초소형 질량 분석기를 위한 이온 발생기의 열전자 방출 시험

論 文
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Hot Electron Emission Test of an Ion Source for a Micro Mass Spectrometer

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Abstract - This paper presents the principle and fabrication of a novel micro mass spectrometer and emission test of hot electron for ionization. A micro mass spectrometer consists of a micro ion source and a micro ion separator. The micro ion source consists of a hot filament and grid electrodes. Electrons emitted from a hot filament are to ionize some sample molecules. The ions are accelerated to an ion detector by an electric field. Mass can be analyzed by using the time of fight depending on the mass-to-charge ratio. The current of hot electron emission from the hot filament is measured for various input voltages.

Key Words : Micro mass spectrometer, Micro ion source, Time-of-flight, Hot filament

1. INTRODUCTION

Measuring masses of molecules in order to understand the structures and components of mixtures is very important in physics, chemistry and other sciences. Moreover, it has wide application areas such as in environmental monitoring, residual gas analyzer, drug analysis and space research. The mass spectrometer uses the difference in mass-to-charge ratio (m/e) of ionized atoms or molecules to separate them from each other. Previous mass spectrometers which are operated with high voltage in high vacuum are bulky and expensive. But the micro mass spectrometer can analyze some samples in low vacuum and reduce its cost.

Recent reports on mass spectrometers fabricated by micromachining technology show the possibility of the miniature mass spectrometer. Feustel first proposed the structure of a micro mass spectrometer by using micromachining[1]. Siebert fabricated a miniaturized mass spectrometer by anisotropic etching and electroplating[2]. But the mass spectrometer of Siebert is difficult to generate plasma. Oleg Kornienko fabricated a miniature ion trap mass spectrometer[3]. Tunstall fabricated the micro quadrupole mass filter by using micromachinig[4]. Tunstall used a bulky conventional ion source. The method of this paper differs from previous methods with respect to the ionization and the ion separation. In this paper, the bulk of the proposed mass spectrometer is small compared with them mentioned above, and a micro ion source fabricated together with an ion separator. The hot electron emission test of the ion source was performed.

2. PRINCIPLE

Figure 1 shows the principle of the micro time-of-flight mass spectrometer(TOFMS). The separation principle of the micro TOFMS is to separate the ions by the acceleration depending on the mass. That is to say, the heavy ions will travel more slowly than the light ones. Electrons produced from the heated filament are accelerated by the pulsed electric field. The accelerated electrons make collisions with some sample molecules and ionize them. Ions generated by collisions are accelerated by the electric field between acceleration electrodes. At all times the acceleration region, d, has an electric field, and the drift region D is a free-field region. Then, ions reach the ion detector in the order of mass increase. There is no need for a magnetic field. The relationship between the time of flight and the mass-to-charge ratio, of the ion is obtained by considering that the velocity of the ion is determined by the electrostatic field applied. The time of flight of the ion is proportional to the square root of its mass value. For the given length of flight and the acceleration voltage, the mass can be calculated from the time taken for the ion to reach the detector.

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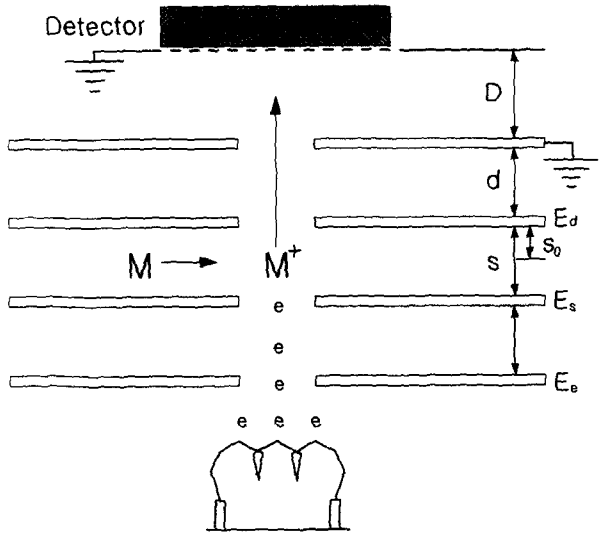

Fig. 1 The principle of the micro time-of-flight mass spectrometer.

3. STRUCTURE

The micro mass spectrometer consists of an ion source, an ion separator and an ion detector. Figure 2 illustrates the structure of the ion source and separator for the micro TOFMS. The ion source and ion separator consist of a tungsten filament, tungsten electrodes and four pairs of nickel electrodes on silicon substrate. The tungsten filament is to emit hot electron. Nickel electrodes are to focus and accelerate ions. There is a cavity under the tungsten filament. The cavity reduces the heat loss by conduction heat transfer. The repeller electrode is used in the test of the electron emission. The width and height of nickel electrodes are 80 μm and 15 μm, respectively. The total size of the ion source and separator of the micro TOFMS is 10 × 10 × 1 mm^3.

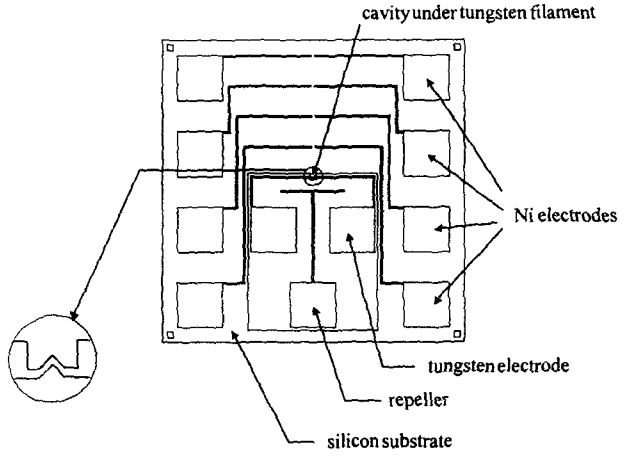

Fig. 2 The structure of the ion source and separator.

4. FABRICATION PROCESS

Figure 3 shows the fabrication process of the ion source and separator of the micro TOFMS. The starting material of the micro mass spectrometer is 525 ± 10 μm-thick 4 inch n-type <100> silicon wafer. First, a 0.7-μm-thick thermal oxide layer for an etch mask is grown. To make a step between the tungsten filament and nickel electrodes, the front side of the silicon wafer is etched by 10 μm with TMAH solution. This step is to locate the tungsten filament at the center height of nickel electrodes. After the residual oxide is etched off, a 0.7-μm-thick thermal oxide is grown again. The Cr/Au (300 Å/3000 Å) as a seed layer for electroplating is deposited by thermal evaporation process. To make grid electrodes and acceleration electrodes, a thick PR (AZ 4620) mold is fabricated. Then, nickel electroplating is performed. The thickness of nickel electrode is about 20 μm. After removing thick PR and Cr/Au, the tungsten layer (3000 Å) is deposited by sputtering and patterned by photolithography. The tungsten filament is fabricated by tungsten etch solution (34g KH_2PO_4, 33g $K_3Fe_3(CN)_6H_2O$, 13.4g KOH to 1 liter). The etch rate of tungsten is 1600 Å/min. The Cr layer, etch mask layer is thermally-evaporated by 0.3 μm and patterned. To make a cavity below the tungsten filament, the silicon substrate is etched by 30 μm with EPW (Ethylendiamine : Pyrocatechol : DI Water = 250 ml : 40 g : 80 ml) solution. Finally, the Cr layer is removed.

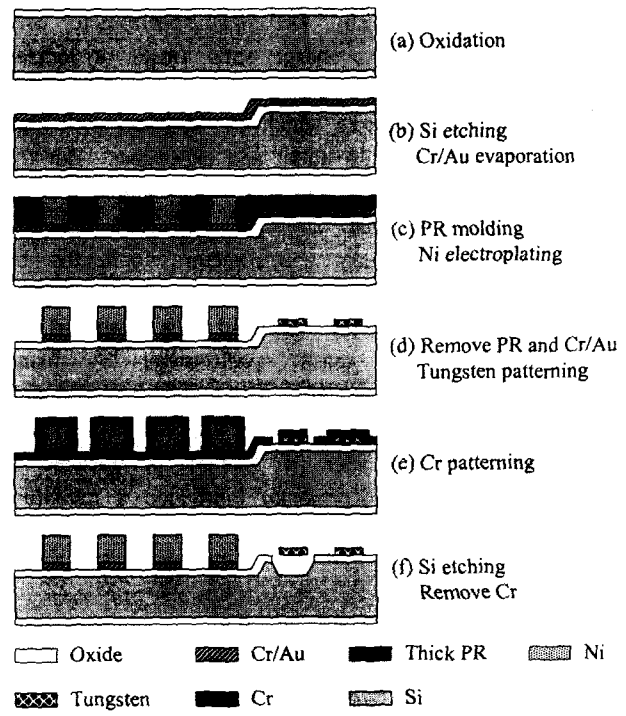

Fig. 3 The fabrication process of the ion source and separator.

The photograph of the fabricated ion source and separator of the micro TOFMS compared with a coin is shown in Figure 4. Figure 5 shows the SEM image of the fabricated tungsten filament. The width and thickness of the tungsten filament are 20 μm and 0.3 μm, respectively.

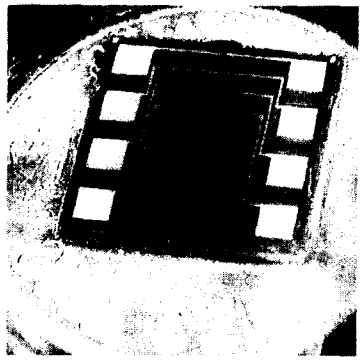

Fig. 4 The photograph of the fabricated ion source and separator.

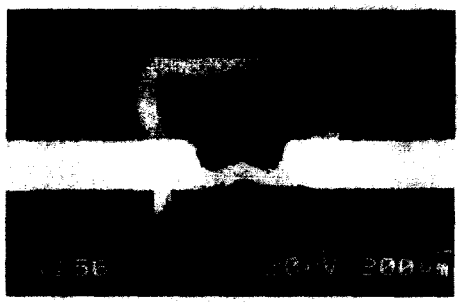

Fig. 5 The SEM image of the fabricated tungsten filament.

5. EXPERIMENTAL RESULTS

Figure 6 illustrates the measurement setup for the tungsten filament characteristic test of the fabricated ion source for the micro TOFMS. To protect oxidation of tungsten filament, the fabricated micro mass spectrometer is installed in a vacuum chamber equipped with a turbo molecular pump and a mechanical pump. At this time, the pressure of a vacuum chamber is 1.0×10^{-6} Torr. If the voltage is applied to both electrodes of the tungsten filament, heat is generated at the tungsten filament. If heat is generated at the tungsten filament, hot electrons are emitted. To detect emitted current, a pico ammeter is connected at repeller electrode behind the filament electrode. Figure 7 shows the photograph of the incandescent light of the working tungsten filament in vacuum. The resistance of the tungsten filament is 1.6 kΩ at room temperature. Figure 8 is the plot of measured electron current vs. the applied voltage. As the applied voltage increases from 2.5 V to 35 V, the electron current increases from 0.7 pA to 7 nA.

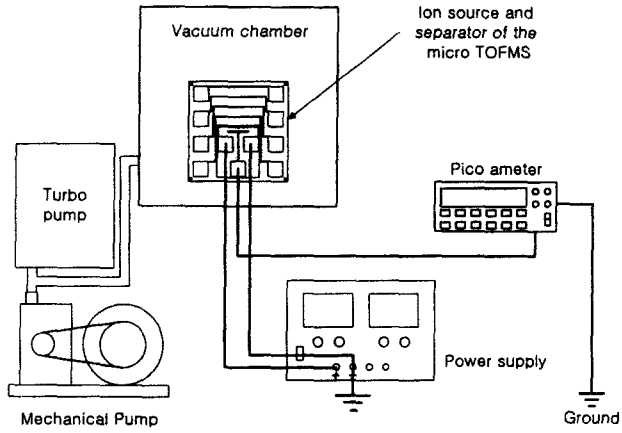

Fig. 6 The measurement setup for the tungsten filament characteristic test of the fabricated ion source of the micro TOFMS.

Fig. 7 The photograph of the working hot filament.

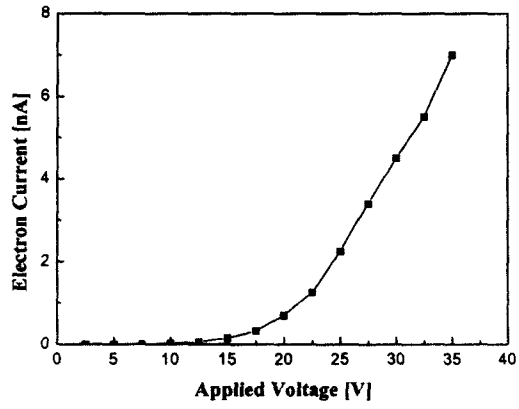

Fig. 8 The plot of the electron current vs. the applied voltage.

6. CONCLUSIONS

In this paper, the ion source and separator of the micro TOFMS has been fabricated and the hot electron emission of a tungsten filament has been tested. A micro mass spectrometer consists of a micro ion source with a tungsten filament and a micro ion separator with nickel electrodes. To identify the emission of the hot electrons from the tungsten filament, the voltage was applied to the tungsten filament and hot electron emission current was measured. In the near future, the test of the gas molecule ionization by the hot electron emission will be performed.

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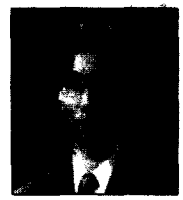

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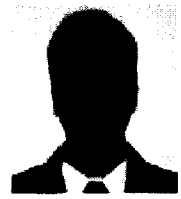

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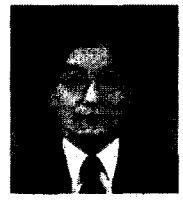

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