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Effects of ultrasonic vibration on the silver deposition in powder mixed EDM for antibacterial surface modification

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Abstract

Previous studies have demonstrated that silver nano-powder mixed electrical discharge machining (PMEDM) is a potential technique for concurrent ablative shaping medical implants and generating antibacterial layers on the surfaces. PMEDM modified surfaces with deposited silver offer excellent antibacterial properties along with very good biocompatibility. However, for medical applications, PMEDM is still facing challenges providing sufficiently uniform distribution of deposited silver and preventing spattered layers that might lead to delamination from the modified surface.

It was indicated that ultrasonic vibration (UV) has remarkable effects on the fluid dynamics in the spark gap, which positively affect the silver distribution and reduce the generation of spattered layers. Consequently, in this study, a Ti6Al4V workpiece is vibrated with an ultrasonic frequency of 22.3 kHz and an amplitude of 2.5 µm during the PMEDM process. The effect of vibration direction in *X* and *Z* axis on the silver deposition is evaluated while discharge energy is varied up to 125 µJ. For comparative analyses also PMEDM without vibration assistance is investigated. Ti6Al4V hollow tool electrodes with an outer diameter of 3 mm and an inner diameter of 2.5 mm are utilised for internal flushing with a pressure of 4 bar. A hydrocarbon-based dielectric fluid is mixed with silver nano-powder at a concentration of 15 g/l. Results show that UV assisted PMEDM increases the content of deposited silver up to 1.7 times compared to PMEDM without vibration. UV with vibration in *Z* direction and pulse energies of 55 µJ and 125 µJ results in uniform silver distribution and significantly decreases spattered layer formation, whereas the use of vibration in *X*-direction leads to negative effects. By carefully selecting machining conditions such as vibration direction and amplitude, wall thickness of hollow tool electrode and powder concentration, PMEDM provides surfaces with promising characteristics for medical applications regarding silver deposition and antibacterial properties.

Keyword: powder mixed EDM, ultrasonic vibration, discharge energy, silver deposition, antibacterial effect

1. Introduction

The demand for implantable devices is rapidly increasing while the medical implant industry is still facing a major challenge of implant-associated-infections. Recent techniques for antibacterial surface modification such as physical vapor deposition, chemical vapor deposition and magnetron sputtering until now are solely used for coating an antibacterial layer on implant surfaces, therefore additional manufacturing processes for shaping the implants are required.

Electrical discharge machining (EDM) has been widely used for machining medical devices. By suspending silver nano-powder in the dielectric fluid, PMEDM has demonstrated its applicabilities for ablative shaping of medical implants and concurrently generating antibacterial layers on the implant surfaces [1]. In comparison to the polished surfaces and EDM surfaces without silver, PMEDM surfaces containing silver exhibited a significant reduction in both Staphylococcus aureus bacterial numbers and clusters while no negative influence on the attachment and differentiation of osteoblasts was observed [1,2]. Additionally, the machining time can be decreased down to 3 times by mixing silver nano-powder in the hydrocarbon-based dielectric fluid compared to EDM without powder [1]. However, the spattered layer, due to its size and low adhesion to the alloying layer, is not suitable for the medical implant application as illustrated in Fig. 1. In addition, since the silver content affects antibacterial properties significantly, generation of surfaces exhibiting sufficiently uniform distribution of silver is required for uniform antibacterial properties over the modified surface.

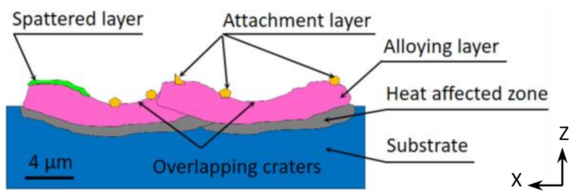

Figure 1. Sketch of the cross-section of a layer generated by PMEDM [1]

It has been reported that through vibrating the workpiece with ultrasonic frequency in a *Z*-axis when using a discharge energy of 10 µJ in PMEDM with side flushing, the generation of spattered layer was reduced. Nevertheless, it can be realized that by utilizing side flushing and a tool electrode with a diameter of 2 mm, UV poses a challenge for silver nano-powders getting deep into the spark gap, which reduces the actual powder concentration [1,3].

Consequently, to comprehensively investigate the effect of UV on the silver deposition in PMEDM, internal flushing is applied in this study whereby the workpiece is ultrasonically vibrated in *Z*-axis and in *X*-axis direction. In addition, the effect of increasing discharge energy on the resulting silver deposition is evaluated.

2. Methodology

The distribution of silver powders in the PMEDM machining gap with and without the vibration assistance is shown in Fig. 2.

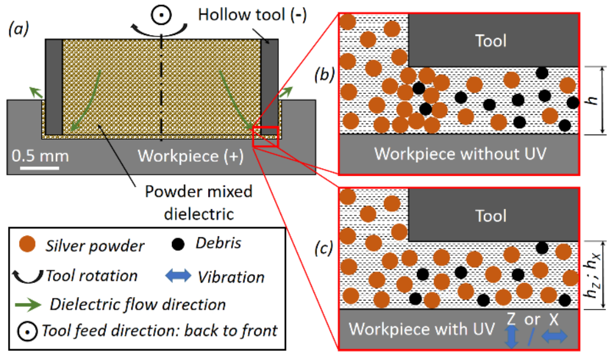

Figure 2. Schematic showing expected effects of UV on the silver powder distribution in the machining gap

Due to narrow spark gaps between 5 µm and 10 µm leading to a congestion of powder particles in the gap [3,4], challenges in transporting the suspended silver powders through the gap were reported when applying internal flushing in PMEDM without UV assistance as illustrated in Fig. 2 (b) [5]. Therefore, the actual powder concentration in the gap is remarkably reduced. To solve this challenge, UV is introduced to the workpiece in *Z*-axis or *X*-axis direction as shown in Fig. 2 (c). Additionally, it is also aimed to improve the uniform distribution of silver powders over the whole spark gap, which is a prerequisite for sufficient uniformity of deposited silver.

The experiments were performed with a Hielscher UIP250 ultrasonic vibration system integrated with a PMEDM circulation system, whereby the Ti6Al4V workpiece was vibrated at a frequency of 22.3 kHz. A Polytec OFV-505 Laser Vibrometer and a Polytec OFV-5000 controller were used to measure the vibration frequency and amplitude. The applied experimental conditions are shown in Table 1.

Table 1 Experimental conditions

Parameter	Value
Machine	Sarix T1T4 µ-EDM machine
Workpiece	Material: Ti6Al4V
Tool electrodes	- Outer diameter 3.0 mm - Inner diameter 2.5 mm - Rotational speed: 300 / min - Material: Ti6Al4V
Dielectric fluid	HEDMA111 oil
Flushing pressure	4 bar
Discharge energy	10 µJ, 55 µJ and 125 µJ
Polarity	Negative tool electrode
Silver powder	- Size: 50-60 nm - Concentration in dielectric: 15 g/L
Ultrasonic vibration	- Frequency: 22.3 kHz - Amplitude ($a_{p\text{-}p}$): 2.5 µm - Vibration direction: *Z* or *X* axis

For each machining condition, three machining repetitions were carried out. The resulting modified area is shown in Fig. 3. Two overlapping machining lines were performed to modify an area of *X* x *Y* = (5 x 10) mm² with a depth of 45 µm.

After machining, an ethanol filled ultrasonic bath was used to clean each sample at room temperature. Energy dispersive X-ray spectroscopy (EDS) was utilized to analyze the elemental composition and material mapping. The surface topography was analysed by scanning electron microscopy (SEM). To get the average deposited silver content of the modified surface, a spectrum of approx. (3 x 3) mm² was analyzed by EDS. The distribution of deposited silver over the surface was evaluated through analyses of 12 smaller spectra numbered from 1 to 12.

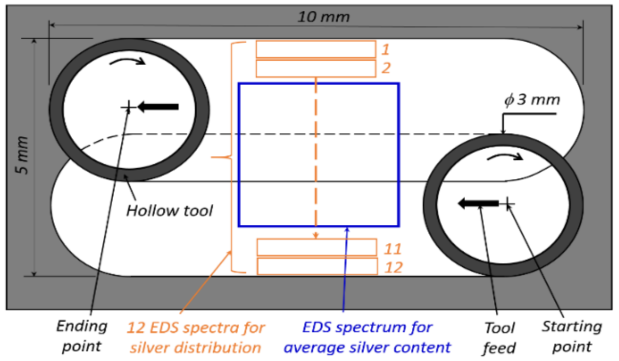

Figure 3. Top view on the geometry of the modified area and locations for EDS analyses

3. Results and discussions

In this section, influences of the vibration direction and discharge energy on the silver deposition are presented and discussed. This focuses on the resulted silver distribution over the modified surface and the prevention of spattered layer.

3.1. Effects of the vibration direction

Fig. 4 shows the average content of the deposited silver. It can be observed that UV plays a vital role in improving the deposited silver content when utilizing internal flushing in PMEDM. The application of UV in *Z*-axis direction results in the highest deposited silver content. An increase of the average silver content from 2.5 % to 4.2 % has been realized with using a discharge energy of 125 µJ compared to PMEDM without UV. This is due to the fact that the actual powder concentration in the machining gap is significantly increased by introducing the vibration to the workpiece.

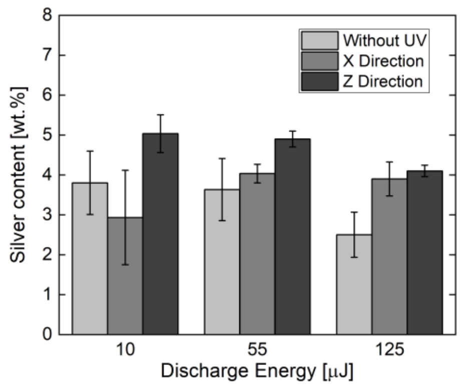

Figure 4. Average content of the deposited silver depending on the vibration direction and discharge energy

As regards the effects of the vibration direction, by applying UV in *Z*-axis direction the cyclical change of the gap size with an ultrasonic frequency is assumed to break the congestion of the powder particles in the gap and works as an additional pump, which significantly improves the transportation of the silver powder through the machining gap. The application of UV in *X*-axis direction leads to a remarkable increase of the deposited silver content by applying 125 µJ discharge energy compared to PMEDM without UV. However, at 10 µJ discharge energy which leads to decrease the spark gap [4], the deposited silver content is much lower with a wide standard deviation. Introducing UV to the workpiece in *X*-axis direction seems to have a positive effect on breaking the congestion of the powder particles in sufficiently wide gaps, while in case of smaller gaps, it aggravates this congestion.

Effects of the vibration direction on the distribution of deposited silver over the modified surface are shown in Fig. 5.

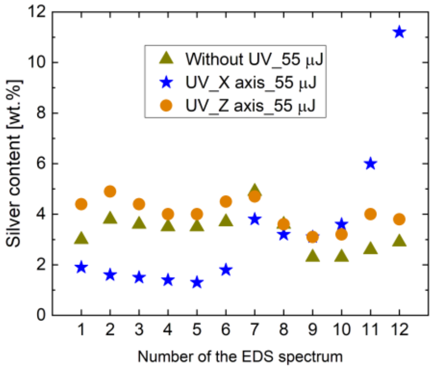

Figure 5. The distribution of deposited silver over the surface when applying different vibration directions at a discharge energy of 55 µJ

It can be observed that the deposited silver distribution tends to increase from the center to the outside of the machining line. A remarkable difference of the silver content between two machining lines is realized. Without UV assistance, the deposited silver is distributed in a range of $3.31^{+1.6}_{-1}$ %, whereas it is $4.1^{+0.8}_{-0.9}$ % through introducing UV to the workpiece in *Z* axis, therefore generating modified surfaces exhibiting a uniformer silver distribution. However, vibrating the workpiece along *X*-axis leads to a negative effect in uniformity of the silver distribution.

As shown in Fig. 6, influences of the vibration direction on the spattered silver are represented.

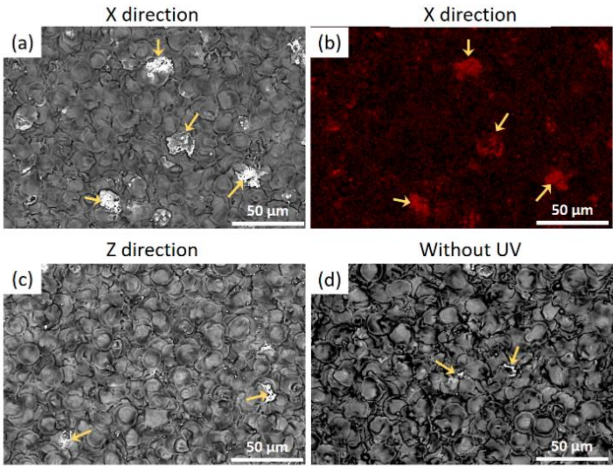

Figure 6. (a,c,d) SEM images and (b) silver mapping of (a) of the surfaces modified by PMEDM with and without UV assistance using discharge energy of 55 µJ (arrows point on spattered silver; an acceleration voltage of 20 kV and the working distances from 6.6 mm to 7.8 mm were used for SEM analyses)

A SEM image and a silver mapping analysis (identical area) of the modified surface applying UV in *X* axis are shown in Fig. 6 (a) and Fig. 6 (b), respectively. A large amount of the spattered silver can be observed. In comparison to the surface modified without UV assistance shown in Fig. 6 (d), introducing the vibration in *X* axis to the workpiece increases the spattered silver significantly. In case of UV assistance in *Z* direction shown in Fig. 6 (c) the modified surface shows a small amount of spattered silver. However, in comparison to surfaces modified without UV, the effect of UV in *Z* direction on the spattered silver is not clear when using a discharge energy of 55 µJ. Since discharge energy affects the machining gap significantly [4], impacts of UV in *Z* direction on the distribution of deposited silver are also investigated when the discharge energy is varied from 10 µJ to 125 µJ.

3.2. Effects of discharge energy on the silver distribution

Fig. 7 shows the distribution of deposited silver over the modified surfaces with 10 µJ and 125 µJ pulse energies.

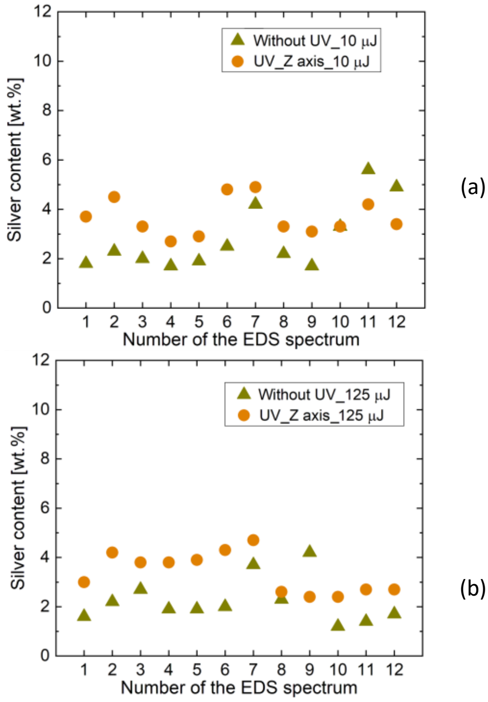

Figure 7. Distribution of deposited silver over the modified surface when applying (a) 10 µJ and (b) 125 µJ discharge energies without UV and with UV in *Z*-axis direction

In comparison to the application of 55 µJ discharge energy in PMEDM (Fig. 5), the utilization of lower and higher discharge energies decreases the uniformity of deposited silver over the surface both without and with UV assistance in *Z* direction. Specifically, without vibrating the workpiece, the silver content is distributed in a range of $2.84^{+2.7}_{-1.2}$ % and 2.23^{+2}_{-1} % when applying 10 µJ and 125 µJ, respectively, whereas they are $3.68^{+1.2}_{-1}$ % and $3.38^{+1.3}_{-1}$ % when the workpiece is ultrasonically vibrated. From Fig. 5 and Fig. 7, it can be realized that at different levels of the discharge energy, the assistance of UV enhances the uniformity of silver distribution over the surface significantly. The utilization of 55 µJ discharge energy results in the most uniform distribution.

Silver mapping images of the modified surfaces using different discharge energies without UV and with UV in *Z* axis are represented in Fig. 8. It can be observed that in all cases silver is deposited to the whole analyzed workpiece surfaces. The deposited silver is more densely distributed over the surface when UV is applied. A higher powder concentration in the machining gap is one reason, which leads to a higher deposited silver content. Additionally, effects of UV in Z axis on improving the silver powder amount attaching on the alloying layer when this layer is still in the molten phase also result in this dense distribution [1,3,6]. The bacteria's sizes are in micro scale, e.g. Staphylococcus aureus has diameter of 0.5 µm - 1.5 µm, therefore more dense distribution may improve antibacterial properties of the modified surface.

Regarding the spattered silver, the assistance of UV in *Z* direction with discharge energy of 10 µJ shows its remarkable influence on reducing the generation of spattered layer. The amount of spattered silver has been significantly decreased. However, the effect of UV on the spattered silver at discharge energies of 55 µJ and 125 µJ is still not clear since relatively similar small amounts of the spattered silver can be observed on surfaces modified with and without UV assistance. It should be noted that a higher powder concentration owing to UV assistance results in a higher deposited silver content as shown in Fig. 4, however it also increases the generation of spattered silver. Therefore, UV may have effects on preventing the increase of this spattered silver. The spattered silver is still not completely eliminated. However, due to influences of tool geometry on decreasing the generation of spattered layer in UV assisted PMEDM [1,3], hollow tool electrodes with a thicker tool wall should be used for the elimination of the spattered layer. Additionally, lower powder concentrations such as 5 g/L and 10 g/L are also recommended.

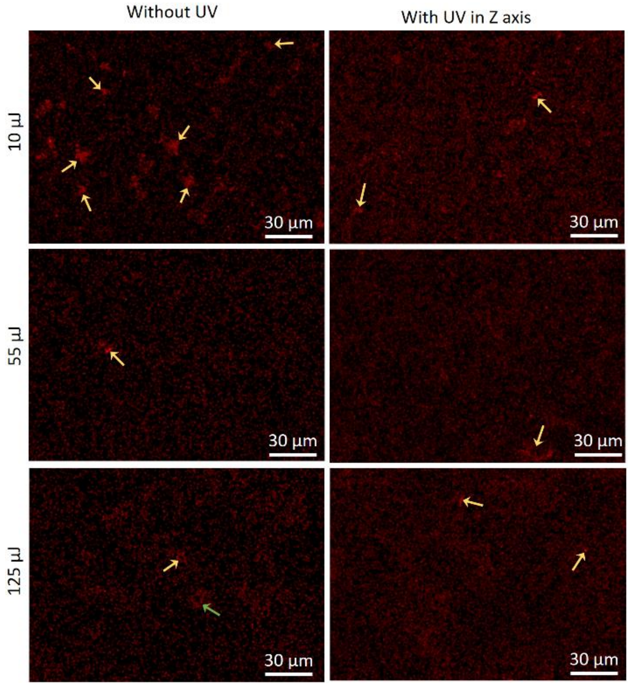

Figure 8. Silver mapping of the surfaces modified using different discharge energies with and without UV (arrows point on spattered silver; mapping analyses with acceleration voltage of 10 kV)

It can be also observed from Fig. 8 that increases of discharge energy to 55 µJ and 125 µJ leads to a reduction of the spattered silver. It is assumed that larger spark gaps at higher discharge energies facilitate the flushing condition which decreases the resolidification of molten spattered silver on the surface.

Using a higher discharge energy leads to an increase in surface roughness and changes in surface topography. The roughness value *Ra* after PMEDM with UV assistance in Z direction is increased from $(0.26 \pm 0{,}02)$ µm to $(0.34 \pm 0{,}01)$ µm and to $(0.65 \pm 0{,}03)$ µm when increasing discharge energy from 10 µJ to 55 µJ and 125 µJ, respectively. The formation of larger and deeper craters at higher energy as can be seen in Fig. 9 is a main reason for this [1]. The results also show that the roughness of surfaces modified with UV in Z direction is similar to surfaces modified without UV assistance. For comparison with a regular EDM results, further experiments were performed using copper tool electrode without powder and without UV assistance. The results showed even rougher surfaces of Ra = 0.8 ± 0.03 µm when utilizing a discharge energy of 125 µJ.

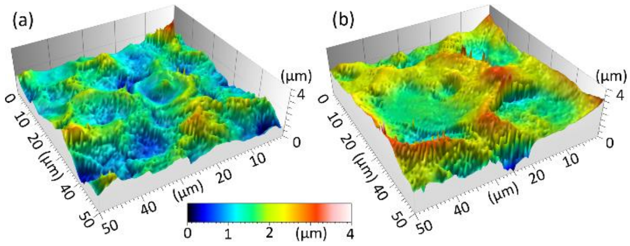

Figure 9. Topography of surfaces modified by UV assisted PMEDM in Z direction applying (a) 55 µJ and (b) 125 µJ discharge energies

4. Conclusion

The present study was undertaken to investigate the influence of ultrasonic vibration on the silver deposition in PMEDM for antibacterial surface modification. The ultrasonic vibration with vibrated direction in *X* and *Z* axis direction has been introduced to the Ti6Al4V workpiece during the PMEDM process while discharge energy was varied up to 125 µJ. From the results, the following summaries can be drawn:

- The content of deposited silver has been significantly increased up to 1.7 times with the assistance of ultrasonic vibration compared to PMEDM without vibration.
- The application of ultrasonic vibration to the workpiece with vibrated direction in *Z* axis improves the uniformity of silver distribution over the modified surface and significantly decreases the generation of spattered silver in comparison to surfaces modified without vibration.
- In contradiction, the utilization of ultrasonic vibration with the workpiece's vibrated direction in *X* axis results in non-uniformly deposited silver distribution and an increase of the spattered layer.
- Discharge energy plays a vital role in the silver deposition. Pulse energies of 55 µJ and 125 µJ with an ultrasonically vibrated workpiece in *Z* axis is recommended to achieve surface properties, which meet the requirement for medical application with respect to the silver deposition and antibacterial properties.
- It is possible to provide sufficiently uniform distribution of deposited silver and significantly decrease the spattered layer by carefully selecting machining conditions.

Recommendation

Further studies are recommended to evaluate the effects of ultrasonic vibration with larger vibrated amplitudes on the silver deposition. Thicker hollow tool walls and lower powder concentrations up to 10 g/L should be used.

Acknowledgements

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