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ABSTRACT

Surface polishing of hard diamond coatings is a key technique for applying such coatings in industry. In this study, we demonstrated surface modification of polycrystalline diamond films with minimal volume loss of the films by vertical irradiation of KrF excimer laser beams. Optimized laser scanning selectively removed surface asperities and reduced the surface roughness from ∼0.1 to ∼0.05 μm. Raman spectroscopic measurements revealed that laser polishing involves the phase transformation of diamond to amorphous carbon phases and thermal oxidation or evaporation of the amorphous phases. The residual amorphous carbon was almost completely removed by subsequent laser irradiation at appropriate fluences. We consider that the surface texture inherent to polycrystalline diamond films plays an important role in the concentration of laser beams on the nearby film surface. From reciprocating slide-type friction tests, we found that laser polishing decreased the initial friction coefficient from 0.5 to 0.1–0.3 against alumina balls and reduced the abrasion of the mating materials.

Diamond is a common coating material in industrial applications, including cutting tools,[1] sliding members,[2] heat spreaders,[3] and chemical electrodes.[4] This is because of diamond's excellent physical and chemical properties, such as its high hardness, high wear resistance, high thermal conductivity, chemical inertness, and biocompatibility. Diamond coatings are conventionally deposited as polycrystalline films using chemical vapor deposition (CVD) techniques, such as hot-filament CVD and microwave plasma-enhanced CVD.[5,6] CVD diamond grows with a columnar structure from crystal nuclei to cover a base material, and the grown films are composed of randomly oriented crystal grains.[7] An increasing film thickness corresponds to a large grain size and rough surface, which substantially deteriorates the film's friction and wear properties.

To obtain diamond films with a smooth surface, there are two main approaches. One is grain fining during film growth, and the other is surface polishing of the grown films. Grain fining proceeds by modulating the growth parameters to promote re-nucleation, including the source gas concentration, process pressure, and bias treatment.[8] The films are either nanocrystalline diamond (NCD) or ultra-nanocrystalline diamond (UNCD), depending on the grain size.[9] NCD films (and especially UNCD films) have a smooth surface and exhibit low friction efficiency.[10,11] There are reports on their coating performance for cutting tools[12,13] and mechanical seals.[14] However, reducing the grain size results in an increase in the contents of graphitic phases and hydrogen in the films.[15,16] These changes degrade the wear resistance of the films and limit the film thickness up to several micrometers owing to the accumulation of intrinsic stress in the films.[17,18] Maintaining a large grain size prevents the deterioration of diamond's inherent physical properties[19,20] and also enhances adhesion between the films and substrates.[21,22]

Surface polishing is a promising solution for overcoming the trade-off between the surface morphology and film properties of CVD diamonds. Polishing can be either with or without contact.[23,24] Diamond-polishing techniques have been developed as contact methods

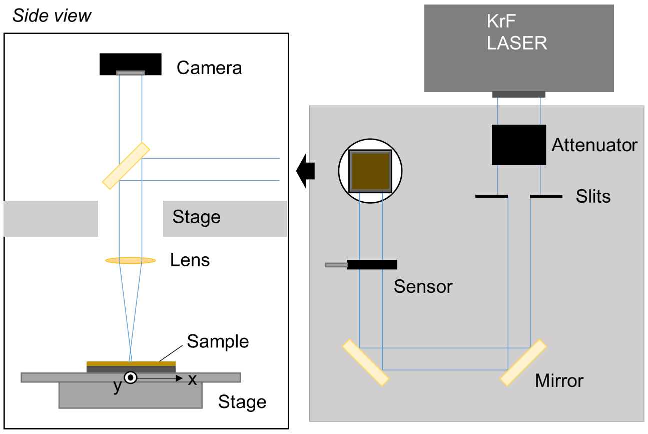

FIG. 1. Schematic illustration of a KrF laser-irradiation system for surface polishing of polycrystalline diamond films.

based on mechanical polishing with chemical and thermal assistance. These methods provide rough and finishing polishing for the planarization of two-dimensional objects. However, contact methods are time-consuming, and there are problems of microcracking and impurity contamination on the film surfaces.[25–27]

Laser polishing, which is a noncontact method,[24] can be used for overall machining of diamond materials in air (i.e., cutting and microstructuring as well as polishing).[28] This polishing method is applicable for three-dimensional objects regardless of their size and shape as long as the laser can fully access the surface. Laser polishing is advantageous for the processing of coating films, whereby it is necessary to maintain the designed shape of the processing objects (e.g., the cutting edge of the drill and the surface of the mold). Although there are reports of laser polishing of polycrystalline diamond films, most such films have a high roughness and thickness like in the bulk state,[23,29] because the thickness must be sufficiently greater than the ablation depth to form smooth surfaces.[30,31] This paper reports surface polishing with a pulsed krypton fluoride (KrF) excimer laser for polycrystalline diamond films prepared on cobalt-cemented tungsten carbide (WC-Co) substrates. We discuss the laser polishing with little loss of film thickness and describe the friction properties of the laser-polished films based on the results of reciprocating sliding tests.

Polycrystalline diamond films with thicknesses of $\sim$10 μm were prepared on WC-Co substrates by hot-filament CVD. Figure 1 shows a schematic illustration of the laser-irradiation system. A KrF excimer laser (Gigaphoton, Inc.) with a wavelength of 248 nm ($h\nu = 5.0$ eV) and a pulse duration of $\sim$15 ns was used without optical pulse stretchers. The films were placed on an x–y axis movable stage. Laser beams emitted from the excimer laser were guided to the films with mirrors and were focused on the film surface with a focusing lens. The beams were irradiated from the direction perpendicular to the film surface. Laser irradiation was carried out with scanning at 0.3 mm s^{-1} in the direction of the y axis in an ambient atmosphere; the repetition rate of the laser pulses was 100 Hz or 1 kHz. The laser spot had a rectangular shape (dimensions: 0.5×0.2 mm^2). The fluence of the pulsed beam was up to 4 J cm^{-2}.

The surface morphology of the as-grown and laser-irradiated diamond films was observed using an optical microscope and a scanning electron microscope (SEM). The cross-sectional images of the samples were obtained with the SEM. The arithmetic mean roughness (R_a) and maximum height (R_z) were estimated using a laser confocal scanning microscope (OLS4100, Olympus). The crystalline structures of the diamond films were characterized using a Raman spectrometer (NRS-5100, Jasco) at room temperature. The excitation wavelength was 532 nm and the spectral resolution was 0.4 cm^{-1}. The friction properties of the films were evaluated with a reciprocating slide-type friction tester (FPR2100, Rhesca) with a ball-on-plate configuration. Alumina (Al_2O_3) ceramic balls with a diameter of 3 mm were used as counterpart materials. The normal load was 0.49 N, which caused a maximum Hertzian contact stress of $\sim$0.6 GPa in the initial state. The friction test was performed at room temperature (24–25 °C) in air with 34% relative humidity (RH). The linear velocity was 25 mm s^{-1} for a stroke length of 5 mm, and the sliding test time was 600 s. The friction coefficient was calculated as a moving average from the measured friction force.

Figure 2 shows typical cross-sectional SEM images of the as-grown and laser-irradiated films. Structural changes resulting from laser irradiation were evident on the film surface. Whereas the as-grown films exhibited a pyramidal texture structure that was attributable to crystalline facets, the laser-irradiated films exhibited a flat surface. The laser irradiation selectively removed the asperity structures within $<$2 μm of the surface (i.e., with minimum loss of film thickness and volume). After laser irradiation, the R_a of the films decreased to $\sim$0.05 μm (originally 0.1 μm) and the R_z decreased to 0.1 μm (originally 0.2 μm). These roughness values are some of the lowest values obtained by laser polishing.[23,29,32] However, the surface roughness after laser polishing depends on the state of the pristine films (e.g., the grain size, crystal orientation, and film thickness).

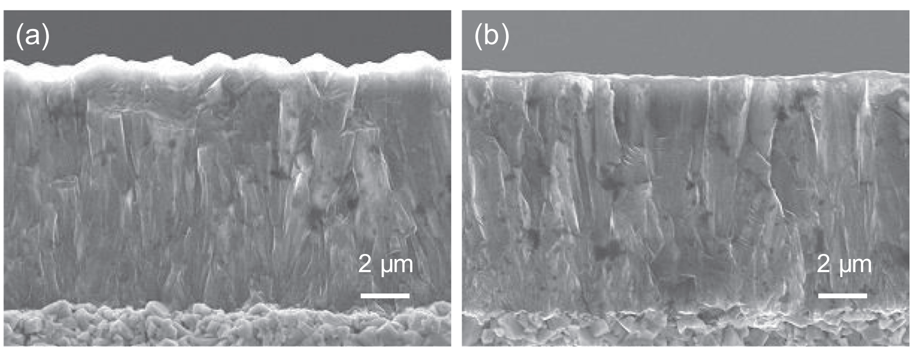

FIG. 2. Cross-sectional scanning electron microscopy images of (a) as-grown and (b) laser-irradiated films (fluence, 3.5 J cm^{-2}; frequency, 100 Hz).

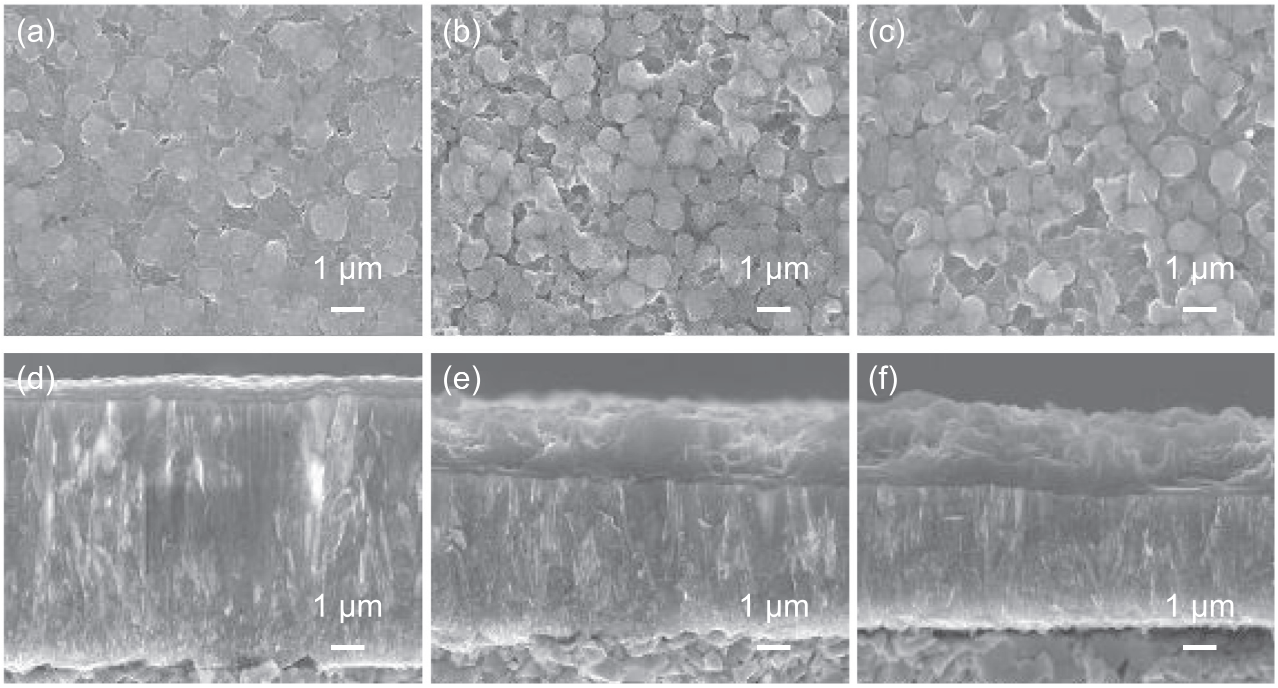

FIG. 3. [(a)–(c)] Surface and [(d)–(f)] cross-sectional scanning electron microscopy images of laser-irradiated diamond films at various fluences (frequency: 1 kHz): [(a) and (d)] 3.0, [(b) and (e)] 3.6, and [(c) and (f)] 4.0 J cm^{-2}.

We investigated the effects of the laser-irradiation parameters to understand how the surface modification and material removal proceeded. Figure 3 shows surface and cross-sectional SEM images of the laser-irradiated diamond films at fluences of 3.0, 3.6, and 4.0 J cm^{-2} under 1-kHz operation. There were no evident structural changes of the film surfaces by irradiation lower than 2.8 J cm^{-2}. The threshold for surface modification was ~3 J cm^{-2}, which is close to the ablation threshold for polished polycrystalline diamond.[33] Laser irradiation at ≥3 J cm^{-2} formed ball-shaped grains that covered the film surfaces [Figs. 3(a)–3(c)]. We did not observe local machining, such as the elongation of ablating tails by oblique laser irradiation.[34] Increasing the fluence from 3.0 to 4.0 J cm^{-2} reduced the film thickness and led to the formation of residues at the film surfaces [Figs. 3(d)–3(f)]. The excessive irradiation completely removed the films and exposed the substrate surfaces. The uniformity of the laser processing depends on the experimental parameters, such as the energy distribution in the laser beam and variations of the laser incident angle.

We characterized the crystalline structure of the films using Raman spectroscopy. Figure 4 shows typical Raman spectra of the as-grown and laser-irradiated films. The spectrum of the as-grown film [Fig. 4(a)] was mainly characterized by a sharp peak at ~1335 cm^{-1} and a broad band centered at ~1520 cm^{-1}. The sharp peak is assigned to a typical diamond peak,[35] and the board band to the G-band (related to sp^2-bonded carbon phases).[36] We did not observe spectral shapes unique to nanoscale diamond, which have peaks at ~1150 and 1450 cm^{-1}.[37] The spectra of the laser-irradiated films [Figs. 4(b)–4(d)] were characterized by two broad bands centered at ~1330 and 1530 cm^{-1}, which are assigned to G- and D-bands, respectively. The diamond peak at ~1335 cm^{-1} disappeared after laser-induced modification. This is because the intensity of the graphite spectra due to sp^2 states was ≥50 times larger than that of the diamond spectra due to sp^3 states.[38] The laser-irradiated areas are usually covered by graphitized layers.[39,40]

We preferentially removed the amorphous carbon residues that remained on the film surfaces by further laser irradiation at a lower fluence (2 J cm^{-2}) than the threshold for surface polishing with the same laser-irradiation system, resulting in glossy surfaces. The top of the film surfaces was still covered by non-diamond phases identified in Figs. 4(e)–4(g). In this case, diamond peaks were difficult to observe in the Raman spectra.[41] The Raman spectra had two broad peaks centered at ~1330 and 1570 cm^{-1}, similar to the spectrum of glassy carbon.[35] Such non-diamond phases may be removable by plasma etching or thermal annealing.[30,39,42,43] For single-crystalline diamond, irradiation at a much lower fluence than the graphitization threshold leads to the removal of non-diamond phases and exposure of the pristine diamond surface.[31]

We evaluated the friction properties of diamond films using reciprocating friction tests with alumina balls. The friction coefficients of the as-grown and laser-polished films exhibited different behavior, especially at the beginning of the sliding test. The friction coefficient of the as-grown films was initially 0.5 and then gradually decreased before stabilizing at 0.1–0.3; by contrast, the friction coefficient of the laser-polished films was stable at 0.1–0.3 from the beginning of the test. In general, diamond films exhibit relatively high initial friction coefficients in the run-in period.[11,30,42] This behavior depends on the kind of mating materials, size of the loads, and the peripheral environment.[10,11] The surface of the as-grown films prepared by CVD would be terminated by hydrogen atoms,

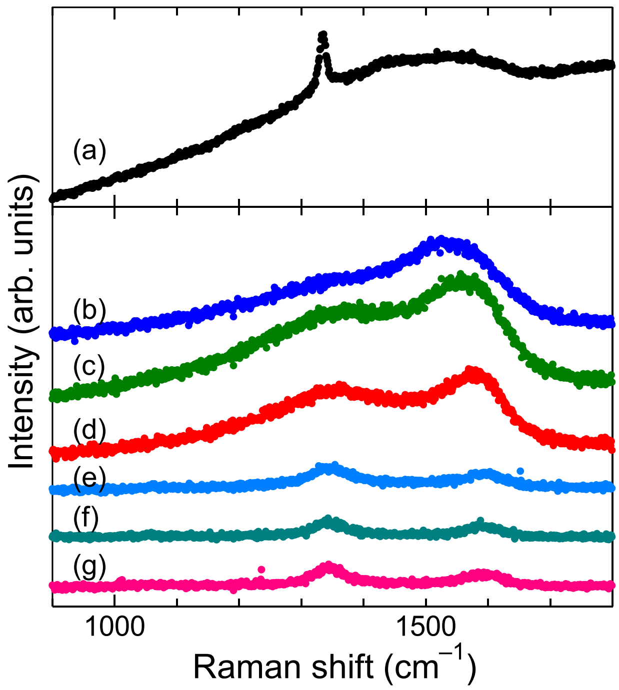

FIG. 4. Raman spectra. (a) As-grown film. [(b)–(g)] Laser-irradiated films with the following parameters: [(b) and (e)] 4 J cm^{-2} and 100 Hz, [(c) and (f)] 3 J cm^{-2} and 1 kHz, and [(d) and (g)] 4 J cm^{-2} and 1 kHz. Spectra (b)–(d) are of asperity structures damaged by laser irradiation under the aforementioned conditions. Spectra (e)–(g) are taken after removal of the amorphous carbon residues covering the film surfaces by laser irradiation at 2 J cm^{-2}.

whereas laser-polished films have a carbon layer, which may act as a lubricant.[30,42]

The wear volume of the alumina balls markedly decreased from $\sim 2 \times 10^{-4}$ mm^3 for the as-grown films to $3–6 \times 10^{-5}$ mm^3 for the laser-polished films. For the as-grown films, a rough surface decreased the actual contact area at the interface between the balls and the films owing to the local concentration of contact pressure on the tip of the surface asperities of the films. This caused a high friction coefficient in the initial state and abrasion and plowing of the mating surfaces during sliding tests, resulting in the wear of the alumina balls. As the wear proceeded, the apparent contact area was substantially enlarged, and the local concentration of contact pressure was relaxed. The reduction in the average contact pressure is reflected in the friction behavior.[10] During the sliding test, the water in the ambient air (34% RH) favorably dissociated and passivated the diamond surface.[44] This passivation effect also contributed to the low friction and wear,[10] in addition to carbon phases on the sliding surfaces. From these results, laser polishing improved the friction properties of the polycrystalline diamond films and reduced mechanical damage to the mating material by providing a highly stable equilibrium state of the contact interface.

The mechanism of laser-induced surface flattening for polycrystalline diamond films has been discussed in the literature.[23,33,45,46] The photon energy of a KrF excimer laser (5.0 eV) is lower than the bandgap energy of diamond (5.47 eV). Laser processing involves vaporization or chemical etching via the phase transition of diamond by a photothermal process. The attenuation of the laser-light intensity in the films generally follows the Beer–Lambert law. The optical absorption coefficient for KrF laser light in CVD diamond is on the order of 10^3 cm^{-1},[47,48] which indicates that the laser light is almost absorbed within $\sim 10\,\mu$m. The experimental results obtained in this work reveal that the laser-induced modification of diamond films with a ~ 10-μm thickness proceeds from the film surface in the depth direction, gradually reducing the film thickness. The incident light is efficiently absorbed within a shallow depth in the vicinity of the film surface to modify the surface structure when the laser fluence is higher than the graphitization threshold of diamond.

The optical absorption process probably changed after the formation of amorphous carbon residues on the film surfaces by the phase transition. The laser-induced modification can be divided into two stages: (1) the absorption stage due to the surface texture (which consists of coarse grains); and (2) the absorption stage due to laser-induced absorption near the film surface. In the initial stage, the textured surface morphology plays a substantial role in surface flattening with minimal volume loss of the films. Figure 5 shows a schematic illustration of the laser-polishing mechanism of the polycrystalline diamond films. The surface texture of the as-grown films affects the optical absorption of incident light by[49] (i) reducing the reflectance from the surface by multiple reflections (i.e., an anti-reflection effect), (ii) extending the optical path length, and (iii) optical confinement in the films. In addition, the short wavelength of the incident light increases the refractive index.[50] These effects lead to the penetration of laser beams with low reflection and concentration on the film surfaces [Fig. 5(a)]. Thus, laser beams are efficiently absorbed near the film surfaces (which exhibit asperities structures) by multi-phonon absorption or absorption at defective sites in the bandgap, and the laser energy is converted into thermal energy. The thermal energy preferentially increases the surface temperature, causing the conversion of diamond. This process probably depends on the texture density and periodicity,

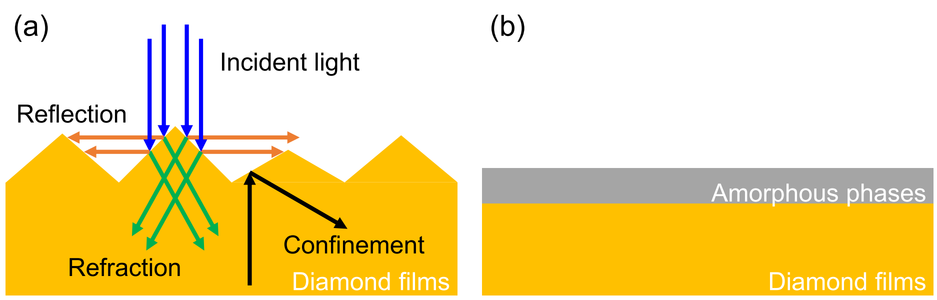

FIG. 5. Schematic representation of the surface-polishing mechanism for polycrystalline diamond films with a KrF excimer laser. (a) As-grown surface and (b) laser-irradiated surface of the films.

which are determined by the grain size and the crystal faces exposed at the film surfaces.

Once surface modification had occurred by laser irradiation, the film surfaces were covered by amorphous carbon residues, which have higher absorption coefficients than diamond [Fig. 5(b)]. The process of laser-induced structural changes (Fig. 3) indicates that the conversion of diamond consistently occurred at the surfaces of the diamond films underneath the amorphous carbon residues. In addition, some absorption centers (including defects and bonding states) appeared to form near the surface of the diamond films during initial surface modification. Interestingly, at a high fluence (e.g., 3.5 J cm^{-2}), transformation of diamond to amorphous phases progressed, and the amorphous residues consistently remained on the surface. At a low fluence (e.g., 2 J cm^{-2}), elimination of amorphous carbon residues was dominant rather than the transformation of diamond. The selective removal of amorphous residues on diamond occurs at much lower fluence than the graphitization threshold.[31] Further investigation is required to verify the specific surface-modification phenomena that depend on the fluence.

In conclusion, we polished polycrystalline diamond films prepared on WC-Co substrates using a KrF excimer laser. Laser irradiation perpendicular to the film surfaces preferentially removed surface asperities and produced a smooth surface (surface roughness: $\approx$0.05 μm). The laser-induced surface modification involved the transition of diamond to amorphous carbon phases, followed by oxidization or evaporation of the amorphous phases. The surface texture that is inherent to the polycrystalline diamond films probably plays a key role in achieving efficient optical absorption, which results in surface flattening. Reciprocal sliding tests revealed that laser polishing imparted a low initial friction coefficient and reduced the wear of the mating materials. These results indicate that a KrF excimer laser (and other lasers with a similar wavelength) can be used to polish diamond coatings with a low loss of film thickness. Designing the irradiation system with optimization of the parameters, such as the photon energy, pulse duration, and ambient atmosphere, is expected to improve the polishing efficiency and provide the diamond surface with desired functional groups. Laser processing overcomes the tribological issues that are attributable to a film's rough surface (and in so doing improves the performance, including wear resistance, lifetime, and machining accuracy of diamond-coated cutting tools) and expands the applications of diamond coatings.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Yuki Katamune: Conceptualization (equal); Data curation (equal); Funding acquisition (equal); Investigation (equal); Writing – original draft (equal); Writing – review & editing (equal). **Kouki Murasawa:** Conceptualization (equal); Data curation (equal); Investigation (equal); Resources (equal). **Tsuyoshi Yoshitake:** Supervision (equal); Writing – review & editing (equal). **Toshifumi Kikuchi:** Investigation (equal). **Kaname Imokawa:** Investigation (equal). **Hiroshi Ikenoue:** Resources (equal); Supervision (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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