DIAMANT
VOLLSTÄDT DIAMANT GmbH

Operating instruction

Friability –Test

The Friability Tester measures the impact strength value (Toughness Index - TI) of diamond.

Attention: *With the Friability Tester ST1 cannot tests be performed on abrasives whose friability index is less than 20 because it is not possible to set less than 500 cycles.*

Remarks for the **Friability Tester VERSION ST4**

Version ST4 can be used to measure the friability index in diamonds, with significant differences in impact strength, and in cBN.

Contents:

IMPORTANT NOTICE

The manufacturer will not be held liable for any damage arising from the improper use of the equipment, for actions taken by unauthorized personnel and for any operation that does not correspond to those recommended and described in this manual.

1 THEORY AND LIMITS OF THE FRIABILITY INDEX MEASUREMENT

In the majority of diamond tools for machining stone materials the cutting action is roughly similar to hammering, i.e. it acts by crushing pression waves. Hence it is paramount to know their shock strength, that is, how hardness and resiliency are balanced.

Besides this, the cutting properties of a tool are also, though not only, affected by the correct combination of the diamond properties with the bonding agent hardness and resiliency.

This is why synthetic diamonds are supplied complete with the applicable commercial grading, dividing them according to their application area, substantially based on impact strength and the decay of that property as a result of temperature and sintering time.

From the above it comes out dear that for an independent, state-of-the-art design and control of diamond tools for machining stone materials it is particularly crucial to thoroughly know, besides the bonding agent properties, also the diamond specifications, measured directly before and after sintering.

A measurement can be reliable, hence enabling the operator to correctly evaluate the conditions he is investigating, only provided that measurement is repeatable and predictable.

Repeatability means that measuring according to the same procedures equal materials, the applicable numerical resu1ts are the same or with a deviation as slight as possible. A tolerance acceptable to the operator is often within the range of some per cent units.

A measurement is regarded as predictable when changing some parameters of the measuring method, quality or type of material to be examined, it is possible to foretell the new numerical value. The latter requirement can be complied with only provided that measurement is supported by a well-grounded theory.

The final numerical value of measurement should be separately considered.

Actually, measurements can be divided into absolute or relative.

Measurements are regarded as absolute when, once defined a standard, the physical specifications of the sample under investigation can be directly measured.

For instance, once defined the length standard: the meter, everybody knows what measuring a sample length means.

Relative measurements are those where the obtained datum is not directly related to the physical condition of the sample under examination but, for instance, it is an expression of a property of the material that does not fall under direct observation. Of course, in this case standards and methods can be the most varied. For instance, hardness measurement defines the relationship between the penetration of the inching tool and an assumed number intended for classifying the hardness property. Just think about the various methods: Brinnel, Rockwell, Vickers, ... Each one of these methods makes use of different measurement systems, based on their own relative scale, features a specific area of application, expresses properties that are similar, but not exact1y the same, and sometimes for the same material measurements are not comparable.

The impact strength value or diamond, expressed by the F.I. friability index, is a repeatable, predictable and relative measurement. It expresses a diamond property that cannot be directly evaluated with physical parameters, but must be set in relationship with the average or combinations hardness-resiliency of a limited number of diamond grains assumed as representative sample of the material under investigation.

Let us assume to consider a diamond sample consisting of a sufficient1y large number of crystals sharing similar shape, dimensions and quality and to undergo it, under controlled conditions, a series of destructive impacts. Crystals are assumed to have uniform quality when they contain in the same number, dimensions and positions: carbon inclusions, joints and graphitizings along the cleavage planes.
From an experimental standpoint the variation in the number of unbroken crystals is in accordance with the statistical equation

$$dN = -KNdE \qquad [1]$$

where

dN	-	variation in the number of unbroken crystals
N	-	number of sound crystals
dE	-	variation in the destructive impact energy
K	-	constant of impact diamond breakage probability.

The **–** sign proves that as the breakage energy increases the number of unbroken crystals decreases.

Summarizing the whole thing, when diamond crystal are broken by impact a relationship exists between breaking energy and number of unbroken crystals, and this relationship depends on a K factor that can be experimentally determined, and if the material is uniform it is constant.

Integrating, and calculating the ancillary conditions, a law is obtained, that gives the number of unbroken crystals as a function of the breaking energy supplied to the system

$$N(E) = N(0)e^{-KE} \quad [2]$$

where:

$N_{(E)}$	-	number of unbroken crystals as a function of the destructive impact energy E
$N_{(0)}$	-	number of originally unbroken crystals
E	-	destructive impact energy
e	-	base of natural logarithms (2.718..).

Let us assume now that the diamond sample consists only of perfectly octahedral crystals with the same dimensions and uniform quality.

Hence, all diamonds feature the same density and therefore a weight unit (i.e. 1 mg) always contains the same number thereof.

Based on this assumption the number of crystals can be simply "counted" weighing them(according to the same principle of the piece counter balance), thus formula [2] becomes:

$$P_{NF} = P_T \cdot e^{-KE} \quad [3]$$

where:

P_{NF}	-	weight of the uncrushed diamonds
P_T	-	original diamond weight

It should be now considered the way in which the crushing energy is supplied to the diamond.

The system experimentally consists of a capsule with a suitably shaped inner cavity, into which the diamond is placed together with a ball.

The capsule is high-frequency oscillated in the direction of the larger axis of the cavity and hence at each oscillation (back and forth) the hall is violently projected against the bottom capsule walls "squashing" the diamond that is between the wall and the ball.

At every oscillation two impacts are produced between the ball and the diamond grains.

We can hence assume that at each impact the diamond is given a small constant quantity of breaking energy, and thus, by further simplification, since it is easier experimentally counting the oscillation cycles with respect to every single impact, we can replace the energy value with the number of shaking cycles, of course adequately changing the K constant.

The formula [3] can thus be re-written

$$P_{NF} = P_T \cdot e^{-HC} \qquad [4]$$

where

C	-	shaking cycles
H	-	new constant of probability of diamond impact breaking setting the relationship between shaking cycles and the destructive impact energy

Fig. 1 shows the curve expressing the trend of formula [4].

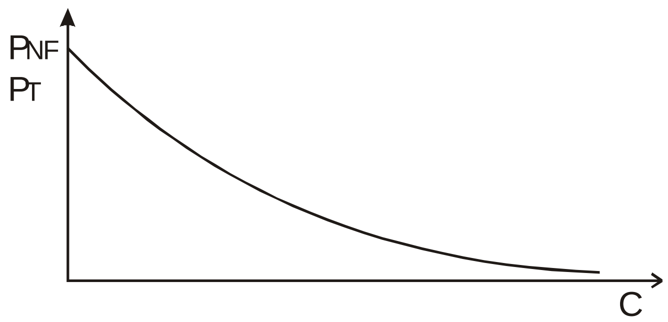


Fig. 1

It can be observed how PNF features an exponentially decreasing trend as a function of the shaking cycles.

The above reported law should be a more theoretical exercise if it was not experimentally observed that synthetic diamonds can be produced and selected, having different impact strength, and this practically allows for classing the various diamond commercial grades. This difference is mathematically expressed by different H values and hence different breaking curves result for every diamond quality; in detail, the „best„ diamonds feature - being the supplied energy equal - higher breaking curves, as shown in Fig.2.

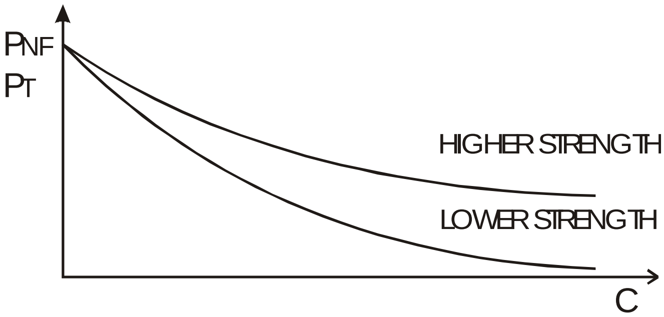


Fig.2.

So determining H permits to know the „quality „ of the diamond under examination. From [4]

$$\frac{P_{NF}}{P_T} = e^{-HC}$$

therefrom

$$\ln \frac{P_{NF}}{P_T} = -HC$$

and, based on the logarithm properties

$$\ln \frac{P_T}{P_{NF}} = HC$$

therefrom

$$H = \frac{\ln \frac{P_T}{P_{NF}}}{C}$$

The study of function [5] shows how 1-1 decreases as the diamond „quality„ increases, tending to 0 the more the diamond withstands impact breaking. Even if mathematically correct, this trend is but poorly practical because opposite to the usual concept of qualification, that gives increasing values to the improvement of properties. This is why it was preferred to define the F.I. friability index as the opposite of formula [5]

$$F.I. = \frac{C}{\ln \frac{P_T}{P_{NF}}} \qquad [6]$$

It was thus defined a number (F.I.) that qualifies the diamond as a direct function of the capability to withstand impact breaking.
Of course, should the above number be used as it was defined, it is not possible to compare data surveyed by different measurement systems in that the value scale depends on the geometric and operating conditions of the testing equipment.
Furthermore, even it is true that every point surveyed on the curve permits to calculate F.I., when PNF value is low the error involved by weighing excessively affects the results, generating too poor accuracy in F.I. determination.
To remedy these limitations an arbitrary multiplication factor and a PNF range within which measurements are reliable were defined.
Taking a sample with original quantity to be undergone breaking PT = 400 mg ± 0.5 %, it was observed that the F.I. determination is sufficiently accurate, with the adopted weighing technique (accuracy ± 1 mg) when PNF = 200 mg ± 5 %.

It was then resolved to establish for F.I. the condition according to which:

F.I. = 100 when the uncrushed diamond PNF is equal to 50% of the original diamond PT, and the number of cycles required for this condition to take place is C = 2500.

From the above conditions it results

$$F.I. = \frac{0{,}027726 \cdot C}{\ln \frac{P_T}{P_{NF}}} \qquad [7]$$

Replacing in formula [7] the values PT = 400, PNF = 200 and C = 2500 it results F.I. = 100. The problem to be faced now is how determining the number of cycles C in which crushing of *50%* of the original diamond takes place.
lt must then be defined a procedure for determining the *C5O* value, i.e. the number of cycles by which 50% of the material is crushed, in case that with the first measurement PNF does not fall within the acceptable range.

After a first determination with a number of cycles arbitrarily set as a function of grain size distribution and of the natural or synthetic diamond type, PNF is weighed and F.I. calculated.

Since F.l. is a constant, by a simple equation it results

$$\frac{0{,}027726 \cdot C}{\ln \frac{P_T}{P_{NF}}} = F.I. = \frac{0{,}027726 \cdot C_{50}}{\ln \frac{P_T}{50\% P_T}}$$

therefrom with simple algebraic passages it results

$$C_{50} \cdot = \frac{0{,}693148 \cdot C}{\ln \frac{P_T}{P_{NF}}} \qquad [8]$$

Making use of formula [8] it is then possible, in case the first test did not result in PNF values within the preset validity range (200 mg ± 5%), calculating the number of cycles required to perform a second test in which the PNF value is as close as possible to 200 mg, and hence the calculated F.I. value is affected as little as possible by the errors involved by the weighing system.

A thorough examination of the theory on which the above mentioned measurement is grounded might arouse many doubts about its applicability to, and correspondence with an examination of commercial diamonds available on the market.

In fact, the properties requested to the diamond under examination are too many and too limitative, whereas the theory applied to the ball motion has too much simplified an approach; furthermore the interference in the process of the crushed diamond should not be neglected.

Nevertheless, considering the results achieved in 15 years of the commercially diamonds analysis, any more thorough procedure is not necessary.
The F.I. measurement using the RE.TEK. method, despite the significant simplifications introduced, was found to be reliable and repeatable, providing results that have effectively helped our customers to distinguish among the various diamonds available on the market.

The one and only attention is to be paid to the determination of the grain size of the diamond under examination, since this test is reliable and comparable only for whole grain size analysis values (i.e. 40/50 mesh) or down to half grain (i.e. 30/35 mesh).

2 FRIABILITY TESTER

2.1 GENERAL DESCRIPTION

The Friability Tester has been redesigned using today's most advanced electronic technologies (2005), while its original mechanics have remained unchanged. This upgrade has made the equipment even more versatile and now four standards are possible at the same time and not two as in the previous version. In addition, equipment operation is even more reliable and does not require any calibration checks.

The alternating movement of the capsule is generated by a piston connected to a connecting rod-crank system. This system is driven by a step motor, controlled by a specific electronic circuit, which ensures constant motion and absolute repeatability of the set cycles.

A magnetic sensor, activated by the crank counterweight, and thus independent from the motor control, displays the number of cycles completed, making it easier to check efficiency.

All functions of the Friability Tester are managed by a microprocessor, supplied with an auto-diagnostic program, which isolates itself while shaking cycles are being carried out.

2.2 HARDWARE

The Friability Tester consists of a special mechanics to ensure the necessary forces affecting the diamonds by means of the steel ball. The number of cycles can be preset.

For the measures of the TTI (Thermal Toughness Index) procedures we recommend the DiaHeat .

The special furnace is designed for the thermal treatment of industrial diamond grit under protective atmosphere up to 1100°C.

DiaHeat - Hardware:

Central part of the system is the sealable quartz tube which shields the sample from the outer atmosphere. Sample holder and the sealing for the related end of the quartz tube are build up as one unit, which is moveable on the central rail of the furnace. On the same rail also the heater block is moveable. These moveable parts make handling and treatment of the samples fast and easy. A microprocessor unit controls and stabilizes the heater temperature.

DiaHeat - Principle of operation:

For the determination of thermal strength of industrial diamonds a treatment between 800°C and 1100°C under protective atmosphere is necessary for the subsequent friability testing (TTI) or single crystal testing (CFF, CFS).
At first the heater control is set to the working temperature. Then a ceramic dish with a diamond sample is placed onto the sample holder and brought on the holder into the quartz tube. After closing the sealing the protective gas stream is allowed to flow through the tube. While the furnace is away from the samples place in the tube we have to wait 10 minutes for preheating and loading the diamond grit with Argon.
Then the furnace is moved over the sample for 20 minutes.
After treatment the furnace is moved away from the sample, so it can cool down for 10 minutes, until it´s taken out of the tube. Treatment of the next sample can start immediately.

and we recommend to use the ***Analytical Sieve Shaker AS200***

The AS 200 analytical shaker is used for grain size determination and separation by wet or dry sieving. It works on an electromagnetic drive. This drive assures a three-dimensional throw movement that distributes the sieving material evenly over the entire surface of the sieve. The advantages of this are high endurance, extremely quiet running and short sieving times with clean separation.

2.3 PRINCIPLE OF OPERATION

This method is commonly used to get a general information of the quality of a diamond sample. The value expresses a diamond property that cannot be directly evaluated with physical parameters, but must be set in relationship with the average of combinations hardness-resiliency of a limited number of diamond grains assumed as representative sample of the material under investigation.

2.4 TECHNICAL SPECIFICATIONS

Measurement range	:	500...7000 shaking cycles
Stability and repeteability	:	± 0.06 %
Operating temperature range	:	10 to 40 ^{0}C
Power supply	:	230 V, 50 Hz (optional 60 Hz)
Absorbed power		
Stand by mode	:	37 W
during the cycle	:	90 W
Protection class	:	IP 54
Dimensions	:	405 x 255 x 195 mm (approx.)
Weight	:	approx. 31 kg

Distributed by : **Vollstädt-Diamant GmbH**
Kiefernweg 7
D-14554 Seddiner See, Germany
Tel: +49-33205-74620, Fax: +49-33205-74621
email: service@vdiamant.de, web: www.vdiamant.de

3 CONTROLS

3.1 BASIC OPERATING ELEMENTS

POWER Illuminated power switch

START Shaking cycle start button

EMERGENCY STOP Self-retaining emergency stop button. Turn the button clockwise to release it. If the button is pressed while the device is carrying out the haking cycle, the motor will stop, performing the braking ramp. If pressed during stand-by, it prevents the shaking cycle from being carried out, disabling the START control.

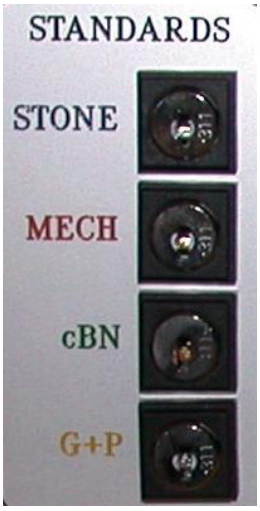
SHAKING KEYBOARD Used to set (PRESET) the shaking cycles. It is disabled while the shaking cycles are being carried out. The number of cycles is set (PRESET) by pressing the buttons in sequence, waiting no more than two seconds between pressing operations. Two seconds after the last button is pressed, the data entered are acquired permanently, until a new setting has been made.

3.2 ADDITIONAL OPERATING ELEMENTS - Version ST4

Version ST4 can be used to measure the friability index in diamonds, with significant differences in impact strength, and in cBN. With the basic version ST1, as explained in the first section of the special warnings, tests cannot be performed on diamonds whose friability index is less than 20 because it is not possible to set less than 500 cycles.

However, there are many types of diamonds on the market with lower friability values, diamonds used for applications that require abrasives, and binders, which are "softer" than those used to work stones.

Over the years, to meet the specific requirements of some customers, we have developed three new standards to perform comparative impact strength tests on these materials. These standards are now all available in version ST4. In particular, the equipment can perform tests on the following types of materials:



Version ST4:

-Diamonds for working stones

-Diamonds for mechanical operations

-CBN

-Diamonds for working glass and polishing

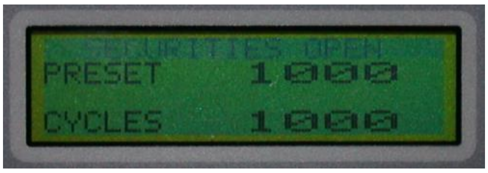
Standards can be varied by reducing the impact energy of the ball, i.e. the motor rotation frequency. The frequency is reduced gradually with regard to the standards indicated above, in other words, from the highest STONE to the lowest G + P.

To select the required standard insert the key supplied into the corresponding switch, one of the four on the left of the SHAKING, and switch it by turning it clockwise. Once the switch has been commuted, the key cannot be removed.

If no standard is selected (the key is not inserted or is not turned), the equipment operates with the STONE standard.

If two keys are deliberately inserted, i.e. the one supplied and the extra one, the equipment will operate with the lowest standard, e.g. for STONE and cBN, the equipment operates with cBN.

3.3 SHAKING DISPLAY



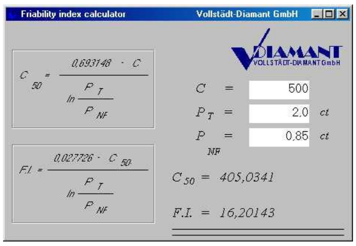
There are four lines of information.

LINE 1	Malfunction information. Flashing.
SECURITIES OPEN	The plug is open and/or the EMERGENCY STOP button is pressed. Check and reset normal conditions.
DRIVER FAULT	Indicates an electronic malfunction. Contact the manufacturer.
MECHANICAL FAULT	Indicates a mechanical malfunction. Contact the manufacturer.
LINE 2 (PRESET)	Indicates the number of cycles set. The PRESET must range between 500 and 7000. For settings outside the range, the relative alarm message will be displayed on line three after two seconds. The number of cycles set remains in memory even when the equipment is turned off and powered back on.
LINE 3	Incorrect PRESET setting information. Flashing. When the following messages appear, the START control is disabled.
UNDER RANGE	The PRESET value is less than 500.
OVER RANGE	The PRESET value is more than 7000.
LINE 4 (CYCLES)	Indicates, in real time, the number of cycles executed. It clears when the equipment is turned on or at the beginning of a new cycle of shaking operations controlled by START.

3.4 ACCESSORIES

- ***Measurement capsule and 100 balls***

- ***Torque wrench***



- ***Friability index Calculator***

  The *Friability index Calculator* is a special software to this calculate of F.I. and

3.5 ADDITIONALLY NEEDED ACCESSORIES

Analytical Sieve Shaker

special furnace - DiaHeat

Sieves

Electronical balance

3.6 INSTALLATION

Check that the power outlet is equipped with an efficient ground and that the line is not subject to disturbances and strong voltage variations.

It is recommended to install the Friability Tester on an insulated and very stable surface.

- Remove the Friability Tester and accessories from its packing.
- Insert the power cord into the socket in the rear of the equipment.
- Insert the three-pole plug of the aforementioned power cord into a power outlet with ground.

3.7 PERFORMING TESTS

After using the illuminated POWER switch to turn on the Friability Tester, refer to chapter 5 – FRIABILITY INDEX MEASUREMENT, PERFORMING TESTS section.

3.8 SPECIAL WARNINGS

The CYCLES digital indicator displays the number of cycles completed under normal operation conditions, without counting the cycles carried out during the acceleration and deceleration phases that, in any case, are very small and thus do not have any effect on the F.I. calculation. To minimize the error, the PRESET should not be set to less than 500 cycles. This restriction, electronically set, involves loss of accuracy in the correspondence between the set shaking number and the number of actually performed shaking, hence it is possible, even if not always, that the shown number - CYCLES - is different fro the preset number - PRESET - of a unit.

While carrying out a shaking cycle, if it becomes necessary to stop the motor for any reason, only the EMERGENCY STOP control can be used.

Do not turn off the equipment while the motor is turning.

The safety plug located on the front panel acts on a microswitch that checks that the plug is correctly screwed into the equipment, and, therefore:

If the safety plug is not screwed into its seat, it inhibits the START control, and, on the first line of the shaking display, the words “SECURITIES OPEN” will appear.

If the safety plug is unscrewed from its seat while carrying out a shaking cycle, it triggers the cycle stop control.

4 MEASUREMENT CAPSULE

4.1 GENERAL

The measurement capsule is the core of the measurement system. It contains the calibrated cavity within which the diamond is subjected to destructive impacts with the ball. The dimensional stability of the cavity, and the construction precision, are indispensable to ensure that measurements are repeatable.

The gas nitrided undeformable steel capsule is supplied already preconditioned based on a special procedure designed to prepare the measurement chamber and to ensure that it is uniform.

The same serial numbers are engraved on the casing, the plug and the pad inside the capsule to avoid having to replace the various components when using multiple capsules.

The capsule is equipped with 101 selected tempered steel balls, which must be changed after each measurement. Therefore 100 tests can be performed (one ball is a spare), a limit within which, in general, the dimensional stability of the calibrated cavity guarantees measurement repeatability with an error of less than one percent.

4.2 CAPSULE CLEANING

The capsule must always be carefully cleaned before and at the end of each test.

To clean as thoroughly as possible, rub clean fine crepe paper carefully and repeatedly: - on the fine thread outside the body, - on the internal thread of the plug, - inside the body's inner chamber, and in particular on the pad striker surface, - and on the cylindrical and concave parts of the pad.

To make it easier to remove the diamond microdust from the inner cavity of the body, carefully turn the body upside-down over a surface covered with crepe paper.

Do not use compressed air to blow on the parts of the capsule unless you are absolutely certain that the air does not contain any oil droplets

4.3 SPECIAL INSTRUCTIONS

Do not use the capsule for more than 100 measurements. Preventive tests have shown that after 150 tests, the F.I. calculation error, due to an alteration of cavity geometry, is 8 %, and 12 % after 200 tests.

Always check that the plug is fully screwed onto the capsule body and that the torque wrench doesn't click due to seizure of the threads caused by diamond microdust. In this case, carefully unscrew the plug and, after having extracted the pad and removed the ball and the diamond, again carefully and thoroughly clean the various parts of the capsule.

5 SAMPLE PREPARATION

5.1 DIAMOND SAMPLE PREPARATION

The careful preparation of the diamond samples is a basic condition for both the repeatability of measurements and a correct comparison of the F.I. values surveyed with known commercial samples values, assumed as standards.

Make sure that the grain size distribution of the prepared samples cover the entire considered range.

To be sure of the above, during sieving at least some grains must remain onto the larger sieve (smaller mesh number) and at least some grains must pass from the smaller sieve (larger mesh number).

1) Weight 1 to 1.2 grams of diamond to be examined.
2) Place the larger mesh sieve (smaller mesh number) onto a sheet of paper.
3) Pour the diamond onto the sieve.
4) Take the sieve between the forefinger and the thumb of the left hand, slightly inclining it upwards from the palm side.
5) Making use of the brush supplied for sieve cleaning, gently tap rhythmically the cylindrical sieve case in the transversal direction. If the operation is correctly performed, the diamond, can be seen, if not passing through the meshes, getting over the sieve accumulating in its upper section.
6) Turn the sieve - keeping it level - by 180° in the transversal direction and grab it again as directed under item 4).
Now the diamond is accumulated in the lower sieve section.
7) Repeat the Operation as directed under item *5).*
8) Repeat the operations outlined under items 4), *5),* 6) and 7) cyclically until when no more grains pass through the sieve meshes. As previously stated, at least some grains must remain over the sieve. If not, sampling cannot be regarded as effective, and operations must be repeated with the sieve immediately smaller (mesh number immediately higher). Of course in that case the range under examination must be reviewed, and it cannot be at any rate the one originally set.
9) Temporarily remove the diamond passed through the sieve collecting it on the sheet of paper.
10) Place the sieve upside-down on a new sheet of paper and, with the aid of the brush supplied with the unit, clean the sieve meshes from residual grains, if any, restrained therein, thus collecting the entire diamond fraction not passed through the sieve. This Operation must be carried out very gently carefully avoiding to beat the mesh with stiff objects or to touch it with the fingers.
11) Set apart the diamond collected as directed under item 10, that exceeds the set measurement range.
12) Place the smaller mesh opening sieve (larger mesh number) onto a new sheet of paper.
13) Pour the diamond fraction passed through the larger sieve and temporarily set apart (item9) on the smaller sieve.
14) Take the sieve between the forefinger and the thumb of the left hand, slightly inclining it upwards from the palm side.

15) Making use of the brush supplied for sieve cleaning, gently tap rhythmically the cylindrical sieve case in the transversal direction. If this operation is correctly performed, the diamond can be seen , if not passing through the meshes, getting over the sieve accumulating in its upper section.

16) Turn the sieve - keeping it level - by 180° in the transversal direction and grab it again as directed under item 14). Now the diamond is accumulated in the lower sieve section.

17) Repeat the Operation as directed under item 15).

18) Repeat the Operations outlined under items 14), 15), 16) and 17) cyclically until when no more grains pass through the sieve meshes. However, as previously stated, at least some grains must remain over the sieve. If not, sampling cannot be regarded as effective, in that there is no certainty that the diamond grain dimensions cover the entire considered range. The above may mainly take place for the grain ranges 30 to 40 and 40 to 50 mesh. In this case the examined diamond mainly consists of the 30 to 35 and 40 to 45 mesh grain ranges.

19) Temporarily remove the diamond collected on the sheet, that makes up the fraction passed the considered sieve mesh.

20) Place the sieve upside-down a new sheet of paper and, with the aid of the brush supplied with the unit, clean the sieve meshes from residual grains, if any, restrained therein, thus collecting the entire diamond fraction not passed through the sieve. This operation must be carried out very gently carefully avoiding to beat the mesh with stiff objects or to touch it with the fingers.

21) The collected diamond makes up the sample for the friability test.

5.2 <u>PREPARATION OF THE PNF SAMPLE</u>

Upon completion of the shaking cycles, as directed under chapter MEASUREMENT OF THE F.I. FRIABILITY INDEX, the uncrushed diamond must be separated from the crushed fraction. To that purpose, proceed as directed:

1) Place the smaller sieve (larger mesh number) onto a sheet of paper.

2) Unscrew the measurement capsule plug and gently pour the diamond and the ball onto the sieve.

3) Remove the ball

4) Take the sieve between the forefinger and the thumb of the left hand, slightly inclining lt upwards from the palm side.

5) Making use of the brush supplied for sieve cleaning, gently tap rhythmically the cylindrical sieve case in the transversal direction. If this operation is correctly performed, the diamond can be seen, if not passing through the meshes, getting over the sieve accumulating in lts upper section.

6) Turn the sieve - keeping it level - by 180° in the transversal direction and grab it again as directed under item 14). Now the diamond is accumulated in the lower sieve section.

7) Repeat the Operation as directed under item *5).*

8) Repeat the operations outlined under items 4), 5), 6) and 7) cyclically until when no more grains pass through the sieve meshes.

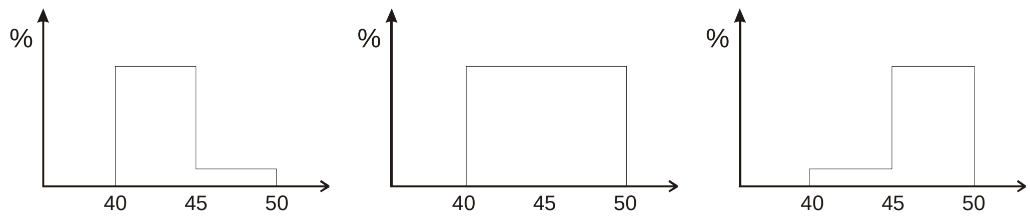
9) Temporarily remove the crushed diamond collected on the sheet.

10) Place the sieve upside-down on a new sheet of paper and, with the aid of the brush supplied with the unit, clean the sieve meshes from residual grains, if any, restrained therein, thus collecting the entire diamond fraction not passed through the sieve. This operation must be carried out very gently carefully avoiding to beat the mesh with stiff objects or to touch it with the fingers.

11) The uncrushed diamond fraction gathered under item 10) makes up the PNF.

5.3 SPECIAL INSTRUCTIONS

- To the purpose of correct test interpretation it was implicitly assumed that the percent distribution of the single grains within the grain size distribution range under examination is constant, even if this is not always true since any producer can develop the most varied distribution. For instance, the 40 to 50 mesh grain cannot be created with the more varied proportions of the 40 to 45 / 45 to 50 mesh grains as shown hereinafter.



  Furthermore, there is no proof that for the semi-grains no diamonds with different F.I. were used. This is an additional reason for which it is required to perform tests with as strict as possible grain size distribution.
- In the presence of non-homogeneous commercial samples (i.e., 35 to 50 mesh or 45 to 60 mesh, etc.) to carry out a correct comparative test sample must be divided into every single grain size range (i.e., for the 45 to 60 mesh, into the 45 to 50 and 50 to 60 mesh) performing tests for every fraction.
- It is strongly recommended that sieves are stored in a sheltered place, free from dirt, never touching wire meshes with the fingers.
  Sieves shall be cleaned wiping them with a brush soaked into suds, next rinsing with plenty lukewarm water, then with distilled water, drying them up in oven at a temperature not exceeding 40 ^{0}C.

6 FRIABILITY INDEX MEASUREMENT

For correct evaluation of the friability index F.I. it is essential to determine experimentally the number of cycles C by which 50 % of the examined diamond is crushed.

The F.I. value ca1culated by means of tests not complying with this condition is to be regarded as merely indicative, and its possible deviation with respect to the real value can be as higher as larger the deviation of uncrushed diamond weight PNF is from 50% of the total weight PT.

Tests can be carried out for two purposes:

A) checking the F.I. value in diamonds with known quality

B) determining the F.I. value in diamonds of unknown quality.

In case A) being the quality of the investigated material known, one test is sufficient. In fact, it is enough to set on the Friability Tester the number of cyc1es C50 previously determined on other diamond specimen with the same quality, and to perform shaking cycle. Should the weight of uncrushed diamond PNF be equal to 50 % ± 5 % of total weight PT, the examined diamond can be c1assified as standard (F.I. may vary by ± 7 % as a maximum).

In case B) procedure is by trials, and therefore at least two measurements are required, except where 50 % crushing takes immediately place. First measurement is aimed at roughly determining the diamond quality, whereas the second precisely determines the F.I. value. Should the first determination largely deviate from 50 %, a third test may be required to determine the correct F.I. value.

6.1 PERFORMING TESTS

1) Fill-in the FRIABILITY TEST table (**6-**2) with the relevant data to the test number, date, diamond type and/or any additional information useful for identification of the specimen, the grain size fraction.

2) Weight 1 to 1.2 g. of the diamond to be examined.

3) Pass sample through a sieve with the required mesh opening and collect the passed fraction. Make sure that you have slightly excesses the minimum 800 mg quantity required for 2 tests.

4) Weight 400 mg ± 0.5 % of the sieved sample (398 to 402 mg).

5) Report the exact weight value in the PT column.

6) Clean the measuring capsule.

7) Insert a new ball into the cavity, handling it with the fingers as little as possible.

8) Place the weighed diamond into the cavity, making sure that no crystals have remained on the inner striker surface. If so, shake lightly or use the brush supplied to make them fall into the cavity.

9) Close the cavity with the pad using the tweezers, and screw and tighten the plug. To do this, insert the capsule body, large thread side, into the torque wrench and tighten the plug until the wrench begins clicking. In this way you will always be certain of having applied the proper torque.

10) Extract the capsule from the torque wrench and reinsert it on the plug side.

11) Screw the measurement capsule onto the piston inside the Friability Tester. Then, screw the safety plug on the machine and make sure that the writing SECURITIES OPEN is no longer indicated on the first line of the SHAKING DISPLAY. If not, follow the instructions indicated on page 3-2.

12) Set the required shaking cycle number **(PRESET)**:

- if the diamond quality is known, set the previously determined value C, after having acquired the standard values from a special table on page (6-1) of the table section.
- if the diamond quality is unknown, set the C value obtained from the following table:

GRAIN SIZE	C (SYNTHETIC)	C (NATURAL)
30/40	1250	575
40/50	2000	750
50/60	2150	875
60/70	2250	900

The values of C in the first column currently (2005) relate to the values detected in synthetic diamonds suitable for marble cutting with an excellent quality. In this way, with the first test already, a general quality indication can be achieved for synthetic diamonds.

In fact, if:

- the weight of uncrushed diamond (PNF) subsequent to the shaking cycle ranges 190 to210 mg the diamond is of a quality very suitable for marble,

- the weight of uncrushed diamond (PNF) subsequent to the shaking cycle is less than 190 mg the diamond is of standard quality suitable for marble,

- the weight of uncrushed diamond (PNF) subsequent to the shaking cycle exceeds 210 mg the diamond is of quality suitable far granite.

13) Note in column C of the table the number of set cycles.

14) Press the START pushbutton of the Friability Tester to carry out the pre-set shaking cycle number.

15) Remove the measuring capsule from the Friability Tester and unscrew it with the torque wrench after unscrewing and removal of the relevant security plug.

16) Extract the capsule from the torque wrench and reinsert it on the large thread side, and unscrew the plug, gripping the torque wrench by the hex cap.

17) Place the diamond and the ball on the lower sieve (larger mesh number) of the size being analyzed. To make it easier to detach the diamond from the cavity of the capsule body, it can be struck with a wooden or plastic stick, turning it upside-down above the sieve.

18) Remove the ball and thoroughly sieve the diamond.

19) Weigh the diamond that did not pass through the sieve.

20) Note down in the PNF column of the table the exact weighed value.

There are two different alternatives:

A) The diamond weight (PNF) ranges190 to 210 mg.

Making use of the formula

$$F.I. = \frac{0{,}027726 \cdot C}{\ln \frac{P_T}{P_{NF}}}$$

calculate the value of the friability index and write it in column F.I. of the table. In this case test is completed, since the calculated value meets the condition required by the measurement theory. The C50 value, calculated by the formula given under item B) shall deviate from the C preset value by ± 7% as a maximum, therefore it is unnecessary to report it in the table

B) The diamond weight is less than 190 mg or in excess of 210 mg.

Making use of the formula

$$C_{50} \cdot = \frac{0{,}693148 \cdot C}{\ln \frac{P_T}{P_{NF}}}$$

calculate the number of theoretical cycles with which 50 % of diamond is crushed and write in column C50 of the table. In this case as well we can calculate the F.I. value by means of the formula reported under item A), but for merely indicative purposes only. If it is desired to write it into the F.I. column of the table, the applicable number should be followed by the F.C. (out of field) remark in order not to mix up exact values with approximate values

21) In case B), as stated in the introduction, a second test shall be performed, starting again from item 4), provided that enough material is available. Should this not be the case, start again from item 2). Though, as soon as item 12) is reached, a number of shaking cycles C must be set-up, equal to the theoretical C50 value, as it has been calculated by the formula reported under item B). In short, the C value of the new test coincides with the C50 value calculate by the previous test.

22) Proceed with the second test up to item 20). This test typically gives a PNF value ranging 190 to 210 mg, hence it is possible now, making use of the formula under item A), to calculate the correct F.I. value, noting it down in the special table column.

23) Should neither the second test produce crushing of 50 % ± 5 % of the examined diamond, but this fact happens very seldom, a third determination is required, to be carried out according to the procedure outlined starting from item B) of the item 20).

6.2 SPECIAL WARNINGS

Whenever during the tests for F.I. determination of an unknown diamond it turns out to be difficult to determine the correct C50 value, and PNF continue to change, and in the meantime it is not possible to steadily fall within the requested tolerance (190 to 210 mg), the above circumstances should lead you to suspect that the examined sample is a non-homogeneous mix of two or several very different diamond qualities, and that when weighing, conditions are produced, such as to have from time to time grains of one type prevailing versus the other.

This phenomenon is especially likely to occur when a mix of natural and synthetic diamonds is examined. Actually, based on how easily synthetic diamonds roll with respect to natural diamonds, if the utmost care is not exerted, weighing with different per cent ratios of the two types can result, with different F.I. values of course.

Should the quality of he diamond under examination be unknown, and if the quantity of diamond available is not large enough for several tests, we should recommend, in order to reduce the F.I. error when the latter is determined out-of-field to negligible values, to try to asses aforeside, by means of a microscope examination, even though this is not always probative, the specimen quality, and to set up, on the grounds of your own experience, a C value as close as possible to C50.

This method, unfortunately affected by the personal expertise, allows to make highly reliable evaluations by one test only.

When the typological analysis carried out, that is, the determination of the average F.I. and C50 values to be reported in the standard value tables, it is recommended to perform seven repeated measurements giving to C the value of C50 that was calculated from the previous measurement. In this way you shall remark that, provided that material is homogeneous, the PNF value tends to be closer and closer to 200 mg. Thus, the values from first two measurements are rejected and the arithmetic average of the remaining values is made. The average F.I. and average C50 values are assumed as typical values for that diamond quality, and reported in the standard value table (page 31).

Table TYPOLOGICAL ANALYSIS (page 32) of typological analysis also reports a schedule where weights of the different grain size fractions are to be reported, in order to allow far calculation of the per cent distribution of the different grains in the examined sample. These data are very important to correctly compare the two diamond samples when full-grain diamonds are being tested (e.g. 30/40 mesh).

Sometimes correct evaluation of diamond quality with some F.I. may require microscopic examination of the crushed diamond, that shall not show excessive presence of micro-powder. Actually, being the measured F.I. values equal, the behavior in diamond tools of diamonds that crushed in the customary way and those that break into micro-powder is definitely different.

If two diamonds with the same F.I. have different cutting reactions in the same tool, a bit of experience will make it possible, just by observing the broken dust through a microscope, to determine which of the two contains excess microdust, and therefore that it consists of a mix of two or more diamond qualities with very different F.I. values.

7 TI/TTI procedures (Example for 40/50 mesh powder)

7.1 TI

- Sieve the sample with sieves 40/45/50/60 mesh (other grain sizes according to the size)
- Take the fractions on the 45 and 50 mesh sieves, mix them.
- Weigh exactly 2,0 cts. from this fraction.
- Give these 2 cts. into the capsule of the friatest machine and put the capsule into the machine.
- Set the machine on 1600 cycles (for other grain sizes according to the operating instruction of the machine) and start it.
- After finishing the cycles, take the capsule out of the machine and empty it on the sieves.
- Sieve the samples thoroughly with the same sieves as above.
- Take the fractions remaining on all sieves (even the 60 mesh) and weigh it.
- The percentage of the remaining amount will be calculated (i.e. *100/2) and is the TI.

7.2 TTI

- Heat up an gas-tight tube-oven (protective gas Argon) to 1100°C.
- Sieve the sample with sieves as above (40,45,50,60 mesh)
- Take the fraction on the 45 and 50 mesh sieves, mix them.
- Weigh 5 cts. from this fraction.
- Put these 5 cts. to a porcelain boat and give it into the oven, at the colder end of the tube to heat up the sample slowly (10 min) and to cover it with Argon.
- Push the sample to the hot zone of the oven (under protective gas) and heat it 20 minutes.
- Pull the sample back to the end of the tube and cool it down (10 min).
- Open the tube and take the sample out of the oven, cool it further 10 min.
- Repeat the TI-procedure as described above, to get the TTI.

7.3 Friability Index (T50 or FI)

- After the TI procedure as described above. If the uncrushed residue is 50% (±5%) (i.e. 1 ± 0,05 ct.), you can directly calculate the Friability Index by formula:

$$T_{50} = \frac{0{,}027726 \cdot C}{\ln \frac{P_T}{P_{NF}}}$$

where: C_{50} - number of cycles, where 50% of the sample has been destroyed
P_T - total diamond weight
P_{NF} - weight of uncrushed diamond portion
ln - natural logarithm

- If the residue is out of this value, then you have to calculate the number of cycles, at which 50% of the sample should be destroyed, by the formula:

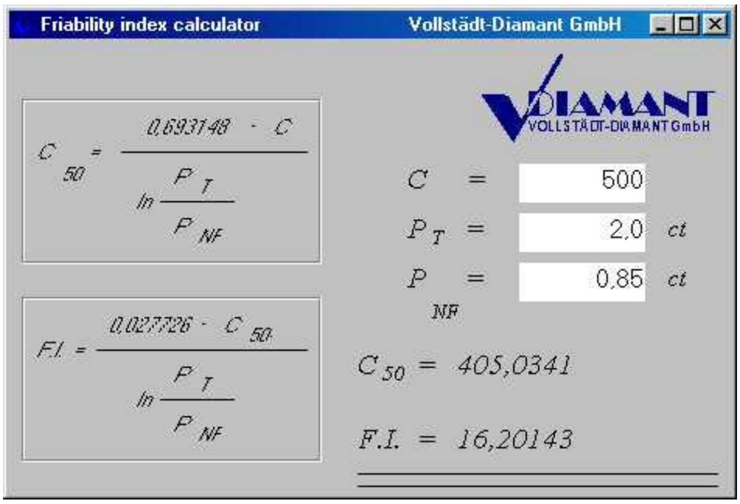
$$C_{50}. = \frac{0{,}693148 \cdot C}{\ln \frac{P_T}{P_{NF}}}$$

where: C - number of cycles performed

- Repeat the TI- procedure with this calculated C50 number of cycles.
- If the weight of the uncrushed diamond residue is now 50% ± 5%, then calculate the Friability Index as described above.
- If the weight of the uncrushed diamond residue is more or less, then calculate again the C_{50} and repeat the procedure until you get 50% ± 5% uncrushed residue and can calculate the T_{50} value.

Friability Test Table

Nr.	Date	Type	Size	C	P_T	P_{NF}	C_{50}	F.I.	Notes



OPERATIVE WEIGHTS AND ALLOWANCES

PT	**=**	**400 mg ± 0,5%**	**398 to 402**
PT	**=**	**200 mg ± 0,5%**	**190 to 210**

STANDARD VALUES TABLE

SIZE (mesh)

TYPE	**30/35**	**30/40**	**35/40**	**40/45**	**40/50**	**45/50**	**50/60**	**60/70**	
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									
F.I.									
C									

TYPOLOGICAL ANALYSIS

MESH	g	%

ANALYSIS N. ______________ OF ______________
TYPE ______________________________
F.I. ______________ C50 ______________

Size	C	P_T	P_{NF}	C_{50}	F.I.	Notes

TYPOLOGICAL ANALYSIS

MESH	g	%

ANALYSIS N. ______________ OF ______________
TYPE ______________________________
F.I. ______________ C50 ______________

Size	C	P_T	P_{NF}	C_{50}	F.I.	Notes