughness in the imprint is decreased. This can be explained by the development of hydrodynamic pressure between the ball and the coating that prevents the direct contact between the surfaces, hence, reducing frictions and eliminating the abrasion mechanisms. In this way, no film failure occurs at impact loads equal or lower than the fatigue critical loads.

An overview of the conducted inclined impact tests, under various loads, number of impacts and dry or lubricated conditions is presented in figure 17. The film failure extent in the imprint is described through the failed area ratio FR. In the upper left part of the figure, at impact loads higher than the fatigue critical load, the failed area ratio growth is decelerated by the lubrication, as the coating removal due to micro abrasion is decreased. On the other hand, at impact loads equal or lower than the fatigue endurance critical load of 140 N, the coating failure mechanism, due to micro abrasion, is eliminated up to one million impacts, under dry or lubricated conditions. Finally, if lubricant is applied, at loads equal or lower than the film fatigue critical load, the coating withstands a theoretically infinite number of impacts, as in the case of the under-critical perpendicular impact test without lubrication [12].

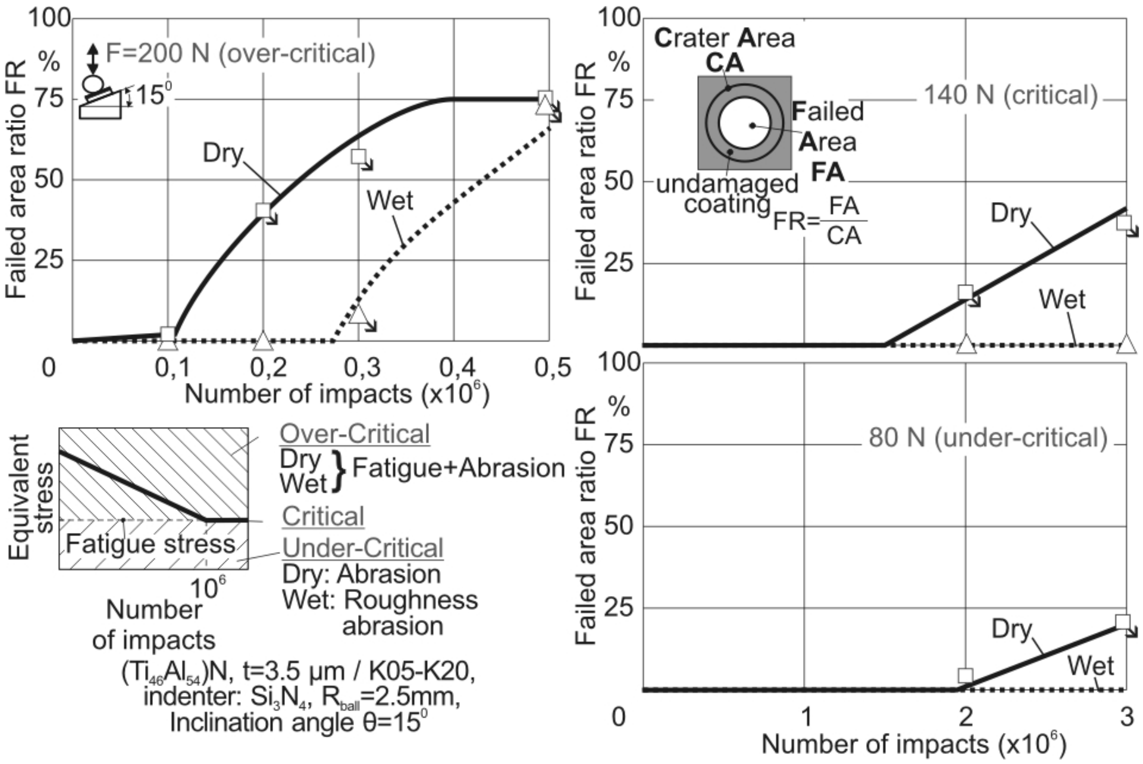


Fig. 17. Failed area ratio FR versus the number of impacts at various impact loads, under dry and lubricated conditions

Coating impact resistance at elevated temperatures

The impact tests at elevated temperatures were carried out in a special device developed for this purpose. The developed experimental set-up is presented in figure 18. The specimen heating device, as well as the special compressed air heating device, warms up and supplies the air to the impact contact area, in order to remove fragments of the ball indenter or the coating, whereas the extra heating maintains the specimen temperature to the desired level.

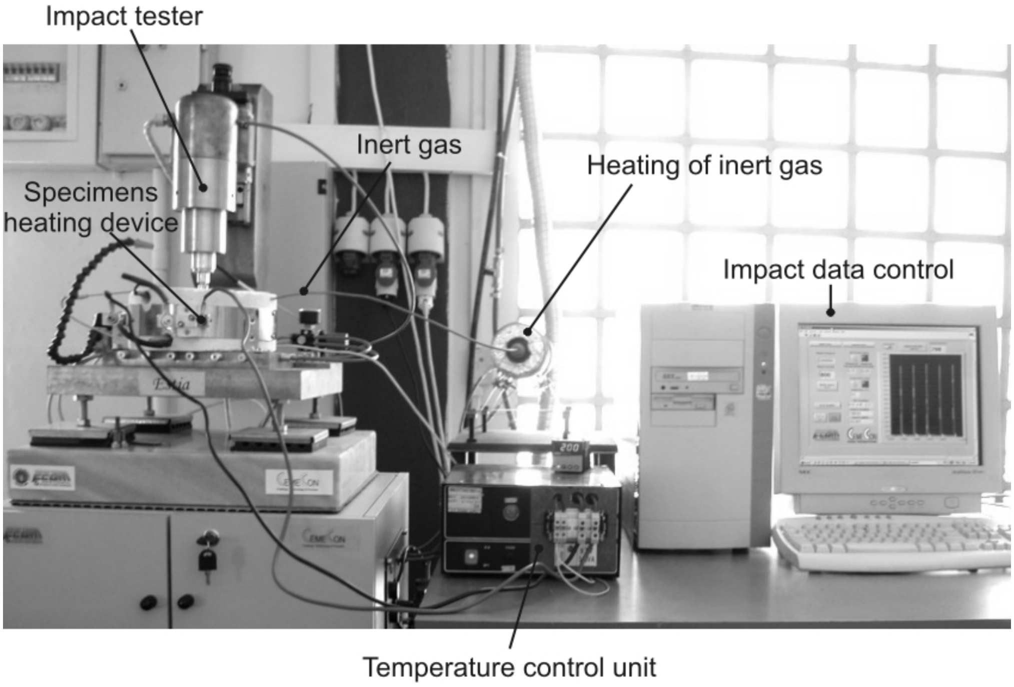


Fig. 18. The developed impact test set-up operating at elevated temperatures

The coating wear propagation during the impact test was studied at operational temperatures up to 600 ^{0}C and it was monitored in terms of the coating fracture ratio (FR) versus the applied impact force F (see figure 19). Each plotted point corresponds to test duration of 10^6 impacts.

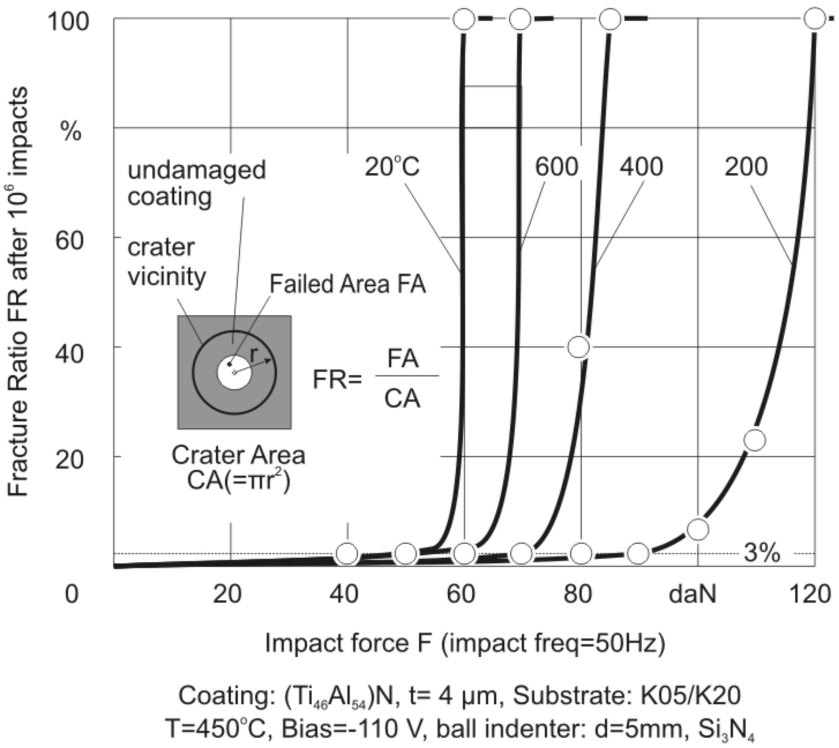


Fig. 19. Coating fracture ratio during the impact test at various temperatures and impact loads

The profile of the coating FR, under various operational temperatures, captures the coating removal rate versus the impact force. The worst performance appears at 20 ^{0}C and the overall best at a temperature level between 100 ^{0}C and 200 ^{0}C. At that temperature level the coating withstands up to almost 1200 N to be totally removed (FR = 100%) after 10^6 impacts.

The impact test performance up to a fracture ratio FR of approximately 3%, for various temperatures is presented in figure 20. The enhancement of the coating impact resistance is obvious through a temperature increase up to around 150 ^{0}C. The related critical impact load that corresponds to a FR-value of 3%, increases from 500 N at room temperature to a level of more than 1000 N. An additional temperature increase over ca. 150 ^{0}C results to a gradual reduction of the critical impact loads; this for example, can be seen clearly at temperatures of 300 ^{0}C, 400 ^{0}C and 600 ^{0}C, as the critical impact load decreases non-linearly to 500, 700 and 600 N respectively. SEM micrographs of the impressions after 10^6 impacts, corresponding to test loads, just to avoid a coating failure (FR<3%) and to activate a film fatigue breakage of various fracture ratios, are illustrated at the bottom of figure 20. The film FR increase after the coating fracture initiation is less intense at impact temperatures in the region of 100 ^{0}C to 200 ^{0}C, compared to all other temperatures, for the same impact force growth of 100 N, over the corresponding critical load. Thus, the film can withstand repetitive impacts, at temperatures between 100 ^{0}C and 200 ^{0}C, for a longer period up to its total removal (FR=100%).

The obtained results documented a gradual increase of the coating fatigue strength and thus of the mechanical strength properties for temperatures up to approximately 150 ^{0}C. This effect may be explained by the dislocations' movements, which are developed within the PVD film under load and the deceleration of their nucleation start, associated with a yield stress increase

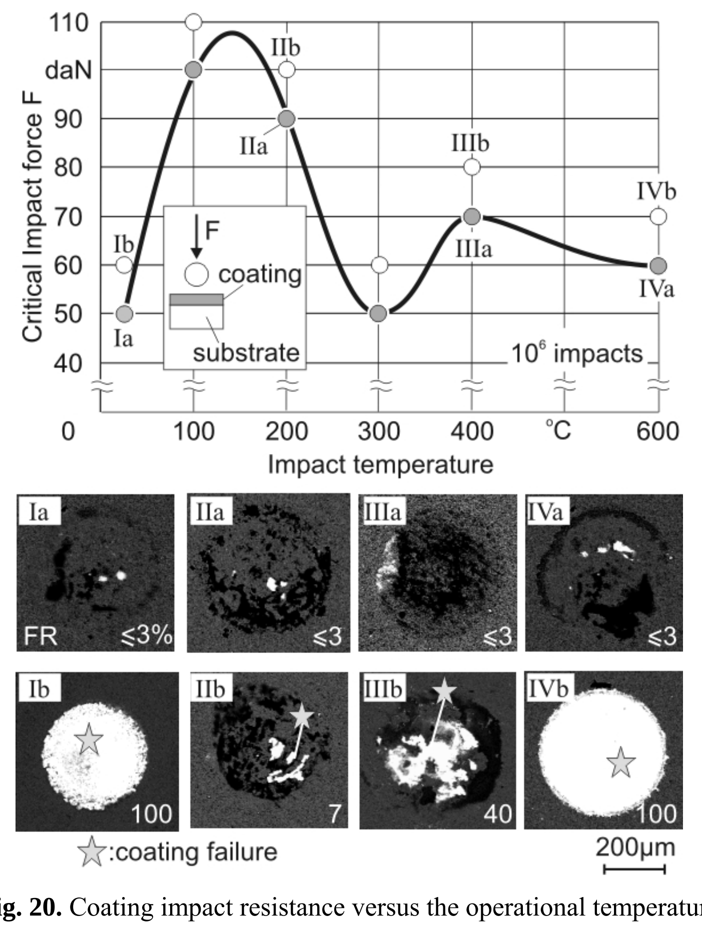


Fig. 20. Coating impact resistance versus the operational temperature

up to a temperature of ca. 150 ^{0}C [13-15]. Over this temperature, free atoms diffusion mechanisms antagonistic to the dislocation's movements facilitate the dislocation's nucleation and deteriorate the impact resistance up to approximately 300 ^{0}C. Moreover, up to ca. 400 ^{0}C, the film strength properties are improving due to the increasing of the atoms kinetic energy, as in the case of the steel blue brittleness. Over 400 ^{0}C, the coating strength properties and impact resistance are affected.

Conclusions

The paper presents some innovative methods developed by EEDM for characterizing the coatings' properties. These methods were based also on experimental set-ups developed by EEDM; the corresponding experimentation was supported by FEM supported computational approaches, facilitating the interpretation of the acting phenomena leading to film fracture and wear. Moreover, the presented results render the impact test, perpendicular or inclined, at room or elevated temperatures, as a very efficient method for detecting coating resistance against impact micro abrasion, with or without lubrication and according to the test conditions for the synchronous contribution of fatigue and thermal phenomena to the film fracture and wear. All these methodologies aim at isolating the parameters affecting the film performance, thus enabling the elucidation of the film fracture and wear phenomena

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Impact test, o tehnica inovativa pentru caracterizarea diverselor proprietati ale acoperirilor

Rezumat

Scopul principal al cercetarilor cu ajutorul unui impact test este caracterizarea proprietatilor de coeziune si adeziune ale acoperirilor in mediu uscat sau lubrifiat, la temperaturi ale mediului ambiant sau ridicate. Evaluarea acoperirii poate fi de asemenea corelata cu alte proceduri experimentale, ca de exemplu procesele de aschiere (frezare, strunjire). Aceasta metoda experimentala este sprijinita de simularea cu ajutorul metodei elementelor finite (FEM), care considera proprietatile elasto-plastice ale materialelor componente si prevede distrugerea acoperirii la diverse conditii de testare.