

Publishable Summary for 15SIP02 ISOChemDepth

An International Standard for Reliable Chemical Depth Profiling of Organic Materials

Overview

The aim of this project was to develop an international standard for measuring the average sputtering yield volume in sputter depth profiling of organic materials. This capitalised on the outputs of EMRP JRP NEW01 TReND project which developed the underpinning metrology of sputter depth profiling of organic materials. The average sputtering yield volume permits the depth of chemical features observed in depth profiles to be measured. The international standard, produced through ISO/TC201, encapsulated the best practice and is intended to help ensure that reported yields are consistent, clear and transferrable between different instruments and techniques.

Need

The primary supporter, Hidehiko Nonaka, chair of ISO TC201 (surface chemical analysis) requested the project maximise the impact of NEW01 TReND by writing and publishing an international standard for determining the average sputtering yield volume in sputter depth profiling of organic materials using secondary ion mass spectrometry. These needs directly relate to the outputs of the JRP NEW01. The primary supporter and the project partners recognized that being able to measure the distribution of chemical components in the near-surface region of materials is essential to understanding and optimising material performance and manufacture. Sputter depth profiling provides this and is routinely used for the quantitative analysis of semiconductor materials. However, in organic materials different analytical challenges exist. Following industry consultation, a high-priority on the action plan for ISO/TC 201 was the development of an international standard for organic sputter depth profiling using cluster ion beams so that the wider community in industry, academia and manufacturers can fully benefit from recent developments and uptake in ion source instrumentation in combination with X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectrometry (SIMS). Currently, approximately 60 % of new XPS and SIMS instruments are equipped with these and many existing instruments have had these sources installed. It is estimated that there are more than 200 instruments worldwide with GCIB and approximately 100 new instruments are sold every year. The main applications are polymers and coatings, including packaging materials, photoresists, drug delivery systems, organic light emitting diodes and organic photovoltaics. Other significant applications are in the medical and pharmaceutical sector where the method provides, for example, a direct visualisation of the location of drug molecules and metabolites within tissue.

XPS and SIMS depth profiling using argon cluster sputtering have developed into routine analytical services. Subsequently, there is a strong requirement for reference materials and documentary standards through which analytical laboratories can demonstrate conformance and reliability. The purpose of this SIP was to provide industry with a documentary standard which enables them to state the uncertainty in their measured layer thicknesses or the depth of a feature in a sample.

Objectives

The objective of the SIP was to maximise the impact of NEW01 TReND specifically;

1. To publish an international standard for determining the average sputtering yield volume in sputter depth profiling of organic materials using secondary ion mass spectrometry.

Results

Objective 1: To publish an international standard for determining the average sputtering yield volume in sputter depth profiling of organic materials using secondary ion mass spectrometry

This objective was fully achieved. ISO 22415 Surface chemical analysis -- Secondary ion mass spectrometry -- Method for determining yield volume in argon cluster sputter depth profiling of organic materials was published in May 2019. The activities and results of this project are split into three main areas. These and the details of the results under each one is given below.

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Publishable Summary

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The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

1. Write a draft international standard for determining the average sputtering yield volume in sputter depth profiling of organic materials using secondary ion mass spectrometry

A draft international standard for determining the average sputtering yield volume in sputter depth profiling of organic materials using secondary ion mass spectrometry (SIMS) has been written. This was reviewed and improved on by the experts and National Physical Laboratory (NPL). It was submitted for a new work item ballot and approved. It was updated and submitted to a committee ballot and again passed with no objections. The document then passed two final ballots, draft international ballot and final draft international ballot with minor comments and updates at each ballot stage. At the ballot stages, the document was subject to unexpected delays by ISO in launching the ballots. The document was then typeset by ISO central secretariat before being proofread by the project team and published on 10 May 2019.

2. Prepare and submit a New Work Item proposal for ballot in ISO/TC 201 and engage with the committee to bring the standard towards publication

The documentation for the new work item proposal – a draft document and an ISO new work proposal form were written. This was submitted to the ISO TC201/SC6 (SIMS) secretary on 19 December 2016 for a new work item ballot. The ballot was launched on 12 January and closed on 6 April. It was approved with helpful comments from the experts. The document was updated based on these comments. The committee experts then improved the document. It was then submitted for a new work item ballot and approved with comments again received from the experts. It was circulated amongst the experts and the draft updated. When the experts were satisfied, it was submitted for a committee ballot and again passed with no objections. The document was again updated by the project team in consultation with the international experts from organisations such as NIST (USA), Pacific Northwest National Laboratory (USA), NIM (China) and AIST (Japan). The document then underwent two more ballots. At both of these ballots the committee members commented on the document and improved it.

3. Engage with international experts and the end user community throughout the process to ensure maximum impact and support.

The project team has attended four international ISO TC 201 meetings as part of the project. At the first meeting, in Autumn 2016, the international experts were informed about the aims of the 15SIP02, ISOChemDepth project. In September 2017, the document was again discussed, firstly at the annual meeting of ISO TC201/SC6 in Krakow with 18 SIMS experts from 7 countries. The same year, the plenary meeting on surface chemical analysis standards was hosted by NPL in Teddington on 21-23 September 2017. This 3-day meeting was attended by 80 delegates from 10 countries and included a session on the project. Finally, in October 2018 international experts and the user community commented on the document and the project in general at the annual TC201 meeting in Mexico.

In order to increase impact four presentations on the project have been given at international conferences and meetings involving the end user community. In September 2017, at the annual conference on secondary ion mass spectroscopy, an invited talk on SIMS and how to collect useful data was given by a project member. An invited talk on organic depth profiling and standards was given at the large AVS symposium in the USA in October 2017. At the international conference on surfaces, materials and vacuum in September 2018, two talks on international standardisation were given by project members.

In addition, throughout the lifetime of this project, the project team engaged with additional experts and end-users throughout the development of the standard via discussions and electronic communication.

In summary, the objective was fully achieved.

Impact

Highlights of Dissemination activities:

An ISO international standard has been published, 4 international talks have been given and a summary peer-reviewed publication is published.

In detail, the international standard will be used by the primary supporter, ISO TC 201 (international standards committee on surface chemical analysis) to provide surface analysts with a standard method for measuring the sputtering yield volume in order to determine the depth of interfaces. The ISO standard will further serve

the wider SIMS and surface analysis community as guidance for reliable sputtering depth profiling of organic materials for which there is currently no standard available. This will have economic impact in areas such as organic electronics, photovoltaics, medical devices and advanced manufacturing where the distribution of chemical components in the near-surface region affect the performance of materials and devices. This will filter down and gain wider community benefit in social, environmental and healthcare areas. Two examples of this are, quality of life will be improved via new medical devices and environmental benefits will happen via improved solar panels. Thus, the sputter depth profiling standard will improve the analytical measurement reliability that underpins and accelerates innovation across these industries, contributing to economic growth and development of environmentally friendly and low-cost technologies.

Consultations with industrial laboratories and commercial analytical service providers indicated that there will be a strong uptake of this standard. For example, the analytical laboratories of major industrial concerns have shown a keen interest. The standard will enable them to provide meaningful data with a depth scale, rather than an 'ion dose' scale. This is important for the understanding of their analyses, and ultimately their products. Currently, sputtering yield volumes for a small number of materials are available from a range of institutions, measured through a number of different methods, the reliability of which is unclear. Furthermore, the protocols used to measure the depth of interfaces require clarification and the standard will contain guidance on the best methods to use.

The ISO TC 201 plenary and sub-committee meetings were attended in September 2016 and the experts were informed and discussed the aims of the 15SIP02, ISOChemDepth project. The aims and outline of the draft standard were presented and were well received by the members which included NMIs, instrument vendors, measurement service providers and industry stakeholders. Following this a draft standard was written and submitted to ISO TC 201 for a new work item ballot. This was balloted amongst 9 participating countries and 10 observing countries and their experts. The project was successfully approved as a new work item with extremely helpful comments received at this ballot stage. The document was again discussed firstly at the annual meeting of ISO TC 201/SC6 (secondary ion mass spectroscopy) held in Krakow on 10 September with 18 SIMS experts from 7 countries, including the UK, Germany, USA, China, Korea and Japan. A second meeting of ISO TC 201 on surface chemical analysis standards was hosted by NPL in Teddington on 21-23 September 2017. This 3-day meeting was attended by 80 delegates from China, Germany, Italy, Japan, Korea, Mexico, Sweden, Switzerland, UK and USA including the primary supporter and 20 delegates from the UK. Finally, in October 2018 ballot international experts and the user community commented on the document and the project in general at the annual TC 201 meeting in Mexico.

An invited talk on 'Quantitative Organic Depth Profiling and 3D Imaging using Secondary Ion Mass Spectrometry' was given at the large AVS symposium in the USA in October 2017. In addition, a presentation was given as part of the 21st International conference on SIMS, Krakow, Poland, 10-15 Sept 2017. At the international conference on surfaces, materials and vacuum in September 2018, two talks on international standardisation were given by project members. The project team also engaged with additional experts and end-users throughout the development of the standard. To increase awareness of the international standard, a peer-reviewed summary article on the standard was published in the journal Surface and Interface Analysis.

In summary, the main output of this project is the international standard, which will become the reference point for reliable sputtering depth profiling of organic materials in areas such as organic electronics, medical devices and advanced manufacturing helping to improve products in these key industrial sectors and accelerate innovation.

List of publications

1. A G Shard, R Havelund, M P Seah, C A Clifford, Summary of ISO/TC 201 Standard: ISO 22415—Surface chemical analysis—Secondary ion mass spectrometry—Method for determining yield volume in argon cluster sputter depth profiling of organic materials, *Surface and Interface Analysis*. [Online]. Available: <https://doi.org/10.1002/sia.6686>

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Project website address: N/A				
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