Quality improvement of deteriorated cutting fluid treated by atmospheric-pressure plasma jet and in-liquid plasma

Junji MIYAMOTO*, Ryo TSUBOI*, Masashi YOSHIDA** and Koichiro NAMBU***

* Department of Mechanical Engineering, Daido University
10-3 Takiharu-cho, Minami-ku, Nagoya, Aichi 457-8530, Japan
E-mail: j-miya@daido-it.ac.jp
** Department of Mechanical Systems Engineering, Daido University
10-3 Takiharu-cho, Minami-ku, Nagoya, Aichi 457-8530, Japan
*** Toyota Technological Institute
2-12-1 Hisakata Tenpaku-ku, Nagoya, Japan

Received: 27 February 2020; Revised: 13 July 2020; Accepted: 17 August 2020

Abstract
In this study, we proposed a sterilization technique for cutting fluids using plasma treatment under atmospheric pressure and in-liquid and investigated the characteristics of sterilization and the fluids’ surface properties (e.g., wear resistance and wettability). The results show that the number of bacterial colonies in the fluid sterilized by atmospheric-pressure plasma and in-liquid plasma was reduced by more than 90% compared with the number in the untreated fluid. The lubricating properties of the plasma-treated cutting fluids were well improved compared with those of the untreated fluid, as determined from a comparison of the results of specific wear rate tests. The adhesive energy of the plasma-treated cutting fluids was greater than that of the untreated fluid, as revealed by the results of sliding angle measurements. However, the adhesive energy decreased over time; that is, the duration of the effect was limited. The results of this study demonstrate that the life of a cutting fluid can be prolonged by plasma treatment, with an associated improvement in the fluid’s tribological properties. This research can help reduce the frequency of maintenance required for coolants used in cutting applications.

Keywords : Cutting fluid, Plasma treatment, Wear, Surface, Microbiological properties

1. Introduction

Friction between a tool and chip, and the heat generated, often limit machining in metal cutting operations. Coolants and lubricants are used in great quantities to reduce friction at the cutting area. However, the cost of cutting fluids is increasing because of the increasingly stringent environmental standards for fluid handling and disposal, and these standards are likely to be further tightened in proposed national and international legislation (Weinert and Inasaki, 2004). Although dry and semi-dry machining is an attractive option, it is currently unavailable in many operations because cooling cannot be achieved (Tasdelen et al., 2008; Werda et al., 2016). Therefore, cutting fluids will continue to be required.

As a fluid deteriorates, its cutting power decreases and the work environment becomes polluted. Techniques that extend the life of cutting fluids are therefore needed. One of the key causes of deterioration is decomposition due to bacterial growth (Rabenstein et al., 2009; Griffiths, 1978). Bacteria enter the fluid from both the air and the chip. Because preventing bacteria from contaminating the cutting fluid is difficult, chemical additives are widely used for bacterial control. The widespread use of oil additives has led to the emergence of resistant bacteria, and the additives change the characteristics of the cutting fluid. Therefore, we proposed a sterilization technique using plasma treatment under atmospheric pressure (Miyamoto et al., 2018). Moreover, we suggested that the molecular structure of the fluids was not affected by plasma jet treatment (Miyamoto et al., 2018). In our previous study, the number of bacterial colonies in fluid sterilized using atmospheric-pressure plasma was reduced by more than 90% compared with the number in an untreated fluid (Miyamoto et al., 2018). From this report, however, it is unclear whether the remaining bacteria and surface...
characteristics such as tribological characteristics of the cutting fluid sterilized by atmospheric-pressure plasma change over time. That is, the duration of the atmospheric-pressure plasma treatment effect and surface characteristics such as tribological characteristics remains poorly understood. Therefore, in the present work, we investigated whether the remaining bacteria and characteristics of the cutting fluid sterilized by atmospheric-pressure plasma vary over time. Moreover, because the treatment time of atmospheric-pressure plasma is long, we propose a sterilization technique based on in-fluid plasma treatment as a more rapid treatment method. Specifically, we investigated the state of the cutting fluid over time after atmospheric-pressure plasma treatment and proposed a method for the plasma sterilization of cutting fluid using in-liquid plasma. The excited species in the in-liquid plasma were investigated spectroscopically to investigate the mechanism of sterilization.

2. Experimental apparatus and methods

2.1 Cutting fluids and experimental apparatus

Commercial water-soluble mineral cutting oil (JIS K2241 Class A1) with an added surfactant was used as the test fluid. The density of the fluid before use was 0.913 g/cm$^3$ at 15 °C, and its pH was 9.3. The cutting oil used for plasma sterilization was similar to that used for actual processing. For use as cutting oil, the fluid was diluted tenfold with water and then used to machine JIS A5056 malleable aluminum alloy and JIS S45C steel (cutting speed 80–150 m/min, feed 0.1–0.3 mm/rev., depth of cut 0.5–2 mm). The cutting oil was extracted at the same time under all experimental conditions so that the properties of plasma sterilization and the cutting oil remained unaffected.

In this research, the jet and in-liquid methods of plasma sterilization were used. The experimental apparatus used for the plasma jet treatment is shown in Fig. 1. Plasma was generated by a pulsed voltage biased between two electrodes (one outside the glass tube and the other inside it). The cutting fluids were stirred during treatment to ensure that bacteria near the bottom of the fluid were exposed to the plasma. The glass tube was forked to allow the flow of argon and nitrogen gases. During testing, the fluid was isolated in an acrylic chamber to prevent exposure to atmospheric oxygen and limit bacterial growth.

The experimental apparatus for the in-liquid plasma treatment is shown in Fig. 2. Plasma was generated by a pulsed voltage biased between electrodes in the cutting fluid. Bubbles were generated in the cutting fluid under the applied voltage. Plasma was assumed to be generated in the bubbles (Shiraishi et al., 2018; Rahim et al., 2015; Syahrial et al., 2015).

Photographs of the plasma treatment via the jet and in-liquid methods are shown in Fig. 3, where Fig. 3 (a) shows the plasma jet treatment and Fig. 3 (b) shows the in-liquid plasma treatment. In all cases, the cutting fluid was irradiated with plasma. However, the emission color of the plasma differed between the two treatments. The emission spectrum of plasma depends on the excited species in the plasma. This observation of different emission colors indicates that the excited species may differ between the plasma jet and the in-liquid plasma.

2.2 Experimental methods

The experimental conditions used in the atmospheric-pressure plasma treatment are given in Table 1. To generate plasma, the electrodes must be negatively or positively biased. In our experiments, bipolar pulses of $V_{pp} = 6$ kV were used. The test fluids were exposed to the plasma jet for 45 min. The fluid was maintained at room temperature while being irradiated. The flow rates of argon and nitrogen were 10 and 0.2 slm, respectively. A distance of 8 mm was maintained between the nozzle tip and the liquid. The number of bacterial colonies was found to increase in fluids treated without isolation in the acrylic chamber and oxygen purging; this colony growth was attributed to the presence of facultative anaerobic bacteria, which can grow when exposed to oxygen (Madanchi et al., 2017; Voloski et al., 2016). We decomposed these additives, such as oiliness and extreme pressure agents, by the propagation of the bacteria, which affects the tribological properties and cutting oil rots. The residual oxygen was therefore purged using argon gas at 10 slm for 10 min to prevent bacterial growth prior to the plasma treatment.

The experimental conditions for the in-liquid plasma treatment are given in Table 2. To generate plasma, bipolar pulses of $3 \leq V_{pp} \leq 6$ kV were used. The frequency of the bipolar pulses was 10, 20, or 30 kHz. The pulse width of the
bipolar pulses was 1.2 μs. The test fluids were exposed to the plasma for 10 or 35 s. The electrode material for generating plasma was tungsten, and a distance of 1 mm was maintained between the electrodes. The number of colonies in the plasma-sterilized fluid was measured using film culture medium and an incubator. For culturing, 1 mL of the fluid was diluted 1000- and 10,000-fold with water. The culturing time was 48 h. Excited molecular, atomic, and ionic species were identified by spectroscopic observation of the plasma generated to investigate the plasma sterilization mechanism of bacteria in the cutting fluids. In the case of the atmospheric-pressure treatment, the emissions from the plasma were observed at a distance of approximately 2 mm from the center of the glass tube. In the case of the in-liquid plasma treatment, the emissions from the plasma could not be observed because plasma was generated in the cutting fluid. Therefore, the emissions were observed in a quartz tube inserted into the cutting fluid.

The friction coefficient of the cutting fluids was investigated using a rotating tribometer. The measurement results of the friction coefficient are shown in the previous report (Miyamoto et al., 2018). The sample material was SKH51 (JIS G 4403) tool steel with a diameter of 30 mm and thickness of 10 mm. SKH51 was selected because it is a widely used cutting material. The hardness of the samples was 360 HV, and the surfaces of the samples were ground and polished successively using silicon carbide abrasive paper and alumina powder. The friction coefficient of the samples was investigated using 10 mm SUJ2 (JIS G 4805; hardness 850 HV) balls in a lubricant atmosphere. SUJ2 was selected because it is the most widely used material in friction tests. The cutting fluids were supplied at 70 μL as the lubrication condition. The applied load was 98.1 N, and the samples were rubbed for 5 min at 10 rpm at a radius of 8 mm and a room temperature. The average contact pressure $P_{\text{mean}}$ before wear was calculated to be 1.46 GPa. We investigated the specific wear rate by measuring wear track after the friction test using a surface roughness meter.

Contact angle measurements on the surfaces of all of the samples were carried out using a contact angle measuring instrument. Each metal sample was mounted onto the adjustable stage of the measuring instrument. A drop of cutting fluid was delivered via needle as the wetting liquid onto the surface of the metal sample at room temperature. An optic device equipped with a high-speed digital video camera was used to monitor the dropped cutting fluid. In the experiment, 10 μL droplets of the cutting fluids were used, and the metal sample material was SUJ2 to achieve the same conditions as those in the friction tests. The surface roughness parameters of the samples were $R_a = 0.03 \, \mu m$ and $R_z = 0.18 \, \mu m$. The samples were ultrasonically cleaned in acetone for 5 min and were then immediately placed on the stage for measurement.

The adhesive energy of each cutting fluid was investigated using the sliding method. This method is used to measure the angle at which a droplet dropped onto a sample flows downward. The liquids used in the measurements were unused,
untreated, and plasma-treated cutting fluids. The sliding angle was obtained from a side-view photograph captured by a high-speed camera.

3. Results and discussion

Fig. 4 shows photographs of the film culture medium after culturing with untreated and atmospheric-pressure-plasma-treated cutting fluids. Fig. 4 (a) shows the untreated fluid, and Figs. 4 (b), (c), and (d) show the fluid immediately after treatment, 1 week later, and 2 weeks later, respectively. Bacteria in the unused cutting fluid were not detected as a result of examination. The cutting fluids observed immediately after treatment with atmospheric-pressure plasma appeared to contain a smaller number of bacterial colonies than the untreated fluid. Here “unused” denotes fluid that had never been used for cutting, and “untreated” indicates the fluid used for sustained cutting of aluminum but not plasma-treated. However, the number of colonies in the cutting fluid treated with atmospheric-pressure plasma increased over time. Under all of the investigated test conditions, the colonies spread on the culture medium. These results are attributed to the bacteria in the cutting fluids.

Fig. 5 shows the colony numbers in the cutting fluids treated with atmospheric-pressure plasma. The colony forming unit (CFU) is the number of colonies produced when bacteria are cultured in a medium. A substantial decrease in the number of colonies was observed in the fluids cultured immediately after plasma treatment. A CFU decrease greater than 90% was observed in fluids treated with plasma. These results suggest that the plasma treatment achieved sterilization within a short time. However, the number of colonies tended to increase with time after the plasma treatment. The number of colonies in the cutting fluid 2 weeks after treatment had increased to the same level as in the untreated fluid. The plasma-treated cutting fluid was preserved at room temperature (20–30°C) to simulate the conditions in actual industrial sites. Therefore, this result was speculatively attributed to multiplication of surviving bacteria in the cutting fluid.

Table 1: Experimental conditions for the atmospheric-pressure plasma treatment

| Parameter                        | Value       |
|---------------------------------|-------------|
| Discharge voltage [kV]          | 6           |
| Treatment time [min]            | 45          |
| Treatment temperature           | Room temperature |
| Mass flow rate [slm]            | Ar : 10, N₂ : 0.20 |
| Distance from nozzle [mm]       | 8           |
| Amount of cutting fluid [mL]    | 20          |
| Rotation speed [rpm]            | 200         |

Table 2: Experimental conditions for the in-liquid plasma treatment

| Parameter                        | Value       |
|---------------------------------|-------------|
| Discharge voltage [kV]          | 3–6         |
| Frequency [kHz]                 | 10, 20, 30  |
| Pulse width [μs]                | 1.2         |
| Distance between electrodes [mm] | 1           |
| Amount of cutting fluid [mL]    | 20          |
| Treatment time [s]              | 10, 35      |

Fig. 4 Photographs of the film medium (atmospheric-pressure plasma)

(a) Untreated
(b) Immediately
(c) 1 week later
(d) 2 week later

Fig. 5 Number of colonies in the fluids treated with atmospheric-pressure plasma
results indicate that periodic treatment is necessary to maintain low colony numbers when cultivation conditions are established.

Fig. 6 shows the optical emission spectra of the plasma generated using the atmospheric-pressure plasma jet. The spectra show the emissions of the N\(_2\) second positive system, the N\(_2\) first positive system, OH (309 nm), NO (236 nm), and Ar. The spectral peaks of positive ions (N\(_2^+\), N\(^+\), etc.) and N atoms reported for conventional low-pressure plasma treatments were not detected (Nagamatsu et al., 2013). The absence of these species is attributed to recombination processes arising from the extremely short mean free path under atmospheric pressure. For this reason, the dielectric barrier discharge plasma sterilization process investigated in the present work may produce species such as excited nitrogen rather than charged particles such as the ions produced in low-pressure plasma. Ultraviolet (UV) irradiation has been used to sterilize medical instruments for more than 60 years (Putt et al., 2012; Zhao et al., 2010). The UV light generated by the plasma may therefore play an additional role in sterilization. As a result of examining the influence of UV sterilization, we found that UV rays do not affect sterilization under the experimental conditions used in the present work (Miyamoto et al., 2018). We concluded that the sterilization observed in the study was caused by the reaction of excited N\(_2\), NO, OH, and Ar.

Fig. 7 shows photographs of the film culture medium after culturing with untreated and in-liquid plasma treated cutting fluids. Fig. 7 (a) shows the untreated fluid, and Figs. 7 (b), (c), and (d) show the fluids treated at 10, 20, and 40 kHz for 35 s, respectively. The cutting fluids treated under all conditions contain a smaller number of bacterial colonies than the untreated fluid. The number of colonies in the cutting fluid treated with in-liquid plasma appears to be smaller than that in the cutting fluid treated with atmospheric-pressure plasma.

Fig. 8 shows the colony numbers in the cutting fluids treated with in-liquid plasma. A substantial decrease in the number of colonies was observed in the fluids treated under all conditions. A decrease greater than 90% was observed in fluids treated for 10 s. These results suggest that the in-liquid plasma treatment achieved sterilization substantially faster than the atmospheric-pressure plasma treatment. This rapid sterilization is attributed to the effects of heat in addition to the effects of direct plasma irradiation of the cutting fluid. Bubbles were generated in the cutting fluid, indicating that the cutting fluid in which in-liquid plasma was generated reached a high temperature.

Fig. 9 shows the optical emission spectrum of the in-liquid plasma. The spectrum shows emissions of OH (309 nm), CH (431 nm), H\(_\beta\) (486 nm), C\(_2\) Swan band (440–620 nm), Na (330 nm, 569 nm, and 589 nm), O\(_2\) (637 nm), H\(_\alpha\) (656 nm), K (766 nm and 770 nm), and O (777 nm). The sodium and potassium signals were assumed to originate from the surfactant additives in the cutting fluid. In the untreated cutting fluid, water and oil were separated by demulsification; by contrast, the plasma-treated cutting fluid was emulsified. We attribute this difference to the presence of sodium and potassium. Moreover, the detection of C\(_2\), OH, O, O\(_2\), and H indicates that water and oil were decomposed by the plasma.

Fig. 10 shows the specific wear rates of SKH51 samples tested under cutting-fluid lubrication. The specific wear rate of SKH51 with unused (fluid that had never been used for cutting) and untreated cutting fluids were 2.2 × 10\(^{-6}\) and 3.7 × 10\(^{-6}\) mm\(^3\)/Nm, respectively. However, the specific wear rates of the SKH51 tested with cutting fluid subjected to
atmospheric-pressure and in-liquid plasma treatments were $3.25 \times 10^{-6}$ and $3.1 \times 10^{-6}$ mm$^3$/N·m, respectively. The wear resistance of the samples prepared using cutting fluids treated with atmospheric-pressure plasma and in-liquid plasma improved in magnitude compared with that of the sample prepared using untreated cutting fluid. If the specific wear rate is high, the tool life is shortened. The results presented here suggest that sterilization by plasma treatment is an effective approach to improving the tribological properties of a cutting fluid. From the results in Fig. 9, we are considered that part of the oil was decomposed by the plasma. The wear resistance of the samples prepared using cutting fluids treated with plasma improved compared with that of the sample prepared using untreated cutting fluid. This effect could be attributed to differences in the resulting emulsion in both cases.

Fig. 11 shows photographs of cutting fluid dropped onto the sample surface. Figs. 11 (a) and (b) show the unused fluid and the untreated fluid, respectively. Figs. 11 (c) and (d) show the fluids subjected to atmospheric-pressure plasma and in-liquid plasma treatments, respectively. The contact angle of the unused cutting fluid and the untreated cutting fluid was 37° and 31°, respectively. However, the contact angle of the cutting fluid treated with atmospheric-pressure plasma (47°) and that of the fluid treated with in-liquid plasma (45°) were greater than the contact angles of the unused and untreated fluids. The cutting oil used contains additives like surfactants and oiliness agents. We considered that these additives are decomposed by the propagation of bacteria, which affects the wettability and water sliding property.

Fig. 12 shows the sliding angle of each cutting fluid. The sliding angle of the unused and untreated cutting fluids was 28° and 9°, respectively. The results show that the adhesive energy of the untreated fluid was lower than that of the unused fluid. A low sliding angle implies that the liquid flows easily on the surface; i.e., the cutting fluid film thickness tends to easily decrease because of the outflow between the ball and the disc. Therefore, this result was attributed to the ease of decrease in the oil film thickness between the ball and the sample. The sliding angle of the plasma-treated cutting fluid was greater than that of the untreated cutting fluid. Cutting fluids with added functional groups tend to remain on
the disk surface by bonding with it; a decrease of the specific wear rate is assumed to be caused by a difficult cutting fluid outflow between the ball and disc as a result of these modifications. Therefore, more plasma-treated cutting fluid likely remained between the ball and the disk compared with the amount of remaining untreated cutting fluid, thereby resulting in a lower specific wear rate. Thus, we attribute the lower specific wear rate observed with the plasma-treated cutting fluid compared with that observed with the untreated cutting fluid to the plasma-treated cutting fluid’s greater adhesive energy.

Fig. 13 shows photographs of the plasma-treated cutting fluids dropped onto a sample surface. Figs. 13 (a) and (b) show photographs of the cutting fluid immediately and 1 month after treatment, respectively. The contact angle of the cutting fluid immediately after plasma treatment was 47°; however, the contact angle 1 month after the plasma treatment decreased to 28°. This decrease in contact angle is attributed to the remaining bacteria in the treated cutting fluids proliferating or reacting with oxygen in the air.

Fig. 14 shows the sliding angle of each cutting fluid. As previously mentioned, the sliding angles of the unused and untreated cutting fluids were 28° and 9°, respectively. Moreover, the sliding angle of the cutting fluid immediately after treatment was 20°. However, the sliding angle of cutting fluid 1 month after treatment was 6°, or approximately the same as that of the untreated cutting fluid. This result shows that the effect of plasma treatment on the cutting fluid is time-limited.

4. Conclusions

We proposed a cutting-fluid sterilization technique using plasma treatment under atmospheric pressure and in-liquid and investigated the fluid’s characteristics of sterilization and its the tribological properties (i.e., lubrication and wettability). From the results, the following conclusions were drawn:

1. Substantially fewer bacterial colonies were observed in the cutting fluids treated by in-liquid plasma. After treatment for 10 s, the number of colonies decreased by more than 90%.
2. The number of colonies in the fluid sterilized by atmospheric-pressure plasma increased over time.
3. The spectra of the atmospheric-pressure plasma jet showed characteristic emissions of the \( \text{N}_2 \) first positive system, \( \text{OH} \), \( \text{NO} \), and \( \text{Ar} \). The spectra of the in-liquid plasma showed the emissions of the \( \text{OH} \), \( \text{CH} \), \( \text{H\beta} \), the \( \text{C}_2 \) Swan band, \( \text{Na} \), \( \text{O}_2 \), \( \text{H\alpha} \), \( \text{K} \), and \( \text{O} \).
4. The contact angles of the cutting fluid treated by atmospheric-pressure plasma and in-liquid plasma was increased compared to those of the unused and untreated samples.
5. The sliding angle of the untreated cutting fluid was substantially lower than that of the unused fluid. Furthermore, the sliding angle of the plasma-treated cutting fluid was greater than that of the untreated fluid. The sliding angle of the fluid sterilized by atmospheric-pressure plasma decreased over time.

We conclude that the sterilization effect in this research was caused by the reaction of excited \( \text{N}_2 \), \( \text{N} \), \( \text{OH} \), \( \text{NO} \), and \( \text{Ar} \). The study demonstrated that the life of a cutting fluid can be prolonged by plasma treatment, with an associated
improvement in tribological properties. The results of this research can help reduce the frequency of maintenance required for coolants used in cutting applications compared to the conventional methods.

Acknowledgments

This work was financially supported by a research grant from the Toukai Foundation for Technology.

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