TECHNICAL REPORT

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Synthetic industrial diamond grit products — Single-particle compressive failure strength — "DiaTest-SI" system

Produits en diamant synthétique industriel — Résistance à la compression des particules — Méthode «DiaTest SI»

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 24857 was prepared by Technical Committee ISO/TC 29, *Small tools*, Subcommittee SC 5, *Grinding wheels and abrasives*.

Introduction

A study has been performed to evaluate the suitability of the Vollstädt “DiaTest-SI” system for the single particle compressive strength testing of synthetic industrial diamond particles.

Four distinct saw grit diamond products were measured repeatedly by six test centres, in order that the variation in results between the centres and the variation in results within each centre could be established.

The principal measurement examined was the median single particle strength of a sample (that is, half of the particles in the sample have a strength below this value). It was concluded from the study that within each test centre, the median strength of a saw grit diamond product could be measured with a high degree of repeatability: the average “scatter” of the medians being around 2 % to 4 %. Examining variations between test centres, there were small systematic differences in the results from each test centre's strength testing machine, their measurement “biases” being between −2 % and +5 %. The combination of between-centre and within-centre variations resulted in an estimated experimental error of between ±7 % and ±15 %.

Synthetic industrial diamond grit products — Single-particle compressive failure strength — "DiaTest-SI" system

1 Scope

This Technical Report gives the results of a study to determine the feasibility of the "DiaTest-SI"1) single particle strength tester as a system for measuring the compressive strength of synthetic industrial diamond grit products. Issues that were addressed included: the range of grit products (in terms of both size and strength) for which the "DiaTest-SI" system was appropriate, the choice of distribution statistics with which to describe diamond strength, and the similarities (at a statistically significant level) of the results from various test centres.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5725-1, ISO 5725-2 and the following apply.

3.1
analysis of variance
ANOVA
statistical method used to determine the influence of various assignable causes on experimental results

3.2
compressive failure force
CFF
force (in newtons) applied to a particle which results in its failure

3.3
single particle strength
SPS
alternative term for the compressive failure force (CFF) of a particle

1) "DiaTest-SI" is the trade name of a product supplied by Vollstädt-Diamant GmbH, Schlunkendorfer Strasse 21, 14554 Seddiner See, Germany. This information is given for the convenience of users of this Technical Report and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

3.4
polycrystalline diamond
PCD
intergrown mass of randomly orientated diamond particles in a metal matrix, synthesized at high temperature and high pressure

NOTE PCD offers very high hardness, toughness and abrasion resistance.

3.5
US mesh
size of a diamond product determined by the mesh sizes of the sieves used to separate the diamond particles

NOTE In the US mesh system, a sieve size is defined by the number of lines per inch of that sieve; see ISO 6106 [1] for details.

4 General principles of the single particle strength testing of diamond

Industrial synthetic diamond products may be tested for strength using a variety of techniques. Perhaps the most established of these techniques is the friability test (or friatest [2]), which measures the resistance of a diamond sample contained within a capsule to multiple impacts by a steel ball. Whilst the friatest may be a robust technique, being conceptually simple and having a high level of repeatability, it yields only one “figure of merit” strength value, and cannot be used to describe the distribution of particle strengths within a diamond sample.

The strength of an individual diamond particle may be measured by subjection to an increasing compressive force, the threshold force (in newtons) at which the particle “fails” being its recorded strength. This form of measurement, which is known as single particle strength (SPS), compressive failure force (CFF) or static strength (as distinct from the dynamic strength of the friatest), is therefore a valuable complementary technique to the friatest because of the information it provides on the particle strength distribution. At present, single particle strength testing is most conveniently performed on grit sizes coarser than size D213 (70/80 US mesh).

There are two principal methods for the single particle strength testing of diamond:

— particles may be either crushed between rotating cylinders [3], or

— between vertically aligned anvils.

The second of these two methods is substantially more widespread than the first, and is commercially available in the form of such systems as the “DiaTest-SI” by the German manufacturer Vollstädt [4].

In the DiaTest-SI system (and others of a similar design), diamond particles are aligned on an adhesive “carrier” tape and are subsequently transported between the anvils. An image analysis camera may be positioned before the anvils in order to measure the size and shape characteristics of the particle. The upper anvil is attached to a pneumatically (or mechanically) driven piston, whilst the lower anvil is attached to a load sensor. The anvils may be manufactured from polycrystalline cubic boron nitride (PcBN) or polycrystalline diamond (PCD), with PCD offering a longer anvil life (this is important, as over-used anvils can have a significant effect on results).

As the upper anvil is driven downwards, the particle is subjected to a compressive force, and this force is transmitted through the particle to the lower anvil and the load sensor. Eventually the particle will “fail” in that some disintegration will occur, and there will be an instantaneous reduction in the force detected by the load sensor. The nature of this reduction in force is dependent on the defect structure of the particle: a particle with a high perfection will tend to withstand high compressive forces before disintegrating catastrophically, whilst a particle with numerous significant defects is more likely to break in several stages. Complex algorithms are used to examine the force-time characteristics of a crush and to assign an appropriate failure strength value to the particle.

5 Design of the experiments

5.1 General conditions

The Vollstädt “DiaTest-SI” system is capable of measuring the single particle strength distributions of virtually all common grades of saw grit diamond in the common sizes. Experiments were therefore chosen to evaluate the performance of the machine over a range of operating conditions in accordance with ISO 5725-1 and ISO 5725-2.

a) Title: Synthetic industrial diamond grit products — Single-particle compressive failure strength — “DiaTest-SI” system

b) Name and location of the laboratories:

- Centre 1 Germany
- Centre 2 Ireland
- Centre 3 China
- Centre 4 Germany
- Centre 5 Austria
- Centre 6 Germany

c) Measuring equipment: Vollstädt “DiaTest-SI” system using unified and optimized software

d) Anvil and (pneumatic) piston Each test laboratory received three sets processed from the same PCD discs:

Abrasive, monocrystalline synthetic diamond macrogrit with the following sizes, properties and sievings:

1) high-strength grade, coarse grit (narrow sieving) 30/35 US-mesh

2) high-strength grade, medium-size grit (broad sieving) 40/50 US-mesh

3) medium-strength grade, medium-size grit (broad sieving) 40/50 US-mesh

4) low-strength grade, fine grit (broad sieving) 60/70 US-mesh

Each test laboratory was provided with three samples each of the particle sizes defined in 1) to 4), each sample consisting with approximately 500 particles.

5.2 Additional conditions

A second phase of the study was performed in the same manner, with each laboratory receiving a further three sets of PCD anvils and a further three sets of each of the four diamond samples.

For all tests to be carried out, the test laboratories appointed a measuring instrument operator.

The respective three sets of anvils (anvil and piston) were employed in such a manner that one set of anvils was used for high-strength grade in size 30/35, and another set of anvils was used for the high-strength grade in size 40/50. The third set of anvils was used to test both the medium-strength grade in size 40/50 and the low-strength grade in size 60/70.

These test series were designed to evaluate the accuracy of the Vollstädt measuring equipment in terms of the correctness and precision of strength measurements. The parameter to be tested was the so-called CFF value (compressive failure force, in newtons).

5.3 Results

The following values were determined.

a) Mean strength, S_{mean}

$$S_{\text{mean}} = \frac{\sum F_{\text{take out}}}{n}$$

b) Median strength, S_{med}

$$S_{\text{med}} = F_{\text{take out, med}}$$

where

$F_{\text{take out}}$	is the compressive failure force (CFF), in newtons, remaining after all unquantifiable particle crushes (given the arbitrary strength value 9,999 N by the DiaTest-SI system) have been removed from the data set;
$F_{\text{take out, med}}$	is the middle value of $F_{\text{take out}}$ when sorted in ascending order;
n	is the number of particles (quantifiably) tested.

NOTE If the number of $F_{\text{take out}}$ values is even, the median strength is the average of the middle pair of $F_{\text{take out}}$ values.

Four grades of saw grit diamond were used for the study:

- HS601: a high-strength grade, in size D601 (30/35 US mesh)
- HS427: the same high-strength grade, in size D427 (40/50 US mesh)
- MS427: a medium-strength grade, in size D427 (40/50 US mesh)
- LS251: a low-strength grade, in size D251 (60/70 US mesh)

In all four diamond grades, the particle sizing and particle strength distributions were typical of those found in standard industrial diamond products.

For each grade, the many samples sent to the various centres were extracted from a single larger “batch”. Each sample consisted of around 500 particles, and the sample extraction process was performed using well-established proprietary random-splitting equipment. It is therefore believed that the best possible measures were taken to ensure that individual samples were the same, and representative of the larger batch. Furthermore, test centres were instructed to test all particles in a sample, rather than a fixed number, to remove associated sample selection variations.

Six samples of each grade were analysed by each of the six centres — three samples were tested in the first phase of the study, and the remaining three samples were tested in a subsequent second phase.

Particular efforts were made to minimise the effect of anvil variation on single particle strength results. Polycrystalline diamond discs were carefully chosen to ensure homogeneity, processed into anvils, and distributed to the test centres for use with specific diamond samples.

For the first phase of the study a particular disc was processed into anvils for use with the 18 samples of HS601 (three samples for each centre), a second disc produced anvils for use with the 18 samples of HS427, and a third disc produced anvils for the 18 samples of MS427 and the three samples of LS251. This approach ensured that possible disc-to-disc structural variations did not affect the results either *within* a test centre or *between* test centres for a particular diamond grade. Regrettably the limited size of such polycrystalline discs necessitated the processing of new discs for the second phase of the study. However, the same method was used for the distribution of anvils in the second phase.

6 Assignable causes of variations in single particle strength

The results of the many tests (144 in total) were analysed with the aim of determining the general variation in the single particle strength measurement and the "assignable causes" of the variation [5]. Assignable causes of variation in a measurement system may be summarized in the mnemonic:

- Man: the effect of different machine operators
- Machine: different units giving different results
- Materials: differences or inhomogeneities in the materials used in the test
- Method: differences in the measurement procedure

Some of these assignable causes were investigated by the statistical analyses of the results, whilst other assignable causes were minimized in their effect by judicious experimental design.

The contributions to test variability of *man* and *machine* were combined by ensuring that each test centre used only one person, operating only one "DiaTest-SI" unit, for the entire study.

The category *materials* should perhaps be separated into two components: the test saw grit diamond samples and the polycrystalline diamond anvils. The contributions to test variability of the test diamond samples took the form of systematic differences in strength between different grades, and random variations in the strength from different samples of the same grade. The contributions to test variability of the polycrystalline diamond anvils took the form of variations in compressive strength (or other behaviour under loading) of different anvils from the same disc, and variations in strength between discs. As mentioned earlier, the effects of between-disc variations were eradicated within each of the two phases of the study by the use of specific discs with specific diamond types, and the effects of within-disc variations on results from different samples of the same diamond type were minimized by careful selection of polycrystalline diamond discs according to their structural homogeneity.

Finally, variations in the *method* were addressed by each test centre using the same, strictly defined, measurement procedure.

7 Statistical analyses of the results

A common statistical technique for analysing an experiment such as this is analysis of variance (ANOVA) [6]. ANOVA evaluates differences in results in terms of the various assignable causes – if there is simply one factor that is changed between tests (e.g. machine) then one-factor ANOVA may be employed, whilst for changes in several factors (e.g. machine, material) multi-factor ANOVA should be employed.

In the single particle strength experiment reported here, there were three factors that changed between tests: test centre, diamond type and run ("repeat").

However, a fundamental requirement of ANOVA that prohibits its use for this experiment is that the *random variations within each test be normally distributed*. Here, these random variations correspond to variations in strength of the particles in each "repeat". As will be apparent, single particle strength distributions of diamond products are not necessarily normal (Gaussian) in form, and so the form of the basic data captured in this study invalidates the assumptions of ANOVA. Therefore, a different statistical approach was required in order to obtain an ANOVA-type evaluation of the important factors that contribute towards variation in single particle strength.

Non-normal single particle strength distributions are best described by non-parametric statistics, and so the median was chosen as the descriptor of distribution location, and non-parametric significance tests were used to determine the statistical significance of differences between distributions. An introduction to distribution statistics and an exercise to prove the appropriateness of non-parametric statistics are presented in Annex A.

Recalling Clause 6, it is expected that the assignable cause that will have the most significant effect on strength measurements (other than the systematic differences deriving from the different diamond types) is man/machine – other assignable causes have been hopefully minimized by careful experimental design.

Two fundamental measurement characteristics of each test centre's man/machine are precision and bias. If a man/machine has the ability to perform repeated measurements with little variation in results, it has high precision. If a man/machine is able to obtain a measurement result that does not differ much from the “true” result, it has low bias. (In this study it is difficult to know the “true” strength distribution of a diamond type, so it is taken to be the average of the distributions from all the test centres.)

The statistical analyses performed here fall into two basic categories: analyses of *between-centre* variations and analyses of *within-centre* variations.

Between-centre variations derive from differences in strength measurement between machines of different test centres, and so are informative of the bias of the machines. These variations were assessed by comparing results across test centres, having firstly combined within each test centre the results from its repeats.

Within-centre variations derive from a single machine's ability to measure results consistently, and so are informative of the precision of the machine. These variations were assessed by considering the six repeats for each diamond type individually, calculating the “scatter” in their results.

Further details of the analytical approaches are given in Clause 8, together with the results and discussion.

8 Results and discussion

8.1 Between-centre variation: all diamond types combined

The effect of the factor test centre (i.e. the assignable cause *man/machine*) was evaluated by combining all tests performed within each test centre. Each test centre performed 24 tests (6 repeats on each of 4 diamond types) and, when combined with equal weighting, these formed a “master” single particle strength distribution for the test centre with a median that can be called the overall centre median.

By comparing the six overall centre medians (both in terms of simple percentage differences, and by statistical significance tests such as the Mann-Whitney U test [7], described in Annex B), an appreciation of the fundamental differences in the results from each test centre (i.e. the underlying bias in the test centre's *man/machine*) was obtained. For ease of reference, all figures associated with between-centre variations are found in Annex C.

Table C.1 shows that the “master” distributions from each of the six centres were quite similar in terms of their principal statistics (in this table and others of a similar format, “P10” is used as an abbreviation of “10th percentile”, and so on). The medians of these distributions, the six overall centre medians, all lay within approximately ±2 % of the average overall centre median (found in the right column of the table).

Mann-Whitney U tests were performed to determine which overall centre medians were statistically significantly different from each other. The results are presented in Table C.6. As Annex B explains, a p value of less than 0,05 indicates a statistically significant difference (at the 95 % confidence level) between the two medians being compared.

Here, it was found that in 5 (out of the possible 15) comparisons the two medians were statistically significantly different. Whilst this initially seemed a surprisingly high number (given the apparent similarities between the distributions), it was most probably due to the high number (many thousands) of strength values

