Publishable Summary for 17SIP07 Adlab-XMet
Advancing laboratory based X-ray metrology techniques

Overview

This project aims at the dissemination and exploitation of the project results from EMRP project NEW01 TReND. In particular, the produced and characterised organic and inorganic material systems (e.g. atomic layer depositions of Al2O3 or ion implants) and the knowledge achieved on reliable TXRF (Total reflection X Ray Fluorescence analysis), GIXRF (Grazing Incidence X-Ray Fluorescence analysis) and XRR (X-Ray Reflectometry) based characterisation of such materials including a novel fast and automatable alignment strategy will be applied to the laboratory tools of Bruker Nano GmbH, which acts as the primary supporter of this project.

Need

Many applications of TXRF (Total reflection X-Ray Fluorescence analysis) and GIXRF (Grazing Incidence X-Ray Fluorescence analysis) are used for contamination analysis, elemental depth profiling, nanoparticle and thin layer characterisation, as well as, related existing quantification. Consequently, well characterised and appropriate reference materials are required for calibration to ensure that the results are reliable and quantitative. Specific instrumental parameters such as the effective solid angle of detection are crucial when extending towards GIXRF, thus enabling depth-resolved elemental analysis. Although, calibration standards such as dried droplet samples exist, these standards often suffer from inhomogeneous and partially unknown elemental distributions which severely degrade the reliability of the quantification. For the continuation towards GIXRF analysis, the droplet standards are no longer suitable as their inhomogeneities may result in a non-interpretable GIXRF fluorescence profile. Therefore, there is a clear need for well-defined calibration samples which do not suffer from such drawbacks. The Primary supporter, Bruker, intends to use parts of the well-characterised organic and inorganic material systems from the EMRP NEW01 TReND project; in order to gain a better understanding of laboratory capabilities; based on TXRF, GIXRF and XRR instruments. This is achieved by comparing experimental data from these instruments with the synchrotron radiation-based data obtained from the TReND project, allowing for a reliable determination of relevant parameters of the laboratory tools. In-depth knowledge on such instrumental parameters is a key aspect for the modelling of experimental data that enables a significantly widened applicability of the laboratory-based instruments.

In addition, further dissemination of the more conceptual and theoretical findings from the TReND project activities with respect to TXRF, GIXRF and XRR will be crucial for laboratory tools. This includes the transfer and adoption of an advanced and fast alignment procedure, which will provide reliable control of relevant instrumental parameters of the primary supporter's lab tools.

Objectives

The overall aim of the project is the dissemination and exploitation of the project results from EMRP project NEW01 TReND. The specific objectives of the project are the following:

- To provide novel and more reliable calibration methods for laboratory TXRF and GIXRF instruments, which will be established with the use of the well characterised (with respect to their lateral homogeneity and their in depth elemental distributions) calibration samples from the EMRP project NEW01 TReND.
- To transfer and disseminate the developed alignment and optimisation procedures to the laboratory X Ray instrumentation for TXRF, GIXRF and XRR (X-Ray Reflectometry) in order to eventually make this knowledge available to users of these equipment.

Results

Objective 1: Novel and more reliable calibration methods for laboratory TXRF and GIXRF instruments
With support from PTB, the Primary Supporter has been using the well characterised material systems developed in the EMRP project NEW01 TreND, to perform TXRF, GIXRF and XRR based experiments on laboratory instruments. During these experiments the elemental analysis will enable the transfer of calibration and determination of relevant instrumental parameters; from PTB's unique SI traceable and reference free X-Ray spectrometry (XRS) instrumentation; to the laboratory instruments that are used by Bruker.

Additionally, a set of well characterised samples was distributed to Bruker and the transfer of the calibration and determination of the solid angle of detection was successfully performed. A publication showcasing the methodology and the obtained results including a demonstration of thereby enabled quantitative depth profiling is currently being peer-reviewed.

Objective 2: transfer and disseminate the developed alignment and optimisation procedures to the laboratory X-Ray instrumentation

The transfer of the alignment strategy from the development stage in EMRP NEW01 TReND, towards being an automated tool for the laboratory, based instruments for TXRF, GIXRF and XRR is expected to provide a faster and more reproducible sample alignment for many types of samples (e.g. wafers, glass slides or quartz carriers) and will thus decrease the necessary time to data. The status of the current sample alignment strategy has been evaluated and the necessary changes have been implemented and tested.

Impact

A presentation was given at the European PRORA Conference in November 2019 and two internal presentations were given in company seminars that were held at BRU and Bruker AXS. The project has prepared three papers related to: GIXRF-XRR, the determination of the solid angle of detection and the project results that were applied to industrially relevant multilayer samples (one paper has been published and two papers are undergoing peer-review). Furthermore, a training session on GIXRF basics was provided to new PhD students in February 2020.

The impact of this proposal for the primary supporter, as a producer of advanced XRF metrology tools, will be direct and significant. The planned transfer of calibration procedures from PTB's SI traceable and reference free XRS instrumentation to their own laboratory instruments will establish possibilities for high- accuracy quantitative measurements, e.g. for nanolayer or sub-monolayer elemental depositions. The additional determination of important characteristics such as the convolution of solid angle of detection and the beam profile of the excitation source, will considerably improve the primary supporter's understanding of these parameters. Both outputs will be transferred into a guidance document for the assembly and pre-sale characterisation of new TXRF, GIXRF and XRR instruments. The results of the proposed activities will better position Bruker's instruments in the competitive market and disseminate further the key GIXRF related outputs of EMRP NEW01 TReND project into many scientific and industrial communities worldwide. Moreover, the technology transfer from the project will be a benefit to the industrial (e.g. other Bruker subsidiaries) and non-NMI participants and stakeholders (e.g. TU Berlin and Helmholtz Centre Berlin) of current and future EMPIR JRPs (e.g.16ENG03 HyMet and 16ENV07 AEROMET) in which both synchrotron radiation based as well as laboratory based applications of TXRF, GIXRF and XRR methodologies are being investigated for their metrological capabilities.

The X-ray metrology techniques addressed in the project are very relevant for various applications in the semiconductor industry, material sciences, environmental research and health related research. The reliability of quantitative analyses depends on the calibration procedure which may suffer from inappropriate calibration specimens such as no homogeneously dried droplet standards. An improvement of the calibration strategies is already a field of several research activities. In addition, the transfer and application of the alignment procedures as well as the determination of the instrumental parameters will significantly broaden the scope of application fields for the TXRF and GIXRF techniques. As these are key parameters for performing a reliable experiment and allow for a meaningful modelling, this proposal paves the way for a successful application of GIXRF-based depth profiling for analytical nanometrology offering potential uptake by the ISO TC201 (surface analysis) SC10 committee on XRR and XRF. This will increase the characterisation possibilities from a simple quantification of the amount of substance to a sensitive and quantitative characterisation of the elemental distribution, which is crucial for many applications, e.g. dopant depth profiling.

List of publications

1. P. Hönicke, B. Detlefs, E. Nolot, Y. Kayser, U. Mühle, B. Pollakowski, B. Beckhoff, Reference-free grazing incidence X-ray fluorescence and reflectometry as a methodology for independent validation of X-ray reflectometry on ultrathin layer stacks and a depth-dependent characterization, J. Vac. Sci. Technol. A (2019) 37, 041502 https://arxiv.org/abs/1903.01196 , https://doi.org/10.1116/1.5094891

Project start date and duration:	1st June 2018, 36 months	
Coordinator: Philipp Hönicke, PTB Tel: +49 30 3481 7174 E-mail: Philipp.hoenicke@ptb.de Project website address: https://www.researchgate.net/project/Advancing-laboratory-based-X-ray-metrology-techniques		
Primary Supporter: Ullrich Waldschläger, Bruker		
Internal Funded Partners: 1. PTB, Germany	External Funded Partners:	Unfunded Partners: 2. Bruker, Germany