CHAPTER 5

Discussion and Conclusion

In the first part of this project, we study the principles of operation of the quadrupole mass spectrometer, construction of the quadrupole mass spectrometer system and the conceptual design of the system. From the conceptual design, three important parameters of the quadrupole mass spectrometry are radio frequency, a.c. voltage and d.c. voltage. These parameters are important for determining the equipment which uses to operate the quadrupole. The designed mass range is 1-1,000 amu and the maximum resolution is about 3,300 at ΔM = 0.3 amu. From the resolution and the mass range, the range of radio frequency is 0.5 - 2 MHz. But with this range of frequency, the value of U is less than 1 volt at mass 1 amu. However the lower limit of the voltage is 1 volt. To analyze this mass, the operation has to do at higher value of radio frequency. The chosen frequency is 2.5 MHz at which U is 1.83 V. Then the range of frequency with the designed mass range and ΔM = 0.3 amu is 0.5 – 2.5 MHz. The values of maximum d.c. voltage and maximum a.c. voltage are 1,000 volt and 5,700 volt, respectively. The resolution at mass 1 amu (~ 3.34) or low mass on this SIMS system is very low. To improve the resolution there be must more study on the operation in the second (D area in Figure 2.15) and third (B area in Figure 2.15) Mathieu stability region. (Dawson, 1984a ; 1984b, Du, 1997 ; 2000, Titov, 1998a ; 1998b). The work is study to improve the resolution at low m/z and complete the whole system by the installation of the electronic parts of the quadrupole such as high frequency generator, quadrupole controller and high-voltage supply.

In the second part, we study the sputtering from biological cells (onion) by the quadrupole gas analyzer (only gas molecule can be detected by the quadrupole). Before the measurement of onion cell, we calibrate the quadrupole gas analyzer by argon gas to improve the resolution of the mass spectrum, test the ability of the quadrupole gas analyzer by nitrogen gas and carbon dioxide and measure the

sputtering of three kinds of solid, plastic scintillator and pure carbon. The spectra of both gases are consistent with the spectra from National Institute of Standards and Technology data (see Appendix A). The spectrum of pure carbon shows a peak at m/z equal to 28 which is the carbon monoxide (CO) peak. Carbon peak cannot be observed because carbon atoms are very active when they sputtered from sample then immediately combined with oxygen atoms in chamber to form carbon monoxide molecules. The spectrum of plastic scintillator shows the peak at m/z equal to 2 which is the peak of hydrogen (H_2), but no carbon peak. The spectrum of silicon dioxide shows a base peak at m/z equal to 28 which is the peak of silicon and second height peak at m/z equal to 32 which is the peak of oxygen (O_2).

Some monoatomic sputtered ions cannot be detected because the sputtering is a physical process whereby atoms in a solid target material are ejected into the gas phase due to bombardment but the sputtered atoms are not in their thermodynamic equilibrium state. Therefore, they tend to condense back into the solid phase upon colliding with any surface in the sputtering chamber.

Mass spectrum of the onion cell shows hydrogen, oxygen, water (m/z=18) and carbon monoxide peaks. Carbon monoxide is the base peak and the explanation of carbon monoxide peak is the same as pure carbon sample. Second and third height peaks are oxygen and hydrogen, respectively. The lowest peak is water, normally water is the main composition in onion but this experiment used dry onion so that the peak of water is less than usual. The spectrum shows hydrogen, carbon and oxygen as we expect from living organism.

The cell wall of plant cells are composed of cellulose which has a chemical structure of $C_6H_{12}O_6$ (Voet and Voet, 1990). In previous experiments, the cell wall was bombarded by 30 keV Ar ion. The sputtering yields from TRIM simulation of the cell wall by assuming the cell wall to be the solid material are $Y_C = 0.18$, $Y_H=1.87$ and $Y_O= 7.65$ (Yu and others, 2002). SEM micrographs of the onion surface are shown in Figure 5.1. Compared to the unbombarded cell surfaces (Figure 5.1 a, d) which are smooth and even, the ion-bombarded surfaces (Figure 5.1 b, c, e) are severely etched and show undulations and scattered ‘holes ’. Close-ups of these holes show crater-like

structures as shown in Figure 5.1 c,e. From the SEM micrographs, the cell wall is inhomogeneous and preferential sputtering will occur.

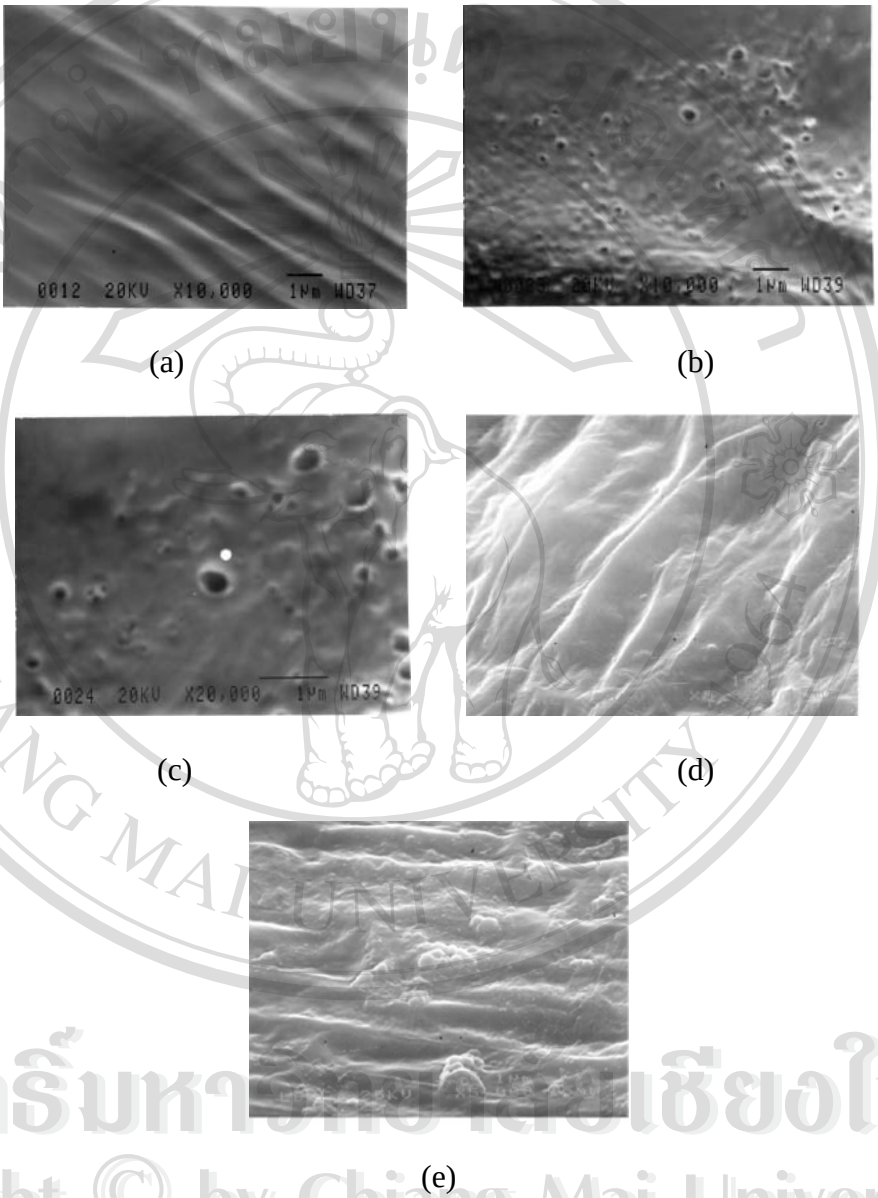

Figure 5.1 SEM photographs of the onion cell surfaces. Ion bombardment conditions are 30 keV Ar ions. (a) onion cell surfaces (unbombarded control) (b) ion bombarded onion cell surfaces (c) ion-bombarded onion cell surfaces at higher magnification (d) onion cell surfaces at high resolution (unbombarded control) (e) ion-bombarded onion cell surfaces at high resolution, at glancing angle (Yu and others, 2002).

The difference between the spectrum of cell wall bombarded by 30 keV Ar ion and 30 keV N ion is, the spectra of 30 keV Ar bombarded (observed by Dycor _ quadrupole gas analyzer Model 100, AMETEK, Pittsburgh, PA, USA) show the H, C and O peaks (Yu and others, 2002), but the spectra of 30 keV N bombarded (observed by same Quadrupole mass analyzer) show the CO, O_2, H_2 and H_2O peaks.

One reason why the mass spectrums of onion cell dose not show many peaks is that the sputtered ions from onion cell involve big clusters, having high molecular weight, but the maximum mass-to-charge ratio of the quadrupole is 100.

A mass spectrum will usually be presented as a vertical bar graph, in which each bar represents an ion having a specific mass-to-charge ratio (m/z). The most intense ion is referred to as the base peak. Most of the ions formed in a mass spectrometer have a single charge, so the m/z value is equivalent to mass itself. Mass spectrometers easily distinguish (resolve) ions differing by only a single atomic mass unit (amu), and thus provide completely accurate values for the molecular mass of a compound. The highest-mass ion in a spectrum is normally considered to be the molecular ion, and lower-mass ions are fragments from the molecular ion.

Advantage of study on the sputtering from biological cell is referred to detecting the interaction between cell and bombarding ions to measure quantity and depth of penetrate ions.