1 Publishable executive summary

emdpa
GD - TOF - MS

Material Science: ultra fast depth profiling of surfaces and interfaces

The final meeting of the STREP project EMDPA – www.emdpa.eu - took place at HORIBA Jobin Yvon premises in France on July 29, 2009 gathering 27 participants from all partners' sites and the EU Commission.

Consortium partners	Acronym	Country of origin
HORIBA Jobin Yvon	HJY	France
The University of Manchester	UoM	United Kingdom
National Institute of Lasers, Plasma and Radiation Physics	NILPRP	Romania

Consortium partners	Acronym	Country of origin
Institute for Analytical Sciences	ISAS	Germany
Swiss Federal Institute for Materials Science and Technology	EMPA	Switzerland
Laboratory of Plasma and Energy Conversion (formerly CPAT)	Laplace	France
TOFWERK AG	TW	Switzerland
University of Oviedo	UNIOVI	Spain
University of Catania	UNICT	Italy
ALMA Consulting Group	ALMA	France

The coordinator of EMDPA, Patrick Chapon, can be reached at patrick.chapon@horiba.com. The scientist in charge of the new instrumentation, Agnès Tempez, can be reached at agnes.tempez@horiba.com .

The acronym **EMDPA** stands for **E**lemental and **M**olecular **D**epth **P**rofiling **A**nalysis.

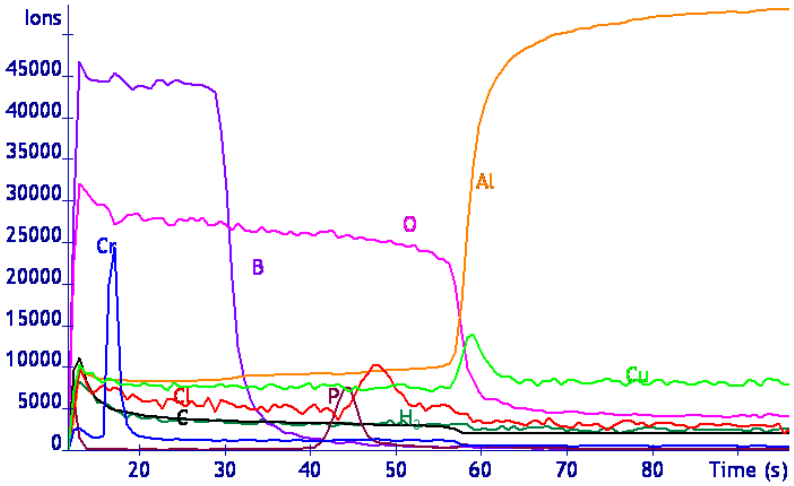
EMDPA is a STREP project that aims to input radically new knowledge into the development of a pulsed RF GD-TOF mass spectrometer dedicated to surface and depth profiling analysis (with nanometre (nm) depth resolution) of advanced materials with conductive and non-conductive thin layers.
EMDPA should also provide a "multi-dimensional" characterization, allowing direct simultaneous elemental and molecular measurement.

The key objectives of the project have been reached. That notably reflects in the number of presentations given at international conferences and local meetings as well as in the 30 peer reviewed papers published in strong cooperation between partners, ranging from plasma characterisation and surface/plasma interactions studies to the introduction of new concepts and advanced applications that have come along with the instrument development.

Publications demonstrate that the EMDPA consortium has successfully explored all aspects of the project (full references are available on our web site at www.emdpa.eu). The results have generated interest in the scientific community and a strong dissemination effort has been conducted. First domains of applications embrace corrosion science, solar cells manufacturing and molecular electronics. The instrument interests several labs involved in research, development, characterisation and measurement on thin/thick films.

Major achievements

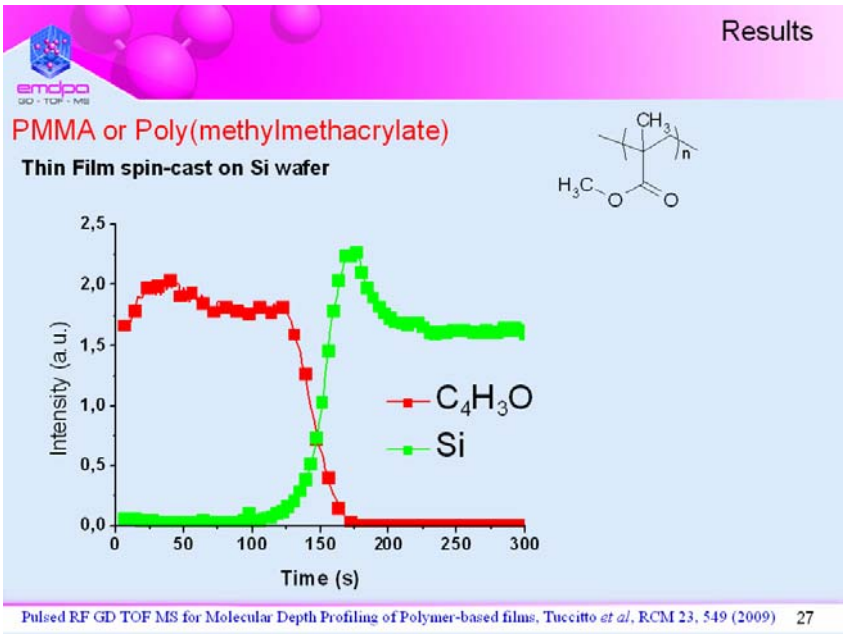
a) Depth profiling with nanometer depth resolution has been achieved on reference samples. An example is given below. It features a 2 nm Cr delta layer embedded in a 360 nm anodized Al on Al.



This result and several other published ones also show that the new instrument has a key benefit for depth profiling – the **speed of analysis** with measurements in seconds.

b) Molecular depth profiling, impossible to perform with OES, has been demonstrated with the new instrumentation.

By combining pulsed operation with soft conditions minimizing damage and timely gated detection in the afterglow regime, relevant molecular profiles have been obtained both from positive and negative ions. Two articles report this breakthrough:

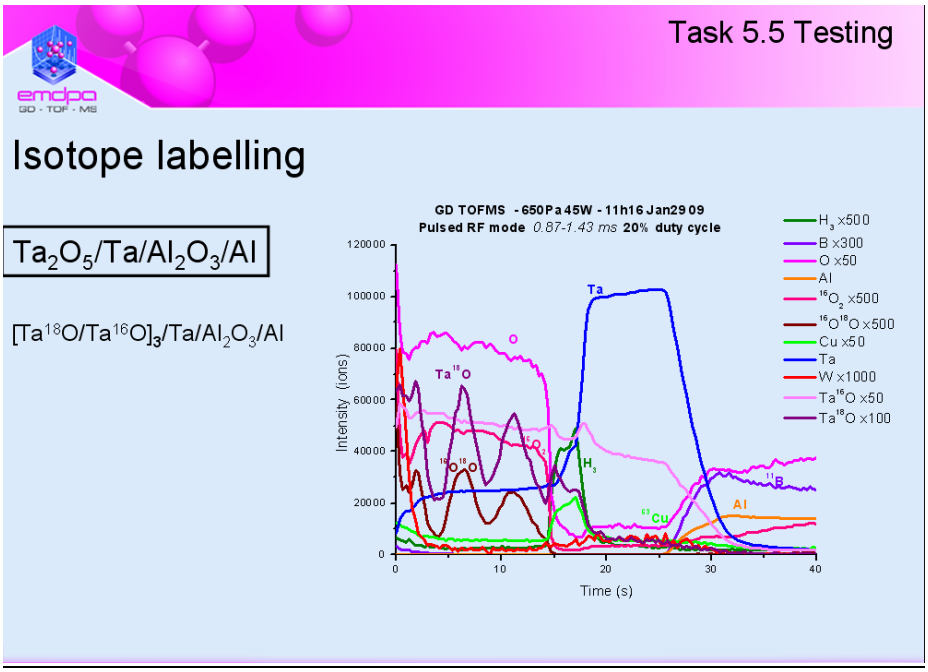


A paper entitled "*Pulsed radiofrequency glow discharge time-of-flight mass spectrometry for molecular depth profiling of polymer-based films*" coauthored by Nunzio Tuccitto, Lara Lobo, Agnès Tempez, Ivan Delfanti, Patrick Chapon, Stela Canulescu, Nerea Bordel, Johann Michler, Antonino Licciardello published in Rapid communications in Mass Spectrometry, **23**, *p 549-556, (2009).*

A paper entitled "*Potential analytical applications of negative ions from a pulsed radiofrequency glow discharge in argon"* coauthored by Stela Canulescu, James Whitby,

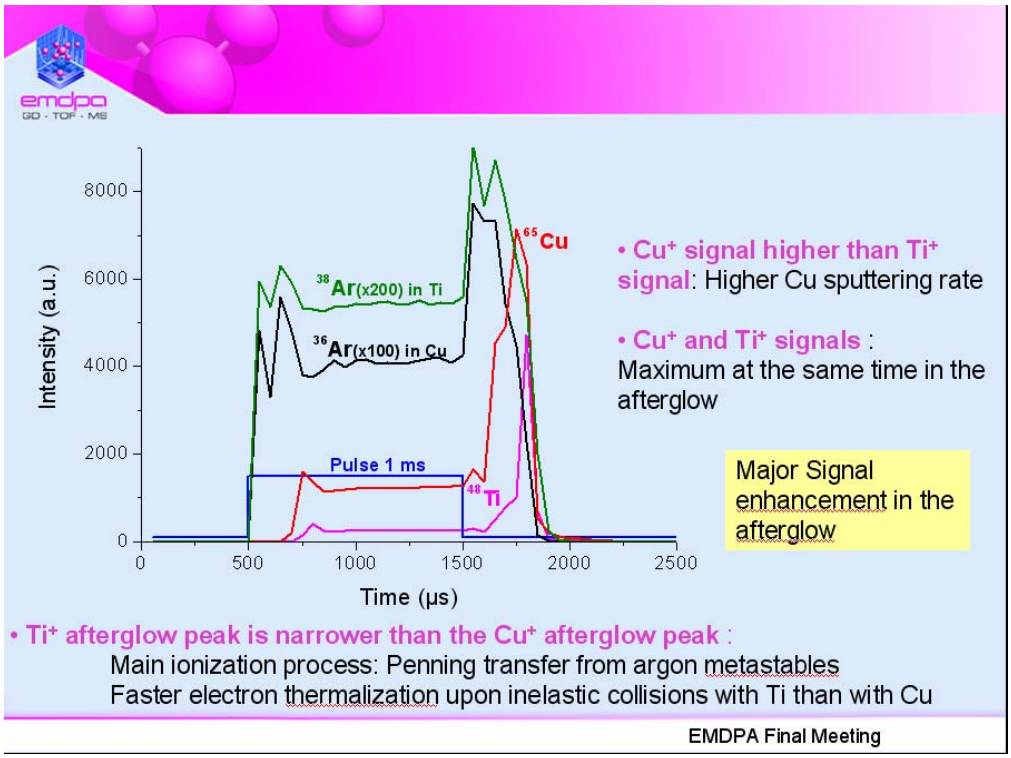
Katrin Fuhrer, Markus Hohl, Marc Gonin, Thomas Horvath and Johann Michler published in Journal of Analytical Atomic Spectrometry, ***24***, *p 178-180, (2009).*

c) Isotopic depth profiling has been shown of great interest for the understanding of transport mechanisms in corrosion studies notably.



Several papers on this major breakthrough have been written notably the one entitled: "*18O/16O isotopic separation in anodic tantala films by glow discharge time of flight mass spectrometry"* and coauthored by Agnès Tempez, Stela Canulescu, Igor Molchan, James Whitby, Lara lobo, Johann Michler, George Thompson, Nerea Bordel, Patrick Chapon, Peter Skeldon, Ivan Delfanti, Antonino Licciardello and Nunzio Tuccitto that has been accepted in Surface and Interface Analysis Journal.

d) Major scientific knowledge has been put in the understanding of the ionisation mechanisms in pulsed operation – specially the significant signal enhancement in the afterglow, of major relevance for analytical purposes when using a time gated detection as the TOFMS permits.



Several publications have been made on this topic including a review paper entitled *"pulsed glow discharge for analytical applications"* co-authored by Philippe Belenguer, Mihai Ganciu, Philippe Guillot and Thomas Nelis and published in Spectrochemica Acta part B 2009.

Research was also conducted on the use of magnetic fields for coupling efficiency and ion transport enhancement with **two patents** filed. Some of the analytical benefits are presented in a paper entitled "*Spectroscopic evaluation of a compact magnetically boosted radiofrequency glow discharge for time-of-flight mass spectrometry"* coauthored by P. Vega, J. Pisonero, N. Bordel, A. Tempez, M. Ganciu, A. Sanz-Medel published in Analytical and Bioanalytical Chemistry *(2009).*

Contamination issues have been studied and an innovative approach – called plasma cleaning – has been shown. Corresponding paper was published in JAAS in June 09 and EMDPA was asked to design the front cover for this specific edition.

e) Instrument

Two instruments with evolving configuration have been used during the project and a final prototype has been designed that includes the various developments studied during EMDPA – horizontal lamp design for easy handling and positioning, RF coupler, blanking –with the possibility to blank several masses simultaneously which is crucial for depth profiling – positive and negative ion modes and fully operational softwares.

This instrument is now at HJY for demonstration.

f) Conclusion

The EMDPA consortium was asked to contribute to a review MS handbook to be published by Wiley in 2010 with a chapter on thin and thick films analysis that will notably detail the major outputs of our project.

The new GD TOFMS gives the unique possibility to get the full mass spectrum at any depth in a depth profile analysis and any time within a pulse in case of pulsed operation offering a unique characterisation tool for advanced multilayer materials and achieves the objectives of the EMDPA project.