Letters to the Editor

Mass Spectra of Oxygen–Nitrogen Plasma by Introduction of Organic Solvent in a Microwave Induced Plasma Mass Spectrometer

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An inductively coupled plasma mass spectrometer (ICP/MS) has been used in routine analyses of ultra trace elements.^{1,2} Unfortunately, calcium, magnesium and chromium are generally difficult to analyze by the ICP/MS, because their mass numbers (m/z) coincide with the m/z of argon and its related compounds and with the m/z of the compounds^{3,4} of carbon. Furthermore, the m/z spectrum of magnesium is interfered with by C_2^+ and C_2H^+ radicals which are formed when organic solvents are used.⁵ Argon–oxygen mixed gas (98%:2%) is used in the ICP mass spectrometry when organic solvents are introduced, because this mixed gas can remove graphite material which builds up on the orifice of an interface of ICP/MS. But this mixed gas can not lessen the interference caused by carbon related compounds. To find something which will lessen the interference is a key issue in the analyses with the ICP/MS.

Since the microwave induced plasma mass spectrometer (MIP/MS)^{6–8} can be operated by nitrogen, the interference originating from argon was eliminated perfectly. But, the nitrogen plasma also has a limitation for applying to many kinds of samples. So we studied another plasma formed by the use of oxygen as a carrier gas.

Experimental

A Hitachi Model P-7000 MIP/MS was used for this experiment. The MIP, as reported already, is operated at a frequency of 2.45 GHz, the mass spectrometer is constructed with a quadrupole mass spectrometer. Pure water, acids and ethanol used were the same grade as semiconductor manufacturing ones. The standard solutions were commercial reagents prepared by E. Merck; these were diluted with nitric acid. The instrument was installed in a clean room to prevent environmental contamination.

Nitrogen is the plasma forming gas, and oxygen (100%) or oxygen–nitrogen (20%:80%) mixed gas is used as a carrier gas for each sample.

Results and Discussion

Figure 1 is the MIP/MS spectra for the nitrogen plasma whose carrier gas consists of the oxygen–nitrogen mixed gas. Neither C_2^+ nor C_2H^+ signal peaks corresponding to m/z of 24 and 25 were observed, even though ethanol was introduced into the plasma. It is thus not startling that ArC^+ ($m/z=52$) is not observed. Then the measurement of Mg^+ ($m/z=24$) or Cr^+ ($m/z=52$) could be carried out without any interference from the organic

Fig. 1 Background spectra of ethanol by oxygen–nitrogen plasma.

Fig. 2 Mass spectra of magnesium and chromium in ethanol solution: Mg, 10 $\mu\text{g/l}$; Cr, 10 $\mu\text{g/l}$.

Fig. 5 Mass spectra of YO^+ by 100% oxygen carrier gas plasma.

Fig. 3 Background spectra of pure water by nitrogen plasma.

Fig. 6 Mass spectra of chromium in ethanol solution.

Fig. 4 Background spectra of ethanol by 100% oxygen carrier gas plasma.

solvent, as shown in Fig. 2.

As Fe^+ ($m/z=56$) coincided with N_4^+ ($m/z=56$) in the nitrogen plasma, it was difficult to distinguish between Fe^+ and N_4^+ , as shown in Fig. 3. This $m/z=56$ was greatly lessened when 100% oxygen was the carrier gas, as shown in Fig. 4. Then the measurement of Fe^+ could be performed easily.

In this plasma, Y^+ ($m/z=89$), YC^+ ($m/z=101$) and YN^+ ($m/z=103$) were not found, even though an ethanol was used. Only YO^+ ($m/z=105$) appeared as shown in Fig. 5. In contrast with these phenomena, species of CrO^+ ($m/z=68$), CrC^+ ($m/z=64$) and CrN^+ ($m/z=66$)

seemed not to be formed in this plasma, and these signals could not be detected. But Cr^+ ($m/z=52$) was clearly recognized, as shown in Fig. 6.

In conclusion, when oxygen was applied, as the carrier gas, the signals originating from carbon (or an organic solvent) and nitrogen, such as C_2^+ , C_2H^+ , N_4^+ and ArC^+ , were minimized.

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