CCQM WG on Surface Analysis (SAWG)

Report on the activities April 2019 – April 2020

# General

Toshiyuki Fujimoto was appointed as the new chair of SAWG at the 25th meeting of CCQM for the period of 2019 to 2023. Alex Shard was appointed as the deputy chair. Alex Shard has agreed to become the new KCWG representative of SAWG.

Instead of the spring meetings which were cancelled as a consequence of the spread of COVID-19 around the world, a 3 hour virtual meeting was held on June 5th 2020 with the assistance of BIPM. 25 people from 19 institutes participated, which is highest record of participation.

At the beginning of the virtual meeting, Li-Lin Tay was appointed as the rapporteur of the meeting.

The minutes of last SAWG meeting, held on April 8/9 2019 BIPM, Sèvres, Paris had previously been circulated as SAWG\_19-14 were approved by the participants without any correction.

# Progress of P-190, Thickness Measurement of HfO2 films

Kyung Joong Kim, PL presented the final report on the pilot study P-190. Objective of P-190 is to measure the thickness of HfO2 films in 6 different samples of HfO2 film (1-4nm)/SiO2(2 nm)/Si(100). There are 11 participating institutions. Measurement methods used are: XPS, XRR, TEM, MEIS, SE, XRF and RBS. The PL proposed to use a Mutual Calibration using the averaged XPS and XRR data to determine the reference thicknesses. There was considerable discussion on the reliable measurement ranges for XPS and XRR, respectively. The PL will circulate a revised draft based on the discussion. Participants in P - 190 agreed to hold another web meeting to finalize P-190 after the circulation of the revised draft.

# Report of the UNIMM and Tubitak Bilateral BET comparison results and proposal for a new KC

A key comparison for the amount of gas adsorption on surfaces at specific conditions and the specific surface area estimated using the BET method had been carried out for nonporous and mesoporous materials as K-153 and K-136. Comparative gas adsorption measurements for microporous materials are also required. The result of a bilateral comparison for microporous materials by UNIIM-Tubitak was reported and a new KC was proposed by Egor Sobina. The material to be used for the comparison is a zeolite. The measurands proposed in the new KC are specific adsorption of Ar and specific surface area. The protocol and timeline of the KC were also presented. Currently 5 Potential participants with 2 -3 potential participants to be confirmed. All members agree to initiate a new KC with microporous materials.

# Report from Task Group on Quantitative Measurements with Raman

Li-Lin Tay presented the T&R of Raman task group. The T&R was presented to the 25th CCQM plenary. SAWG expect to receive a feed back from the CCQM president to continue the activities formally.

Steps towards pilot study on the traceable quantitative Raman measurements with mixed polymer films were also presented. Tay presented updated draft plan using two miscible polymers, polystyrene (PS) and polyphenylene oxide (PPO). The suggested measurand is mass fraction of the polymers. Samples still need further improvements before launching a pilot study. The protocol for the comparison is under development at NRC. After the sample and the draft protocol are ready, Li-Lin will invite a partner NMI to confirm the samples and protocols are suitable for a pilot study.

Li-Lin also provided a brief update on three other Raman interlaboratory comparison that is currently underway in VAMAS. 1. Structural characterization of CVD-grown graphene: coverage on substrate, number of layers and level of disorder project under VAMAS TWA-41 is co-led by NPL (Andrew Pollard) and NIM (Lingling Ren). All measurements are completed and data from 16 NMIs submitted to NPL for analysis. 2. The second VAMAS project is on the measurement of lateral resolution of Raman microscopy with InAs nanowire. The project runs under TWA-42 and is led by Dr. Sebastian Wood (NPL) and launched in Jan. 2020. There are 15 participants signed up for this interlaboratory comparision. 3. The third VAMAS project is led by Dr. Andrea Rossi (INRIM) on the Raman spectroscopy of TiO2 nanoparticle mixture. Dr. Rossi gave a brief update on the project and its tentative start date. The project will use Raman spectroscopy and chemometric analysis to quantify and classify different phases of TiO2 binary mixture.

# Planning: Pilot study for Amount of material in a buried organic layer

Paulina Rakowska provided a current status of the proposal. The sample structure being considered for the study is a 3-layered films with an Ir(ppy)2(acac) layer sandwiched between two Irganox 1010\* layers on Si substrate. The measurand is the amount of substance in an Ir(ppy)2(acac) layer in an organic layer stack on a Si substrate, expressed as the thickness of Ir(ppy)2(acac). Several calibration samples and a test sample are being prepared by NPL. The study will benchmark the current techniques in the analysis of organic layers and as a step towards establishing traceability for chemical depth profiling of organic layers by using surface analysis methods. A formal proposal to launch the pilot study will be submitted in due course.

\*Irganox 1010: Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate)

# Planning: Pilot study on “Homogeneous surface composition of an ionic liquid

Alex Shard and Joerg Radnik presented a draft plan and preliminary results. It has a focus on an analysis of the “Homogeneous surface composition of an ionic liquid”.

There is a strong need to underpin measurement services performed by SAWG members. A CMC for “equivalent homogenous composition” would be very useful in this regard. For this purpose the sample for a KC should have known composition, be homogeneous with no surface layers and have very low vapor pressure to be compatible to UHV. Some ionic liquids meet these requirements. A protocol is under development by BAM and NPL. BAM and NPL is will perform a bilateral study and the results will be reported at the SAWG meeting in 2021 Spring.

# Next meeting

In order to maintain a high level of activity, the chairman proposed to hold an interim SAWG meeting in autumn. The meeting will be held in virtual space. The proposal was supported unanimously.