# CHARACTERISATION OF SINGLE-CRYSTAL DIAMOND GRIT FOR CONSTRUCTION APPLICATIONS

#### Dr S. Nailer, T. Klein, M. Müller, Dr. S. Grasberger Development Diamond Insert Tools, Hilti Corporation, 9494 Schaan, Liechtenstein stuart.nailer@hilti.com

#### ABSTRACT

The last ten years have seen a step-change in the ability to position diamond particles in metalmatrix segments for drilling and sawing applications. Advances in segment assembly technology allow diamonds to be positioned at specific locations within the segment. Performance of the segment can be optimized when the diamond particles are themselves selected to best exploit arranged-diamond segment design and other cutting conditions (such as tool drive parameters).

Diamond 'grit' products for stone and construction applications each comprise particles of various sizes, shapes, strengths and purities. Consequently, effective selection of diamond types is dependent upon measurement using appropriate laboratory 'characterisation' techniques. As many of these techniques yield results on individual particles rather than bulk 'figures-of-merit', effective interpretation of these results is dependent on appropriate statistical analyses.

This paper will introduce the key characteristics of diamond products for construction applications, provide insights into appropriate laboratory characterisation techniques, and describe how their results may be best interpreted to facilitate understanding and consequent diamond selection.

#### **KEYWORDS**

Diamond, diamond segment, drilling, sawing, particle characterisation, particle size, particle shape, diamond strength, friability, image analysis

#### INTRODUCTION

In the conventional method of segment manufacture for stone and construction applications, diamond particles are mixed with metal powders and pressed to form segments. The diamonds are 'randomly distributed' (or 'statistically distributed') and efforts are made to reduce the probability of the 'clustering' of multiple diamonds in a small region. Recent developments in segment assembly technology allow diamond particles to be positioned at specific locations in the segment. As well as eliminating the possibility of diamond clustering, such 'arranged-diamond segment' technologies enable the diamonds to be assigned to a suitable position for the application conditions (such as the expected drilling/sawing performance, drive parameters of the tool motor, and 'base material' to be drilled/sawn). The performance of the diamond 'insert tool' (core bit, saw blade) can be optimized when the diamond particles are themselves selected to best exploit the design of the arranged-diamond segment and the application conditions. That is, the *right particles* come into operation in the right place, at the right time.

Particulate diamond products used in stone and construction sawing and drilling applications (commonly known as 'saw grit') each comprise particles of various sizes, shapes, strengths and purities. It is far from the case that all particles in a saw grit product exhibit similar characteristics. Consequently, the effective selection of diamond types for use in arranged-diamond segments is dependent on the quality of the laboratory measurements used to quantify the particle characteristics. Whilst some of these laboratory techniques provide simple single-value 'figures-of-merit', other techniques yield results from every individual particle. Thus, effective interpretation is dependent on the correct statistical analysis of these 'distributions' of results.

#### 1. DIAMOND SAW GRIT AND ITS CHARACTERISTICS

Synthetic diamond grit products (produced by high-pressure, high-temperature conversion from graphite) are normally graded and selected according to the key characteristics of size and strength. The size band of the product is selected according to whether the priority is surface finish (as in 80µm diamond for polishing) or material removal rate (as in 600µm diamond for drilling) [1]. The strength (or 'grade') of the product is then selected according to the characteristics of the base material to be cut and the drive parameters of the tool to be used. Diamond grit works most effectively by maintaining sharp cutting edges. The optimal diamond strength should therefore be high enough to prevent premature fracture and thus short working life, without being too high that the particles polish to a smooth surface and lose their cutting ability.

The term 'diamond characterisation' may be defined as the laboratory measurement of the properties of diamond believed to play important roles in behaviour in application. Two of the most important behaviours of insert tools for construction applications are speed (typically the depth drilled or area sawn per unit time) and lifetime (the total depth drilled or area sawn before the segments are fully worn). As previously implied, speed may be considered to be mostly dependent on particle size, whereas lifetime may be considered to be mostly dependent on particle strength (and its contributing factors, particle shape and metallic inclusion content).

The following sections describe in greater detail these key diamond characteristics, together with laboratory techniques for their quantification and the mathematical/statistical forms of their results.

#### 2. CHARACTERISATION OF PARTICLE SIZE

There are perhaps three important methods of characterising or expressing the size of diamond particles: sieving, image analysis, and particles per carat. Particles per carat ('PPC') describes the number of particles per unit mass (one carat being 0.2g), and is valuable to the segment manufacturer for expressing how a certain mass of diamond put into a segment translates into the number of available particles. Systems for measuring PPC are not widely commercially available, so particles are commonly counted manually and then weighed. Sieving and image analysis are described in further detail below.

#### 2.1 SIEVING

Sieving uses sieves of sizes defined by international standards [2] to physically separate diamond particles into size fractions. Sieves are traditionally defined by the number of lines per inch (the 'US mesh' system), with a corresponding aperture size specification in microns. Due to its principle of physical separation, sieving is used for the creation of diamond size bands (as well as their measurement). US mesh sizes may take the form of 'half sizes' or 'full sizes'. A half size is defined by a pair of sieves where the coarser ('upper defining') sieve has an aperture size 25-30% larger than that of the finer ('lower defining') sieve. For example, a size 30/35 US mesh half size diamond product is that which sits between a 30 mesh (645µm) sieve and a 35 mesh (505µm) sieve. Adjacent half sizes may be blended together to form a full size.

Whilst the particle size distributions of the graded half sizes are approximately 'normal' (Gaussian) in form, the blended full sizes tend to exhibit 'bimodal' particle size distributions containing the two modes (peaks) of the component half sizes. A graph showing the theoretical particle size distribution of a 30/40 US mesh full size is shown in Fig. 1.

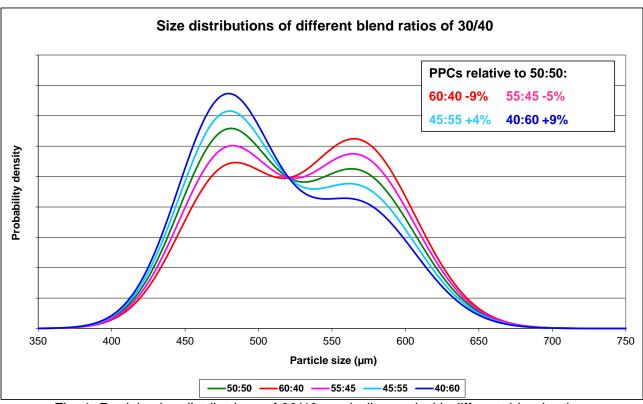


Fig. 1. Particle size distributions of 30/40 mesh diamond with different blend ratios.

The green curve in Fig. 1 represents the particle size distribution from 30/35 and 35/40 blended together in a 50:50 ratio. However, such blend ratios are not specified by international standards. Any blend of the two half sizes is therefore permitted, provided that the 'oversize' and 'undersize' fractions (in this case, coarser than 30 mesh and finer than 40 mesh) do not exceed a few percent.

Five curves representing five different blend ratios are shown in Fig. 1. It can be seen that, even within this fairly narrow range of blend ratios (60:40 to 40:60), the size distributions appear quite different. More significantly, there is a difference of around 18% in the particles per carat (PPC) of the 60:40 and 40:60 blend ratios, which could result in marked differences in application between the two blends even though they would both be correctly classified as '30/40 mesh'. As has been previously demonstrated [3], it is clear that understanding the underlying size distribution (and not just the nominal mesh size) is important for controlling diamond behaviour in application.

# 2.2 IMAGE ANALYSIS

Image analysis uses a computerised optical microscope to capture digital images of individual particles, which are then measured using various size and shape parameters. Measurement of individual particles enables full particle size distributions to be recorded. However, it should be noted that image analysis generates two-dimensional images, the third dimension not being visible.

A commonly-favoured image analysis size parameter is 'equivalent circle diameter' (illustrated schematically in Fig. 2). The equivalent circle diameter is calculated from the projected area of the particle, and is defined as the diameter of a circle with the same projected area as the particle. Equivalent circle diameter is sensitive to the longer visible dimension of the particle, and so the measured particle size distributions are larger than might be expected from sieving results. However, equivalent circle diameter, being derived from the area, has a good measurement resolution and gives smooth size distributions where small size differences are easily detected.

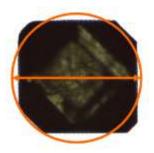


Fig. 2. Schematic illustration of equivalent circle diameter.

Particle size distributions of US mesh half sizes are typically normal (Gaussian), and can be described by the well-known distribution statistics of the 'mean' (average) and 'standard deviation' (spread). However, when a US mesh full size is created by blending two half sizes, the resulting size distribution is 'bimodal' (it has two peaks, as previously shown in Fig. 1). Equivalent circle diameter distributions of typical 30/35 and 35/40 half sizes are shown in Fig. 3, together with the 30/40 full size (blended using a 50:50 ratio of the half sizes). These distributions are shown in cumulative form. The lines entitled 'raw' show the as-measured data, and the lines entitled 'normal fit' show normal distributions with the same means and standard deviations as the 'raw' data.

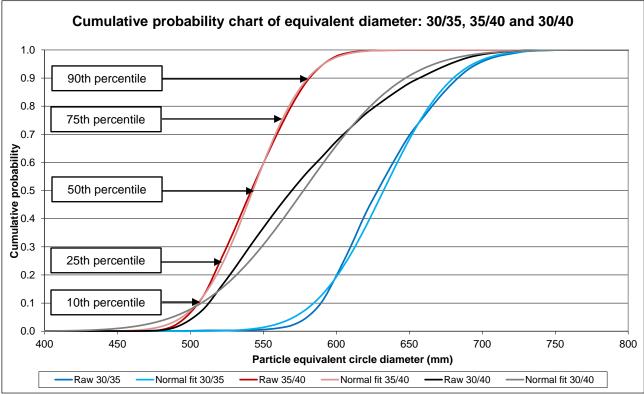


Fig. 3. Cumulative probability chart of equivalent circle diameter for US mesh half and full sizes.

It can be seen in Fig. 3 that the normal fits match the as-measured distributions quite well in the half sizes, but the normal fit for the 30/40 full size deviates substantially from the as-measured data. This is because means and standard deviations always assume a normal (Gaussian) symmetrical distribution shape, and are therefore inappropriate for describing distributions that have multiple peaks or are excessively peaked, flattened or skewed. A more appropriate method of describing distributions of any shapes or number of peak is to use 'percentiles', where the X<sup>th</sup> percentile is the value below which X% of the data points lie (when ordered smallest to largest). The most commonly-used percentile is the 50<sup>th</sup> percentile ('median'), which represents the middle value in the distribution. By using additionally the 10<sup>th</sup>, 25<sup>th</sup>, 75<sup>th</sup> and 90<sup>th</sup> percentiles, a distribution shape can be conveniently described by five 'non-parametric' statistics.

# 3. CHARACTERISATION OF PARTICLE SHAPE

The particle images used to measure particle size can also be used to measure particle shape. A useful shape parameter for saw grit diamond is compactness, defined as the ratio of the actual perimeter and the perimeter of a circle of the same area as the particle. A particle that appears circular (such as a highly crystalline cubo-octahedral diamond) will have a compactness near to 1, whereas particles which appear less round (for example, diamonds with elongation or lower crystallinity) will have compactness values higher than 1 (see Fig. 4).

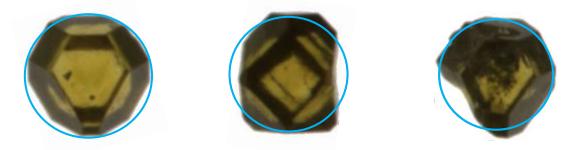


Fig. 4. Schematic illusion of compactness, with highly crystalline (left), elongated (centre) and less crystalline (right) diamond particles.

Compactness measurements on four adjacent saw grit product grades in size 30/35 are now demonstrated. Fig. 5 presents example images of these products, ranging from Grade 1 (a top-grade product) through to Grade 4 (a medium-grade product). It can be seen from the small selections of particle images in Fig. 5 that there are no dramatic differences in particle shape from Grade 1 to Grade 4. More noticeable are the increased populations of darker particles in the images to the right, and the reason for this is discussed later.

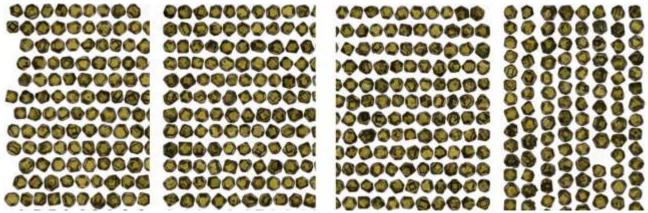


Fig. 5. Images of four 30/35 saw grit products: Grade 1 (left) to Grade 4 (right).

These observations of particle shape are confirmed by the distributions of compactness in Fig. 6. The compactness distributions of the four grades are close to one another, and furthermore, Grade 4 appears to have a better shape than Grade 3 (the Grade 4 compactness distributions being further to the left and therefore closer to the minimum compactness value 1). From this graph it may be concluded that adjacent saw grit products are not substantially different in their particle shapes because they are differentiated only by small differences in the amounts of well-shaped or poorly-shaped particles. Also, particle shape does not always rank correctly with the perceived product grade.

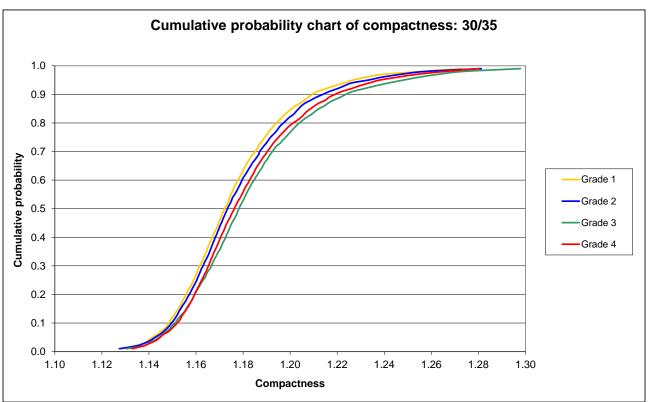


Fig. 6. Cumulative probability distributions of compactness of four saw grit grades in size 30/35.

# 4. CHARACTERISATION OF PARTICLE STRENGTH

Diamond product strength is typically characterised by two techniques: friability (or 'toughness') testing, and compressive strength testing.

# 4.1. FRIABILITY (TOUGHNESS) TESTING

Friability testing [4] measures the resistance of a bulk diamond product to cyclical impacts. The product is sieved to remove 'oversize' and 'undersize' particles, and the remaining 'on-size' particles are put into a steel capsule with a steel ball and subjected to a fixed number of oscillations ('cycles'). The product is then removed from the capsule and poured over a slightly finer 'post-impact' sieve. The diamond that has survived sits on this sieve, and is called the 'residue'. A stronger product registers a higher percentage residue (also called 'toughness index').

Friability testing is typically performed at a given number of cycles. For example, size 30/35 saw grit products are commonly tested using 1000 cycles. In this case, the test delivers a single percentage residue (toughness index) value – a figure-of-merit of product strength. However, all diamond products contain a distribution of particle strengths, and this distribution can be explored by testing at a range of different cycles. This provides a friability 'residue-time' curve, such as that in Fig. 7, which shows the friability residue-time curves of the four saw grit grades in size 30/35. At all cycles, the four grades show the 'correct' ranking (Grade 1 being the strongest, Grade 4 the weakest), but their relative strengths vary as a function of the number of cycles. The data points at different cycles may be connected by fitting a negative exponential curve according to the 'Rosin-Rammer equation' [5] (named after two scientists that studied the crushing behaviour of coal). The Rosin-Rammer equation is shown in Fig. 7, the constants *k* and *n* being characteristics of the diamond product. As well as allowing the prediction of percentage residue at any number of cycles, this equation enables the prediction of the number of cycles required for 50% residue – the 'half-life'.

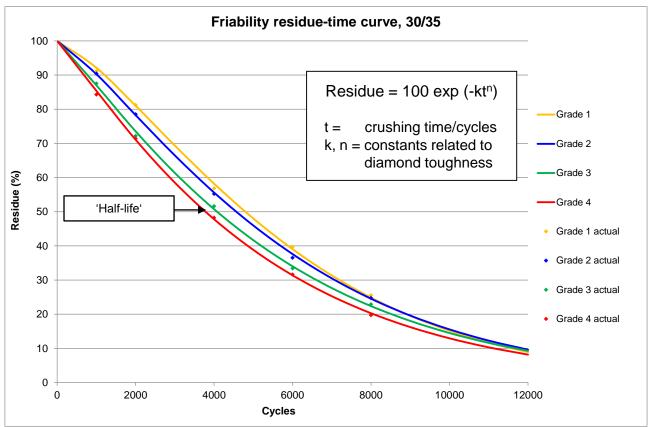


Fig. 7. Friability residue-time curves of four saw grit grades in size 30/35.

# 4.2. COMPRESSIVE STRENGTH TESTING

Compressive strength testing of diamond measures the force required to crush individual particles. Particles are crushed between two polycrystalline diamond 'anvils', and the load cell connected to the lower anvil measures the force required to fracture each diamond particle [6]. This method provides a distribution of particle strengths ('fracture forces') for each product. Fig. 8 shows the strength distributions of the four saw grit grades. For easier comparison with the friability residue-time curves, the vertical axis shows the probability of survival (rather than failure). Furthermore, the as-measured results have been replaced by Weibull distributions, which tend to fit compressive strength distributions well [7]. In this graph, the four grades rank as expected, with Grade 1 having the highest median compressive fracture force and Grade 4 the lowest.

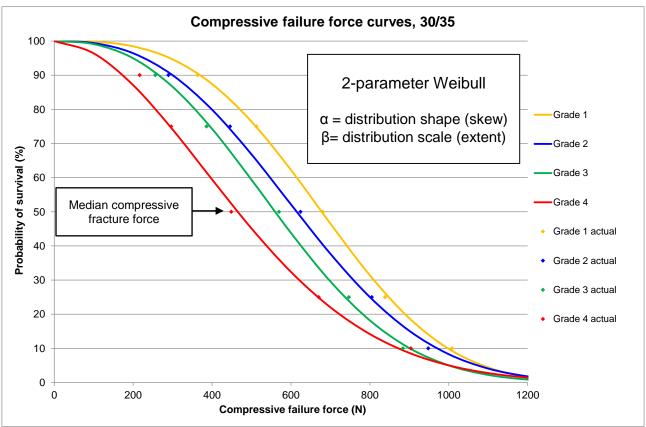


Fig. 8. Compressive strength distributions of four saw grit grades in size 30/35.

Whilst these two methods of strength testing can give complementary results, there are practical reasons for selecting one method or the other. Friability testing at one cycle selection is comparatively quick (around 10 minutes) but will only provide a single value figure-of-merit. The measurements required for a full residue-time curve would require over an hour. However, friability testing does have a good experimental error (around  $\pm 1\%$ ), and is therefore commonly used for quality control. Compressive strength testing enables a full distribution from 500 particles to be obtained in around 30 minutes, but the experimental error is usually greater due to damage of the polycrystalline diamond anvils influencing the fracture force measurements [8].

# 5. CHARACTERISATION OF METALLIC INCLUSION CONTENT

Saw grit diamonds are synthesised by the high-pressure, high-temperature conversion of graphite to diamond. Transition-metal alloys (typically iron-nickel) are used to lower the pressures and temperatures required for this conversion, and small traces of these alloys ('inclusions') can be trapped in the diamond crystals during the synthesis process (see Fig. 9 (left)). These inclusions can adversely affect diamond strength during high-temperature segment manufacture (by promoting re-graphitisation), but even at room temperatures the inclusions can act as 'weak points'. As these inclusions are ferromagnetic, their levels can be quantified in terms of 'magnetic susceptibility'. By measuring the magnetic dipole moment of the bulk diamond sample **M** in response to a magnetic field **H**, the (unitless) magnetic susceptibility  $\chi_{\nu}$  can be obtained. Fig. 9 (right) shows the magnetic susceptibility values of the four saw grit grades in size 30/35. These results show that Grades 1 and 2 have similar inclusion levels, whilst Grade 4 (with the highest magnetic susceptibility) has much greater inclusion content than Grade 3. This contributes to the darker appearance of many of the Grade 4 particles in the images shown in Fig. 5.

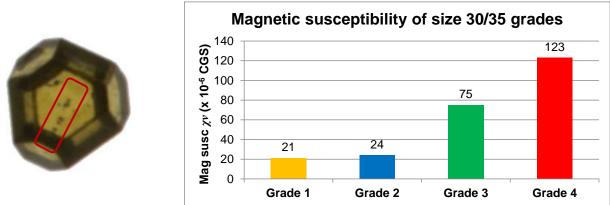


Fig. 9. Metallic inclusions in diamond (left, in red) and magnetic susceptibility results (right).

# 6. DISCUSSION AND CONCLUSIONS

Comparison of the shape, strength and inclusion measurements from these four grades enables the influences on diamond strength to be understood. These grades were ranked by the diamond manufacturer in the order 1, 2, 3 and 4 (strongest to weakest), and the results of both friability (toughness) and compressive strength confirm this. Whilst image analyses suggested that the particle shapes of Grade 4 were slightly better than those of Grade 3, magnetic susceptibility measurements showed the metal content of Grade 4 to be much higher. Consequently, Grade 4 delivered lower strength values than Grade 3.

The detailed results presented above focused on the mesh size 30/35. Particle shape, magnetic susceptibility and friability (toughness) results have also been obtained from the same grades in sizes 35/40 and 40/45, and compiling all these results leads to the contour plot in Fig. 10.

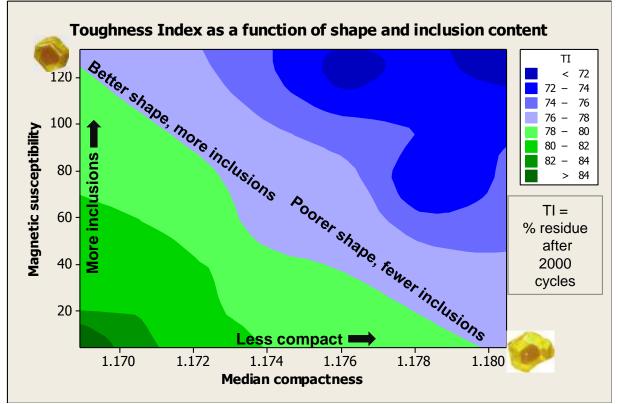


Fig. 10. Toughness index (friability residue) as a function of particle shape and inclusion content.

In Fig. 10 the vertical axis shows magnetic susceptibility and the horizontal axis the median compactness. The colours in the contour plot represent the toughness index (percentage residue), from less than 72 (dark blue) to greater than 84 (dark green). It can be seen that the highest toughness comes from products with the lowest magnetic susceptibility (inclusion content) and the lowest median compactness (most circular particle shape). Furthermore, by following the border between light blue and light green (a toughness index of 78), it can be seen that the same toughness values are obtained by products with low median compactness but high magnetic susceptibility and products with low magnetic susceptibility but high median compactness. From this, it can be concluded that saw grit product strength is primarily influenced by a combination of particle shape and inclusion content.

# REFERENCES

[1] Gielisse, P.J., 1998, 'Mechanical properties of diamond films and related materials'. In Gielisse, P.J. (ed.), Diamond and Diamond-Like Film Applications, Technomic, Pennsylvania, pp. 281-296.

[2] 'Abrasive products – Checking the grain size of superabrasives', ISO 6106:2013.

[3] Engels, A., 2003, 'The role of particles per carat in diamond tool behaviour', Industrial Diamond Review, 2/03, pp. 39-45.

[4] Belling, N.G. and Dyer, H.B., 1964, 'Impact strength determination of diamond abrasive grit', Industrial Diamond Information Bureau booklet, London.

[5] Rosin, P. and Rammler, E., 1933, 'The laws governing the fineness of powdered coal', Journal of the Institute of Fuel, pp. 29-36.

[6] Vollstädt, H. and List, E., 2003, 'Controlling the stability of the properties of superabrasive powders', 4<sup>th</sup> Zhengzhou International Superhard Materials and Related Products Conference.

[7] Gallagher, J., Scanlon, P. and Nailer, S.G., 2006, 'Characterisation techniques for the study of high-strength, coarse diamond', Industrial Diamond Review, 3/06, pp. 58-65.

[8] Nailer, S.G., 2006, 'Synthetic industrial diamond grit products – Single-particle compressive failure strength: "DiaTest-SI" system', ISO Technical Report ISO/TR 24857:2006.

# ACKNOWLEDGEMENTS

The authors express their gratitude to Messrs Markus Stengele and Stefan Fabbro for laboratory testing at Hilti AG, and to Vollstädt Diamant GmbH for provision of equipment and further measurements.