CSM Instruments
Advanced Mechanical Surface Testing

CSM Instruments

APPLICATIONS BULLETIN

Physical characterization of coated surfaces Part II : Scratch Test

Since part I of this AB covered the calculation of true mechanical coating properties by nanoindentation, all mechanical values are known which are required to fine-tune a scratch test for specific surface structures.

The standard scratch test is a widely used method to test the mechanical stability of coatings on different types of substrates and has become a sensitive technique to control the reliability of the manufacturing process. It is based on various standards [12, 13].

A diamond stylus (normally spherical diamond tip geometry) is utilized to apply a normal load F_N onto the sample surface. Simultaneously, the sample is displaced at a constant speed while the normal load is increased. At some point, the resulting stresses in the surface structure at the coating-substrate interface or between the coatings cause flaking or chipping of the coating. The critical load (Lc) at which a specific failure event occurs can be measured from the fluctuation in the tangential force, from the acoustic emission signal or can be observed as specific surface deformation in the optical microscope. Lc can also be detected as a discontinuity (step) in the post-scan surface. However, it can be difficult or impossible to calculate generic material properties, e.g. critical stresses of each failure mode, from such standardized tests, because they are not tailored to the surface structure under investigation and, therefore, do usually not create critical stresses in the proximity of the coating structure, but deep down in the substrate. Consequently, the conventional scratch test should be properly dimensioned at first.

Dimensioning of scratch tests

With the generic mechanical material properties calculated in the previous Application Bulletin (n37) by physically analyzing nanoindentation measurements, a subsequent scratch test can be dimensioned according to the flow chart in Fig. 2. Only the TR sample was investigated in this study (see details in our previous Application Bulletin n°37). The goal of dimensioning a scratch test is to get as much measurement information from the coating of interest as possible, so that a physical analysis of such tests can explain failure mechanisms of the intended applications – like mode-I fracture or mode-II fracture which are much closer to what happens in a contact situation from practice than a single load-component indentation. In order to achieve this, the most relevant degrees of freedom of a scratch test, which are the indenter geometry and applied normal force, have to be determined. The indenter geometry is defined by the indenter radius with respect to a spherical indenter (Rockwell) which is a common scratch test stylus. As FilmDoctor® Studio allows the modeling and simulation of lateral forces and resulting tilting, this software can also be used to dimension a scratch test where these contact conditions are relevant. Therefore, three different scratch situations with spherical indenters of 20 µm, 50 µm, and 200 µm radii as well as normal loads of 1 N, 20 N and 80 N, respectively, are modeled and the resulting Von Mises stress distribution is calculated as shown in Fig. 1 based upon previously determined elastic modulus of the layers (EC1, EC2) and the substrate (ES). These scratch test parameters may be chosen based upon experience or calculated stress distribution of the indentation measurement and in accordance to the measurement equipment at disposal. For this first dimensioning, the tangential force may be chosen according to coefficient of friction values from literature and the surface is assumed to be plane. It is obvious that these different scratch parameters result in a completely different stress distribution, locations of maxima, and values of maxima.

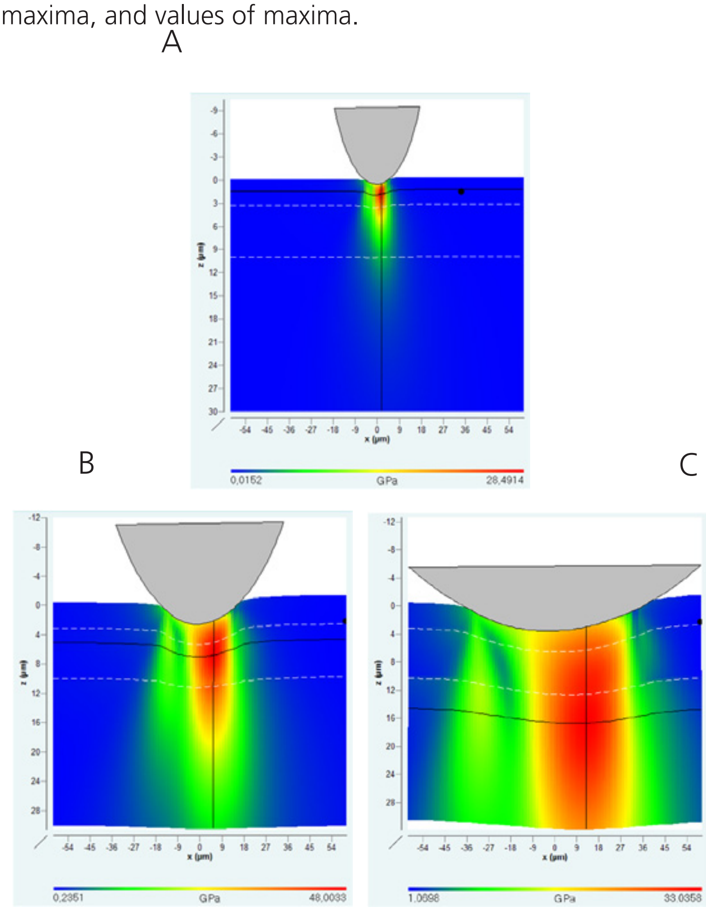


Fig. 1: Simulation of distribution of Von Mises stress for three different kinds of scratches with spherical tips: 20µm radius with 1N normal load (a), 50µm radius with 20N normal load (b), and 200µm radius with 80N normal load (c). The interfaces are indicated by the white dashed lines. The block cross hairs mark

the von-Mises stress maxima.

While the von Mises stress in Fig. 1c is concentrated in the substrate, the maxima of Von Mises stress are in the first and second layer of the coating in Fig. 1a and Fig. 1b, respectively. The von-Mises stress maxima should be in good agreement with the depth of interest, because it is expected that the sensitivity of subsequent scratch tests will be in these depth ranges. Additionally, the maximum should sufficiently exceed the yield strength of the constituent of interest within the limits of the relevant application situations to ensure that a failure will happen.

Physical analysis of scratch tests

A scratch tester has to measure certain information for a physical analysis of a scratch test. Apart from the progressively loaded scratch itself, during which the normal load, the penetration depth under this load, and the lateral force are measured, the profile of the surface prior to the scratch has to be obtained. Because the contact situation and, consequently, the stress-strain fields are significantly different depending on where and how the stylus is in contact with the sample surface (see Fig. 2). A three-dimensional topography of the sample surface for instance from an AFM would be perfect, because it makes also a difference whether the stylus hits an asperity exactly in the center and will scratch over (very rare case) or if the stylus hits the asperity at a flank side and, hence, will deflect and result in an multi-axial inclined contact situation. For reasons of simplicity, only a 2D surface profile as obtained by a simple pre-scan will be used here.

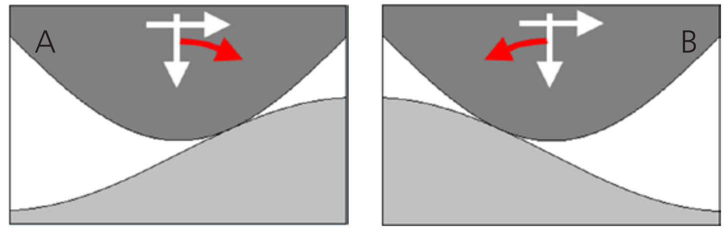


Fig. 2: Schematic of two different inclined contact situations: when the stylus moves upwards the flank of an asperity (a) and downwards (b), the dark gray upper shape denotes the stylus and the light gray lower shape the asperity of the sample surface. The white arrows denote the applied forces and the red arrow the tilting of the stylus due to the asperity.

Additionally, the surface profile after the scratch has to be measured to distinguish the plastic deformation from the elastic deformation as it results in a different contact situation (e.g. size and location of contact area) and is therefore critical for the simulation. Just like the pre-scan scratch surface, a 2D profile of the residual surface can be obtained by a simple post-scan or a 3D topography by an AFM measurement for instance. All these measurement data are considered for the calculation of the stress-strain field developing during the scratch test enabling a physical analysis of the scratch test, because specific critical mechanical properties of the constituent of interest (layers/substrate) can be determined. The critical load of failure (Lc) is also determined and will be correlated to the simulated contact field in order to find out why the layer failed at that very moment (see Fig. 3).

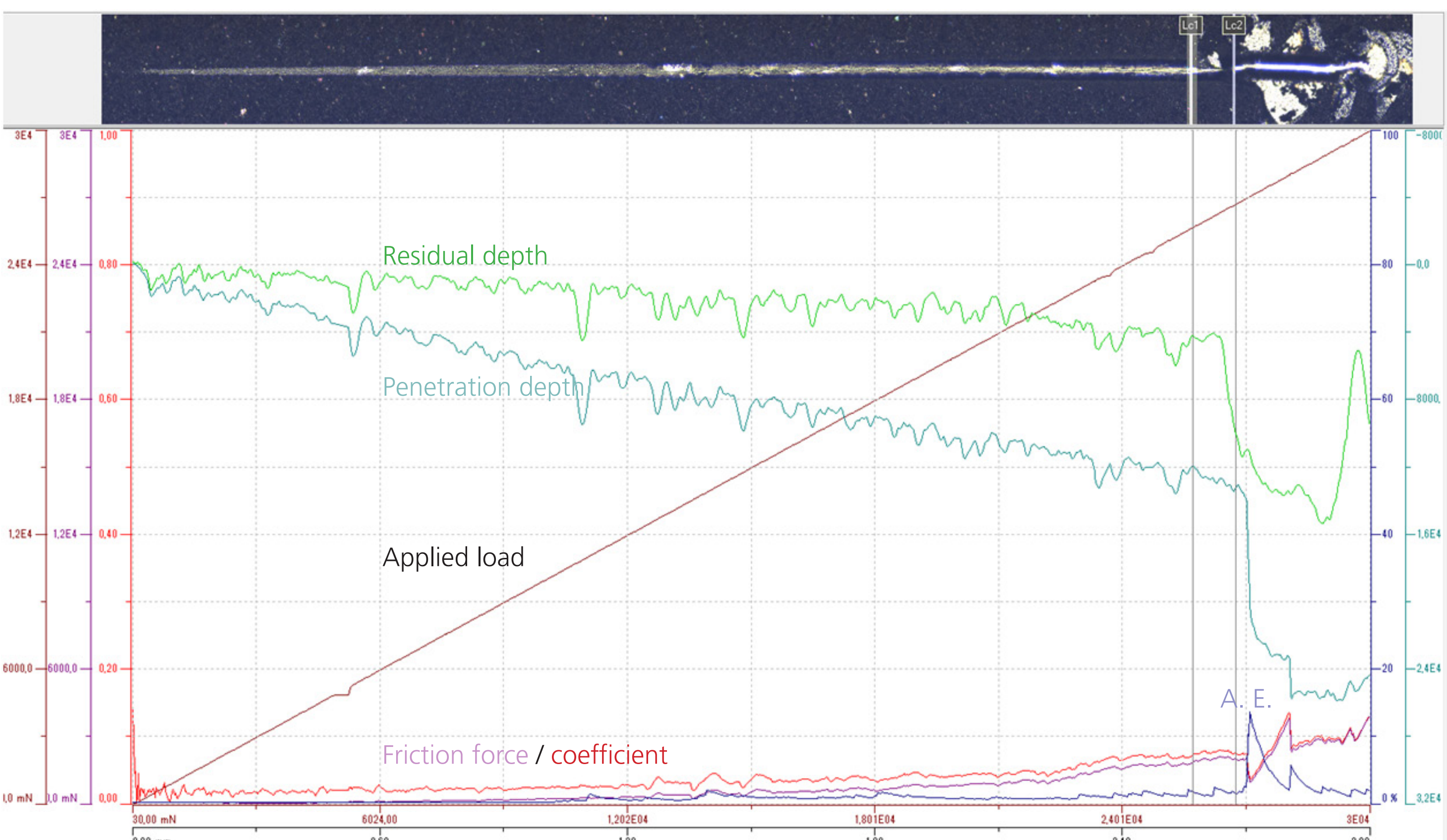


Fig. 3: Profiles of measured information along the scratch track together with an aligned panorama image of the scratch track on top.

The software SSA®, ISA, or FilmDoctor® calculate more than 28 field components based upon these measurement data. The Von Mises stress shown in Fig. 4 and the normal stress in scratch direction shown in Fig. 5 are only two of them, but the most relevant ones for the typical failure mechanisms mode I fracture and mode II fracture. At the beginning of the pre-dimensioned scratch test the stresses are definitely subcritical (6.9 GPa maximum Von Mises stress and 2.2 GPa maximum tensile stress) and are concentrated in the top layer as shown in Fig. 5a and Fig. 5a. During the scratch test the maximum of the von-Mises stress moves downwards to the substrate due to the increasing normal load. In between the beginning of the scratch test and the Lc moment, the maximum von-Mises stress (25.8 GPa) is concentrated in the interlayer as shown in Fig. 4b. As shown in Fig. 2 at this point, plastic deformation occurs, because the residual depth is well below the pre-scan surface. But note that the yield strength of the interlayer is 30 GPa, so there is no yielding happening in the interlayer. Instead, the von Mises stress in the substrate (22.1 GPa) has exceeded the substrate's yield strength (22 GPa) at the interface and, hence, plastic deformation in terms of yielding takes place in the substrate. In this case, this is the initial failure of the system which will result in total failure of the coating later. Consequently, the coating progressively loses its support from the substrate and is more and more stretched at the surface. The tensile stress at the surface behind the stylus already is 8.2 GPa according to Fig. 6b. Eventually, the substrate has yielded so much in the moment of failure (Lc = 26.3N), that the top layer cracks (mode I fracture) from the surface straight down to the coating-substrate interface behind the stylus. This failure mode is schematically illustrated in Fig. 6a together with an optical picture of the corresponding failure location on the scratch track in Fig. 6b. Hence, the calculated tensile stress of the top layer as 10.2 GPa is its critical tensile stress. Note that it is necessary to know the LC values in order to link them to the calculated stress-strain fields. This allows one to determine generic mechanical properties like the critical tensile stress by means of scratch testing.

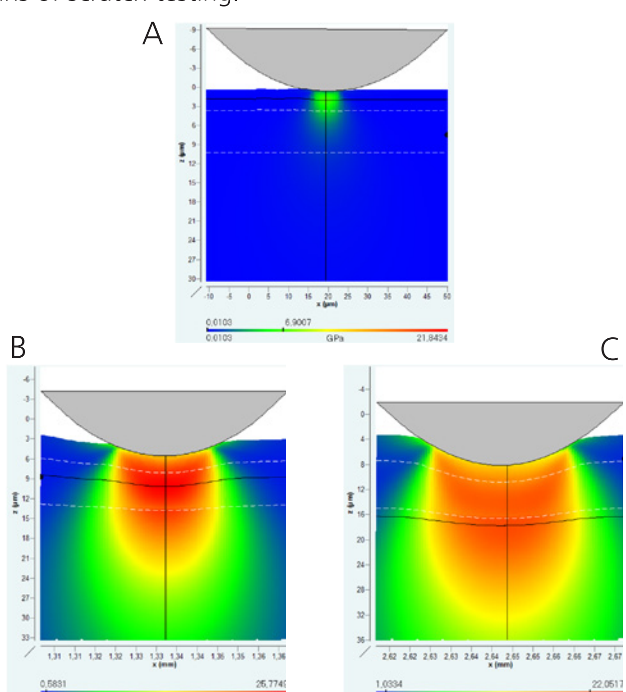


Fig. 4: The evolution of von-Mises stress during the scratch test shown at three measurement points: (a) at the beginning of the scratch test, (c) in the moment of LC failure, and (b) in between. The black cross hairs indicate the location of the maximum.

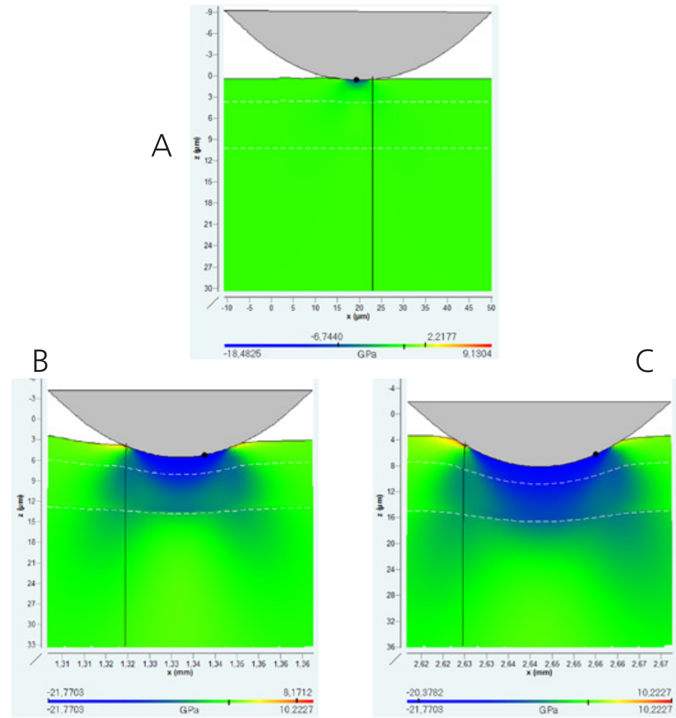


Fig. 5: The evolution of normal stress in scratch direction illustrated at three measurement points: (a) at the beginning of the scratch test, (c) in the moment of LC failure, and (b) in between. The black cross hairs indicate the location of maximum tensile stress.

Hence, such a physical analysis of mechanical contact measurements like instrumented indentations and scratch tests enable sone to find out why a surface structure fails in a certain moment. These results provide indications on how the investigated coating structure can be improved. But the optimization of coated surfaces would be beyond the scope of this work.

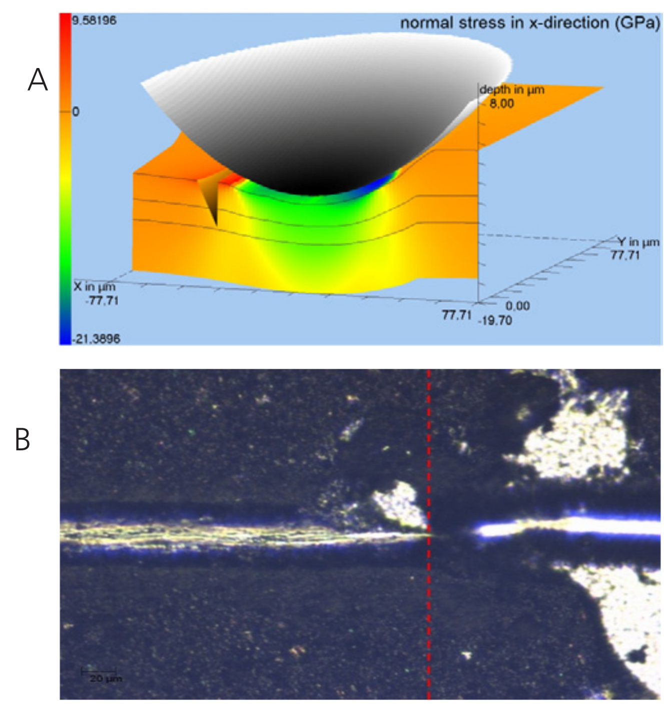


Fig. 6: Illustrative scheme of the failure mechanism (a) and an optical image of the post-scratch surface (b) in which the corresponding LC position is marked in red.

Example of application: for cutting tools

Advanced design of decorative and wear resistant coatings hinges on the optimization of the mechanical properties (Elastic modulus, yield strength, adhesion, intrinsic stresses, fracture, fretting, wear resistance...) of the coating-substrate system.

The goal is to find material and structural solutions which keep the resulting stress-strain field under typical application conditions below the stability limits of the system.

Based on nanoindentation measurements obtained from the coating-substrate system which should be optimized, a scratch test is dimensioned as function of the load range and the indenter geometry. The measured data from this "Physical Scratch Test" is used to compute spatial or 3D stress profiles of, for instance, the von Mises stress and the normal stress (tensile/compressive) in scratch direction.

To improve and understand better the coating-substrate interface, we follow the flow chart below.

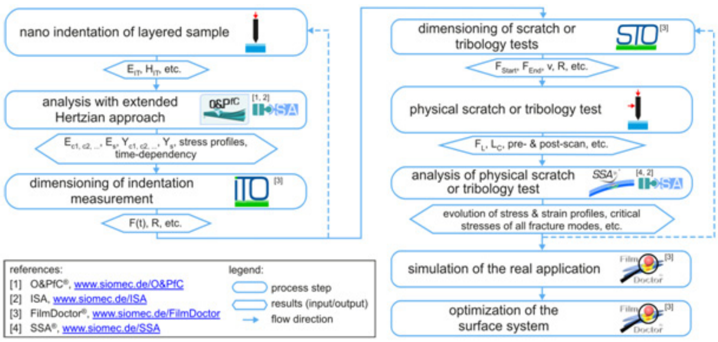
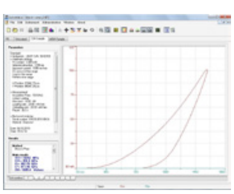
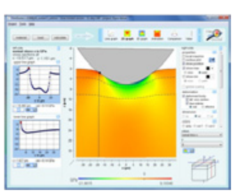


Fig. 7: A flow chart of the procedure of mechanical characterization and optimization of arbitrary structured surfaces

Practical example



Step 1



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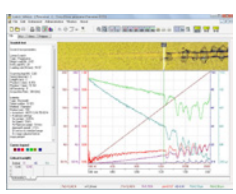

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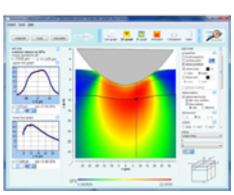

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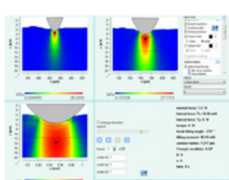
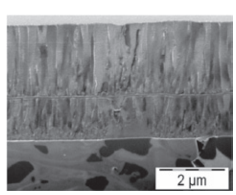


Step 5



Step 6

> Step 1:
A nanoindentation test is performed to accurately measure the materials properties of the substrate and coatings.

> Step 2:
With our partner software and the materials properties previously measured by Nanoindentation, an analysis is made for perfect dimensioning of the scratch test.

> Step 3:
Scratch testing with the defined conditions is performed.

> Step 4:
An advanced analysis with the integration of the residual penetration depth is made with the simulation software in order to know the complete stress field.

>Step 5:
For better understanding, an animated scratch is created.

>Step 6:
Optimize your coating-substrate system.

References

[7] N. Schwarzer, Q.-H. Duong, N. Bierwisch, G. Favaro, M. Fuchs, P. Kempe, B. Widrig und J. Ramm, „Optimization of the Scratch Test for Specific Coating Designs," Surface and Coatings Technology, 2011.

[8] Saxonian Institute of Surface Mechanics, „Material DataBase," [Online]. Available: www.siomec.de/database.

[9] Saxonian Institute of Surface Mechanics, „FilmDoctor," [Online]. Available: www.siomec.de/FilmDoctor.

[10] Saxonian Institute of Surface Mechanics, „Oliver&Pharr for Coatings (O&PfC)," [Online]. Available: www.siomec.de/OPfC.

[11] Saxonian Institute of Surface Mechanics, „Indentation and Scratch Analyzer (ISA)," [Online]. Available: www.siomec.de/ISA.

[12] ISO 20502 Fine ceramics (advanced ceramics -advanced technical ceramics) - Determination of adhesion of ceramic coatings by scratch testing

[13] ASTM C1624 Standard Test Method for Adhesion Strength and Mechanical Failure Modes of Ceramic Coatings by Quantitative Single Point SCRATCH Testing

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This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

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