Fabrication of tungsten micro-rods using electrolyte jet turning method

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Abstract

The electrolyte jet turning (EJT) method was used to fabricate tungsten micro-rods with the neutral sodium nitrate (NaNO₃) aqueous solution as electrolyte. Since the non-conductive oxide film generated on the surface of tungsten interrupts the electrochemical dissolution process, a bipolar current was needed to remove the oxide film during machining. With the bipolar current, sodium hydroxide (NaOH) is generated, when the polarity of the tungsten electrode is negative, enabling the removal of oxide film. A cylindrical nozzle was used for the EJT to jet electrolyte to the surface of workpiece which was rotated with a rotation speed of 3000 rpm. The inner diameter of the cylindrical nozzle determined the diameter of the electrolyte jet, and its influence on the machining characteristics was investigated first. Then, the influences of the duty factor and frequency of pulse voltage on the machining process were investigated to improve the machining accuracy. Finally, tungsten micro-rods with high aspect ratio were fabricated by translating the electrolyte jet on the surface of rod workpiece.

Key words: Electrochemical machining; Electrolyte jet turning; Tungsten micro-rods; Micromachining.

1. Introduction

Electrolyte drilling technique was proposed by Ippolito et al.¹ to produce the holes in diesel motor’s injection valves. The hole diameters of 0.2 mm to 0.5 mm were designed to be drilled on heat-treated steel walls with the thickness of 0.7 mm. With this method, the workpiece is machined only in the area which is hit by the electrolyte jet because of the electrical current supplied through the electrolyte jet. Intricate patterns were machined successfully by translating the electrolyte jet on the workpiece surface with the electrolyte jet machining (EJM) method⁵. Moreover, mirror-like finishing surface was obtained by reciprocating the jet at a high translating speed⁶.

The ECM method can machine many of conductive materials regardless of the hardness. However, strongly passivating materials such as cemented tungsten carbide are difficult to be machined by ECM using neutral electrolyte because of the non-conductive oxide film, which hinders the electrochemical dissolution, generated on the workpiece surface. Hence, cemented tungsten carbide is conventionally machined using an alkaline electrolyte, such as NaOH and KOH aqueous solution, which are toxic and not environment friendly. However, Maeda et al.⁴ reported that when cemented tungsten carbide was machined with neutral electrolyte NaNO₃ aqueous solution using a bipolar current, NaOH was generated, when tungsten carbide electrode was in negative polarity, enabling to remove the oxide film on the surface of tungsten carbide. Then, Miyoshi and Kunieda⁵ developed the electrolyte jet turning (EJT) system to machine cemented tungsten carbide rods using the neutral electrolyte of NaNO₃ aqueous solution and bipolar current, and a micro-rod of 36 μm in diameter and aspect ratio of 20 was machined successfully. Han and Kunieda⁶ machined pure tungsten micro-rod with a minimum diameter of 7 μm using the neutral electrolyte based on the same mechanism proposed by Maeda et al.⁴.

In this research, tungsten micro-rods were machined with the EJT method using neutral electrolyte NaNO₃ aqueous solution. A cylindrical nozzle made of stainless steel was used to jet the electrolyte to the surface of workpiece. The shape of electrolyte jet is kept constant during machining although cylindrical nozzle is worn during machining due to the bipolar current, which is an advantage compared with the slit nozzle previously used by Miyoshi and Kunieda⁷ because the dimension of the flat electrolyte jet made by the slit nozzle is changed due to the wear of the nozzle. Fig. 1 shows the worn slit nozzle which indicates that the shape of the flat electrolyte jet is to be changed due to the wear. In addition, a higher material
removal rate with the cylindrical nozzle can be easily obtained by increasing the inner diameter of cylindrical nozzle compared with the slit nozzle due to the larger contact area between the electrolyte jet and workpiece surface. Hence, the machining results with different inner diameters of nozzle were investigated first. Then the influences of the duty factor and frequency of pulse voltage on the machining characteristics were studied to optimize the machining conditions. According to the above experimental results, tungsten micro-rods with high aspect ratio were fabricated by translating the electrolyte jet on the surface of rod workpiece.

2. Electrolyte jet turning (EJT) equipment

Fig. 2 shows the EJT method. The pulse power supply is coupled to the gap between the nozzle and workpiece. The electrolyte jetted from a nozzle hits the surface of rod workpiece which rotates with a high speed. The current distribution is localized under the electrolyte jet, thereby removing the material only there. The electrolyte jet can be translated on the surface of workpiece to generate any intricate patterns. When machining micro-rods, the feed distance determines the machined length of micro-rod, and the diameter of the micro-rod will be determined by the machining parameters such as the machining time, translating speed, and current density. The details of the EJT equipment used in this study can be found in the previous research by Miyoshi and Kunieda.

3. Influence of inner diameter of cylindrical nozzle

3.1 Experimental method

It is difficult to machine tungsten carbide by ECM because of the passive oxide film generated on workpiece surface. Maeda et al. successfully machined cemented tungsten carbide using the bipolar current and neutral electrolyte of NaNO₃ aqueous solution. The following electrochemical reaction occurs when the tungsten carbide electrode is negative.

\[ 2Na^+ + 2H_2O + 2e^- \rightarrow 2NaOH + H_2 \]  

Because of the NaOH, the tungsten oxide (WO₃), which is generated on the surface of tungsten carbide workpiece, can be dissolved as,

\[ WO_3 + 2NaOH \rightarrow Na_2W_3O_9 + H_2O \]  

Han and Kunieda verified that pure tungsten could also be machined based on the same principle above.

In this study, commercial needle nozzles (Musashi Engineering, INC) were used as the cylindrical nozzle. A pulse voltage generated by a function generator (Agilent, 33250A) was amplified by a bipolar amplifier (NF Corporation, HSA4101) to work as the pulse voltage supply. Bipolar pulse voltage was used with the total amplitude of 100 V and frequency of 1000 Hz and the duty factor was 50%. The workpiece was tungsten rod with the original diameter of 300 μm. The electrolyte was NaNO₃ aqueous solution of 20 wt% in concentration. The initial gap width between the nozzle and workpiece surface was 1 mm. The initial position of the electrolyte jet with respect to the rod workpiece is shown in Fig. 3. The diameter of electrolyte jet depends on the inner diameter of the used cylindrical nozzle. To investigate the influence of the inner diameter on the machining characteristics, four cylindrical nozzles with different inner diameters were used as listed in Table 1. The total machining time was 20 s and the position of electrolyte jet was fixed without translating.

Table 1 Inner and outer diameters of nozzle.

| Nozzle type | A | B | C | D |
|-------------|---|---|---|---|
| Inner diameter [mm] | 0.14 | 0.21 | 0.42 | 1.43 |
| Outer diameter [mm] | 0.32 | 0.42 | 0.72 | 1.83 |

Fig. 3 Initial position of electrolyte jet with respect to rod workpiece.

3.2 Experimental results

Fig. 4 shows the waveforms of the gap current and voltage with inner diameters of 0.14 mm and 0.21 mm. The gap current increased with increasing the inner diameter of cylindrical nozzle because the gap resistance was decreased with the larger diameter of electrolyte jet and constant gap voltage control. It is noted that the current decreased during the positive pulse duration, which is presumably caused by the increase in the thickness of oxide film generated on the workpiece surface, resulting in a higher gap resistance. The oxide film was removed by the NaOH generated in the negative
pulse, although it was generated again during the positive pulse duration.

Fig. 5 shows the micro-rods machined for 20 s with different inner diameters of cylindrical nozzles. The material removal volume increased with increasing the inner diameter of cylindrical nozzle because of the higher current under the constant voltage as shown in Fig. 4. In addition, when the inner diameter was 1.43 mm, the workpiece was shortened because the position of the jet centre from the end of the workpiece was shorter than the radius of the electrolyte jet, leading to the concentration of the current at the end of the workpiece.

Thus, using the inner diameter of 1.43 mm, the machining time was shortened. Fig. 6 shows the experimental results with the machining time of 5 s and 15 s. The significantly smooth surfaces of the micro-rod show the effectiveness of the bipolar current. Moreover, the side surfaces of the micro-rod were straight compared with Fig. 5(d), especially with the machining time of 5 s.

Fig. 5 Micro-rods machined for 20 s with different inner diameters of (a) 0.14 mm (b) 0.21 mm (c) 0.42 mm and (d) 1.43 mm.

4. Influence of duty factor of pulse voltage

4.1 Experimental method

The duty factor was increased from 5 % to 80 % with the corresponding machining time decreased inversely from 200 s to 12.5 s to keep the anodic dissolution time constant at 10 s. Since the pulse period was kept constant at 1 ms, the ratio of positive to negative pulse duration in Fig. 7 was changed with the different duty factors. A cylindrical nozzle with the inner diameter of 0.42 mm was used and the initial gap width between the nozzle and workpiece surface was changed from 0.1 mm to 1.0 mm. The initial position of the electrolyte jet with respect to the rod workpiece was the same as shown in Fig. 3.

Duty factor = \frac{t_a}{t_a + t_b} \times 100\%

Fig. 7 Definition of duty factor of pulse voltage.

4.2 Experimental results

Fig. 8 shows the machining results with different gap widths when the duty factor was 50 %. The material removal volume increased with decreasing the gap width because the current density increased due to the smaller gap resistance, resulting in a higher material removal rate.

Fig. 6 Micro-rods machined with different machining time of (a) 5 s and (b) 15 s. The inner diameter of nozzle was 1.43 mm.

Fig. 4 Waveforms of gap current and voltage with different inner diameters of nozzles.
Fig. 8 Machining results with different gap widths of (a) 1 mm, (b) 0.5 mm, (c) 0.2 mm and (d) 0.1 mm.

Therefore, a small gap width of 0.1 mm was used for the following experiments.

Fig. 9 shows the waveforms of gap current and voltage with the duty factors of 10 %, 40 % and 80 %. It is considered that NaOH was generated when the polarity of tungsten electrode was in negative and removed the oxide film. However, the oxide film was generated again when the tungsten electrode was positive. The current continuously decreased with increasing the positive pulse duration, because the oxide film thickness increased with the passage of time.

Fig. 10 shows the machining results with different duty factors of 5 %, 40 % and 80 %. Fig. 11 shows the material removal volumes with different duty factors. With a lower duty factor, the material removal volume was low. The positive pulse current is first used to complete the charging of the electric double layers formed on the surface of electrodes, and after that used for the faradaic current for the anodic dissolution reactions. Hence, the electrochemical reactions cannot occur until the electric double layers are fully developed in the small electrode gap. Therefore, the fraction of the faradaic current is reduced with decreasing the duty factor, resulting in decreased material removal volume. On the other hand, it is noted that the material removal volume decreased with increasing the duty factor over 50 %. This is because the thickness of oxide film became thicker with increasing the positive pulse duration, resulting in a decrease in the current as shown in Fig. 9. Hence, the optimal duty factor was 50 % to obtain the maximum material removal volume.

Fig. 10 Machining results with different duty factors of (a) 5 %, (b) 40 % and (c) 80 %.

Fig. 11 Material removal volumes with different duty factors.

Fig. 12 shows the used cylindrical nozzle. It was shortened after machining because the cylindrical nozzle was electrochemically dissolved when its polarity was in positive.
Fig. 12 Nozzle wear due to bipolar pulse current.

5. Influence of frequency of pulse voltage

5.1 Experimental method
The frequencies were 100 Hz, 2 kHz, 20 kHz and 100 kHz with the duty factor of 50%. The total anodic dissolution time was the same at 20 s for different frequencies. The position of electrolyte jet with respect to the rod workpiece was fixed as shown in Fig. 3 during the machining process.

5.2 Experimental results
Fig. 13 shows the waveforms of gap current and voltage with different frequencies of 100 Hz, 2 kHz and 100 kHz. The gap current increased with increasing the frequency because the thickness of oxide film generated in the positive pulse duration became thinner with increasing the frequency.

Fig. 13 Waveforms of gap current and voltage with different frequencies.

Fig. 14 shows the machining results with the different frequencies. With the lowest frequency of 100 Hz, the long duration of positive pulse resulted in a thick oxide film and low current density. Therefore, the material removal volume was significantly small. With the highest frequency of 100 kHz, the material removal volume was also low because of the influence of the concentration boundary layer formed on the interface between the tungsten surface and bulk electrolyte. A concentration boundary layer is formed due to the diffusion of dissolved metal ions and electrolyte ions on the anode surface, and a specific time is needed to obtain the fully developed boundary layer\(^9\). Furthermore, it takes some time for the electric double layer to be formed on the electrode surfaces. This would also decrease the material removal rate because start of the faradaic reaction is delayed. Hence, the material removal volume peaked at around 1 kHz, as shown in Fig. 15.

Fig. 14 Machining results with different frequencies of pulse voltage.

Fig. 15 Material removal volumes with different frequencies of pulse voltage.

6. Micro-rods machining by translating electrolyte jet

6.1 Experimental method
To machine micro-rods with high aspect ratios,
the electrolyte jet was translated on the surface of tungsten workpiece. The initial position of the electrolyte jet with respect to the rod workpiece is shown in Fig. 16. The jet was translated twice along the rod axis with the translation distance of 5 mm. According to the above experimental results, the optimized frequency of 1 kHz and duty factor of 50% were used due to the higher material removal rate. The total amplitudes of the bipolar voltage were 100 V and 40 V, which were used for the first and second translating process, respectively. The voltage was decreased in the second translating process because the machined rod was easily shortened in the second translating process due to the stray current flowing through the end of the rod while the end is penetrating the jet. The inner diameter of the cylindrical nozzle was changed as 1.43 mm and 0.42 mm to investigate the influence of the jet diameter on the machinability of micro-rods with higher aspect ratio. With the inner diameter of 1.43 mm, the translating speeds of the jet were 0.2 mm/s and 0.1 mm/s for the first and second translating process, respectively. With the inner diameter of 0.42 mm, the translating speeds were 0.15 mm/s and 0.1 mm/s in the first and second translating process, respectively. Since the contact area of the jet with the workpiece was reduced with decreasing the inner diameter of cylindrical nozzle, the translating speed in the rough machining was slightly reduced to 0.15 mm/s compared with the inner diameter of 1.43 mm to get a comparable material removal volume in the first translating process.

Fig. 16 Initial position of electrolyte jet with respect to rod workpiece.

6.2 Experimental results

Fig. 17 shows a micro-rod machined using the inner diameter of 1.43 mm. The surface was significantly smooth owing to the electrochemical dissolution. The average diameter of micro-rod was 85 μm and the aspect ratio was about 530. A large taper was formed at the end of the micro-rod as shown in Fig. 17(c) because the current density was higher in the vicinity of the rod end due to the stray current when the rod end is penetrating the electrolyte jet as shown in Fig. 18. However, the influence of the stray current on the end could be decreased by decreasing the inner diameter of cylindrical nozzle. Fig. 19 shows the micro-rod machined with the inner diameter of 0.42 mm, and it is found that the tapered rod length was shorter compared with Fig. 17. This is because the influence of the stray current disappears after the rod end has penetrated the electrolyte jet, reaching a steady state. Moreover, it is noted that the rod in section (a) was tapered as shown in Fig. 17 and Fig. 19. This is because the machining time is not uniform in section (a), of which the length is equivalent to the jet diameter.
7. Conclusions

Tungsten micro-rods were machined with the electrolyte jet turning (EJT) method using a neutral electrolyte of NaNO₃ aqueous solution and bipolar current. A cylindrical nozzle was used to jet the electrolyte perpendicularly to the rod workpiece so that both axes intersect each other. The gap width between the nozzle and workpiece was adjusted manually and switching on/off the machining power supply was synchronized with the translating motion of the electrolyte jet along the axis of the rod. The diameter of micro-rods machined could be controlled by changing the machining time and the amplitude of the bipolar voltage. The following conclusions were obtained.

1) The material removal rate was increased with increasing the inner diameter of cylindrical nozzle because the current was increased with the increased machining area. However, the machining accuracy was reduced with increasing the jet diameter when the end of the rod was inside the jet because the distribution of current density was not uniform along the rod.

2) The current density was influenced obviously by changing the electrode gap widths under the constant voltage condition. The cylindrical nozzle was worn due to the bipolar pulse current because it was dissolved when the nozzle was in positive polarity.

3) There was an optimal duty factor of the bipolar pulse to obtain the maximum material removal rate. With a low duty factor, the percentage of the faradaic current was decreased with decreasing the duty factor resulted in a lower material removal rate. With a significantly high duty factor, thicker oxide film was generated, resulting in a lower material removal rate.

4) The frequency of pulse voltage was optimized to obtain the maximum material removal rate. The thickness of the oxide film becomes thick with a long duration of the positive pulse. With excessively higher pulse frequencies, the faradaic reaction cannot start due to the time needed for the full charge of the electric double layer and ion rearrangement in the concentration boundary layer, resulting in a decreased material removal rate.

5) Translating the electrolyte jet along the rod axis, micro-rods with the average diameter of 85 μm and high aspect ratio of 530 were machined. The end of the rod was tapered due to the stray current. However, the influence was decreased using a smaller jet diameter.

References

1) Ippolito R, Tornincasa S, Capello G, Electron-Jet Drilling-basic Involved Phenomena. CIRP Annals-Manufacturing Technology, 1981, 30(1): 87-89.
2) Kunieda M, Yoshida M, Yoshida H, Akamatsu Y, Influence of Micro Indents Formed by Electro-chemical Jet Machining on Rolling Bearing Fatigue Life, ASME, PED, 1993, 64: 693-699.
3) Kawanaka T, Kunieda M, Mirror-like Finishing by Electrolyte Jet Machining, CIRP Annals – Manufacturing Technology, 2015, 64(1): 237-240.
4) Maeda S, Saito N, Haishi Y, Principle and characteristics of electro-chemical machining. Technical report of Mitsubishi Electric, 1967, 41(10): 1267-1279 (in Japanese).
5) Miyoshi K, Kunieda M, Fabrication of Micro Rods of Cemented Carbide by Electrolyte Jet Turning, Procedia CIRP, 2016, 42: 373-378.
6) Han W, Kunieda M, Fabrication of tungsten micro-rods by ECM using ultra-short-pulse bipolar current, CIRP Annals-Manufacturing Technology, 2017, 66(1): 193-196.
7) Yoneda K, Kunieda M, Numerical Analysis of Cross Section Shape of Micro-Indents Formed by the Electrochemical Jet Machining. Journal of JSEME, 1996, 29(63): 1-8. (in Japanese)
8) Masuzawa T, Fujino M, Kobayashi K, Suzuki T, Kinoshita N, Wire Electro-Discharge Grinding for Micro Machining. CIRP Annals – Manufacturing Technology, 1985, 34(1): 431-434.
9) Van Damme S, Nelissen G, Van Den Bossche B, Deconinck J, Numerical model for predicting the efficiency behavior during pulsed electrochemical machining of steel in NaNO₃, J. of Applied Electrochemistry, 2006, 36: 1-10.
10) Schuster, R., Kirchner, V., Allongue, P., Ertl, G, Electrochemical micromachining. Science, 2000, 289(5476): 98-101.
11) Anasane, S. S., Bhattacharyya, B, Parametric analysis of fabrication of through micro holes on titanium by maskless electrochemical micromachining. The International Journal of Advanced Manufacturing Technology, 2019, 105(11): 4585-4598.