AE MONITORING FROM CVD-DIAMOND FILM SUBJECTED TO MICRO-INDENTATION AND PULSE LASER SPALLATION

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Abstract

We monitored AE signals during micro-indentation and pulse laser spallation test of CVD diamond film deposited on sintered SiC, and classified the fracture type by Lamb wave AE analysis. In the indentation test with a Rockwell sphere indenter, we observed Hertzian ring cracks and simultaneously detected AE signals from Mode-I fracture. Crack types were classified from polarity distribution of the first arrival So-mode Lamb waves. Fracture strength of the diamond film was estimated as 7.1 GPa by FEM analysis, using the critical indentation force, indentation depth and Hertzian crack diameter. In the laser spallation test, strong pulse expansion wave was produced by the pulse laser ablation of the confined silicone grease on the opposite surface of the diamond film and was utilized to cause Mode-I delamination of diamond film. Critical tensile interfacial stress to cause the spallation was estimated as 222 MPa. We attempted to detect the spallation initiation by monitoring the in-plane motion of So-mode Lamb wave AE using broadband pinducer mounted on the edge planes. Waveform change due to the Mode-I spallation was detected as high-frequency AE.

Keywords: Diamond film, indentation test, Hertzian ring crack, fracture strength, laser spallation, interfacial strength

1. Introduction

CVD-diamond-coated tools are widely used for machining graphite, ceramics and Al-Si alloys. Wear performance of diamond film as a cutting tool has been demonstrated to be excellent. However, decohesion (fracture) of the film during machining has remained a problem. This is more serious than the antifriction problem of the film. In order to study the fracture mechanism, fracture strength of the film and film/substrate interface must be correctly evaluated.

Indentation fracture test using spherical indenter gives us much useful information about micro-fracture. When this test is applied to hard materials, Hertzian ring crack is caused by the surface tensile stress, as given by the Hertz theory [1]. Fracture strength of ceramic coating has been evaluated using the threshold load to cause the first ring crack and FEM analysis [2,3]. Here, accurate determination of crack initiation load is critical. We monitored progression of micro-cracks during loading using AE system. We classified fracture types by analyzing the polarity distribution of the first arrival So-mode Lamb wave. This AE system is different from the conventional AE system utilized for indentation [4, 5] and scratch test [6, 7].

We developed a pulse laser spallation method to measure the absolute adhesion strength of hard coatings. It utilizes strong shock waves produced by pulse laser breakdown of confined grease or liquid. For the measurement of adhesive strength, scratching and indentation methods have been adopted so far [8 - 10], but could not measure the absolute adhesion strength. The strength estimated by these conventional methods changes depending on the testing conditions, such as the surface roughness, shape and stiffness of the indenter. Furthermore these methods
can hardly be applied to diamond film. Contrary to this, the laser spallation technique [11] can estimate the Mode-I adhesion strength when both the out-of-plane displacement of expansion wave and critical laser energy to cause micro-spallation are correctly measured.

We first conducted an indentation test to CVD-diamond film to estimate the fracture strength of the film, and next estimated the interfacial adhesion strength of the film utilizing the pulse laser spallation technique. AE was successfully utilized for damage detection and fracture mode classification.

2. Experimental

2.1 Indentation test

We prepared polycrystalline diamond films with grain size of 10 to 20 µm deposited on sintered SiC substrate by hot-filament CVD method of 3% methane and 97% hydrogen gas mixture. The films were polished using diamond wheel before the indentation and spallation tests of the diamond films. A diamond film of 35-µm thickness on a 20 x 20 x 5 mm SiC substrate was submitted to the indentation test. For the spallation tests, a diamond film of 78-µm thickness deposited on a 35 x 35 x 5 mm SiC substrate was used.

Experimental setup for the indentation test is shown in Fig. 1. We used a diamond braille indenter with spherical tip (radius: 0.2 mm and angle: 120 degree). Maximum indentation load, $F_{\text{max}}$, were selected at 98, 196, 294, 392 and 490 N. Loading rate and holding time are controlled at 0.49 N/s and 10 s, respectively. Four small AE sensors (PAC, PICO, center frequency: 500 kHz) were mounted on the four edge surfaces of the substrate. Sensor outputs were amplified by 60 dB using a pre-amplifier (NF9913, NF Circuit Co.), digitized by an 8-channel fast A/D converter (Gage CompuScope, CS12100) and fed to a personal computer. Sampling interval and sampling points were set as 50 ns and 2048, respectively.

The AE system monitors the Lamb (or plate) wave AE. We classified fracture modes or crack types by analyzing the polarity distribution or radiation pattern of So-mode Lamb wave. Two types of crack, the Mode-I fracture with a crack opening vector parallel to the surface corresponding to the ring crack, and the Mode-II fracture or lateral crack at the film/substrate interface, were examined. When all polarities of the first arriving So-mode waves are positive,
this is produced by a Mode-I crack. When two opposite polarities are found for the initial AE waveforms among 4-channels of AE signals, this is induced by a Mode-II crack. Detail of polarity distribution analysis can be found elsewhere [12].

2.2 Laser spallation test

Experimental setup for the laser spallation test is shown in Fig. 2. A high-energy pulse from a Q-switched Nd:YAG laser (New Wave Research, Tempest300, maximum energy: 300 mJ, pulse duration time: 3-5 ns, wavelength: 1064 nm) was irradiated on the uncoated surface of the substrate, opposite the diamond film under test. Silicone grease containing fine MoS$_2$ particles was painted on the surface as an energy-absorbing layer. Thickness of the layer was controlled precisely using a thickness gage to 20 µm and confined rigidly by a 6-mm-thick silica plate with anti-reflection coating. Impact wave was excited in the substrate by the laser breakdown of the grease. The beam diameter was controlled to be 1 mm by changing the defocusing distance. Laser energy was changed from 0.7 to 75 mJ. A heterodyne-type laser interferometer (He-Ne laser, 532 nm) was used to measure the out-of-plane displacement of the stress wave at the epicenter of the source on the diamond film. In order to detect the spallation initiation by AE, a small broadband sensor (Valpey Fisher, pinducer) was glued on a side surface of the substrate. Sensor to source distance was kept as 14.5 mm. Leaked laser light was used as a trigger signal for the laser interferometer and AE system. Interferometer and AE signals were digitized and stored in a high-resolution digital oscilloscope at sampling interval of 0.4 ns with sampling points of 8192. Output of the AE sensor (pinducer) was directly fed to the digital oscilloscope.

Fig. 2 Experimental setup for laser spallation and AE monitoring.

3. Results and Discussion

3.1 Indentation test

We observed Hertzian ring crack generation during the indentation test. The number of ring cracks increased with maximum indentation load $F_{\text{max}}$ and expanded outward into area around the first ring crack. Figure 3 shows the indentations produced by the test of $F_{\text{max}}$: 98 N, and the indentation force $F$ vs. indentation depth $h$ with AE timing. We detected frequent AE signals from the Mode-II fracture (open triangles). These AE signals were, however, considered to be the contact noise. Only one Mode-I AE, from the ring crack was detected as indicated by solid triangle at 39.1 N. The number of the Mode-I AE signals increased with $F_{\text{max}}$. Here we focused our attentions on the load to cause the first ring crack in order to evaluate the tensile strength of the diamond film. Table 1 shows the load at which the first Mode-I AE was detected. The load was almost the same, except that (127.4 N) for $F_{\text{max}}= 196$ N. AE signals from the Mode-I ring
crack at the load of 39.1 N for $F_{\text{max}} = 98$ N are shown in Fig. 4. Polarities of first $S_0$ mode waves were all positive, and indicate the initiation of Mode-I Hertzian crack.

Fig. 3 Laser microscopic image of indentation (a) and indentation curve with AE timings (b), for the test of $F_{\text{max}} = 98$ N.

Fig. 4 AE waves from the Mode-I Hertz crack during the indentation test of $F_{\text{max}} = 98$ N.

Next we calculated the film stress at the load of 39.1 N by FEM. Figure 5 (left) shows the axi-symmetric model for the FEM. We calculated the film stress for the indentation depth of 2.7 µm at 39.1 N. Elastic properties of the same CVD-diamond measured by laser ultrasonic technique [13] were used. Figure 5 (right) shows the distribution of radial stress. The maximum tensile stress is computed as 7.1 GPa to the contact radius of 23 µm. This radius is consistent with the measured radius in Fig. 3. Thus the tensile fracture strength of the CVD-diamond film was estimated to be 7.1 GPa.

### 3.2 Laser spallation test

Preliminary test showed that the diamond film suffers spallation at laser energy of around 46 mJ. Figure 6 shows microscopic images of spallated area. Spallation occurred above laser energy of 46.1 mJ. We observed very small delamination of 0.3 mm diameter at 46.1 mJ, though not clear in the photo. Delaminated circle expanded with laser energy. In order to estimate the adhesion strength correctly, we must determine the delamination initiation. Owing to the transparency
of the polished diamond film, we can detect the spallation by eye inspection when a finite size delamination occurs, however, much accurate detection technique is needed for non-transparent and rough surface films.

We are now using two methods. One is waveform analysis of bulk waves detected by the interferometer. Figure 7 compares the outputs of laser interferometer for the case of film spallation (b) and no-spallation (a). Very clear waveform differences exist at the arrival of the first P-waves. Wave oscillation of 28-33 ns period in (a) is due to the multi-reflection of the P-wave (1400 m/s) in the confined grease layer (20 µm thick). This oscillation disappears when spallation occurs. This is because the film spallation was induced by the first P-wave arrival and the subsequent P-waves cannot be transmitted to the delaminated film.

We next estimated the critical interfacial stress to cause spallation. We detected out-of-plane displacement of the stress wave at the epicenter on the substrate (without CVD diamond film). Figure 8 shows the waveforms as a function of laser power from 4.8 to 46.1 mJ. At low energies from 4.8 to 15 mJ, the compressive wave or down-shooting wave is the major component. The
expansion component appears above 35.5 mJ and increases with laser energy. Tensile stress of the expansion component is given by Eq. (1) [14].

\[
\sigma = -\rho V_l \frac{\partial u(t)}{\partial t}
\]  

where \( \rho \) is the density of the medium and \( V_l \) denotes the velocity of the longitudinal wave. \( \rho \) and \( V_l \) of sintered SiC are measured as 3150 kg/m\(^3\) and 11.80 km/s, respectively. The partial differentiation of displacement with respect to time defines the particle velocity of the longitudinal wave. Since the thickness of the diamond film is negligibly small, the same calculation procedures were applied to the diamond-film spallation experiments. Figure 9 shows the stresses calculated for compression (filled circle) and expansion (open circle) waves. Here compressive stress was designated by negative values. Tensile stress increased with increasing the laser energy. Data scattering appears to be due to the difficulty in determination of the particle velocities. Three data points represent the case of spallation. From this, the interfacial adhesive strength of the diamond film is estimated as 222 MPa.
In order to determine the spallation initiation accurately, we utilized AE monitoring. We previously detected the impact–induced cracks in the PMMA hit by flying objects [15]. For such case, weak AE by fracture is hidden by strong impact induced AE and could not be detected by the AE sensor mounted on the plate surface. However, it is feasible to detect the fracture-induced weak AE by monitoring the in-plane motion of the So-Lamb waves using the sensors mounted on the edge plane of the target [15]. We used this technique and detected Lamb-wave AE from the spallation using a broadband (1 to 25 MHz) pinducer mounted on the edge (see Fig. 2).

Figure 10 (top row) shows detected Lamb-wave AE signals as a function of laser energy. In this sample, the diamond film started to spall at 69 mJ, but it did not spall at 67 mJ. Two left waves represent non-spallation cases, while the right two waves are for the spallation. The corresponding frequency spectra of the waves are given in the bottom row. Significant signal
power exists at 2 – 6 MHz. When no spallation occurs, signal power at 13 – 17 MHz is strong. The peak height at 16 MHz is comparable to that at 2.5 MHz for 37 mJ pulse. When the spallation occurs, the high frequency peak diminishes rapidly. At both 69 and 75 mJ tests, 14-MHz peak decreases almost to the noise level. Large reduction of the ratio of signal power at 13 – 17 MHz range to that of 2 – 6 MHz is a good indicator of the presence of spallation.

4. Conclusion

We utilized AE for detecting and classifying the film fractures during Rockwell micro-indentation and laser spallation test of CVD-diamond films deposited on SiC plate. Results are summarized below:

1) Mode-I Hertzian ring crack during Rockwell indentation was detected by advanced AE system utilizing four AE sensors around the indenter. Critical indentation load, indentation depth and ring crack diameter to cause the first Hertz crack were determined and submitted to FEM analysis. Tensile strength of the diamond film was estimated to be 7.1 GPa.

2) Laser spallation technique can determine the absolute interfacial strength of CVD-diamond film on SiC substrate by measuring the fast out-of-plane displacement of the expansion stress wave. Adhesion strength of the test sample was estimated as 222 MPa.

3) In order to detect the spallation initiation, we monitored in-plane motion of the So-mode Lamb wave by broadband small AE sensor (pinducer) mounted on the distal planes. The sensor detected the AE from spallation through the loss of high-frequency (13-17 MHz) components.

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