

Surface textured diamond particles - properties and applications

Mesh and micron size surface textured (ST) monocrystalline diamond particles with various degree of surface texture (etching) were produced using a proprietary High Temperature Water Vapor (HTWV) etching process (Patent Application No. 12/982,444).

Surface textured diamond particles exhibit less angular particle shape, rougher surface (increased surface area) and increased crushing strength. In addition, while sharing similar surface chemistry, the surface treated diamond particles exhibit lower levels of surface species (cleaner surface) compared to untreated diamond particles. Paper by I. C. Benea, B. R. Rosczyk and L. M. Fitzgerald.

Diamond powders are mainly used in abrasive applications, in which diamond particles are bonded on a variety of tools for drilling, sawing, grinding, cutting and slicing applications, or bonded to form diamond films or incorporated into slurries and compounds for lapping and polishing applications. Diamond powders are also used in high pressure-high temperature (HPHT) sintering of diamond particles into an abrasive body/compact (polycrystalline diamond compact or PCD), which is a non abrasive application.

The production of high performance diamond tools requires that diamond particles be firmly retained in the bonding material in which they are embedded. A variety of bonding materials are used for the manufacturing of diamond tools, including metals and metal alloys, glass, ceramics and synthetic resins. Bonding between diamond particles and bonding material (bonding matrix) can be mechanical or chemical. While mechanical bonding is a result of bonding material enveloping the diamond particles, chemical bonding is a result of diamond particles reacting with the bonding material to form chemical compounds at the diamond-bonding material interface.

Well developed monocrystalline diamond particles, exhibit smooth surfaces available for bonding. It is known that the bonding strength improves if diamond particles exhibit rougher surface which translates into increased surface area available to bonding. Consequently, when bonded on a tool, the surface textured diamond particles are thought to provide increased bond retention compared to regular monocrystalline diamond particles. Lapping and polishing of advanced materials wafers (i.e. sapphire, silicon carbide, gallium nitride, gallium arsenide, etc), widely used in electronics industry require that virtually scratch free, high surface finishes be obtained by using micron and sub-micron size diamond slurries.

Monocrystalline diamond particles are generally characterised by a reduced number of sharp cutting points and edges, thus being prone to random scratching. In contrast to regular monocrystalline diamond particles, the micron size surface etched monocrystalline diamond particles exhibit an increased number of less sharp cutting points and edges. Therefore, compared to regular monocrystalline diamond particles, the surface textured monocrystalline diamond particles are presumed capable

of delivering improved surface finishes and less random scratching in lapping and polishing applications.

High pressure-high temperature (HPHT) sintering of diamond particles into a coherent polycrystalline diamond body/compact (PCD) is achieved either by infiltration of metal (i.e. cobalt from tungsten carbide-cobalt substrate) or by mixing the diamond particles with a metal powder (i.e. cobalt) or combinations of metal powders (i.e. cobalt, tungsten carbide, etc.). Irrespective of HPHT sintering technique, when compacted under high pressure, the surface textured diamond particles are expected to provide better powder packing and less breakage and chipping of corners and edges, due to a less angular particle shape.

Surface chemistry is another key diamond characteristic to a strong bonding with bonding materials, as well as, to good particle dispersion in slurries and compounds. Moreover, surface chemistry is of paramount importance in high pressure-high temperature sintering of diamond particles, which essentially consists in forming of diamond to diamond "bridges" via covalent bonding of sp³ hybridized carbon atoms.

At elevated temperature, diamond is not chemically inert and it can be eroded by oxygen and oxygen compounds, molten metals (i.e. Mn, Ni, Fe, Co, Pt, Ti, Zr, Ta, W), and hydrogen. There are a number of known processes used to etch the surface of diamond particles. Thermo-chemical etching of diamond requires heating the diamond particles in oxygen or KNO₃ [1, 2]. Etching can also be achieved by heating the diamond particles in a non-oxidizing atmosphere or vacuum [3]. Other methods involve heating the diamond particles embedded in metal powder while exposed to hydrogen or hydrogen-containing gas [4], or heating the diamond particles in air in the presence of metal, or metal oxides [5].

The majority of these techniques do not provide a good control over the etching process, and lead to changes in surface chemistry of diamond coupled with contamination of the diamond surface. Particularly, heating of diamond particles in potassium nitrate, or embedded in metal powders while exposed to hydrogen or hydrogen-containing gas, involve chemical reactions that are difficult to control, and require additional processing steps to remove the resulting chemicals/chemical compounds and clean the recovered diamond particles.

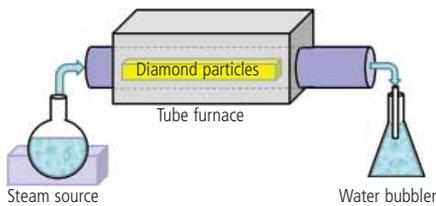


Fig 1 Schematic diagram of the equipment for carrying out the high temperature water vapour (HTWV) etching process according to the present invention

Property	Technique
Particle size distribution	Electrical sensing zone
Particle shape and surface	Scanning electron microscopy (SEM)
Surface chemistry	Time of Flight Secondary Ions Mass Spectroscopy (TOF SIMS)
Surface graphitisation	Raman spectroscopy
Mechanical strength & Fracture characteristics	Crushing strength (US Patent 7,275,446) & Particle size distribution of crushed powder
Bond retention	Shear strength @ Diamond-electrodeposited Ni interface

Table 1 Properties and techniques

Fig 2a Overlay of particle size distributions of 10-20 MB diamond powder before (green) and after HTWV treatment (red)

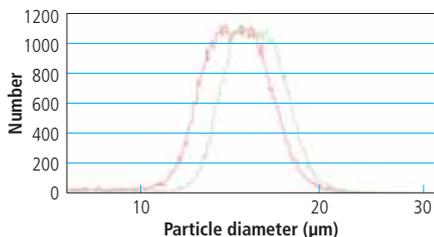
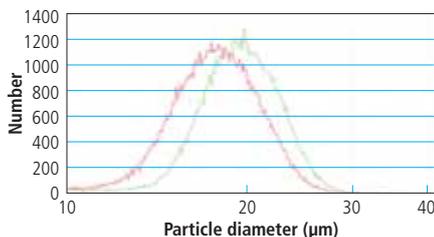


Fig 2b Overlay of particle size distributions of 15-25 MB diamond powder before (green) and after HTWV treatment (red)



	5%	50%	95%	99.9%
15-25 µm MB – untreated	15.07	19.43	24.46	29.10
15-25 µm MB – treated	13.31	17.73	22.88	28.27
10-20 µm MB – untreated	12.79	15.47	18.69	24.98
10-20 µm MB – treated	11.70	14.47	17.72	21.95

Table 2 PSD data for 20 microns and 15 microns metal bond diamond powders subjected to HTWV treatment

Experimental

The high temperature water vapor (HTWV) process for etching the surface of diamond particles (Patent Application No. 12/982,444), consists in essence, of heating the diamond particles to temperatures in excess of 700 °C in the presence of water vapour. The experimental set up is presented in Fig 1.

The HTWV etching process is very flexible and can be conducted in a desired manner to obtain diamond particles exhibiting slightly textured to heavily textured surface.

Resin bond (RB) and metal bond (MB) micron size monocrystalline diamond powders, as well as metal bond grinding grade (G) and metal bond saw grade (S) mesh size monocrystalline diamond powders ranging in size from 4-8 µm to 600-770 µm (25/30 mesh), were treated at high temperatures ranging from 700 °C to 1,100 °C, for a dwell time ranging from 1 minute to 240 minutes, in the presence of water vapour

Following the high temperature water vapour treatment, diamond particles were recovered and analysed for changes in diamond weight, particle size (particle size distribution), particle shape and surface roughness (topography), surface graphitisation (graphite to diamond ratio), surface chemistry (ionic species), and mechanical strength (crushing strength) and fracture characteristics, using the techniques summarized in Table 1, left.

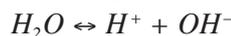
A large number of experiments have been conducted in an attempt to understand the mechanism of high temperature water vapour etching of diamond particles and to establish the effect of each process parameter on etching of the diamond particles. As a result, correlations between the etching process parameters (temperature, dwell time, water vapour flow), and the outcome of the etching process with respect to changes in characteristics of diamond particles (diamond weight, particle size, particle shape, surface roughness and mechanical strength) have been established.

Results and Discussion

Diamond etching mechanism

While a rigorous explanation of the kinetics of the process of water vapour etching of diamond cannot yet be given, it is suggested that this process is a result of the water molecules adsorption on the diamond surface, whereas most of them undergo dissociation when colliding with the diamond surface forming chemical bonds: C-H; C=O and C-O-H

Dissociation of water molecules when colliding with one another (self ionisation) and with diamond surface, takes place according to chemical reaction below:



However, hydrogen ions have negligible existence as protons and are produced already hydrated, as hydroxonium ions (H₃O⁺). Therefore, this equation may be better written as:



Next, hydroxonium ions (strong acid) and hydroxide ions (strong base) interact with diamond surface and form chemical bonds: C-H; C=O and C-O-H

It needs to be noted that etching of diamond by water vapour is much slower and requires a higher temperature compared to etching by molecular oxygen.

Particle size (particle size distribution)

In one set of experiments metal bond (MB) type monocrystalline diamond powders with average particle size of 15 and 20 microns respectively, were subjected to HTWV treatment at 1050 °C for 120 minutes. Using a Beckman Coulter Analyser – Multisizer 3, the particle size distribution (PSD) was measured before and after treatment. The overlay of particle size distributions before and after treatment is depicted in Figs 2a and 2b, while the PSD data is presented in Table 2.

As expected, following the HTWV etching process, the size of diamond particles is reduced (particle size distribution of the powder is shifted finer). Evidently, the PSD shift is directly related to the degree of surface etching (texturing). In addition, smaller diamond particles, which possess higher surface area, are likely to be affected more by the HTWV treatment, with very small particles being most likely completely consumed during the process. To substantiate this effect the particle size distribution of 0.125 microns metal bond diamond powder was measured before and after treatment at 925 °C for 120 minutes, using a centrifugal sedimentation analyser (CPS), and the PSD overlay is presented in Fig 3.

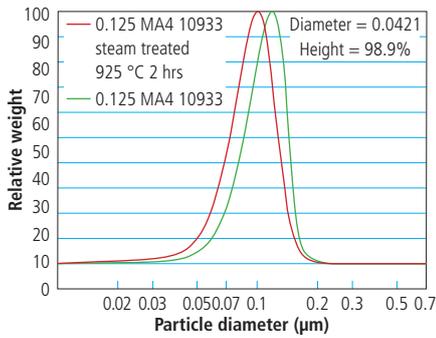


Fig 3 PSD overlay of 0.125 micron diamond powder before (green) and after HTWV treatment (red)

Particle shape and surface

In another set of experiments, MB diamond particles having an average size of 20 microns and 35 microns respectively were treated at 1050 °C for 120 minutes. SEM micrographs of unetched diamond particles are presented in Figs 4a and 4b, while SEM

micrographs of etched diamond particles are presented in Figs 4c through 4f.

Similarly, metal bond grinding grade diamond particles with an average size of approx. 100 microns (140/170 mesh), as well as saw grade diamond particles with an average size of approx. 350 microns (40/50) and approx. 685 microns (25/30 mesh), were treated at 1050 °C for 120 min. SEM micrographs of unetched and etched 140/170 mesh grinding grade diamond particles are displayed in Figs 5a, and 5b respectively. SEM micrographs of 40/50 mesh saw grade diamond particles are exhibited in Figs 6a through 6c (unetched particles) and 6d through 6g (etched particles).

SEM micrographs of unetched 25/30 mesh saw grade diamond particles are exhibited in Figs 7a through 7c, while SEM micrographs of etched 25/30 mesh saw grade diamond particles are exhibited in Figs 7d through 7f.

Fig 4a SEM micrograph of unetched 15-25 MB diamond particles (1.0kX magnification)

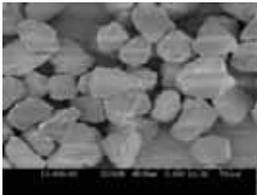


Fig 4b SEM micrograph of unetched 15-25 MB diamond particles (2.0kX magnification)

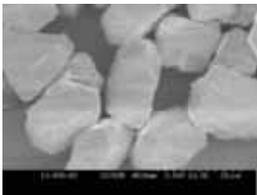


Fig 4c SEM micrograph of surface etched 15-25 MB diamond particles (1.0kX magnification)

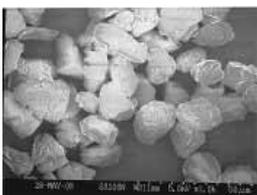


Fig 4d SEM micrograph of surface etched 15-25 MB diamond particles (3.5kX magnification)

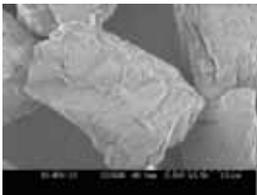


Fig 4e SEM micrograph of surface etched 15-25 MB diamond particles (4.0kX magnification)

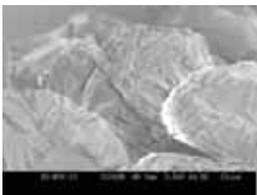


Fig 4f SEM micrograph of surface etched 15-25 MB diamond particles (4.5kX magnification)

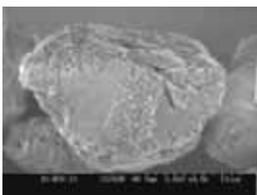


Fig 6a SEM micrograph of unetched 40/50 mesh saw grade diamond particles (150X magnification)

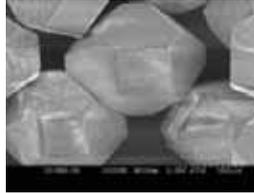


Fig 6b SEM micrograph of unetched {100} face of a 40/50 mesh saw grade diamond particle (400X magnification)

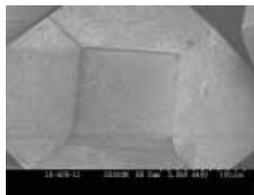


Fig 6c SEM micrograph of unetched {111} face of a 40/50 mesh saw grade diamond particle (400X magnification)

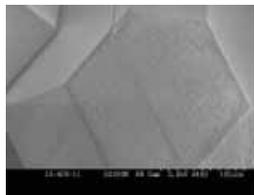


Fig 6d SEM micrograph of surface etched 40/50 mesh saw grade diamond particles (200X magnification)

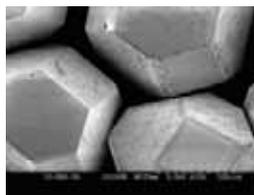


Fig 6e SEM micrograph of surface etched 40/50 mesh saw grade diamond particles (250X magnification)



Fig 6f SEM micrograph of heavily etched 40/50 mesh saw grade diamond particles (250X magnification)

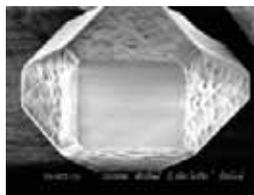


Fig 6g SEM micrograph of heavily etched 40/50 mesh saw grade diamond particles (250X magnification)

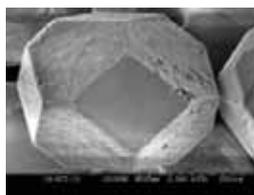


Fig 7a SEM micrograph of unetched 25/30 mesh saw grade diamond particles (20X magnification)

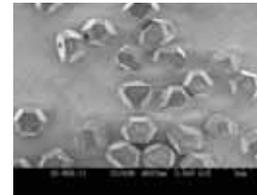


Fig 7b SEM micrograph of unetched 25/30 mesh saw grade diamond particles (50X magnification)

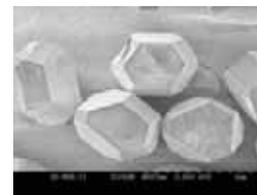


Fig 7c SEM micrograph of unetched 25/30 mesh saw grade diamond particles (100X magnification)

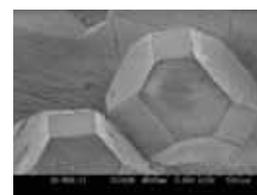


Fig 7d SEM micrograph of surface etched 25/30 mesh saw grade diamond particles (50X magnification)

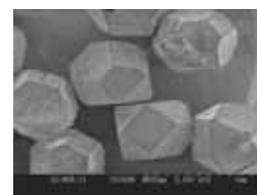


Fig 7e SEM micrograph of surface etched 25/30 mesh saw grade diamond particles (50X magnification)

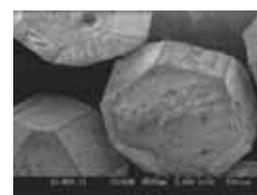


Fig 7f SEM micrograph of surface etched 25/30 mesh saw grade diamond particle (150X magnification)

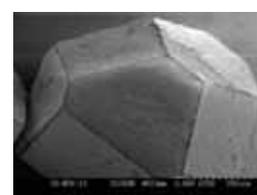


Fig 5a SEM micrograph of unetched 140/170 mesh grinding grade diamond particles (1.0kX magnification)

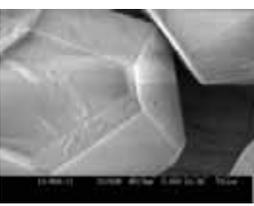
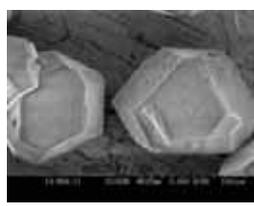


Fig 5b SEM micrograph of surface etched 140/170 mesh grinding grade diamond particles (500X magnification)



To reveal the etching pattern of diamond surface, SEM analysis was performed on etched {111} and {100} faces of 40/50 mesh saw grade diamond particles.

SEM analysis, reveals that the etching pattern on {111} faces consists of triangular pits (trigons), as depicted in Figs 8a and b, while the etching pattern of {100} faces consists of square and hexagonal pits, as depicted in Figs 9a and b. While triangular pits on {100} faces are deeper and can be detected at low magnification (i.e. 400X), the square and hexagonal pits on {100} faces are much shallower and can only be detected at high magnification (i.e. 12,000X). This represents clear evidence that the etching rate of diamond surface is much faster for {111} faces compared to {100} faces.

Surface chemistry

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) uses a pulsed primary ion beam to desorb and ionize species from a sample surface. The resulting secondary ions are accelerated into a mass spectrometer, where they are mass analysed by measuring their time-of-flight from the sample surface to the detector. Time of Flight – Secondary Ion Mass Spectrometry was used to analyse untreated and treated (surface etched) MB diamond particles having 17 microns average size, as well as, saw grade (S) diamond particles with average size of approx. 350 microns (40/50 mesh).

TOF-SIMS spectra of untreated and surface treated diamond particles with an average size of 17 microns and 350 microns are presented in Figs 10a and 10b, respectively. Both spectra have been normalised with respect to Carbon peak which should not change due to treatment.

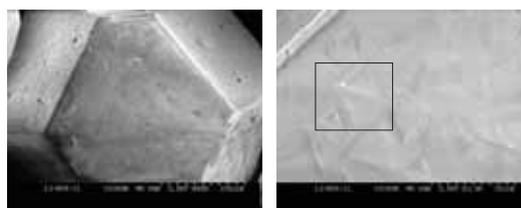
As depicted in Fig 10a, the surface of diamond particles before treatment shows significant peaks at 1, 16 and 17 atomic mass units, which are attributed to H, O or CH₄, and OH. After treatment, the peaks at 16 and 17 atomic mass units are much smaller compared to carbon peak. This is likely due to removal of O or CH₄ and OH species from the surface. On the other hand, after treatment, the peak at 1 atomic mass unit is higher which seems to indicate that the H atoms terminate the surface carbon atoms.

The TOF SIMS spectrum of 40/50 mesh saw grade diamond particles presented in Fig 10b, shows that the surface of diamond particles before treatment shows significant peaks at 1 and 13 atomic mass units, which are attributed to H and CH. After treatment, these peaks are much smaller compared to carbon peak. This is likely due to removal of H and CH species from the surface. After treatment, the peak at 16 atomic mass unit, which corresponds to O or CH₄, is slightly higher. This seems to indicate a slightly elevated level of O or CH₄.

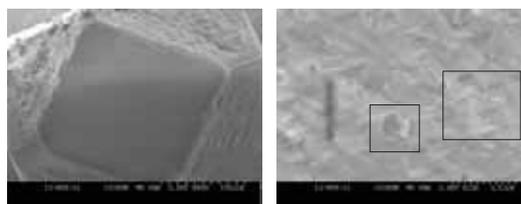
The TOF SIMS analysis results indicate that, in addition to surface etching, the HTWV etching process also removes the ionic contaminants from the surface of diamond particles.

Surface graphitisation

Raman spectroscopy is very sensitive to different forms of carbon. The Raman spectrum of diamond exhibits a sharp first order peak at ~1333 cm⁻¹, while a typical Raman spectrum of amorphous carbon consists of two broad bands; a D band (D for disordered) at approximately 1350 cm⁻¹, and a G band (G for graphite) at around 1580 cm⁻¹. Both graphite and amorphous carbon have Raman scattering efficiencies greater than that of diamond. This means that for identical numbers of sampled atoms in the three carbon forms - diamond, graphite, and amorphous carbon, the graphite and amorphous carbon samples would give a much larger Raman

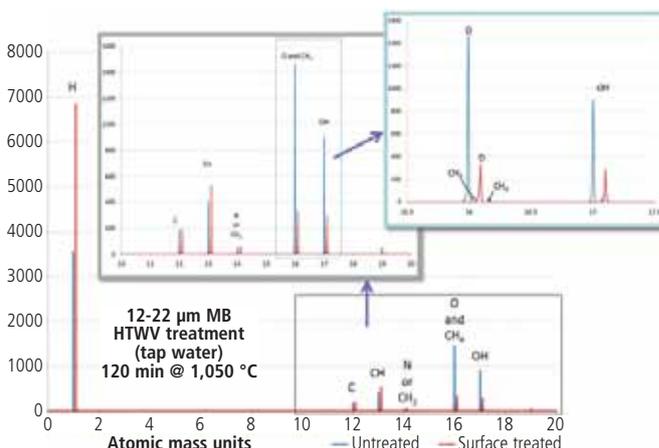


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Figs 8a and b SEM micrograph of a 25/30 mesh saw grade diamond particle reveals the etching pattern on {111} face consisting of triangular pits or trigons (400X and 2.0kX magnification)

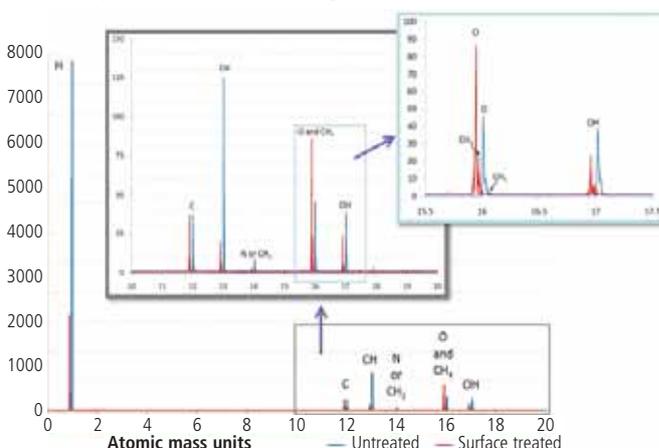


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Figs 9a and b SEM micrograph of a 25/30 mesh saw grade diamond particle reveals the etching pattern on {100} face consisting of shallow square pits (400X and 2.0kX magnification)

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Fig 10a TOF-SIMS spectra of untreated and surface treated diamond particles with an average size of 17 microns



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Fig 10b TOF-SIMS spectra of untreated and surface treated diamond particles with an average size of 350 microns



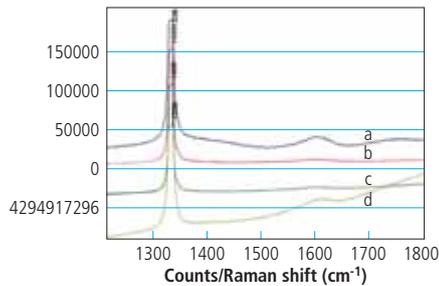
signal than the diamond sample. Intensity ratios can, however, be used as a measure of relative concentration.

MB diamond particles 17 microns average size were treated at 1,050 °C for 240 minutes, in two different ways: in Argon and in water vapour. Following treatment, four diamond particles from

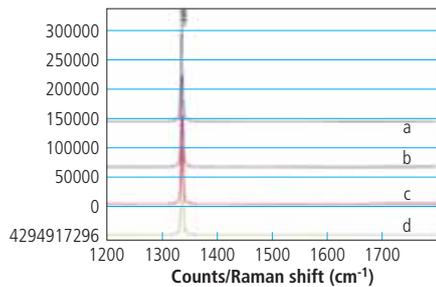
each treatment were analysed by Raman spectroscopy to detect the presence of graphite and the amount of graphitisation, if present, as expressed by graphite to diamond ratio.

As shown in Fig 11a, the Raman spectrum of the diamond sample treated at high temperature in the presence of water vapour shows only the diamond peak

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Fig 11a Raman spectrum of a 12-22 MB diamond sample treated in Argon at 1050 °C for 120 minutes



♦♦♦
Fig 11b Raman spectrum of a 12-22 MB diamond sample subjected to HTWV treatment at 1050 °C for 120 minutes



at 1333 cm⁻¹; no peaks due to graphitic or amorphous carbon were detected at 1595 cm⁻¹.

By contrast, as depicted in Fig 11b, the Raman spectrum of the diamond sample treated at high temperature in Argon a weak band due to graphitic or amorphous carbon was detected at 1595 cm⁻¹, and a diamond to graphite ratio of 244 was calculated. Furthermore, the graphite to diamond ratio was calculated as 1/244 = 0.04 or 0.4%.

Based on Raman spectroscopy analysis, it is concluded that following the HTWV etching, the surface of diamond particles does not graphitise, as opposed to similar treatment in argon.

The mechanism of HTWV etching process is not yet understood. However, it is hypothesised that hydrogen atoms are preferentially etching the sp² graphitic bonds and can terminate the surface carbon atoms to maintain sp³ hybridization and prevent graphite formation.

Mechanical strength and fracture characteristics

It is known that, mainly due to metallic impurities which are trapped inside during HPHT crystal growth process, the mechanical strength of diamond particles decreases when exposed to high temperature in excess of approx. 900 °C. However, it is also known that when exposed to high

temperature, the mechanical strength of diamond particles increases, due to annealing of internal defects (redistribution of metallic impurities and mechanical stresses). Therefore, by choosing the appropriate temperature range for the etching process, in relationship with the size of the diamond particle and the level of residual crystal growth defects (RCGD), it is possible to achieve etching of diamond particles without the decrease of mechanical strength.

To compare the mechanical strength of untreated and treated (surface etched) diamond particles, the crushing strength index (CSI) of diamond powder, was measured using the technique and apparatus described in (6). A close up of the apparatus is depicted in Fig 12.

During the crushing test, diamond particles are subjected simultaneously to compression and shear as illustrated in the drawing embedded in Fig 12.

The CSI is calculated from the particle size distribution (PSD), by taking into account the number of “on-size” particles (particles with diameter ranging between 50% and 95% of the PSD), in the initial (uncrushed) and resulting (crushed) diamond powders, as illustrated in Fig 13: $CSI = ROS/IOS \times 100$ where:

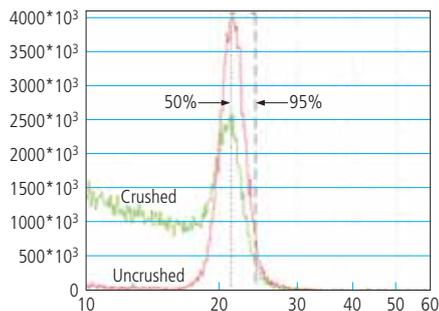
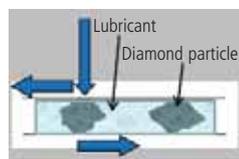
IOS is the initial number of on-size particles (uncrushed powder)

ROS is the resulting number of on-size particles (crushed powder)

The PSD of uncrushed and crushed diamond powders was measured using a Beckman Coulter Multisizer 3 analyser. Knowing that for any given size, CSI is sensitive to the PSD width (i.e. wide vs. tight), and that after HTWV treatment the size of the diamond particles is reduced (PSD is shifted finer), the untreated diamond was graded to match the PSD of the treated diamond.

The crushing strength measurements were performed on three MB micron diamond powders having the average particle size of 17 microns, 20 microns and 35 microns. Depending on size, the diamond mass was adjusted to have approx. same surface area (as calculated from PSD), under the piston. A known amount of diamond and lubricant mixture is placed on the cup surface (a PCD blank). Next, the compression force is applied simultaneously with the shear force, which is generated by rotating the piston (PCD blank) and the cup (PCD blank) in opposite directions at a pre set rpm for a pre set time. Crushing strength test parameters are presented in Table 3.

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Fig 12 Apparatus for measuring the crushing strength of micron diamond

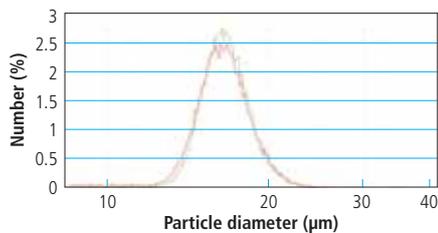


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Fig 13 Particle size distribution of uncrushed and crushed diamond powder

Diamond			Load (lb)	RPM (piston & cup)	Time (sec)
Type	Size (µm)	Mass (mg)			
MB	12-22	25	20	10	10
MB	15-25	30	20	10	10
MB	30-40	52	20	10	10

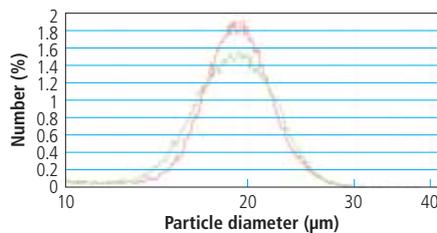
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Table 3 Crushing strength test parameters

Diamond technology



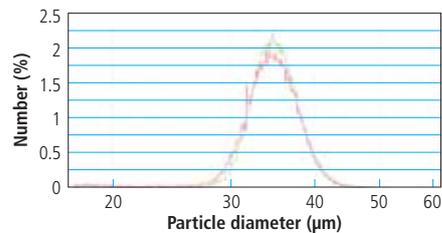
Sample	Diameter on % (µm)			
	5%	50%	95%	99.9%
Treated 12-22 MB	13.57	16.41	19.9	25.35
Untreated 12-22 MB	14.04	16.54	19.78	24.33

Fig 14 PSD match for 12-22 MB diamond untreated (green) and treated (red)



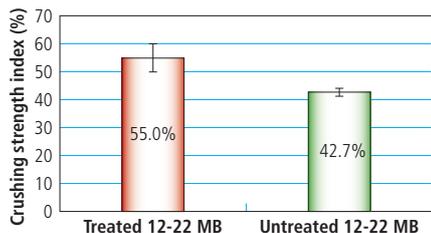
Sample	Diameter on % (µm)			
	5%	50%	95%	99.9%
Treated 15-25 µm MB	14.14	18.92	23.97	29.72
Untreated 15-25 µm MB	14.83	19.08	23.81	30.64

Fig 15 PSD match for 15-25 MB diamond untreated (green) and treated (red)



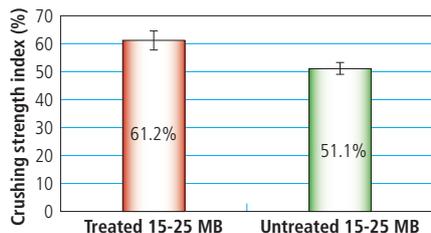
Sample	Diameter on % (µm)			
	5%	50%	95%	99.9%
Treated 30-40 µm MB	29.62	34.52	39.67	45.23
Untreated 30-40 µm MB	30.64	34.76	39.67	45.41

Fig 16 PSD match for 30-40 MB diamond untreated (green) and treated (red)



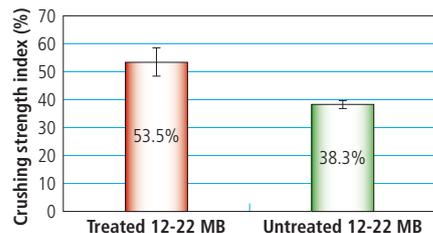
12-22 MB	CSI (%)			CSI (%)	
	Trial 1	Trial 2	Trial 3	Avg	Std Dev
Treated	52.46	60.80	51.86	55.0	5.0
Untreated	43.56	41.26	43.33	42.7	1.3

Fig 17 CSI of 12-22 MB diamond untreated (green) and treated (red)



15-25 MB	CSI (%)			CSI (%)	
	Trial 1	Trial 2	Trial 3	Avg	Std Dev
Treated	64.16	57.65	61.75	61.2	3.3
Untreated	52.19	48.83	52.42	51.1	2.0

Fig 18 CSI of 15-25 MB diamond untreated (green) and treated (red)



30-40 MB	CSI (%)			CSI (%)	
	Trial 1	Trial 2	Trial 3	Avg	Std Dev
Treated	49.31	50.48	60.65	53.5	6.2
Untreated	41.44	39.79	33.76	38.3	4.0

Fig 19 CSI of 30-40 MB diamond untreated (green) and treated (red)

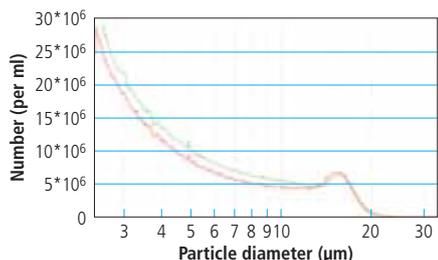


Fig 20 Fracture characteristics of 12-22 MB diamond untreated (green) and treated (red)

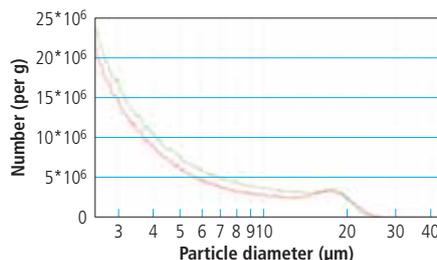


Fig 21 Fracture characteristics of 15-25 MB diamond untreated (green) and treated (red)

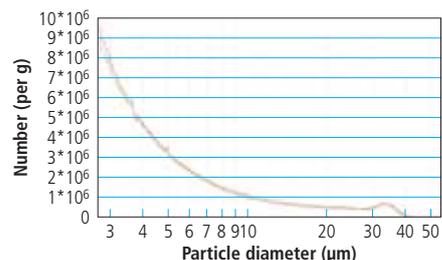


Fig 22 Fracture characteristics of 30-40 MB diamond untreated (green) and treated (red)

Each measurement was repeated three times and average CSI value and standard deviation were calculated.

Figs 14, 15 and 16 show the PSD match for each diamond size measured. CSI data and charts of the powders under investigation are presented in Figs 17, 18 and 19.

Particle size distributions of the crushed powders, regarded as a qualitative measure of the fracture characteristics, are displayed in Figs 20, 21 and 22.

Crushing test results clearly demonstrate that, if carefully controlled, the HTWV treatment does not lead to decay of mechanical strength (crushing strength) of diamond particles. On the contrary, following the HTWV treatment the mechanical strength of diamond particles increases slightly. The increase in mechanical strength of diamond particles subjected to HTWV treatment is explained mainly based on the reduced incidence of breakage and

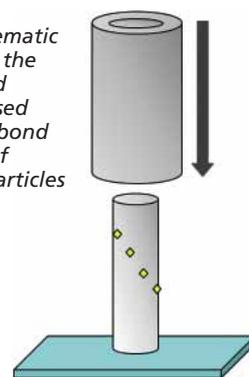
chipping of corners and edges coupled with the annealing of internal defects (residual crystal growth defects).

The analysis of the crushed diamond powder reveals that, when subjected to compaction and shear, surface textured diamond particles generate less fine particles compared to untreated diamond particles. It is therefore obvious that surface textured diamond particles exhibit less breakage and chipping of corners and edges when subjected simultaneously to compaction and shear.

Bond retention

The bond retention of surfaces textured diamond particles compared to untreated diamond particles was assessed using an Instron test machine Model 4204, equipped with a cylinder-rod assembly, designed to test the shear strength at the interface

Fig 23 Schematic drawing of the cylinder-rod assembly used to test the bond retention of diamond particles



diamond particle-electrodeposited Ni bond. A schematic drawing of the cylinder-rod assembly used to test the bond retention of diamond particles is shown in Fig 23. The actual diamond electroplated rods and respective cylinders are displayed in Fig 24, while Fig 25 represents a detailed drawing of the diamond electroplated rod used in these experiments.



Fig 24 Actual diamond electroplated rods and respective cylinders

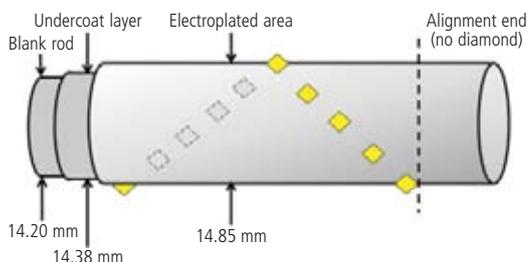


Fig 25 Detailed drawing of the diamond electroplated rod

Details regarding bond retention test are presented below:

- ◆ Diamond type: saw grade
- ◆ Diamond size: 25/30 mesh (average size = approx. 685 μm)
- ◆ Static compression load rate: 0.125 inch/min
- ◆ Electroplated rod's dimensions:
 - ◆ rod (blank): 14.20 mm
 - ◆ rod with electroplated Ni undercoat layer: 14.38 mm
 - ◆ rod with final electroplated Ni layer: 14.85 mm
- ◆ Ni bond thickness: 235 μm ($14.85 - 14.38 = 0.47/2 = 0.235$ mm)
- ◆ Coverage of diamond particles: 31%-39% (95% of particles in size range 600-770 μm ; avg. size = 685 μm)

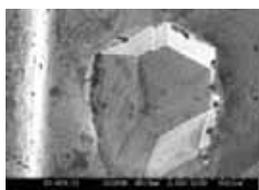


Fig 26a SEM micrograph of a pull-out (100X magnification)

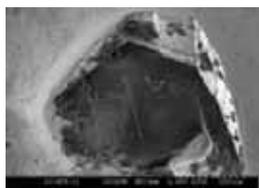


Fig 26b SEM micrograph of a sheared diamond particle (150X magnification)

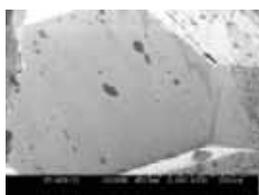


Fig 27a SEM micrograph of the cavity from which a surface treated diamond particle was pulled out (250X magnification)

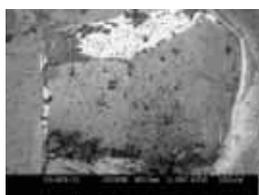


Fig 27b SEM micrograph of the cavity from which an untreated diamond particle was pulled out (150X magnification)

As described in Fig 24, under an applied static compression load, the cylinder moves down over the electroplated rod and dislodged diamond particles (one by one) and debris fall down freely. The bond retention test was performed using a total of six cylinder-rod assemblies: three electroplated rods incorporated untreated diamond particles and three electroplated rods incorporated surface treated diamond particles.

Following the shear test, electroplated rods were inspected to identify the failure mode at each diamond particle-electrodeposited Ni bond interface, using optical microscopy and SEM.

The mode of failure was assessed using the following criteria:

- ◆ **Pull-out:** bonding between diamond particle and Ni bond failed under the shear force and the particle was pulled-out (dislodge) from the bond
- ◆ **Sheared:** diamond particle failed (fractured) before the shear force was able to dislodge it from the Ni bond (stronger bonding)
- ◆ **Mixed:** some portion of the diamond particle remained in the cavity, but the majority of the particle was lost through shear

SEM micrographs of a pull-out and of a sheared diamond particle are presented in Figs 26a, and 26b, respectively. Examination of the SEM micrographs taken from cavities from which a surface treated diamond particle was pulled out, Fig 27a, and an untreated diamond particle was pulled out, Fig 27b, reveals that while the pulled out untreated diamond particle leaves a cavity with smooth and clean surface, the surface treated diamond particle leaves a cavity with a rough surface (as it mirrors the surface roughness of the diamond particle) and debris is also present.

Based on the mode of failure criteria defined above, the bond retention exhibited by surface textured diamond particles versus untreated diamond particles is presented in Fig 28. It needs to be noted that different colour shades were used for each rod.

The results of the bond retention test demonstrate clearly that under the same shear force, the surface textured diamond particles are more likely to shear rather than to be pulled out from the Ni bond, as opposed to untreated diamond particles which experience significantly increased number of pull outs. This indicates increased bond retention of the surface textured diamond particles compared to untreated diamond particles.

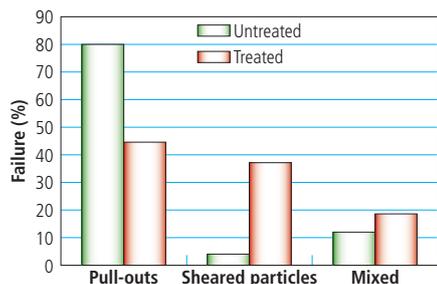


Fig 28 Failure mode of surface textured diamond particles versus untreated diamond particles

DIAMOND/ CBN WHEEL TRUEING & DRESSING MACHINE



MODEL FC-250EX

FC-250EX is equipped with **ExVision**, a computer-driven vision system with a software program specifically designed for the FC-250EX and its various applications. **ExVision** is user-friendly, and contains easy-to-use menus for selecting standard as well as user-customized wheel shapes.

Included are menus for drawing lines, arcs and basic, as well as complex forms. The system is easy for those who are not familiar with CAD functions.

FC-250EX is equipped with servo motor for auto-swivelling and auto-infeed; also the function of auto-oscillation can be entered. Ease and efficiency in trueing and dressing flats, angles, and radii on diamond and CBN single wheel, or multiple wheel packs. Simple to operate, highly accurate and cost effective.

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Tel: 886-4-25665231, 25665237, 25684491

Fax: 886-4-25662049

E-mail: farman55@ms41.hinet.net

Website: www.farman.com.tw

Conclusions

Main characteristics of surface textured diamond particles

Shape and surface:

- ◆ Less angular particle shapes
- ◆ Textured particle surface (etching pits); increased surface roughness; increased surface area

Surface chemistry:

- ◆ Unchanged surface chemistry: H; OH; O; CH & CH₄. Lower levels of some surface species (process particularities dependent)
- ◆ No surface graphitisation. Hydrogen atoms preferentially etch away graphite and can terminate the surface C atoms to maintain sp³ hybridization and prevent graphite formation

Crushing strength and fracture characteristics:

- ◆ Increased crushing strength primarily due to less breakage and chipping of corners and edges and to annealing effect of residual crystal growth defects.

Bond retention:

- ◆ Increased bond retention due to increased surface area available for bonding.

Targeted applications for surface textured diamond particles

Surface textured diamond particles are regarded as likely candidate for improved performance when bonded in sintered and electroplated tools, when incorporated in slurries and compounds and when used as feedstock for high pressure – high temperature (HPHT) sintering of polycrystalline diamond (PCD) products (bits, blanks, dies, etc.).

When incorporated in bonded tools via mechanical and/or chemical bonding, surface etched diamond particles are expected to provide increased bond retention, compared to regular monocrystalline diamond particles whose surface is not altered. When incorporated in slurries and/or compounds for lapping and polishing applications, surface textured diamond particles, are presumed to provide higher surface finishes and less random scratching. Furthermore, surface etched diamond particles, are considered likely to provide better powder packing and less breakage and chipping of corners and edges when compacted under high pressure during the HPHT sintering process of PCD products. ◆

◆ Authors

Dr. **Ion C. Benea**, **Benjamin R. Rosczyk** and **Laura M. Fitzgerald**
all work for the Engis Corporation, 105 W. Hintz Road,
Wheeling, IL 60090, USA. www.engis.com

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