

# Experimental Study on Preparation of Ultra-Hard Brittle Materials Based on Electrochemical Processing

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It is of great engineering significance to study methods for processing and preparing ultra-hard brittle materials with low processing cost and high processing efficiency. In this paper, the traditional Electrical Discharge Machining (EDM) ultra-hard brittle materials technology is improved, a method for processing the ultra-hard brittle materials by spray electrochemical method is proposed, and this method is applied to the processing of a typical ultra-hard brittle material monocrystalline silicon, and then the influence of processing peak voltage, pulse width/interval, electrolyte concentration and other parameters on the processing effect of the ultra-hard brittle materials is analyzed. Ultra-hard brittle materials can be etched by electrochemical etching and chemical dissolution. In this study, the conventional EDM-electrochemical technology is improved, and the liquid electrolyte is converted into a fog state by using an atomization device. The interrupted ratio of the metal wheel is set to  $\eta=0.55$ ; the thickness is set to 2 mm, and the rotate speed is set to 1800 r/min. The test results show that the larger the peak voltage, the larger the pulse width, the smaller the pulse interval, the larger the metal wheel rotate speed, and the larger the removal rate and surface roughness of the ultra-hard brittle material. The removal rate and surface roughness of the ultra-hard brittle material both decreased first and then increased with the increase of the electrolyte concentration. The research conclusions can provide a theoretical reference for the electrochemical processing of ultra-hard brittle materials.

## 1. Introduction

Ultra-hard brittle materials include semiconductor materials (monocrystalline silicon, cuprous oxide, gallium arsenide, silicon carbide, etc.), optical glasses (colorless/colored optical glass, radiation-proof optical glass, radiation-resistant optical glass, etc.), engineering ceramics (aluminium oxide, zirconium oxide, silicon nitride ceramics, etc.). Ultra-hard brittle materials have broad application prospects in electronics, various civil industries, transportation, national defense and other fields (Aprea et al., 2018; Tian et al., 2009; Djilali, 2017). Early preparation and processing methods for ultra-hard brittle materials were mainly physical machining, including outer diameter (OD) cutting, band saw cutting, scroll saw cutting, cutting machining, grinding preparation, etc. Due to the high brittleness of the ultra-hard brittle materials, the above-mentioned physical processing methods can easily cause micro-cracks and various defects to the ultra-hard brittle materials. Therefore, finding a method with low processing cost and high processing efficiency has important engineering significance for the preparation of high-performance ultra-hard brittle materials.

In recent years, researchers have proposed various composite processing methods for ultra-hard brittle materials, and its core technique is to use heat energy, light energy, electrochemical energy, etc. to achieve processing of various types of ultra-hard brittle materials, including ultrasonic processing, laser preheating processing, EDM, etc. (Zhang et al., 2011; Kang, 2011; Liu and Wei, 2013; Liu et al., 2015, Lu, 2018). Among them, the EDM technology is widely used, its basic principle is to generate gas barrier film by electrolysis, and to etch and prepare the ultra-hard brittle materials by the breakdown effect of EDM (Peng and Liao, 2004; Bamberg and Rakwal, 2008; Ali et al., 2018). However, EDM has a large wear on the slotted metal wheel and high production costs.

In view of above defects, this paper improves the traditional EDM ultra-hard brittle material technology, and innovatively proposes a spray electrochemical method for the preparation of ultra-hard brittle materials. The typical ultra-hard brittle material monocrystalline silicon is processed, and the effects of processing peak

voltage, pulse width/interval, electrolyte concentration and other parameters on the processing effect of ultra-hard brittle materials are analyzed. The research conclusion can provide a theoretical reference for the electrochemical processing of ultra-hard brittle materials.

## 2. Heat transfer model for electrochemical processing of ultra-hard brittle materials

The processing principle of ultra-hard brittle materials by the spray electrochemical method proposed in this paper is shown in Figure 1. The metal wheel and the electrolyte are respectively an anode and a cathode, between the metal wheel and the electrolyte is the gas film. Under normal conditions, the gas film will isolate the metal wheel from the electrolyte, so that chemical reactions do not occur in the device, while after the gas film is broken down by spark ignition, a discharge channel shown in the figure is formed between the metal wheel and the electrolyte. Due to the high-speed rotation of the metal wheel, the residual energy of the electric spark after the electrolyte is broken can directly act on the surface of the ultra-hard brittle material. The material melts and vaporizes, forming pits of different sizes under the action of electrochemical discharge. Multiple ignition of electric sparks can effectively impact the surface of the ultra-hard brittle material, and finally achieve etching of the ultra-hard brittle material (Li and Ren, 2018).

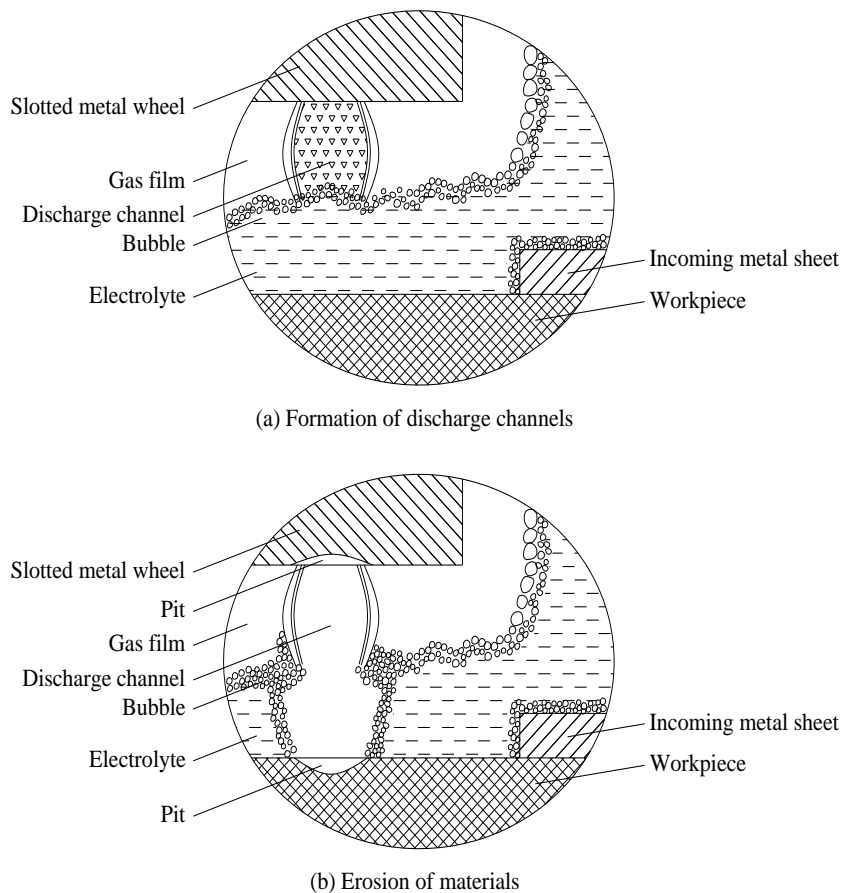


Figure 1: Principle of electrochemical processing of ultra-hard brittle material

The heat transfer model for electrochemical processing of ultra-hard brittle material is shown in Figure 2. After the spark discharge, a large amount of heat is generated. Some of the heat is dissipated in the air due to the high-speed rotation of the metal wheel, and a part of it is absorbed by the electrolyte, causing the temperature of the electrolyte to gradually increase. When the temperature is increased to a certain extent, the electrolyte is vaporized and the underlying ultra-hard brittle material is exposed, making the material melt and corroded areas of different sizes appear. Due to the high-speed rotation of the metal wheel, the etched areas of the ultra-hard brittle material are constantly moving, and the molten gasification area of the ultra-hard brittle material is always controlled within a reasonable range.

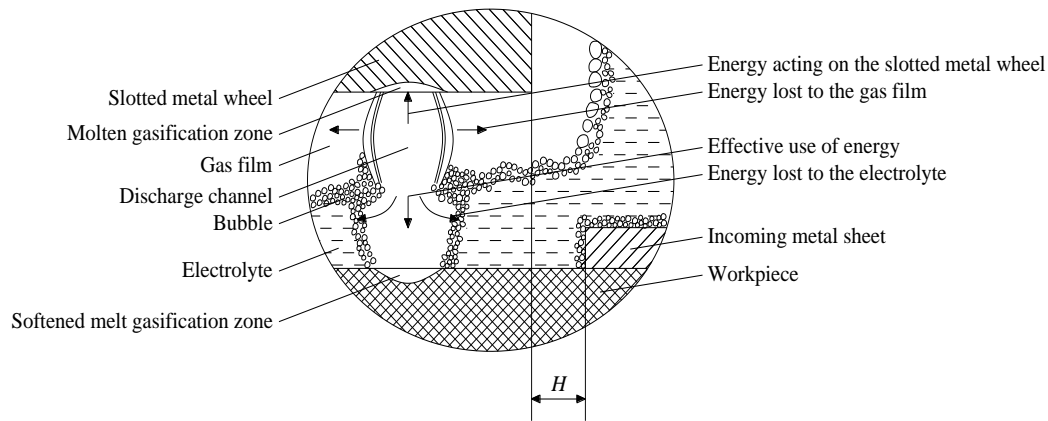


Figure 2: Heat transfer model for electrochemical processing of ultra-hard brittle material

The traditional method based on EDM-electrochemical technology for the processing of ultra-hard brittle material has very low energy utilization rate. This paper optimizes it according to the following method:

(1) Use an atomization device to convert the liquid electrolyte into a fog state, then the processing medium of the ultra-hard brittle material changes a gas-liquid two phase. After atomization of the electrolyte, the atomized electrolyte greatly reduces the energy absorption in the device, and the energy required for the formation of the metal wheel gas film is also significantly reduced, thereby increasing the useful work in the device.

(2) Redesign the structure of the metal wheel, as shown in Figure 3. The interrupted ratio of the traditional slotted metal wheel is  $\eta=0.6-0.8$ . In order to make the gas film around the metal wheel form faster, the interrupted ratio of the metal wheel is reduced to  $\eta=0.55$ ; and the thickness of the metal wheel is reduced to 2mm to increase the energy density after EDM.

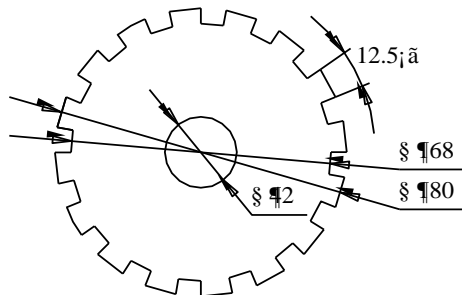


Figure 3: Optimized metal wheel structure

(3) Set the rotate speed of the metal wheel to 1800r/min, at which the air flow field around the metal wheel is in an optimal state.

### 3. Test results and analysis

#### 3.1 Test methods

The DC power supply is used as the external power supply of the test device, after energizing, the reactions occur at the cathode and the anode in the electrolyte are:

Reaction at the cathode:



Reaction at the anode:



The amount of hydrogen generated is much larger than that of oxygen, so the ultra-hard brittle material is processed by the method of positive electrode processing. Monocrystalline silicon, the most common ultra-hard brittle material, is taken as an example to test the improved test method.

Figure 4 shows the effect of different types of electrolyte on the discharge waveform of monocrystalline silicon. As can be seen from the figure, since the monocrystalline silicon has a certain conductivity, when the electrolyte is water, the current in the device exhibits a typical semiconductor current waveform, that is, an overall periodic climbing waveform.

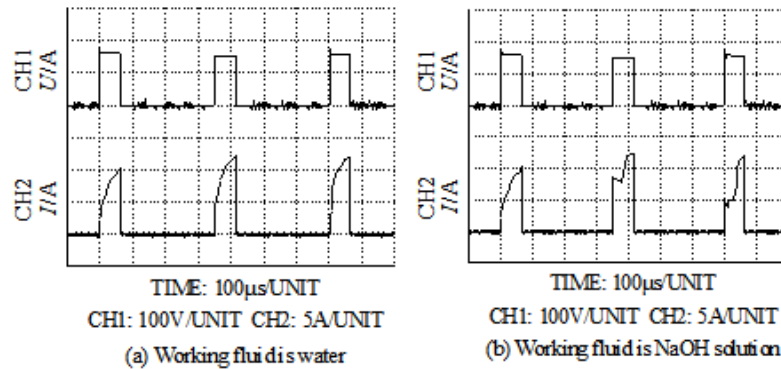


Figure 4: Effect of different kinds of electrolytes on the discharge waveform of monocrystalline silicon

When the electrolyte is sodium hydroxide, the discharge waveform is significantly different from that of water. Since the generated gas film completely separates the ultra-hard brittle material, when the electric energy is increased to a certain extent, the gas film is broken down, and spark discharge is formed in the gas film. After being broken, the gas film cannot completely isolate the positive and negative electrodes, so the resistance between the positive and negative electrodes is drastically decreased, and the current rapidly rises. According to the chemical reaction formula 1 and formula 2, after a certain period of time, the hydrogen gas generated after the reaction in the device will fill in the gas film and make it dense again, so that the electric resistance between the electrodes is increased again, and the current becomes smaller. It can also be seen from the figure that there is more fluctuation in the current growth and decline when the electrolyte is sodium hydroxide than when the electrolyte is water.

### 3.2 Influence of different parameters on the preparation and processing of monocrystalline silicon

Further explore the influence of different parameters on the processing effect of monocrystalline silicon during processing. Figure 5 shows the effect of peak voltage on the removal rate and surface roughness of the ultra-hard brittle material. It can be seen from the figure that the removal rate and surface roughness of the ultra-hard brittle material increase with the increase of the peak voltage, which is because the discharge energy of the device is also significantly increased after the voltage is increased, its ability to work on the ultra-hard brittle material is also increasing gradually, therefore, the increase in voltage is conducive to the complete elimination of impurities in the ultra-hard brittle material.

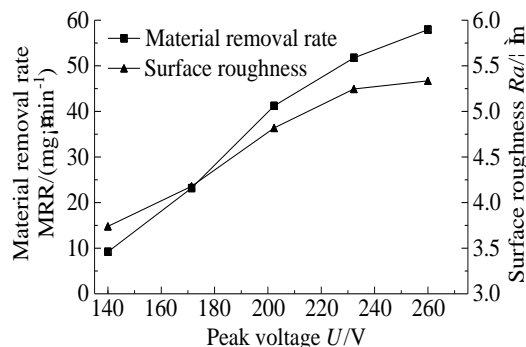


Figure 5: Effect of peak voltage on removal rate and surface roughness of ultra-hard brittle material

Figure 6 shows the effect of pulse width on the removal rate and surface roughness of the ultra-hard brittle material. It can be seen from the figure that the increase of the pulse width also causes the removal rate and

surface roughness of the ultra-hard brittle material to increase, wherein the material removal rate first increases rapidly and then slowly increases, and the surface roughness of the material increases rapidly at first and then remains basically unchanged.

Figure 7 shows the effect of pulse interval on the removal rate and surface roughness of ultra-hard brittle material. It can be seen from the figure that the larger the pulse interval, the lower the removal rate and surface roughness of the ultra-hard brittle material. Increasing the pulse interval reduces the number of electrochemical discharges in the device, resulting in reducing in the amount of energy acting on the ultra-hard brittle material per unit time.

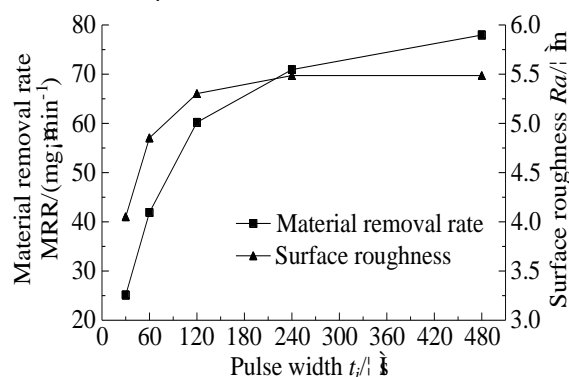


Figure 6: Effect of pulse width on removal rate and surface roughness of ultra-hard brittle material

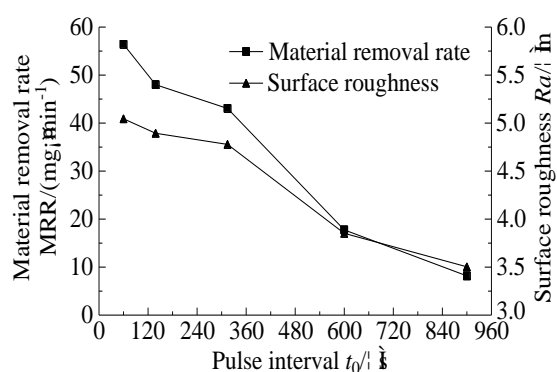


Figure 7: Effect of pulse interval on removal rate and surface roughness of ultra-hard brittle material

Figure 8 shows the effect of electrolyte concentration on the removal rate and surface roughness of the ultra-hard brittle material. It can be seen from the figure that the removal rate and surface roughness of the ultra-hard brittle material tend to decrease first and then increase with the increase of the electrolyte concentration. When the electrolyte concentration is 0, the energy generated by the spark discharge directly acts on the surface of the ultra-hard brittle material, and the energy utilization rate is higher, and at this time, both the removal rate and the surface roughness of the material are larger. When the electrolyte concentration is 5%, part of the energy generated by the spark discharge is absorbed by the electrolyte, so that the range of energy acting on the ultra-hard brittle material is increased, and the surface roughness of the material is improved. When the electrolyte concentration is further increased, the energy absorption in the electrolyte reaches a saturated state, the excess energy directly acts on the ultra-hard brittle material, so the removal rate and surface roughness of the ultra-hard brittle material increase again.

Figure 9 shows the effect of metal wheel rotate speed on the removal rate and surface roughness of the ultra-hard brittle material. The greater the rotate speed of the metal wheel, the greater the removal rate and surface roughness of the material. This is because the high-speed rotation of the metal wheel accelerates the formation of the gas film, which improves the processing efficiency of the ultra-hard brittle material.

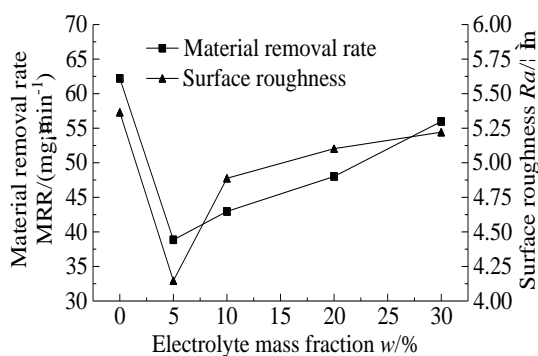


Figure 8: Effect of electrolyte concentration on removal rate and surface roughness of ultra-hard brittle material

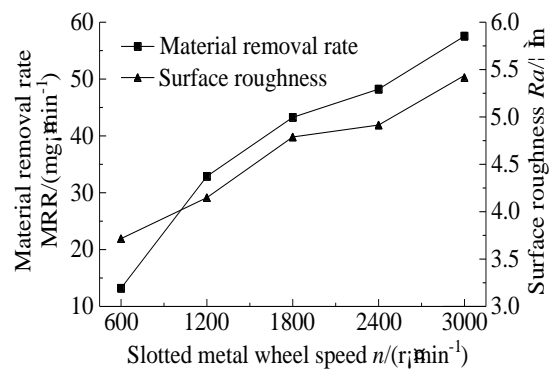


Figure 9: Effect of metal wheel rotate speed on removal rate and surface roughness of the ultra-hard brittle material

#### 4. Conclusion

This paper improved the traditional EDM ultra-hard brittle material technology, and innovatively proposed to process and prepare the ultra-hard brittle material by spray electrochemical method. It processed monocrystalline silicon, a typical ultra-hard brittle material, and analyzed the effects of processing peak voltage, pulse width/interval, electrolyte concentration and other parameters on the processing effect of the ultra-hard brittle material. The research conclusions are as follows:

(1) This study used electrochemical etching and chemical dissolution to etch the ultra-hard brittle material and improved the traditional EDM-electrochemical technology. It used an atomization device to convert the liquid electrolyte into a fog state, and set the interrupted ratio of the metal wheel to  $\eta=0.55$ , thickness to 2 mm, rotate speed to 1800 r/min.

(2) The larger the peak voltage, the larger the pulse width, the smaller the pulse interval, and the larger the rotate speed of the metal wheel, the greater the removal rate and surface roughness of the ultra-hard brittle material. The removal rate and surface roughness of the ultra-hard brittle material both decreased first and then increased with the increase of the electrolyte concentration.

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