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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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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

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[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 in each “master” distribution — as the sample size increases so does the confidence in the results, and hence even minor differences can become statistically significant.

In summary, perhaps the only notable case of bias in a test centre was that of Centre 1: the 10th, 25th and 90th percentiles of Centre 1 were higher than those of the other centres, and 4 of the 5 statistically significant differences between medians involved the median of Centre 1.

8.2 Between-centre variation: individual diamond types

Similar analyses were performed on individual diamond types — within each test centre the six repeats on a particular diamond type were combined to form a larger data set. By comparing the data sets from the six test centres, the bias of each test centre *as a* function of diamond type was examined. In other words, perhaps a particular test centre had a *man/machine* that showed systematic bias when measuring a particular diamond type. The equivalent study in ANOVA would be the interaction between test centre and diamond type.

Tables C.2 and C.7 show the distribution statistics and Mann-Whitney U test p values respectively for the diamond type HS601. The differences between the medians were greater here than for all diamond types combined — the six centre medians for HS601 were within around ±4 % of the average centre median. Centres 1 and 4 recorded generally high strength results, and Centre 3 recorded generally low strength results. This greater scatter also increased the number of medians that were statistically significantly different to each other (11 out of 15 comparisons).

The distribution statistics and Mann-Whitney U test results for diamond type HS427 are shown in Tables C.3 and C.8 respectively. For this diamond type, the six centre medians were within ±3 % of the average centre median, and medians were found to be statistically significantly different in 7 of the 15 possible comparisons.

The distribution statistics and Mann-Whitney U test results for diamond type MS427 are shown in Tables C.4 and C.9 respectively, whilst the distribution statistics and Mann-Whitney U test results for diamond type LS251 are shown in Tables C.5 and C.10 respectively.

The findings for the diamond type LS251 are notable in that the strength results from Centre 1 are substantially higher than those from the other centres. The medians from Centres 2 to 6 are very similar, and Table C.10 shows that when comparing medians from these centres, only two pairs of medians are significantly different. However, Centre 1's median is about 15 % higher than the average of the others, and is statistically significantly different from the others. It was confirmed that Centre 1's median value was not distorted by one or two “rogue” repeats; results from the six repeats were reasonably similar, and so it should be concluded that the offset of Centre 1's median compared to the other centre medians is a “real” effect.

The tables of “p values” presented in Tables C.6 to C.10 may be conveniently summarized non-numerically in tables of “homogeneous groups”. Tables of homogeneous groups for each of the diamond types are presented together in Table C.11. In such a table, each centre's median is represented by an X (or a row of Xs). Where different centres have Xs in the same column(s), there are no significant differences between their medians.

For example, at the right-hand side of Table C.11 there is a table of homogeneous groups for diamond type LS251. The X corresponding to Centre 1 (in the top row) does not overlap with any of the other Xs, and so this infers that Centre 1's median is significantly different from all of the other centres' medians (this is confirmed by the very low p values in Table C.10). The X corresponding to Centre 6 overlaps with those of Centres 2, 3 and 4, and hence their medians are not significantly different. However, Centre 6's X does not overlap with Centre 5's X, and so these two medians are significantly different. Again, these similarities and differences are confirmed by the p values in Table C.10. Other observations may be made in the same way.

The interaction effect between test centre and diamond type becomes evident when comparing the relationships between centre medians for one diamond type, and then for another diamond type. Centre 3, for example, recorded the lowest median strength for HS601 but the highest median strength for HS427. Centre 1, for example, recorded a particularly high median strength for LS251 and yet a very “average” median for HS427. That is, the respective biases of the test machines (as examined by considering all diamond types combined) changed according to the diamond type.

This interaction is perhaps best communicated by the table and graph in Table C.12 and Figure C.1, respectively. These show the bias (as a percentage) of a centre median from the average centre median for each diamond type, and for all diamond types combined. The biases were calculated simply from the median (P50) strengths in the Tables C.1 to C.5.

From Tables C.11 and C.12, two cases of centre bias are seen. Centre 1's five centre medians all lay on or above the average centre medians (the 0 % line on the y-axis), and Centre 5's medians all lay on or below the average centre medians. The medians of the other four centres were reasonably evenly distributed around the 0 % line.

Having gained an understanding of the measurement *bias* of each test centre (and the extent to which these biases are affected by diamond type), the measurement *precision* of each test centre was then examined.

8.3 Within-centre variation

Within-centre variation was analysed for each diamond type by comparing the six tests (“repeats”) within each centre. By examining only the relationship between the six repeats within a centre (without any comparison between centres) the effect of test centre *bias* was eliminated, thus isolating the effect of test centre *precision*.

Results of the six repeats from the six centres for diamond types HS601, HS427, MS427 and LS251 are presented in Annex D in Tables D.1, D.2, D.3 and D.4 respectively. For the purpose of brevity, only the strength distribution medians are given (rather than the selection of percentiles given in earlier tables). The average of the six medians for each centre is shown in the bottom row of each table.

There are two points of note here. Firstly, some values are absent from the tables. During strength testing for this study, test centres occasionally reported measurement difficulties (e.g. computer malfunctions, equipment faults) that either prevented completion of the test or invalidated the results. Results were only excluded from analysis where there were reported equipment problems; unexpected results obtained whilst the equipment was seemingly working normally have been retained in the analysis.

Secondly, during the analysis of within-centre variations it became apparent that for diamond type LS251 there was a systematic difference between the results from the first phase of the study (repeats 1 to 3) and the second phase of the study (repeats 4 to 6). This can be seen from the median strengths in Table D.4.

The only experimental factor that changed between the two phases was the polycrystalline disc from which the anvils were manufactured. In the specified experimental procedure, a pair of anvils used to test a sample LS251 were previously used to test a sample of MS427. It might be the case that the set of anvils used for these two diamond types in the second phase were more prone to chipping than those in the first phase. If so, the testing of MS427 might have induced additional anvil damage in the form of rough surfaces, which are known to result in lower recorded particle strengths.

As this difference was reported by most test centres (as would be expected if the anvil material was the source of the problem) the exact procedure for analysing results for LS251 was modified to compensate (further details of this are given shortly). It should be stressed that the systematic nature of this difference did not compromise the findings of the between-centre variations, as all centres were affected equally. Furthermore, statistical significance tests showed that for the other three diamond types (HS601, HS427 and HS251) there were no significant differences between the results from the first phase and the second phase of the study.

Statistical tests were performed to determine whether, for a particular diamond type in a particular centre, the medians from the six “repeats” were significantly different. For this, the Kruskal-Wallis H test (a version of the Mann-Whitney U test for multiple samples, and described briefly in Annex B) was used. The results of these tests (expressed as p values) are tabulated in Annex D, Table D.5.

Table D.5 shows that for each of the four diamond types measured by Centre 1, the six repeats were found to be insignificantly different. On the other hand, the p values from Centre 3 were less than 0,05 (indicating statistically significant differences) for all four diamond types.

It is unsurprising that the p values for the repeats of LS251 were less than 0,05 for Centres 2 to 6. This indicates that the scatter within the six repeats was high, and this was due to the systematic difference between the results from phase one of the study (repeats 1 to 3) and phase two (repeats 4 to 6). To compensate, the results from these two phases were analysed separately.

Average medians and p values for LS251 from the two phases have been included in the Tables D.4 and D.5. Table D.5 shows that the test centres generally had high consistency of results for LS251 when considering repeats 1 to 3 and repeats 4 to 6 separately; only two of the 12 relevant p values are less than 0,05.

The *precision* (consistency) of the results from each centre are perhaps best communicated by the Table D.6 and in Figure D.1, respectively. For each diamond type in each centre, the differences between the median strengths of individual repeats and the average of the six repeat medians were calculated. These six differences were then expressed as percentages of the average median. Obviously some of these percentage differences were negative values (for medians below the average) and the remainder were positive values (for medians above the average). In order to prevent the average of these being 0, the absolute values of the six differences were calculated (i.e. negatives values were converted to positives), and the mean (average) of these was taken.

This value may be defined as the “mean deviation of the median from the average median” for a particular diamond type in a particular centre, but has been called “scatter” for simplification. Larger numbers signify greater scatter within the group of six medians, and so the greater the scatter, the poorer the precision.

There is a certain correlation between the p values in Table D.5 and the scatter values in Table D.6, but the relationship is complex: six repeat medians could have a low scatter by consisting of five very similar medians, but the presence of one “outlier” could prevent all six being deemed to be from the same population.

8.4 Comparison of between-group and within-group variations

The measurement capability of each test centre (the assignable cause *man/machine*) can be summarized for all diamond types by calculating an average value for bias and an average value for scatter (inversely related to precision).

To calculate the average bias for each test centre, the average was taken of the bias values for HS601, HS427, MS427 and LS251. To calculate the average scatter for each test centre, the average was first taken of the scatter values for LS251 1–3 and LS251 4–6, and then the average was taken of this and the scatter values for HS601, HS427 and MS427 (this approach was taken to compensate for the offset in results from LS251 from the two phases of the study, and to ensure equal weighting for the four diamond types).

The average bias and average scatter for each test centre are presented graphically in Figure E.1 in Annex E. Overall, Centre 2 had the lowest scatter (and hence, best precision) and (together with Centre 6) the smallest bias. Centre 1's measurement bias was significantly affected by its results on LS251; in both phases of the study its results on this diamond type were much stronger than those of the other centres, and reasons for this should be investigated. Excluding its results on LS251, Centre 1's average bias would be approximately 2 %.

Another interesting observation was made by calculating the average bias and scatter as a function of diamond type (rather than centre). As the average bias for each diamond type was (by definition) 0, individual bias values were made absolute (all positive) prior to calculation of the average. A graph of average absolute bias against average scatter for the four diamond types is shown in Figure E.2.

There appears to be a relationship between bias and scatter: the diamond type which was measured with the greatest repeatability (precision) within each centre (HS427) was also measured with the least bias across the centres. Conversely, the diamond type measured with the highest within-centre variation (LS251, even compensating for the differences between the results of the two phases) also reported the highest between-centre variation.

It might reasonably be expected that the greatest measurement variation would derive from the diamond type with the broadest strength distribution (HS601), because the width of the strength distribution is a possible source of sampling variation, but that does not seem to be the case. The broader particle size distributions in HS427 and MS427 (another source of sampling variation) do not appear to have adversely affected measurement variation either.

Diamond type LS251 is at the edge of the “operating window” of the DiaTest-SI system (in terms of particle size and strength), and this is perhaps the reason for its greater measurement variation.

8.5 Estimation of accuracy of the single particle strength test

A final mathematical exercise was performed to estimate the overall accuracy of the single particle strength test for each diamond type. The aim of this was to produce a “bottom line” experimental error for each diamond type, such that in the “everyday” scenario of two DiaTest-SI users (e.g., a diamond manufacturer and a diamond toolmaker) comparing results, the extent to which the results are similar could be understood.

For each diamond type, the 36 medians (i.e. 6 repeats from each of 6 centres) were collected, and these formed a sampling distribution of the median for that diamond type (the concept of the sampling distribution is introduced in Annex A), which was theoretically normal. In a normal distribution, 95 % of the data points lie within ±1,96 standard deviations around the mean. Therefore, by constructing the sampling distribution of the median for a given diamond type, it is expected that (theoretically) when a sample of diamond is tested by any of these test centres, 95 % of the time the median will fall within the mean of the sampling distribution ±1,96 standard deviations. Converted to a percentage value, this 95 % interval is an appropriate measure of the overall experimental error of the single particle strength test according to this study.

The 95 % intervals of the sampling distributions of the medians for the four diamond types are shown in Figure F.1 in Annex F. Again, an adjustment has been made to the medians of diamond type LS251 to compensate for the offset in results from the two phases of the study.

The Table F.1 shows that for the three diamond types HS601, HS427 and MS427 95 % of the medians are expected to be within approximately ±8 % of the “true” median (the average of all 36 medians). For diamond type LS251, this rises to ±15 %. These numbers give good estimations of the accuracies to which DiaTest-SI machines in various centres can determine the median single particle strength of diamond (although perhaps in “everyday” scenarios where the diamond samples and anvils are less rigorously controlled the errors would be greater).

9 Consequences for a standard

For testing the diamond type LS 251 (lower strength/finer particle size) the test machine comes close to its limitation. It is physically possible to study this type but an experimental error of around ±15 % might be too large for reliable results.

10 Conclusions

A controlled study has been made of the feasibility of the Vollstädt “DiaTest-SI” system as a means of testing the single particle compressive strength of synthetic industrial diamond. Six test centres each performed six “repeat” measurements on each of four diamond types.

It was proven that non-parametric statistics were the most appropriate statistics for describing the strength distributions obtained, and so the median was chosen as the principal descriptor of distribution location.

Statistical analyses of the measured strength distributions showed that the experimental error of the test (expressed as the range within which 95 % of the recorded medians were expected to lie) was approximately ±8 % for the three coarser diamond types and approximately ±15 % for the fine, weak diamond type.

This variation was analysed in terms of contributions from measurement precision and measurement bias. Measurement precision (the repeatability of results within each centre) was expressed as the average “scatter” of the median strengths of a particular diamond type. This was found to be 2 % to 4 %, depending on the test centre. Measurement bias (the relationship between results from different centres) varied from −2 % to +5 %.

It was found that the machines of the six test centres yielded statistically significantly different results when considering all diamond types together, and, when considering individual diamond types, the patterns of results between different centres changed according to diamond type. From this, it was concluded that the factor *man/machine* and the interaction between *man/machine* and *material* (diamond type) had significant effects on the results.

The factor *material* (diamond type) had an obvious and entirely expected significant effect on the results (to the extent that this was not statistically tested in its own right). The other contribution to the factor *material*, the polycrystalline diamond anvils, did not significantly affect results except those of the fine, weak diamond type in the second phase of the study (repeats 4 to 6). It is possible that the lower strength results observed in the second phase derived from less chip-resistant anvils, and they demonstrate the importance of strictly-controlled conditions when testing the strength of such a superhard material.

Annex A
(informative)

Use of parametric and non-parametric statistics

A.1 Introduction to distribution statistics

In order that the distribution of particle strengths obtained for a sample be described correctly, its distribution statistics should be chosen carefully. Distribution statistics are values that describe the characteristics of a distribution, such as location, spread and shape.

Perhaps the most well-known distribution statistics are mean (or average) and standard deviation, which are measures of the location and spread respectively. These are so-called parametric statistics, in that these values are the parameters in a known probability distribution function (for example, entering the values for mean and standard deviation into the probability distribution function of a normal (Gaussian) distribution allows the calculation of the probability of occurrence of any value in that distribution).

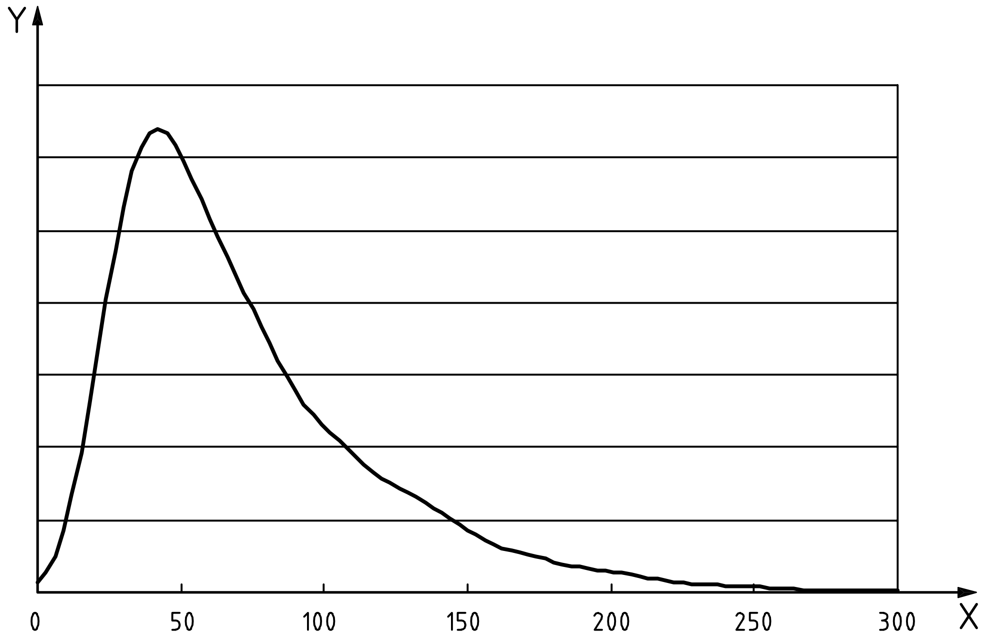
However, single particle strength distributions of diamond do not necessarily conform to any known probability distribution (they can be symmetrical or skewed, single-peaked or multi-peaked, depending on the design of the product). Consequently, knowledge of the mean and standard deviation is not sufficient to describe the entire distribution, and if used can lead to significant misinterpretation of the diamond strength distribution.

An effective way to summarize any distribution (irrespective of whether it conforms to a known probability distribution) is the use of non-parametric statistics, such as percentiles. By describing the values below which certain percentages of the results lie, percentiles essentially give the positions of various points along the cumulative probability distribution curve. For example, the 50th percentile (also called the median) is the value (in this case, the strength) below which 50 % of the results lie. The median, when used with the 10th, 25th, 75th and 90th percentiles, gives five “points” along the distribution curve which should be sufficient to describe the location, spread and shape of most strength distributions. In this case, the median replaces the mean as the measure of location, and the interquartile range (the difference between the 25th and 75th percentiles, also known as the 1st and 3rd quartiles) replaces the standard deviation as a measure of spread.

Whilst non-parametric statistics are still appropriate for describing symmetrical, normal-like strength distributions, they become particularly important for describing highly skewed strength distributions. The robustness of these statistics can be demonstrated by examining the sampling variations of a selection of parametric and non-parametric statistics, as has been done in the following exercise.

A.2 Sampling variations of selected distribution statistics

For this exercise, the entire data set (over 13 000 strength values, obtained by the six test centres) of diamond type LS251 was considered. The underlying strength distribution of this product, shown in Figure A.1, was highly skewed. From this population, a random sample of 500 data points was taken, and the distribution statistics of this sample were recorded. The process of taking a random 500-point sample and recording its distribution statistics was repeated another 49 times. Hence, 50 sample means were obtained, and these formed their own sampling distribution (which is theoretically normal) with its own mean and a standard deviation. The smaller this standard deviation (expressed as a percentage of the sampling distribution mean) was, the smaller the sampling variation in the mean. 50 sample standard deviations, 50 sample medians and 50 sample interquartile ranges were also obtained. Each of these statistics had its sampling distribution, and so its sampling variation could be calculated.



Key

X single particle strength, N

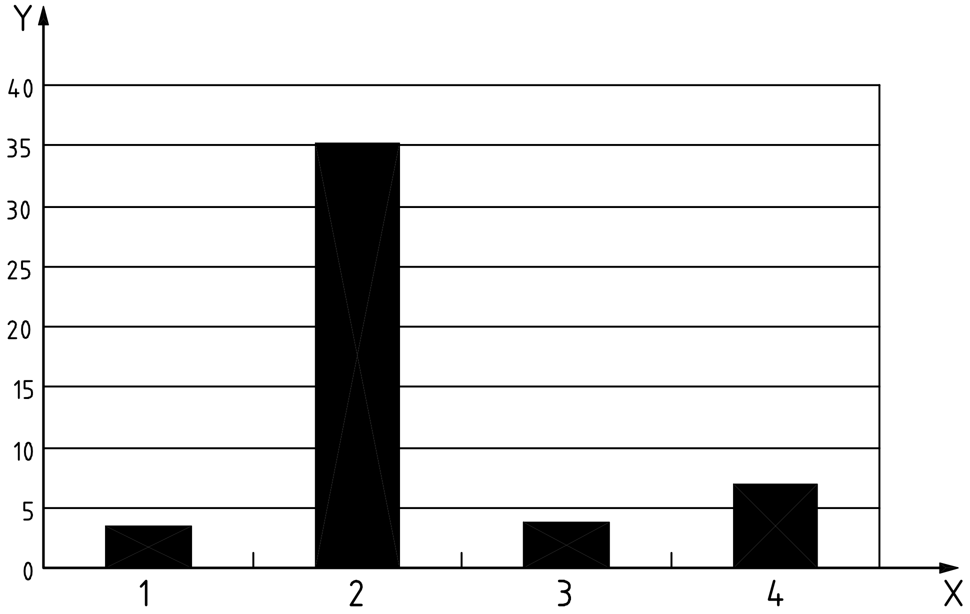
Y probability density

Figure A.1 — Population strength distribution of diamond type LS251

In such an exercise, the more robust distribution statistics are likely to change little from one sample to the next, and hence their sampling variations will be small. Other, less suitable, statistics may show much greater sampling variations. Figure A.2 shows the sampling variation (the sampling standard deviation as a percentage of the sampling mean) of the mean, standard deviation, median and interquartile range of the strength distribution of LS251.

In Figure A.2, the sampling variations of the mean and median are similar (the median is perhaps disadvantaged by the fact that all such percentiles are actual strength values, rather than calculated values). The most notable result is the sampling variation of the standard deviation, which is a very large 35 %. This shows that for such highly skewed strength distributions the standard deviation is an inappropriate measure of the spread of the distribution: first, when combined with the mean it might be used to recreate the underlying strength distribution on the assumption that it is normal (which it is not, as Figure A.1 demonstrates), and secondly, it can vary significantly from one sample to the next, suggesting a large batch-to-batch variation that is actually only simple sampling error.

As a strength distribution becomes more normal, the sampling variations of parametric statistics become more similar to those of non-parametric statistics. However, a consistency of approach is required for all types of strength distribution, and consequently it was decided that non-parametric statistics be used to describe the strength results obtained in this study, with the median being the chosen descriptor of distribution location.



Key

1 mean
2 StdDev
3 median
4 IQR

X distribution statistic
Y sampling variation, %

Figure A.2 — Sampling variations of four statistics used to describe the single particle strength distribution of diamond type LS251

Annex B
(informative)

Statistical significance tests

Statistical significance tests are used to determine whether two (or more) samples are different (or, put more correctly, whether two (or more) samples are taken from the same population). The most commonly known significance test is perhaps the Student's t test, though this is a parametric test and assumes that the underlying data are Normally distributed. However, it is also possible to perform non-parametric significance tests, and these are more appropriate given the non-normal nature of most strength distributions.

The Mann-Whitney U test (or simply, the U test) [7] is a rank sum test for comparing two samples. First, it combines the data points from the two samples, arranges them in ascending order, and assigns a rank to each value. Then, the ranks assigned to the data points from sample 1 are summed, and compared to the sum of the ranks assigned to the data points from sample 2. A significant difference between the rank sums from samples 1 and 2 implies a significant difference between the samples.

The parameter U itself is the total number of times that sample 2 values follow sample 1 values when all values are combined and placed in ascending order. From the U parameter (using mathematics that are outside the scope of this Technical Report) it is possible to calculate the p value, which states whether the two samples are significantly different.

The p value may be defined as “the probability of incorrectly rejecting the null hypothesis that the two samples come from the same population”. Hence, as the p value increases, rejecting the null hypothesis (concluding that the two samples are different) becomes more and more incorrect.

In order to make a statistical decision a “threshold” p value is chosen, below which the two samples are concluded as being different (because the probability of being incorrect is suitably low), and above which the two samples are concluded as being from the same population. The threshold p value chosen for this study was 0,05; this is commonly used and corresponds to what is known as the “95 % confidence level”.

The Kruskal-Wallis H test is a generalized form of the U test for evaluating whether two or more samples come from the same population. Whilst the mathematical intricacies of the H test are different from those of the U test, the two tests perform the same basic rank sum operation.

In practical terms, for a given diamond type the many samples tested were extracted from the same population using a robust random-splitting procedure, and so testing on a statistical basis whether samples derived from the same population gave an indication of the influences of other factors (“assignable causes”) on the results.

Annex C
(informative)

Between-centre variations

Table C.1 — Strength statistics for all diamond types combined

Values in newtons

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6	Average
Mean	318	310	308	312	303	312	310
P10	54	48	47	50	46	49	49
P25	118	110	104	107	105	110	109
P50	264	263	257	259	257	265	261
P75	449	439	440	450	437	441	443
P90	689	668	658	680	654	675	671

Table C.2 — Strength statistics for diamond type HS601

Values in newtons

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6	Average
Mean	620	599	575	609	573	595	595
P10	325	288	237	307	266	289	285
P25	468	450	416	460	426	427	441
P50	623	605	583	620	589	603	604
P75	770	755	742	763	736	757	754
P90	902	888	876	882	859	884	882

Table C.3 — Strength statistics for diamond type HS427

Values in newtons

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6	Average
Mean	326	321	336	320	324	332	327
P10	182	175	180	162	162	174	173
P25	240	234	243	229	232	244	237
P50	316	307	324	310	313	322	315
P75	398	394	413	405	405	410	404
P90	492	489	501	486	501	499	495

Table C.4 — Strength statistics for diamond type MS427

Values in newtons

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6	Average
Mean	250	248	245	243	246	249	247
P10	103	99	96	96	99	101	99
P25	158	153	144	146	146	153	150
P50	233	233	220	219	227	233	228
P75	322	327	317	316	324	326	322
P90	413	417	427	415	418	417	418

Table C.5 — Strength statistics for diamond type LS251

Values in newtons

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6	Average
Mean	77	73	76	75	69	71	73
P10	29	27	29	28	26	30	28
P25	42	39	39	39	37	40	40
P50	67	59	59	58	57	57	60
P75	101	92	88	91	88	90	92
P90	141	138	134	134	128	130	134

Table C.6 — “p values” from Mann-Whitney U tests between pairs of medians, for all diamond types combined

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
Centre 1						
Centre 2	0,036					
Centre 3	0,001	0,183				
Centre 4	0,031	0,970	0,187			
Centre 5	0,000	0,085	0,695	0,084		
Centre 6	0,121	0,554	0,059	0,571	0,021	

NOTE A p value of less than 0,05 represents a significant difference between the two medians at the 95 % confidence level.

Table C.7 — “p values” from Mann-Whitney U tests between pairs of medians, for diamond type HS601

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
Centre 1						
Centre 2	0,001					
Centre 3	0,000	0,000				
Centre 4	0,153	0,050	0,000			
Centre 5	0,000	0,001	0,738	0,000		
Centre 6	0,000	0,490	0,003	0,009	0,009	

NOTE A p value of less than 0,05 represents a significant difference between the two medians at the 95 % confidence level.

Table C.8 — “p values” from Mann-Whitney U tests between pairs of medians, for diamond type HS427

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
Centre 1						
Centre 2	0,068					
Centre 3	0,019	0,000				
Centre 4	0,104	0,901	0,000			
Centre 5	0,512	0,298	0,005	0,378		
Centre 6	0,072	0,000	0,554	0,001	0,018	

Table C.9 — “p values” from Mann-Whitney U tests between pairs of medians, for diamond type MS427

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
Centre 1						
Centre 2	0,773					
Centre 3	0,009	0,028				
Centre 4	0,002	0,007	0,686			
Centre 5	0,141	0,259	0,253	0,124		
Centre 6	0,851	0,924	0,020	0,006	0,207	

Table C.10 — “p values” from Mann-Whitney U tests between pairs of medians, for diamond type LS251

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
Centre 1						
Centre 2	0,000					
Centre 3	0,000	0,810				
Centre 4	0,000	0,842	0,696			
Centre 5	0,000	0,050	0,134	0,036		
Centre 6	0,000	0,986	0,855	0,786	0,041	

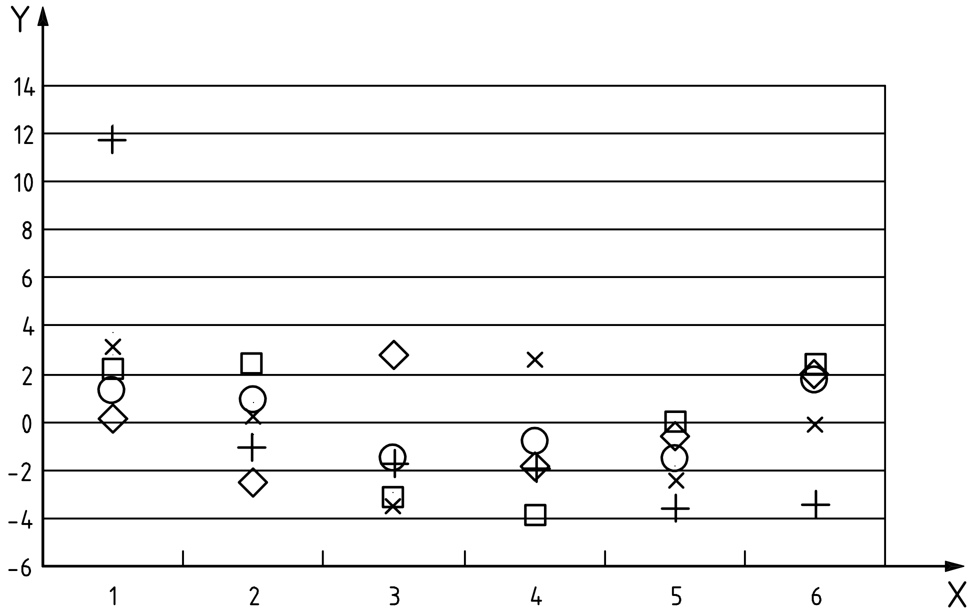
Table C.11 — Tables of “homogeneous groups” of centre medians, for each diamond type

	All types			HS601				HS427			MS427		LS251		
Centre 1			X				X	X	X			X			X
Centre 2	X	X			X	X		X				X	X	X	
Centre 3	X	X		X						X	X		X	X	
Centre 4	X	X				X	X	X			X		X		
Centre 5	X			X				X			X	X		X	
Centre 6		X	X		X				X	X		X	X		

NOTE X marks in the same column indicate no significant differences between centre medians for that diamond type.

Table C.12 — Biases of centre medians from average centre medians

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
HS601	3,11 %	0,23 %	− 3,50 %	2,65 %	− 2,42 %	− 0,08 %
HS427	0,11 %	− 2,48 %	2,82 %	− 1,84 %	− 0,60 %	1,99 %
MS427	2,24 %	2,47 %	− 3,18 %	− 3,85 %	− 0,06 %	2,38 %
LS251	11,78 %	− 1,09 %	− 1,69 %	− 1,92 %	− 3,63 %	− 3,46 %
All types	1,28 %	0,88 %	− 1,52 %	− 0,84 %	− 1,50 %	1,69 %



Key

X test centres 1 to 6

Y bias of median strength from average, %

×	HS601
◇	HS427
□	MS427
+	LS251
○	all types combined

Figure C.1 — Biases of centre medians from the average of the centre medians, for each diamond type

Annex D
(informative)

Within-centre variations

Table D.1 — Strength medians for diamond type HS601

Values in newtons

	Centre 1	**Centre 2**	**Centre 3**	**Centre 4**	**Centre 5**	**Centre 6**
Repeat 1	627	576	591	618	587	632
Repeat 2	610	614	576	614	581	590
Repeat 3	631	589	620	627		564
Repeat 4	637	621	603	614	575	601
Repeat 5		612	597	608	589	615
Repeat 6	601	613	521	635	605	621
Average	621	604	585	619	587	604

Table D.2 — Strength medians for diamond type HS427

Values in newtons

	Centre 1	**Centre 2**	**Centre 3**	**Centre 4**	**Centre 5**	**Centre 6**
Repeat 1	320	308	338	321	312	341
Repeat 2	305	304	321	304	315	316
Repeat 3	306	309	309	311	331	330
Repeat 4	313	305	322	308	302	312
Repeat 5	327	311	326	309	312	319
Repeat 6	323	307	340	307	309	321
Average	316	307	326	310	314	323

Table D.3 — Strength medians for diamond type M427

Values in newtons

	Centre 1	**Centre 2**	**Centre 3**	**Centre 4**	**Centre 5**	**Centre 6**
Repeat 1	237	227	231	215	233	217
Repeat 2	229	229	233	213	220	234
Repeat 3	235	236	223	218	224	232
Repeat 4	247	234	203	240	226	241
Repeat 5	226	230	214	202	241	235
Repeat 6	227	237	211	239	228	240
Average	233	232	219	221	229	233

Table D.4 — Strength medians for diamond type LS251

Values in newtons

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
Repeat 1	71	65	60	62	52	58
Repeat 2	68	62	63	64	56	57
Repeat 3	68	59	62	57	54	65
Repeat 4	61	59	56	58	60	57
Repeat 5	63	55	59	56	64	53
Repeat 6	67	57	52	54	53	53
Avg 1–6	66	59	58	59	57	57
Avg 1–3	69	62	61	61	54	60
Avg 4–6	64	57	56	56	59	54

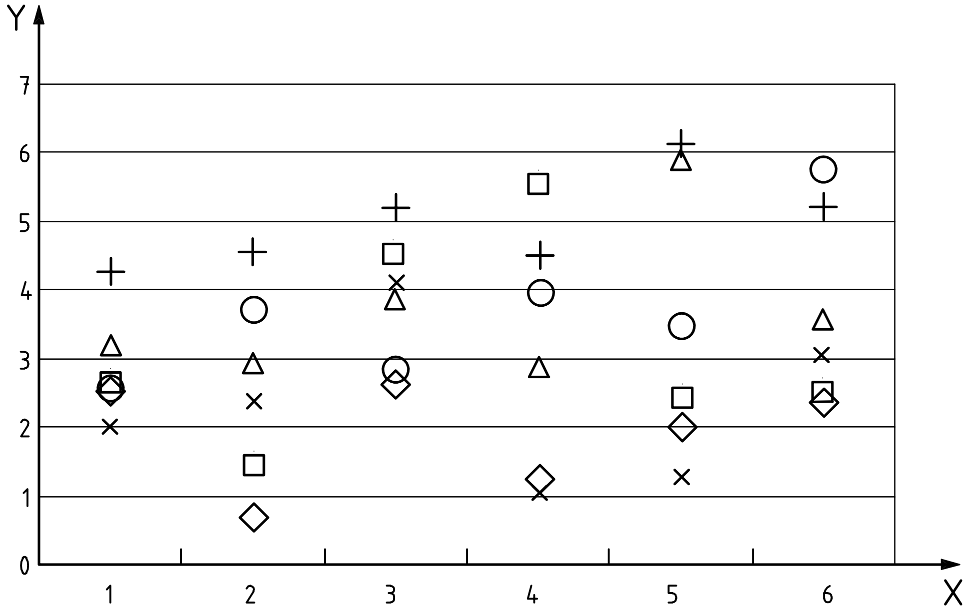
Table D.5 — "p values" from Kruskal-Wallis H tests comparing the six "repeat" medians within each centre, for each diamond type

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
HS601	0,477	0,177	0,000	0,357	0,127	0,018
HS427	0,361	0,642	0,020	0,662	0,009	0,039
MS427	0,759	0,956	0,002	0,002	0,073	0,425
LS251	0,144	0,042	0,023	0,028	0,002	0,000
LS251 1–3	0,533	0,547	0,478	0,622	0,451	0,001
LS251 4–6	0,313	0,816	0,107	0,139	0,000	0,515

NOTE A p value of less than 0,05 represents a significant difference between the medians at the 95 % confidence level.

Table D.6 — Scatter of each centre's medians for each diamond type, expressed as the mean deviation of the six medians from their average

	Centre 1	Centre 2	Centre 3	Centre 4	Centre 5	Centre 6
HS601	2,00 %	2,38 %	4,11 %	1,24 %	1,27 %	3,13 %
HS427	2,50 %	0,70 %	2,65 %	1,25 %	2,02 %	2,53 %
MS427	2,62 %	1,46 %	4,53 %	5,55 %	2,40 %	2,54 %
LS251	4,28 %	4,55 %	5,20 %	4,50 %	6,13 %	5,21 %
LS251 1–3	2,55 %	3,70 %	2,84 %	3,96 %	3,50 %	5,74 %
LS251 4–6	3,14 %	2,88 %	3,80 %	2,82 %	5,83 %	3,51 %



Key

X test centres 1 to 6

Y scatter of the six medians from their average, %

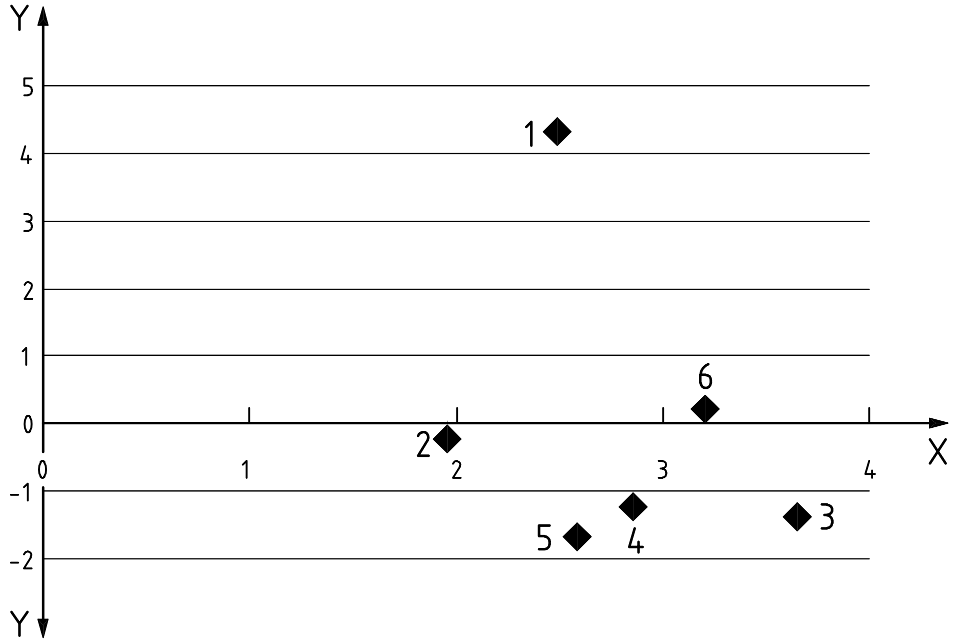
× HS601 ◇ HS427 □ MS427

\+ LS 251 ○ LS251 1-3 △ LS251 4-6

Figure D.1 — Scatter of each centre's medians for each diamond type, expressed as the mean deviation of the six medians from their average

Annex E
(informative)

Summary of between-centre and within-centre variations



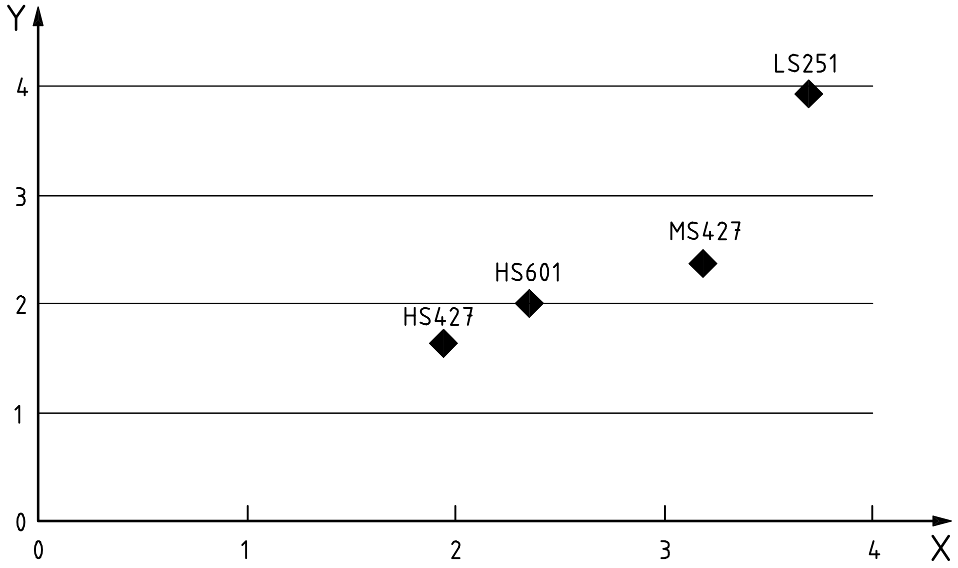
Key

X average scatter, %

Y average bias, %

1	Centre 1	2	Centre 2	3	Centre 3
4	Centre 4	5	Centre 5	6	Centre 6

Figure E.1 — Bias and scatter of each centre, averaged over the four diamond types



Key

X average scatter, %

Y average absolute bias, %

Figure E.2 — Bias and scatter of each diamond type, averaged over the six centres

Annex F

(informative)

Estimation of the experimental error of the single particle strength test

Table F.1 — Standard deviation and the 95 % interval of the sampling distribution of the median, for each diamond type

	Standard deviation	**95 % interval**
HS601	3,9 %	7,7 %
HS427	3,4 %	6,6 %
MS427	4,8 %	9,3 %
LS251	7,6 %	14,9 %
NOTE The 95 % interval is a good measure of experimental error.		

Bibliography

[1] ISO 6106:2005, *Abrasive products — Checking the grit size of superabrasives*

[2] BELLING N.G. Friatesting and Diamond Strength: A Review. *Industrial Diamond Review*, **52**, No. 550, 1992

[3] Diamond Characterisation. *General Electric Superabrasives Brochure*, GES1278E, 2000

[4] VOLLSTÄDT H. and LIST E. Controlling the Stability of the Properties of Superabrasive Powders. *Proceedings of 4th Zhengzhou International Superhard Materials and Related Products Conference*, 2003

[5] WHEELER D.J. and CHAMBERS D.S. *Understanding Statistical Process Contro*l, SPC Press, Knoxville, Tennessee, 1992

[6] MONTGOMERY D.C. *Introduction to Statistical Quality Control.* 3rd edition, Wiley, New York, 1997

[7] SPIEGEL M.R. and STEPHENS L.J. *Theory and Problems of Statistics.* 3rd edition, Shaum's Outline Series, McGraw-Hill, New York, 1999

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