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Controlling the stability of the properties of superabrasive powders

Heiner Vollstaedt and Einar List
Vollstaedt-Diamant GmbH, Seddiner See, Germany

Abstract

The need for extensive and thorough quality control in the manufacturing, the distribution and the consumption of superabrasive powders is discussed.
The commonly used parameters for synthetic diamond are shown along with the appropriate equipment for the measurement.

Introduction

50 years ago the first man made diamond was created at the ASEA Laboratories in Stockholm. Since then industrial diamond remained to be an exotic and costly raw material for long time. The high price was compensated by the excellent characteristics of the material, thus opening new possibilities for machining difficult objects.
At the end of the last century the conditions changed dramatically when the diamond manufacturers from Asia have entered the global marketplace. As one result of the strong competition the price for the established brands of diamond powders dropped deeply. As a second result the new suppliers had to increase and stabilize the quality of their powders. And as third result the former comfortably high profit margins melted when the high tech product became partly close to cheap commodity /1/.
The market still grows and the competition between the diamond manufacturers is expected to continue in a battle on the fields of price, performance and quality, and - not to forget – customer care and technical support.
As an example of the really new situation may serve what the managing director of Element Six, Christian Hultner, said at the officially opening of the expanded diamond synthesis plant in Springs, South Africa“: „ .. we will continue to drive down costs to become the world’s lowest cost producer.“ /2/
The beneficiary of this process is the diamond tool maker and the construction industry. But at the toolmakers site it requires some effort to take advantage from the situation. New material has to be ranked into the known range of products, so that a quick thumb rule can judge about the price-performance ratio. Some experiments and an optimisation process will follow. At the end a recipe will be developed along with the set of process parameters and application rules for the tool.

From now on the raw material will influence the success of the manufacturing process via two factors:

- constant availability in time and quantity
- uniformity of the properties.

It becomes clear that uniformity and constancy of the product properties are at absolutely the same level of importance as its price / performance ratio. Looking at the European market these factors should be considered in a logical AND combination, if one of them fails then the product will not succeed.
Consequently all measures were taken to ensure an objective and precise evaluation of the product properties, based on the experience of the technical personnel, but without the variations in human decisions.
The following chapters will show the current state of measuring and test methods and the related equipment introduced to the market.
Regardless of the usability of methods and equipment for the characterization of other superabrasives and abrasives the paper will look at synthetic diamond only.

Properties and methods for measuring

When excluding all measuring methods not suitable for the „daily use“, there is only one basic parameter directly accessible for the measurement: the particle size. All other basic properties of the diamond particle are hidden for routine measurement. **Fig. 1** shows basic properties of the diamond and the viewing angle of common measuring techniques.

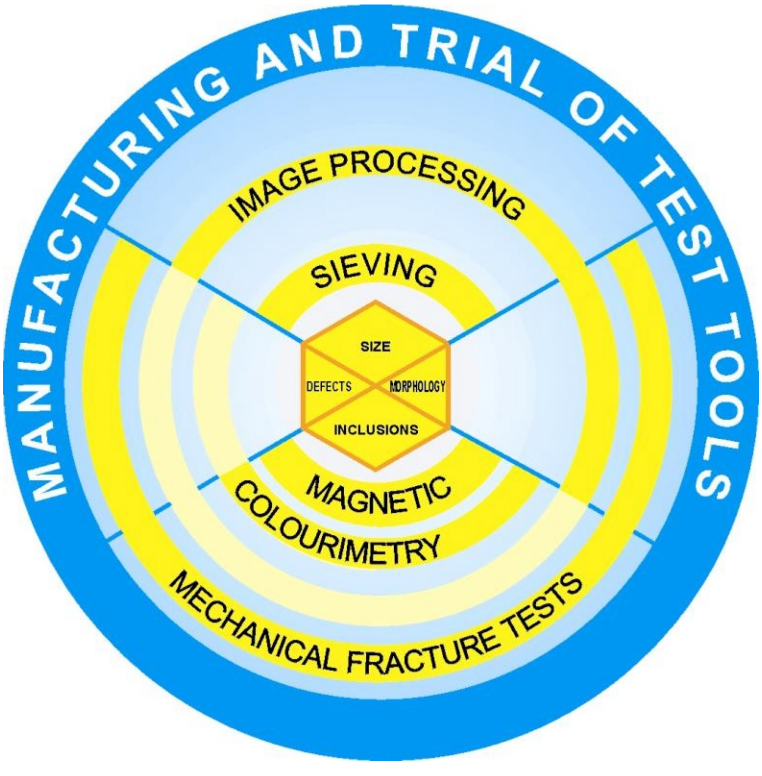

Figure 1 : Basic properties and the viewing angle of measuring techniques

It is clearly visible that none of the measuring principles or techniques can cover all aspects of the particle properties. The final shell in the picture which really integrates all particle properties in relation to their dedicated task remains the manufacturing and the trial of a test tool. But, as derived from the picture, there is no gap in the observation orbit around the basic particle properties. Every change or distortion in the basic properties will cause a measurable change in at least one of the observers outputs. This will early indicate that a different behaviour of the final product should be expected. Consequently, if changes and distortions in the material cannot be excluded for sure, the client will definitely make use of all affordable testing methods.

The following chapters will pass the available and commonly used devices revue. Remarks will be made regarding the ease of use, the resolution and the stability of the results.

Size measurement

Sieving as the standard procedure is well standardized and will not be discussed in detail. If the hardware is maintained carefully there is few chance to mismatch.
As an alternative image acquisition and processing systems are more and more in use. The main difference of the above methods is that sieving applies a set of two-dimensional criteria onto all orientations of all three-dimensional objects, while the image acquisition measures a dimension property on only one two-dimensional projection of every three-dimensional object. Without correction the results from sieving and the size check in the image show a systematic difference. The percentage of difference itself depends on the crystal shape and can be corrected by a fixed factor for a given type of material.
Size check based on image processing belongs to the single particle tests. In addition to the limit check also the particle size distribution is measured. **Fig. 2** shows particle size distributions for samples from different manufacturers, which have both passed the limit check for the 40/50 mesh range.

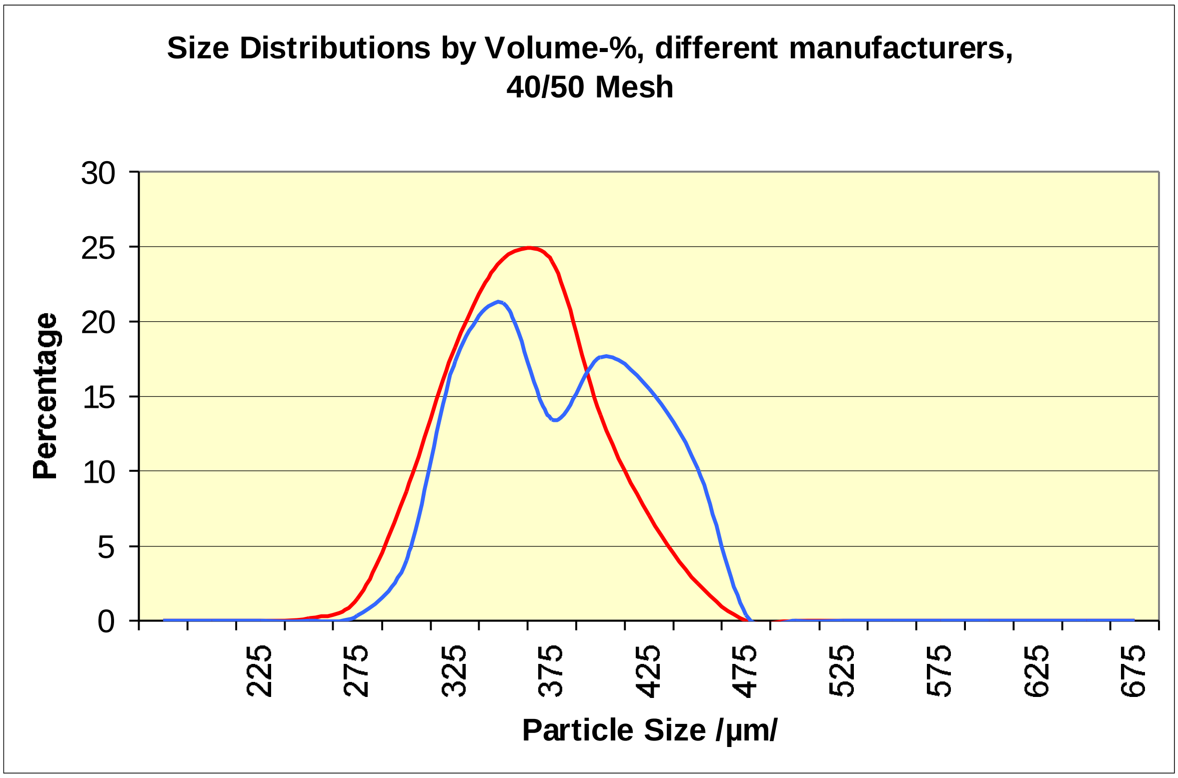

Figure 2: Particle size distributions measured with an image processing system (DiaInspect from Vollstaedt-Diamant)

Image processing is sensitive to a wider range of basic properties and will be discussed later in more detail.

Magnetic Inclusions – Magnetic Susceptibility

Impurities (Inclusions) have a direct and strong influence on the fracture behaviour of the particle. The total amount, the type and the distribution of the inclusions is of interest, but hard to measure with a simple method. If the inclusions are totally or partly magnetic, at least the total content of magnetic material can be estimated by means of a susceptibility meter.
A modern device requires an amount of 8 carat powder only to produce a precise and reproducible result. Sample preparation and measurement itself takes few seconds only, so that the method is well appreciated as quick check for stable material properties.
Due to the robustness of battery powered susceptibility meters they are suitable also at the diamond manufacturers site for in-process control close to the magnetic sorting machines. **Fig. 3** shows values for the magnetic susceptibility for a complete product range of synthetic saw grit diamond from low price up to high price. Some discontinuity is visible, but the general suitability of the susceptibility meter as a sensitive monitoring device becomes obvious.

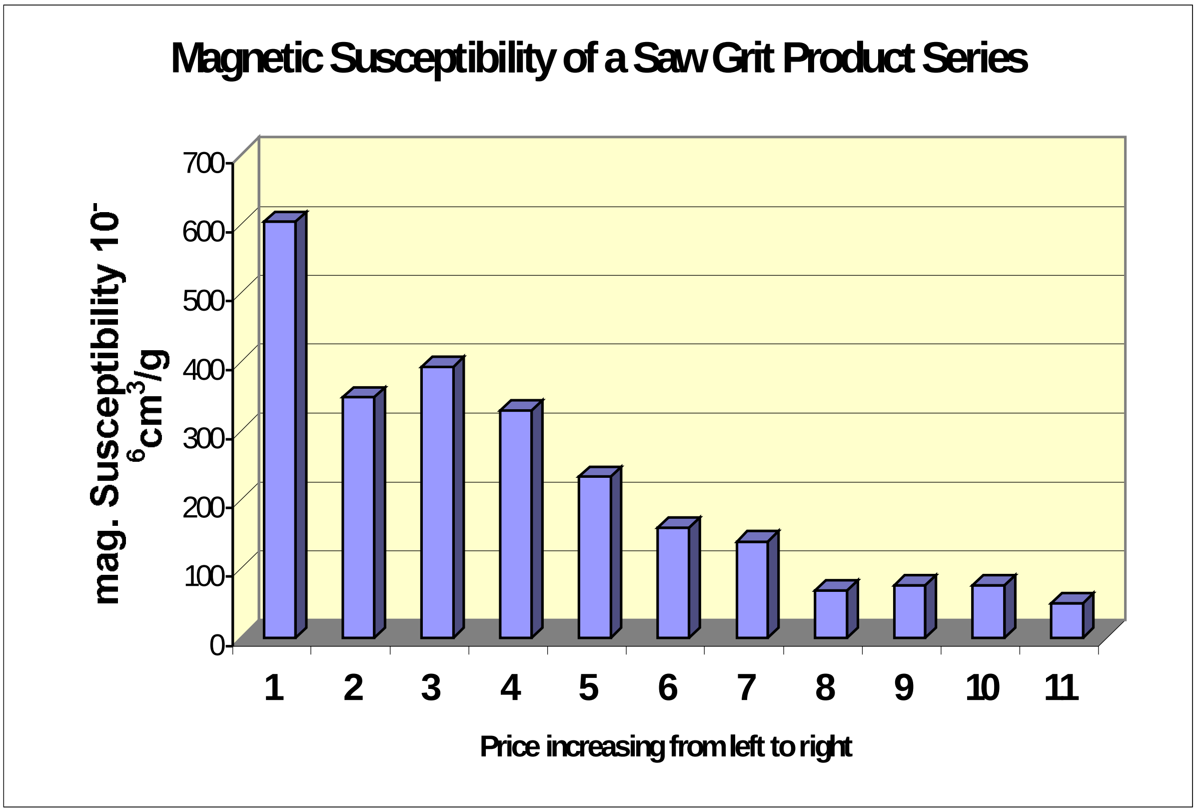

Figure 3: Magnetic susceptibility of a saw grit series

Colour

There is no generally established method of colour measurement on superabrasive powders. Although the particles colour is directly influenced by the amount, type and distribution of impurities or inclusions it is hard to establish a standard configuration for professional colour measuring instruments. The gleam and the graniness of the powder require a careful set up of the sample. There is also no agreement about the colour space to be used. Nevertheless there are colourimeters with special mounts for the diamond powder in use.

The subjective “colour” impression when looking at different powders is usually expressed in terms as “bright yellow” or “slightly greenish” or “dark”, which from the history correlates with an idea about the quality and maximum price of the batch. Recent investigations on colour image processing systems show that some of these rules should be revised.

Colour image processing is able to determine colour coordinates at certain regions of single particles. Investigating a powder described as “slightly greenish” it was found a colour coordinate located at deep yellow, but much less in brightness than a powder described as “bright yellow”, which was in fact more colourless, but more bright.

“Colour” in terms of coordinates is an indicator which can be watched for deviations, while the absolute visual impression of colour depends on the observer and will vary between different observers and also from time to time.

Image processing

Computerized image acquisition and processing picks up the task of the qualified employee and tries to support him in judging by the appearence of the powder. Prerequisite for meaningful image processing results is a precise and reliable image acquisition which produces calibrated pictures from a least several hundreds of particles.
For the size range of saw grit down to approximately 100 µm this task can be done by high resolution slide scanners. With a maximum resolution of 4000 dpi (dots per inch) the smallest unit of the picture is roughly 6 µm in square. The colour response can be calibrated by standards, allowing for quite stable colour evaluation on single particles. **Fig. 4** shows pictures taken from rough saw grit with a 4000 dpi autofocus slide scanner.

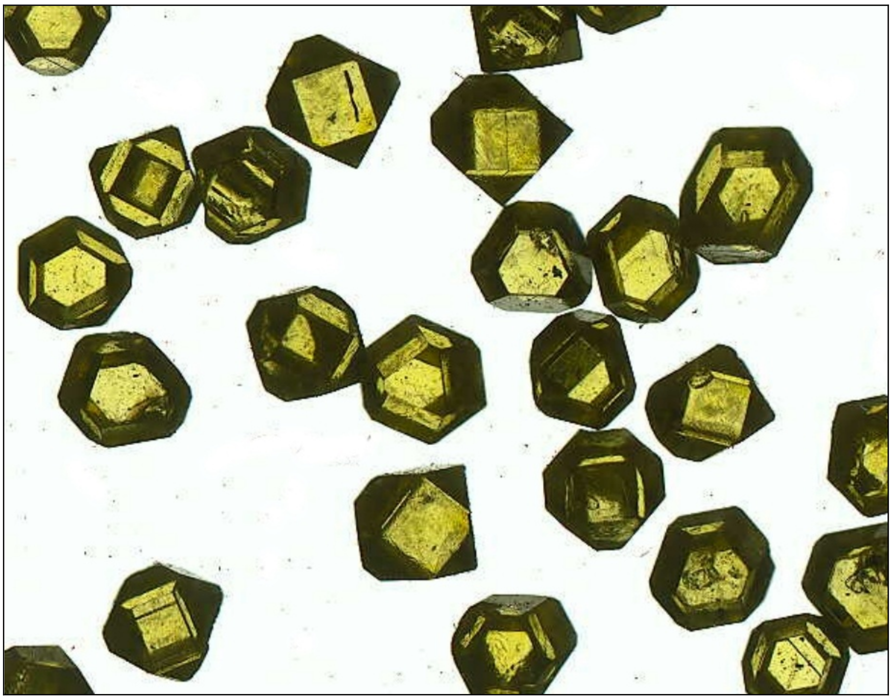

Figure 4: Part of a picture from saw grit, captured on a 4000 dpi slide scanner

If smaller particle sizes are of interest a microscope with an electronic camera can be used. One can overcome the small viewing area and the limited particle count inside the picture by using a computer controlled scanning table. In such way the image processing programme can investigate a large series of pictures, taken at different places of the object plate, keeping the particles data and its pictures for further computations. **Fig. 5** shows a computerised microscope, producing pictures shown as example in **Fig. 6**.
Once the particle images are available for image processing the programme will measure an extensive set of parameters for every single particle:

- size parameters like minimum, maximum and average diameter and total area
- shape parameters derived from perimeter, convex perimeter, area and moments of inertia
- parameters describing the transparency or the “darkness” of the crystal

- parameters describing the colour of certain regions inside the particle.

Figure 5: Computer controlled XYZ scan microscope DiaInspect.OSM (Vollstaedt-Diamant GmbH)

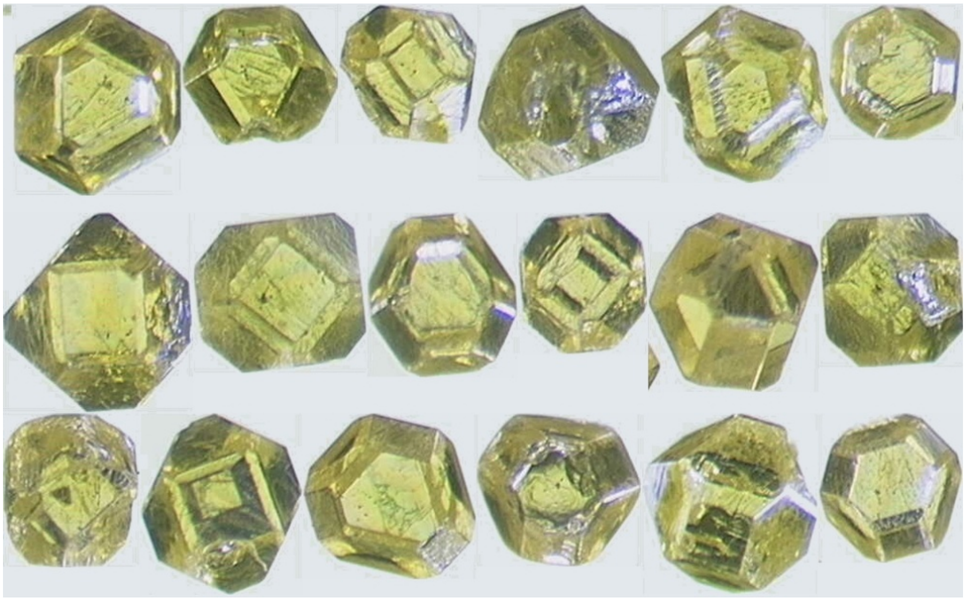

Figure 6: Collection of saw grit particles processed by DiaInspect.OSM

Assuming 700 particles have been investigated by the image processing the whole set of data will consist of 16100 numbers. It was found that simply compressing the data into averages for the single parameters will neglect valuable information about the batch. A flexible system was developed that provides easy access to every parameter for every crystal as well as simple to handle customizing for final reports. The most demanding task of reducing the amount of data without loss of information was solved by the introduction of a so called “virtual sorting machine”. As the basis serves the particle distribution in relation to two or three parameters. Sorting classes can be defined in the coordinate system which given by the actual parameters. As result of the virtual sorting procedure the programme will assign a class number to the data set of every particle. The number of particles inside a class can now be shown in a histogram. Following this procedure the set of several thousands of digits can be compressed into a histogram with 10 columns (**Fig. 7**). It is possible to achieve a good reflection of verbal descriptions like “dark”, “clear”, “blocky” or “unsymmetrical” in the bars of the histogram.

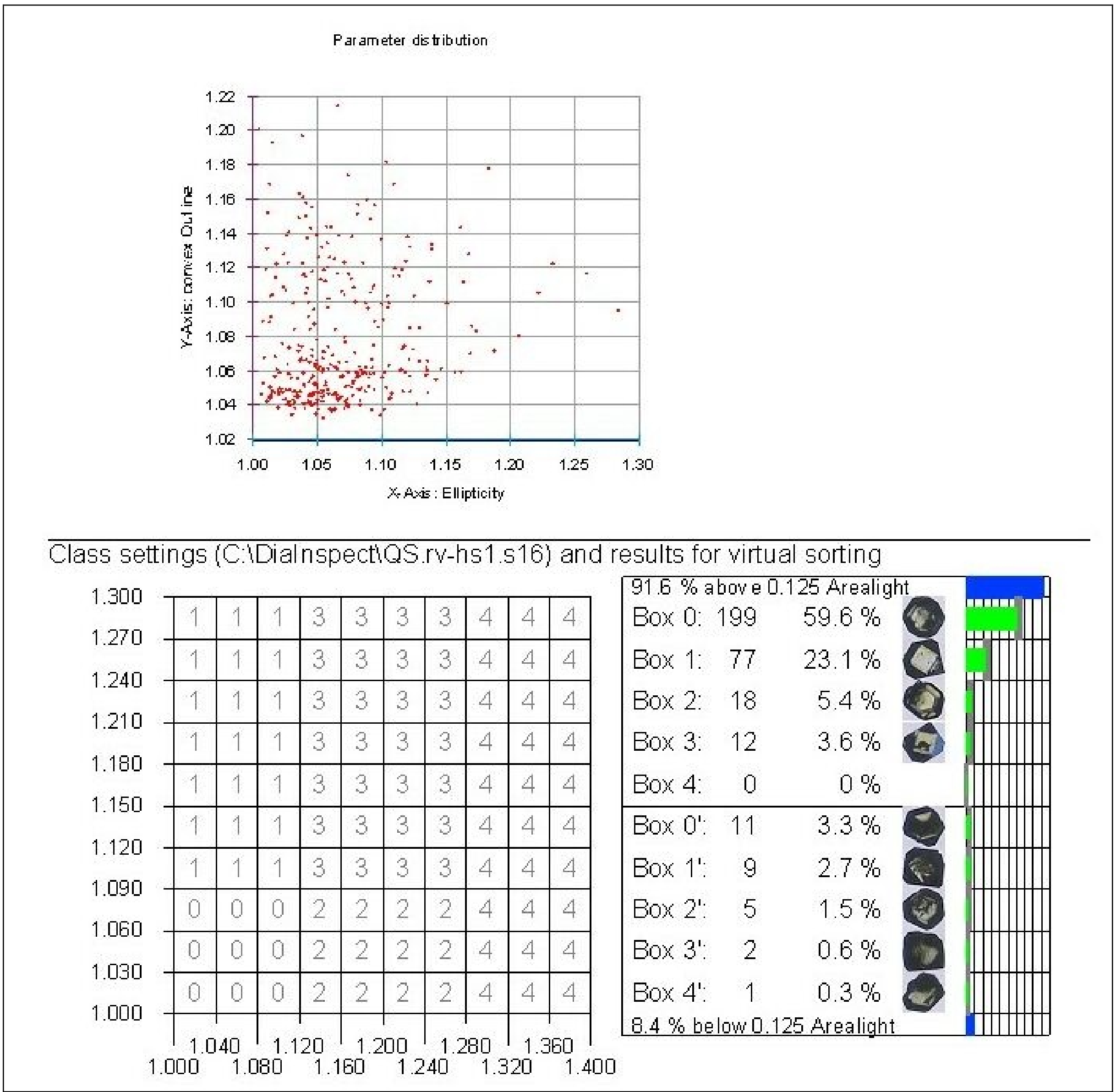

Figure 7: Result presentation from the “virtual sorting machine”

The most sophisticated programme part based on geometrical models will also find the morphology class the crystal relates to. Of course this will work only on well shaped particles with not too much defects.
Image processing was found a quick and flexible tool, allowing for sensitive monitoring of crystal size, shape and colour.

Mechanical fracture test – Friability test

The toughness index (TI) and the friability index (FI) along with these values after thermal stress of the material (TTI) are very popular measures for the impact strength of a diamond batch.
Looking for the reason for this popularity it is obvious that the stress applied to the particles seems similar to the situation in the diamond tool. And, fortunately, the rules for the friability test lead to a stable result. As visible from **Fig. 8** the usual capsule with 2 carat diamond in it applies the test to several thousand of particles. This provides high statistical accracy. The red columns in **Fig. 8** indicate critical particle counts of less than 500 when investigating 20/30 mesh and bigger stones in the standard Friatest.

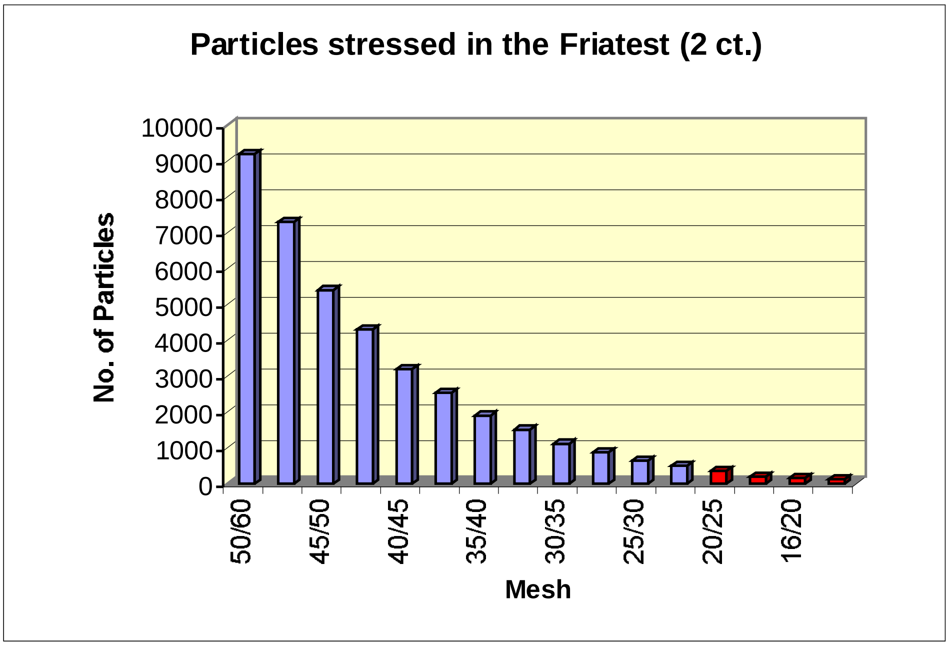

Figure 8: Number of particles in Friatest capsule of 2 carat

Apart from the manageable result there are two disantvantages, at first the bad comparability between Friatest machines from different manufacturers and as a second the limitation to a single number as result. The distribution of stress resistance values of the individuals inside the batch remains unknown.
Nevertheless the Friatest is a standard procedure used all over the world.

Mechanical fracture test – Single particle static compression test

In contrast to the bulk testing method described above the single particle compression test should give an impression about the spread of the strength property inside the batch. The simple layout of the experiment should also lead to comparable results between different laboratories and machines.
In most cases, Chinese diamonds were graded by a test of the static compression force to crush a single crystal, 40 grains were tested. The crash forces were recorded to calculate the average force, and abandon the ones that exceed 2 times the average value, then recalculate the average of the rest. The result is expressed by Kg force, which represents the static strength of the diamond sample /4/. The russian standard recommends to test 50 grains. **Fig. 9** shows results of repetitive fracture force measurements according the chinese standard on high quality 45/50 mesh synthetic material.

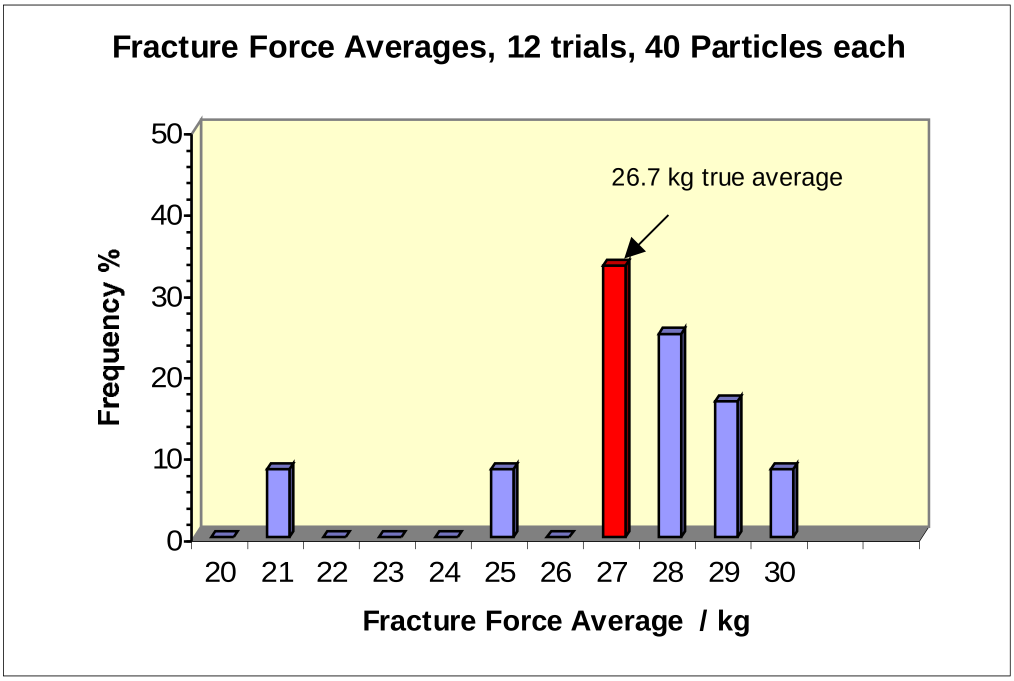

Figure 9: Distribution of the results from 12 fracture force measurements, 40 crystals each, high quality 45/50 mesh diamond

Looking at the statistics coming from these tests on 40 or 50 particles it becomes clear that much more particles have to be tested to get a meaningful distribution. Several machine designs were published for this purpose. The “roller crusher” from General Electric Corp. /5/ is able to crush a sufficient amount of stones and produces fracture force distributions. However the results are not directly comparable to the standard compressive fracture force procedure because of the different load application to the crystal. Moreover the device is not available on the market. Recently a computer controlled single particle fracture force tester was introduced to the diamond industry, which combines image processing capabilities and static compression test on single particles.
The DiaTest-SI (Vollstaedt-Diamant GmbH, **Fig. 10**) is a combination of a computer controlled single particle fracture force tester with size and shape inspection of the single particles. The fracture force tester measures the maximum uniaxial compressive fracture force (CFF [N]) necessary to crush a particle. To generate the constantly rising force a load cell with a pneumatic force generation system is applied. For measuring diamonds a load cell in the range from 0 N to 2000 N

(resolution of force measurement in whole device: 6 N, anvils: polycrystalline diamond) is being used whereas for non-superabrasives a load cell from 0 N to 500 N (resolution of force measurement in the whole device: 1 N, anvils: polycrystalline c-BN) is being operated. The obtained CFF-values are statistically processed and the test is stopped when the statistical deviation is less than 5%. The particles are fed to the device in a carrier stripe which is processed automatically.

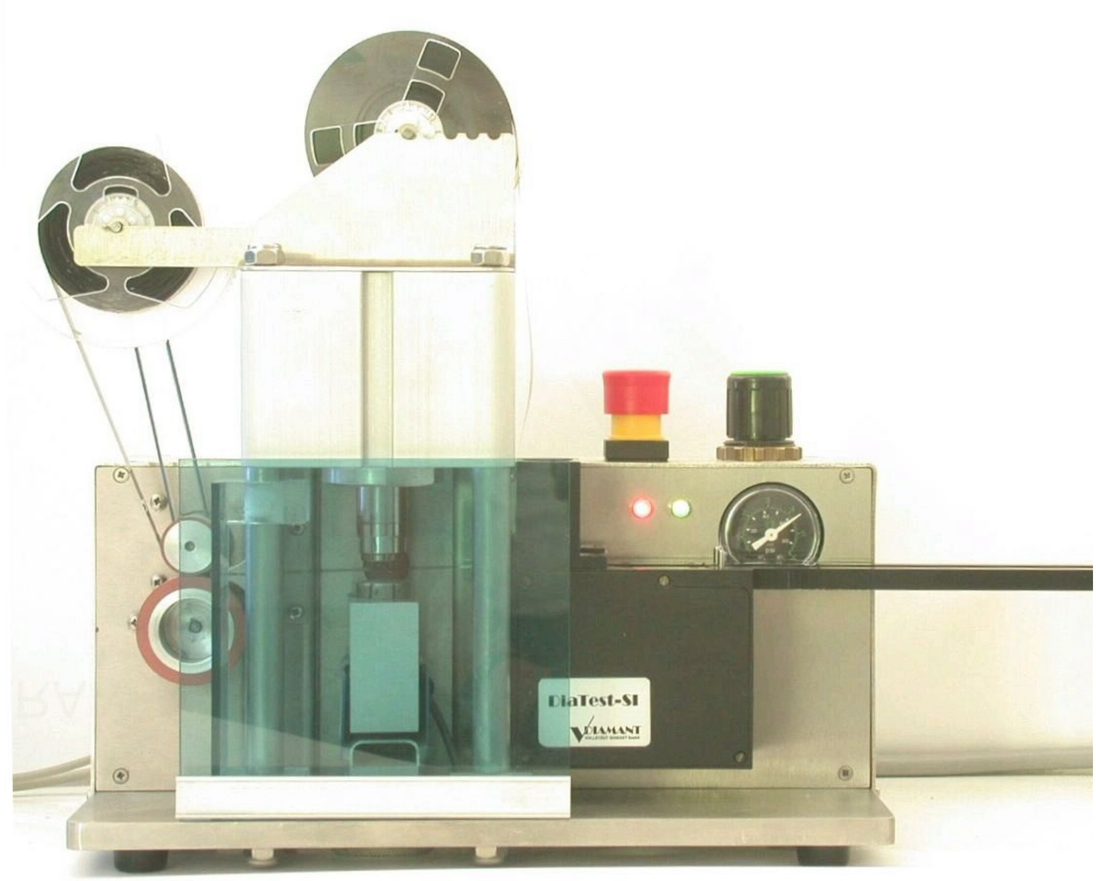

Figure 10: Static Fracture Strength Tester DiaTest-SI (Vollstaedt-Diamant GmbH)

Before being crushed the particles pass through a built in microscope connected to an image acquisition an processing system. That way the program can record size and geometrical properties along with the fracture force for every particle. From fracture force and particle size also the fracture strength is calculated.
The reports show the distribution of fracture forces, the size distribution and the scatter of particles in a size –strength diagram. The whole set of data for all particles is available as EXCEL-spreadsheet for individual calculation.

Extensive studies were carried out on how the results of static compression testers can be compared. Basic condition is a statistical sufficient number of crushed particles. If this prerequisite is given the biggest influence will have the design of the experimental conditions in forms of the anvils who contact the crystal. Ideal conditions would provide an ideal smooth, flat, and never wearing pair of parallel anvils. This is quite far from reality. The anvil material commonly used is tungsten carbide, cBN and PCD. The fracture force values measured on WC anvils were found always slighly higher than these on the superhard cBN or PCD. For crushing several hundreds of diamonds on a small place of the anvil PCD has the best performance.

Wear of the PCD anvil surface will cause a dropping of the fracture forces. Quite the opposite one can find when using cBN or WC anvils.
If the precautions regarding anvil material and anvil wear were kept the single particle strength test gives reproducible and comparable results.
Of special interest for the diamond tool maker is the access to the distribution of parameters instead of depending on averages. **Fig. 11** illustrates the advantage of dealing with distribution parameters. Two diamond samples were measured on DiaTest-SI, the average fracture force between them differed by less than 1% only. But looking at the fracture force distribution it becomes evident that the two samples are totally different in behaviour. The red plot indicates the narrow distribution of a uniform material, while the blue line shows much wider spreaded properties bewteen high strength and very friable. As a conclusion the results of fracture force investigations should be expressed by means of distribution parameter and graphical plots of distributions instead of compressing the results into an average value only.

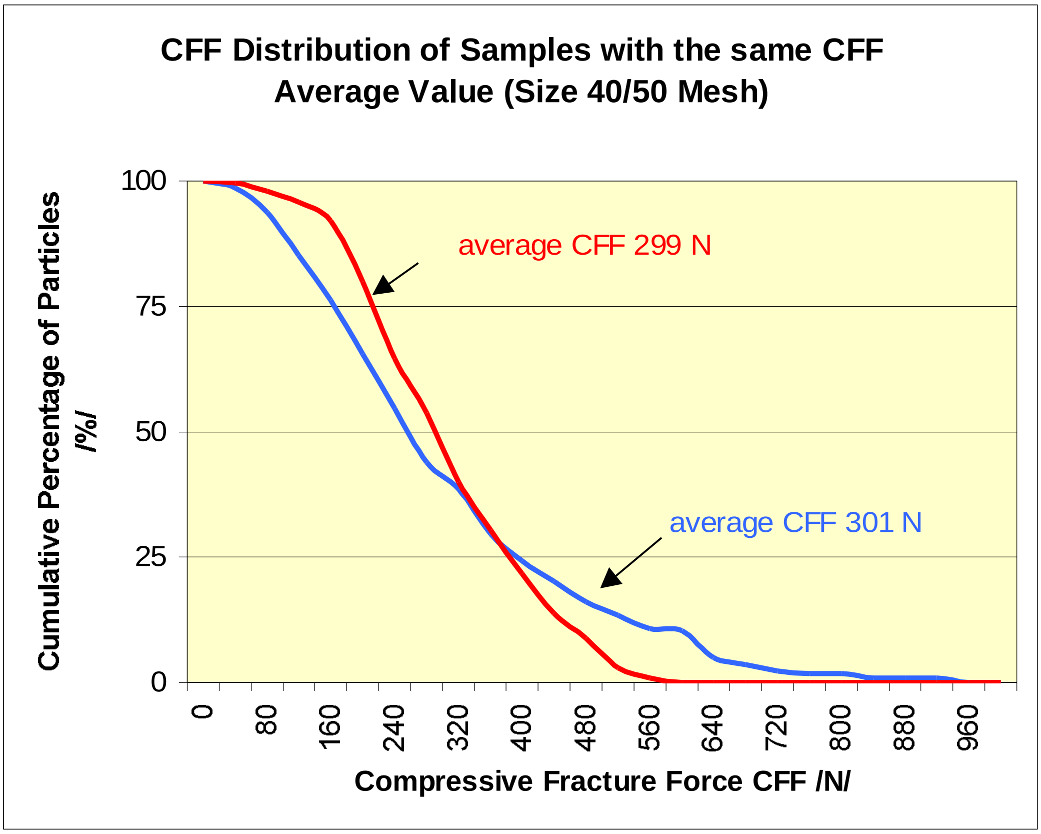

Figure 11: Fracture force distribution of samples with the same CFF average

Conclusion

Methods and apparatus are available for both ranking and control of the properties of synthetic diamond. Most of them are suitable also for other abrasives and superabrasives.
The request for tightly specified raw material can be met when introducing the advanced methods into the control of the manufacturing and sorting steps and the final quality control of superabrasives.

In addition to national standards in China and Russia for the compressive fracture force it is desirable to extend the product description by parameters for the shape of distributions.
There are no agreed standards for magnetic susceptibility, colour or shape parameters. In the opinion of the authors there is no real need for this at the moment as long as these methods are not used for absolute rankings. For monitoring the stability of product properties they are already perfectly suited.

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