A new System for Single Particle Strength Testing of Grinding Powders

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A new automated system for the single particle strength testing of abrasive and superabrasive powders is presented. It is shown, how the system design meets the requirements for stable test conditions and automated operation. The size and shape parameters acquired by the digital imaging subsystem are listed. Single fracture experiments are used for the discussion of the force vs. time signal in the crushing subsystem. A flexible method for fracture data collection is derived from the experiments. Crush experiments using different anvil materials are used to show the interaction of anvil material and test specimen and their general influence on the fracture force. The calculation of compressive fracture strength is discussed with regards to irregular shaped particles. A collection of mean size and fracture strength values from different superabrasive and conventional abrasive powders in the size range from 325/400 to 40/50 mesh demonstrates the measuring range and the resolution of system prototype.

1. Introduction

Fracture strength, fracture behaviour or wear resistance are key properties of abrasive and superabrasive powders. Along with the particle size distribution, these parameters are crucial for the application of the material. Tight control of the properties is necessary for the manufacturing of high precision and high performance tools. The toughness index or friability index test procedures for superabrasives and ball mill tests for conventional abrasives have been well established for many years. These tests are applicable for a wide range of particle sizes, but depending on the toughness of the material, the parameters of the test environment have to be adapted. The method produces a quite stable integral result, representing several thousand single particles. But one has to pay the price for the stable result: the results are comparable only if measured under the same conditions, and the spread of properties among the single particles remains hidden.

Beside the dynamic toughness test procedures, the static compressive fracture test is well adopted especially for the description of man-made diamond [1,2]. The simple method of applying static uniaxial force onto a particle until it breaks can be realised in many different ways without changing the results very much. The insight into the strength distribution is considered the most valuable result of the test. Another advantage is the possibility to put all results onto the common fracture force scale, or, using the ratio of fracture force and particle area, onto a fracture strength scale. This makes the method attractive for the ranking of different types and qualities of superabrasives and conventional abrasives [3].

The currently available single particle fracture force testers are focused on the test of saw grit diamond; particles smaller than 0.25 mm in size cannot be processed by the semi-automatic machines [4].

The current paper will continue the idea of [5] overcoming the actual limitations with regards to particle size and force resolution along with the not exactly predictable anvil surface wear. It will describe the layout of a fully automated fracture strength tester, which combines optical size and shape analysis with a high resolution crushing apparatus.

2. Requirements for the testing machine and their influence on the system layout

The projected size range for the test specimen covers the finest grinding powder (325/400 mesh) as well as coarse grit (18/20 mesh). This makes the particle by particle handling more difficult than in the actual machines, where a carrier tape is loaded with a series of particles by means of linear sieves with different hole apertures. At the smallest particle sizes even the thinnest available carrier tape will prevent the particles from being crushed between the anvils. The semi-automatic separation method by positioning the particles through holes in a screen will also fail when the particles get too small. There is no other way out than the implementation of an automatic single particle pick-up-and-place procedure.

Size and shape measurement has to be carried out before the particle is moved onto the anvils. The size range in question requires a system with adjustable magnification and automatic focus capabilities. Monochromatic lighting with black/white cameras instead of white light and colour cameras will decrease costs and allow for highest resolution pictures.

Special precautions are necessary with regard to the crushing mechanism, especially the anvil surface. The compressive fracture force of perfectly-made coarse synthetic diamonds can reach 2000N and more; mean values of 1200N for a batch are possible. These high forces need to be generated by the system, but this is not a real problem. What will happen when the diamond gets as small as 50µm or less must be looked at in more detail. Of course the fracture forces will be much lower. To measure those values with good precision should not be a problem. The dramatically higher strength of the particle will have more influence on the measuring result, i.e. its fracture force in relation to the particles cross section area. Diamonds of 250 µm in diameter can show a mean fracture strength of 5,000 Nmm^{-2} in comparison to 2,500 Nmm^{-2} of good quality 600 µm stones. At the low end of the projected size range, the fracture strength could easily reach 10,000 Nmm^{-2}, with dramatic consequences on the anvil wear. While working in the saw grit range, the first types of DiaTest-S [2] used polycrystalline cBN as anvil material. With the upcoming high strength diamond qualities, this was changed to high grade polycrystalline diamond. Even these super hard anvils need to be observed carefully early enough in order to detect anvil surface degradation and its harmful influence on the measuring results. So the probability is high that even the most wear-resistant anvils will be damaged very early when crushing very small diamond. To prevent the ongoing anvil degradation from influencing the measuring result, the principle of “new anvil surface for every particle” was developed. The machine design must allow for crushing every particle on a new, undamaged part of the anvil surface.

The last block of planning was a more economical one. The above considerations require a machine with an automatic pick-and-place mechanism for the particles and an automatic feeding mechanism for the anvils to supply new anvil surface for every particle. Based on this hardware, the processing of one sample can then run without the operator’s interference. Only a few more technical prerequisites should be necessary for making batch processing possible. All that is needed is an automatic sample feeder along with an automatic supply of anvils. In this way every sample can be processed by its own set of anvils. The machine should now allow for the operator’s free processing of several samples in line.

The system software should provide a maximum of information, i.e. size and shape parameters, the particle pictures and the fracture information obtained from the crushing mechanism. The data should be stored in a standard database format in order to make the access as easy as possible.

The next chapter will show how the requirements are met with the system design.

3. System design

3.1. Image acquisition and digital image processing

The imaging system has to supply pictures with a lateral resolution of around 2% of the smallest particle size in question. A resolution of 1 µm per pixel meets this requirement and makes the size and shape values precise enough even for the finest powder. At the same time the field of view must be large enough to show coarse particles and a significant part of their neighbourhood. To meet both requirements, a system of two coaxial cameras using the same optical path and focus mechanism was chosen. The high resolution camera gives 1.2 µm per pixel resolution at 1280x1028 image size; the low resolution camera gives 4.5 µm per pixel on a 768x576 image. The imaging system works with transmissive monochromatic lighting and a motorized focus, both controlled by the program. Figure 1 gives an impression of the particle sizes from the smallest to the largest. Their appearance in the imaging system is shown in Figure 2.

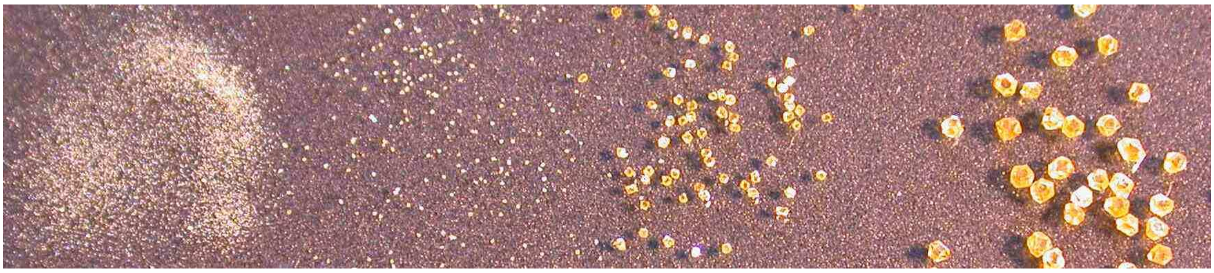

Fig 1. Diamond sizes from D46 to D1118

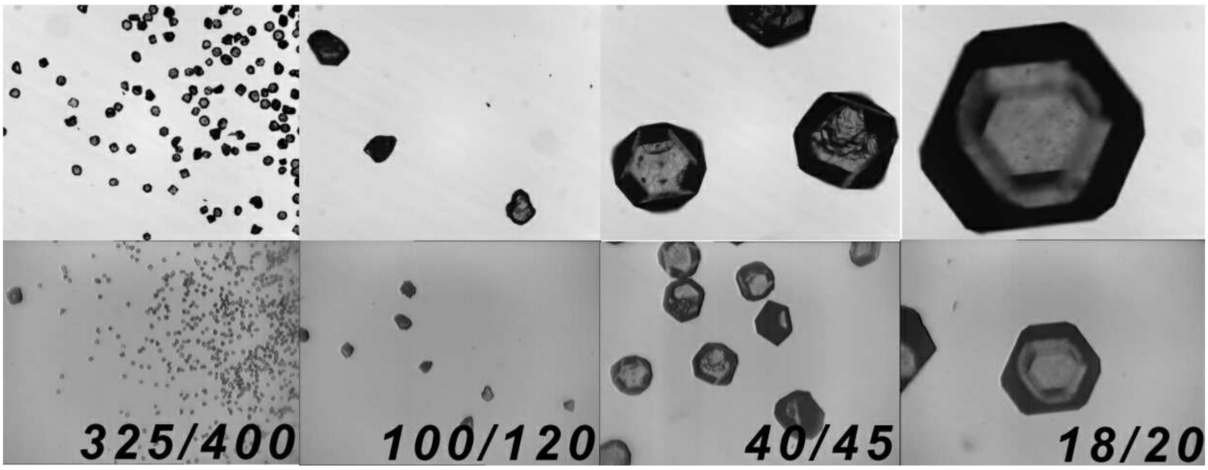

Fig 2. Digital images ready for processing in the program

The digital image processing part of the program performs the separation of the particles from the background, the measurement of the particles properties and the extraction of separated, single particles which are accessible by the pick-up mechanism. The detection of freestanding single particles is performed by first measuring the size and shape parameters for every object in the image and then comparing the results with a set of decision rules, which is specific for a certain size range and the product under test. The program also checks if the neighbour particles are not too close in proximity. Once a particle is selected to be picked up, its picture along with its digital parameters is stored into the result database. The database will be completed later on with the crushing results.

Table 1 gives the main shape parameters along with their calculation methods.

Table 1. Size and shape parameters acquired by digital image processing

Description	Method of measurement or calculation
Minimum feret diameter given in µm	Let a surrounding rectangle rotate around the object, record the minimum distance between two parallel lines of the rectangle
Diameter of the area equivalent circle	The diameter of a circle which has the same area A as the particle (useful for irregular shaped particles): $\sqrt{\frac{4 \cdot A}{\pi}}$
Convex Outline	Derived from the convex perimeter (p_c) and area (A) of the object, it is equal to: $\frac{p_c^{\,2}}{4 \cdot \pi \cdot A}$ A circle has the minimum convex Outline value (1.0).
Compactness	Derived from the perimeter (p) and area (A) of the object, it is equal to: $\frac{p^2}{4 \cdot \pi \cdot A}$ The more convoluted the shape, the bigger the value. A circle has the minimum compactness value (1.0).
Ellipticity	A very stable measure of the total symmetry of the object, derived from the moments of inertia: $\sqrt{\frac{MomentOfInertia_{Max}}{MomentOfInertia_{Min}}}$ An object like a square or a circle has the minimum Ellipticity value (1.0).
Feret Elongation	The ratio between the maximum and minimum feret elongation. A circle will have a value of 1, a square 1.4.
Roughness	A measure of the roughness of an object contour, it is equal to: $\frac{Perimeter}{convexPerimeter}$ A smooth convex object, such as a circle or ellipse, has the minimum roughness value (1.0).

The collection of shape parameters will be completed in future.

3.2. Sample preparation and handling

Agglomeration problems are not critical even at the low end of the grain size range. Therefore a simple spreading of the powder onto a glass slide by means of a small spoon is sufficient as a preparation method. The machine can hold up to 8 slides with different samples. The slide holder is mounted onto a XY planar drive which can move each of the samples into the viewing range of the optics. The planar drive is floating on a thin aircushion, which allows for absolutely wear-free operation. Its comparably low absolute positioning precision is compensated by an adaptive positioning control, which uses the object recognition capabilities of the imaging subsystem. The selected particle can be positioned with an accuracy of 10µm onto the pick-up point. The pick-up device itself serves as a needle with an adhesion layer at the contact point. The needle is mounted onto a rotation unit and can be moved up and down by means of a direct magnetic drive. Figure 3 shows the sample holder and the pick-up mechanism inside the optical system. The lighting unit is located below the sample holder; the focus lens is visible above. The program controls the focus level automatically.

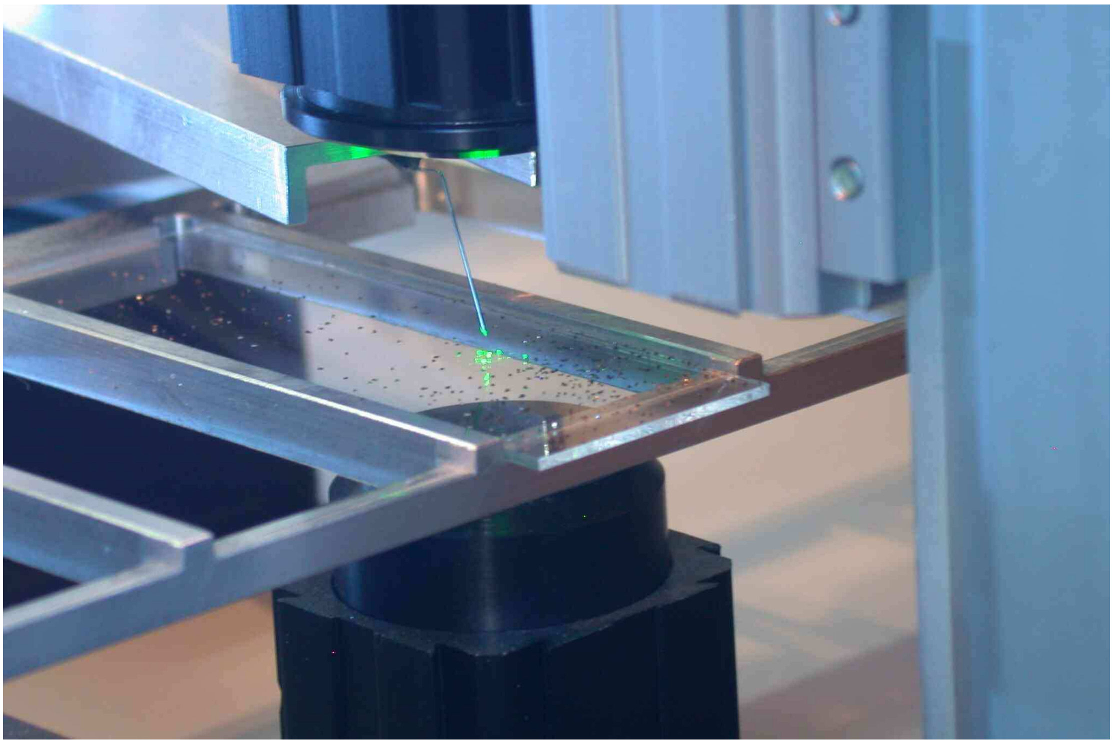

Fig 3. Sample holder, pick-up mechanism and optical system

3.3. Crushing unit and data acquisition

The crushing unit is equipped with 300 mm long anvil bars; the cross section is square shaped. The upper and lower anvils are mounted rectangular to each other. The overlapping area of the upper and lower anvils is the place where the specimen has to be placed. A pushing mechanism ensures that both anvils will move forward a bit after crushing a particle, thus offering a new surface part for the next particle. The step width of the anvil advance is program controlled and makes best use of the anvil material. The lower anvil bar is directly mounted onto one of the force sensors. Two of them with force ranges of 2000N and 200N are available in the system; the usage is controlled by the program. The upper anvil bar is mounted onto a pneumatic cylinder which acts as counter bearing during the crushing process. The lower anvil bar along with the force sensor is driven by a special combination of pneumatic and hydraulic cylinder, which can be switched from high movement speed to high stiffness at very low speeds.

This high stiffness along with a high resolution real-time data acquisition of the force is necessary for the reliable fracture detection of the specimen. The data acquisition subsystem provides 16 bit resolution with programmable measuring ranges. It samples the force sensor signal with 10,000 readings per second. The readings are corrected for the actual sensor excitation voltage and offset values in the software. Noise filtering is also applied digitally.

Figure 4 shows the lower anvil bar mounted on the force sensor, the needle placing a particle, and above, the upper anvil bar mounted onto the counter bearing. The second force sensor is visible on the right-hand side; the background shows one of the observation cameras.

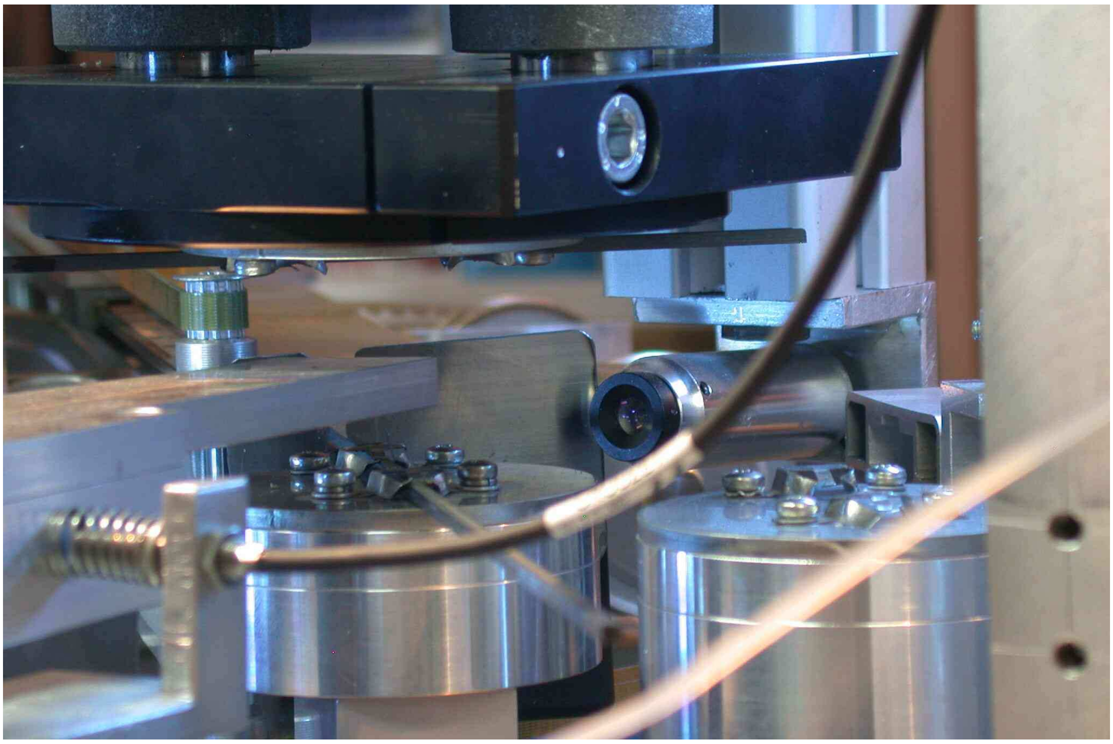

Fig 4. Crushing unit

4. Interpretation of force data

The current crushing system supplies a stream of force values, which can be interpreted as a force vs. time plot. A typical plot from the test of a blocky diamond is shown in Figure 5.

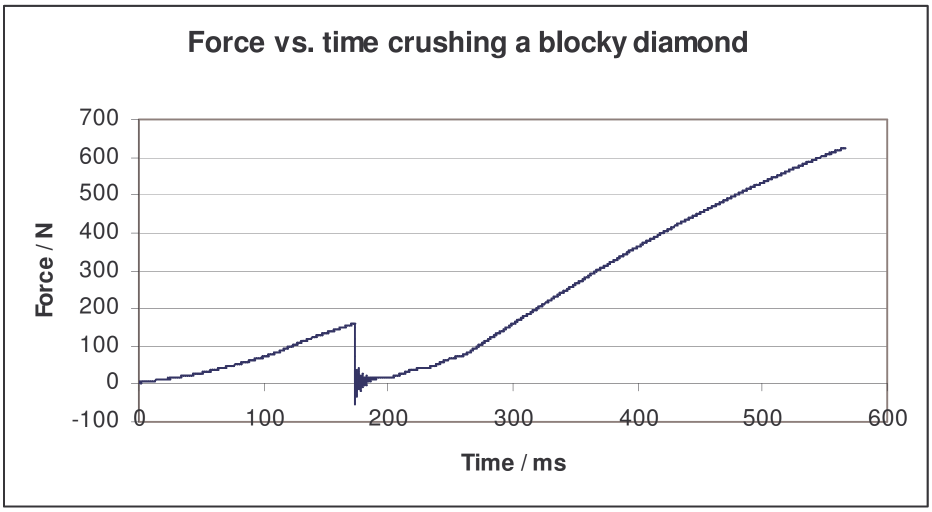

Fig 5. Force vs. time plot showing the fracture of a blocky diamond

It is clearly visible that after reaching a maximum force, the value drops suddenly when the crystal disintegrates. For a short moment there is no mechanical connection between the anvils, the force is nearly zero. The occurrence of that type of fracture is easily detectable. The compressive fracture force CFF is the maximum force recorded before the crush.

More attention needs to be paid to the interpretation of a force vs. time plot showing the fracturing of a corundum particle. Figure 6 might serve as a typical example for the data stream.

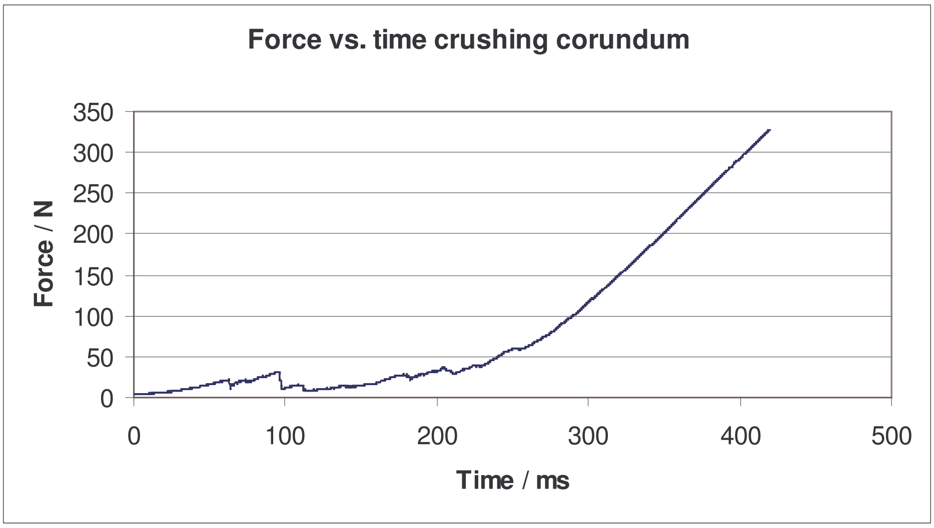

Fig 6. Typical force vs. time plot of the fracture process of corundum

In contrast with the fracturing of a blocky diamond, this shows a series of fracture occurrences. At first glance it seems that a small dent in the plot indicates that only a small piece of the particles has splintered off from the main part. A deeper valley might be caused by the destruction of a major part. To be more certain in this field, some of the corundum particles were crushed between two glass plates using a very stiff press. Pictures were taken after the occurrence of the first, the second, and the following cracks. Figure 7 shows the results of this experiment on one particle of sintered corundum.

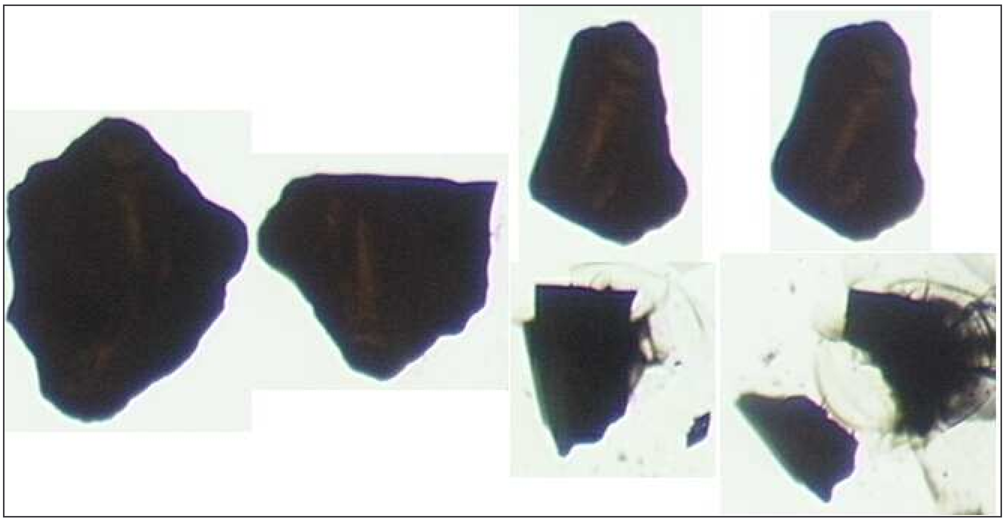

Fig 7. Stepwise fracturing of a particle of sintered corundum

It is visible that the first as well as the successive cracks indicate the break of one bigger particle into a few smaller ones. In most cases, the process shown in Figure 6 produces immediately a lot of fine debris, while the process in Figure 7 needs a series of fracture occurrences to destroy the whole particle.

From the above results, it was necessary to decide, which fracture detection algorithm should be applied by the software and which results should be stored in the database. There was no doubt that the first fracture had to be recorded, but for compatibility reasons with previous testing machines, the algorithm should also detect the sudden explosion of a blocky crystal. The count and the average value of successive fractures might also be of interest. At the moment the program records the compressive fracture force which causes the first fracture and also the force which caused the "explosion" of the specimen (if occurred). The extraction of some more information from the data stream seems possible and desirable. The authors will be thankful for recommendations concerning the data collection algorithm.

5. Influence of the anvil properties on the measuring results

It is well known that the interaction of anvil and specimen plays an important role in the fracture process. Ideal conditions for the fracture tests are flat, smooth and well-defined contact areas between anvil and specimen, while the anvils should be much harder than the specimen. This, of course, is hard to achieve when testing very hard material. It is known from the long-term usage of single particle strength testers with PCD anvils that ongoing anvil degradation results in dropping values for the compressive fracture force. Having a closer look at the anvil degradation it shows an increasing roughness of the anvil surface. As a result, the contact area between anvil and particle becomes smaller, and the stress is introduced more unevenly into the particle. As a result, the particle will reach the critical stress already at lower forces.

A similar behaviour will be observed when the anvil surface remains in the smooth and flat ideal state, but the particle surface becomes more uneven or rough. The particle will break earlier, at lower forces.

Assuming that the hardness of the anvil material is less or much less than that of the specimen, we should find the opposite effect. Small edges or lumps might be pressed into the anvil material. The stress is applied now much more evenly, causing the fracture to happen at higher force values.

The experimental results point in the same direction. A blocky diamond material (40/45 mesh), which was tested on PCD anvils with a median compressive fracture force of 300N showed on tungsten carbide anvil bars a median value of 360N. Two extremely different anvils were used for a test on corundum (size F60), namely tungsten carbide and brass. On the brass anvils the corundum showed median CFF values of 24N compared to 17N on tungsten carbide anvils.

Two conclusions can be drawn:

- comparable results can be achieved only when using the same anvil material
- the usage of different anvil material for tests of the same sample might deliver interesting insights into the contribution of the surface morphology to the fracture behaviour

Figure 8 and 9 illustrate the influence of the anvil properties on the fracture results. Both pictures of the particles footprints on the tungsten carbide and the brass surface were taken with the multifocus option of DiaInspect.OSM, which produces a high resolution 3D picture of an opaque surface. The intrusion depth of the particles is approximately 10 µm for the diamonds (230/270 mesh) on tungsten carbide and 65 µm for the corundum particle (F60) on brass.

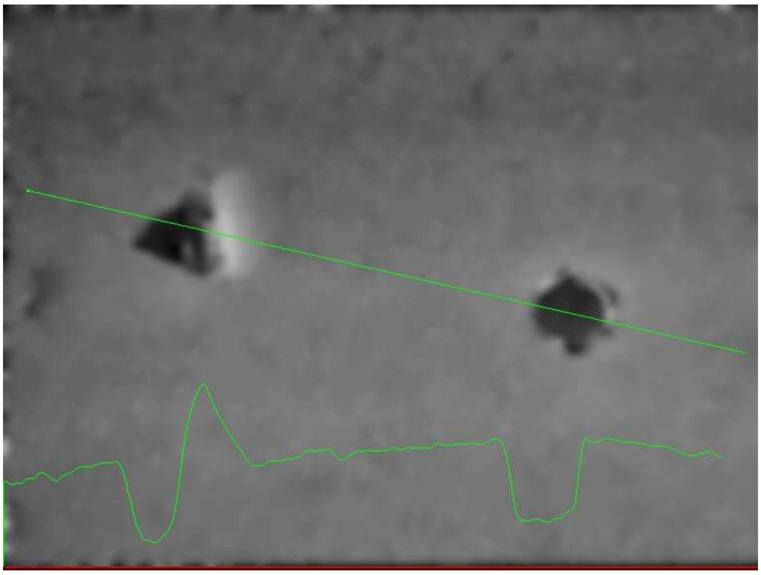

Fig 8. Footprints of diamonds (230/270 mesh) on tungsten carbide, intrusion depth approx. 10 µm

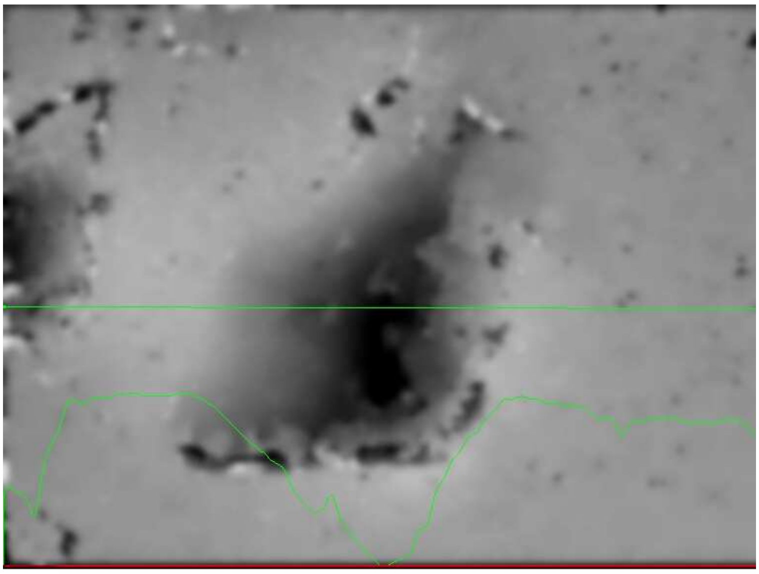

Fig 9. Footprint of a corundum particle (F60) on brass, intrusion depth approx. 65 µm

6. Results from different superabrasives and conventional abrasives

The prototype was used to test a series of different abrasives and superabrasives which covers almost the whole range of grain sizes which the system was designed for. For these first tests only the 2000N sensor was used. The investigation, if and how the increased sensitivity of the 200N sensor (along with its lower mass) should be considered in the detection algorithms, is not yet finished. Tungsten carbide served as anvil material.

The cameras and processing rules for the imaging subsystem were chosen according to the particle sizes and shapes of the samples. The size and shape information collected from the samples will not be shown and discussed here, because it is not specific to the current machine. It can be obtained in the same way using the separate particle analysers DiaInspect and DiaInspect.OSM.

For the presentation of fracture results it is useful to set the particle size as the X axis. For the Y axis we took the fracture force which caused the first fracture of the particle. All these values are medians from a batch of several hundreds of particles. Figure 10 shows the fracture forces for the first fracture in relation to the particle sizes.

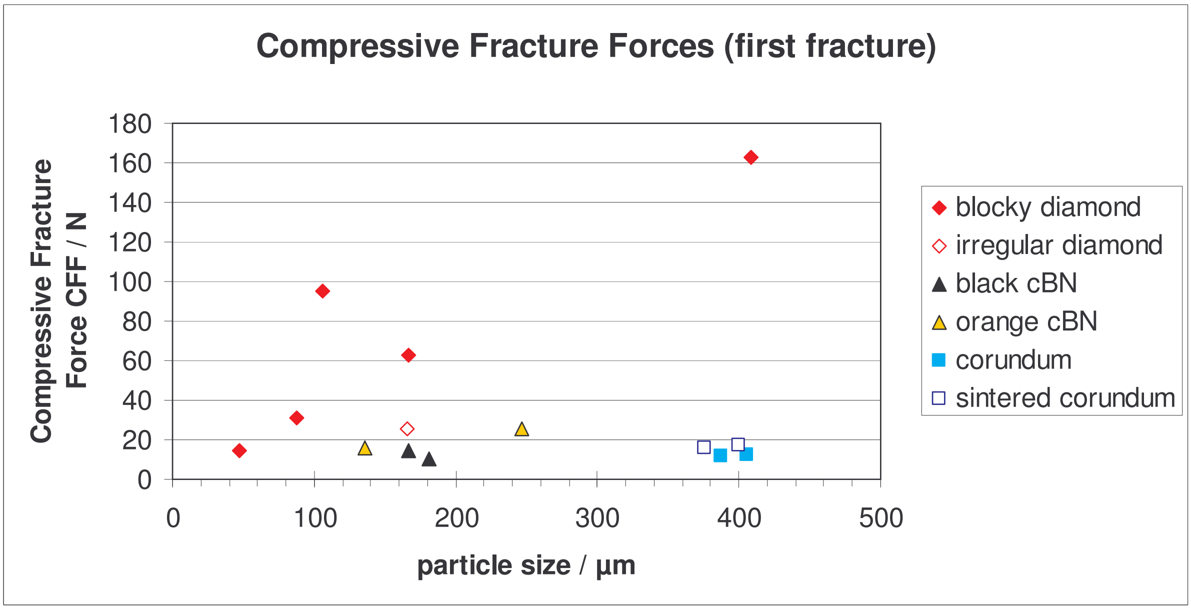

Fig 10. Compressive fracture forces for different superabrasives and conventional abrasives at different grain sizes, the first fracture is considered

The graph does not seem very impressive; most of the fracture force values are found in the range from 10 to 30N. The picture changes dramatically if the fracture force is used in relation to the particles cross section area.

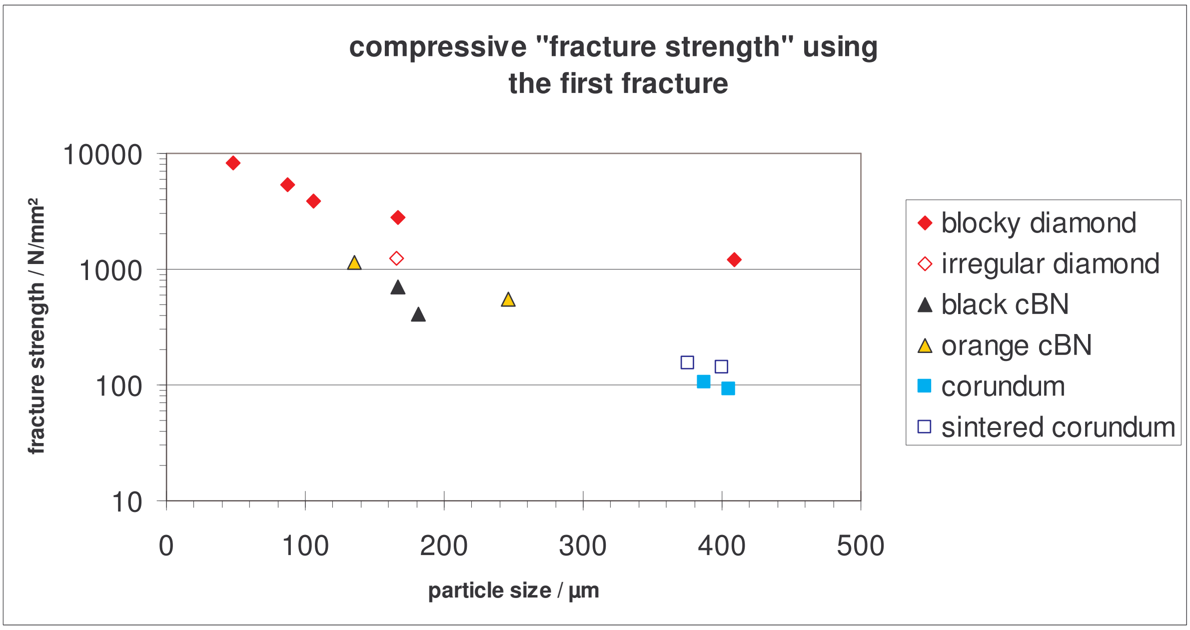

Fig 11. Compressive fracture strength for different superabrasives and conventional abrasives at different grain sizes, the first fracture is considered

The ratio of force and area is defined as the stress, and the maximum stress a material can withstand is its strength. In terms of material science, it is not correct to use the said ratio of fracture force and cross section area as a measure for the strength, because the exact distribution of the stress inside the particle is not known. The reasons were discussed already in conjunction with the influence of the anvils. Nevertheless, the cross section area is directly linked to the particle's mass and its volume. A better parameter is missing at the moment and therefore we use the cross section area to calculate a "Compressive Fracture Strength (CFS)", as imperfect as it might be. Figure 11 shows the plot of CFS against the particle size. A logarithmic scale for CFS is necessary to show the relations clearly.

The missing points for conventional abrasives at the low end of grain sizes will be filled in future using the high sensitive 200N sensor.

7. Conclusions and Outlook

The Abrasive Powders Single Particle Fracture Strength Tester (AfraTest) has been proven as an instrument for the measurement of fracture force and fracture strength in a wide range of abrasive and superabrasive products, covering the sizes from fine grinding powder up to coarse grit. The introduction of the principle "new anvil surface for every particle" gives experimental freedom for the deeper investigation of the fracture of irregular shaped material.

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