T060609

First Step to TDS
Thermal Desorption Spectrometer

Thermal Desorption Spectrometer detects the desorbed gas from a sample using the quadrupole mass spectrometer, when a sample is heated in the vacuum chamber.
The sample is heated from the room temperature to 1200 ℃ by absorbing the infrared rays from a halogen lamp.

The instrument detects gas in high sensitivity that are desorbed in this temperature range.
The instrument can detect the order of 1.0×10^{13} molecules on the sample.

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1 What is TDS

1.1 Brief Overview

TDS is the abbreviation of Thermal Desorption Spectroscopy. It heats a sample in the vacuum chamber and detects gas that are desorbed every temperature from a sample.
The desorbed temperature of gas depends on the adsorption energy to a sample of the molecules. Molecules that have a strong adsorption energy desorbed at high temperature, and molecules that have a weak adsorption energy deosrbed at low temperature.
A mass spectrometer is used for the detector generally. We use a quadrupole mass spectrometer as the detector in our TDS instrument.
The thermal desorption curve that has X-axis as the temperature and Y-axis as the ion intensity as shown in figure 1 is provided with this composition. One thermal desorption curve at every mass number is porovided.

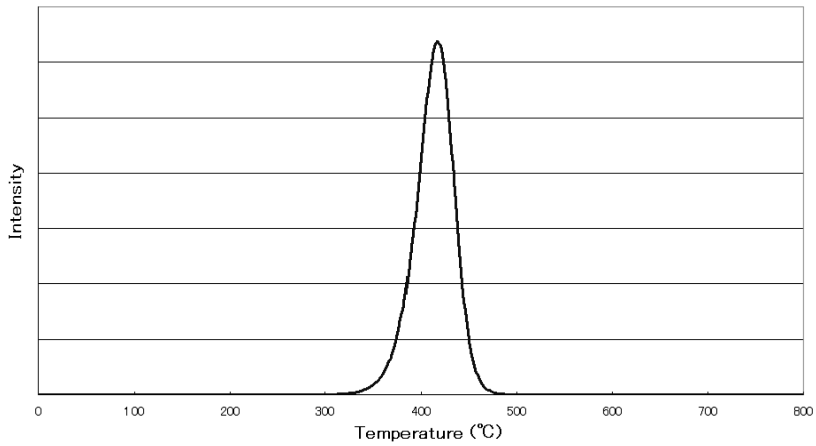

Figure 1: Thermal Desorption Curve

Two kinds of the measurement method with the mass spectrometer show different data. One is the MID (Multi Ion Detection)[1] data, and another is the Scan data.

It is possible to confirm the signal of which mass number is appeared at which temperature in the Scan data. The Scan data are supeiror than the MID data in this view point. But the measuring gain of every mass number cannot be adjusted separately, because one channel is used for measuring signal of all scanned mass number. Signal of all mass number are measured at the same gain, and then the small signal that is hidden in the large signal cannot be detected.
Therefore, the Scan data is used for searching an outline of desorbed gas from a sample. It is necessary to get the MID data for analyzing a sample in detail.

1.2 MID Data

The signal change of the specified mass number that has X-axis as the temperature and Y-axis as the ion intensity is called as the thermal desorption curve. Multiple channels are set to different mass number of a mass spectrometer, and signals of mass number

[1] It is called also as the SIM(Selected Ion Monitor).

are measured while heating a sample. The example of the thermal desorption curve of $m/z = 18$ is shown in figure 2.

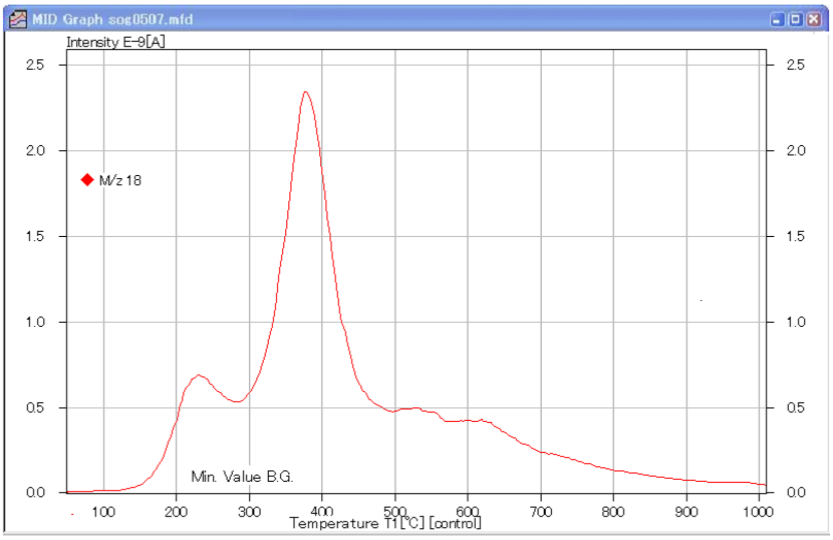

Figure 2: Thermal desorption curve of $m/z = 18$

The MID data is the thermal desorption curves of multiple mass number that are measured at the same time (Figure 3∼Figure 4). Each mass number is set to separate channel and the signal of each channel is measured. The measuring gain of each channel can be adjusted separately, then the dynamic range of the measurement is large and the accuracy of the measurement is excellent.
The number of the thermal desorption curves that can be measured at the same time is confined to the number of channels of the quadrupole mass spectrometer.
It is possible to analyze what kinds of molecules are desorbed and the amount of molecules with setting appropriate mass number to each channel.

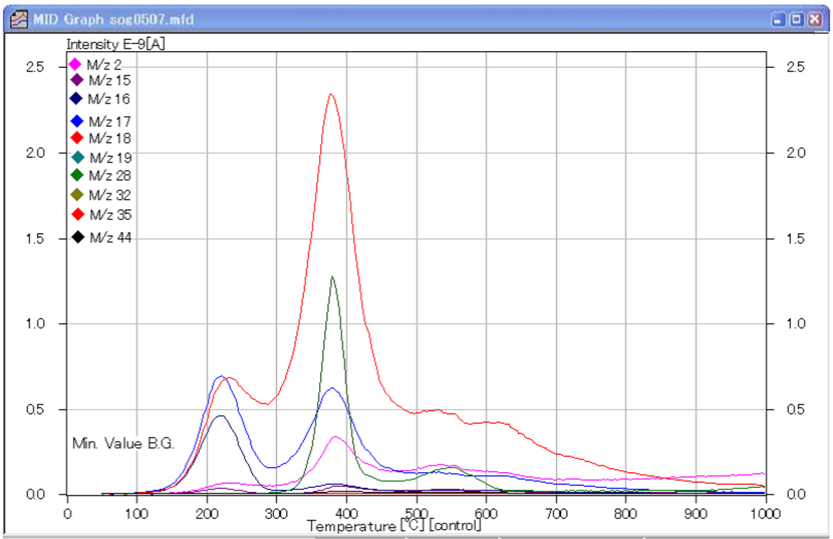

Figure 3: Multiple thermal desorption curve of 10 channels

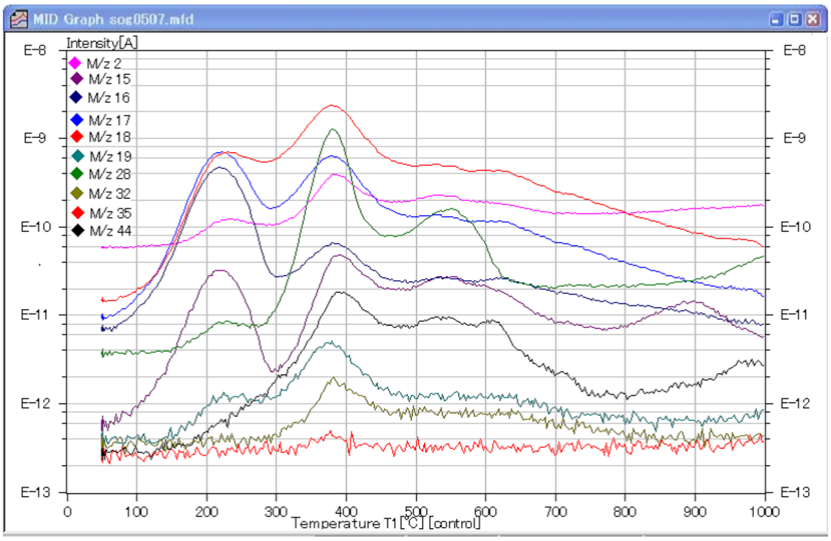

Figure 4: Multiple thermal desorption curve of 10 channels (Log display)

1.3 Scan Data

The Scan data is the thermal desorption curves measured for all mass number that has X-axis as the temperature and Y-axis as the ion intensity.
All mass range is scanned and signals of every mass number are measured using one channel of the quadrupole mass spectrometer while heating a sample.
The mass range of the quadrupole mass spectrometer in our TDS system is 1 to 200[2].
The example of the three dimensional display of the Scan data is shown in figure 5.

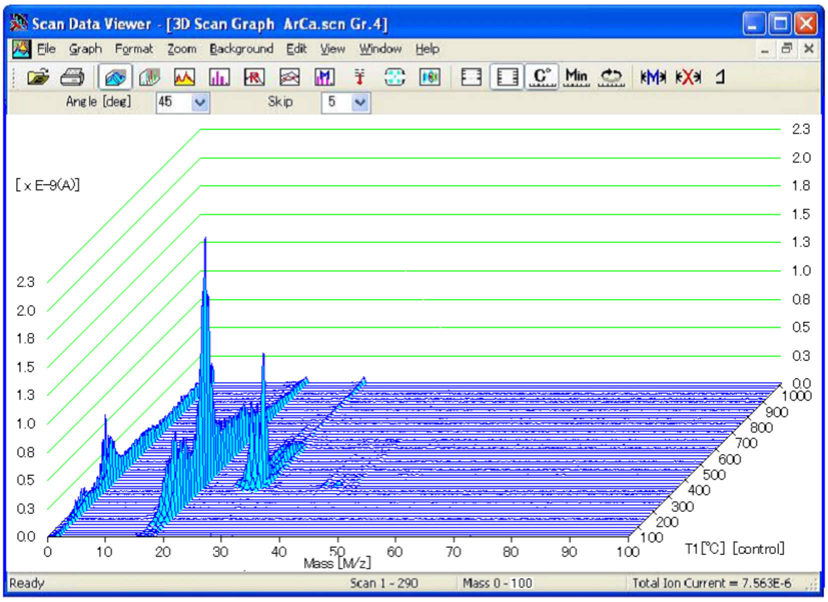

Figure 5: Three dimensional display of the Scan data

[2]1〜1024 of the mass range is available in option.

2 Composition of TDS System

2.1 Appearance and Composition

The appearance of EMD-WA1000S/W that is the main model of our TDS system is shown in figure 6.

Figure 6: Appearance of EMD-WA1000S/W

The right part of the instrument is the operation unit, in which the computer unit is located.
The left part of the instrument is the measurement unit, which is composed of four blocks.

1. The loadlock chamber is located on the upper left side of the measurement unit that is for loading a sample into the main chamber.
2. The main chamber is located on the upper right side of the measurement unit, in which the quadrupole mass spectrometer is installed. The controller of the quadrupole mass spectrometer is located in the operation unit.
3. The vacuum control system for the main chamber and the loadlock chamber, and operation panel are located in the lower left side of the measurement unit.
4. The I.R.lamp for heating a sample and the temperature controller are located in the lower right side of the measurement unit.

2.2 Mechanism of Sample Heating

A sample is heated by absorbing the infrared rays that is radiated from a halogen lamp. The blockdiagram of the heating mechanism is shown in figure 7.

- A halogen lamp is located in the light collector, of an ellipsoid mirror structure.

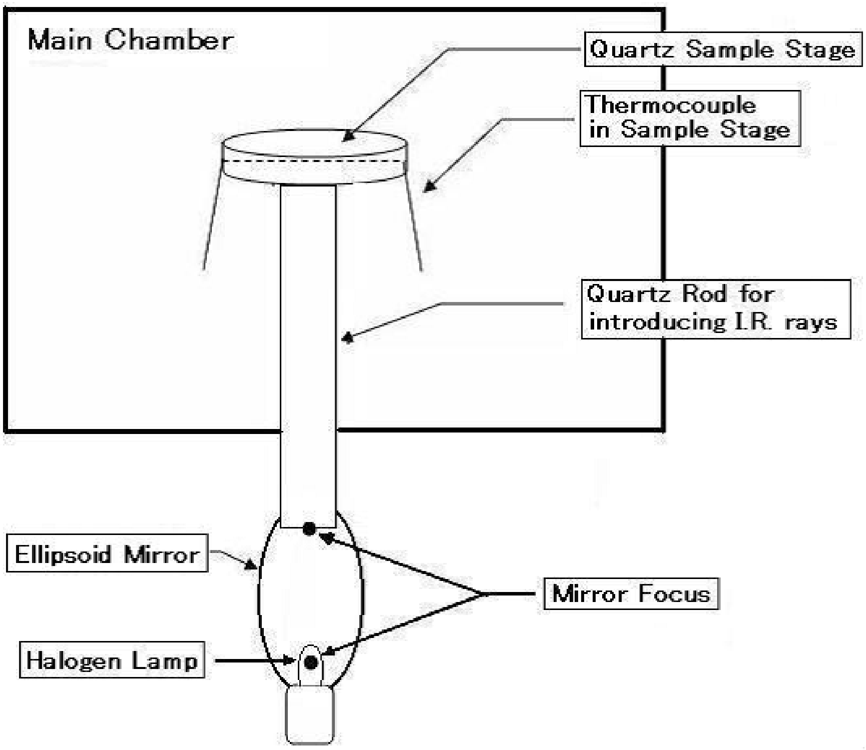

Figure 7: Blockdiagram of Sample Heating Mechanism

- The light collector has two focuses. The halogen lamp is located at one of focus.
- The quartz rod that has a diameter of 2cm is inserted in the light collector at the major axis direction, and the edge of the quartz rod is located in another focus of the light collector. The infrared rays from the halogen lamp focus into the quartz rod.
- The infrared rays that focus into the quartz rod go through the rod by total reflection to another edge of the rod.
- The quartz rod is inserted into the main chamber at a buttom. Therefore, the infrared rays are conducted into the main chamber from the light collector located outside of the chamber. The advantage of this construction is that a lamp exchange is easy and no generation of gas by the lamp.
- The conducted infrared rays go to the sample stage that is located on the top of the rod. The sample stage is made of a quartz. The infrared rays go through the sample stage, and then irradiate a sample on the sample stage.
- A sample is heated by absorbing the infrared rays.
- A diameter of the sample stage is 3cm, and a diameter of the quartz rod is 2cm. Therefore, only the central part of the sample stage is heated. Consequently, a sample should be located in this part.
- A sample that does not absorb the infrared rays cannot be heated. But the indirect heating of the sample is possible using the black sample stage.

2.3 Mechanism of Temperature Measurement

Two kinds of temperature measuring mechanism are equipped in EMD-WA1000S/W.

2.3.1 Measurement by Sample Stage's Thermocouple

One of temperature measurement is performed by the thermocouple in the sample stage. The thermocouple is called as the sample stage's thermocouple. This thermocouple is located between the quartz rod and the sample stage as shown in figure 8.

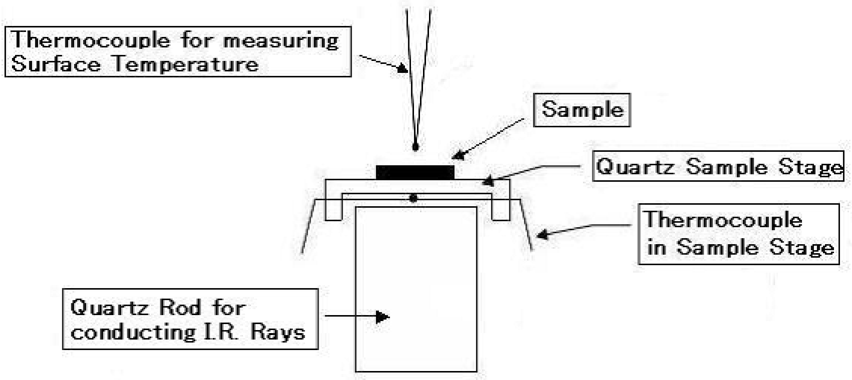

Figure 8: Quartz rod and Sample stage

2.3.2 Measurement by Upper Thermocouple

Another temperature measurement is performed by touching the thermocouple to a sample's surface. The thermocouple is called as the upper thermocouple for measuring surface temperature. The thermocouple for measuring surface temperature is mounted on the top of a linear motion drive that is mounted on the center of the main chamber's upper flange. It can be pulled down for touching with the sample's surface as shown figure 8.

2.3.3 Temperature Control

Temperature control is normally performed with the temperature of the sample stage's thermocouple. Temperature control with the temperature of the upper thermocouple is possible by changing a thermocouple's wiring.

2.3.4 Temperature Calibration

There is a limit with an aspect of accuracy in measuring the temperature in vacuum by a thermocouple.
The temperature calibration method was developed using the comparative chart with the temperature of a two kinds of TDS signal. The desorbed temperature of a molecule in some circumstances that has been measured accurately and the TDS signal's peak temperature in our TDS system that is measured by the sample stage's thermocouple are compared in this calibration method.

2.4 Mechanism of Signal Detection

A quadrupole mass spectrometer is used for detecting the desorbed gas in our TDS system. The quadrupole mass spectrometer is installed in the main chamber as shown in figure 9.

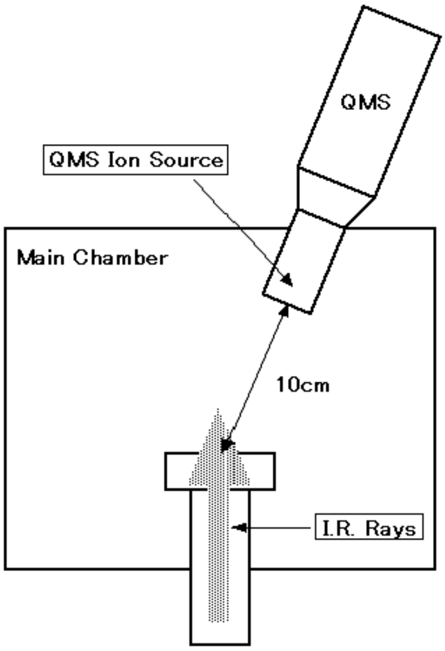

Figure 9: Installing position of Quadrupole Mass Spectrometer

The ion chamber of the quadrupole mass spectrometer is located above the sample stage and close to the sample stage in about 10 cm. Therefore the desorbed molecules that have the long stay time on a chamber inside wall such as polymer organic or metallic substance can be introduced to the ion chamber at a high rate.

2.5 Sample Transfer Mechanism

A sample is transferred to the sample stage of the main chamber through the loadlock chamber. As a result, a sample can be inserted into the main chamber without stopping the vacuum system of the chamber.

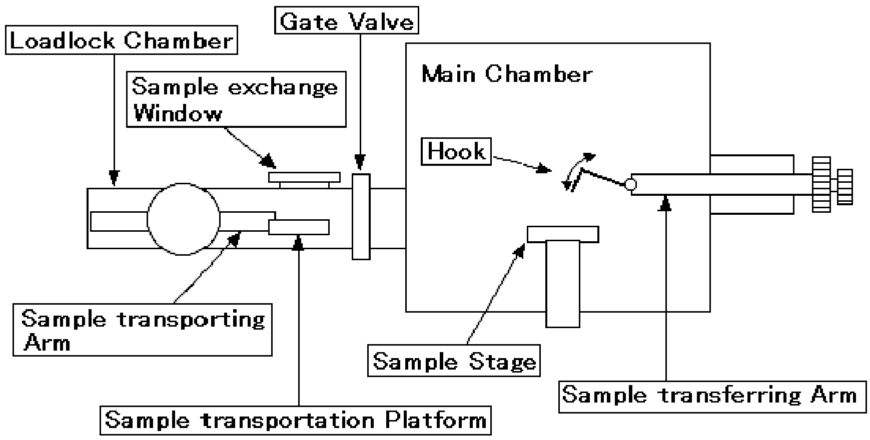

Figure 10: Mechanism of sample insertion and transfer

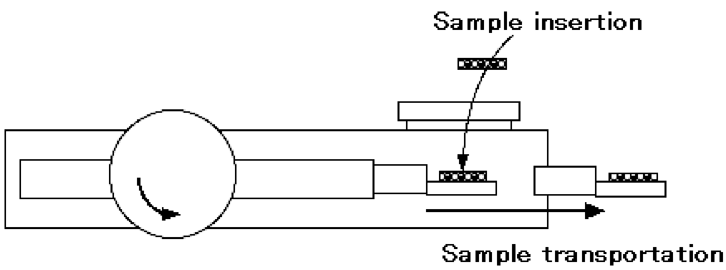

Figure 11: Mechanism of sample transport

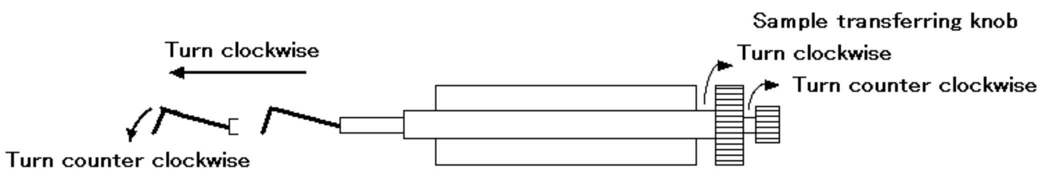

Figure 12: Mechanism of sample transfer

2.5.1 Sample insertion

Sample insertion is performed as following procedure.

1. Vent the loadlock chamber to the atmospheric pressure by Nitrogen gas. Then open the sample exchange window and insert a sample on the sample transportation platform.

2. Close the sample exchange window, and then exhaust the loadlock chamber to the pressure of 10^{-3} Pa order (about 10^{-5} Torr order). The gate valve that is located between the loadlock chamber and the main chamber can be opened at this pressure level.

3. After opening the gate valve, move the sample transporting arm into the main chamber by rotating the knob that is located in front of the loadlock chamber. And move the sample transportation platform toward the sample stage until it comes in contact with the sample stage.

4. Move the sample transferring arm until the hook is located to the left side of a sample on the sample transportation platform, by rotating the large knob that is located right side of the sample transferring mechanism.

5. Lower the hook so as to transfer a sample to the sample stage from the sample transportation platform, by rotating the small knob that is located right side of the sample transferring mechanism.

6. Transfer a sample to the center of the sample stage from the sample transportation platform by rotating the small knob.

7. Raise the hook by rotating the small knob and then retract the sample transferring arm by rotating the large knob. The sample transferring arm should be retracted completely above the sample stage.

8. Measurement can be started after the sample transporting arm is retracted into the loadlock chamber and the gate valve is closed.

2.5.2 Sample removal

Sample removal is performed as following procedure that is the reverse procedure of the sample insertion.

1. Open the gate valve, and then move the sample transporting arm into the main chamber until the sample transportation platform comes in contact with the sample stage.

2. Move the sample transferring arm and locate the hook to the right side of a sample on the sample stage, and then lower the hook.

3. Move the sample transferring arm and transfer a sample to the sample transportation platform from the sample stage.

4. Raise the hook and keep its position for next sample insertion.

5. Retract the sample transporting arm into the loadlock chamber, and then close the gate valve.
6. Flow Nitrogen gas into the loadlock chamber to vent the chamber to the atmospheric pressure.
7. Open the sample exchange window, and then remove a sample.

3 Sample

3.1 Ordinary sample shape and size

The ordinary sample shape is a plate and the size is 10 × 10 mm and 1 mm in thickness. This sample shape and size is decided because the instrument is developed for measuring the desorbed gas from Silicone wafer tip in the semiconductor industry.

3.2 Samples that do not absorb Infrared rays

Samples that do not absorb infrared rays can be heated and measured using the black sample stage. In this case, the black sample stage is heated by absorbing infrared rays, and then a sample is heated indirectly by the heat radiation and conduction from the sample stage. The material of the black sample stage is that has the characteristics of lower gas adosorption.
We have the stage change mechanism that is enable to change over between the transparent sample stage and the black sample stage without opening the main chamber.

3.3 Powder Samples

Powder samples can be measured using the sample boat. The material of the sample boat is a quartz and the size of the boat is 10 × 10 mm and 2 mm in thickness with a 10 mm hole. It is called as the transparent sample boat.
We have another sample boat of the black quartz material. It is called as the black sample boart. The transparent sample boat is adequate to measure a sample that absorbs infrared rays. On the contrary, the black sample boat is adequate to measure a sample that does not absorb infrared rays.

Figure 13: Sample Boat