euspen's 19th International Conference & Exhibition, Bilbao, ES, June 2019
www.euspen.eu

Using the Confined Etchant Layer Technique to process the 3D micro-structures by adjusting the voltage

Xiaole Wang[1*],Lianhuan Han[123], Dongping Zhan[2], Yongda Yan[1*]

[1] *Center for Precision Engineering, Harbin Institute of Technology, P.O. Box 413, Harbin 150001, China*
[2] *State Key Laboratory of Physical Chemistry of Solid Surfaces (PCOSS), Collaborative Innovation Center of Chemistry for Energy Materials (iChem), Engineering Reseach Center of Electrochemical Technologies of Ministry of Education, and Department of Chemistry, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen 361005, China*
[3] *Department of Mechanical and Electrical Engineering, School of Aerospace Engineering, Xiamen University, Xiamen 361005, China*

**18645109561@163.com*

Abstract

As one of the most important semiconductor materials, GaAs has many applications in fields of producing optoelectronic materials and ultra-high-speed integrated circuits. The Confined Etchant Layer Technique is one of methods to fine finish the GaAs. This way has the advantages of high efficiency, high resolution. In order to apply SECM (Scanning electrochemical microscopy) to process three-dimensional micro/nano structures, the tip current/positioning close-loop mode was found. We proposed the way by directly adjusting the voltage to process three-dimensional morphology, now. The processing range is increased. The machining profiles are more consistent, and the machining efficiency is higher. This method was used to verify the necessity through static and dynamic methods and this system was improved by processing sinusoidal surfaces. We will detailed describe the method of machining and machining results.

adjusting the voltage, micro-structures, electrode

1. Introduction

As a comparatively mature electrochemical micro-machining technique, SECM takes the advantages of no tool wear, no residual stresses, less heat, no surface and subsurface damages, high efficient and low cost [1–4]. At present, the corresponding 3D microstructures can be processed through the previously proposed tip current/ positioning close-loop mode. In SECM, the electrode's voltage is also one of the important factors determining the tip current besides the position factor. By adjusting the voltage, we can also process the corres- ponding three-dimensional microstructures on n-GaAs substrates. Compared with the method of adjusting the position, there are three advantages of adjusting volts. At first, the scope of machining of the former is less than the latter. The maximum machining range of the two is the same, but the minimum machining value of the former must be greater than 0 while the latter can achieve the minimum machining value of 0. Secondly, the method of adjusting the position requires controlling the signal of the position and moving the pzt to the specific position, while the latter directly controls the electrochemical workstation to adjust the machining volts directly. When there is a large fluctuation in the machining profile, the latter's efficiency is higher and its feedback time is shorter. Finally, with the change of the position of the tip, the machining profile will be changed. By adjusting the volts, the profile at different volts remain the same after normalization, so that the machining resolution is higher. Thus, we process larger and more complex three- dimensional structures by directly adjusting the electrode voltage.

2. Experimental section

2.1. Experimental materials

Silicon doped GaAs single crystal (n-GaAs , n = 0.8×10^{18} - 2.31×10^{18} cm-3), provided by China Crystal Technologies Co., China. Before using, the substrate surface of the n-GaAs is cleaned with acetone, ethanol and ultrapure water, and then dried with nitrogen. The experimental solution were NaBr and H_2SO_4 prepared with deionized water.

2.2. Experimental instruments and measurement

The working electrode is a tapered 100-µm-diameter (RG=2) Pt disk microelectrode. It was characterized by optical micro-scope and steady-state voltammetry curve, respectively. The counter electrode is Pt wire, and the reference electrode is Ag electrode. The SECM/ECMM instruments used in the experiment are all homemade which has been reported previously [5]. As shown in Fig. 1, the tuned voltage signal is input through the PID controller. It converts them into the electrochemical workstation and controls the volts up and down.

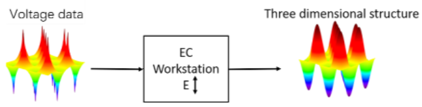

Figure 1. Schematic diagram of the tip voltage adjusting mode of SECM for electro-chemical micromachining.

2.3. Characterization methods

After processing, the 3D micro structure and the depth of the surface are characterized by the scanning force microscopy (SFM). The probe is a diamond probe with a radius of 10 µm.

Firstly, approaching the diamond tip to the substrate surface using the auto-approaching procedure until the force value changes more than 0.5 mN. Next, set the value of the piezoelectric ceramic at the constant of 2 mN. At last, scan the surface with scan speed of 20 μm / s and feed of 10 μm.

3. Results and discussion

3.1. The relationship between electrode voltage and etching depth under quasi-static

The 100s-static-state machining results of series of volts obtained by SFM scanning (Fig. 2-a) indicate that as the electrode's voltage increases, the machining depth increases. The result is not completely consistent with the cylindrical shape of the processing electrode due to the diffusion. The result is not completely consistent with the cylindrical shape of the processing electrode due to the diffusion. Compared with the simulation results performed by Comsol Multiphysics[24-25], the maximum error is only 0.069 μm. The middle width of the machining contours are slightly greater than 100 μm and the normalized-slightly-flawed-machining contours are the same. Corresponding to the energy equation in the electrochemical reaction, the current and voltage are Boltzmann relations: "$I(\mu A)=6.972(1+1/(1+e^{(37.95\times(E(V)-0.9819))}))$. Select the average value of the bottom surface of the center profile as the maximum machining depth. The electrode's voltage and the processing depth are Boltzmann relations: $d(\mu m)=-2.494(1+1/(1+e^{(40.73\times(E(V)-0.9822))}))$ (Fig. 2-b). The same as the previous conclusion, the processing current is linear with the processing depth:" $d(\mu m)=(I(\mu A)+0.0241)/2.7787$ (Fig. 2-c). It can be seen that the three-dimensional structure can be machined by changing the current of the tip by adjusting the volts and the fluctuation of results can be directly determined by the change of current.

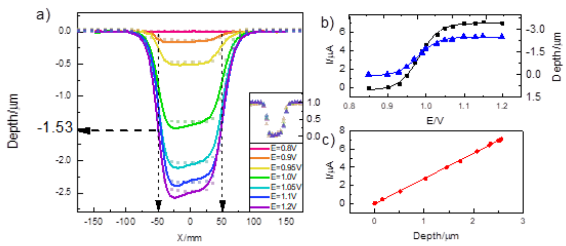

Figure 2. (a) The 100s-static-state machining results of series of volts obtained by SFM, the corresponding simulation results(dash line) and normalized results. (b) The relationships of the current (black line) and the etching depth (blue line) to the tip-volts. (c)The correlation between the etching depth and the current.

3.2. The relationship between electrode voltage and etching depth under quasi-static

Compared with static machining contours, there is a reduction in machining depth due to increased passivation during dynamic machining.The maximum reduction in depth is 0.21 μm and the middlewidth of the the machining contours are slightly smaller than 100 μm, but the trend of depth change at different volts is consistent with static conditions (Fig. 3-b). The volts' changing pattern(0.5 mm) is shown in Fig. 3-c and it is consistent with the machining contour cycle(0.502 mm). The maximum etch depth is 2.31μm , which is 0.18μm smaller than the static result. The relationship between processing depth and current is not a linear relationship, not only because of passivation, but also related to the volts changing. Ignoring the non-conductive point of the potential change curve, the potential and the depth are linear: $d(\mu m)=-(I(\mu A)+0.098)/3.0436$ (Fig. 3-d).The maximum deviation is 1.67μA . Therefore, increasing the rate of changing volts and setting the current feedback to adjust the electrode's voltage is very effective for eliminating passivation errors.

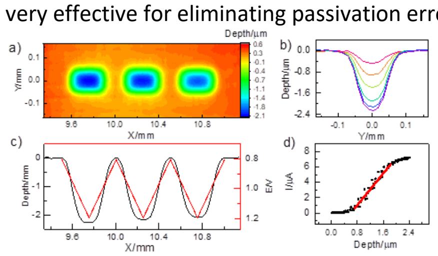

Figure 3. (a) The dynamic machining results of series obtained by SFM. (b) The dynamic etching profiles at different volts of Y. (c) The curve of volts changing and the contours of X. (d) The correlation between the etching depth and the current (square points)and the fitting curve(red line).

3.3. Processing 3D micro structure based on modulating the probe's current

The simpler sinusoidal surface was machined by modulating the electrode's voltage (Fig. 4). In order to avoid the electrolysis of water, the processing current range is reduced. The maximum depth of the three-dimensional sinusoidal surface machined by this system is 2.675μm and the minimum depth is almost 0. However, due to the passivation effect on the surface of the tool electrode, the etching effect of the tool electrode by adjusting voltage becomes worse with the increasing of time. It can be seen from that as the processing time increases, the structure of sine gradually deteriorates.

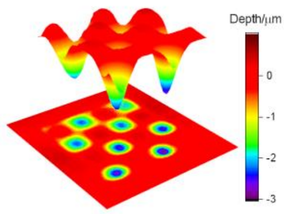

Figure 4. The SFM detection results processed by Origin software.

4. Conclusion

Due to the influence of passivation, there is a large error in setting the volts to directly machine a specific three-dimensional structure. Thus, new methods may be considered to eliminate errors caused in real time and realize the processing of specific three-dimensional microstructures. Because the entire theory is based on a linear relationship between the machining voltage and the depth of machining, the machining topography can be controlled by directly adjusting voltage. Mathematically adjust the machining voltage so that the curve of voltage is smoother and the machining results corresponds more closely to the theoretical results. The more optimized system, the better processing results.

Acknowledgements

Financial support of the National Natural Sience Foudation of China (21827802, 21573054, 21327002) are appreciated. The authors declare no competing financial interests.

References

[1] Bard A and Mirkin M 2001 *J. Scan. Electrochem. Microsco.* **38** 1049

[2] Hüsser O, Craston D,and Bard A 1989 *J. Scan. Electrochem. Microsco.* **136** 3222-29

[3] Tian Z, Fen Z,Tian X, et al 1992 *J. Fara. Discucc.* **94** 37-44

[4] Meltzer S and Mandler D,Tian X, et al 1995 *J. Chem. Soc.* **91** 1019-24

[5] Zhan D, Han L, Zhang J, et al 2016 J. Acc. Chem. Res. 49 2596-604