Quality control of superabrasives: complexity of tests vs. sample size and sampling frequency

List, E., Vollstaedt, H., Frenzel, J.
Vollstaedt-Diamant GmbH, Schlunkendorfer Strasse 21, D-14554 Seddiner See, Germany

Abstract

Both the producer and the consumer of superabrasive powder spend a not negligible amount of investment, labour and consumables for the quality control of superabrasive powders. Complexity and cost of the tests are always the limiting factors for the sampling frequency. On the other hand the limited sample size used in most of the tests causes limitations in the expressiveness of their results. In many cases the test is made on a few dozen to several hundreds of particles, and the result is expressed as a median value which is sometimes accompanied by an interquartile range. The actual spread of the properties amongst the individual particles remains unknown.

The paper shows, how the actual set of test methods can be extended by a bulk testing method which targets large sample sizes and high sampling frequency at the same time. Due to the large sample size this method is able to deliver not only basic statistical data, but also stable distribution curves for many parameters. Using the whole distribution curves from a variety of parameters makes the method sensitive for slight changes in the product. Examples from tests on superabrasive powders are shown, which prove precision and repeatability of the method.

Introduction

Development of superabrasive powder products as well as development of tools based on these products require a deep knowledge and a complex description of the product properties. Once this relation is established the main task of quality control teams on every point of the supply chain is to guarantee stable properties of the product. Cost of tests and solidity of the "guarantee of stability" which is issued by the quality control team are in direct relation.

The current paper shows, how the "guarantee of stability" can be brought to a higher level of confidence at drastically lower costs.

The square root problem

Most of the tests for superabrasive powders deliver as result an average value (or median value for non-gaussian distribution), sometimes along with the value for the standard deviation (or interquartile range).

The result R as average (or median) is computed from a number N of particles which participated on the test. The precision of the result R depends on the distribution width of the individual values and on N. Unfortunately there is no linear relation of the error U with N [equ 1].

\[ U = \frac{\sigma}{\sqrt{N}} \]  
\[ \sigma = \sqrt{(1/N - 1) \sum (a_i - \text{avg}(a))^2} \]
Equations 1 and 2 are for gaussian distributions. According to the "Guide to the expression of uncertainty in measurement" [1] U is called "standard uncertainty of the mean". For normal distributed values the true value of R will be in the interval R +/−U with a probability of 68%. Also for non-gaussian distributions as found on our particles the number of particles works by its square root.

Picture 1 shows typical slopes of U/R for the compressive fracture force of diamonds.

![Relative standard uncertainty of CFF tests on diamonds](image)

*Picture 1: Relative standard uncertainty of compressive fracture force tests on different diamond samples*

It clearly visible that a lesser uncertainty of the test requires not only a linear, but a quadratic increasing number of tested particles. The number of tested particles itself goes linear with test time and test cost.

**Sample size and processing time of typical methods**

Table 1 lists a variety of commonly used tests with respect to the number of tested particles, the cost of consumables, the processing time and the expressiveness of their results. The methods with the lowest amount of labour (magnetic susceptibility and color) must be used together with another method because of their limited viewing angle.
Table 1: Test methods for superabrasive powders (Processing time ranges from "very low = 1 minute or less" "high = 1 hour or more")

<table>
<thead>
<tr>
<th>Test method</th>
<th>Particles used</th>
<th>Processing time</th>
<th>Consumables</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Friability (TI, TTI, FI)</td>
<td>100 ... 500,000</td>
<td>Medium</td>
<td>&gt; 3 US-$</td>
<td>average value</td>
</tr>
<tr>
<td>Single particle compression (SPST)</td>
<td>50 ... 1000</td>
<td>Medium ... High</td>
<td>&gt; 3 US-$</td>
<td>distribution</td>
</tr>
<tr>
<td>Magnetic susceptibility</td>
<td>100 ... 500,000</td>
<td>Very low</td>
<td>0</td>
<td>average value</td>
</tr>
<tr>
<td>Color</td>
<td>1000 ...</td>
<td>Very low</td>
<td>0</td>
<td>average value</td>
</tr>
<tr>
<td>Optical size and shape analysis</td>
<td>500 ... 5000</td>
<td>Medium ... low</td>
<td>0</td>
<td>distributions of size, shape, ...</td>
</tr>
</tbody>
</table>

Requirements for a production scale test instrument

A test instrument for the regular use in the quality control should satisfy the following requirements:

- high number of tested particles (10,000 and more)
- low processing time (minutes)
- neglectable consumables cost
- non-destructive test, re-usable sample
- flexible setup for different products
- easy operation for less skilled workers
- thorough result documentation
- expressive results (output of property distributions)
- easy reporting

The only method capable of meeting these requirements is digital analysis of images captured from a stream of moving particles.

Technical realisation

Core of the system is a particle analysis program, which is fed continuously with images from a digital colour camera. A vibration feeder transports a stream of particles onto a rotating transparent disc. On the disc the particles pass through the optical system, which is built from telecentric lenses together with telecentric lighting.

After being photographed the particles are removed from the disc by means of an air nozzle and collected in a separate container.

In order to get clear images of the moving particles the shutter time of the camera is very short (1/25,000 second), with the side effect that ambient light doesn't influence the results.

The control program has two operating modes: one is for the setup of the system and of the products to test, one is for the daily use and requires just a few operation in order to get the test done. The main parts of the system are shown in Picture 2. The only wear part in the system is the transparent plastic disc as sample holder, which needs to be replaced when scratches or dust obscur the image.
Despite of its exceptional high optical and geometrical quality the cost per piece is low.

The operator can chose between two operating modes. The first mode (collection mode) just measures and stores the particle data. The second mode (test mode) additionally compares the distributions of the measured parameters with the distribution limits for the particular product. Picture 3 shows a program screen where not all distributions fall into the allowed range.

**Picture 2: Components of the fast particle analyser "DiaInspect-P"**

**Picture 3: Operating screen of DiaInspect-P, the red distributions are partially outside the allowed range**
**Test results**

The most important parameter of the system is the total processing time for a batch, which is the sum of preparation time, measuring time for the desired amount of particles and the cleanup time. The estimation is based on staff who uses the device regularly:

- preparation time: if necessary exchange the disc, fill the feeder, select the product and click start (10 ... 30 s)
- measuring time: the measuring time slightly depends on the particle size, there can be more smaller particles present on one image, typical values are 5,000 ... 10,000 particles per minute
- cleanup time: reclaim the particles from the container, clean the feeder (30 ... 60 s)

A test of a 10,000 particles batch would consume in between 100 and 210 seconds. If a batch size of 20,000 particles is required the test will be done in 160 ... 330 seconds.

The results are stable due to the automatic system calibration and the large number of tested particles. As an example a batch of cBN was split into fractions of 10,000 particles each which were analysed on Dialnspect-P. Picture 4 shows the distribution of the parameter "Ellipse ratio" for these fractions. The difference in the parameter distribution between the fractions is very small. It proves that the cumulative distributions of the parameters can serve as stable indicators for the product properties.

![Diagram](image.png)

*Picture 4: Cumulative distribution of the ellipse ratio measured on different fractions of one batch of cBN (10,000 particles per fraction)*
Conclusion

It was shown that the analysis of a large number of particles is possible in a short time. It was further shown that the computed parameter distributions are stable and can be used easily to define product properties for the purpose of quality control. The system can be used on every place in the powder supply chain, from the manufacturer to the end user. The coverage and the reliability of tests in the quality control can be increased drastically at reduced total cost of labour, consumables and investment.

The square root of N is no longer a problem.