速 報

SHORT NOTE

Development of Amorphous Silicon Whisker for Field Desorption Mass Spectrometry

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A new method of the growth of Amorphous Silicon Whisker (ASW) for the field ionization and desorption mass spectrometry is developed. The technical processes are mainly reported. Significant progress has been made in the field desorption mass spectrometry, first introduced by Beckey in 1967¹⁾. The benzonitrile dendritic protrusions (carbon emitter) grown on a 10 $\mu\text{m}\phi$ tungsten wire²⁾ are now widely adopted as a standard emitter of FI and FD mass spectrometry. Beside the carbon emitter, the nickel and cobalt emitters have been developed³⁾. According to our experience, the carbon microneedle emitter has two problems. The first is the long growing time which is between ten to fifteen hours. The second is the strength of the emitter, especially the break down due to electric discharge. The new ASW emitter has been developed in order to satisfy the following two conditions: "*Quick Growth and Never Rupture*".

The ASW growing system is shown in Fig. 1. The method of growing is as follows. 76 $\mu\text{m}\phi$ tantalum wire is spot welded on wire supports. A small amount of gold is deposited by evaporation on the part of tantalum surface on which ASW is wanted to grow. The thickness of gold film is several hundred angstroms. The gold coated tantalum wire with its support is put into

Fig. 1. The ASW growing system.

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Fig. 2 a

Fig. 2 b

Fig. 2 Scanning electron microphotograph (x1000) of ASW emitter.
(a) Before FI, FD operation; (b) After five times FD operation of cholesterol.

a small vacuum chamber and evacuated. The wire is pre-heated by direct electric current (0.45V, 0.95A) for about one minute. The temperature of tantalum wire corresponds to nearly 800°C. The silane gas (SiH_4 , 5% + Ar 95%) is filled in the vacuum chamber up to 100 Torr. The wire is heated by supplying electric current (0.45 V, 0.95 A) in the voltage constant mode of power supply. The temperature of tantalum wire is measured by optical pyrometer, which is nearly 700°C. The ASW grows on the gold coated area from thermally decomposed silane after 1~5 minutes.⁴⁾ No high electric voltage is required.

The growth of ASW is at present explained by the vapor-liquid-solid (VLS) mechanism. In this mechanism, the impurity, gold is used in this case, forms a liquid alloy droplet which has relatively low freezing temperature. Because this liquid droplet occludes preferably the silicon vapor, it becomes super-saturated with silicon, and the whiskers grow by precipitation of silicon from the droplet. Detailed discussions are given in ref. 5. Figures 2a, 2b show the scanning electron microphotographs of the ASW. The average length of the whiskers is about 20 μm . The average diameter is about 0.2 μm .

Fig. 3. (a) FD spectrum of cholesterol;
(b) FD spectrum of oligopeptide ester (Z-Ala-Ala-OCH_3).

The high resolution second order double focusing mass spectrometer (MATSUDA TYPE)⁴⁾ was used for taking mass spectrum. The test of ASW emitter was executed, at first, on FI operation using acetone. The pressure of acetone in the ion source was 4×10^{-6} Torr. The total residual gas pressure at the ion source was nearly 2×10^{-7} Torr and that of the main analyzer was less than 5×10^{-8} Torr. The ionization efficiency of ASW emitter for acetone at the detector was 5×10^{-6} A/Torr (accelerating voltage: 3.0 keV, the main slit width: 0.30 mm). The ASW emitters were qualitatively tested on FD operation. Two examples of mass spectra obtained by ASW emitter are shown. Figure 3a shows the field desorption mass spectrum of cholesterol at 320 mA emitter heating current. The ionization efficiency was 9.0×10^{-11} coulomb/ μ g. Figure 3 b shows the mass spectrum of the oligopeptide ester (*z*-Ala-Ala-OCH₃). The ionization efficiency was 2.2×10^{-11} coulomb/ μ g. Detailed comparisons between ASW and carbon emitter are not yet done. The test of ASW emitter for FD operation is under proceeding.

The ASW grows not only on tantalum wire but also on tungsten wire. The growing process and the characteristics are similar to the case of tantalum. The ASW

emitter is expected to offer a strong and economical ion source for FI and FD mass spectrometry.

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Keywords

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