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(54) Title: FRIABILITY TESTING METHOD FOR SUPER-ABRASIVE GRAINS

(57) Abstract: A method of measuring an aggregate strength indication of a test sample comprising a plurality of super-abrasive grains having an aggregate mass. The method includes measuring the size frequency distribution of the super-abrasive grains and providing a friability tester apparatus, which comprises a drive mechanism, a crusher ball, and a capsule having an enclosable cavity for containing the test sample and the crusher ball. The capsule is configured such that the capsule can be moved at a cyclically varying speed for a number of cycles by the drive mechanism. An energy indication value is calculated as an integration over a full cycle of the square of the speed of the capsule, in metres per second, multiplied by half the mass of the crusher ball, in kilograms, multiplied by the number of cycles, divided by the aggregate mass of the grit sample, in grams. The method include enclosing the test sample and the crusher ball within the cavity; subjecting the capsule to the motion, such that the energy indication value is at least about 2 and at most about 50 ("Joules" per gram), to produce a comminuted sample; measuring the size frequency distribution of the comminuted sample; and calculating the aggregate strength indication based on the size frequency distributions of both the comminuted sample and the test sample.

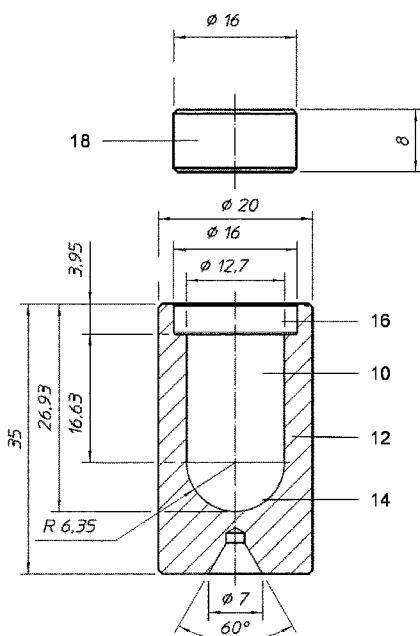


Fig. 1



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FRIABILITY TESTING METHOD FOR SUPER-ABRASIVE GRAINS

This disclosure relates generally to a method for evaluating the strength of super-abrasive grains.

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A comminution device known as a friability tester can be used for providing an indication of the friability of diamond grains. Broadly, the term "friability" refers to the degree to which grains of hard or super-abrasive material tend to fracture when subjected to impact with a body. A friability tester assembly comprises a cylindrical capsule which can be closed at its ends and a ball consisting of a hard, wear resistant material, which can be accommodated by the capsule and move freely within it. The test process includes introducing a plurality of diamond grains having a certain known mass and size distribution into the capsule with the ball, closing the capsule and causing it to be shaken back and forth in the direction of its cylindrical axis, causing the ball and the diamond grains to be violently agitated and the consequent fracturing the diamond grains. The shaking action is maintained for a certain number of cycles, at a certain amplitude and a certain frequency, after which all the diamond grains are recovered and the size distribution is measured (using the same method as used to measure the initial size distribution of the grains). Based on the differences between the initial and final size distributions, a friability index can be computed as the mass percentage of the unbroken grains in relation to the initial mass. The friability index had been used as an indicator of the likely performance of diamond grains of various particular grades in various industrial applications, such as sawing.

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A publication by N.G. Belling and H.G. Dyer, "Impact strength determination of diamond abrasive grit" (Industrial Diamond Information Bureau, London, 1964) explained that the friability test results would be a complex function of the strength of the grains as well as the conditions of operation of the test apparatus, such as the configuration of the capsule and the amplitude and frequency of the agitation. A series of experiments is disclosed to find friability test conditions suitable for standard application and frequencies of 15, 20, 40 and 50 cycles were tested using diamond grains having U.S. mesh size band of 100/120, in which the mean grain size was in the range of about 125 to about 150 microns. It was found that the comminution of diamond grit is very sensitive to the cycle frequency, which would therefore need to

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be rigorously controlled. On the one hand, anomalous results arise if the frequency is too low, and on the other hand, excessive breakage of the grains tends to occur if the frequency is too high. A frequency of 40 cycles per second was found to be satisfactory and was recommended as the standard frequency.

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A publication by N.G. Belling and L. Bialy, "The Friability tester™ - 10 years later" (Industrial Diamond Review, August 1974, pages 285 to 291) discloses an experiment to assess the sensitivity of friability test results to cycle frequency in the region of 40 cycles per second, in which frequencies in the range of 38.3 to 41.7 cycles per second were investigated using diamond grains having mean size of at least about 50 microns.

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United States patent number 5,140,857 discloses an electronically controlled friability testing apparatus, in which the cycle frequency is disclosed as being 40 cycles per second.

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A publication by Zhou *et al.* ("Friability and crushing strength of micrometre-size diamond abrasives used in micro-grinding of optical glass", *Metallurgical and Materials Transactions A*, volume 27A, April 1996, pages 1,047 to 1,053) discloses that no friability test results for diamond grains substantially less than 50 microns had been published prior to 1996. Results of the application of the friability test to diamond grains in the range of 3 to 20 microns using a cycle frequency of 20 cycles per second are disclosed.

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There is a need for a method of providing an indication of the strength of super-abrasive grit, with good discrimination between different grades of the super-abrasive grit having different strength, particularly but not exclusively grit comprising relatively small diamond or cubic boron nitride grains.

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According to a first aspect, there is provided a method of measuring an aggregate strength indication of a test sample comprising a plurality of super-abrasive grains having an aggregate mass;

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the method including:

measuring the size frequency distribution of the super-abrasive grains;

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providing a friability tester apparatus comprising:

a drive mechanism, a crusher ball, and a capsule having an enclosable cavity for containing the test sample and the crusher ball;

the friability tester apparatus configured such that the capsule can be moved at a cyclically varying speed for a number of cycles by the drive mechanism;

5 an energy indication value calculated as

an integration over a full cycle of the square of the speed of the capsule, in metres per second, multiplied by half the mass of the crusher ball, in kilograms, multiplied by the number of cycles, divided by the aggregate mass of the grit sample, in grams; enclosing the test sample and the crusher ball within the cavity;

10 subjecting the capsule to the motion, such that the energy indication value is at least about 2 and at most about 50 ("Joules" per gram), to produce a comminuted sample; measuring the size frequency distribution of the comminuted sample; and calculating the aggregate strength indication based on the size frequency distributions of both the comminuted sample and the test sample.

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Many variations of the method are envisaged by this disclosure, some non-limiting and non-exclusive examples of which are described below.

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In various examples, the super-abrasive grains may comprise or consist of diamond material or cubic boron nitride (cBN) material. Each of the super-abrasive grains may consist of at least one diamond or cBN crystal. At least 80 per cent of the super-abrasive grains may each consist of a single diamond or cBN crystal, or substantially all of the super-abrasive grains may each consist of a single diamond or cBN crystal.

25

In various examples, the super-abrasive grains may have a mean size of at least about 1 micron or at least about 2 microns. The super-abrasive grains may have a mean size of at most about 80 microns, at most about 50 microns, at most about 30 microns or at most about 20 microns. In some examples, the super-abrasive grains
30 may consist of diamond grit having mean size from 10 micron to 20 microns.

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In various examples, the aggregate mass may be at least about 0.1 gram (g), at least about 0.2 gram (g) or at least about 0.3 gram (g). In various examples, the aggregate mass may be at most about 0.8 gram (g), at most about 0.6 gram (g), at most about 0.4 gram (g) or at most about 0.3 gram (g). In some examples, the

aggregate mass may be in the range of about 0.15 to about 0.25 gram (g). In some examples, the aggregate mass may be about 0.20 gram (g).

5 In various examples, the size distribution frequencies of each of the test sample and the comminuted sample may be measured by means of a laser diffractometry apparatus, a Coulter counter apparatus or a sieve device.

10 In various examples, the crusher ball may have a diameter of at least about 6 millimetres (mm), at least about 7 millimetres (mm) or at least about 7.5 millimetres (mm). The crusher ball may have a diameter of at most about 10 millimetres (mm), at most about 9 millimetres or at most about 8.5 millimetres (mm). In some examples, the crusher ball may have a diameter of about 7.9 millimetres. In various examples, the crusher ball may have a diameter in the range of about 6 to about 10 millimetres, about 7 to about 9 millimetres, or about 7.5 to about 8.5 millimetres
15 (mm).

In various examples, the mass of the crusher ball may be at least about 1 gram (g) or at least about 2 grams (g). The mass of the crusher ball may be at most about 4 grams (g) or at most about 3 grams (g). In some examples, the mass of the crusher
20 ball may be about 2.04 grams (g). In various examples, the mass of the crusher ball may be in the range of about 1 to about 3 grams (g) or about 2 to about 3 grams (g).

In some examples, the crusher ball may comprise steel. The surface of the crusher ball may consist of chrome steel, ceramic material or super-hard material. In some
25 examples, the surface of the crusher ball may be defined by a surface of a diamond film or of PCD material. The surface of the crusher ball may have a Rockwell C hardness of at least about 50 HRc.

In some example arrangements, the enclosable cavity may be substantially
30 cylindrical in shape. The enclosable cavity may have a length of at least about 15 millimetres (mm) and at most about 30 millimetres (mm) or at most about 20 millimetres (mm). The enclosable cavity may be substantially cylindrical in shape, having a diameter of at least about 10 millimetres (mm) and at most about 20 millimetres or at most about 15 millimetres (mm).

In some example arrangements, the enclosable cavity may be substantially cylindrical in shape, having a cylindrical axis, and the motion may comprise reciprocation along the cylindrical axis. The motion may comprise at least one cycle in which the speed of the capsule varies substantially as a sinusoidal function of time.

- 5 In some examples, the motion may comprise at least one cycle in which the speed of the capsule varies substantially as a saw-tooth function, substantially as a square and or substantially as a triangular function of time (in other words, when the speed of the capsule is plotted verses time, the shape of the plotted speed appears to be square or triangular, respectively). The speed of the capsule may vary in
10 substantially the same way in each cycle or it may vary in different ways during a single test.

- Some example friability tester apparatuses may comprise a capsule comprising a tube and a pair of stoppers, configured such that the opposite ends of the tube can
15 be securely closed by means of respective stoppers when the capsule is in an assembled condition and both stoppers can be reversibly detached to open the tube at both ends. The diameter of the cavity will be sufficiently great that it can accommodate a crusher ball such that the crusher ball can move freely within the cavity, and the length of the cavity when the stoppers are attached in the assembled
20 condition may be at least double the diameter of the crusher ball, so that the crusher ball can move axially within the cavity. The stoppers may provide substantially planar or concave ends for the cavity when in the assembled condition. The stoppers may provide differently shape respective ends of the cavity.

- 25 In various examples, the number of cycles may be at least about 6,000, at least about 8,000, at least about 10,000 or at least about 12,000. The number of cycles may be at most about 20,000, at most about 16,000 or at most about 14,000. In various examples, the number of cycles may be in the range of about 10,000 to about 16,000, or about 13,000 to about 14,000.

- 30 In various examples, the capsule may be driven to move at the cyclically varying speed for a period of at least about 120 seconds or at least about 240 seconds. In various examples, the capsule may be driven to move at the cyclically varying speed (in other words, the cyclical motion of the capsule may be repeated without
35 interruption) for a period of at most about 500 seconds or at most about 420

seconds. In some examples, the capsule may be driven to move at the cyclically varying speed for about 300 seconds.

5 In various examples, the motion may comprise a plurality of repeated cycles, the frequency being at least about 40 cycles per second, at least about 42 cycles per second or at least about 44 cycles per second. The frequency may be at most about 60 cycles per second, at most about 50 cycles per second to at most about 48 cycles per second. In various examples, the frequency may be about 40 cycles per second, about 45 cycles per second or about 50 cycles per second.

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In some example arrangements, the drive mechanism may comprise an electro-mechanical device. The motion may be controlled by a mechanism comprising piezoelectric component.

15 In various examples, the energy indication value may be at least about 4 Joules per gram of sample grit, at least about 8 Joules per gram of sample grit or at least about 10 Joules per gram of sample grit. In some examples, the energy indication value may be at most about 20 Joules per gram of sample grit.

20 In various examples, the maximum speed of the capsule during each cycle may be at least about 0.5 metres per second (m/s), at least about 0.7 metres per second (m/s) at least about 0.8 metres per second or at least about 0.9 metres per second (m/s). in some examples, the maximum speed of the capsule during each cycle may be at most about 2 metres per second (m/s) or at most about 1 metre per second (m/s).
25 some examples, the maximum speed of the capsule during each cycle may be about 0.72 metres per second (m/s), about 0.81 metres per second (m/s) or about 1.27 metres per second (m/s). In some examples, the maximum speed of the capsule during each cycle may be substantially the same or it may differ.

30 In some examples, the aggregate strength indication may be calculated using the respective median values ($D(v, 0.5)$) of the grain size distributions of the test sample and the comminuted sample, the respective ninetieth percentile values ($D(v, 0.9)$) of the grain size distributions of the test sample and the comminuted sample, and or the differences between the ninetieth percentile value and median values ($D(v, 0.9) -$
35 $D(v, 0.5)$) of each of the grain size distributions of the test sample and the

comminuted sample. In some examples, the aggregate strength indication may be calculated as the difference between the ninetieth percentile value and median value of the comminuted sample divided by the difference between the ninetieth percentile value and median value of the test sample. In some examples, the aggregate strength indication may be calculated using the respective mean volumetric mean values ($D[4,3]$) of the grain size distributions of the test sample and the comminuted sample. In some examples, the aggregate strength indication is calculated as the volumetric mean value of the comminuted sample divided by the volumetric mean value of the test sample.

In some example methods, each crusher ball may be used only once. In other words, each test may be carried out using a new crusher ball, which will not have been previously used in a wear application. This will likely have the effect of enhancing the reliability of the test method, although it is likely that in some example variations, a crusher ball may be used in more than one test, for example in 2 or 3 tests.

In some examples, the maximum speed of the capsule during each reciprocation cycle may be in the range from about 0.5 to about 1.0 metre per second (m/s). For example, the maximum speed of the capsule during each reciprocation cycle may be 0.72 metres per second (m/s). For example, the maximum speed of the capsule during each reciprocation cycle may be 0.81 metres per second (m/s) and the frequency may be about 45 cycles per second.

In some example arrangements, the frequency may be selected such that the median size $D(0.5)$ shifts by a specified degree after a specified time period, given a number of reciprocation cycles and other relevant factors.

In some examples, the method may include heat treating the grit sample by heating it to a temperature for a period of time prior to carrying out a friability using the heat treated sample. For example, the sample may consist of grains comprising or consisting of natural or synthetic diamond and the method may include heat treating the sample at a temperature of at least about 400 degrees Celsius for at least about 30 seconds. In some examples, the temperature may be at most about 1,100 degrees Celsius. The period of time of the heat treatment may be at most about 600

seconds or at most about 300 seconds. The heat treatment may be carried out in an inert or non-oxidising atmosphere, such as argon or nitrogen, and or at a pressure substantially less than 100 kilopascals (kPa).

- 5 Methods that include heat treating the sample grit prior to carrying out the friability test on will likely have the aspect of providing an indication of the thermal stability of the grit sample. In other words, such methods will likely give an indication of the extent to which the strength grit sample will be sustained at elevated temperatures, as may be experienced in certain cutting, grinding, lapping or polishing applications.

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Non-limiting example methods and friability tester apparatuses will be described with reference to the following drawings, of which

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Fig. 1 shows a longitudinal cross section view of a capsule assembly in unassembled conditions, in which the length dimensions shown are in millimetres;

Fig. 2 shows a graph of the velocity and the kinetic energy indication versus time, of a single full cycle of example sinusoidal motion of a capsule;

Fig. 3 shows a graph of three curves of normalised kinetic energy indication as functions of frequency of cyclical motion for three different periods of time.

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With reference to Fig. 1, an example capsule for a friability tester apparatus may comprise a generally tubular body 12 and a stopper 18. The stoppers 18 and the tubular body 12 are configured such that the stopper 18 can be attached to an end 16 of the tubular structure 12 to provide a fully enclosed cavity 10, within which a crusher ball can be contained when in the assembled condition. The stopper 18 is configured such that when it is attached to the end 16 of the tubular body 12, it provides a planar end of the cavity. An opposite other end 14 of the tubular body 12 provides a spherically concave end 14 of the cavity 10.

30

In use, a sample of super-abrasive grit and a crusher ball will be enclosed within the capsule in the assembled condition and the capsule will be driven to oscillate along the cylindrical axis, causing the grit and the crusher ball to be shaken back and forth within the cavity. The cyclical motion will trace a varying axial position of the capsule as a function of time, the instantaneous velocity of the capsule at any point in the cycle being the first derivative of its position with respect to time. The capsule will

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oscillate axially between opposite furthest extents of its position, this distance being the amplitude of the motion. The grains comprised in the grit sample will be repeatedly impacted between the crusher ball and the capsule, and or other grains, and at least some of the grains will likely be fractured in this comminution process.

5

The instantaneous kinetic energy of the capsule, in units of Joules (J), will be half the mass of the capsule in units of kilograms (kg) times the square of its instantaneous velocity in units of metres per second (m/s). An indication or proxy of the kinetic energy of the crusher ball is to be calculated as half the mass of the crusher ball in units of kilograms (kg) times the square of the instantaneous velocity of the capsule. Although this will not be likely to provide an accurate value of the kinetic energy of the crusher ball, since the velocity of the crusher ball will not likely be the same as that of the capsule, units of Joules (J) will be used in relation to the kinetic energy indication value. The kinetic energy indication value may be divided by the mass of the grit sample to provide a normalised kinetic energy indication, expressed in units of Joules per gram (J/g). The instantaneous kinetic energy indication values can be integrated over an entire cycle and over all the cycles comprised in the test, the latter providing the kinetic energy indication value of the friability test according to the disclosed method.

20

The velocity and corresponding kinetic energy indication values of an example capsule and crusher ball subjected to sinusoidal oscillation are shown as functions of time in Fig. 2. In this particular example, the mass of the crusher ball is 0.00204 kilograms (kg), the frequency of the motion is 45 cycles per second and the maximum speed of the capsule is about 1.272 metres per second (m/s). The integrated value of the kinetic energy indication curve over a single full oscillation is 82.5 millijoules (mJ) and the integrated value over 13,500 oscillations, as may be used in a friability test according to this disclosure, is 11.1 Joules (J). In an example friability test, the mass of the grit sample may be 0.2 grams and the normalised kinetic energy integration value for the test is therefore 55.5 Joules per gram (J/g). In an example in which the maximum speed of the capsule during each oscillation is 1 meter per second (m/s), all else being the same, the normalised kinetic energy indication value would be 34.5 Joules per gram (J/g).

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Table 1 shows the integrated kinetic energy indication values for 300 seconds (s) of testing of a diamond grit sample having a mass of 0.2 grams, as well as the corresponding normalised values per gram of sample grit (rounded up to the nearest tenth). The mass of the crusher ball is taken to be 2.04 grams, the motion of the capsule is sinusoidal and the maximum speed of the capsule is 1 metre per second (m/s). Fig. 3 shows the normalised kinetic energy indication values for two different test periods.

Frequency c.p.s.	Integrated kinetic energy indication, J (300 s of operation)		Normalised kinetic energy indication, J/g	
	100 s	300 s	100 s	300 s
40	2.04	6.12	10.2	30.6
43	2.19	6.58	11.0	32.9
45	2.30	6.89	11.5	34.5
47	2.40	7.19	12.0	36.0
50	2.55	7.65	12.8	38.3
55	2.81	8.42	14.1	42.1

Table 1

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A first strength indication value may be calculated as the difference between the value of $D(0.9)$ of the grit sample after comminution and the value of $D(0.5)$ before comminution, divided by the difference between the $D(0.9)$ and $D(0.5)$ values of the grit sample before comminution. This is a measure of the shift in the ninetyth percentile (90'th percentile, $D(0.9)$) of the grain size distribution in relation to the fiftieth percentile (50'th percentile, $D(0.5)$). Clearly, if $D(0.9)$ decreases to less than $D(0.5)$ as a result of the comminution, the indication value will be negative, indicating severe comminution.

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A second strength indication value may be calculated as the ratio of the volumetric mean grain size, $D[4,3]$, after comminution to that before comminution; and a third strength indication value may be calculated as the ration of the median value after comminution to that before comminution.

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The grain size distribution of the super-abrasive grains may be measured by means of a laser diffraction apparatus or a Coulter counter, for example.

5 The disclosed method has the aspect of providing an indication of the strength of very fine super-abrasive grit with sufficient resolution between grades of the grit exhibiting different behaviour in application, the indication being substantially consistent with expectations based on the behaviour. Therefore, the method will likely be helpful in indicating the expected behaviour of very fine super-abrasive grit in certain applications.

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Various aspects of the test operation can be selected, provided that tests are carried out consistently if the results of different tests are to be compared. For example, the capsule may be reciprocated vertically or horizontally, or at some other orientation, and the selection of orientation may potentially have an effect on the results since
15 this may affect the average position and spatial distribution of the sample grit averaged over the test.

In some example methods, the test may comprise carrying out a test for a particular period of time at a certain frequency and aspects of the size distribution of the
20 comminuted grit compared or combined in a particular calculation with that of the sample grit. In other example methods, the time period required to fracture half of the grit sample may be reported as the strength indication value.

Various standards for friability test conditions and or configurations may be specified.
25 Different standard test conditions and or configurations of the capsule, crusher ball (or balls) and or reciprocation motion of the capsule may be specified for grains comprising or consisting of different super-hard material, for grains having different mean sizes (or according to some other feature of the grain size distribution) or for grains of different grades or strengths.

30

In some examples, the friability test procedure may be specified for a particular number of cycles and the result may be expressed in terms of the fraction of grains remaining within a specified size range.

In some examples, the number of cycles may be varied in a series of different tests in order to determine the number of cycles required to achieve some specified change in one or more features of the grain size distribution. For example, the result may be expressed in terms of the number of cycles required to shift the mean size of the grains to half of the mean size value for the test sample prior to comminution.

In some examples, the friability test results may be expressed in terms of a strength indicator *SI* value that may be calculated as the difference between the ninetieth percentile of the size distribution of the comminuted grit $D'(v,90)$ and the fiftieth percentile of the size distribution of the sample grit $D(v,50)$, divided by the difference between the ninetieth percentile of the size distribution of the sample grit $D(v,90)$ and the fiftieth percentile of the size distribution of the sample grit and the sample grit $D(v,50)$, as follows:

$$SI = (D'(v,90) - D(v,50)) / (D(v,90) - D(v,50))$$

In some examples, the results may be expressed in terms of a mean shift *MS* value that may be calculated as the ratio of the volumetric mean of the size distribution of the comminuted grit $D'[4,3]$ to that of the sample grit $D[4,3]$, as follows:

$$MS = D'[4,3] / D[4,3]$$

The disclosed strength tests are likely to have the aspect that they are able to discriminate satisfactorily between high and low strength super-abrasive grit, having mean grain size of at most about 60 microns.

A non-limiting example is described below.

Various aspects of the friability tester apparatus and the test method used in this example are summarised in Table 2. A new crusher ball was used for each test. After the comminution event, the ball and all the grains and powder comprised in the comminuted grit sample are removed from the capsule. The grit sample was combined with water and agitated ultrasonically to break up agglomerations of grains. The respective grain size distributions of both the test grit sample and the

comminuted grit sample were measured by means of a laser diffractometry apparatus.

- Two grades of diamond grit in the size range of 10 to 20 microns were tested by means of the disclosed method. The first grade was MDA™ grit (commercially available from Element Six™) and the second grade was CDA™ grit (commercially available from Element Six™).

Friability tester parameter	Value
Crusher ball diameter	7.94 mm
Crusher ball material	Chrome steel alloy
Capsule cavity length	19 mm
Capsule cavity diameter	12.7 mm
Capsule cavity configuration	Cylindrical tube closed at one end with a planar surface and the opposite end by a spherically concave surface
Frequency	45 cycles per second
Distance amplitude of cyclical motion (peak to peak)	9 mm
Time period of the test	300 seconds
Number of cycles	13,500
Super-abrasive grains	Diamond powder
Mass of test sample aggregation	0.20 grams

Table 2

- The two grades were each tested by means of the friability tester apparatus, in which the frequency was 45 Hz and the number of cycles was 16,500. The sample size was 0.4 grams, the mass of the crusher ball was 2.04 grams and the maximum speed was 1.272 metres per second (m/s). Therefore, the integrated kinetic energy indication was 11.14 Joules (J) and the normalised energy indicator was 27.85 Joules per gram (J/g). A new crusher ball was used for each measurement.

- Parameters of the size distributions of the test grit sample and the comminuted sample produced by the test are shown in Table 3.

Grade and mean size range, microns	Sample grit				Comminuted grit				Median Shift, %
	$D(v,0.1)$	$D[4,3]$	Median $D(v,0.5)$	$D(v,0.9)$	$D(v,0.1)$	$D[4,3]$	Median $D(v,0.5)$	$D(v,0.9)$	
MDA 10-20	8.9	12.7	12.4	17.1	1.1	6.3	5.8	12.4	47
CDA 10-20	8.6	12.4	12.0	16.8	1.0	5.1	4.0	10.8	33

Table 3

The median shift of the MDA sample was substantially greater than that of the CDA sample, indicating that the mean strength of the former grit is higher than that of the latter grit.

Certain terms and concepts as used herein are briefly explained below.

As used herein, friability is an extrinsic property of solid material indicating the degree to which it can to be reduced to smaller pieces when energy is applied to it.

As used herein, comminution is the process in which grains are reduced in size, by crushing, grinding or other processes.

The size range of abrasive grains may be expressed in terms of U.S. Mesh size, in which two mesh sizes are provided, the first being a mesh size through which the grains would pass and the second being a mesh size through which the grains would not pass. Mesh size may be expressed in terms of the number of openings per (linear) inch of mesh.

As used herein, grain sizes expressed in length units such as micrometres (microns), as opposed to mesh size, will refer to the equivalent circle diameters (ECD), in which each grain is regarded as though it were a sphere. The ECD distribution of a plurality of grains can be measured by means of laser diffraction, in which the grains are disposed randomly in the path of incident light and the diffraction pattern arising from the diffraction of the light by the grains is measured. The diffraction pattern may be interpreted mathematically as if it had been generated by a plurality of spherical grains, the diameter distribution of which being calculated and reported in terms of

ECD. Aspects of a grain size distribution may be expressed in terms of various statistical properties using various terms and symbols. Particular examples of such terms include mean, median and mode. The size distribution can be thought of as a set of values D_i corresponding to a series of respective size channels, in which each

5 D_i is the geometric mean ECD value corresponding to respective channel i , being an integer in the range from 1 to the number n of channels used.

Mean values obtained by means of laser diffraction methods may be most readily expressed on the basis of a distribution of grain volumes, the volume mean can be

10 represented as $D[4,3]$ according to a well-known mathematical formula. The result can be converted to surface area distribution, the mean of which being $D[3,2]$ according to a well-known mathematical formula. Unless otherwise stated, mean values of size distributions as used in the present disclosure refer to the volume-based mean $D[4,3]$. The median value $D50$ of a size distribution is the value dividing

15 the plurality of grains into two equal populations, one consisting of grains having ECD size above the value and the other half having ECD size at most the value. The mode of a size distribution is the value corresponding to the highest frequency of grains, which can be visualised as the peak of the distribution (distributions can include more than one local maximum frequency and be said to be multi-modal).

20 Various other values $d(y)$ can be provided, expressing the size below which a fraction y of the plurality of grains reside in the distribution. For example, $d(0.9)$ refers to the ECD size below which 90 per cent of the grains reside, $d(0.5)$ refers to the ECD size below which 50 per cent of the grains reside and $d(0.1)$ refers to the ECD size below which 10 per cent of the grains reside.

25

A Coulter counter is an apparatus for counting and measuring the size distribution of particles suspended in an electrolyte solution. A Coulter counter may have at least one micro-channel connecting two chambers containing the electrolyte solution. As fluid containing particles is drawn through each micro-channel, each particle causes

30 a brief change to the electrical resistance of the liquid. The counter detects these changes in electrical resistance.

CLAIMS

1. A method of measuring an aggregate strength indication of a test sample comprising a plurality of super-abrasive grains having an aggregate mass;
5 the method including:
measuring the size frequency distribution of the super-abrasive grains;
providing a friability tester apparatus comprising:
a drive mechanism,
a crusher ball, and
10 a capsule having an enclosable cavity for containing the test sample and the crusher ball;
configured such that the capsule can be moved at a cyclically varying speed for a number of cycles by the drive mechanism;
an energy indication value calculated as
15 an integration over a full cycle of the square of the speed of the capsule, in metres per second,
multiplied by half the mass of the crusher ball, in kilograms,
multiplied by the number of cycles,
divided by the aggregate mass of the grit sample, in grams;
20 enclosing the test sample and the crusher ball within the cavity;
subjecting the capsule to the motion, such that the energy indication value is at least about 2 and at most about 50 ("Joules" per gram), to produce a comminuted sample;
measuring the size frequency distribution of the comminuted sample; and
25 calculating the aggregate strength indication based on the size frequency distributions of both the comminuted sample and the test sample.
2. A method as claimed in claim 1, in which the super-abrasive grains comprise diamond material.
30
3. A method as claimed in claim 1 or claim 2, in which the super-abrasive grains consist of at least one diamond crystal.
4. A method as claimed in any of the preceding claims, in which at least 80 per cent
35 of the super-abrasive grain consists of a single diamond crystal.

5. A method as claimed in any of the preceding claims, in which substantially all of the super-abrasive grains consists of a single diamond crystal.
- 5 6. A method as claimed in claim 1, in which the super-abrasive grains comprise cubic boron nitride (cBN) material.
7. A method as claimed in any of the preceding claims, in which the super-abrasive grains comprised in the test sample have a mean size of at least 1 micron.
- 10 8. A method as claimed in any of the preceding claims, in which the super-abrasive grains comprised in the test sample have a mean size of at least 2 microns.
9. A method as claimed in any of the preceding claims, in which the super-abrasive grains comprised in the test sample have a mean size of at most about 80 microns.
- 15 10. A method as claimed in any of the preceding claims, in which the super-abrasive grains comprised in the test sample have a mean size of at most about 30 microns.
- 20 11. A method as claimed in any of the preceding claims, in which the grains comprised in the test sample have a mean size of at most about 20 microns.
- 25 12. A method as claimed in any of the preceding claims, in which the grains comprised in the test sample have a mean size of at most about 10 microns.
13. A method as claimed in any of the preceding claims, in which the aggregate mass is at least 0.2 gram (g) and at most 0.2 gram (g).
- 30 14. A method as claimed in any of the preceding claims, in which the aggregate mass is 0.40 gram (g).

15. A method as claimed in any of the preceding claims, in which the size distribution frequencies of each of the test sample and the comminuted sample are measured by means of a laser diffractometry apparatus.
- 5 16. A method as claimed in any of claims 1 to 14, in which the size distribution frequencies of each of the test sample and the comminuted sample are measured by means of a Coulter counter apparatus.
- 10 17. A method as claimed in any of the preceding claims, in which the crusher ball has a diameter of at least 6 millimetres (mm) and at most 10 millimetres.
18. A method as claimed in any of the preceding claims, in which the mass of the crusher ball is 2.4 grams (g).
- 15 19. A method as claimed in any of the preceding claims, in which the crusher ball comprises steel.
- 20 20. A method as claimed in any of the preceding claims, in which the surface of the crusher ball has a Rockwell C hardness of at least about 50 HRc.
21. A method as claimed in any of the preceding claims, in which the surface of the crusher ball consists of ceramic material.
22. A method as claimed in any of claims 1 to 20, in which the surface of the crusher ball consists super-abrasive material.
- 25 23. A method as claimed in any of the preceding claims, in which the enclosable cavity is substantially cylindrical in shape, having a length of at least 15 millimetres (mm) and at most 30 millimetres (mm).
- 30 24. A method as claimed in any of the preceding claims, in which the enclosable cavity is substantially cylindrical in shape, having a diameter of at least 10 millimetres (mm) and at most 20 millimetres (mm).

25. A method as claimed in any of the preceding claims, in which the motion comprises at least one cycle in which the speed of the capsule varies substantially as a sinusoidal function of time.
- 5 26. A method as claimed in any of claims 1 to 24, in which the motion comprises at least one cycle in which the speed of the capsule varies substantially as a saw-tooth function of time.
- 10 27. A method as claimed in any of the preceding claims, in which the number of cycles is at least about 6,000.
28. A method as claimed in any of the preceding claims, in which the number of cycles is at least about 10,000.
- 15 29. A method as claimed in any of the preceding claims, in which the number of cycles is at most about 20,000.
- 20 30. A method as claimed in any of the preceding claims, in which the motion comprises a plurality of repeated cycles having a frequency being at least 42 cycles per second.
- 25 31. A method as claimed in any of the preceding claims, in which the motion comprises a plurality of repeated cycles having a frequency being at least 44 cycles per second.
- 30 32. A method as claimed in any of the preceding claims, in which the motion comprises a plurality of repeated cycles having a frequency being at most 60 cycles per second.
33. A method as claimed in any of the preceding claims, in which the motion comprises a plurality of repeated cycles having a frequency being at most 50 cycles per second.

34. A method as claimed in any of the preceding claims, in which the motion comprises a plurality of repeated cycles having a frequency being at most 48 cycles per second.
- 5 35. A method as claimed in any of the preceding claims, in which the maximum speed of the capsule during each cycle is at least 0.5 metres per second (m/s).
36. A method as claimed in any of the preceding claims, in which the maximum speed of the capsule during each cycle is at most about 2 metres per second (m/s).
- 10
37. A method as claimed in any of the preceding claims, in which the energy indication value is at least 4.
- 15 38. A method as claimed in any of the preceding claims, in which the energy indication value is at least 8.
39. A method as claimed in any of the preceding claims, in which the energy indication value is at least 10.
- 20 40. A method as claimed in any of the preceding claims, in which the energy indication value is at most 20.

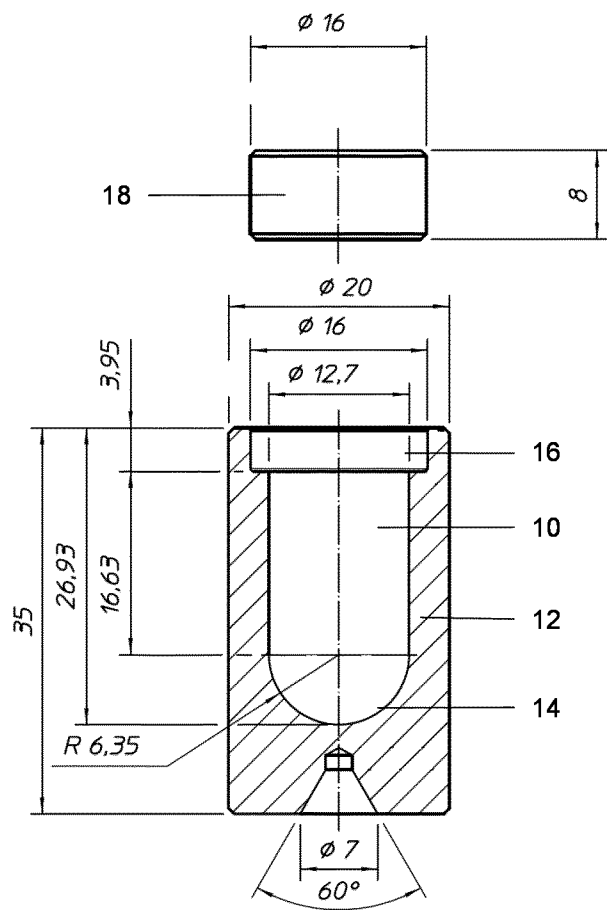


Fig. 1

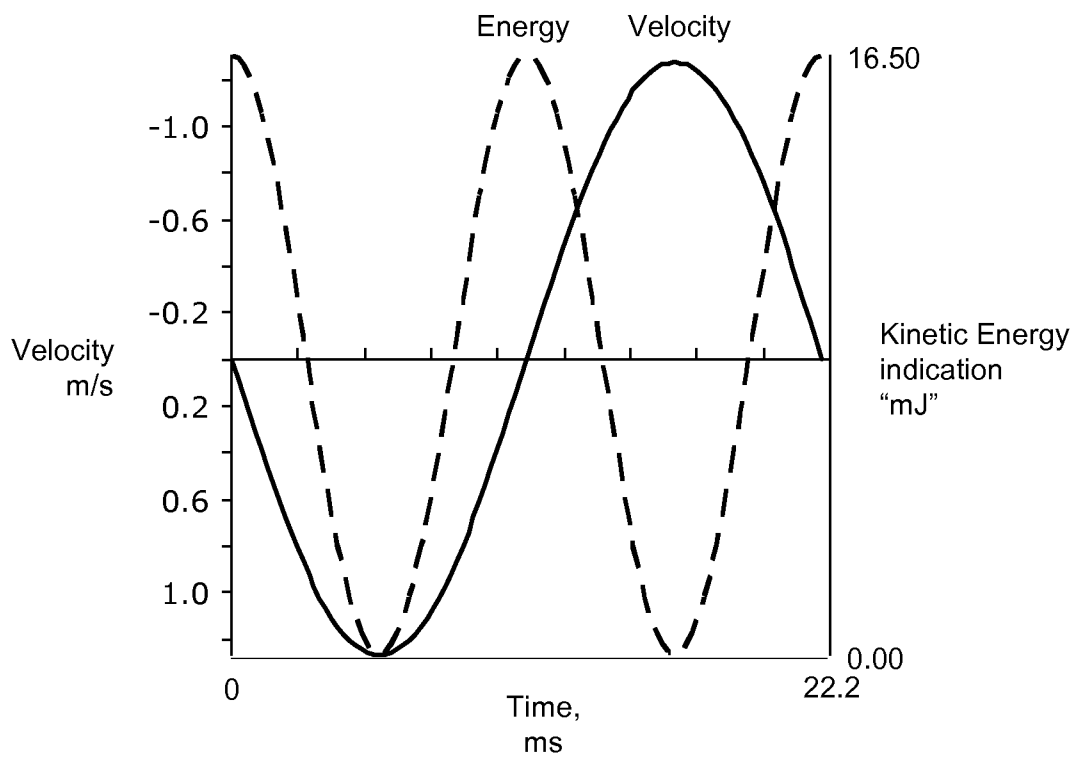


Fig. 2

Normalised kinetic
energy indication,
J/g

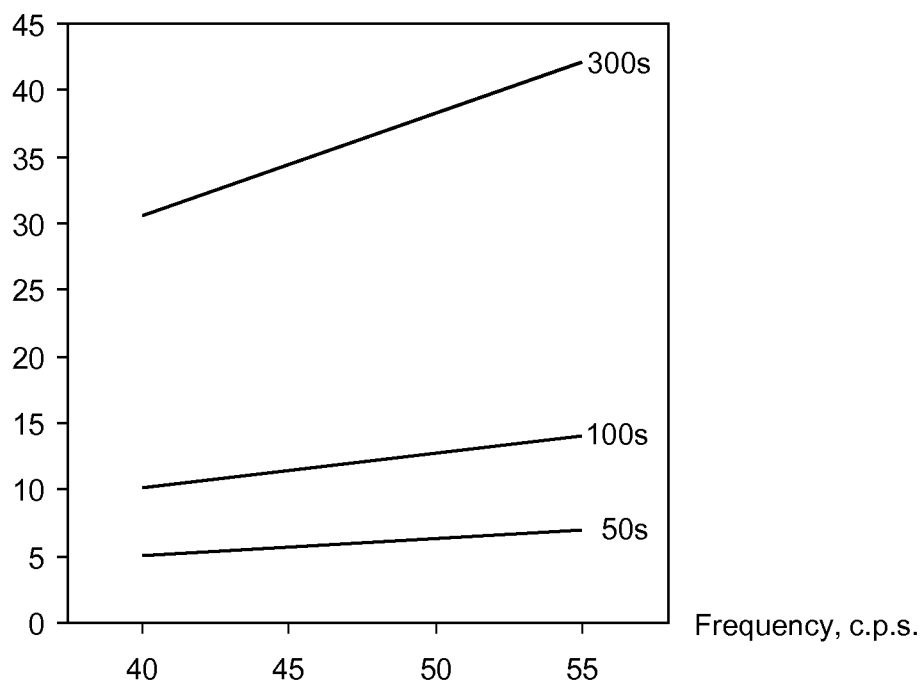


Fig. 3

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2014/058517

A. CLASSIFICATION OF SUBJECT MATTER

INV. B02C17/00 G01N3/56
ADD. G01N15/00 G01N15/02 G01N21/47

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B02C G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	BELLING N G ET AL: "THE FRIATESTER* - 10 YEARS LATER", INDUSTRIAL DIAMOND REVIEW, INDUSTRIAL DIAMOND INFORMATION BUREAU. LONDON, GB, 1 August 1974 (1974-08-01), pages 285-291, XP002045704, page 285, column 2, line 35 - page 286, column 1, line 10 page 286, column 2, lines 8-10 page 287, column 2, lines 30-32 page 287, column 2, lines 25-28 Test Procedure; page 287, column 2 - page 288, column 1; figures 3, 5 page 289, column 1, lines 27-30 page 290, column 1, lines 20-23 Index of impact strength; page 290, column 2, lines 6-52 figure 14 -/--	1-40

☒ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

30 July 2014

Date of mailing of the international search report

05/08/2014

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Bamière, François

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2014/058517

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	----- J. SPINNEWYN, R. LEBOUT AND R. LORENT: "Numerical Simulation of the Diamond Grit Friability Tester", 'DIAMOND TOOLING' PROCEEDINGS OF THE EURO PM 2002 CONFERENCE, LAUSANNE, OCTOBER 7-9 2002, 9 October 2002 (2002-10-09), pages 84-89, XP008170889, ISBN: 1899072179 Retrieved from the Internet: URL: http://www.custodiam.com/pdf/Simul_FRI_ATESTER.pdf the whole document equation (3); page 2 2. The parameters; page 3/6 -----	1-40