Application Note: 31052

The Karlsruhe Micro Nose, KAMINA

Key Words

- Surface Analysis
- ARXPS at Each Point on Linescan
- Automated Small Area Analysis
- Routine Measurement of Thin Films
- Measurement of Large Samples

The growing demand for inexpensive, space saving and intelligent gas sensor systems led to the development of an electronic nose at the Forschungszentrum Karlsruhe.[1] The areas of applications range from the monitoring of air quality, to medical breath analysis, to freshness control of food or the control of firing tools.

The central element of the KAMINA (Karlsruhe Micro Nose) is a gas sensor micro-array, which currently comprises of 38 sensor elements on an area of 4 x 8 mm^2. The array is based on an 150 nm thick SnO_2 or WO_3 layer, the electrical conductivity of which is highly sensitive to the ambient atmosphere. The basic structure of the micro-array is manufactured by RF-magnetron sputtering, applying a shadow masking technique. Parallel platinum strip electrodes for conductivity measurements are sputtered on top of the metal oxide, thus subdividing the latter into the initially identical sensor elements. In order to modify the gas response of individual sensor segments, a gas-permeable SiO_2 membrane, with a thickness variation of approximately 2 to 10 nm was deposited across the device using ion beam assisted deposition. The structure is illustrated in Figure 1.

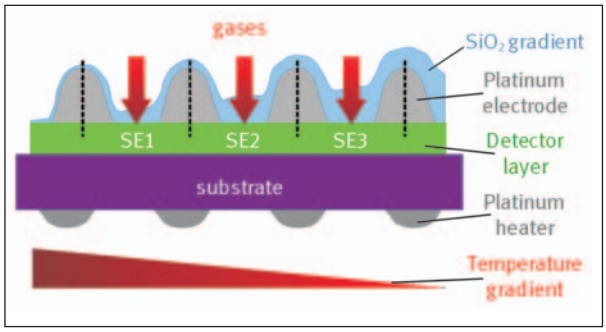

Figure 1: Schematic diagram of the gas sensor micro-array

The silicon dioxide gradient causes a differing gas response over each sensor segment. The ability of the detector to discriminate gases is a function of the steepness and the shape of the gradient membrane. Heater elements are attached to the reverse side of the substrate to raise the temperature to its operating range, 250-350 °C. The heaters can also be used to produce a temperature gradient of 50 °C across the device.

The way in which the thickness of the oxide layer varies with position over the device is crucial to its operation. The thickness is up to a few nanometers and so the measurements can be performed using angle resolved X-ray electron spectroscopy (ARXPS). Ideally, a thickness line scan experiment should be performed. This type of experiment is routine for the Thermo Scientific Theta Probe.

The whole sensor device was mounted on the standard sample holder and loaded into Theta Probe for analysis. Figure 2 shows the device in the analysis position.

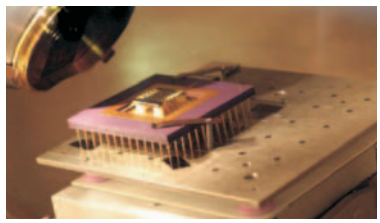

Figure 2: The packaged gas sensor micro-array in the Theta Probe analysis position

Optical images, below, show an overview of the sample (Figure 3) and a close-up view of the analysis area (Figure 4). These images were obtained using the zoom microscope and camera which is exactly aligned with the analysis position.

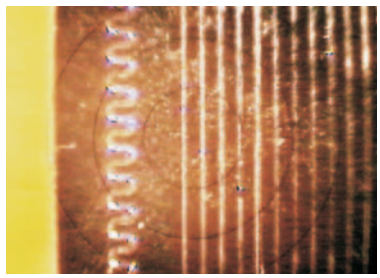

Figure 3: Low magnification view of the analysis position

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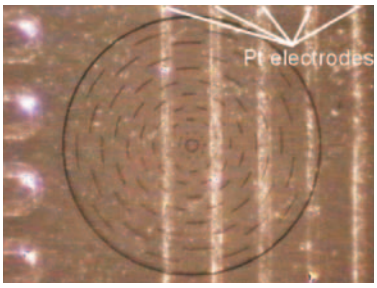

Figure 4: High magnification view

The graticule visible on these images is used for aligning a feature of interest with the source and analyzer focal points. The inner circle on this graticule is 50 µm diameter; the outer ring is 1 mm diameter.

The start and end points of the line scan were selected using the optical views. Two linescans were defined across the sample, one in which the analysis positions were above the Pt electrodes and one where the analysis positions were above the SnO_2 channels between the electrodes. Each linescan was 7 mm long. Data were collected from 38 points.

The microfocus monochromator was set to 50 µm to define the area from which data were collected.

Parallel angle resolved XPS spectra were collected from the Pt 4f, Si 2p, Sn 3d, C 1s and O 1s regions. The 60° angular range available from Theta Probe was divided into 16 channels.

Some of the materials in this specimen are insulating and so charge compensation was required in order to obtain satisfactory ARXPS data.

Using the *Avantage* data system, peak fitting was applied to each spectrum, at each angle and each position in the line scan. Figure 5 shows one of the O 1s spectra, fitted with two components, SiO_2 and SnO_2.

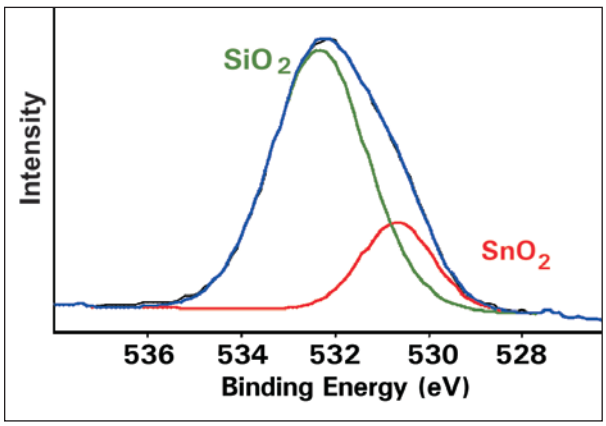

Figure 5: O 1s region of the XPS spectrum showing the peak fitted with two components

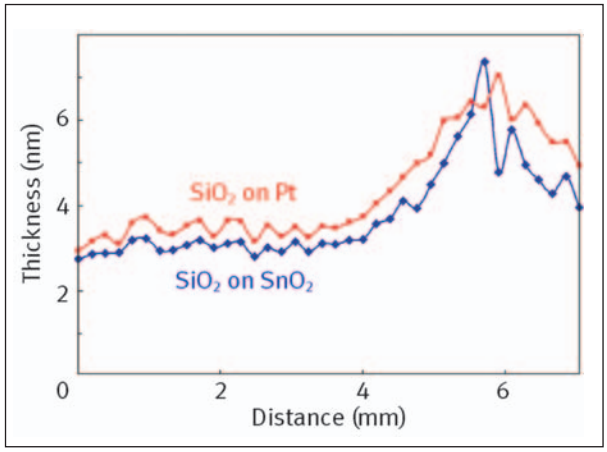

Figure 6: Silicon dioxide thickness as a function of distance across the micro-array

The usual method of the determination for the thickness of an overlayer film of one material on a substrate of another is based on the intensity ratio of the two XPS signals of the overlayer and the substrate, respectively, and on the assumption of an exponential depth distribution function for the signals. The fitted peaks were used to generate the thickness plots.

The angle resolved data were used to calculate the oxide thickness at each point on each line scan. The method used is described in the application note 31014.

The line scan shows that the thickness of the silicon dioxide varies between 3 and 7 nm across the sample.

Only Theta Probe can accomplish this type of measurement having its unique combination of attributes:

- Large sample handling
- Microfocusing X-ray source
- Parallel ARXPS facility
- Effective charge compensation

References:

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