SCRATCH TEST

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CONTENT

INTRODUCTION

Scratching has been a well known tool for obtaining a material hardness since 1812 when the German mineralogist Friedrich Mohs put forth his scale of the mineral hardness. This scale contains ten minerals (1. talc, 2. gypsum, 3. calcite, 4. fluorite, 5. apatite, 6. orthoclase feldspar, 7. quartz, 8. topaz, 9. corundum, 10. diamond), ordered from the softest to the hardest mineral. This scale is based on the simple idea that harder material can visibly scratch another material, but not contrarily. Yet the scale itself might be sufficient to acquire basic idea of minerals hardness it does not meet the requirements of industrial practice.

Hardness is defined as an ability of material to resist penetration or abrasion by other materials. In agreement with this definition can be performed various tests that differ from the technique and value of the hardness. Evaluating of the hardness can be divided into the three main types: *rebound hardness*, *indentation* and *scratch*. Rebound hardness is evaluated by measuring of the bounce of hammer dropped from the fixed height onto the material. Indentation hardness is evaluated according to the dimensions of indent left by the indenter. On the field of engineering is the most common Vickers's, Brinnel's and Rockwell's test. Scratch hardness is often assessed in the case of surface films or as a comparative method. [1] [2]

Hardness can be evaluated on the basis of three different scales: macro, micro and nano scale. Specimen tested on the macro-scale usually undergoes test load higher than 10 N and in this scale are also included aforementioned Vickers's, Brinnel's and Rockwell's tests. Development of the micro and nano-scale tests has been driven by the need of material science to test samples on the smaller scale e.g. hard thin coats, separately test elements of composed material or when only a limited amount of material is available. Macro-scale test would not be possible to perform under such circumstances. Interestingly, it has been observed that due to flaws occurred in a material, hardness measured on the micro and nano-scale is higher than hardness measured on the macro-scale.

This paper is focused on the discussion over the various types of scratch tests for different purposes and is divided into the two sections. In the first section will be discussed scratch test method itself and scratch tests for different purposes according to the tested material. Second part will be devoted to the practical evaluation of the scratch test.

1 THEORY

1.1 SCRATCH TESTERS

In accordance with aforesaid scales of scratch tests, there are corresponding testers that are used for given range of load. These high sophisticated devices are produced by the specialized manufacturers such as CSM, Tribotechnic, Anton Paar or Sheen. For the sake of illustration see Tab. 1 where are mentioned different types of CSM scratch testers including their specifications.

Scale	Nano	Micro	Macro
Normal Force Range	10 µN to 1 N	30 mN to 30 N	0.5 to 200 N
Load Resolution	0.15 µN	0.3 mN	3 mN
Maximum Friction Force	1 N	30 N	200 N
Friction Resolution	0.3 mN	0.3 mN	3 mN
Maximum Scratch Length	120 mm	120 mm	70 mm
Scratch Speed	0.4 to 600 mm/min	0.4 to 600 mm/min	0.4 to 600 mm/min
Maximum Depth	2 mm	1 mm	1 mm
Depth Resolution	0.6 nm	0.3 nm	1.5 nm
XY Stage	120 x 20 mm 245 x 120 mm (OPX)	120 x 20 mm 245 x 120 mm (OPX)	70 mm x 20 mm
XY Resolution	0.25 µm 0.1 µm (optional)	0.25 µm 0.1 µm (optional)	0.25 µm 0.1 µm (optional)
Video Microscope Magnification	200x, 800x, 4000x	200x, 800x	200x, 800x
Video Microscope Camera	Color 768 x 582, high resolution	Color 768 x 582, high resolution	Color 768 x 582, high resolution

(OPX) - Open Platform [3]

Tab. 1 CSM scratch testers

1.2 SCRATCH TEST

In the case of nano and micro-scratch test the surface of the specimen must the flattened and polished. On that scale any disturbance on the surface might cause fluctuation in both force and depth of scratch which might make further correct evaluation complicated or impossible. When ready, the specimen is inserted into the chamber, which during the test separate specimen from external surroundings as it can cause inaccuracy during the test. In the next step the location for the scratch test is chosen and the surrounding area is scanned.

Scratch test itself usually consists of three stages, which follow the same trajectory over the surface:

First - So called prescan is used for measuring the surface. It is performed under the lowest possible pressure so that no permanent damage is made on the surface.
Second - Scratch is performed according to preset conditions (speed, force, depth)
Third – Postscan is performed in order to measure residual topograpfy of the damaged area. Similarly to the first stage, it is performed under the lowest possible pressure.

When the specimen is subjected to the load test, crucial parameters of the test are measured such as vertical and horizontal force, depth and length of the scratch (see Fig. 1). Additionally, surroundings of the scratch test can be mapped and scaled so that precise geometry including the width of the scratch test can be measured. According to the vertical load, scratch test can be performed in three types: Constant, progressive, incremental, see Fig. 1. During the scratch test can occur three types of failure: elastic, plastic and fracture, whilst each is studied for the different purpose. Technical standards of procedures and application methods linked to the indentation and scratch tests are also developed by ASTM organization (American Society for Testing and Materials).

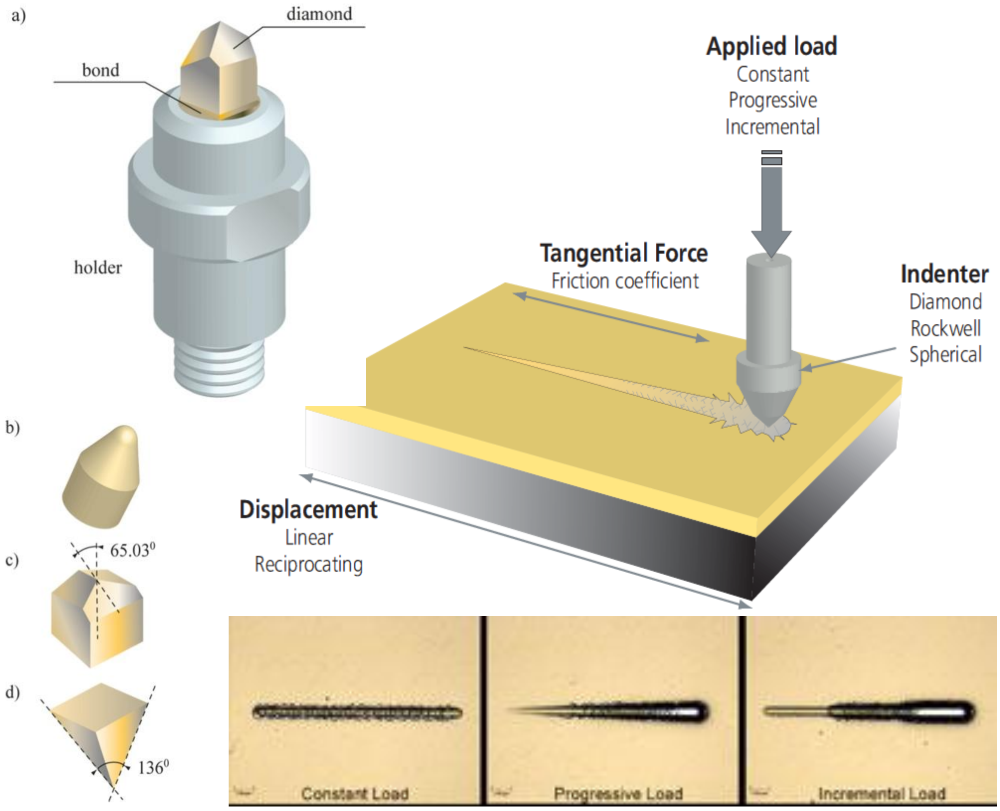


Fig. 1 Scratch test [3] [4]

1.2.1 TESTS OF COATINGS

BASIC CHARACTERISTICS

Test on coatings is often used for comparing and assessing characteristics of several coating samples. ASTM standard [5] recommends performing progressive scratch test at least three-times on the same specimen. As the different conditions affect material characteristics standard [5] also suggest conditions and preset test parameters.

Fig. 3 illustrates typical results of a scratch test and plots vertical displacement (left side) and applied force (right side) versus scratch distance. Desired characteristic values are calculated from the curves $C1$ – $C5$ as follows:

PD [mm] – penetration depth is calculated from the prescan values $C1$ and displacement from the scratch test $C3$.

$$PD = C3 - C1 \tag{1}$$

RD [mm] – residual depth (permanent plastic deformation) is calculated from the values of prescan C1 and values of postscan $C2$.

$$RD = C2 - C1 \tag{2}$$

ER [mm] – elastic recovery defines the value elastic deformation that occurs after the scratch test.

$$ER = C3 - C2 \tag{3}$$

C_f [-] – friction coefficient is calculated as a ration of the tangential $C4$ and normal force $C5$.

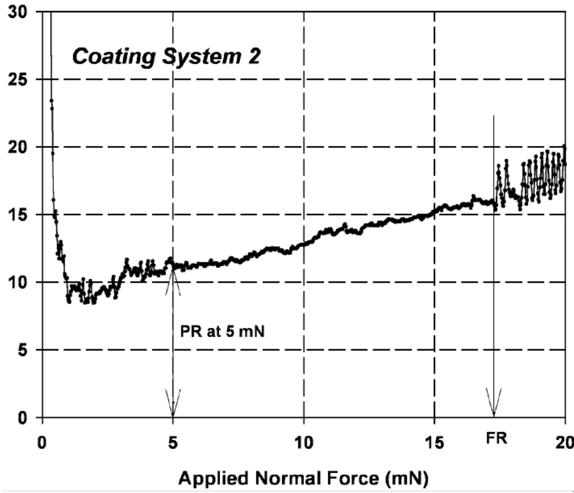
$$C_f = C4/C5 \tag{4}$$

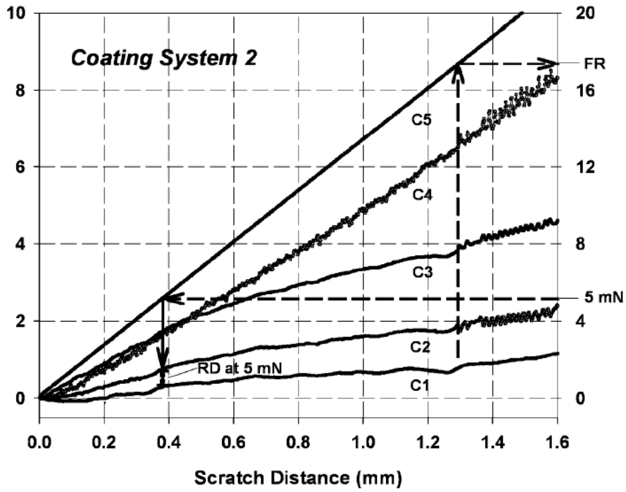
PR [mN/mm] – plastic resistance is calculated from the particular normal force F_N that should be taken from the lower part of the graph which is relatively stable, see

Fig. 2. This stable section is usually followed by the rapid fluctuation which indicates fracture of the material.

$$PR = F_N/RD \tag{5}$$

FR [mN]– fracture resistance can be determined as the value of normal force when the first fracture occurs. This event is followed with wild fluctuation in both force and penetration, see Fig. 2 Plot of the applied normal force Fig. 3 Typical scratch test plot. Fracture can be also followed with sudden change o friction coefficient or acoustic emission [6].





Typical data from a nanoscratch experiment:
C1—Vertical displacement of the indenter during the pre-scan
C2—Vertical displacement during post-scan
C3—Vertical displacement during scratch
C4—Tangential force
C5—Applied normal force **(1)**

Fig. 2 Plot of the applied normal force [5] Fig. 3 Typical scratch test plot [5]

ADHESION

Additionally the adhesion scratch test might be performed. In this test the progressive vertical force is applied. The moment when the scratch penetrates trough the coat is followed with the fracture of material a fluctuation of forces. This force is called *critical force*. [6]

WEAR

The set of laboratory test is often closed with wear test, which measure the number of cycles necessary to wear through the coating. Test is performed with constant force of a small magnitude. [6]

SCRATCH HARDNESS *HS*

In the case of hard thin coatings its hardness might be required to evaluate or compare. Unlike in previous section, in this case is used scratch test with constant load. In order to obtain scratch test hardness is used sharp, usually diamond tip, of known geometry. The resulting width is measured by AFM (atomic force microscope) and material scratch hardness is calculated:

$$HS = \frac{k \times F_N}{W^2} \tag{6}$$

where k is a constant referring to the known geometry of scratching tip (Berkovic indenter k=2.31), F_N is applied normal (vertical) force, W is the width of scratch, A_T is an area which project loading onto the sample. [6]

1.2.2 COHESIVE MATERIALS

Scratch test might become a progressive tool for the evaluation of basic material characteristics in the case of cohesive materials such as rock, cement or bricks. Research dedicated to the scratch tests revealed relation not only between the scratch test measurements and strength properties but also between the scratch test measurements and fracture properties. This laboratory test can be especially valuable when a limited amount of material is available, although the specimen must undergo shaping.

DETERMINATION OF MOHR-COULOMB PARAMETERS

In the [7], [8] and [9] was published method for determination of the unconfined compressive strength UCS, cohesion C and internal friction angle φ. This method is, however, performed with rectangular blade of width W a dragged though the specimen in the depth D, inclined with back-rake angle θ, see Fig. 4. Test was performed under these settings [9]:

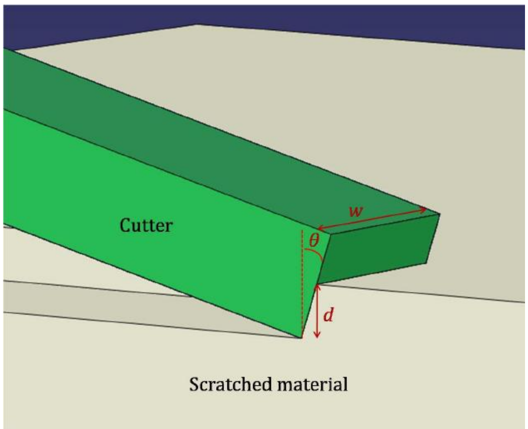


Fig. 4 Geometry of the scratch test

Width: 2.5mm, 5mm, 10mm
Depth: 0.1 – 0.35mm
Normal force: 5 – 15 N
Tangential force: 15 – 35 N

Scratch hardness HS is calculated from:

$$HS = \frac{F_T}{W \times D} \tag{7}$$

, where F_T is horizontal applied load

when considering friction coefficient $\mu = \frac{F_T}{F_V} = \tan\varphi$ (φ – angle of internal friction, F_T – horizontal force, F_V - vertical force), then cohesion C is calculated from Eq.(8) for the Mohr-Coulomb model as

$$HS = 2C\frac{\cos\varphi(1-\sin^2\theta)}{1-\sin\theta\cos\varphi\sqrt{1+(\tan\varphi\sin\varphi)^2}-\sin\varphi\cos^2\theta} \tag{8}$$

In conclusion UCS is calculated by Eq(9)

$$UCS = 2C\frac{\cos\varphi}{1-\sin\varphi} \tag{9}$$

Work on the assumption that $\mu = \tan\varphi$ was also mentioned in [10] as it is in an agreement with the observed behavior that the value of the friction angle on the interface of rock vs. blade is very close to the internal friction angle of the rock itself.

FRACTURE TOUGHNESS K_{IC} OBTAINED WITH RECTANGULAR BLADE

When seeking for new applications for scratch test was found the technique to obtain fracture toughness, see [9] and [11]. This method considers the same rectangular blade as in the previous section, see Determination of Mohr-Coulomb parameters. According to [9] and [11] K_{IC} can by obtained from Eq.(10), when presuming horizontal crack Fig. 5 Scratch test geometry :

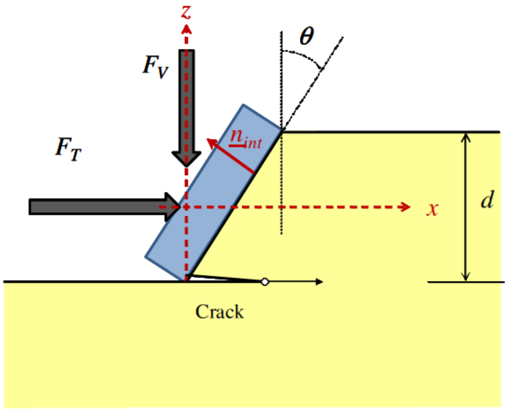


Fig. 5 Scratch test geometry [11]

$$\sqrt{\frac{1}{2}F_T^2+\frac{3}{10}F_V^2} = K_{IC}W\sqrt{D} \tag{10}$$

where F_T and F_V are calculated mean forces, see Fig. 6.

Eq. (10) is based on the formulation of $J - integral$, which is equal to energy release rate G when further development of the horizontal crack is presumed, see [11] for detailed deductions.

$$G = \frac{{K_{IC}}^2}{E} = \frac{\frac{1}{2}F_T^2+\frac{3}{10}F_V^2}{EW^2D} \tag{11}$$

According to [11], Eq. (11) is verified when looking at Fig. 7, where are depicted scratch test for different widths (2.5, 5 and 10mm) and different depths (0.1-0.6mm) performed on the cement paste. Measured slopes of colored lines should be toughness K_{IC} whose different slopes for different widths should indicate scale effect. That means higher toughness for smaller cuts (cutters).

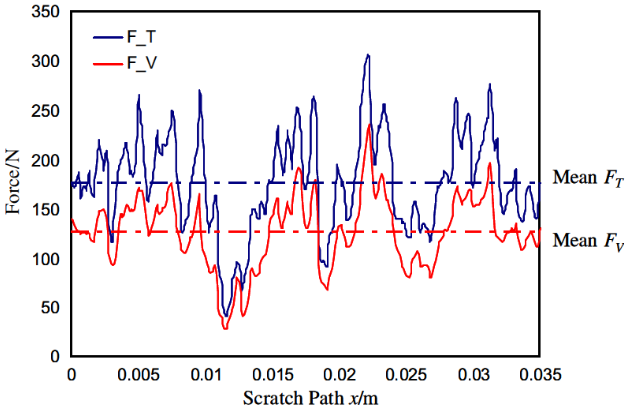


Fig. 6 Record of the scratch test results [11]

As a support for proposed formulation there was mentioned agreement between measured toughness K_{IC} for W = 10mm from Fig. 7 ($K_{IC} = 0.68\ \mathrm{MPa}\sqrt{\mathrm{m}}$) and toughness measured by notched three-point bending test ($K_{IC} = 0.65\ \mathrm{MPa}\sqrt{\mathrm{m}}$). Same experiment was also performed for the sandstone with remarkable estimation. This approach would though provide a direct determination of material toughness, which can be repeated without need for large specimens or extrapolation techniques.

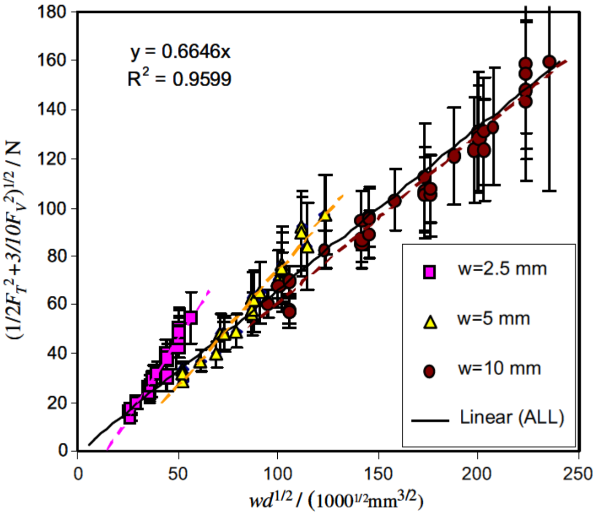


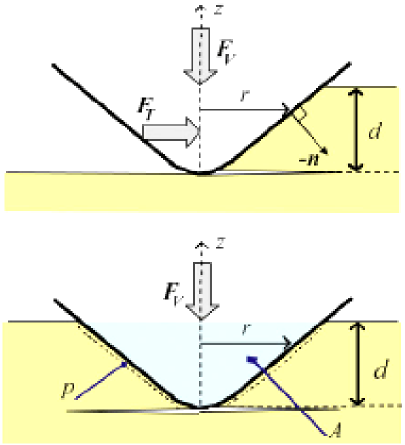
Fig. 7 Determination of the fracture toughness from the scratch test [11]

Although the evaluation of toughness K_{IC} itself is quite straightforward it was discussed later in detail in [12]. There was claimed that experimental data in [9] and [11] might had been incorrectly interpreted.

Firstly, there was implied that during such shallow cuts as 0.1 – 0.6 mm, the plastic yielding is dominating failure criterion and not fracture. Furthermore, there was shown that linear relation between mean force and depth of cut (see Fig. 7) is valid only at small scale beneath 1mm and then becomes scattered.

Secondly, the assumption of horizontal development of the crack in order to use $J - integral$ might be misleading as a chipping occurs when the cutter advances forward. Furthermore, according to observation, crack goes upward and none horizontal crack develops.

FRACTURE TOUGHNESS K_{IC} OBTAINED WITH MICRO-SCRATCH TEST



In order to provide a procedure, which would one enable to obtain the fracture toughness via the micro-scratch test, was published [13]. Similarly to [9] and [11] this technique presume initial circular horizontal crack, see Fig. 8. Furthermore, the affect of vertical force F_V on the fracture is neglected and the stress field ahead the indenter tip is considered uniaxial. According to [13] fracture toughness can be written as:

$$K_{IC} = \frac{F_T}{(2pA)^{1/2}} \tag{12}$$

where function of the indenter f for conical indenter can is written as:

$$f = 2p(d)A(d) = 4\frac{\sin\theta}{\cos^2\theta}D^3 \tag{13}$$

Though Eq. (12) can be rewritten as:

$$K_{IC} = 2\frac{F_T}{\left(\frac{\sin\theta}{\cos^2\theta}D^3\right)^{1/2}} \tag{14}$$

where D is depth of the scratch. Proposed formulation was also supported by comparing of calculated and measured fracture toughness for different materials.

COMPRATATIVE METHOD - NANO-SCRATCH TEST

Scratch test might be also used as a comparative method in medical science, see [14]. In this case, two types of material preparations were compared by nano-indentation and nano-scratch test, while Berkovic indenter was used. When comparing the hardness HS of both specimens the constant loading test was performed while the Eq.6 was used with the coefficient k=2.31. Width W of the scratch test was measured by AFM.

2 EVALUATION OF PERFORMED SCRATCH TEST

In order to examine the aforesaid calculations for cohesive materials a nano-scratch test was performed on the cement paste. Scratch test was controlled by vertical force and performed with constant load. Test is characterized by three following stages: rapid increase in the normal force, stable stage of a maximum normal force (4300µm), rapid decrease in the normal force, see Fig. 9. Slow increase in a depth of the scratch test might imply the less hard matter of the specimen, see Fig. 10. Fig. 12 shows Lateral displacement vs. time, note that zero value of lateral force indicates initial position of indentation tip. Lateral force remains for the time of the test steady without significant fluctuations.

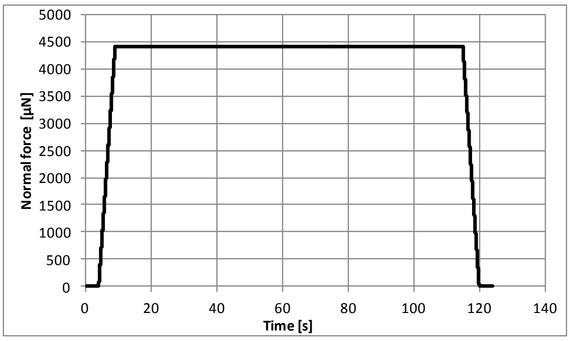


Fig. 9 Normal force

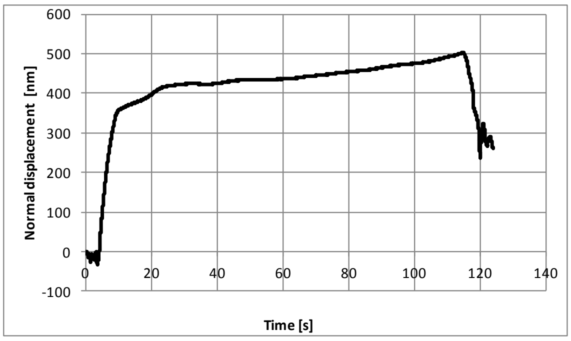


Fig. 10 Normal displacement

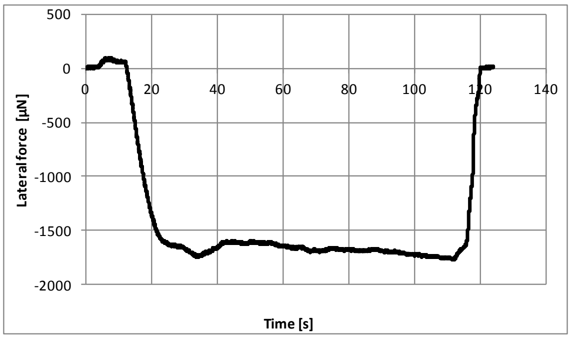


Fig. 12 Lateral force

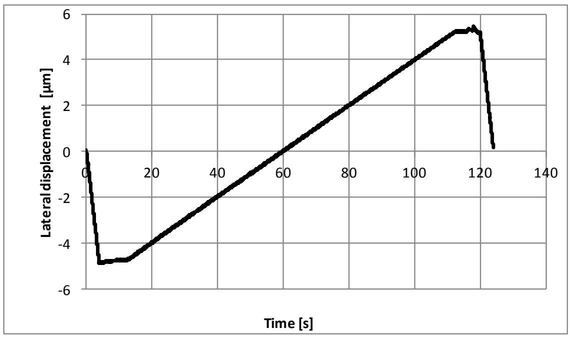


Fig. 11 Lateral displacement

For the sake of illustration here are provided pictures of a scan and measurement of the scratch, see Fig. 13 – Fig 15.

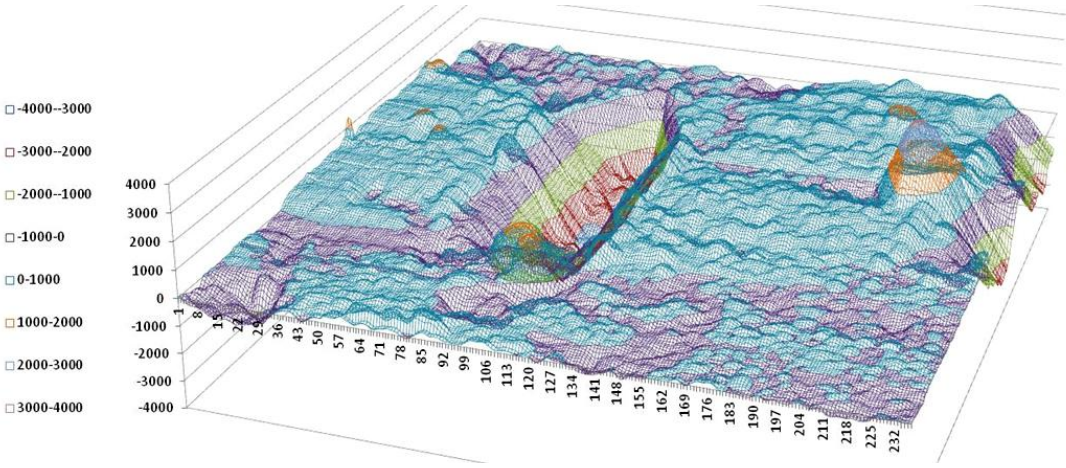


Fig. 13 Projection of the performed scratch

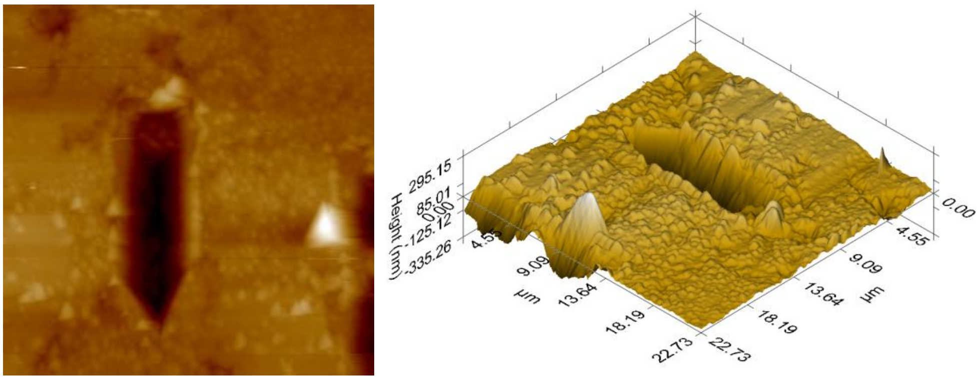


Fig. 14 Scratch – AFM

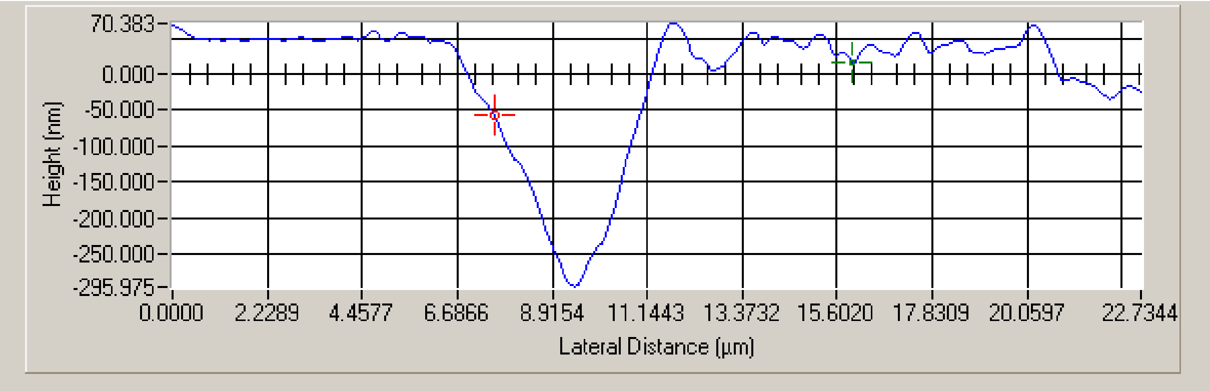


Fig. 15 Measured width of the scratch

SCRATCH HARDNESS

According to Eq.6, scratch hardness can be calculated as:

$$HS = \frac{2.31\ \times\ F_N}{W^2} = \frac{2.31\ \times\ 4\,400}{4.75^2} = 0.450\ \mathrm{kPa}$$

where F_N [µm] is an average normal force calculated from the stable stage of the scratch test. Width of the scratch W is obtained from the Fig. 15 Measured width of the scratch (lateral distance).

FRACTURE TOUGHNESS K_{IC}

Fracture can be calculated as Eq. 12, Eq. 13 and Eq. 14 suggest:

$$K_{IC} = 2\frac{F_T}{\left(\frac{\sin\theta}{\cos^2\theta}D^3\right)^{1/2}} = 2\frac{1\,668}{\left(\frac{0.908}{0.174}0.3^3\right)^{1/2}} = 8.89\ \mathrm{kPa\ m^{1/2}}$$

MOHR-COULOMB PARAMETERS

Even though Eq. 8 and Eq. 9 are suggested for the application of rectangular blade, here is attempt to evaluate parameters C – cohession and φ – internal friction angle of M-C model via those equations. Average friction coefficient is μ = 0.379, where relation between μ and φ can be written as:

$$\mu = \tan\varphi$$
$$0.379 = \tan\varphi => \varphi = 20.7°$$

Half apex angle: $\theta = 65.39$

$$HS = 2C\frac{\cos\varphi(1-\sin^2\theta)}{1-\sin\theta\cos\varphi\sqrt{1+(\tan\varphi\sin\varphi)^2}-\sin\varphi\cos^2\theta}$$

$$8\,890 = 2C\frac{0.935(1-0.826)}{1-0.909\times0.935\sqrt{1+(0.378\times0.353)^2}-0.353\times0.173} = 2C\frac{0.163}{0.081}$$

$$8\,890 = 2C\frac{0.163}{0.081}$$

$$C = 2.208\ \mathrm{kPa}$$

In conclusion UCS is calculated by Eq.(9):

$$UCS = 2C\frac{\cos\varphi}{1-\sin\varphi} = 2\ \times 2\ 208 \times \frac{0.935}{1-0.353} = 6\ 381.7\ \mathrm{Pa}$$

The values calculated from the scratch test can be claimed unreliable as the expected values of hardness HS should lye around the 65 MPa and value of fracture toughness K_{IC} around 0.6 MPa [9] [13][14]. Following values of Mohr-Coulomb parameters are affected by HS and therefore also provide low values. Yet the values might be low, there was not known the age of specimen, which can significantly affect the results of calculations.

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