



## TXRF

# Control of Copper Contaminations through Analysis of Wipe Tissues with TXRF

Application Note # XRF 429

### Introduction

For the production of modern high performance circuits the combination of copper thin films and low capacitance materials is of particular importance. Copper has low electrical resistance and is consequently used in back-end-of-line (BEOL) processes for interconnect metallization. Any crosscontamination in FEOL (front-end-of-line) processes must be strictly controlled and avoided [1].

Wipe tests are the most common technique for contamination control of laboratory surfaces and tools. The wipes containing the contaminants are subsequently digested in acids and analysed using methods like inductively coupled plasma mass spectrometry (ICP-MS).

These methods are very costly and require additional efforts for sample pretreatment and calibration. In this study the more simple and less expensive technique total reflection x-ray fluorescence spectroscopy (TXRF) was used for contamination control.



**Figure 1**  
Wafer production

## Instrumentation

All measurements were performed using the bench top TXRF spectrometer S2 PICOFOX. This instrument is equipped with an air-cooled low power X-ray tube (Mo target), a multilayer monochromator with 80% reflectivity and the liquid nitrogen-free XFlash® Silicon Drift Detector (SDD) with an energy resolution of < 159 eV (Mn K $\alpha$ ).

## Qualitative wipe analysis

One of the major benefits of the TXRF technique is the versatility in terms of sample types. The S2 PICOFOX is not restricted to liquids, but allows the trace element analysis of solids, filters, suspensions, wipes and other kinds of samples.

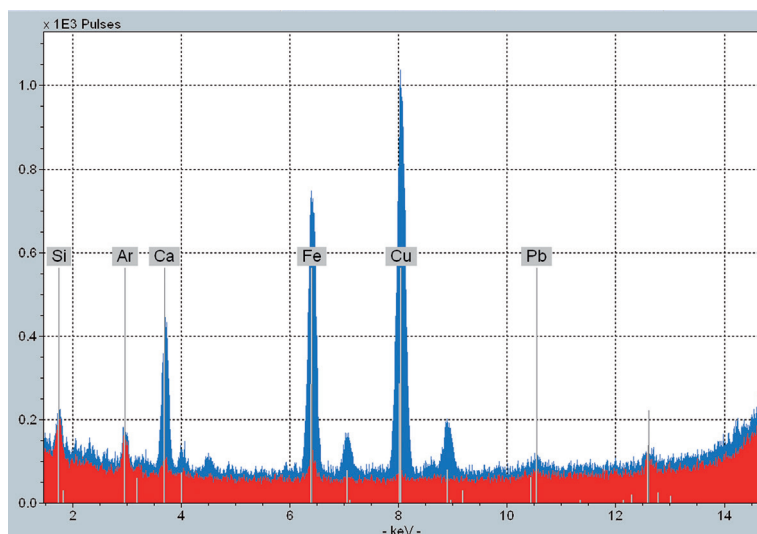
For the analysis of Cu-contaminations the direct measurement of wipes will save time and cost. The suitability of TXRF for this kind of application was tested as described here. A Kim wipe tissue was spiked with 2  $\mu$ l of a 1 g/l Cu standard solution, resulting in a total Cu amount of 2  $\mu$ g. A small part (about 0.5 x 0.5 cm) of the spiked tissue area was cut out and pre-*pared* onto a quartz glass carrier. The sample was fixed by a thin film of Silica grease. A blank tissue was prepared for comparison purposes.

The results of the exclusion test measurements are shown in Figure 1, where the red spectrum represents the blank sample and the blue spectrum the sample spiked with 2  $\mu$ g Cu. Obviously, the detection of Cu in this concentration range is possible.

Contaminations in this concentration range and most presumably far below can be easily controlled. The detection of additional elements like Ca, Fe and Pb are caused by contaminations during sample preparation, which was not performed in a high grade clean room laboratory.

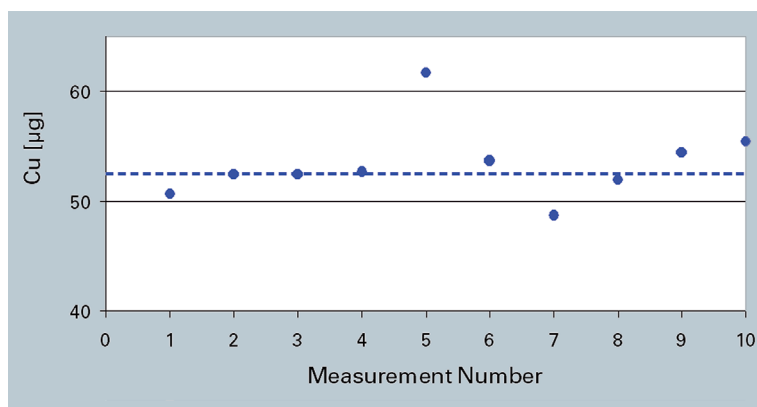
## Quantitative wipe analysis

The reproducibility and accuracy of a quantitative analysis was tested with 10 different tissues spiked with 50  $\mu$ l of a Cu standard



**Figure 1**

TXRF spectra of a blank (red line) and Cu-spiked (blue line) tissue



**Figure 2**

Results of quantitative wipe tissue analyses

solution (Merck, 1 g/l) and one blank tissue. In a thoroughly cleaned glass beaker the tissues were treated with 50 ml of ultrapure nitric acid for 5 minutes in an ultrasonic bath. 500  $\mu$ l of Ga solution (1 g/l) for internal standardisation and 500  $\mu$ l of Ge for control purposes were added before starting the ultrasonic treatment. 10  $\mu$ l of each extract were prepared as triplicates on a quartz glass carrier, dried in a desiccator and subsequently measured for 1000 seconds.

The blank corrected results for the Cu content are displayed in Figure 2. The mean value for Cu has been determined at 52.5  $\mu$ g, which

corresponds to a value of 5.0 % relative deviation from the nominal value. Measurement 5 was excluded from evaluation. The overestimation of this analysis was caused by an error during the addition of the internal standard. This was proven through the control element Ge, which showed a similar overestimation.

The relative standard deviation of the measurements, mainly influenced by sample preparation and instrument stability, is 6.6 %. The method limit of detection, calculated for a confidence level of 99.9 % with a student's t-value of 2.896, is 5.8 µg. Calculating the limit of detection through the concentration, peak and background intensities according to the equation given by Klockenkämper [2] results in a value of 0.2 µg.

## Conclusion

In this study it could be demonstrated that TXRF analysis using the S2 PICOFOX is a time and cost-efficient alternative to techniques like ICP-MS.

Main benefits are the easy sample screening before quantitative analysis and the comparably low analytical effort and cost. Furthermore, the simultaneous analysis of other contamination elements is also possible through TXRF spectroscopy.

## Bibliography

- [1] SINHA, D. & TAYLOR, P. (2002): Preventing Cu contamination from BEOL processing, A2C2 Magazine, 03/2002
- [2] KLOCKENKÄMPER, R. (1997): Total-Reflection X-Ray Fluorescence Analysis, John Wiley & Sons, 245 p

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