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Creation of Smooth and Flat Surface in Micro-machining of Monocrystalline Diamond by Pulsed Laser

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Abstract

Both picosecond and nanosecond pulsed lasers enable unique process to obtain smooth and flat surfaces of Ib type monocrystalline diamond. Picosecond pulsed laser can achieve flat and smooth surface of Ra=0.2 µm at a certain overlap rate of laser shot. This process is mostly performed by series of crack propagation in parallel direction to top surface of diamond, and laser scanning area can keep diamond structure. On the other hand, combination of nanosecond pulsed laser and acid cleaning can perform shiny and flat surface in parallel direction to top surface of diamond at the transitional region of removal of diamond. Its surface roughness is smaller than Ra=0.2 µm, and Raman spectroscopy analysis shows glassy carbon structure of processed surface. Combination of these phenomena would contribute shape creation processes of diamond.

Keywords: monocrystalline diamond; micro machining; smooth surface; pulsed laser

1. Introduction

Diamond has been widely used for cutting tools because of its excellent properties such as high hardness, high conductivity and small coefficient of thermal expansion, as reported by Field et al., 1996 and Berman 1965. However, it is difficult to machine by conventional mechanical methods, because of its high hardness and brittleness. In recent years, laser processing is expected as a processing method for diamond, since it is highly efficient process without mechanical contact. On the other hand, diamond has high transparency at visible wavelength, but energy absorption can be performed by pulsed laser beam with high peak intensity. However, finishing process is necessary on the processed surface where the heat affected layer remains after laser irradiation. Polishing with diamond powder abrasive grains is used as a common method of finishing process, but there are problems, such as high cost and long processing time. Therefore, the cost reduction can be expected, if the polishing time can be shortened by the reduction of surface roughness with laser processing. In addition, high quality laser processing can be expected by the reduction of heat affected layer

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with less thermal load. Therefore, in this study, monocrystalline diamond was irradiated by nanosecond and picosecond pulsed lasers, and its removal process was investigated by observing the processing phenomena, evaluating surface roughness and groove shape on the bottom of processed area.

2. Experimental Procedures

In this study, nanosecond and picosecond pulsed lasers were used, and main experimental conditions are shown in Table 1. In nanosecond pulsed laser experiments, the wavelength, the pulse duration, the pulse energy, and the pulse repetition rate were 1060 nm, 200 ns, 50 μ J, and 50 kHz, respectively. On the other hand, the wavelength of 1064 nm, the pulse duration of 12.5 ps, the pulse energy of 12.5 μ J and the pulse repetition rate of 200 kHz were set in picosecond pulsed laser experiments. Both laser beams have Gaussian mode.

Monocrystalline diamond was classified into 4 types as stated by Breeding, 2009, and Ib type, which is mainly employed in industrial applications, was used as a specimen. The crystallographic orientation of top surface was mainly (111), and the axis of laser beam was set in perpendicular direction to the top surface of monocrystalline diamond. Only when discussing the influence of crystallographic orientation, (110) was set at the top surface of specimen, and binderless polycrystalline diamond (PCD) of nano-grains was also used.

The number of laser scans was once for all experiments, and monocrystalline diamond was processed in air without assist gas. Shot number was defined as the case that the time of laser shot existed within the spot diameter, and the value of shot number was varied by controlling the scanning velocity at the fixed pulse repetition rate.

Table 1. Experimental conditions

Pulse duration	200 ns	12.5 ps
Wavelength	1060 nm	1064 nm
Polarization	Random	Circular
Pulse repetition rate	50 kHz	200 kHz
Pulse energy	50 μJ	12.5 µJ
Focal length	100 mm	100 mm
Spot diameter	45 μm	25 μm

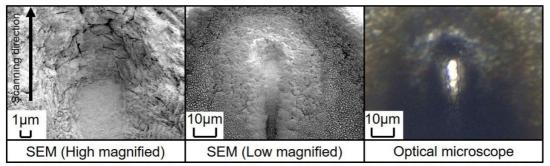
3. Experimental Results and Discussion

3.1. Nanosecond pulsed laser

Figure 1 shows scanning electron microscope (SEM) and optical microscope images of processing front, when the laser beam irradiation of 200 ns was stopped instantly. The center SEM and the right optical microscope images are the same location, and left SEM image is highly magnified one of the center SEM. As shown in the optical microscope image, the reflection of light is obvious at the bottom of processing front, although other areas are low light reflection. In highly magnified SEM image, the slope of processing front seems to be roughened surface, but almost flat surface can be observed at the bottom of processing front.

The slope and the bottom surfaces of processing front were analyzed by Raman spectroscopy as shown in Fig. 2. Both g-band around 1600 cm⁻¹ and d-band at 1333 cm⁻¹ appeared at both surfaces, which are similar spectra to glassy carbon or diamond like carbon (DLC). As shown in Fig. 1, the surface roughness at the

bottom of processing front is smaller than that at slope of processing front. Although material is the same, the intensity of reflection light differs by its surface roughness. Thus, it is considered that the intensity of reflection light at the bottom of processing front becomes strong compared to other areas due to its small surface roughness. The interesting point is the flat surface of processing front, and the processed grooves were observed after the removal of graphite layers.



Pulse duration: 200ns, Wavelength: 1060nm, Repetition rate: 50kHz, Pulse energy: 50μJ, Shot number: 1000shots

Fig. 1. SEM and optical microscope photographs of processing front in the case of nanosecond pulsed laser

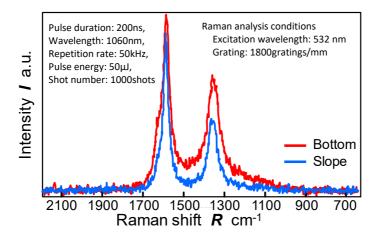
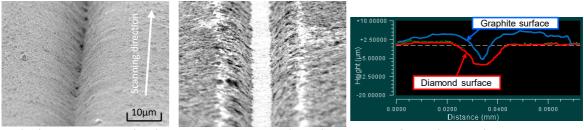


Fig. 2. Raman spectra at the bottom and slope surfaces of processing front in the case of nanosecond pulsed laser

Graphite layer was removed by acid cleaning process, in which nitric acid of 60 % and sulfuric acid of 97 % were mixed by the ratio of 1 to 3, and the processed specimens were kept in this mixed liquid for 1 hour at 300 degrees Celsius. Figure 3 shows SEM images and shapes of processed groove before and after the removal of graphite layer by acid cleaning. Surface profiles of processed groove were measured by using a white light interferometric microscope. As shown in the figure, the processed groove was covered by graphite layer, and the graphite layer was successfully removed by acid cleaning. In addition, the processing front was not covered by the graphite layer, as shown in Fig. 1. However, the processed groove was covered by the graphite layer. Thus, it is considered that diamond was removed by ablation process at the processing front, and scatted material from the processing front was deposited at the back side of processing front. The bottom surface of processing front was almost flat in parallel direction to the top surface, and the smooth surface could be obtained at the bottom surface of groove after acid cleaning. Raman spectroscopy analysis

made it clear that the flat surface at the bottom of groove could keep the diamond structure after acid cleaning. Therefore, combination of nanosecond pulsed laser and acid cleaning can perform shiny and flat surface in parallel direction to top surface of diamond.



 $Pulse\ duration:\ 200ns,\ Wavelength:\ 1060nm,\ Repetition\ rate:\ 50kHz,\ Pulse\ energy:\ 50\mu J,\ Shot\ number:\ 1000shots$

(a) Surface of graphite

(b) Surface of diamond

(c) Shape of processed groove

Fig. 3. SEM photographs and shapes of processed groove before and after removal of carbide layer in the case of nanosecond pulsed laser

These phenomena were observed only at the case when the pulse energy was a little higher than the threshold value of removal. In this case, sublimation phenomenon would be generated at the center area of laser beam due to small difference of laser intensity, and smooth flat surface would appear at the bottom of groove.

Figure 4 shows the surface roughness R_a before and after laser processing with acid cleaning for various shot numbers. Under one processing condition, 10 experiments were conducted, and the average of 10 measurements was recorded as the value of surface roughness. Surface roughness was measured by using a white light interferometric microscope. The surface roughness at the bottom of groove becomes almost half compared to that before processing for all shot numbers. At the same pulse energy condition, the surface roughness smaller than $Ra=0.2~\mu m$ can be performed regardless of shot number. Thus, the large area creation of smooth surface would be expected by controlling scanning path, because this phenomena is insensitive to overlap of laser shot.

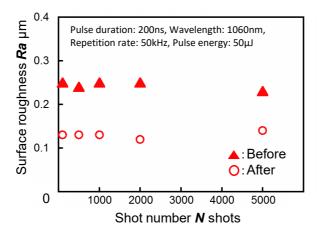


Fig. 4. Surface roughness before and after processing with acid cleaning for various shot numbers in the case of nanosecond pulsed laser

3.2. Picosecond pulsed laser

Figure 5 shows the difference of surface roughness R_a before and after laser process, and SEM images at shot number of 1000 shots are arranged at the right side in the figure. Surface profiles at the bottom area of processed groove were measured by using a white light interferometric microscope. Under one processing condition, 10 experiments were conducted, and the average of 10 measurements was recorded as the value of surface roughness. The surface roughness of processed area normally increased compared to that before laser beam processing, but its value became smaller only at shot numbers of 500 and 1000 shots. However, not all processing areas showed smaller surface roughness at shot numbers of 500 and 1000 shots, and both rough and smooth surfaces appeared at the same irradiation condition as shown in SEM images. The crack propagation was observed at the processing front in the case of smooth surface, and this phenomenon appeared at higher pulse energy more than a certain value. The plane of smooth surface was almost parallel to the top surface of diamond, although the laser irradiation axis is perpendicular to the top surface of diamond. In normal, cracks would propagate in the parallel direction to the laser irradiation as reported by Sakakura et al., 2013, when a laser beam was focused inside transparent materials. This phenomenon is very unique, and stable creation of this phenomenon would contribute high quality laser beam processing of diamond.

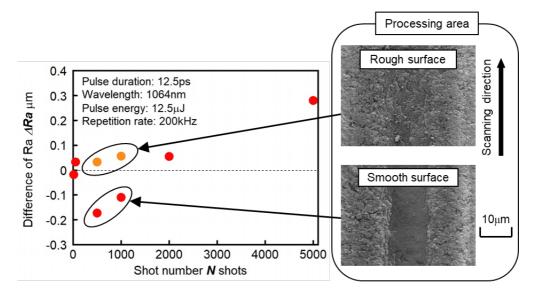


Fig. 5. Difference of surface roughness before and after processing, and SEM images at shot number of 1000 shots in the case of picosecond pulsed laser

Figure 6 shows SEM images of processed areas for (111) and (110) top surface of monocrystalline diamond, and binderless PCD without specific crystallographic orientation. Ramaseshan, 1946 reported that the (111) cleavage was found to be by far the most perfect and most abundant, because that plane has the minimum cleavage energy. Therefore it is considered that crystallographic orientation (111) is easy to be fractured compared to other orientations. On the other hand, similar cleavage phenomena were observed not only for other crystallographic orientation (110) but also for polycrystalline diamond of nano-grains. These cleavage phenomena were confirmed not only in the case of specific crystallographic orientation but also for diamond without specific crystallographic orientation. Therefore, this unique process, in which

smooth surface can be obtained in parallel direction of laser irradiation, would be generated by gathering the small cracks caused by laser irradiations.

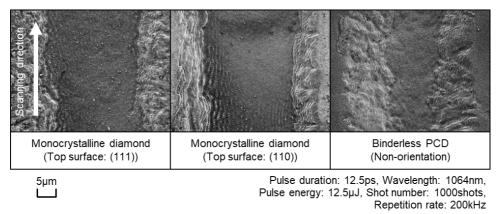


Fig. 6. Generation of smooth surface for various diamond types using picosecond pulsed laser

Conclusions

Main conclusions obtained in this study are as follows:

- (1) In the removal process of diamond by nanosecond pulsed laser, smooth surface can be observed at the bottom surface of groove after acid cleaning of graphite layer.
- (2) In the case of nanosecond pulsed laser, it is suggested that smooth surface can be performed by a surface of low laser absorption rate which is appeared due to sublimation phenomenon.
- (3) In the removal process of diamond by picosecond pulsed laser, two processing types of smooth and rough surface can be observed at the bottom surface of groove at specific shot number and pulse energy.
- (4) In the case of picosecond pulse duration, it is suggested that smooth surface can be performed by cleavage phenomena in parallel plane to the top surface of diamond.

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References

Berman, R., 1965. Physical Properties of Diamond, Clarendon Press, London, p.140.

Breeding, C. M., Shigley, J. E., 2009. The "Type" Classification System of Diamonds and Its Importance in Gemology, Gems and Gemology 45(2), p.96.

Field, J. E., Pickles, C. S. J., 1996. Strength, Fracture and Friction Properties of Diamond, Diamond and Related Materials 5, p.625. Sakakura, M., Ishiguro, Y., Shimotsuma, Y., Fukuda, N., Miura, K., 2013. Modulation of Transient Stress Distributions for Controlling Femtosecond Laser-induced Cracks inside a Single Crystal, Applied Physics A: Materials Science and Processing, 114(1), p.261. Ramaseshan, S., 1946. the Cleavage Properties of Diamond, Proceedings of Indian Academy of Sciences, Section A, 24(1), p.114.