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Influence on Diamonds During the Spraying of Diamond-Bronze Abrasive Coatings

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Detonation spraying provides the opportunity to produce diamond grinding tools for the machining of stone, cement, and concrete. Especially the atmospheric conditions of the spraying process yield in a high production flexibility. However, during detonation spraying, the oxygenic atmosphere as well as the thermal and kinetic energy have an impact on the processed diamond. Despite its importance for the tools' performance, the influence of the spraying process on the superabrasive diamond is predominantly unknown. The potential decrease of the diamond durability and strength due to degradation effects during the production of sprayed diamond-CuSn 85/15 composites has not yet been determined. X-ray diffraction and Raman spectroscopy were used to verify thermally initiated surface reactions of the sprayed diamonds after exposure to the spraying process. Additionally, reference measurements on the degradation of diamonds in oxidizing and inert conditions were carried out to compare the spraying results. Differential thermal and thermogravimetric analyses were employed. To validate the mechanical properties of the diamond superabrasives, friability tests and fracture force tests were performed. It was found that under optimized detonation spraying conditions the thermal and mechanical impact remains low enough to ensure a good reliability of the processed diamonds. The diamond crystal structure endured the spraying process without detectable graphitization or oxidation. Deterioration indicators were not observed in SEM micrographs, x-ray diffraction patterns or Raman spectra. Furthermore, a high durability and strength of the sprayed diamonds were confirmed by mechanical testing.

Keywords diamond degradation, durability, process detonation sprayed, Raman scattering, thermal stress

1. Introduction

The strength of the diamond is one of the most important factors influencing the performance and durability of superabrasive diamond composites for grinding (Ref 1, 2). Diamond deterioration through oxidization, graphitization or chemical reactions with the matrix material reduces its strength (Ref 1). The diamond deterioration is mainly caused by high process temperatures, e.g. the oxidation of diamond in atmospheric conditions to CO and CO_2. However, the reaction does not only depend on the temperature, but also on time, oxygen concentration, reaction catalysts and the surface constitution of the diamond (Ref 1, 3-7).

Bullen et al. investigated diamond degradation during the sintering of compression molded diamond/steel-bronze segments in air. SDA 100 diamonds (De Beers, Düsseldorf, Germany) with a size of 40-50 mesh (300-420 μm) were analyzed under various sintering temperatures. A diamond surface degradation below 700 °C was identified. However, up to 1000 °C, the diamond deterioration was determined to be low. With steel/bronze as metal matrix the weight loss of the diamonds was 7% after a sintering period of 30 min. The decrease in strength (determined in a friability test) was approximately 30%. The influence of the Fe-based matrix was not quantified (Ref 1).

For synthetic monocrystalline diamonds (type 1b, <1% Ni), Beffort et al. determined the mass loss rate and the influence of the particle size on the degradation of diamonds in air at temperatures between 550 and 750 °C (Ref 3). Temperature in the range 500-550 °C was detected as the critical temperature at which the thermal degradation begins. For a diamond size of 10-16 μm, a mass loss rate of 0.0136 mg/min in isothermal conditions at 550 °C was confirmed. Even at 600 °C the mass loss rate of 0.0750 mg/min was negligibly small. At 650 °C, the degradation rate was proved to be significantly higher with 0.2915 mg/min rising to 0.3360 mg/min at 700 °C and

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0.3600 mg/min at 750 °C. Additionally, it was confirmed that the particle size or more precisely the specific surface area of the diamonds influences the degradation. With smaller diamonds with sizes less than 0.25 μm, a higher mass loss rate of 0.4050 mg/min was determined at 750 °C. However, diamonds with a coarser size show a slower deterioration rate. At 750 °C, the mass loss was 0.3530 mg/s for 40-60 μm particles. It was observed that the critical starting temperature for a measurable degradation in air changes to 600-650 °C (Ref 3).

For large SDB 1055 diamonds, as they are used in this research work, no detailed information is given about the durability at elevated temperatures in oxidizing conditions. Only for inert atmospheres the distributor guarantees a thermal resistance of min 1100 °C with a small reduction in the diamonds strength (Ref 2).

In contrast to processes at inert atmospheres or vacuum conditions, the manufacturing of grinding tools in air atmosphere leads to higher flexibility and economical benefits. However, thermal deterioration must be reduced to a minimum, because reduced cutting sharpness and endurance of the diamond superabrasives would be the consequence. In previous research, detonation spraying has demonstrated its suitability for producing diamond-copper and diamond-bronze composites (Ref 8, 9). It was shown that it is possible to spray diamond composite layers with a low porosity and oxidation. During the spraying the diamond superabrasives were injected into the matrix. Due to the high kinetic energy of the detonation they were well embedded. SEM micrographs of coating cross-sections showed no visible damage or cracking of the diamonds, although the detonation process entails high kinetic energy and temperature (Fig. 1).

However, the ground and polished cross-section hides the information about possible surface deterioration of the embedded diamonds. Surface graphitization and erosion initiated by oxidation are not detectable, because indicators of these effects could be removed during the polishing. Despite its importance on the grinding performance and the potential of the alternative manufacturing process, the real amount, type, and reasons for deterioration effects are predominantly unknown for the detonation spraying.

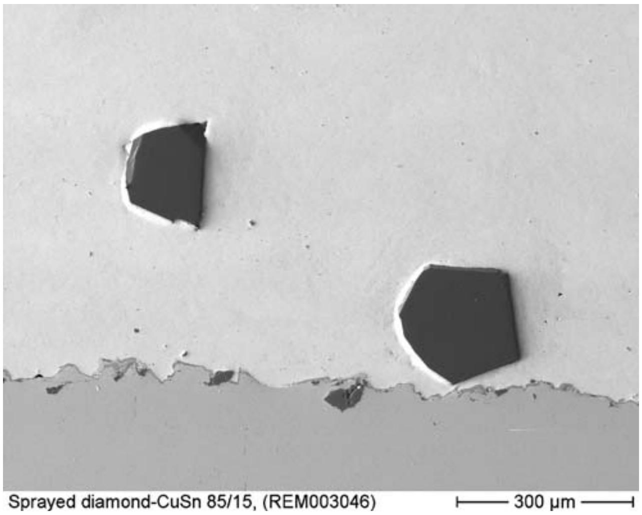

Fig. 1 SEM micrograph of detonation sprayed diamond-CuSn 85/15 layer (Ref 8)

2. Experimental

2.1 Materials

SDB 1055 diamonds of a grain size 60-70 mesh (210-250 μm) (Element Six, Hanau, Germany) were used for the spraying and analyzing experiments. The cubo-octahedral particles contain fine Ni-Fe inclusions (crystal seed) needed for the catalytic synthesis (Ref 2).

CuSn 85/15 with a size of 45-90 μm (GTV, Luckenbach, Germany) was taken as matrix material to manufacture superabrasive diamond composite layers by means of detonation spraying. The coatings were applied on C45-steel (1.0503) substrates with a dimension of Ø 16 × 40 mm. The C45 samples were grit-blasted before coating ($Rz = 73.5 \pm 8.7$ μm, $Ra = 14.4 \pm 0.9$ μm) to enhance the adhesion between substrate and coating.

2.2 Detonation Spraying

Detonation spray equipment (Surface Advanced Technology Inc., Warsaw, Poland) was used for the diamond-bronze spraying. Propane and oxygen were used as process gases. In regard to the process optimization presented in Ref 8, a propane flow of 2.6 L/min and an oxygen flow of 14.5 L/min were constantly supplied to the system. The process gases were ignited in the process chamber with a frequency of 2 Hz.

Diamond and bronze powder were fed into the detonation tube, accelerated and heated by means of the detonation. With two independent powder feeders it was possible to regulate the feeding rate and to ensure the exact diamond input. The powder-feeder (Electro-Plasma Inc., CA, USA) was used to insert the bronze powder (15 g/min), whereas the Single-10C powder feeder (Plasma-Technik AG, Switzerland) was employed for the diamonds (0.2 g/min). A nitrogen gas flow of 20 L/min was constantly applied for the particle feeding.

Substrate heating was employed to soften the matrix material and to increase the diamond implantation efficiency (Ref 8). Therefore, the substrates were heated by induction before and during the coating process. With the induction equipment (HG 3002, Himmelwerk GmbH, Tübingen, Germany) the sample was heated up to 650 °C within 2 min. During the spraying process, a reduced temperature of 590 °C was measured at the coated sample surface due to the cooling air flow after each detonation shock wave. The detonation spray and injection heating setup is illustrated in Fig. 2.

In addition to the spraying of diamond-bronze composites, a second detonation spraying setup was employed to extract thermally and kinetically stressed diamonds for

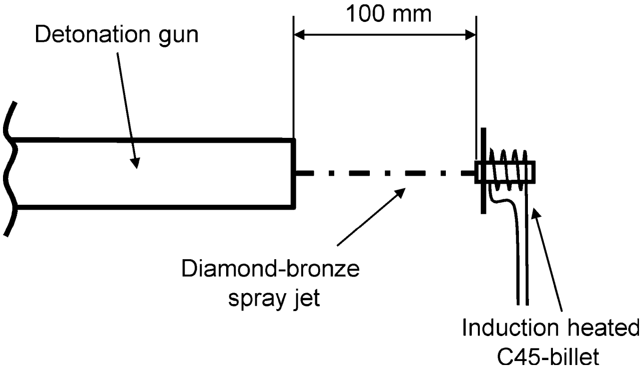

Fig. 2 Detonation spray setup for the coating of diamond-bronze on steel billets

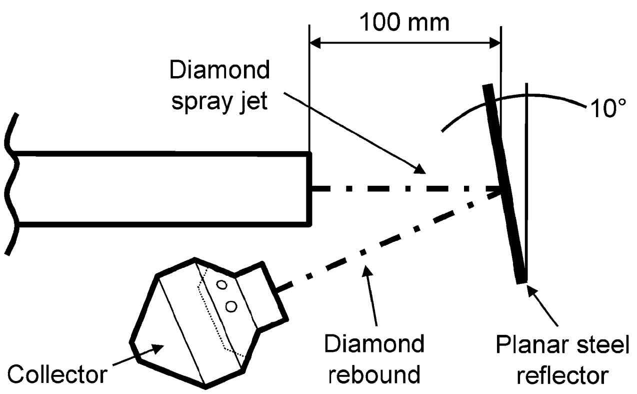

Fig. 3 Spray setup to collect detonation stressed diamonds

further analyses (Fig. 3). Due to the fact that diamond (carbon) does not chemically react with bronze, only diamonds were fed into the detonation gun. However, the bronze powder feeder was not used, the nitrogen gas flow remained constant to achieve comparable detonation temperatures and velocities. Employing the same spraying parameters as for the composite coatings, the diamonds were equivalently accelerated and heated by the detonation.

At a distance of 100 mm in front of the detonation barrel, a planar steel reflector of C45 was installed in an angle of 10° (Fig. 3). Rebounding diamonds were collected in a special interceptor with venting holes and rebound plates.

2.3 SEM/EDX

Scanning electron microscopy (SEM) (JXA-840, Jeol GmbH, Eching, Germany) was employed to visualize the topography of the sprayed composite layers, as well as the roughening and furrowing effects concerning the deterioration of the diamonds' surfaces. Additionally, the elemental analyses of diamond surface features after thermal degradation were carried out via energy dispersive x-ray spectroscopy (EDX).

2.4 DTA/TGA and Thermal Treatment

Differential thermal analyses (DTA) and thermogravimetric analyses (TGA) were performed to analyze the degradation of the processed diamonds in air (STA 409, Netzsch-Gerätebau GmbH, Selb, Germany). A constant amount of 60 mg diamonds was used for each test. The diamonds were heated in Al_2O_3 crucibles. The furnace chamber was heated with a rate of 5 K/min from room temperature to test temperature for the DTA and a heating rate of 5 K/min with an isothermal exposure of 10 min at 1000 °C for the TGA measurements. A constant air flow of 50 mL/min was maintained during all tests. Al_2O_3 has been used as standard for the DTA investigations.

A larger quantity of thermally treated diamonds for the friability and fracture force testing was generated in a tube furnace (SR 70-500/12, Gero Hochtemperaturöfen GmbH & Co. KG, Neuhausen, Germany). The diamond sample temperature was increased at 5 K/min up to 800 °C then held at 800 °C for 10 min followed by furnace cooling. During the annealing, the furnace tube was flushed with a constant air flow of 50 mL/min.

For comparative Raman measurements, an additional diamond sample was heat-treated (partially graphitized) in inert atmosphere. An amount of 60 mg SDB 1055 diamonds were annealed in argon (Ar 99.9999%) atmosphere at normal pressure in a closed hot isostatic press (HIP, ASEA Brown Boveri QIH-3, ABB GmbH, Friedberg, Germany). According to the research results of Seal (Ref 10), Quian et al. (Ref 11), and Fedoseev et al. (Ref 12) who investigated the graphitization of diamond in inert atmospheres, the argon annealing has been carried out at 1600 °C. The chamber was heated with 15 K/min and maintained at 1600 °C for 60 min.

2.5 XRD and Raman Spectroscopy

The crystallographic structure of the diamonds in the as-received condition, after thermal treatment and after detonation spraying, was investigated by x-ray diffraction using a D8 Advance diffractometer (Bruker GmbH, Karlsruhe, Germany) operated in $\theta/2\theta$ mode at 30 kV, 40 mA with a Cu-Kα radiation ($\lambda = 1.5406$ Å). A step size of 0.02° was gauged with a step time of 4 s.

Raman spectroscopy was conducted to investigate the diamond surface degradation in high magnification and local resolution. The LabRAM micro-Raman-spectrometer (Horiba Jobin Yvon GmbH, Bensheim, Germany) was used employing a He-Ne laser with a wavelength of 632.8 nm.

2.6 Friability Tests

The friability test was applied to determine the impact strength of diamonds named the toughness index (TI). The test setup consists of a special capsule containing a steel ball and the diamonds to be tested. Vibrating the filled capsule the steel ball impacts the diamonds. The lower the impact strength of the diamonds, the more diamonds break.

The measurements on the 60-70 mesh SDB 1055 diamonds were performed on friability tester type ST1 (Re.Tek., Italy). According to ANSI B74.23-2002, exactly 400 mg of sieved diamonds with a grade of 60-70 mesh were tested.

After having partially crushed the diamonds with the hardened steel ball, the broken diamonds were sieved out. The TI was calculated as percentage of the grains which were not destroyed, related to the input (400 mg).

2.7 Fracture Force Tests

The fracture strength of the diamonds was determined in a computer based single particle fracture force tester (DiaTest-SI, Vollstädt Diamant GmbH, Germany). Size, shape, fracture strength and fracture pattern were automatically determined for every tested particle. Detonation sprayed 800 °C air-annealed and virgin diamonds were investigated. At least 450 diamonds were tested each to achieve statistic reliable results.

3. Results and Discussion

3.1 Thermal Degradation of Diamonds

In the DTA measurements, the degradation of SDB 1055 diamonds with a size of 60/70 mesh was investigated in air under atmospheric pressure. Figure 4 shows the reaction enthalpy curve starting at room temperature up to 1000 °C.

In the temperature range from room temperature to 630 °C, the behavior of the diamond is endothermic. This indicates the energy absorption for the conversion of the metastable sp^3-structure to the energetically more stable sp^2-structure at the surface of the diamonds.

Oxidation of the diamonds started at ~630 °C, when the character of the curve changes slowly from endothermic to exothermic. However, the amount of oxidation remains low. Cooling down the investigated diamonds after the 700 °C DTA experiment to room temperature a mass loss of 1.68 mg was measured. This means an oxidation of 2.8% of initially processed diamonds.

Elevating the temperature up to ~840 °C, the average conversion energy reaches the local exothermic maximum. Continuously, the amount of diamond oxidation increases. At 800 °C, 22.9% of the diamond mass had reacted to CO and CO_2.

At higher temperatures over 840 °C, the reaction enthalpy decreases. Conversion processes of sp^3 to sp^2 increase faster than the oxidation processes at the diamonds surface. Nevertheless, the amount of oxidation reached 48.0% in the 900 °C DTA experiment.

At 980 °C, the sum of simultaneous endothermic and exothermic reactions approaches zero. The diamond mass loss was quantified to 75.2% at 1000 °C.

In addition to the qualitative mass loss detected in the DTA experiments after heating up and cooling down, the isothermic oxidation rate in air was investigated in the TGA apparatus (Fig. 5). It was observed that the initial weight of the sample started declining at around 630 °C, as expected. At 650 °C, a relative mass loss rate of 0.08 mg/min was confirmed.

In the test temperature range between 650 and 750 °C, the mass loss rate significantly increases to 0.22 mg/min. Above 750 °C, the degradation velocity reveals a regressive growth reaching the fastest oxidation velocity of 0.40 mg/min at the highest temperature of 1000 °C. However, a degradation velocity of 0.40 mg/min was proved in the described TG measurement, the regression of the curve is strongly dependent on the relative surface-oxygen contact. In further experiments, it was detected that, e.g., a slightly increased air flow leads to a higher degradation velocity above 750 °C.

3.2 SEM of Diamonds Before and After Thermal Exposure

The topography of the diamond was investigated by SEM. Diamonds in the as-received virgin condition, after different thermal exposures in the DTA experiments, and detonation sprayed diamonds were compared. The SEM results are given in Fig. 6.

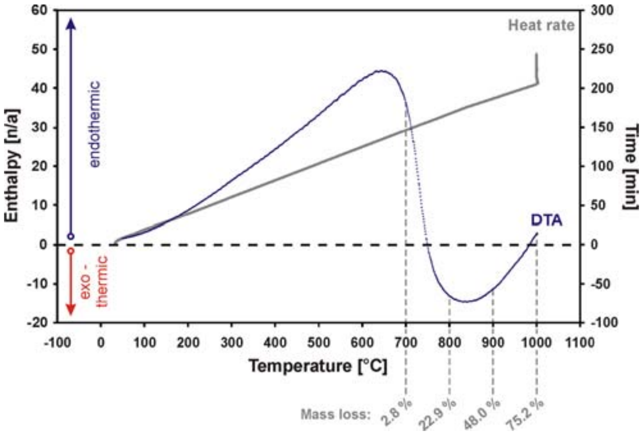

Fig. 4 DTA enthalpy curve of diamond, SDB 1055, 60/70 mesh

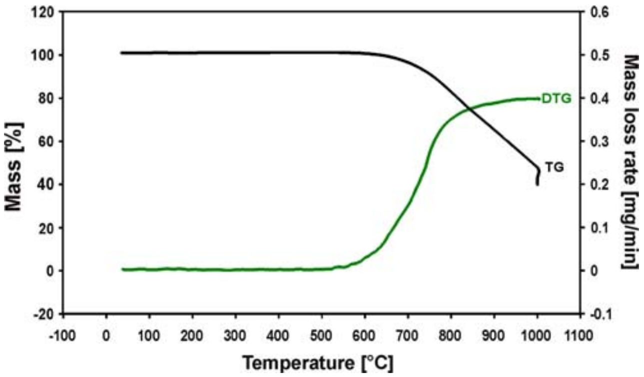

Fig. 5 TGA of diamond, SDB 1055, 60/70 mesh

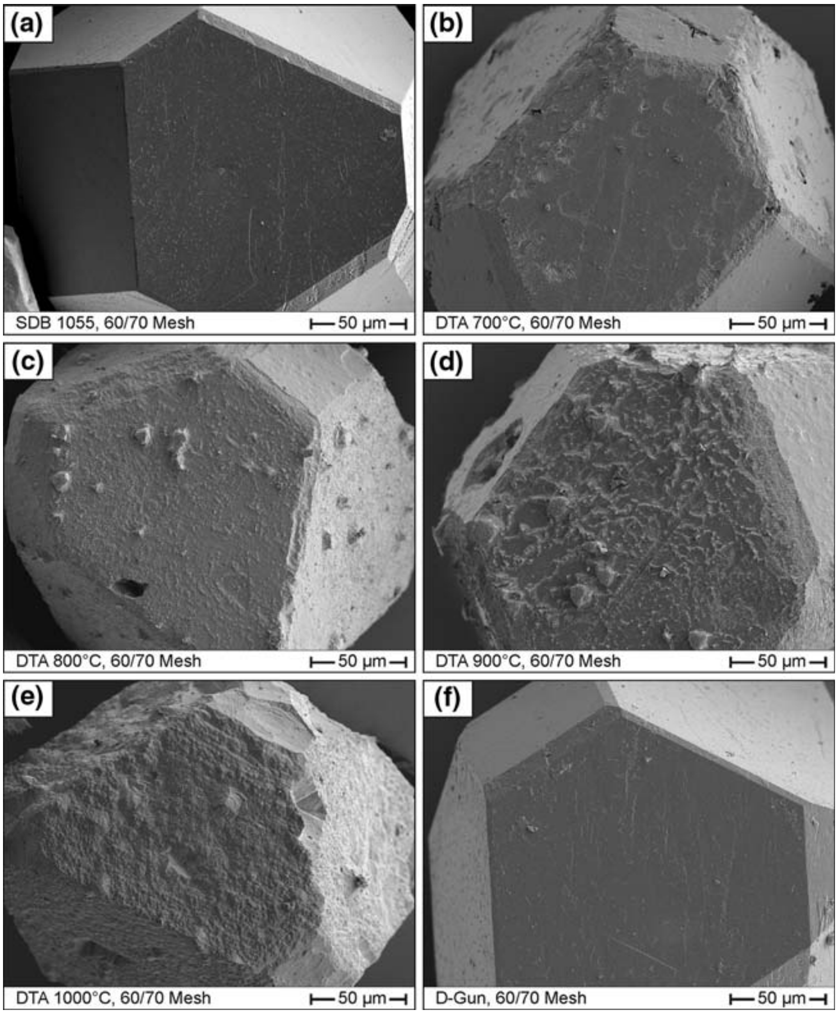

Fig. 6 SEM micrographs of SDB 1055 diamond, 60/70 mesh before and after different thermal treatment. (a) Diamond in the as-received condition. (b) After DTA measurement, 700 °C. (c) After DTA measurement, 800 °C. (d) After DTA measurement, 900 °C. (e) After DTA measurement, 1000 °C. (f) Detonation sprayed diamond

In the virgin condition without thermal treatment, the SDB 1055 quality shows a cubo-dodecahedral shape with a smooth surface and sharp defined edges (Fig. 6a).

Compared to the evident undamaged diamond facade of the virgin diamonds, the 700 °C DTA-annealed crystals featured a much rougher surface due to the initial oxidization in atmospheric air (Fig. 6b). After heating up with 5 K/min to 700 °C and directly cooling down, the edges are rounded and some shallow pits are formed on the side walls of the diamond. However, some unaffected diamonds could be found in the thermal-treated sample.

Raising the process temperature up to 800 °C, the micro- and macro-furrowing effects significantly increase (Fig. 6c). The walls of the diamond crystal become very rough. In addition to some deep pits, elevated spots were found on the surface. Nevertheless, EDX analyses showed no other elements except carbon.

Elevating the test temperature increased the amount of macro-furrowing, pits, and spots (Fig. 6d). At 1000 °C, the former dodecahedral shape of the crystalline diamond is completely destroyed (Fig. 6e). All diamond particles feature degradation effects of huge macro-furrowing all over the surface. A notable reduction of the median diamond size was observed due to the high amount of oxidation.

In contrast to the DTA annealed diamonds, the surface appearance of the detonation sprayed diamonds remains undamaged (Fig. 6f). After the thermal and kinetic impact of the detonation, no visible diamond surface degradation could be detected in the SEM. The smoothness of the walls and the sharpness of the edges remain preserved. No furrowing or pitting was detected. However, it is not known if a surface graphitization might have taken place.

3.3 XRD and Raman Spectroscopy

To verify the lattice form of the carbon structure, XRD and Raman spectroscopy were employed. DTA annealed

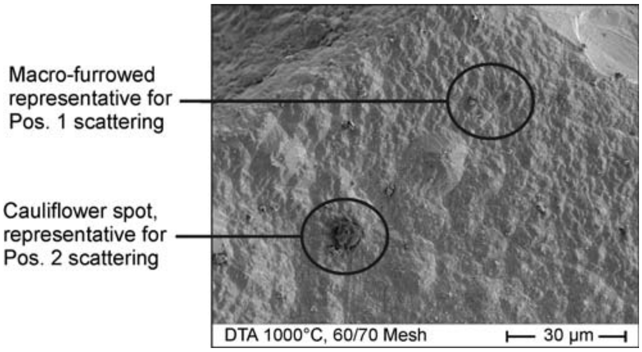

Fig. 7 SEM micrograph of SDB 1055 diamond, 60/70 mesh after 1000 °C DTA annealing in air

(1000 °C in air), detonation sprayed, and virgin diamonds were investigated. Additionally, inert-gas annealed diamonds (1600 °C in argon) were used for comparative Raman scatterings.

The XRD measurements did not show any remarkable differences between the as-received and the sprayed diamonds. Even the spectrum of the highly degraded 1000 °C DTA annealed diamonds showed the ideal peak of 100% crystalline diamond without graphitization. Because of the measurement principle of the used XRD, the result is based on a median value of ~100 diamond particles analyzed in the deep crystalline structure under the deteriorated surface of the diamond.

The micro-Raman spectroscopy is much more sensitive to the surface. In a high magnification, it was possible to investigate specific surface degradation effects like pits or the elevated spots on the annealed diamonds surface (Fig. 7). The result for the tested diamonds with different thermal treatment is illustrated in the Raman spectrum (Fig. 8).

Independent on the thermal treatment each investigated SDB 1055 sample showed its highest peak at 1335 cm^{-1}. This peak corresponds to the Raman diamond line which is 1332.5 cm^{-1} in an undisturbed diamond lattice (Ref 13, 14). The spectral shift of +2.5 cm^{-1} correlates to the diamond growth on Ni-Fe crystal seed (Ref 14). Illustrated in the spectrum of the virgin diamond reference (Fig. 8a) no other band or line could be detected. This indicates the good-quality of the crystalline diamond.

The 1000 °C DTA annealed diamonds predominantly showed equal scatterings as the SDB 1055 reference, which indicates that the surface degradation is mainly affected by oxidation processes without significantly changing lattice structure of the diamond (Fig. 8b). Only on cauliflower formed elevated spots (Fig. 7) some features caused by mechanisms of the allotropic conversion of the metastable sp^3-structure to the thermodynamically favorable sp^2-structure were detected (Fig. 7 and 8c).

The weak feature appearing in the scattering at 1406 cm^{-1} (D-band) and the line at 1505 cm^{-1} are associated with sp^2 hybridized carbon phases (Fig. 8c) (Ref 14). The noticed point defects related to the broad peaks at 1771, 1942, and 2038 cm^{-1} (Ref 14) indicate a distinctive feature of disordered diamond with nondiamond carbon phases on the surface.

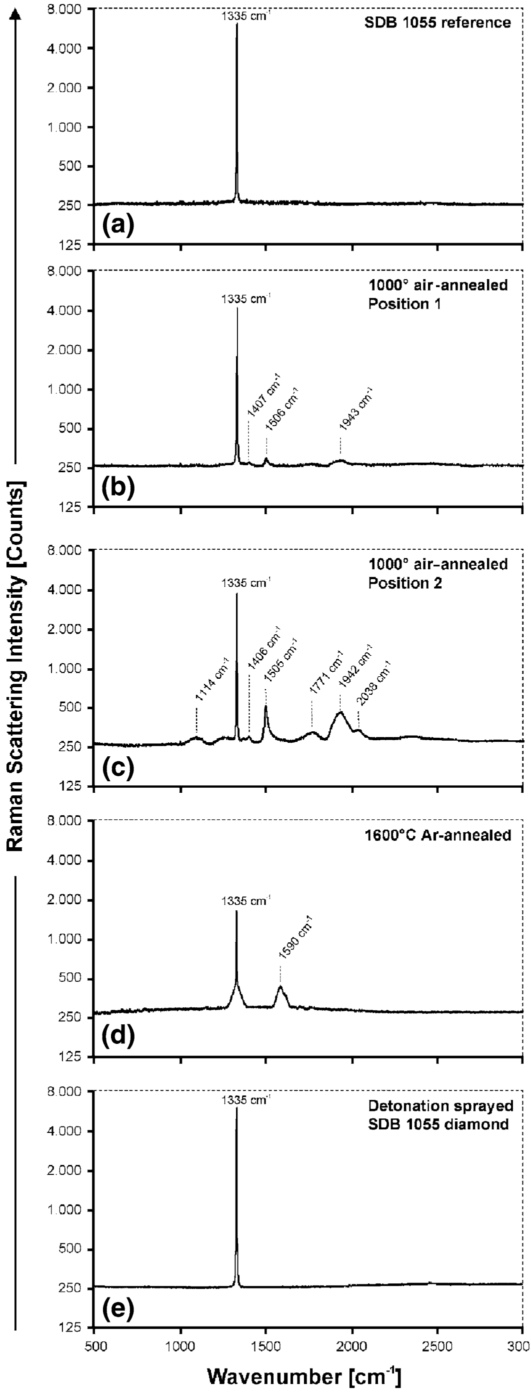

Fig. 8 Raman spectrum of (a) virgin diamonds, (b, c) 1000 °C air-annealed, (d) 1600 °C Ar-annealed, and (e) detonation sprayed SDB 1055, 60/70 mesh diamonds

The weak band at 1114 cm^{-1} is attributed to a thermochemical treatment with the nickel inclusions at moderate temperatures of 700-800 °C (Ref 14).

However, point defects and sp^2 indicators are detected in the Raman scattering of the annealed diamond, the

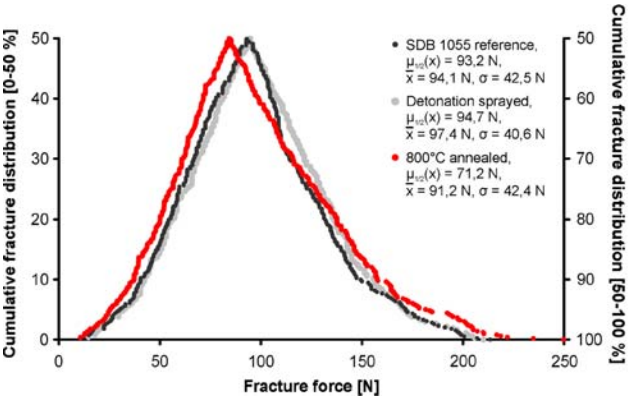

Fig. 9 Fracture force test of diamonds

most important information is particular the missing graphite line (1581-1585 cm^{-1}) (Ref 13, 14) (Fig. 8c). It verifies that no graphite but carbon metastructures of sp^2-sp^3 hybridization are formed on the surface. In the oxygen atmosphere, the metastructures are reacting directly to CO and CO_2.

The comparative Raman scattering of 1600 °C argon-annealed SDB 1055 diamond demonstrates the spectrum of a skin-deep graphitized diamond lattice (Fig. 8d). In addition to the diamond line at 1335 cm^{-1}, the scattergram features an explicit graphite peak at 1590 cm^{-1}.

The detonation sprayed diamonds did not show any effect of structural changes in the diamonds lattice. As illustrated in Fig. 8(e), pure diamond without degradation is indicated in the Raman scattergram.

3.4 Influence of Surface Degradation on Diamond Strength

The DIATest was used to determine size and shape of the diamonds and the compressive fracture force. From the size and fracture force the compressive fracture strength was calculated. As-received, detonation sprayed, and 800 °C air-annealed diamonds were compared. The 800 °C annealed diamonds were selected because their surface degradation was measurable in the previous DTA, TG, and SEM experiments but not as progressed to a more or less complete destruction of the diamond as it was in the 1000 °C annealing experiment. The results are shown in Fig. 9.

The folded cumulative fracture distribution curves of all diamonds tested basically feature the same shape with a slightly positive skewness. Whereas the curves of the SDB 1055 reference and the detonation sprayed diamonds are almost in line with each other, the curve of the 800 °C annealed diamonds is shifted to lower fracture forces. Its median ($\mu_{1/2}(x)$) is with 71.2 N about 23 N lower than the medians for the detonation sprayed diamonds and the reference diamonds. Concerning the arithmetic mean of the fracture forces, the three tested diamond samples showed a smaller divergence compared to the medians. This is due to the fact that the highest positive skewness was detected for the 800 °C annealed diamonds. An arithmetic mean fracture force of 91.2 N was identified for the 800 °C annealed diamonds, 94.1 N for the reference diamond sample in the as-received condition and 97.4 N for the detonation sprayed diamonds.

Table 1 Friability test

Sample	Unfractured, mg	TI ($C = 2000$)
SDB 1055 reference	259	65 (64,75)
Detonation sprayed	262	66 (65,50)
800 °C annealed	249	62 (62,25)

Although the measurements indicate that the 800 °C annealing reduces the diamonds strength and the detonation spraying does not affect it, the high fracture force deviation in all measurements of 40.6-42.5 N reduces the statistical reliability of these results. Nevertheless, the friability test confirmed the result of the diamonds strength. The TI representing the percentage of undestroyed diamonds after testing, related to the input, is given in Table 1. The highest values were measured for the detonation sprayed (TI = 66) and as-received diamonds (TI = 65). The difference between 800 °C thermally treated (TI = 62) and detonation sprayed diamonds revealed that the spraying process had no effect on the diamonds durability and performance.

4. Conclusion

Thermal deterioration of diamonds in air atmosphere is characterized by the mechanisms of the allotropic conversion of the metastable sp^3-structure to the thermodynamically favorable sp^2-structure and the immediate oxidation to CO and CO_2. In DTA measurements, first surface reactions on 60/70 mesh (210-250 μm) SDB 1055 diamonds were determined at 630 °C. However, the oxidation velocity remains extremely low up to a temperature of 650 °C. Raising the temperature to 1000 °C the oxidation rate increases to 0.40 mg/min. Strong surface destructions were detected in the DTA tests above 800 °C corresponding to the long heating time. In SEM, micro- and macro-furrowing effects were noticed reducing the diamond size, and significantly increasing the surface roughness. Additionally, features of shallow pits and elevated spots were formed on the surface. In Raman spectroscopy, metastructures of sp^2 and sp^3 hybridized carbon were confirmed on the upper surface of the diamonds immediately oxidizing to CO and CO_2. In fracture force and durability tests on diamonds annealed at 800 °C, a slight strength reduction of ~5% was determined compared to the as-delivered diamond state.

The analytical and mechanical investigations on detonation sprayed diamonds showed that no degradation effects took place during the spraying process due to the short duration of the detonation impact. Neither in SEM micrographs nor in Raman spectroscopy deterioration indicators were observed on the processed diamonds. Furthermore, the fracture toughness and durability of the diamonds were confirmed to be unaffected by the thermal and kinetic impact of the detonation.

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