# Using power coupling efficiency for ALD thin film oxides quantification

**Damian Frey**
Empa, Laboratory for Mechanics of Materials and Nanostructures, CH-3602 Thun Switzerland

## Motivation

Reference materials for GDOES with high oxygen concentrations providing high oxygen signals are hard to find. There is only one conductive oxide commercially available. In nonconductive samples the erosion rate, and therefore the signal intensity, is strongly dependent on the power coupling efficiency. A complete oxygen calibration curve can be obtained by changing only one parameter which influences the power coupling efficiency. In this study Al2O3 discs of varying thicknesses are used to quantify oxygen in ALD thin film oxides.

We deposit ALD thin films in our lab with a home built ALD reactor and wanted to know if it is possible to measure the composition of these layers with GDOES.

## Power coupling efficiency

In the RF-GDOES of nonconductive samples, a voltage drop develops inside the material. The voltage transfer coefficient is defined as the ratio between the peak voltage in front of the sample (facing the plasma) and the peak voltage applied to the back of the sample.

The power coupling efficiency therefore depends on the sample capacitance, which is defined by its relative permittivity, its surface and its thickness.

As ceramic discs with equal surface can easily be purchased, we decided to use a set of Al2O3 ceramic discs with varying thicknesses from 0.8 mm to 3.8 mm.

## Experimental

GDOES Device: **Horiba JY 5000 RF**
Plasma: Argon
Applied Real Power: 40W
Pressure: 650 Pa

Profilometer: AltiProbe optical profilometer

**Calibration Samples**:
Al2O3 ceramic discs
Several conductive samples (3 steel, 1 aluminum, 1 conductive oxide, 1 silicon wafer)

## Calibration

All samples were sputtered for the same amount of time, each crater depth was measured by profilometry

$x$ = *measured crater depth [um]*
$t$ = *sputtering time [s]*
$\rho$ = *sample density [g/cm$^3$]*
$s$ = *sputtering rate [g/(m$^2$*s)]*
$d$ = *crater diameter [m]*
$m$ = *mass erosion rate [ug/s]*
$c$ = *element concentration [wt%]*
$m_e$ = *element mass erosion rate [ug/s]*
$U$ = *element Signal [V]*

$$U \propto m_e$$

$$s = \frac{\rho * x}{t}$$

$$m = s * \frac{d^2 * \pi}{4}$$

$$m_e = m * 10^6 * \frac{c}{100}$$

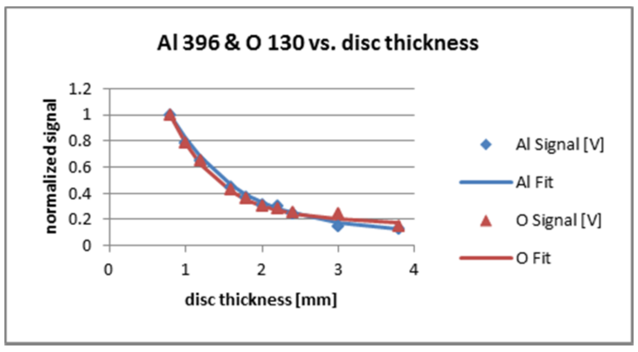

**Figure 1:** *Normalized signal intensities vs. sample thickness*

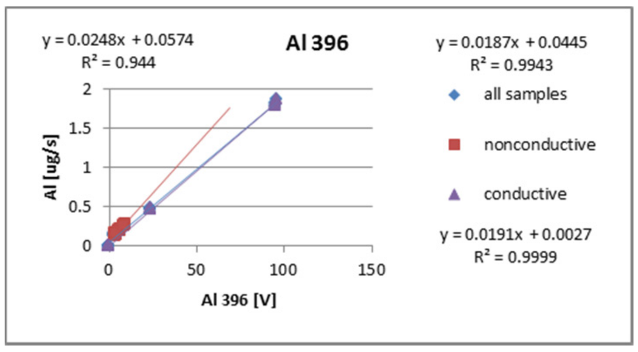

**Figure 2:** *Al 396 calibration lines (non- and conductive samples)*

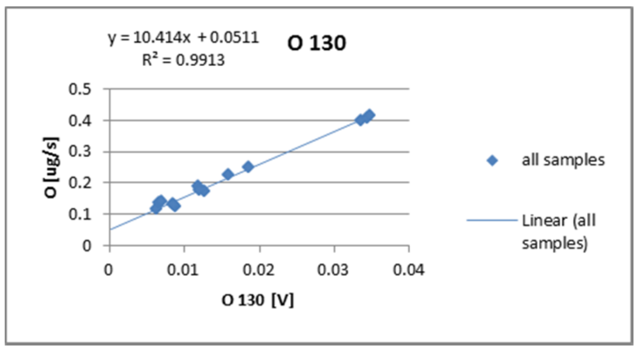

**Figure 3:** *O 130 calibration line*

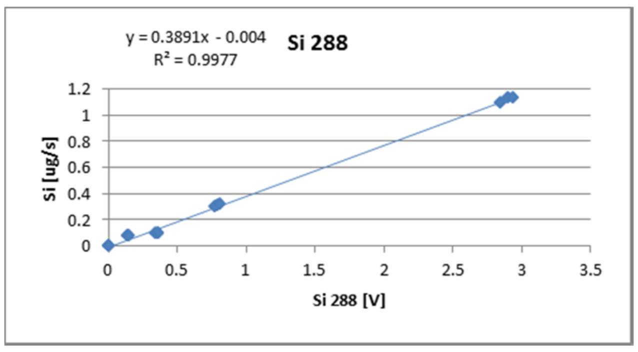

**Figure 4:** *Si 288 calibration line*

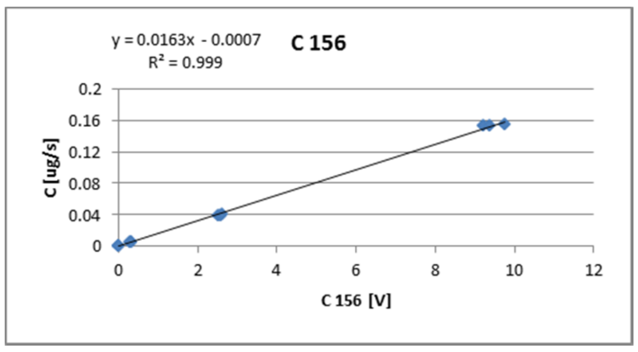

**Figure 5:** *C 156 calibration line*

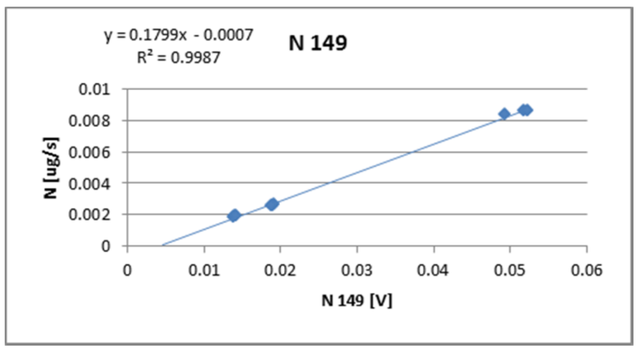

**Figure 6:** *N 149 calibration line*

## ALD thin film

Al calibration line of ceramic samples was used

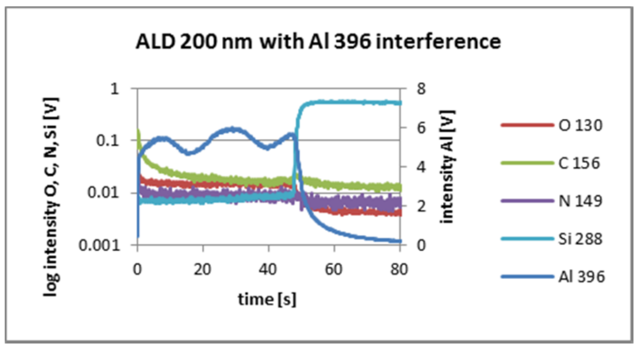

**Figure 7:** *Raw signals showing interference @ Al 396 due to transparent ALD layer, UV lines show no interference*

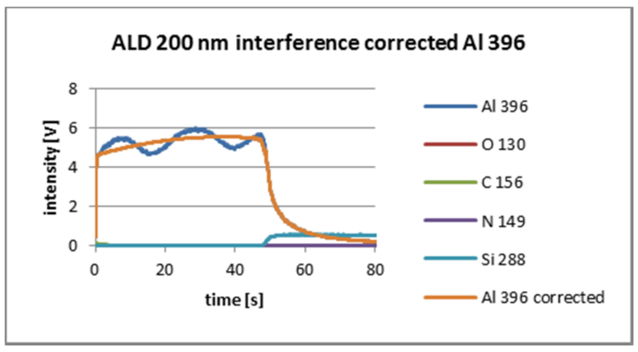

**Figure 8:** *Al 396 corrected for frequency of 0.04 Hz*

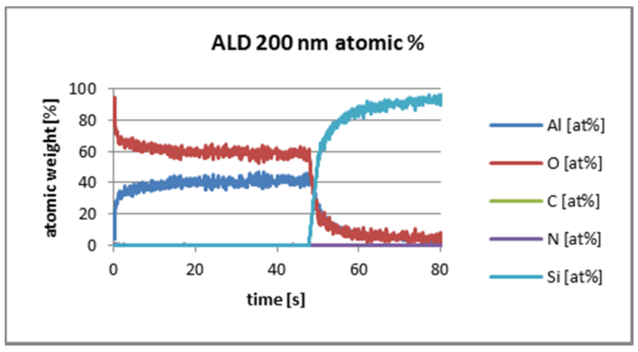

**Figure 9:** *ALD 200 nm layer atomic %*

*Average composition between 20 and 40 seconds:*

***Al: 40.2%; O: 59.7%; C: 0%; N: 0.1%; Si: 0%***

## Conclusions

Unfortunately the Aluminum calibration line of the nonconductive samples does not fit with the line obtained with conductive samples.

Using Al calibration of the noncoductive samples together with the interference correction provides very good results in quantification of the ALD layers.

Next approach would be to incorporate other ceramics like ZrO2 as well as thinner discs (down to 0.3 mm) to see if there is a similar effect.

### References:

Alwyn B. Anfone and R. Kenneth Marcus - Radio frequency glow discharge optical emission spectrometry (rf-GD-OES) analysis of solid glass samples
L. Therese, Z. Ghalem, P. Guillot, P. Belenguer - Improved voltage transfer coefficients for nonconductive materials in radiofrequency glow discharge optical emission spectrometry

### Contact:

**Damian Frey, Email: damian.frey@empa.ch, Phone: +41 58 765 62 87**