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Hydrogen, Carbon and Oxygen Analysis in Thin Films Using Extremly High Vacuum SIMS

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Extremely high vacuum instrument for secondary ion mass spectrometry is developed to perform trace H, C and O analysis in thin films and interfaces. The instrument is equipped with a 20 K-cryopanel surrounding samples to pump out contaminating residual gases. The sample chamber pressure of 2×10^{-9} Pa is achieved during analysis. Detection limits for H and O in thin film analysis are 1/100 that obtained by conventional instruments.

1. Introduction

Evaluation of H, C and O which are contaminating elements in device fabrication is indispensable for recent thin layer materials. Secondary ion mass spectrometry (SIMS) is a highly sensitive depth profiling method for impurities in solids. However, the actual detection limits are not determined by the sensitivity but by the instrumental backgrounds. Serious backgrounds arise from residual gases in the sample chamber vacuum in H, C and O analysis. Since SIMS detects the ions from the outermost layer of the sample surface, residual gas molecules sticking to the surface also constitute secondary ions. Thus, the detection limits for H, C and O are directly influenced by the partial pressure of residual gas species such as H_2O, CO, and hydrocarbons. In this situation, the removal rate of the sample surface is also becomes an important factor. Very high sputtering rates, as fast as several tens of nanometer per second, has been used to achieve low background levels.

On the other hand, a moderate sputtering rate is needed to measure the impurity distribution in thin layers and interfaces. Therefore, the pressure of the contaminating gas in the SIMS sample chamber should be reduced to as great an extent as possible. We have already designed a cryopanel pumping system and succeeded in reducing the residual gas pressure in a SIMS instrument, Cameca IMS-3F.[1] In order to further improve the vacuum condition, we have recently developed an extremely high vacuum (EHV) SIMS instrument using the cryopanel pumping system.[2] In this paper, we present the vacuum characteristics of the instrument and the application of the thin layer analysis.

2. Instrument

The vacuum system of a quadrupole-based secondary ion mass spectrometer, Atomika A-DIDA 3000, is modified to obtain an extremely high vacuum. The sample chamber is pumped with a 220 l/s ion pump. In addition, a cryopanel made of copper is installed in the chamber. The panel has a cylindrical shape which fully surrounds the sample manipulator. The panel is cooled to less than 20 K by using a closed-cycle helium refrigeration unit. At this temperature, any contaminating residual gases in the sample chamber can be fully condensed on the panel surface. To reduce thermal radiation from the sample chamber wall, a second panel is placed between the 20 K-panel and the chamber wall. The second panel temperature is estimated to be 50-80 K. This reduces the thermal load on the 20 K-cryopanel to 1/200. By means of these cryopanels, the sample chamber itself acts as a cryopump.

Another improvement over the original instrument consists of removing or replacing

nonbakeable components. For example, a metal-sealed gate valve is used for the sample transfer system and for the primary ion column. This allows baking the whole system at 250°C.

Two types of ion sources are employed for depth profiling. One is a duo-plasmatron ion source for O_2^+ primary ion production in positive secondary ion detection. The other is a thermal surface-ionization source for Cs^+ primary ion production in negative secondary ion detection. Primary ion beams are raster scanned to form a flat crater bottom on the sample surface. Secondary ions are extracted from the central region of the raster scanning area by electronic gating.

The operating pressure of the ion source is $1x10^{-4}$ Pa for the duo-plasmatron ion source and $3\text{-}10x10^{-6}$ Pa for the Cs ion source. The primary ion column is differentially pumped with a 170 l/s turbomolecular pump.

3. Vacuum Characteristics

The total pressure in the sample chamber was measured using a modulating ion current gauge. After a 24 h bakeout period at 250°C, the base pressure in the sample chamber reached $1x10^{-8}$ Pa by pumping with the ion pump. Under cryopanel operation, the base pressure was lowered to $2x10^{-9}$ Pa. The pressure did not changed during Cs^+ ion source operation. Even during duo-plasmatron ion source operation, the pressure remained $3x10^{-9}$ Pa.

The effect of the cryopanel on the residual gas backgrounds is evaluated under O_2^+ primary ion bombardment. Figure 1 shows the background H^+ intensity variation for Si under O_2^+ bombardment after the refrigeration process started. The temperature of the refrigerator cold head measured by a thermocouple is also shown in the figure. When the cryopanel was not in operation, a high background H^+ intensity was produced due to gases flowing into the chamber from the ion source. The residual gas pressure in the sample chamber was about $1x10^{-7}$ Pa. With the decrease in the cold head temperature, a step-like pump-down curve is obtained. The step corresponds to the condensation of the residual gas on the cryopanel. The H^+ background signals were lowered to 1/100 of the initial level when the

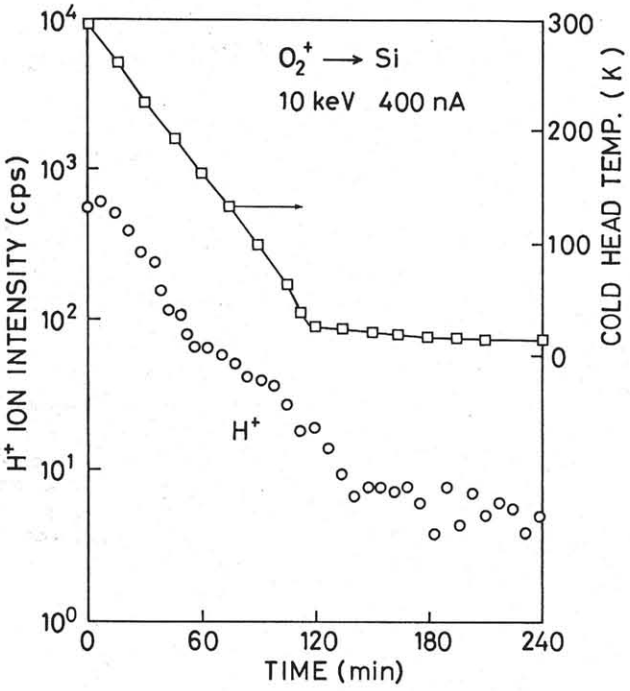

Fig. 1 Variation in H background signal under O_2^+ bombardment after beginning of refrigeration process.

cryopanel was in full operation. During this time residual gas pressure reached $3x10^{-9}$ Pa. This result demonstrates the high efficiency of the cryopanel in residual gas evacuation.

4. Thin Film Analysis

A low background vacuum is useful for depth profiling of H, C and O in thin films and interfaces because a moderate sputtering rate at a lower ion energy and a lower current density is required for this purpose. On the other hand, the residual gas background level is inversely proportional to the primary ion current density.[3] Therefore, higher sputtering rates have been indispensable for achieving low detection limits,[1,2] although this has been accomplished at the expense of depth resolution.

The O background levels under 3.0 keV Cs^+ bombardment are compared between the EHV condition with the cryopanel in operation and the conventional vacuum condition in Fig. 2. The sample was 100 keV O^+-implanted Si to a dose of $1x10^{15}$ ions/cm^2. The sputtering rate was lowered to 0.02 nm/s by using a 3 keV Cs^+ beam with a current of 30 nA. In spite of the very low

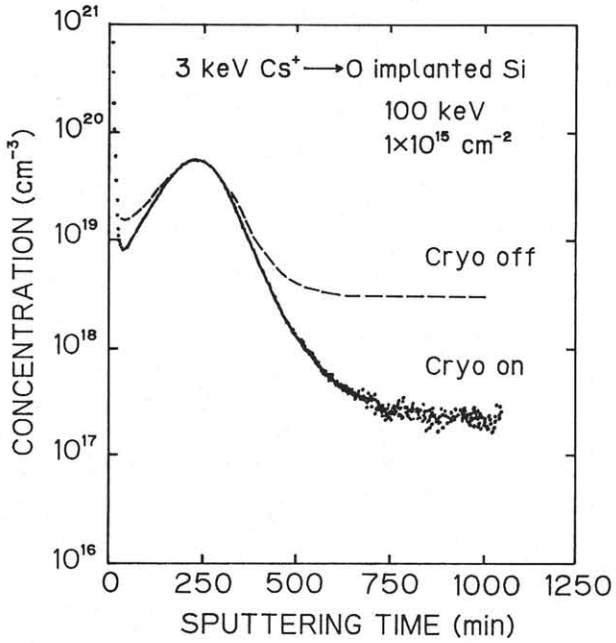

Fig. 2 Depth profiles of O-implanted in Si measured by weak Cs^+ beam. The profile obtained during the cryopanel operation and that obtained without cryopanel operation are compared.

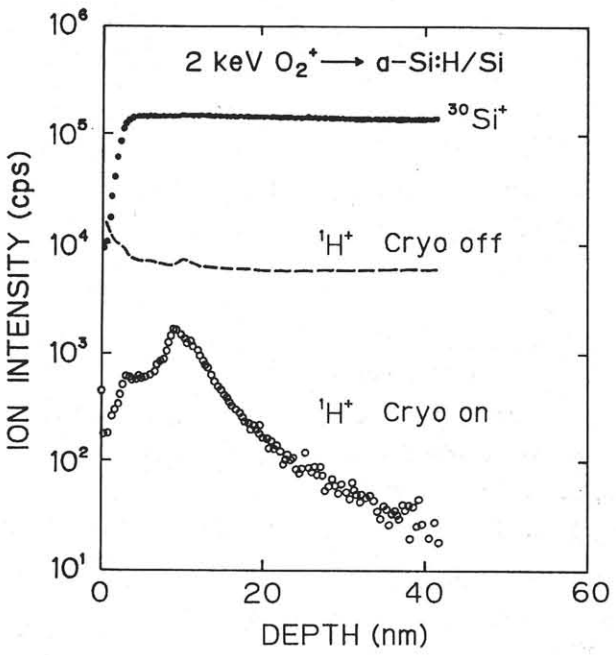

Fig. 3 Depth profile of H in 10 nm a-Si layer on Si measured by weak O_2^+ beam.

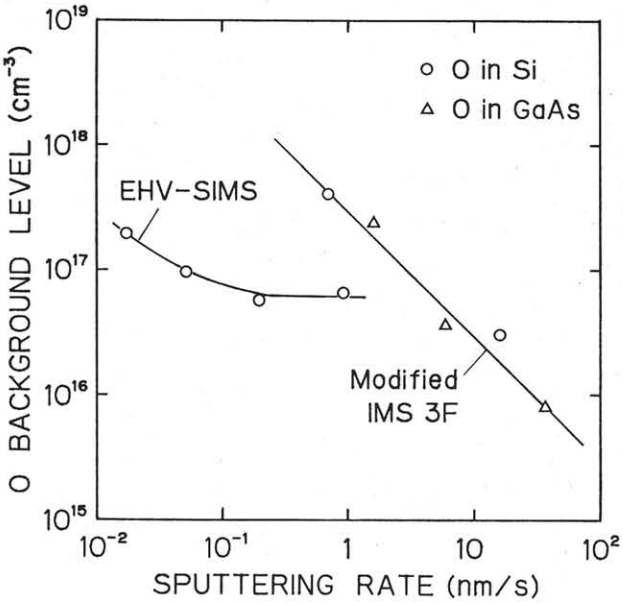

Fig. 4 Relationship between O background level and sputtering rate. Data obtained by the EHV-SIMS instrument are compared with those obtained by the cryopanel-equipped IMS-3F.

sputtering rate, a low O background of about 2×10^{17} atoms/cm^3 is obtained. As shown in the figure, this level is 1/20 of that obtained without cryopanel operation.

Figure 3 shows an example of thin film depth profiling for H in an amorphous Si layer 10 nm in thickness grown on a Si substrate. To evaluate such thin film, a primary O_2^+ energy of 2 keV and a sputtering rate of 0.01 nm/s were employed. Without cryopanel operation, the H profile in the layer could not be obtained, since the H background level due to the residual gas flowing from the ion source was higher than the H concentration in the amorphous Si under the low sputtering rate. In contrast to this, the cryopanel operation made it possible to obtain the H profile by reducing residual-gas-produced signals. The H accumulation at the interface between the amorphous Si layer and Si substrate is clearly measured. The H profile tail in the Si substrate can be explained by mixing of the sample atoms due to primary ion bombardment.

5. Backgrounds in EHV Condition

Figure 4 summarize the relationship between the O background level and the sputtering rate under Cs^+ bombardment. The data obtained by the EHV-SIMS instrument are compared with those obtained by the cryopanel-equipped Cameca IMS-3F.[1,2] The superiority of the EHV-SIMS instrument to the modified IMS-3F is evident in the low sputtering rate region. In this region, a background level 1-2 orders of magnitude lower is

achieved by the EHV-SIMS instrument. It should be noted, however, that in the higher sputtering rate region, the modified IMS-3F yields lower background levels.

This is due to the presence of non-residual-gas-produced backgrounds. These backgrounds are released from the adsorbed layer or from contamination on electrode surfaces in the vicinity of the sample.[4,5] These contaminant layers are bombarded by backscattered primary ions or by sputtered target atoms and produce stray background ions. In the ion microscope type instrument, IMS-3F, the stray ions can be rejected by using secondary ion optics which preferentially extract ions from a central region of the primary ion rastering area. On the other hand, in the ion microprobe type instrument, A-DIDA, secondary ion optics accept ions from a larger area. Ions from the vicinity of the sample as well as secondary ions from the sample surface are allowed to pass into the mass spectrometer. Thus, in the UHV-SIMS instrument, the O detection limit is determined by these stray O ions rather than the residual gases. For other residual gas-related elements, the detection limits are also determined by stray ions in the EHV condition. The background levels for H and C are 5×10^{16} and 3×10^{16} atoms/cm^3, respectively.

6. Conclusion

We have shown that the 20 K-cryopanel pumping system is effective in evacuating residual gases during SIMS analysis. In thin film analysis using a moderate sputtering condition, residual-gas-produced background levels are reduced to 1/100 of that in conventional instruments. In the higher sputtering rate region, however, non-residual-gas-produced backgrounds are found to limit the detection of H, C and O.

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