# Evaluation of contamination of power semiconductor device wafers by total reflection X-ray fluorescence spectrometer

**Hiroshi Kohno***

## 1. Total reflection X-ray fluorescence spectrometry (TXRF)

TXRF spectrometers are widely used as evaluation instruments for measuring contamination in the semiconductor fabrication process. This is mainly because the TXRF technique allows non-destructive analysis for almost all elements (Na~U) in the periodic table.

TXRF spectroscopy is possible due to the property of X-rays as they irradiate a flat surface, such as a Si wafer or glass. Each material has a unique critical angle. If the incident angle of the X-rays is above the critical angle, the X-rays penetrate the surface deeply; if the incident angle is smaller than the critical angle, the X-rays are reflected off the surface totally, or what is termed total reflection. In the total reflection condition, any fluorescence X-rays will occur only from contamination on the surface and the surface material will not add background noise to the measurement. The penetration depth of the incident X-rays under the conditions of total reflection is theoretically around 5 nm, and thus TXRF is classified as a surface analysis. TXRF's outstanding S/N ratio makes the detection of contamination on the surface of a sample possible with high sensitivity. Figure 1 shows the comparison of X-rays penetration depth under the high incident angle condition and at an incident angle where the TXRF condition exists (Called TXRF condition hereafter).

## 2. Metal contamination and influence on device characteristics

Contamination measured by TXRF is mainly metal contamination. There are two types of metal contamination, namely alkali metal and transition metal, and their impact on device characteristics is different (Table 1).

The degree of metal contamination impact on device characteristics depends on the type of device. As CIS (CMOS image sensor), DRAM, flash, etc. are sensitive to metal contamination, a control of $1 \times 10^9$ atoms/cm$^2$ or lower is strictly applied to them. An upper limit of contamination of approximately $1 \times 10^{10}$ atoms/cm$^2$ is commonly applied to logic devices (Fig. 2). Metal contamination evaluation and control standards for SiC

power devices are being established since these next generation power devices utilize a new substrate. For the moment, the evaluation standard currently adopted for Si logic devices is also being applied to SiC power devices.

## 3. Comparison between other measurement methods and TXRF

Besides TXRF, Vaper phase decomposition ICP mass spectrometry (VPD-ICP-MS), atomic absorption spectrometry (AAS), and lifetime measurement are available for the evaluation of metal contamination. Table 2 shows a comparison between TXRF and these other measurement methods. With TXRF, sample pretreatment is unnecessary (non-destructive), mapping measurement is possible, a high-level operator is not required, and it is easily adaptable to automation protocols such as GEM300, GEM200, etc. In contrast, VPD-ICP-MS and AAS require decomposing and condensing oxide films by hydrofluoric acid. It is difficult to apply VPD-ICP-MS to SiC substrate because SiC substrate is chemically stable with hydrophilic surface. Since this reason, TXRF is going to be widely used in the SiC process.

## 4. Usage of TXRF in the semiconductor process

TXRF is used in all processes of semiconductor fabrication. It is especially useful when a new fabrication line is started up. Clean room atmosphere, contamination from fabrication instruments, etc. are evaluated by the TXRF at this stage, and it contributes to early identification of the point at issue of the line and in evaluation of the stabilization of the fabrication process. It is also used, even after startup of the line, for the evaluation of the fabrication instruments before and after maintenance. Moreover, quite a few users employ TXRF as part of an automation system for quality control.

The most significant feature of TXRF is the ability to map the distribution of contamination on the total surface of the wafer by systematically changing the measurement position. TXRF can uniquely detect metal contamination located partially on the wafer surface. On the other hand, the chemical analysis method represented by VPD-ICP-MS carries out the sample pre-treatment, which is decomposition and recovery of the contamination on the total area of the sample by hydrofluoric acid. The measurement result is,

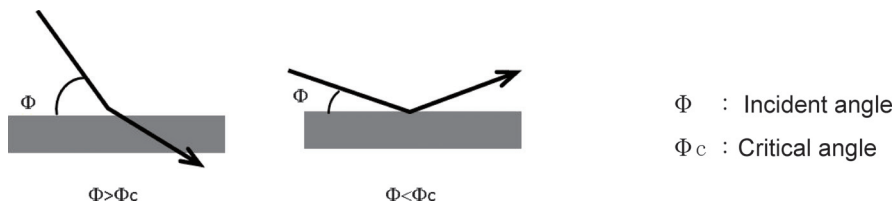* SBU Thin-Film Device Group, X-ray Analysis Division, Rigaku Corporation.

Φ : Incident angle
Φc : Critical angle

**Fig. 1.** High incident angle condition and TXRF condition.

**Table 1.** Relation between type of metal contamination and its impact.

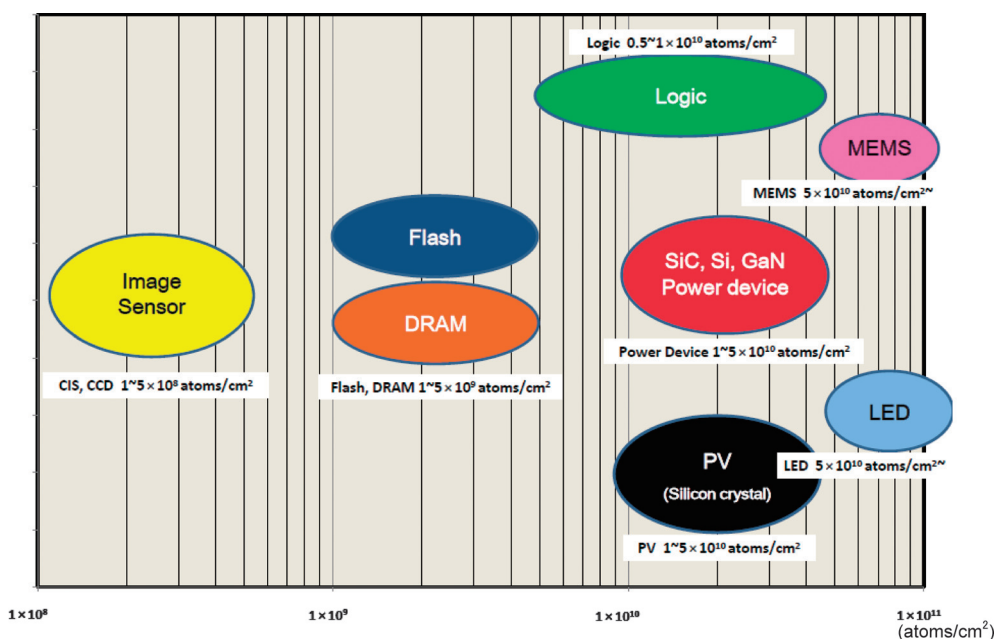| Type of metal contamination | Impact on device characteristics |
|---|---|
| Alkali metal | Gate insulator breakdown<br>Shift of gate insulator threshold voltage |
| Transition metal | Junction leak current<br>Carrier life time degradation<br>Growing of OSF (oxidation induced stacking fault)<br>Gate insulator breakdown |



**Fig. 2.** Semiconductor device and metal contamination control levels.

**Table 2.** Comparison table of analysis methods for metal contamination.

| | TXRF | VPD-ICP-MS | AAS | Lifetime |
|---|---|---|---|---|
| Measurement depth | Surface | Surface | Surface | Bulk |
| Element analysis | Possible (Na-U) | Possible (Except for some) | Possible (Except for some) | Impossible |
| Sample pretreatment | Unnecessary | Necessary | Necessary | Unnecessary (Except for some) |
| Throughput | Average | Slow | Slow | Fast |
| Detection limit | $1 \times 10^9$ atoms/cm$^2$ | $1 \times 10^7$ atoms/cm$^2$ | $1 \times 10^7$ atoms/cm$^2$ | $1 \times 10^9$ atoms/cm$^2$ |
| Mapping | Possible | Impossible | Impossible | Possible |
| Operation | Easy | Need skill | Need skill | Easy |
| Automation | Available | Difficult | Difficult | Available |

therefore, an average of the total area of the wafer. For example, a measurement of $1\times10^{10}$ atoms/cm$^2$ of Fe contamination detected at one spot on the wafer as the result of a TXRF mapping measurement would be measured as $1\times10^8$ atoms/cm$^2$ with VPD-ICP-MS since the measurement only tells you contamination averaged over the whole wafer. This is important to remember when comparing measurements from the two techniques.

## 5. Metal contamination evaluation of SiC power devices

To date, the quality improvement of SiC wafers has mainly been aimed at reduction of macro defects, such as micro-pipes or stacking faults. Micro defects such as metal contamination or dislocation, have not been considered. But SiC wafers having an equivalent quality to Si wafers is starting to be required since SiC power devices need high performance and stable operation. For this reason, metal contamination evaluation of SiC wafers is now important. But as SiC wafers have good chemical stability and are not suitable for surface contamination recovery by use of the chemical solution technique (VPD: Vapor Phase Decomposition), metal contamination analysis by VPD-ICP-MS has generally not been applied to SiC wafers. TXRF, which easily enables non-destructive and highly sensitive metal contamination analysis, is attracting interest.

### 5.1. Metal contamination analysis of SiC power devices by TXRF

The TXRF spectrum of a SiC wafer is almost identical to that of a Si wafer (Fig. 3). Consequently, detection limits and repeatability statistics are the same between SiC and Si wafers.

When a crystalline substrate is involved in a TXRF measurement, it is always important to avoid X-ray diffraction events. SiC has many different polymorphs but there are two hexagonal polytypes, 4H and 6H, which are particularly important. Because of the different polytypes, taking into account the X-ray diffraction events is much more complicated than for <100> Si wafers. The polytype 4H is used for power devices and 6H is used for high frequency devices

(substrate for GaN).

### 5.2. Problems caused by diffraction X-rays

Since the TXRF technique utilizes a monochromatic incident X-ray beam for excitation, diffraction X-rays appears at the same energy level to the excitation X-rays. Specifically, diffraction X-rays from the excitation X-rays of W-L$\beta$ (9.67 keV) interferes with measurement of transition metal fluorescence. Diffracted X-rays cause three problems for a TXRF measurement:

**A) Increase of background**

The excitation X-rays are diffracted by the SiC wafer and the diffracted X-rays appears as a high intensity spectrum to the detector. This gigantic peak increases background widely and makes the detection limit worse.

**B) Increase of escape peak intensity**

As the strong diffraction X-rays of W-L$\beta$ comes into the detector, the escape peak intensity increases proportionately to the intensity of the excitation X-rays. The escape peak of W-L$\beta$ overlaps with Cu-K$\alpha$, therefore, the measurement reliability of Cu contamination decreases.

**C) Increase of detector dead time**

Counting loss takes place when the strong diffracted X-rays of W-L$\beta$ hit the detector. Dead time increases due to correction of counting loss, and throughput falls.

Figures 4 and 5 show two spectra taken from same measurement position of the same wafer but at different incident direction of excitation X-rays. The red spectra are from an orientation of the wafer that maximizes the measurement of diffracted X-rays and the black spectra are based on an orientation that minimizes diffracted X-rays.

### 5.3. Extended diffraction X-ray avoiding mechanism of TXRF3760

The diffraction X-ray problem can be avoided by fixing the direction of the incident beam relative to the flat/notch orientation. For example, a 39° orientation relative to the flat/notch is the proper direction to avoid diffraction X-rays in case of a Si<100> cut wafer. For some time, Rigaku's Total Reflection XRF Analyzer, TXRF3760, has been equipped with a sample stage having an X$-$Y$-\theta$ positioning system to fix
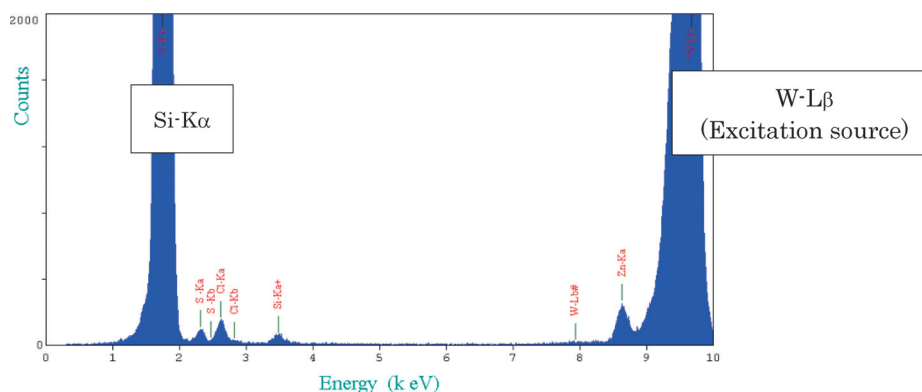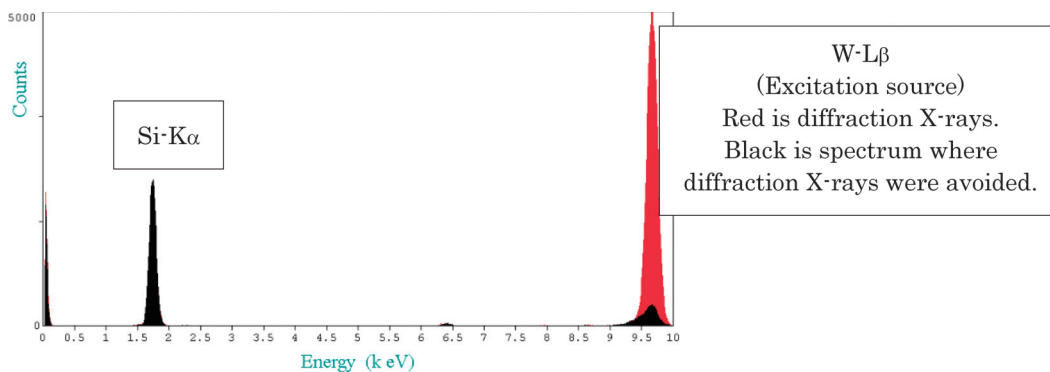


**Fig. 3.** TXRF spectra of SiC wafer.

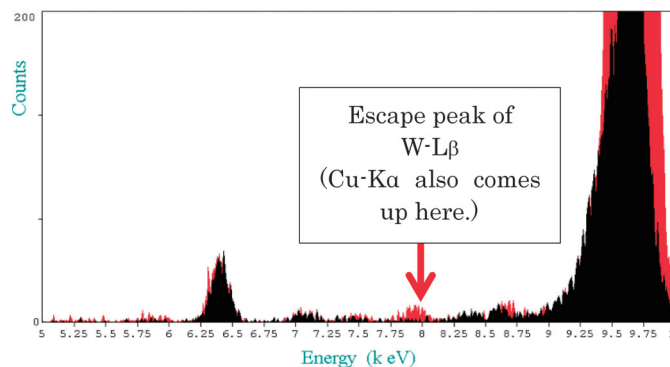**Fig. 4.** Spectra with maximized diffraction X-rays (red) and minimized diffraction X-rays (black).



**Fig. 5.** Spectra with maximized diffraction X-rays (red) and minimized diffraction X-rays (black). (Magnified escape peak)
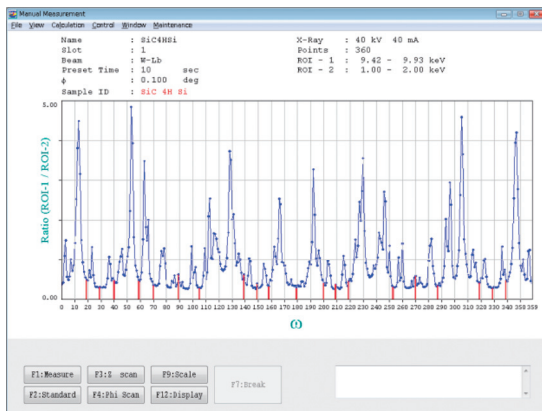


**Fig. 6.** W-L$\beta$ diffraction pattern from 4H-SiC.

One of the directions indicated with a red line can be used as an incident measurement angle. It is difficult to avoid diffraction X-rays as flat ranges are all narrow.
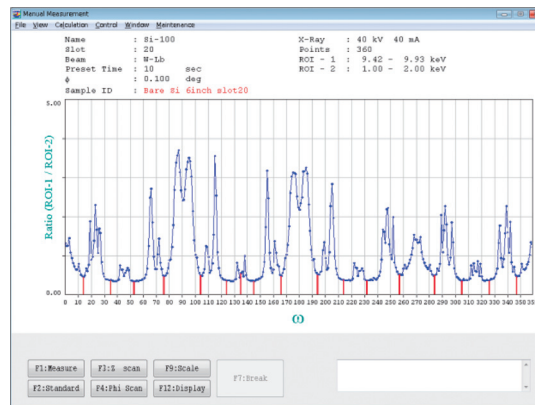


**Fig. 7.** W-L$\beta$ diffraction pattern from Si<100>.

It is easy to avoid diffraction X-rays as flat ranges are wide.

the measurement direction to avoid diffraction X-rays from Si wafers. Recently the TXRF3760 has been modified to cope with the more complicated diffraction from SiC wafers. The new mechanism automatically determines multiple directions of weak diffraction X-rays from the pattern of W-L$\beta$ diffraction X-rays measured beforehand. It then sets the best incident beam direction for avoiding diffraction X-rays when the measurement coordinates are setup. It is possible to cope with diffraction X-rays from all types of substrates

and wafers such as 4H-SiC, 6H-SiC, GaN, etc. Figure 6 shows the diffraction pattern of 4H-SiC. Directions with red lines are diffraction-free directions. Figure 7 is of Si<100> shown for comparison.

### 5.4. Mapping measurement

Currently 4 inch SiC wafers are most prevalent, but 6 inch wafers are starting to be used and the trend is in the direction of bigger wafers. Based on this trend, it becomes important to understand contamination distribution on SiC wafers in order to solve problems of the fabrication process. The TXRF3760 is equipped with

a high-speed entire wafer mapping function "Sweeping-TXRF" and it allows measurement of the whole area of a 6 inch wafer in approximately 30 minutes. Moreover, wafer edge measurements can also be made by a "ZEE-TXRF" function. Figures 8 and 9 show Sweeping-TXRF measurement results of a 4 inch SiC wafer.

## 6. Metal contamination analysis of GaN group power device by TXRF

When a GaN group wafer is measured with W-L$\beta$ excitation X-rays, a broad peak is observed at around
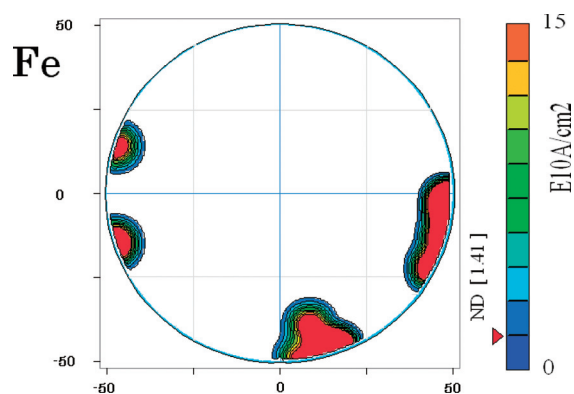


**Fig. 8.** SiC wafer before cleansing.
Fe contamination of more than $1 \times 10^{11}$ atoms/cm$^2$ is detected around the wafer edge.
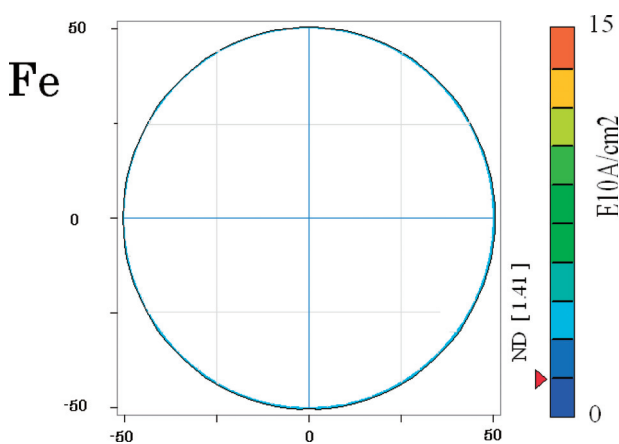


**Fig. 9.** SiC wafer after cleansing.

7.0 keV～8.7 keV (Fig. 10). This is due to Raman scattering that appears when the excitation X-ray energy is slightly lower than the absorption edge energy of the objective element. In the case of GaN, resonant X-ray Raman scattering is observed between W-L$\beta$ (9.67 keV) and Ga-K absorption edge (10.386 keV).

Due to the Raman scattering X-rays, backgrounds for Co, Ni, Cu, etc. for which their spectrum is near the Raman scattering goes up and the detection limit gets worse. Especially, as the peak of the Raman scattering is very close to the Cu-K$\alpha$, it is supposed that the detection limit of Cu on the GaN wafer is 1 significant figure worse than on a Si wafer. Because of this, it is necessary to pay special attention to TXRF analysis of GaN wafers.

However, the impact of Raman scattering on the analysis of the elements of importance like K, Ca, Ti, Cr, Mn, etc. is not serious and same level of contamination measurement as the ordinary Si wafer or the SiC wafer can be made.

Actual measurement example of GaN wafer and quantitative results are shown in Figs. 11 and Table 3.

As mentioned above, the detection limit of the contamination of elements ranging from alkali metals to Fe on the GaN wafer is similar to the case of a Si wafer, although Cu is worse due to the Raman scattering impact. Because of this performance and difficulty to apply chemical analysis represented by the VPD-ICP-MS, contamination evaluation of GaN by the TXRF is starting to be considered, and it has already been introduced to the contamination evaluation of substrate (SiC or sapphire), which grows GaN film.

## 7. The future

Currently, SiC power devices are mostly used for Schottky Barrier Diodes (SBD). However, the SiC market will become much larger in the future when SiC is used for MOS FET devices, where SiC will enable higher speed and finer control. As MOS FET is sensitive to surface metal contamination, evaluation of metal contamination will become much more important.

The movement from 4 inch SiC wafers to 6 inch SiC wafers is seen as a necessary trend in bringing the cost down. The requirement for mapping measurements
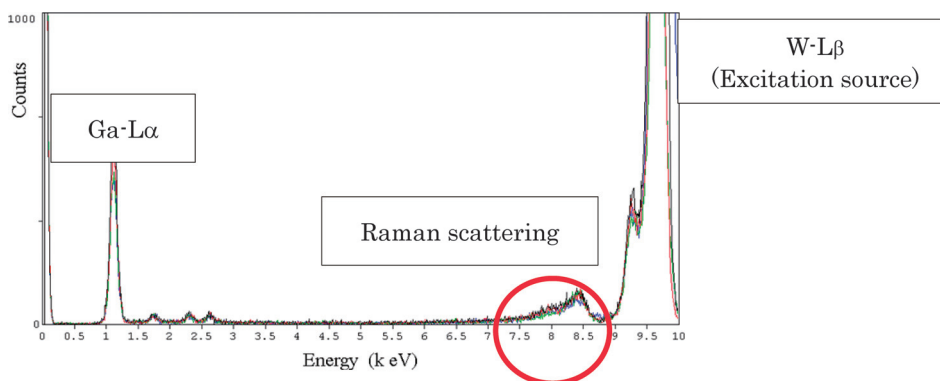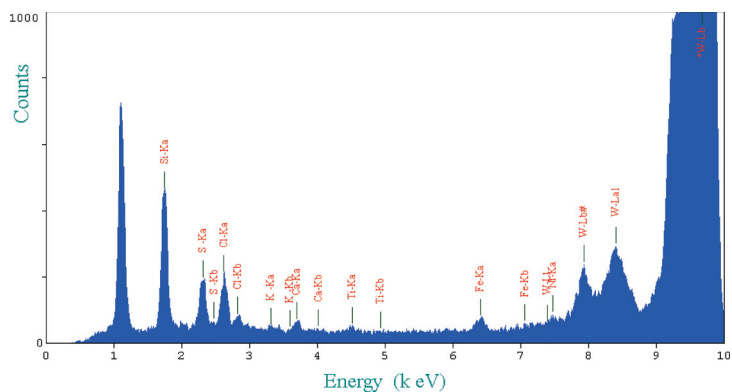


**Fig. 10.** Raman scattering.

**Fig. 11.** Spectra chart of GaN wafer.

**Table 3.** Quantitative result of elements detected.

| Element | Si | S | Cl | K | Ca | Ti | Fe | Ni |
|---|---|---|---|---|---|---|---|---|
| Unit | $10^{10}$ atoms/cm$^2$ | | | | | | | |
| Quantitative result | 9825.33 | 520.97 | 336.03 | 14.95 | 22.96 | 6.88 | 6.06 | 2.34 |

is proportional to size of the wafer, and thus there is an expectation that TXRF will continue to grow in popularity as a technique as more and more 6 inch wafer facilities are built.

Although it depends on the type of the device fabricated in future, it is reasonable to assume that the requirement for metal contamination control level of the SiC process will become $1 \times 10^9$ atoms/cm$^2$ or under, therefore, upgrading the instrument to higher sensitivity by incorporation of a VPD chemical pretreatment is likely to become necessary. Further development of the instrument is to be required in the future as the market grows and matures.